



L'urine humaine en agriculture : des filières variées pour contribuer à une fertilisation azotée durable

Tristan Martin

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Tristan Martin. L'urine humaine en agriculture : des filières variées pour contribuer à une fertilisation azotée durable. Alimentation et Nutrition. Université Paris-Saclay, 2020. Français. NNT : 2020UPASB026 . tel-03350482

HAL Id: tel-03350482

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L'urine humaine en agriculture : des filières variées pour contribuer à une fertilisation azotée durable

Thèse de doctorat de l'université Paris-Saclay

École doctorale n° 581 : agriculture, alimentation, biologie,
environnement et santé (ABIES)

Spécialité de doctorat : Sciences de l'environnement

Unité de recherche : Université Paris-Saclay, INRAE, AgroParisTech, UMR ECOSYS,

78850, Thiverval-Grignon, France

Ecole des Ponts ParisTech, UPEC, UMR LEESU,

77455 Champs-sur-Marne, France

Référent : AgroParisTech

**Thèse présentée et soutenue à Paris-Saclay,
le 18 décembre 2020, par**

Tristan MARTIN

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Professeur, AgroParisTech

Président

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Professeur, AgroSup Dijon

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EcoSys



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Référent : AgroParisTech

Thèse présentée et soutenue à Grignon,
le 18 décembre 2020, par

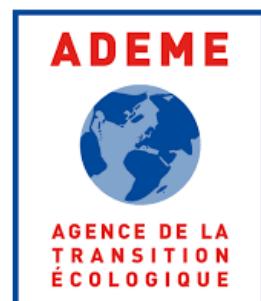
Tristan MARTIN

Composition du Jury

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| Mathieu SPERANDIO | Professeur, INSA Toulouse | Rapporteur & Examinateur |
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Financements

Cette thèse a été soutenue par le projet "Initiative Doctorale Interdisciplinaire (IDI 2017)" financé par l'IDEX Paris-Saclay, ANR-11-IDEX-0003-02. Le projet de recherche Agrocap dans lequel s'inscrit cette thèse est financé par l'ADEME (Agence de l'environnement et de la maîtrise de l'énergie), le SIAAP (Syndicat interdépartemental pour l'assainissement de l'agglomération parisienne) et la SEDE (Veolia).



Remerciements

La thèse est certes un rendu individuel, mais aussi un travail très collectif et je tiens à remercier très chaleureusement toutes les personnes qui y ont contribué.

Je tiens tout d'abord à remercier mes 3 encadrants de thèse : Fabien Esculier, Florent Levavasseur et Sabine Houot. Votre soutien et votre bienveillance m'ont été très utiles et, tout au long de ces 3 ans, ce fut un plaisir de travailler avec vous trois. Fabien, avec ton enthousiasme tu m'as donné l'envie de me lancer dans la recherche ! Ton positivisme et tes encouragements m'ont été très précieux pendant ces trois ans et j'ai beaucoup aimé échanger avec toi sur toute une variété de sujets. Florent, j'ai été très content de travailler avec toi, que ce soit lors des épandages sur le terrain ou lors de toutes les discussions que nous avons pu avoir. Ton pragmatisme a été d'une grande aide pour ce travail. Sabine, merci beaucoup pour ton engagement, tes encouragements dans les périodes de doutes, et tes nombreuses relectures !

Je souhaite remercier les membres de mon jury de thèse, Thierry Doré, Bernard Nicolardot, Mathieu Sperandio et Arnaud Helias, pour avoir accepté d'évaluer mon travail ainsi que pour les échanges très enrichissants que nous avons eus le jour de ma soutenance.

Je souhaite aussi remercier les membres de mon comité de thèse : Christine Aubry, Gilles Billen, Raymond Reau, Joël Aubin et Marie-Line Daumer pour toutes nos discussions qui m'ont beaucoup aidé, en particulier au début la thèse.

Je tiens aussi à remercier l'ADEME, le syndicat interdépartemental de l'assainissement de l'agglomération parisienne, la SEDE, et l'université Paris-Saclay pour avoir soutenu et financé ma thèse et le projet de recherche AGROCAPI.

Un grand merci à Léa Tordera et Enola Gilles que j'ai eu la chance d'encadrer pendant leurs stages de master et avec qui ça a été un plaisir de travailler !

Je souhaite remercier les collègues d'ECOSYS avec qui j'ai travaillé au cours de ces 3 ans : Sophie, Marco, Polina, Baptiste pour les essais volatilisation ; Jean-Christophe, Alain, Fabrice pour les essais agronomiques ; Yolaine et Eric pour votre aide au laboratoire et Marjolaine et Anaïs pour les analyses de micropolluants !

Je remercie aussi tous les collègues du LEESU, Annick et Mohammed, pour leur aide. Merci à tous les membres du programme OCAPI pour tous nos échanges : Marine, Bruno, Alessandro, Bernard, Aurélie, Etienne, Louise, Florent.

Un grand merci aux équipes de la Chambre d'agriculture d'Ile-de-France et en particulier à Christophe et Lucie pour leur aide et leur implication sur tous les essais. Merci aussi aux agriculteurs avec qui nous avons travaillé et sans qui les essais n'auraient pas été possibles.

Merci au réseau de l'assainissement écologique pour tous les échanges très enrichissants ainsi que pour les dizaines de m³ d'urine !

Merci à Joël Aubin et Julie Auberger de m'avoir formé sur l'ACV et pour leur aide sur cette partie de mon travail.

J'ai eu l'occasion de passer 3 mois en Suède, à l'Université d'Uppsala, lors de ma deuxième année. Je souhaite remercier toute l'équipe Kretsloppsteknik de m'avoir accueilli pendant l'hiver suédois : malgré le froid et la tombée de la nuit à 14h, votre accueil a été très chaleureux et j'ai vécu un bel échange ! Un merci particulier à Bjorn Vinnerås, Prithvi Simha, Jennifer McConville et Pernilla Tidåker, avec qui j'ai eu l'occasion de travailler au cours de ces 3 mois. Tack !

Merci à Damien, Victor, Diego, Anaïs, Paul, Marie-Liesse et Pauline pour les pauses déjeuner sportives et les bons moments passés ensemble !

Un grand merci au bureau 0B021 et tous ses occupants qui se sont succédé. Marianne, pour ses cours intensifs de Québécois, Sixtine qui derrière son côté autoritaire renferme un grand cœur, Anaïs pour les heures de rigolades et de jardinage, Mazette Marine qui s'est découvert une vocation de chanteuse à 15h00, Vlad pour m'avoir laissé le battre au tennis, Aurore pour sa bonne humeur communicative, et plus récemment président Florent qui est arrivé super motivé pour sa thèse.

Je souhaite remercier ma famille et mes amis qui m'ont encouragé et soutenu dans la dernière ligne droite.

Merci à Joëa pour ta présence et ton aide tout au long de ces 3 ans, de m'avoir supporté au quotidien (surtout à la fin). Sans toi, je n'aurais même pas pu faire un dixième de ce travail.

Liste des publications

Les chapitres 2, 3, 4 et 6 de cette thèse sont écrits sous la forme d'articles scientifiques :

Chapitre 2:

Martin TMP, Esculier F, Levavasseur F, Houot S (2020). Human urine-based fertilizers: a review.

Publié dans *Critical Reviews in Environmental Science and Technology*

Chapitre 3:

Martin TMP, Levavasseur F, Dox K, Tordera L, Esculier F, Smolders E, Houot S. Nitrogen use efficiency of nine different human urine-based fertilizers under greenhouse conditions

Soumis dans *Journal of Soil Science and Plant Nutrition*

Chapitre 4:

Martin TMP, Levavasseur F, Dion C, Vidal M, Esculier F, Houot S. Nitrogen fertilizing efficiency of human urine-based fertilizers is similar to mineral fertilizer but with potentially high volatilization of ammonia

À soumettre dans *Agronomy for Sustainable Development*

Chapitre 6:

Martin TMP, Gilles E, Aubin J, Auberger J, Esculier F, Levavasseur F, Houot S. Comparative study of environmental impacts related to cereal production with human-urine based fertilizers versus classical fertilizers

À soumettre dans *Journal of Cleaner Production*

Autres communications

Levavasseur F, Martin T (2020). De l'urine recyclée pour les futurs engrais. The conversation. theconversation.com/de-lurine-recyclee-pour-les-futurs-engrais-129945

Martin T, Levavasseur F, Esculier F, Houot S (2019). Détermination de l'efficacité fertilisante et des émissions gazeuses de fertilisants à base d'urine humaine. 14èmes Rencontres de la fertilisation raisonnée et de l'analyse (COMIFER), 20-21 Novembre 2019, Dijon, France (Poster)

Martin T, Levavasseur F, Esculier F, Houot S (2018). Human urine as a nitrogen fertilizer: a greenhouse experiment. 20th N Workshop, 25-27 Juin 2018, Rennes, France (Poster)

Table des matières

| | |
|--|-----------|
| Chapitre 1 Introduction générale..... | 14 |
| 1. Production agricole et fertilisation..... | 15 |
| 1.1. Les besoins des plantes | 15 |
| 1.2. Le modèle dominant actuel : la fertilisation minérale | 15 |
| 1.3. Alternatives aux engrains minéraux | 19 |
| 1.4. Gestion de la fertilisation azotée | 20 |
| 2. La gestion actuelle des excréts humains..... | 22 |
| 2.1. Le système alimentation/excrétion..... | 22 |
| 2.2. Composition des eaux usées..... | 23 |
| 2.3. Traitements des eaux usées | 24 |
| 2.4. Un système linéaire | 25 |
| 3. L'urine humaine en agriculture | 27 |
| 3.1. Une ressource autrefois valorisée..... | 27 |
| 3.2. La séparation à la source de l'urine et les filières de valorisation..... | 28 |
| 4. Cadre et objectifs de la thèse | 31 |
| 4.1. Le projet Agrocapi | 31 |
| 4.2. Objectifs et structure de la thèse..... | 31 |
| References | 34 |

Partie 1 Caractéristiques des traitements de l'urine et des urinofertilisants..... 39

| | |
|---|-----------|
| Chapitre 2 - Revue des urinofertilisants..... | 41 |
| Abstract..... | 43 |
| Key words..... | 43 |
| 1. Introduction | 44 |
| 1.1. Disconnection between sanitation and agriculture | 44 |
| 1.2. Human urine, the missing link | 44 |
| 1.2.1. <i>An essential part of wastewater</i> | 44 |
| 1.2.2. <i>Fresh urine</i> | 45 |
| 1.2.3. <i>Source separation and treatment</i> | 46 |
| 1.3. Scope of the study | 47 |
| 1.4. Literature recovery | 48 |
| 2. Characteristics of the treatments..... | 48 |
| 2.1. Storage..... | 48 |

| | |
|--|-----------|
| 2.2. Acidification..... | 49 |
| 2.3. Alkalization..... | 50 |
| 2.4. Nitrification | 51 |
| 2.5. Mixture with solid organic substrates | 51 |
| 2.6. Phosphorus precipitation..... | 51 |
| 2.7. Volume reduction post-treatments | 52 |
| 2.7.1. <i>Evaporation</i> | 54 |
| 2.7.2. <i>Distillation</i> | 54 |
| 2.7.3. <i>Forward osmosis</i> | 55 |
| 2.7.4. <i>Reverse osmosis</i> | 55 |
| 2.7.5. <i>Freezing-thawing</i> | 55 |
| 2.7.6. <i>Lyophilization</i> | 56 |
| 2.7.7. <i>Membrane distillation</i> | 56 |
| 3. Comparisons between treatments and resulting fertilizers | 56 |
| 3.1. Nutrient concentration..... | 56 |
| 3.1.1. <i>Nitrogen</i> | 56 |
| 3.1.2. <i>Phosphorus and other nutrients</i> | 58 |
| 3.2. Fertilizing efficiency and related impacts | 59 |
| 3.2.1. <i>Nitrogen and phosphorus use efficiencies</i> | 59 |
| 3.2.2. <i>Application</i> | 62 |
| 3.2.3. <i>Gaseous emissions</i> | 63 |
| 3.2.4. <i>Other impacts linked to field application</i> | 63 |
| 3.3. Contaminants..... | 65 |
| 3.3.1. <i>Trace metals</i> | 65 |
| 3.3.2. <i>Pharmaceutical residues</i> | 65 |
| 3.3.3. <i>Pathogens</i> | 67 |
| 3.4. Treatment inputs..... | 69 |
| 3.4.1. <i>Energy</i> | 69 |
| 3.4.2. <i>Chemicals and organic matter inputs</i> | 73 |
| 4. Synthesis and perspectives..... | 74 |
| References | 76 |
| Supplemental material..... | 91 |
| SM 2-1. Nutrient extraction and other urine treatments..... | 91 |
| SM 2-2. Pharmaceuticals post-treatments | 92 |
| References | 93 |

Partie 2 - Efficacité fertilisante azotée et impacts associés 98

| | |
|---|------------|
| Chapitre 3 - Efficacité fertilisante azotée en conditions contrôlées | 101 |
| Abstract..... | 103 |
| Key words..... | 103 |
| 1. Introduction | 104 |
| 2. Material and methods | 104 |
| 2.1. Urine-based fertilizers..... | 104 |
| 2.2. Greenhouse experiment..... | 106 |
| 2.3. Nitrogen use efficiency and mineral fertilizer equivalent..... | 107 |
| 3. Results and discussion..... | 108 |
| 3.1. Urine-based fertilizers characteristics | 108 |
| 3.2. Biomass production and nitrogen uptake | 111 |
| 3.3. Mineral fertilizer equivalent..... | 113 |
| 4. Conclusion..... | 114 |
| References | 115 |
| Supplementary material | 118 |
| SM 3-1. Detailed soil analysis..... | 118 |
| SM 3-2. Analysis method for plant and fertilizers | 119 |
| SM 3-3. Nitrogen residues at the end of the experimentation..... | 120 |

Chapitre 4 - Efficacité fertilisante azotée et mesures de volatilisation ammoniacale en conditions réelles 121

| | |
|--|------------|
| Abstract..... | 123 |
| Key words..... | 123 |
| 1. Introduction | 124 |
| 2. Material and methods | 126 |
| 2.1. Fertilizers tested | 126 |
| 2.2. General characteristics of the field experiments | 127 |
| 2.3. Mineral fertilizer equivalent experiments | 130 |
| 2.4. Substitution experiments | 131 |
| 2.5. Nitrogen use efficiency in organic agriculture | 131 |
| 2.6. Ammonia volatilization..... | 131 |
| 2.7. Statistical analyses..... | 132 |
| 3. Results and discussion..... | 133 |
| 3.1. Physico-chemical characteristics of the fertilizers and pathogens | 133 |
| 3.2. Mineral nitrogen equivalent for organic and urine-based fertilizers | 135 |

| | |
|--|------------|
| 3.3. Mineral fertilizer equivalents of urine-based fertilizers and comparison to other organic fertilizers | 138 |
| 3.4. Fertilization strategies | 141 |
| 3.4.1. Complete substitution of mineral fertilizers using urine-based fertilizer is possible..... | 141 |
| 3.4.2. Use of urine-based fertilizers in organic agriculture..... | 142 |
| 3.5. Ammonia volatilization..... | 144 |
| 4. Conclusion..... | 146 |
| References | 147 |
| | |
| Chapitre 5 - Mesures de volatilisation en conditions contrôlées | 150 |
| 1. Introduction | 151 |
| 2. Matériel et méthode..... | 152 |
| 2.1. Fertilisants testés | 152 |
| 2.1. Dispositif expérimental | 156 |
| 2.2. Expérimentations..... | 157 |
| 2.3. Statistiques | 158 |
| 3. Résultats et discussion..... | 159 |
| 4. Conclusion..... | 166 |
| Références | 167 |

Partie 3 - Impacts des filières de valorisation **170**

| | |
|--|------------|
| Chapitre 6 - Évaluation des impacts environnementaux de l'utilisation de trois urinofertilisants en comparaison des engrains classiques pour la production de céréales par analyse du cycle de vie..... | 172 |
| Abstract..... | 174 |
| Key words..... | 174 |
| 1. Introduction | 175 |
| 2. Method..... | 177 |
| 2.1. Goal and scope of the study | 177 |
| 2.1.1. Goal of the study | 177 |
| 2.1.2. Functional unit..... | 177 |
| 2.1.3. Boundaries of the system..... | 178 |
| 2.2. Life cycle inventory analysis and description of the scenarios | 179 |
| 2.2.1. Common hypothesis and background data | 181 |

| | |
|--|------------|
| 2.2.2. Reference scenario of fertilizer production..... | 183 |
| 2.2.3. Stored urine..... | 184 |
| 2.2.4. Nitrified concentrated urine..... | 185 |
| 2.2.5. Alkalinized dehydrated urine | 186 |
| 2.2.6. Grain production..... | 186 |
| 2.2.7. Sensitivity analysis | 192 |
| 2.3. Life cycle impact assessment | 192 |
| 3. Results and discussion..... | 195 |
| 3.1. Results by impact categories | 195 |
| 3.1.1. Climate change..... | 195 |
| 3.1.2. Acidification | 196 |
| 3.2.3. Particulate matter | 197 |
| 3.2.4. Eutrophication..... | 199 |
| 3.2.5. Water resource depletion | 200 |
| 3.5.6. Mineral, fossil and renewable resources depletion | 201 |
| 3.5.7. Cumulative energy demand..... | 202 |
| 3.2. Maize production..... | 203 |
| 3.3. Sensitivity analyzes | 204 |
| 3.3.1. Transport distance | 204 |
| 3.3.2. Number of drying units..... | 205 |
| 3.4. Discussion | 205 |
| 4. Conclusion..... | 209 |
| 5. Perspectives..... | 209 |
| References | 211 |
| Supplementary Material..... | 217 |

| | |
|--|------------|
| Chapitre 7 - Conclusion générale..... | 229 |
| 1. Synthèse des résultats..... | 230 |
| 1.1. L’urine humaine en agriculture | 230 |
| 1.2. Quels sont les traitements possibles de l’urine et quelles sont les caractéristiques des urinofertilisants résultant de ces traitements ? | 230 |
| 1.3. Quelle est l’efficacité fertilisante azotée des urinofertilisants en tant qu’engrais azotés ? | 234 |
| 1.4. Quels sont les impacts associés à l’apport d’urinofertilisants au niveau de la parcelle ?..... | 235 |
| 1.5. Quels sont les impacts environnementaux des différentes filières de valorisation de l’urine ?..... | 237 |
| 1.6. Synthèse de quatre filières parmi les plus développées..... | 238 |

| | |
|---|------------|
| 2. Perspectives..... | 241 |
| 2.1. Impacts agronomiques..... | 241 |
| 2.2. Freins et leviers sociotechniques (en France)..... | 242 |
| 2.3. Passage à l'échelle territoriale..... | 244 |
| Références | 247 |

Chapitre 1

Introduction générale

1. Production agricole et fertilisation

1.1. Les besoins des plantes

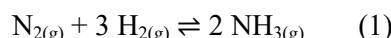
Aujourd’hui, la majeure partie de notre alimentation repose sur la production végétale pour nourrir directement les êtres humains ou les animaux d’élevage qui seront consommés (Mazoyer and Roudart 1997). Les plantes au contraire sont des organismes autotrophes qui produisent leurs propres tissus organiques à partir de molécules inorganiques prélevées dans leur environnement : les nutriments. Les végétaux contiennent une grande partie d’eau. À l’échelle atomique, au moins 17 nutriments essentiels au développement des plantes ont été recensés (Frausto da Silva and Williams 2001; Danforth 2009). Parmi eux, le carbone (45% de la masse sèche, fixé lors de la photosynthèse), l’oxygène (45% de la masse sèche, impliqué dans la respiration) et l’hydrogène (6% de la masse sèche, impliqué dans la balance osmotique, les réactions biochimiques, etc.). Ces trois éléments proviennent de l’air et de l’eau absorbés par la plante. Ensuite, les trois nutriments primaires pour la croissance des plantes (Danforth 2009; Raven 2014) sont l’azote (1,5% de la masse sèche, constituant des protéines, de la chlorophylle et des acides nucléiques), le phosphore (0,2% de la masse sèche, impliqué dans de nombreuses réactions biochimiques et constituant de l’ADN) et le potassium (1% de la masse sèche, impliqué dans la photosynthèse et de nombreuses autres fonctions). D’autres nutriments secondaires (calcium, magnésium, soufre) et micronutriments (bore, chlore, cuivre, fer, manganèse, molybdène, zinc et nickel) sont aussi nécessaires à la croissance des plantes (Danforth 2009) et composent les 1,3% restant de la masse sèche (Raven 2014). Selon la loi de Liebig sur le minimum (Liebig 1855), la croissance d’une plante est limitée par le nutriment qui vient à manquer en premier. Étant donné leur importance pour le fonctionnement des plantes et leur disponibilité naturelle dans les sols, les 3 éléments limitant la croissance des plantes sont principalement l’azote (N), le phosphore (P) et le potassium (K). Par exemple, la production de 100 quintaux de grains de blé exporte avec le grain environ 180 kg N, 65 kg P exprimé en P_2O_5 et 50 kg K exprimé en K_2O (calculé d’après COMIFER 2013, 2019). Toute société basant sa subsistance sur l’agriculture doit donc trouver le moyen de restituer la fertilité des sols cultivés et compenser les exports de nutriments des récoltes pour garantir une production alimentaire pérenne.

1.2. Le modèle dominant actuel : la fertilisation minérale

La pratique classique pour satisfaire les besoins en nutriments des récoltes consiste à utiliser des fertilisants ou engrais, c’est-à-dire des matières comprenant des nutriments nécessaires à la croissance des plantes. Au cours du XXe siècle, la production agricole a très fortement augmenté par l’utilisation de fertilisants minéraux d’origine industrielle (Erisman et al. 2008). En effet, chaque année, environ 200 millions de tonnes d’engrais minéraux sont consommés et cette consommation est susceptible de continuer à augmenter dans les prochaines années (FAO 2019). Les principaux engrais utilisés sont les

engrais azotés (environ 110 millions de tonnes), les engrais phosphatés (environ 50 millions de tonnes, exprimées en P₂O₅) et les engrais potassiques (environ 40 millions de tonnes, exprimées en K₂O).

L'azote compose 78% de l'atmosphère, mais sous une forme inutilisable par les plantes (diazote, N₂), hormis pour les plantes associées à des microorganismes pouvant fixer l'azote atmosphérique. En 1908, Fritz Haber a développé un procédé (Equation 1) permettant de transformer le diazote de l'air en ammoniac (NH₃). Cette forme réactive de l'azote peut être ensuite transformée en diverses formes d'azote constituant les engrais de synthèse (urée CO(NH₂)₂, ammonium NH₄⁺ ou nitrate NO₃⁻).



Ce procédé a été développé à l'échelle industrielle par Carl Bosch en 1913 et est aujourd'hui connu sous le nom de procédé Haber-Bosch. On estime que l'utilisation d'engrais azotés de synthèse a permis d'augmenter les rendements de 30 à 50% et a accompagné le passage d'une population de moins de 2 milliards d'habitants au début du XX^{ème} siècle à plus de 7 milliards aujourd'hui. Actuellement, environ 50% de la population mondiale dépend du procédé Haber-Bosch pour se nourrir (Figure 1-1., Erisman et al. 2008) et l'azote issu du procédé Haber-Bosch représente environ 70% de l'azote injecté dans les systèmes agricoles (Sutton et al. 2013).

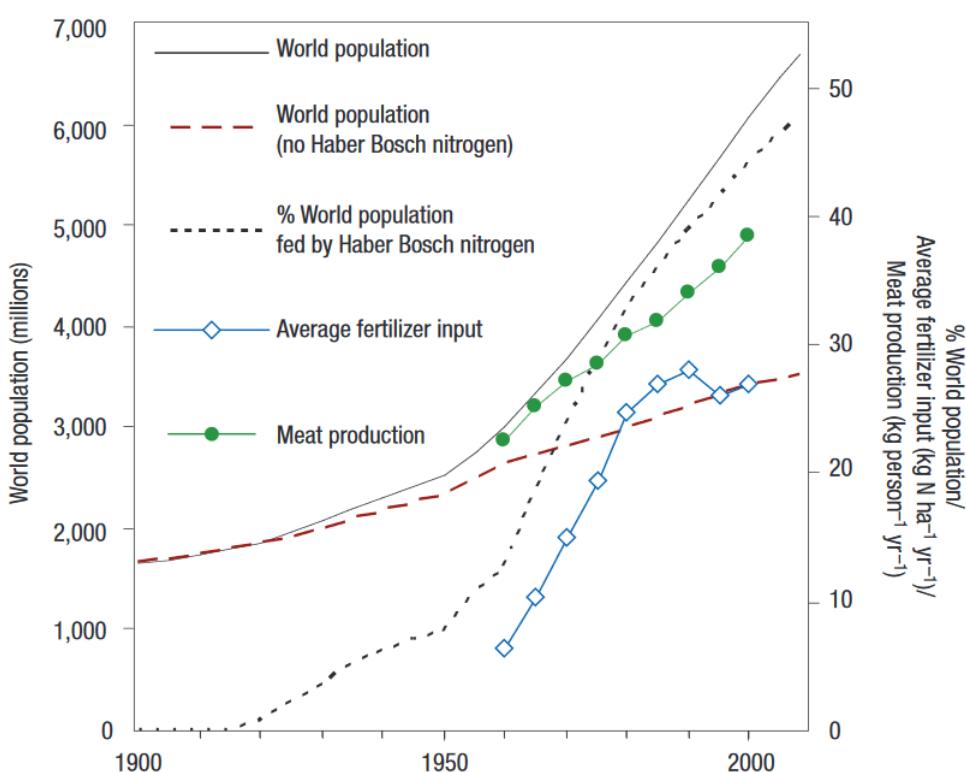


Figure 1-1. Évolution de la population humaine (mesurée et modélisée sans l'azote Haber-Bosch), de la consommation d'engrais azotés et de la production de viande au cours du XX^{ème} siècle (Erisman et al. 2008).

Cependant, du dihydrogène (H_2) est nécessaire à la réaction. La production de ce dihydrogène (Équation 2) est très majoritairement dépendante de l'utilisation de combustible fossile et en particulier de gaz naturel (Cherkasov et al. 2015). Ainsi, le procédé Haber-Bosch est un procédé énergivore avec environ 45 MJ/kg N synthétisé (Maurer et al. 2003) qui représente 1 à 2% de la consommation énergétique mondiale (Matassa et al. 2015).



Dès lors, la production des engrains azotés se déroule majoritairement dans les pays disposant de gisement de gaz naturel. Seulement 34% de l'azote consommé en France est produit en France et, même dans ce cas, sa synthèse est basée sur l'utilisation de gaz naturel importé (UNIFA). Le prix de ces engrais est aussi corrélé à celui des combustibles fossiles et est donc volatile (Figure 1-2).

Indice des prix des engrais azotés simples et du gaz

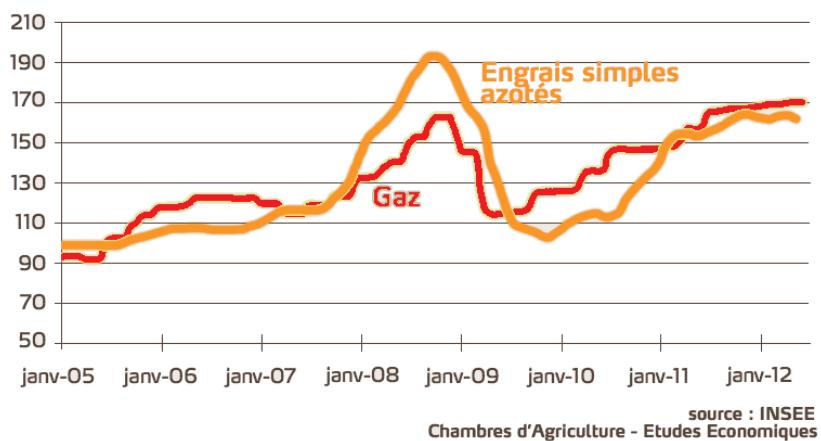


Figure 1-2. Indice des prix des engrais azotés simples et du gaz naturel entre janvier 2005 et janvier 2012 (adapté de Yara 2020).

Si l'azote minéral est aujourd'hui plutôt bon marché (entre 500 et 750 euros la tonne d'azote, web-agri.fr), ce coût a récemment augmenté dans les dernières décennies et représente en moyenne 11% des coûts de production par hectare pour les grandes cultures (Lecuyer et al. 2013).

À côté de la fertilisation azotée, l'agriculture actuelle est aussi basée sur l'utilisation de phosphore issu de roches phosphatées fossiles. La consommation d'engrais phosphaté a augmenté très fortement au cours de la deuxième moitié du XX^{ème} siècle (Cordell et al. 2009). Cependant, c'est une ressource non renouvelable extraite de mines. Certains auteurs mettent en évidence un possible pic de production des engrais phosphatés d'ici à la fin du siècle (Cordell et al. 2009). Les estimations des réserves vont de 30 à 300 ans (Figure 1-3), mais le pic de production pourrait entraîner un prix des fertilisants phosphatés plus élevé et une qualité moindre contenant des teneurs supérieures en métaux lourds (Cordell et White 2011). De plus, les trois quarts des réserves mondiales de roches phosphatées sont situés au Sahara occidental (U.S. Geological Survey 2019). Très peu de mines étant présentes en

Europe, l'Union Européenne a récemment classé le phosphore comme une matière première critique présentant un risque élevé de pénurie d'approvisionnement (EU commission 2017).

Enfin, comme le phosphore, le potassium est issu de mines dont les réserves sont concentrées dans quelques pays (U.S. Geological Survey 2019). La tension sur cette ressource semble moins mise en avant que pour le phosphore en raison de réserves disponibles en Europe et de l'absence de pollution liée au potassium dans les masses d'eau comme c'est le cas pour l'azote ou le phosphore. Cependant, c'est aussi une ressource fossile dont les réserves sont limitées. Les réserves de potassium connues pourraient être épuisées avant celles du phosphore au rythme actuel de consommation (93 ans de réserves, Figure 1-3, Jönsson 2019).

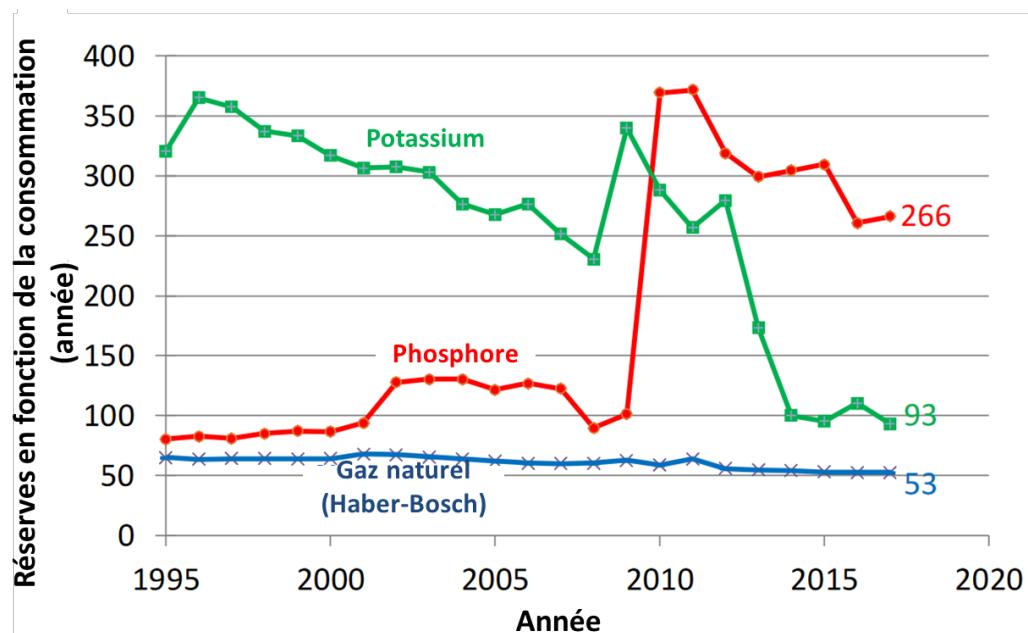


Figure 1-3. Réserves économiques de gaz naturel (utilisé pour le procédé Haber-Bosch), de phosphore et de potassium en fonction de la consommation actuelle (adapté de Jönsson 2019).

Les engrains minéraux sont donc largement utilisés, mais leur usage n'est pas durable, en raison de leur caractère fossile, de l'énergie nécessaire à leur production et des impacts environnementaux résultant de leur utilisation. En effet, l'utilisation massive des engrains minéraux depuis le XX^{ème} siècle perturbe très fortement l'environnement. Pour le cas spécifique de l'azote, une fois introduit dans les systèmes agricoles sous forme réactive (formes autres que N₂), l'azote va se retrouver dans de nombreux compartiments environnementaux par « cascade biogéochimique » (Billen et al. 2011). Différents impacts environnementaux causés par l'introduction d'azote réactif en excès ont été listés par Sutton et al. (2011) : acidification des sols et des masses d'eau, diminution de l'ozone stratosphérique, augmentation de l'ozone troposphérique, formation de gaz à effet de serre, diminution de la qualité de l'air et formation de particules fines, eutrophisation terrestre, des eaux douces et des océans liée au déséquilibre du milieu par excès de nutriments.

Steffen et al. (2015) ont défini 9 limites planétaires à ne pas dépasser pour rester dans un système terrestre stable. Parmi les frontières dépassées, nous trouvons les cycles biogéochimiques de l'azote et du phosphore (Figure 1-4). Ces frontières sont basées sur les flux de phosphore vers l'océan et les quantités de phosphore et d'azote injectés vers les systèmes agricoles. Ces excès de nutriments génèrent la possibilité d'événements anoxiques de grande échelle dans les masses d'eau marines entraînant la disparition de nombreux organismes vivants ainsi que l'eutrophisation diffuse des cours d'eau (développement excessif des végétaux dans l'eau et appauvrissement de la teneur en oxygène de l'eau entraînant de nombreux impacts sur la qualité de la masse d'eau).

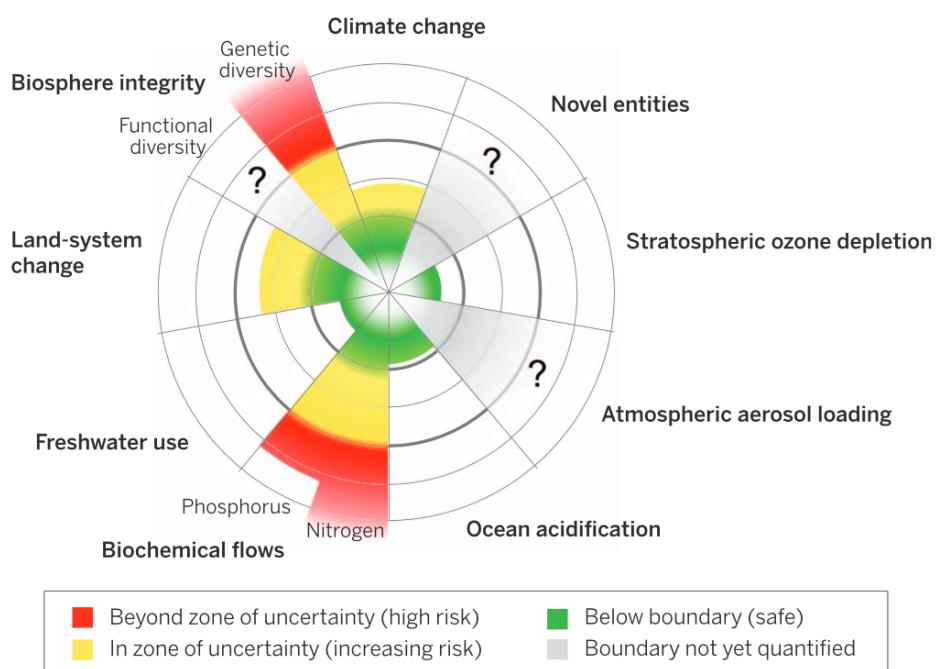


Figure 1-4. État actuel pour 7 des 9 limites planétaires (Steffen et al. 2015).

L'usage massif des engrains minéraux de synthèse est ainsi un des marqueurs de l'entrée dans l'Anthropocène. Le dépassement de ces frontières étant principalement dû aux apports de phosphore d'origine fossile et à l'azote des engrais, il est nécessaire de limiter et de réduire ces apports.

1.3. Alternatives aux engrains minéraux

Des alternatives aux engrains minéraux existent. C'est par exemple le cas des effluents d'élevage qui représentent environ 275 millions de tonnes en France. Cependant, ils sont déjà en très grande partie valorisés directement à la pâture ou après récupération dans les bâtiments d'élevage (Houot et al. 2014). De plus, les fourrages et grains utilisés pour nourrir les animaux sont en partie fertilisés avec des engrains minéraux (Le Noë et al. 2017) : les nutriments des effluents d'élevage viennent donc en partie et indirectement des engrains minéraux. De nouvelles ressources potentielles ont été identifiées, en particulier issues des déchets urbains conventionnels (biodéchets, déchets verts, etc.). Cependant,

cela ne représente que quelques millions de tonnes au niveau de la France et des flux insuffisants d'azote, de phosphore et de potassium à substituer aux engrains minéraux, hormis autour de certaines zones urbaines (Verger et al. 2018). Les boues de stations d'épuration sont quant à elles déjà valorisées en agriculture à hauteur de 60-70% en France (Houot et al. 2014). Elles permettent le recyclage d'une part non négligeable de phosphore, mais représentent néanmoins des flux mineurs à l'échelle nationale à la fois pour l'azote et le phosphore (Le Noë et al. 2018).

Une autre stratégie (seulement valable pour les apports d'azote) peut être l'utilisation de légumineuses qui permettent de fixer directement l'azote de l'air (Billen et al. 2012). Elles peuvent être intercalées entre différentes cultures non fixatrices d'azote et fournir une partie de l'azote à travers leurs résidus de culture. Cependant, ces restitutions sont généralement de l'ordre de quelques dizaines de kilogrammes d'azote par an (Peoples et al. 2009; Chambre d'agriculture de Seine-et-Marne 2017), ce qui est souvent insuffisant pour couvrir les besoins de cultures à haut potentiel de rendement. Le développement des légumineuses en parallèle d'un changement de régime alimentaire reste toutefois un élément clé pour réduire les besoins en fertilisants azotés des systèmes de culture (Billen et al. 2012).

1.4. Gestion de la fertilisation azotée

Dans les systèmes conventionnels des climats tempérés, la gestion de la fertilisation azotée se fait en grande partie à l'année. Par exemple, pour la production de céréales, les apports sont généralement faits au printemps, période à laquelle les céréales ont besoin d'azote pour leur croissance. En France, la méthode du bilan est largement utilisée pour calculer les apports d'azote (COMIFER 2013). Cette méthode prend en compte les besoins de la culture ainsi que les fournitures du sol qui correspondent principalement aux reliquats en azote dans le sol à la sortie de l'hiver, à la minéralisation de la matière organique du sol, des résidus de culture du précédent et des produits organiques éventuellement apportés au cours de la culture. Le bilan des besoins et de la fourniture du sol donne la dose complémentaire d'azote minéral à apporter pour atteindre un rendement annuel visé. Dans les systèmes biologiques, la gestion de la fertilisation azotée se fait plutôt à l'échelle de la rotation en utilisant des légumineuses et des fertilisants organiques et à long terme en entretenant la fertilité du sol (Küstermann et al. 2010).

Un point important pour la gestion de la fertilisation azotée est l'efficacité des fertilisants. En effet, les cultures absorbent l'azote sous forme minérale : ammonium (NH_4) ou nitrate (NO_3). En opposition aux engrains azotés minéraux qui ne contiennent que des formes minérales de l'azote (l'urée est considérée comme une forme minérale étant donné son hydrolyse rapide), les fertilisants organiques (digestat, lisier, fumier...) contiennent une proportion plus ou moins grande d'azote organique. Ainsi l'intérêt fertilisant de ces produits organiques va dépendre de leur teneur en azote minéral et de la vitesse de minéralisation de leur azote organique pour pouvoir être absorbé par les cultures. Cette

fraction organique peut être plus ou moins stable et difficile à dégrader, résultant en une efficacité en tant que fertilisant azoté plus ou moins grande l'année de l'apport (Agehara et Warncke 2005; Gaskell et Smith 2007).

Outre la dynamique de minéralisation de la fraction organique, l'efficacité fertilisante va également dépendre du risque de volatilisation de la fraction azotée sous forme ammoniacale (Trochard et al. 2019). Celle-ci se produit principalement au moment de l'épandage et est favorisée par la teneur en azote ammoniacal du fertilisant (au moment de l'épandage ou après hydrolyse de l'urée), des pH du sol et du fertilisant élevés, et par la durée de contact du fertilisant avec l'air (elle est plus longue pour les produits solides). De plus, l'ammoniac est un polluant de l'air qui a un impact négatif sur la santé à travers la formation de particules fines et sur l'environnement par acidification et eutrophisation (Sutton et al. 2011).

Les différents fertilisants azotés (minéraux et organiques) peuvent avoir des efficacités fertilisantes différentes. Le coefficient apparent d'utilisation de l'azote (CAU) et le coefficient d'équivalence engrais (KEQ) sont souvent utilisés pour quantifier cette efficacité fertilisante d'un engrais sur une saison culturelle (Figure 1-5, Réseau PRO 2015). Le CAU correspond à la proportion apparente de l'azote apporté par le fertilisant absorbé par la culture. Le coefficient d'équivalence engrais correspond au rapport entre le CAU du fertilisant testé par rapport à celui d'un engrais minéral. Il indique à quelle dose d'engrais minéral correspond le même apport d'azote avec un fertilisant organique. Par exemple, avec un KEQ de 50% pour un engrais organique, deux fois plus d'azote devront être apportés pour obtenir la même efficacité qu'avec un engrais minéral sur une saison culturelle. Le KEQ d'un fertilisant est souvent très fortement corrélé à sa teneur en azote minéral, ainsi qu'à la rapidité de la minéralisation de l'azote organique qu'il contient. Ainsi, les digestats ayant une proportion élevée d'azote minéral vont avoir un KEQ élevé de 40 à 60% (Gutser et al. 2005) quand d'autres fertilisants ayant une forte proportion d'azote organique auront un KEQ plus faible, par exemple 10 à 20% pour un fumier bovin (Gutser et al. 2005). Plus le KEQ sera élevé, plus le comportement du fertilisant se rapprochera de celui d'un engrais minéral et plus la dose d'azote total à apporter pour une même efficacité immédiate sera proche de la dose d'azote efficace nécessaire. Ces KEQ peuvent être utilisés directement par les agriculteurs pour l'établissement de leur plan prévisionnel azoté (COMIFER, 2013). Ils ne restent cependant que des ordres de grandeur, la valeur fertilisante azotée des produits résiduaires organiques (PRO) dépendant de nombreux facteurs (sols, climat, pratiques et conditions d'apports...).

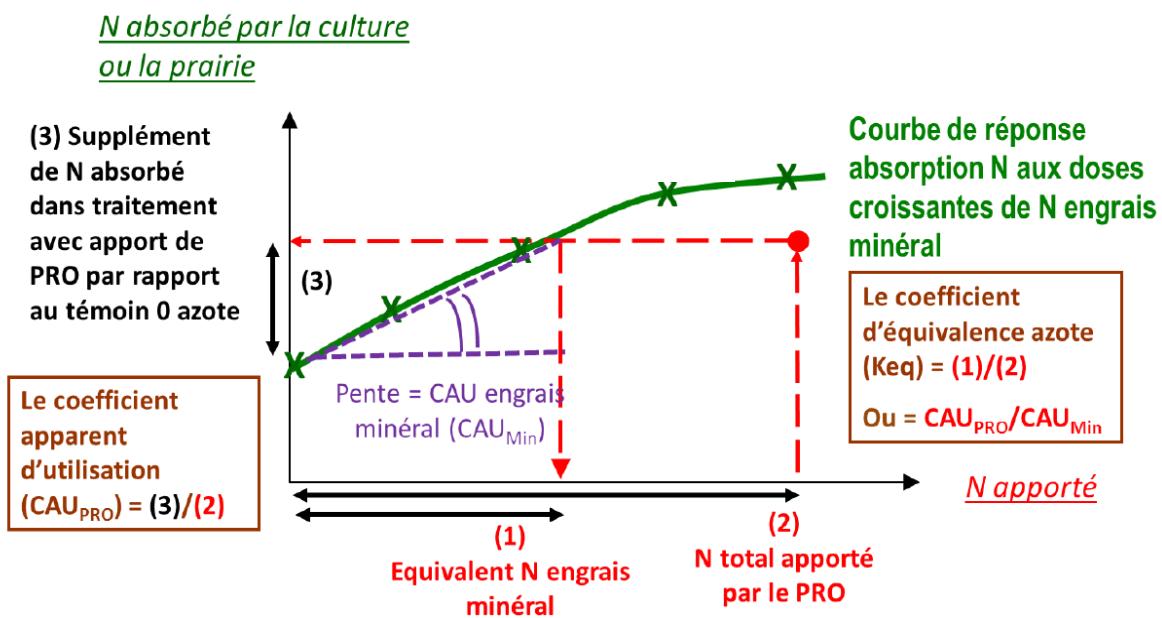


Figure 1-5. Courbe de réponse à l'azote (N absorbé pour N apporté) et calculs des CAU et KEQ (adapté de Réseau PRO 2015).

2. La gestion actuelle des excréts humains

2.1. Le système alimentation/excrétion

Le corps humain n'accumulant pas de nutriments, excepté en faible quantité lors des périodes de croissance, les êtres humains ont besoin d'excréter les nutriments absorbés via l'alimentation. Ces deux besoins physiologiques – se nourrir et excréter – ainsi que toutes les activités qui y sont reliées, forment le système alimentation/excrétion d'une société (Esculier 2018). Chaque année, environ 4,6 kg d'azote et 550 g de phosphore sont absorbés et excrétés par personne dans le régime alimentaire actuel des sociétés occidentales (Figure 1-6.).

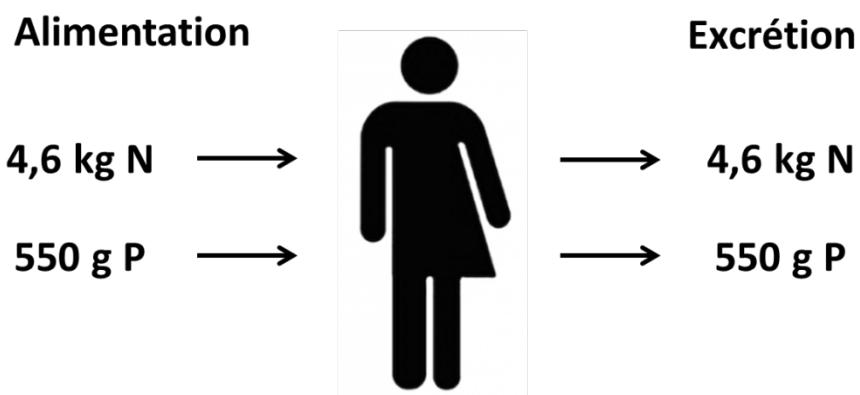


Figure 1-6. Flux d'azote et de phosphore du système alimentation/excrétion par personne et par an. Adapté de Esculier (2018) et basé sur les données de Friedler et al. (2013).

2.2. Composition des eaux usées

Dans les sociétés industrielles, les excréts humains sont usuellement évacués des habitations en mélange avec les différentes eaux domestiques, formant ensemble les eaux usées. Les composantes des eaux usées sont principalement les « eaux jaunes » (urine et chasse d'eau), les « eaux marrons » (matières fécales, papier toilette et chasse d'eau) et les « eaux grises » (eaux de douche, de baignoire, de lavabo, de cuisine, de lavage, etc.). En moyenne, 148 L d'eaux usées domestiques sont produites par personne et par jour dans les pays industrialisés (Friedler et al. 2013).

Environ 0,5 m³/personne/an d'urine est excrété. L'urine est composée à 95% d'eau et à 5% de sels dissous (Friedler et al. 2013). La fraction organique, caractérisée par la demande chimique en oxygène (DCO), présente dans l'urine est assez faible avec environ 4,7 kg DCO/personne/an. Environ 4 kg N, 0,3 kg P et 0,9 g K/personne/an sont excrétés dans l'urine, ce qui représente environ 5 kg de nutriments/personne/an. Les matières fécales représentent 73 kg/personne/an (Friedler et al. 2013). La matière organique présente dans les matières fécales est plus élevée que pour l'urine avec environ 11,3 kg DCO/personne/an. Le flux de nutriments est plus faible que l'urine avec 0,5 kg N, 0,2 kg P et 0,3 g K/personne/an (Friedler et al. 2013). La composition des eaux grises varie énormément en fonction des pratiques individuelles. Cependant, elle représente la majeure partie des eaux usées domestiques, soit environ 39 m³/personne/an et 73% du volume total (Friedler et al. 2013). Les eaux grises représentent une partie significative des flux de matière organique avec 18,6 kg DCO/personne/an (Friedler et al. 2013). Cependant, les flux de nutriments sont faibles en comparaison de l'urine avec environ 0,3 kg N, 0,2 kg P et 0,1 kg K par personne et par an. Ainsi, l'urine représente la plus grande partie de l'azote, du phosphore et du potassium présents dans les eaux usées (respectivement 79, 47 et 71%, Figure 1-7.) dans moins de 1% du volume total des eaux usées (Friedler et al. 2013).

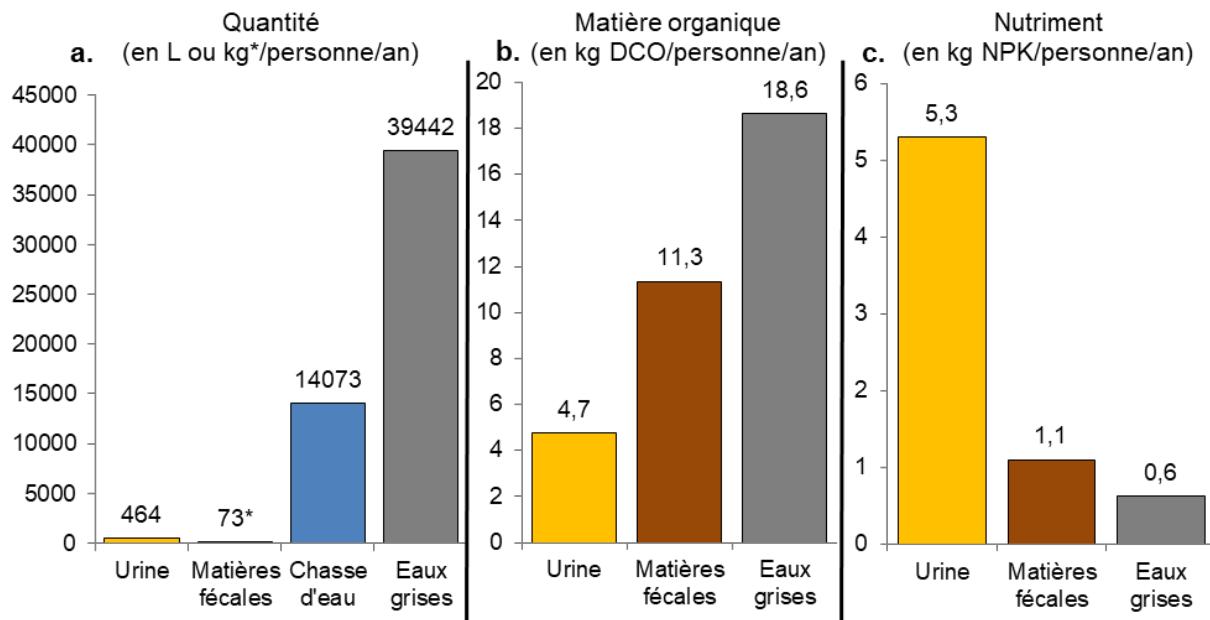


Figure 1-7. Quantité (a.), flux de matière organique (b.) et de nutriments (c.) par personne et par an pour les différentes composantes des eaux usées domestiques. Basé sur les valeurs de Friedler et al. (2013).

2.3. Traitements des eaux usées

La gestion des excréts et des eaux usées est très inégalement mise en œuvre selon les pays. En 2017, 55% de la population mondiale, soit 4,2 milliards d'habitants n'ont pas accès à une bonne gestion sanitaire des excréts (WHO et UNICEF 2019). Dans les pays industrialisés, les eaux usées dans les zones densément peuplées sont majoritairement collectées par un égout et théoriquement orientées vers les stations d'épuration pour être traitées. Depuis la seconde moitié du XX^{ème} siècle, les traitements se sont complexifiés et ont permis une efficacité de traitement des eaux usées de plus en plus élevée et une diminution des rejets vers les milieux aquatiques. Les traitements des eaux usées sont très variés. Le traitement le plus simple (traitement primaire) correspond à une simple décantation des eaux. Dans la plupart des stations d'épuration, cette décantation est généralement complétée par un traitement aux boues activées. Ce traitement consiste à cultiver des bactéries qui dégraderont la matière organique dans un bassin aéré (milieu aérobie). Il permet principalement de dégrader le carbone des eaux usées. Au fur et à mesure de l'évolution des technologies et en fonction des stations d'épuration, différentes étapes se sont rajoutées comme la précipitation des phosphates par ajout de fer ou d'aluminium.

Pour ce qui est de l'azote, la dénitrification (transformation de l'azote en diazote gazeux) est considérée comme une bonne pratique. La plus grande partie de la matière organique est dégradée lors des premières étapes des traitements. La majorité de l'azote se retrouve donc sous forme ammoniacale (NH_4^+). La première étape du traitement consiste donc en une nitrification biologique de l'azote

ammoniacal en azote nitrique par des bactéries nitrifiantes dans un bassin aéré (Larsen et al. 2013). La seconde étape correspond à la dénitrification hétérotrophique. C'est une réaction anaérobie qui peut nécessiter l'ajout d'un substrat carboné (p. ex., méthanol) car la majorité du carbone a été dégradée lors des étapes précédentes. Cette réaction correspond à la réduction de l'azote nitrique en diazote gazeux (N_2) qui est renvoyé vers l'atmosphère. L'azote restant en solution est rejeté vers les eaux de surface (Larsen et al. 2013; SIAAP 2020). La très grande majorité de l'azote étant émise vers l'atmosphère ou les eaux de surface, ce traitement empêche le recyclage de l'azote vers l'agriculture.

Les obligations réglementaires de traitement des eaux usées dans l'Union Européenne sont régies par la directive 91/271/EEC (1991) relative au traitement des eaux urbaines résiduaires. Le pourcentage minimal de réduction du phosphore total par rapport à l'entrée de la station d'épuration dans les zones sensibles à eutrophisation est fixé à 80% (ou rejet en dessous de 1 à 2 mg P/L en fonction de la taille de l'agglomération) et à 70-80 % pour l'azote total (ou rejet en dessous de 10 à 15 mg N/L en fonction de la taille de l'agglomération). Aucune précision n'est donnée dans la réglementation en fonction de la taille de l'agglomération pour le pourcentage de réduction de l'azote entrant à la station d'épuration, ce qui se traduit généralement en pratique par une efficacité minimale de traitement exigée de 70% pour l'azote. En considérant le cas de Paris avec ses 10,5 millions d'habitants et un taux d'abattement de 80% (supérieur à la réalité pour l'azote, légèrement inférieur pour le phosphore), c'est l'équivalent en azote et en phosphore de l'urine de 2,1 millions de personnes qui resterait dans les eaux après traitement et serait rejeté vers la Seine.

Les boues issues des différentes étapes des traitements peuvent être recyclées. En France, la majorité des boues d'épuration (60 à 70% au niveau national et 50% dans le cas de l'agglomération parisienne) est valorisée en agriculture, soit directement, soit après compostage (Reverdy and Pradel 2010; Esculier 2018). Cependant, cette pratique de recyclage est interdite dans différents pays comme en Suisse, principalement en raison des contaminants potentiellement présents dans les boues, obérant le recyclage des nutriments des eaux usées vers l'agriculture. Quand le phosphore est traité dans les eaux usées, les boues d'épuration contiennent la majorité du phosphore, leur épandage en agriculture permet donc un bon recyclage du phosphore des eaux usées. Elles contiennent cependant une très faible partie résiduelle de l'azote et du potassium (qui n'est pas traité) des eaux usées, leur épandage en agriculture ne permet donc pas de recyclage significatif de ces deux éléments. Le traitement des eaux usées à la station d'épuration a aussi des impacts négatifs sur l'environnement en raison de la consommation d'énergie (Maurer et al. 2003) et des émissions de gaz à effet de serre (Kampschreur et al. 2009).

2.4. Un système linéaire

En raison de la gestion actuelle des excréts humains, le système alimentation/excrétion sur sa partie urbaine peut être considéré comme majoritairement linéaire. Par exemple, seulement 4 % de l'azote,

41 % du phosphore et 2% du potassium excrétés sont actuellement recyclés vers l'agriculture via les boues d'épuration en Ile-de-France (Esculier 2018). Si les valeurs de recyclage du phosphore peuvent être plus élevées dans certains cas favorables, les valeurs de recyclage de l'azote et du potassium sont quasiment toujours très faibles et ces valeurs sont totalement nulles quand les boues ne vont pas sur des sols agricoles. Les flux pour l'azote excrété et leur gestion sur la partie urbaine sont représentés dans la Figure 1-8 dans le cas de Paris. Ce système linéaire entraîne à la fois la nécessité de traiter les eaux usées avant rejet vers l'environnement et l'utilisation d'engrais minéraux pour la fertilisation. Afin de ne pas dépasser les limites planétaires (Steffen et al. 2015), l'émission de nutriments dans l'environnement pourrait être diminuée en partie par le recyclage des nutriments des eaux usées aujourd'hui rejetés vers l'environnement.

kgN/cap/y

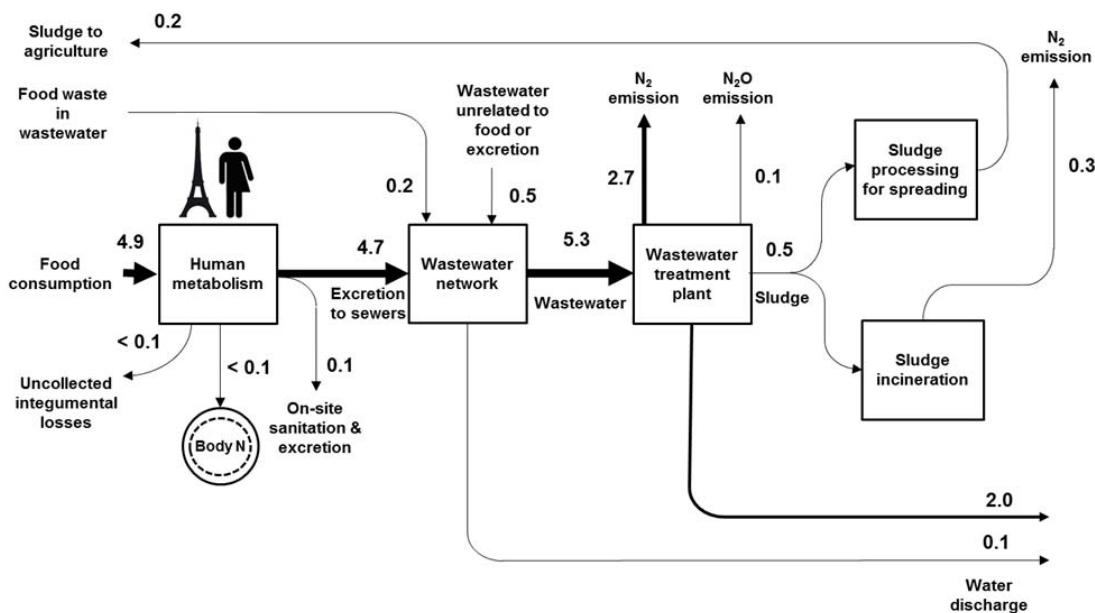


Figure 1-8. Flux d'azote urbains liés aux excréptions d'un habitant de l'agglomération parisienne (Esculier 2018).

Comme vu précédemment, l'urine concentre la majeure partie des nutriments des eaux usées (79% de l'azote, 47% du phosphore et 71% du potassium des eaux usées). En fonction du contexte géographique (urbain et agricole), les nutriments des excréts pourraient couvrir une grande partie des besoins des cultures. Par exemple, dans le cas de l'agglomération parisienne qui comporte 10,5 millions d'habitants et est entourée par 569 000 hectares de terres cultivés majoritairement en grandes cultures, l'urine des habitants de l'agglomération pourrait couvrir 140% de la consommation actuelle d'azote, 75% pour le phosphore et 156% de la consommation de potassium (calculé à partir des données de Esculier et al., 2018 et l'Union des industries de la fertilisation, 2018).

3. L'urine humaine en agriculture

3.1. Une ressource autrefois valorisée

Le recyclage des nutriments présents dans les excréts n'est pas une pratique nouvelle. En effet, différents cas de pratiques de valorisation de l'urine et matières fécales depuis plusieurs milliers d'années sont rapportés, en particulier en Asie comme en Chine (Paulet 1853; Xue 2005; Ferguson 2014; Esculier 2018). Dans le cas particulier des villes françaises, la gestion des excréts humains semble être majoritairement linéaire à l'échelle collective avant le XIX^{ème} siècle. Cependant, c'est à partir de ce moment et ce jusque pendant la première partie du XX^{ème} siècle que se développent, dans certaines villes, des systèmes circulaires et que se met en place un « mutualisme fécond », notamment entre l'agriculture et la ville (Barles 2005; Esculier 2018). Les matières sont principalement récupérées au pied des immeubles dans des fosses d'aisance (matières fécales et urine mélangées) et épandues sur les cultures après un traitement plus ou moins élaboré (Figure 1-9).



Figure 1-9. Épandage de matière de fosses d'aisance (Girardin 1876).

De manière intéressante, il existait une forte dynamique sur les différents traitements possibles des excréts et de l'urine à cette époque comme c'est actuellement le cas au XXI^{ème} siècle (voir chapitre 2.), par exemple par extraction de l'azote sous forme de sulfate d'ammonium, ou précipitation de struvite (Esculier 2018). Plus de 140 brevets sur le traitement des excréts pour en faire des fertilisants ont été listés par Adler (2020). Les instituts d'agronomie sont aussi très impliqués sur la valorisation agricole des excréts humains, comme par exemple à l'école d'agriculture de Grignon (Dehéain 1873).

Avec le développement concomitant du tout-à-l'égout et des engrains industriels à la fin du XIX^{ème} siècle et au XX^{ème} siècle, la majorité des villes perd l'engrais humain qui est envoyé en rivière.

Toutefois, certaines villes augmentent un temps leur circularité. C'est le cas de Paris où les eaux usées récupérées sont directement valorisées sur des champs d'épandage. Un pic de recyclage de l'azote et du phosphore est ainsi observé au début du XX^{ème} siècle. À ce moment, près de 65% du phosphore et 45% de l'azote de l'agglomération parisienne sont recyclés (Figure 1-10, Esculier et Barles 2019).

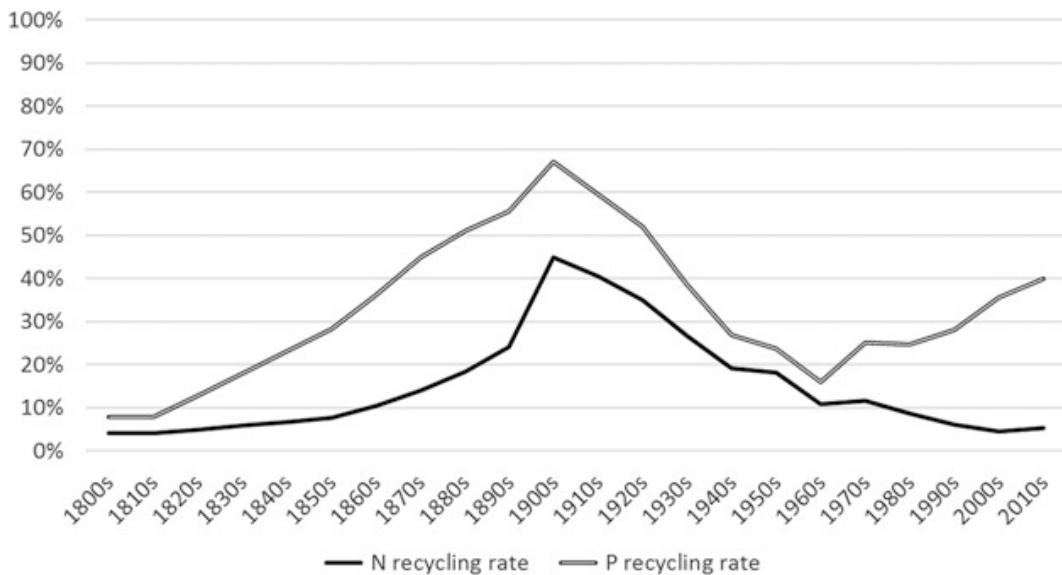


Figure 1-10. Taux de recyclage de l'azote et du phosphore des excréptions humaines de 1800 à 2010 à Paris (Esculier et Barles 2019).

Cependant, cette pratique ne se faisait pas sans risque, en raison par exemple de la présence de pathogènes (Adler 2020), ni sans impacts environnementaux liés aux métaux lourds dans les sols (Védry et al. 2001). En raison de divers facteurs tels que la disponibilité nouvelle des engrains minéraux, les coûts opérationnels des champs d'épandage, cette pratique a rapidement décliné au cours du XX^{ème} siècle. Le rejet direct des eaux usées en rivière puis leur traitement en station d'épuration ont alors été privilégiés (Esculier et Barles 2019). Nous pouvons cependant noter une amélioration depuis 50 ans dans le recyclage du phosphore avec une augmentation du taux d'eaux usées effectivement traitées puis la mise en place de sa récupération dans les boues d'épuration par précipitation et l'épandage agricole de ces boues (Esculier 2018). Aujourd'hui, l'urine humaine n'est généralement plus considérée comme un engrain dans l'imaginaire collectif (urine blindness, Drangert 1998).

3.2. La séparation à la source de l'urine et les filières de valorisation

Les eaux usées étant actuellement collectées avec un égout et orientées vers les stations d'épuration, il pourrait être intéressant pour parvenir à une plus grande circularité de transformer les stations d'épuration en station de récupération des ressources (water resources recovery facilities, Besson 2020). Cependant, une fois toutes les composantes des eaux usées mélangées, il devient difficile d'extraire la plupart des nutriments. En effet, aujourd'hui seul le phosphore peut être récupéré

efficacement par précipitation (récupéré à 84% dans les boues d'épuration au niveau de l'agglomération parisienne, Esculier 2018). Cependant dans le cas de l'azote, les meilleures technologies disponibles ne permettent qu'une récupération limitée au niveau de la station d'épuration, souvent inférieure à 30% (Esculier 2018; Besson 2020) et sont énergivores. C'est cependant un champ de recherche dynamique. D'autres éléments fertilisants d'intérêt, comme le potassium, ne sont pas considérés comme des polluants pour les masses d'eau et ne sont donc ni traités, ni recyclés.

Le mélange de l'urine, qui contient la plupart des nutriments, avec les autres composantes des eaux usées (matières fécales, eaux grises) entraîne différents impacts négatifs, par exemple la présence de pathogènes, de métaux, la dilution des nutriments avec les volumes élevés des eaux grises. Cependant, ces impacts peuvent être évités par la séparation à la source de l'urine (Rossi et al. 2009). Comme pour un tri sélectif des déchets, cette pratique consiste à séparer les différentes composantes des eaux usées en fonction de leurs caractéristiques et potentielles utilisations. Ainsi, la séparation à la source permet de séparer l'urine des eaux grises pour éviter la dilution. Elle permet aussi de séparer l'urine des matières fécales car leur composition et leurs caractéristiques physiques (liquide ou solide) sont très différentes. En outre, des traitements adaptés à chaque type de matière se révèlent particulièrement efficaces pour valoriser au mieux ces ressources (Esculier, 2018). La séparation à la source peut être réalisée à l'aide de différents types de toilettes (Figure 1-11).



Figure 1-11. Différents types de toilettes permettant la séparation à la source. a. Urinoir féminin «Marcelle», b. Toilette à séparation «Roediger» installé à l'EAWAG c. Principe de fonctionnement d'une toilette à séparation «save!, Laufen». Credits: Tristan Martin, Louise Raguet, EOOS.

L'urine étant peu contaminée en pathogènes, la séparation à la source de l'urine permet aussi de limiter sa contamination par les autres composantes des eaux usées et en particulier par les matières fécales. Cependant, en fonction du type de toilette utilisée, une contamination fécale croisée peut arriver (Höglund et al., 1998). La contamination de l'urine en métaux est aussi faible en comparaison des autres composantes des eaux usées (Ronteltap et al. 2007). Néanmoins, environ 70% des résidus de pharmaceutiques sont excrétés dans l'urine et des questions subsistent quant aux risques pour la

santé et l'environnement qu'ils entraînent et la nécessité de les traiter (Lienert et al. 2007; Winkler 2009; WHO 2012).

La séparation à la source de l'urine est une pratique très ancienne. Elle a connu un regain d'intérêt en Suède au début des années 1990 et différents quartiers équipés de toilettes à séparation ont été construits (Kirchmann et Pettersson 1995; Johansson et al. 2000). Cette pratique s'est ensuite diffusée progressivement au cours des années 1990 et 2000 dans l'Europe scandinave (Danemark, Gómez-Muñoz et al. 2017) et germanique (Suisse, Allemagne, Larsen et Gujer 1996; Otterpohl 2003). La séparation à la source de l'urine est aujourd'hui étudiée mondialement (États-Unis, Brésil, Chine, etc. Jiang et al. 2017; Mullen 2018; Medeiros et al. 2020). Dans le cas particulier de la France, elle a pris un essor important depuis 2015 et plusieurs quartiers équipés de séparation à la source de l'urine sont actuellement à l'étude. D'un point de vue réglementaire, le statut de l'urine pour un usage agricole n'est pas explicitement précisé. Cependant, il est à ce jour possible, *a minima*, d'assimiler l'urine à des matières de vidange de l'assainissement non collectif relevant du décret n° 97-1133 du 8 décembre 1997 relatif à l'épandage des boues issues du traitement des eaux usées et son épandage en agriculture pourrait relever de la mise en place de plans d'épandage.

Une fois l'urine séparée, de nombreux traitements sont possibles. La plupart de l'azote est excrété dans l'urine sous forme d'urée, cependant cette urée s'hydrolyse rapidement en azote ammoniacal qui est sensible à la volatilisation. Certains traitements ont pour objectif de **stabiliser l'azote** dans une forme qui limite ces pertes par volatilisation. L'urine étant une solution peu concentrée en comparaison des engrains minéraux, certains traitements ont pour but d'augmenter la concentration en nutriments et de **réduire le volume**. Des traitements ont pour objectif de **réduire la contamination en pathogènes/résidus de pharmaceutiques**. Enfin, certains traitements ont pour objectif d'**extraire un ou plusieurs nutriments de l'urine**. Les fertilisants résultants de ces traitements sont appelés « urinofertilisants ».

La mise en place de ces filières de valorisation de l'urine en agriculture pose de nombreuses questions. En effet, même si de plus en plus d'études sont conduites ces dernières années, la plupart des urinofertilisants et leurs filières de valorisation restent pour la plupart peu étudiés et peu connus. Par exemple, les urinofertilisants présentent des caractéristiques variées (forme et concentration de l'azote, liquide/solide), mais il existe très peu d'essais agronomiques comparant leurs efficacités. Différents impacts associés à leur épandage restent aussi très peu étudiés, c'est par exemple le cas de la volatilisation qui peut potentiellement être élevée, l'azote étant sous forme ammoniacale dans de nombreux urinofertilisants. Afin de pouvoir comparer les bénéfices de l'utilisation de l'urine en comparaison des pratiques actuelles associant l'utilisation d'engrais minéraux et le traitement de l'urine à la station d'épuration, des évaluations globales au niveau de la filière de valorisation sont nécessaires en prenant par exemple en compte les impacts du traitement et du transport.

4. Cadre et objectifs de la thèse

4.1. Le projet Agrocapi

Cette thèse s'inscrit dans le projet Agrocapi (programme OCAPI : www.leesu.fr/ocapi) qui a pour but d'étudier différentes filières de valorisation de l'urine en agriculture. En plus des aspects environnementaux et agronomiques inclus dans cette thèse, il vise aussi à étudier les contaminants et en particulier les résidus de pharmaceutiques présents dans les urinofertilisants ainsi que leur devenir après apport au champ. Les freins et leviers à l'emploi d'urinofertilisant et l'appropriation de cette pratique par les agriculteurs sont également étudiés dans le cadre du projet (Brun, 2018).

4.2. Objectifs et structure de la thèse

Nous avons vu que l'urine contient, dans un faible volume, la majeure partie des nutriments des eaux usées, et son utilisation comme fertilisant semble pertinente pour participer au bouclage des cycles biogéochimiques. Cependant, de nombreuses questions se posent pour les diverses filières de valorisation de l'urine possibles. **L'objectif de cette thèse est de caractériser les filières envisageables de valorisation de l'urine humaine en agriculture au niveau agronomique et leurs impacts environnementaux.** Étant donné la présence de nombreux macro et micro nutriments dans l'urine (Etter et al. 2015) et l'importance de clore tous les cycles biogéochimiques, nous nous sommes intéressés principalement aux traitements de l'urine qui conservent tous les nutriments (traitements conservatifs). Étant dans une perspective d'efficacité fertilisante azotée, nous nous sommes aussi majoritairement concentrés sur les procédés gardant l'azote sous forme minérale et aboutissant à la production d'engrais.

L'organisation du manuscrit est présentée dans la Figure 1-12. L'objectif de la thèse a été décomposé avec les questions suivantes :

1/ Quels sont les traitements possibles de l'urine et quelles sont les caractéristiques des urinofertilisants résultant de ces traitements?

La première partie de cette thèse correspond à une revue de la littérature des différents traitements possibles de l'urine et des urinofertilisants résultant de ces traitements. La revue s'est concentrée principalement sur les traitements de l'urine qui conservent la plupart des nutriments. Les traitements ont été caractérisés en fonction de leurs impacts sur les nutriments et les contaminants ainsi qu'en fonction de leur consommation d'énergie et de réactifs. Les urinofertilisants ont été caractérisés en fonction de leur concentration en nutriments, de leur efficacité fertilisante et des impacts associés au champ.

2/ Quelle est l'efficacité fertilisante des urinofertilisants en tant qu'engrais azotés ?

La seconde partie de la thèse vise à quantifier l'efficacité fertilisante azotée de ces différents urinofertilisants. Cette efficacité a d'abord été étudiée en conditions contrôlées en serre (Chapitre 3) afin de caractériser dans les mêmes conditions pédoclimatiques l'efficacité fertilisante en tant qu'engrais azoté de 9 urinofertilisants aux caractéristiques variées. Dans un second temps (chapitre 4), cette efficacité fertilisante a été mesurée au champ pour 4 urinofertilisants en conditions « réelles » d'application. Dans toutes les conditions, l'efficacité des urinofertilisants a été comparée à d'autres engrains organiques dont le lisier de bovins et à un engrais minéral.

3/ Quels impacts associés à l'apport d'urinofertilisants ? Focus sur la volatilisation ammoniacale

Une partie des impacts associés aux apports d'urinofertilisants concernent les contaminants (teneurs en éléments traces, chapitre 3 et pathogènes, chapitre 4). Ces contaminants ont été analysés dans l'ensemble des urinofertilisants utilisés en serre et au champ. La recherche des résidus de pharmaceutiques fait l'objet de travaux spécifiques dans le projet Agrocapi. Nous nous sommes focalisés dans cette thèse sur le risque de volatilisation de l'ammoniac (chapitre 4 et 5). Elle a été mesurée en conditions contrôlées pour l'ensemble des urinofertilisants étudiés et au champ pour l'urine stockée.

4/ Quels impacts environnementaux associés aux différentes filières de valorisation de l'urine ?

Enfin, le chapitre 6 porte sur une évaluation des impacts environnementaux de la production de céréales fertilisée ou non avec des urinofertilisants. Nous avons considéré trois filières de valorisation de l'urine et deux modes de production agricole (conventionnelle et biologique). Les impacts environnementaux de la toilette à la production de grains ont été pris en compte. Cette évaluation a été réalisée en utilisant la méthodologie de l'analyse de cycle de vie (ACV).

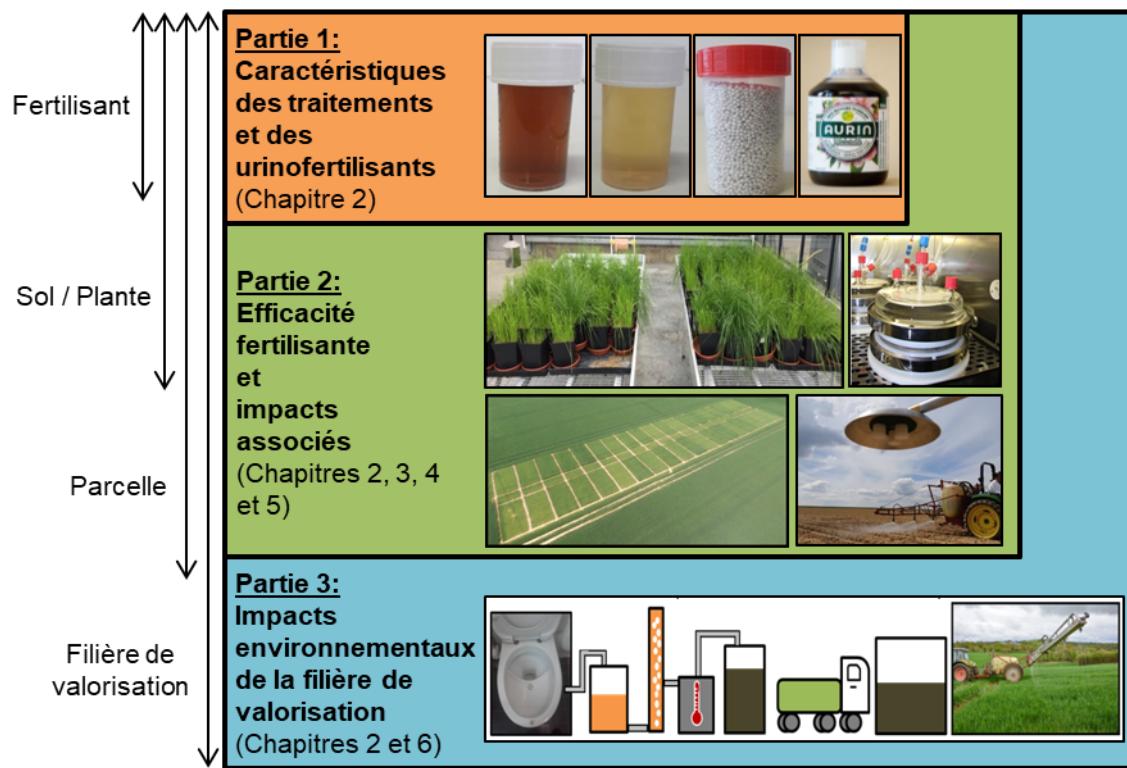


Figure 1-12. Organisation des différents chapitres du manuscrit. Credits : Tristan Martin, Christophe Dion.

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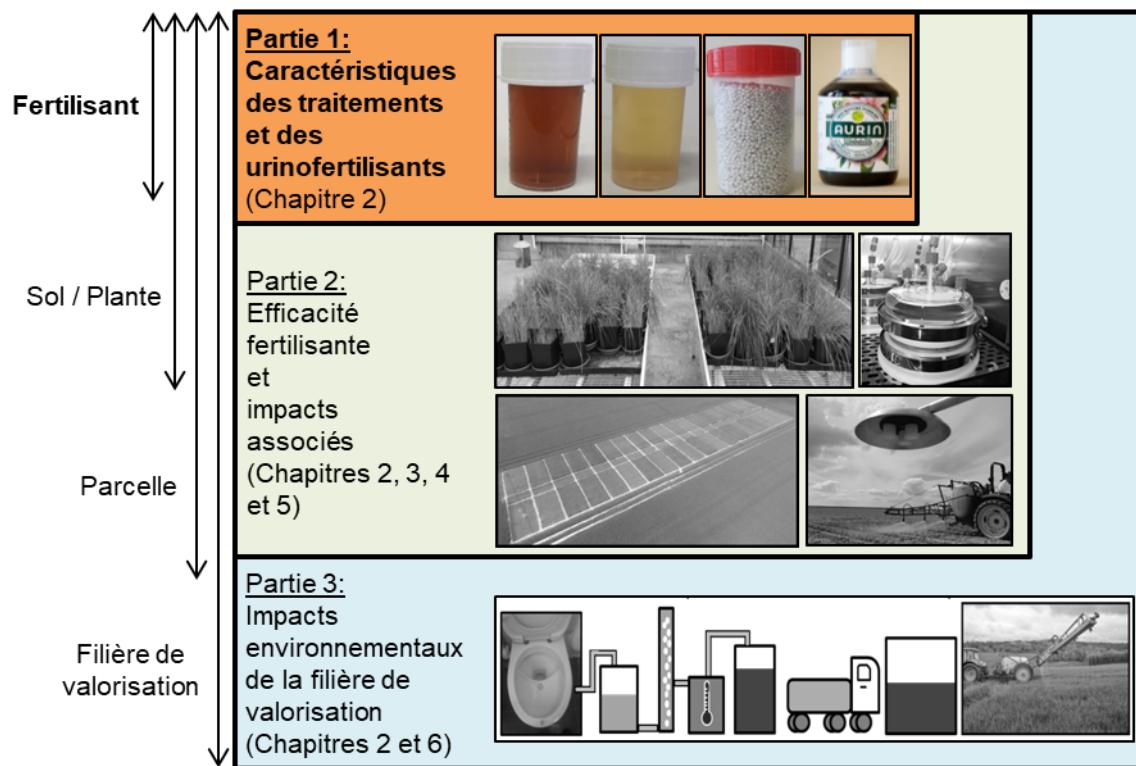
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Partie 1

Caractéristiques des traitements de l'urine et des urinofertilisants

Les traitements possibles de l'urine sont nombreux. La première partie de cette thèse correspond à une revue de la littérature des différents traitements possibles de l'urine et des urinofertilisants résultant de ces traitements (chapitre 2). La revue s'est concentrée principalement sur les traitements de l'urine qui conservent la plupart des nutriments. Les traitements ont été caractérisés en fonction de leurs impacts sur les nutriments et les contaminants ainsi qu'en fonction de leur consommation d'énergie et de réactifs. Les urinofertilisants ont été caractérisés en fonction de leur concentration en nutriments, de leur efficacité fertilisante et les impacts associés à leur apport au champ.



Chapitre 2

Revue des urinofertilisants

Human urine-based fertilizers: a review

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Abstract

Urine contains most of the nutrients excreted by humans. They are mainly released into the environment and contribute to the strong disruption of planetary biogeochemical cycles. These nutrients could be separated at source and used in agriculture. Once the urine is separated different treatments can be applied to stabilize nitrogen, reduce the volume, treat contaminants or extract nutrients. These treatments and the resulting fertilizers have different characteristics and environmental impacts that must be assessed.

We reviewed the characteristics of six treatments and the resulting fertilizers. The studied treatments were restricted to those that retain the most nutrients: storage, acidification, alkalization, nitrification and mixture with organic substrate. Phosphorus precipitation was also included, as it is one of the most developed treatments. Volume reduction post-treatments were also considered.

We compared these treatments and the resulting fertilizers in terms of nutrient forms and concentrations, fertilizer efficiency, contaminant removal, energy and chemical consumption. Although some parameters, such as fertilizer efficiency or pharmaceutical removal were in the same range, the nutrient concentrations and the energy and chemical consumption requested by fertilizer production varied widely. Holistic studies and pilot facilities implemented at a larger scale are needed in order to assess the whole value chain.

Key words

Contaminants; Human urine; Nutrients; Urine-based fertilizer; Urine recycling; Urine treatment;

1. Introduction

1.1. Disconnection between sanitation and agriculture

Today, in high-income countries, most wastewater is collected, but treatments vary in their efficiency, leading to various environmental impacts such as eutrophication (Sutton et al., 2011). In many cases, the denitrification of nitrogen is considered as the best sanitation practice. However, it is an energy-intensive treatment (Maurer et al., 2003) that hinders nitrogen recovery for agricultural use and emits greenhouse gases as nitrous oxides (Bollon et al., 2016). Phosphorus can be recovered from sewage sludge and thus be recycled in agriculture, whereas potassium is usually not treated and only little recovered. The nutrients present in wastewater have been largely considered pollutants for water bodies since the middle of the 20th century (Barles, 2005) and are only partially recycled in agriculture. Less than 10% of phosphorus present in wastewater is currently recycled worldwide (Cordell et al., 2009), and the figures are much lower for nitrogen, e.g. for a high-income country, less than 5% in Paris (Esculier et al., 2018).

Since the 20th century, an increasing amount of the nitrogen used in agriculture comes from atmospheric nitrogen fixed by the Haber-Bosch process; this process introduces twice as much nitrogen as biological fixation today (Sutton et al., 2013) and represents 70% of the nitrogen used in agriculture. However, this reaction requires a substantial amount of energy and represents approximately 1 - 2% of the current total world energy consumption (Matassa et al., 2015). Moreover, phosphorus and potassium fertilizers are fossil resources extracted from mines which are geographically unequally distributed (e.g. about three quarters of phosphate rock reserves are located in Morocco and Western Sahara, U.S. Geological Survey, 2019). This implies risks for food security and food resilience of countries. Some authors highlight that a peak production may be reached in the coming decades, especially for phosphorus (Cordell et al., 2009). Such situation may imply a higher fertilizer price and a higher trace metals content in phosphorus fertilizers due to lower quality phosphate rock (Cordell & White, 2011). The use of Haber-Bosch-produced nitrogen and mined phosphorus for crop production together with the poor recycling of nutrients from wastewater at the end of the food chain are the main disruptors of these open biogeochemical cycles and are currently transgressing the safe operating space of planetary boundaries (Steffen et al., 2015).

1.2. Human urine, the missing link

1.2.1. An essential part of wastewater

Urine is responsible for most of the nutrients present in wastewater, with 79% of the nitrogen, 47% of the phosphorus (even higher when phosphorus detergents are banned) and 71% of the potassium

(Friedler et al., 2013). These nutrients from urine are concentrated in a small volume (approximately 0.5 m³ of urine per year per person) in comparison to the whole volume of wastewater (approximately 55 m³ of wastewater per year per person in high-income countries). Urine is about 100 times more concentrated than domestic wastewater (about 9 g N/L in urine versus less than 100 mg N/L in wastewater, calculated from Friedler et al., 2013). The nutrients present in urine represent a huge amount of the total nutrient supply worldwide. Recycling all urine-excreted nutrients would represent about 19% of the current worldwide inorganic nitrogen inputs (recalculated from Trimmer et al., 2019, assuming 88% of human-excreted N nutrients in urine) and about 11% of the current global phosphorus demand (assuming 50% P of human-excreted nutrients in urine, Mihelcic et al., 2011). For example, in the Paris region (12 million inhabitants and 569 000 ha of cultivated lands, including 82% field crops), the nutrients present in the urine of the inhabitants of the metropolis could cover all nitrogen and half of the phosphorus currently spread in the fields (calculated from Esculier et al., 2018 and Union des industries de la fertilisation [UNIFA], 2018).

1.2.2. Fresh urine

Composition: Approximately 1.3 L (1.0 L to 1.5 L) of urine is excreted daily by the human body (Friedler et al., 2013). About 85% of the nitrogen is excreted as urea, 5% as ammoniacal nitrogen and 10% as other organic N, which is mainly composed of creatinine, uric acid and creatine (Rose et al., 2015; Udert et al., 2006). Phosphorus is mainly present in a dissolved form as phosphate (Udert et al., 2006). The pH of fresh urine is slightly acidic, approximately 6.2 (Udert et al., 2006). If the urine is not diluted, its daily mean concentration in high-income countries usually ranges between 8.8 - 9.2 g N/L for nitrogen, 0.74 - 2 g P/L for phosphorus and 2.2 - 2.7 g K/L for potassium (Maurer et al., 2006; Udert et al., 2006). The load of nutrients is correlated with the diet (Trimmer et al., 2019). Urine also contains many micronutrients that are needed for plant growth (Etter et al., 2015).

For organic matter, the chemical oxygen demand (COD) of urine is about 10 g O₂/L and 85% of the COD is easily degradable (Udert et al., 2006). Fresh urine contains more than 3,000 different compounds (urine metabolome, Bouatra et al., 2013), actually few studied.

Contaminants: Urine is not sterile at excretion for healthy adults and it can includes approximately 240 bacterial operational taxonomic units from diverse orders (Lahr et al., 2016). Some pathogens, such as *Schistosoma haematobium*, *Salmonella typhi*, *Salmonella paratyphi*, *Leptospira interrogans* can be naturally excreted in urine (Feachem, 1983). However, contamination of urine in pathogens mostly comes from fecal cross-contamination, which can occur at the diverting toilet (Höglund et al., 1998). Usually, urine concentrations of trace metal elements are low and lower compared to manure and mineral fertilizers (especially phosphate fertilizer, Ronteltap et al., 2007). Among the different contaminants found in urine, pharmaceutical residues seem to be of greatest concern because of their ecotoxicological potentials and relatively high concentration compared to other compounds. Lienert,

Bürki et al. (2007) have done a screening of the excretion pathways of 212 pharmaceutical active ingredients. On average, 64% of active ingredients were excreted in urine, of which 42% were in metabolized forms, generally less toxic (Lienert, Güdel et al., 2007). However, these proportions vary extremely depending on the compound. Pharmaceutical residues are present in “average urine” at concentrations from zero to several hundred µg/L (Winker et al., 2008) and even reach concentrations higher than a few mg/L for individuals under medication (Bischel et al., 2015). The need for specific treatments to remove pharmaceuticals before the application of urine to land is currently a topic of debate (World Health Organization, 2012; Winker, 2009).

1.2.3. Source separation and treatment

Source separation is needed to separate urine from other types of wastewater and to avoid the dilution of nutrients or contamination with pathogens (Johansson et al., 2000). Source separation can be performed in various types of toilets and urinals. The most common are waterless male urinals and urine-diverting toilets (Rossi et al., 2009). Some dilution can occur in diverting toilets equipped with a flush (Maurer et al., 2006). After separating the urine, treatments are possible to create different fertilizers that could be used in agriculture. The name “urine-based fertilizer” (UBF) identifies these fertilizers as resulting from urine treatments. Dozens of these treatments were developed since the 2000s, with different objectives as presented in Maurer et al. (2006):

Nitrogen stabilization: Most nitrogen is excreted in urine as urea but is rapidly transformed during urine storage into ammoniacal nitrogen due to the ubiquitous presence of urease enzyme secreted by microorganisms (Friedler et al., 2013). Ammoniacal nitrogen is very sensitive to volatilization due to the high pH in hydrolyzed urine which is close to the pKa of the $\text{NH}_4^+ / \text{NH}_3$ couple (9.2). Ammonia is responsible for the nitrogen losses after application and for a part of the malodor of urine (Rodhe et al., 2004; Hashemi & Han, 2017). Stabilization corresponds to processes that keep nitrogen into a form that limits losses during treatment and allows further volume reduction or limits losses during the other steps of the value chain such as the spreading on crops. Nitrogen can be stabilized for example, as urea, nitrates, ammonium or organic nitrogen.

Volume reduction: The water content of urine represents approximately 95% of the weight of fresh urine (Rose et al., 2015). Urine is a low-concentration fertilizer compared to typical mineral fertilizers. Volume reduction is useful for storage, transport and application in the field.

Nutrient extraction: Nutrient extraction may be used to separate nutrients from the influent and produce a concentrated fertilizer. Generally, these treatments aim to recover one specific or a few nutrients from the influent.

Pharmaceutical residues and / or pathogens treatment: The possible pharmaceutical residues or pathogens contamination in urine can be reduced through adequate treatments such as filtration or sorption processes.

This review focused on the first two objectives. Nutrient extraction and treatments for pharmaceutical residues are briefly presented in SM 2-1 and SM 2-2.

1.3. Scope of the study

The objectives of this study were to review existing treatments for urine and to analyze the properties of the resulting UBFs in terms of nutrient content and fertilizing efficiency as well as their contaminant content and other environmental impacts. As we choose to focus on using urine as a fertilizer in agriculture, we excluded treatments related to other types of uses such as electricity generation or water reuse from urine.

Various reviews were carried out on treatments allowing the use of urine nutrients as fertilizer (e.g. Chipako & Randall, 2020; Harder et al., 2019; Maurer et al., 2006; Udert et al., 2016). The recent review by Harder et al. (2019) presented a comprehensive review on all possible ways to recover nutrients. We decided to focus on treatments that produce fertilizers specific to urine and detailed them as it was done by Maurer et al., (2006) with updated data from recent researches. We wanted to put forward technical and practical information on the treatments and the resulting fertilizers including their effectiveness as fertilizers.

Most of the treatments were developed in high-income countries. Although they can be applied in developing countries (e.g. Pronk & Koné, 2009), this review focussed on the context of high-income countries. We highlighted the use of UBFs for grain crops as they use large amount of fertilizers over large areas.

The treatments were separated into two categories as done by Harder et al. (2019). Conservative treatments retain most of the nutrients in the final product. If a volume reduction is carried out, the water will be extracted and not the nutrients. On the opposite, extractive treatments aim to recover one or several nutrients present in the urine to obtain a concentrated or contaminant-free fertilizer. We focussed on conservative treatments since they offer the possibility of closing all nutrient cycles and limiting waste discharge. The treatments studied were storage, acidification, alkalinization, nitrification and the mixtures with solid organic substrates. Phosphorus precipitation was also included as it is one of the most studied extractive treatments. Conservative volume reduction post-treatments to remove water were also considered.

1.4. Literature recovery

Relevant articles were collected from the Web of Science and Google Scholar databases. The search terms “urine fertiliz[ser]” and “urine agriculture”, both separated by the boolean operator ‘OR’, were used. No restrictions on language, publication date or type of publication (e.g. gray literature, research project reports) were used. For literature on contaminants, the search terms “pharmaceutical”, “metal” and “pathogens” were added to the search string. Finally, the articles found in the reference lists of the first identified papers were added.

2. Characteristics of the treatments

The studied treatments and their value chains are summarized in Figure 2-1.

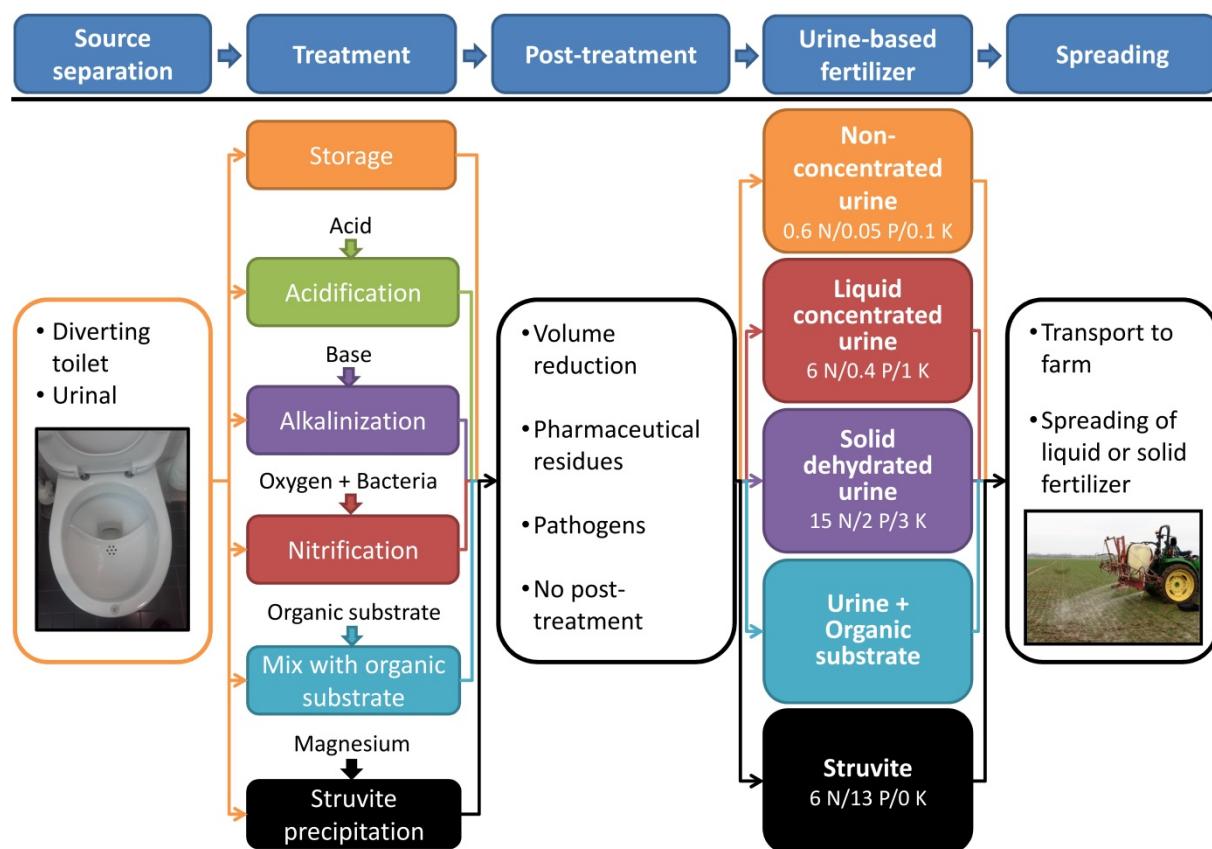


Figure 2-1. Summary of treatments and value chains studied. The nutrients concentrations (in % mass or g N/100 g raw material) in UBFs are based on average values from literature (see 3.1.1. Nitrogen) and theoretical pure dry struvite (Ahmed et al., 2018).

2.1. Storage

Urine storage was used since ancient times in several countries (Paulet, 1853) and more recently in high-income countries, for example, in ecovillages in Sweden or with waterless urinals during temporary events in France (e.g. Brun, 2017; Johansson et al., 2000). During storage and without

additional stabilization treatment, urea is rapidly hydrolyzed to ammoniacal nitrogen. The half-life of urea ranges from 40 years without urease enzyme to a few millisecond in its presence in high concentration (Senecal & Vinnerås, 2017). Hydrolysis of urea occurs more quickly in cases of fecal cross-contamination or presence of biofilms on pipe surfaces (Vinnerås, 2002). Usually, it occurs within a few days of storage (Udert et al., 2003). Storage in this study thus refers to the hydrolysis of urea and then the storage of hydrolyzed urine. The storage time ranges from a few hours (e.g. before treatment such as nitrification) to several months before the application on crops. After urea hydrolysis, approximately 90% of the nitrogen in urine is in ammoniacal form (Udert et al., 2006). The remaining nitrogen is in organic form. The hydrolysis of urea raises the urine pH up to 9.1 (Udert et al., 2006). Ammonia volatilization can occur during storage since the pH is close to 9.2, the pKa of the $\text{NH}_4^+ / \text{NH}_3$ couple. Ammonia exchange with air is slow, but losses can be high if the storage tank is not sealed (Udert et al., 2006). The increase in pH also promotes phosphorus precipitation (Friedler et al., 2013). Compared to fresh urine, approximately 30% of phosphorus precipitates as struvite [$\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$] and hydroxyapatite [$\text{Ca}_5(\text{PO}_4)_3(\text{OH})$] together with the calcium and magnesium present in urine, forming sludge at the tank bottom or deposits in urine pipes (Höglund et al. 2000). The sludge is usually mixed with the liquid part but it can potentially be managed separately from the liquid part as a phosphorus fertilizer, however no studies on this technique were found. Due to the presence of ammonia after urea hydrolysis and of various volatile compounds, the odor of stored urine is strong (Hashemi & Han, 2017; Troccaz et al., 2013).

Depending on the importance of nitrogen gaseous losses and phosphorus, magnesium and calcium precipitation during storage, the content in nutrients differs more or less from fresh urine. Depending on the collection method (e.g. flush toilet, waterless urinal), the concentration ranges between 1.8 - 9.2 g N/L for nitrogen, 0.1 - 0.5 g P/L for phosphorus and 0.8 - 3.3 g K/L for potassium (Maurer et al., 2006).

2.2. Acidification

Acidification stabilizes the nitrogen in urine. Experimental pilot or laboratory experiments were carried on this treatment (e.g. Boncz et al., 2016, Antonini, Nguyen et al., 2012). When applied on fresh urine (Hellström et al., 1999), acidification to below pH 4 prevents urea hydrolysis during storage and maintains nitrogen as urea (Boncz et al., 2016). The addition of approximately 60 mEq H⁺/L is recommended to inhibit urea hydrolysis for more than 100 days. Various strong or weak acids can be used such as sulfuric acid, acetic acid or citric acid (Antonini, Nguyen et al., 2012; Boncz et al., 2016; Saetta & Boyer, 2017). For concentrated strong acid such as sulfuric acid (e.g. 96%), 60 mEq H⁺/L represents about 3 g/L. A larger quantity of weak acids than of strong acids has to be added to provide the same amount of mEq (Boncz et al., 2016). Lactic fermentation can also be used to decrease the pH of fresh urine below 5 by adding easily degradable organic matter and lacto-

fermenting bacteria (Andreev et al., 2017). Stored urine can also be acidified to maintain the ammoniacal nitrogen in the NH_4^+ form rather than the NH_3 form (Antonini, Nguyen et al., 2012; Kirchmann & Pettersson, 1994). The optimal pH of acidified stored urine to avoid losses during further dehydration is 4 (Jiang et al., 2017). However, 10 times more acid (600 to 650 meq H^+/L) is needed to acidify stored urine due to the high buffer capacity of the hydrolyzed urine caused by the ammonia and carbonates formed after urea hydrolysis (Udert et al., 2006). Acidification may lower the organic content of the urine (Antonini, Nguyen et al., 2012). As odor is linked to the NH_3 content of urine (Hashemi & Han, 2017), the odor of the acidified urine is lower than that of the stored urine.

Theoretically, phosphorus precipitates may be redissolved if the stored urine is acidified, due to the pH decrease (Wang et al., 2006). However, the kinetics for dissolution are not known. Then, most of nutrients should be found in liquid phase. Dilution can occur when a diluted weak acid such as vinegar is used. Nutrients can also be added through acidification depending on the chosen acid (e.g. phosphoric acid).

2.3. Alkalization

Another way to stabilize nitrogen and keep it as urea form is to alkalinize the urine. Some pilot-scale prototypes are currently being field-tested in Finland, France and Sweden (e.g. Karlsson, 2019). Alkaline dehydration is moving toward the commercialization stage with a spin-off company from the Swedish University of Agricultural Sciences (SLU): Sanitiation360. An adjusted pH above 10 by adding a base inhibits the urease enzyme (Geinzer, 2017; Kabdaşlı et al., 2006). The base should be added to fresh urine (Simha et al., 2018); if added to hydrolyzed urine, the pH increase promotes NH_3 volatilization. Different types of base or alkaline media can be used, such as calcium, magnesium or potassium hydroxides, wood ash and alkalinized biochar (e.g. Dutta & Vinnerås, 2016; Simha et al., 2018). Urine can also be alkalinized using an anion exchange resin (Simha et al., 2018). An example of a readily available base is calcium hydroxide $[\text{Ca}(\text{OH})_2]$. The addition of 10 g $\text{Ca}(\text{OH})_2$ per liter of fresh urine represents a good safety margin for stabilization (Randall et al., 2016). This represents approximately 5 kg of $\text{Ca}(\text{OH})_2$ per year per capita. Wood ashes should be added at rates of 5 to 10% (weight to weight). In this case, 25 to 55 kg of ash is needed per year per capita (calculation from Senecal & Vinnerås, 2017; Simha et al., 2018). When $\text{Ca}(\text{OH})_2$ is used, 95% of the phosphorus precipitates, mostly as calcium phosphate (Flanagan & Randall, 2018). As for stored urine, the tank bottom sludge could potentially be managed separately as a phosphorus fertilizer. The odor of alkalinized urine in liquid or solid form is less strong than that of stored urine (personal test).

The concentration of nitrogen in the liquid phase of alkalinized urine is expected to be approximately the same as that in fresh urine. Since phosphorus dissolution depends on the pH, the phosphorus concentration in the liquid phase will vary accordingly.

2.4. Nitrification

To reduce NH₃ volatilization and odor and make volume reduction possible, nitrogen in stored urine can be stabilized through nitrification. A pilot plant was installed at the Swiss Federal Institute of Aquatic Science and Technology (EAWAG). Nitrification followed by distillation is moving toward the commercialization stage with a spin-off company from EAWAG: Vuna (<http://www.vuna.ch/>). This reaction acidifies the urine. Approximately 50% of the ammonium content of urine can be converted into nitrate before approaching pH 5.5, the limit for nitrifying bacteria (Etter et al., 2015). Bases such as NaOH or Na₂CO₃ can be added to complete nitrification (e.g. Jiang et al., 2011). Due to the supply of oxygen and aerobic conditions during nitrification, heterotrophic bacteria develop and degrade approximately 90% of the organic substances contained in urine (Fumasoli et al., 2016), decreasing the odor of urine (Etter et al., 2015). This also reduces the interactions between organic matter and any optional following treatments (e.g. adsorption onto an activated carbon filter to remove pharmaceutical residues), increasing their efficiency.

As for the acidification of urine, phosphorus precipitates may be redissolved due to pH decrease during nitrification (Wang et al., 2006).

2.5. Mixture with solid organic substrates

Mixing with organic substrates is widely applied in many contexts (e.g. dry toilets in all types of countries). Urine can be mixed with various organic substrates, such as compost (Shrestha et al., 2013), wood chips (Brun, 2017), sawdust (Brun, 2017), biochar (Bai et al., 2017) or rice straw (Hashemi & Han, 2018). Processes range from a simple impregnation of the organic substrates (e.g. adding urine to wood chips in dry toilet) to a complete transformation e.g. through composting (Brun, 2017; Shrestha et al., 2013). Mixing with organic substrates could be a way to reduce odor and ammonia emissions, as it is also used for the management of animal manure with straw or sawdust (van der Weerden et al., 2014). Nitrogen stabilization may occur due to the interactions with organic matter.

The organic substrate-urine mixtures generally contain fewer mineral nutrients than the other UBFs but contain more nutrients in organic forms. Therefore, the nutrient concentration in the fertilizer depends on the organic substrate used.

2.6. Phosphorus precipitation

Phosphorus precipitation is already implemented in some wastewater treatment plants (WWTPs), and pilot facilities on urine are running (e.g. Zamora et al., 2017). Phosphorus precipitation occurs due to oversaturation resulting from high pH and the presence of magnesium (Ronteltap, 2009) or calcium ions (Randall et al., 2016). In hydrolyzed urine, approximately 30% of phosphate precipitates

spontaneously due to pH increase after urea hydrolysis (Udert et al., 2006). Struvite is a salt composed of phosphate, magnesium and nitrogen that can precipitate in stored urine. Struvite precipitation can be increased by adding magnesium to the urine (Zamora et al., 2017). Then, the struvite precipitate can be filtered, washed with water and dried (Etter et al., 2011; Ganrot et al., 2008; Huang et al., 2019). Different forms of magnesium can be used, such as MgO, Mg(OH)₂, MgCl₂, and MgSO₄. Magnesium must be added at a ratio between 1:1 and 1.5:1 Mg:P to recover up to 99% phosphorus (Antonini et al., 2011; Etter et al., 2011; Ronteltap, 2009; Wilsenach, et al., 2007). It is also possible to use sea water, brine or even wood ash as magnesium sources for phosphorus precipitation (Liu et al., 2013; Sakthivel et al., 2012). Phosphorus precipitation can also be increased by adding calcium base in fresh urine to precipitate phosphorus as calcium phosphate but not in stored urine because it reacts with the carbonates resulting from urea hydrolysis, forming calcium carbonate (Randall et al., 2016). Potassium can also precipitate and be recovered with phosphorus as K-struvite [MgKPO₄·6H₂O] once there is no more ammonium in the solution (Wilsenach et al., 2007).

The precipitation of struvite allows the recovery of phosphorus but only a small fraction of nitrogen. The N:P ratio is about 0.5 in struvite when it is more than 10 in fresh urine (Ahmed et al., 2018). Pure dry struvite theoretically contains 5.7% nitrogen and 12.5% phosphorus (Ahmed et al., 2018). Phosphorus in struvite is more than 100 times more concentrated than it is in fresh urine (Friedler et al., 2013). However, the concentration and crystal structure can vary depending on the precipitation and drying processes, e.g. from 7.2 to 18% of P and the presence of dittmarite or nesquehonite in the crystal structure in examples from Antonini, Arias et al. (2012).

2.7. Volume reduction post-treatments

The water content of urine represents approximately 95% of the weight of fresh urine (Rose et al., 2015). Strauss (1985) mentioned that nitrogen represents 14 - 18% and phosphorus and potassium represent both 3.7% of the weight of dry solids in urine. The nitrogen concentration in dry urine can also be lower when chemicals are added. In the case of nitrified urine, the concentration of dry solids can go up to 24% of nitrogen and possibly higher since there is no more carbonate and less organic matter. (Udert & Wächter, 2012). Simha et al. (2020), also mentioned 24% of N in the total solid of urine. Post-treatments occur after storage and other treatments mentioned previously. These volume reduction post-treatments are characterized by their concentration factors, expressed as the ratio between the output and the input nitrogen concentrations. Considering the mean urine at 8.7 g N/L (Friedler et al., 2013), the theoretical concentration factor to reach 24% of nitrogen [dehydration] (Udert & Wächter, 2012) is 28. If the urine is flush-diluted to 1.8 g N/L (Etter et al., 2015), the concentration factor is 134 times. Various methods were developed for volume reduction for which energy use is a key factor. Their concentration factors and energy consumption are presented in Table 2-1.

Table 2-1. Concentration factor, energy and primary energy (PE) consumption of volume reduction post-treatments and some treatment compared to the production of nitrogen fertilizer, treatment of nitrogen in WWTP and transport.

| Process | Energy kWh/m ³ | Primary energy MJ/m ³ | MJ/kg N | Concentration factor | Comments | Reference |
|-------------------------------------|---------------------------|----------------------------------|---------------|----------------------|--|--|
| Evaporation | Solar still | 0 | 0 | Dehydration | 5.5 - 8.3% N in final product. Adding acid for stabilization. | Antonini, Nguyen et al., 2012 |
| | Non-optimized prototype | 3483 - 6333 | 40452 - 73548 | 3548 - 6452 | 5 Urine at 11.4 g N/L. 5.6% N in final product. Adding base for stabilization. 1100 - 2000 MJ/kg N*. | Karlsson, 2019 |
| | Pilot scale | 100* | 1161 | 208** | Dehydration 13.2% N in final product from Meyer et al. (2018). Adding base for stabilization. | Swiss Federal Institute of Aquatic Science and Technology (n.d.) |
| Distillation | Without energy recovery | 710* | 8245 | 1198 - 3450 | 35 - 67 Urine at 2.4 - 6.9 g N/L. 99.2% water removed. 16 - 24% N in final product. | Udert et Wächter, 2012. |
| | | 611* | 7095 | 1273** | Dehydration To remove 1m ³ . 2200 MJ/m ³ *. Considering electricity. | Tettenborn, 2011 |
| | | 722 | 8387 | 1505** | Dehydration To remove 1m ³ water. 2600 MJ/m ³ *. Considering electricity. | Maurer et al., 2006 |
| | | 3737 | 389* | 10 | Urine 9.6 g N/L. 9.7% N in final product. | Maurer et al., 2003 |
| | With energy recovery | 107* | 1243 | 302 - 699 | 20 - 25 Urine at 1.8 - 4.1 g N/L. 4.8% N in final product. Energy consumption recovered on graph. | Fumasoli et al., 2016 |
| | | 110* | 1277 | 186 - 534 | 35 - 67 Urine at 2.4 - 6.9 g N/L. 99.2% water removed. 16-24% N in final product. | Udert et Wächter, 2012 |
| | | 80* | 929 | 516 | 28 Urine at 1.8 g N/L. 5% N in final product. | Etter et al., 2015 |
| | | 77 - 110 | 894 - 1277 | 103- 147** | Dehydration 96% water removed. 277 - 396 MJ/m ³ * | Maurer et al., 2006*** |
| Forward osmosis | | 326 | 34* | 10 | Urine 9.6 g N/L. 9.7% N in final product. | Maurer et al., 2003*** |
| | | 42 - 50 | 184 - 581 | 87 - 104** | Dehydration Large scale. To remove 1 m ³ of water. 150 - 180 MJ/m ³ *. Considering electricity. | Maurer et al., 2006*** |
| | | 111 | 1290 | 231** | Dehydration To remove 1 m ³ of water. 400 MJ/m ³ *. Considering electricity. | Maurer et al., 2006*** |
| Reverse osmosis | 0.84* | 10 | 0.82 | 2 - 3 | Urine at 11.9 g N/L. Volume concentration factor (recovered on graph) is 2 to 4. Considering 20% N losses. | Zhang et al., 2014 |
| | 5 - 10* | 58 - 116 | 19 - 37 | 5 | Urine at 3.1 g N/L. | Maurer et al., 2003 |
| Freeze-Thaw | 30* | 348 | 97 | 5 | Urine at 3.6 g N/L. Volume concentration factor is 5. Considering 5% N losses. Adding acid for stabilization. | Ek et al., 2006 |
| | 1200* | 13935 | 2500** | 4 - 5 | Laboratory scale. Volume concentration factor. No N losses. | Gulyas et al., 2004 |
| | 244* | 2834 | 508** | 4 - 5 | 5000 cap scale. Volume concentration factor. No N losses. | Gulyas et al., 2004*** |
| Lyophilization | 30* | 348 | 62** | 4 - 5 | 500000 cap scale. Volume concentration factor. No N losses. | Gulyas et al., 2004*** |
| | 2511 | 29160 | 5231** | Dehydration | To remove 1 kg of water. 8100 kWh/m ³ PE*. | Udert et Wächter, 2012*** |
| Nitrification | 20 - 242 | 230 - 2809 | 128 - 685 | - | Urine at 1.8 - 4.1 g N/L. 11 - 59 kWh/kg N*. | Fumasoli et al., 2016 |
| | 50* | 581 | 323 | - | Urine at 1.8 g N/L. | Etter et al., 2015 |
| Struvite production | | 25* | - | | | Maurer et al., 2003 |
| N Fertilizer production | | 45* | - | | | Maurer et al., 2003 |
| Nitrification/Denitrification WWTP | | 45 - 109* | - | | | Maurer et al., 2003 |
| Transport of non-concentrated urine | 10 km | 48 | 6 - 27** | - | Non-concentrated urine at 1.8 - 8.7 g N/L. 4.8 MJ/ton/km. | Wilsenach & van Loosdrecht, 2006 |
| | 100 km | 480 | 55 - 267** | - | | |

Table footnote: Data recovered from literature are noted with *. The mean influent nitrogen concentration of all reviewed experiments in Table 2-1 was 5.6 g N/L. This value was used when the influent nitrogen concentration was not given in order to calculate the energy consumption per kg of nitrogen. Recalculated values using urine at 5.6 g N/L are noted with **. Conversion into primary energy is based on the European electricity mix (efficiency of 0.31, Maurer et al., 2003). Electricity was taken, if no energy source was given. References are noted with *** when process was not applied on urine. The energy consumption was recalculated in the same units for all treatments. The concentration factor was calculated as the ratio between the nitrogen concentration in the final products and the corresponding influents. When the concentration factor is given as a volume concentration factor, it was recalculated taking into account the expected nitrogen losses during the process.

2.7.1. Evaporation

Nitrogen is stabilized before evaporation to prevent gaseous ammonia emissions and losses of nitrogen. Then, the water can be evaporated by managing the temperature, pressure and hygrometry (e.g. passive evaporation, Bethune et al., 2015). Evaporation can be a simple volume reduction process (the final product stays liquid) or can continue until dehydration (the final product is a solid). When urea hydrolysis is inhibited, the evaporation should take place below 40 °C to 60 °C to prevent chemical degradation of the urea and losses (Randall et al., 2016; Simha et al., 2018, 2020). Nitrogen losses can reach about 5 to 30% during evaporation operating at temperature up to 60°C in a pilot experiment using alkalinized urine or solar still with acidified stored urine (Antonini, Nguyen et al., 2012; Simha et al., 2018, 2020). Chemical urea hydrolysis and volatilization are linked to the temperature and the duration of the treatment at this temperature. Reducing the temperature (Randall et al., 2016) or increasing dehydration rate (Simha et al., 2020) will reduce nitrogen losses.

After dehydration, the nitrogen concentration in the final product can be approximately 5 - 13% (Antonini, Nguyen, et al., 2012; Meyer et al., 2018) depending on the amount of chemical added (e.g. acid sulfuric, lime) and nitrogen losses.

2.7.2. Distillation

Water distillation consists of evaporation at high temperature (e.g. 80 °C) and low pressure (e.g. 500 mbar). To perform distillation, nitrogen has to be stabilized in a form other than NH₃ (to prevent volatilization) or urea (to prevent chemical hydrolysis at high temperature) with treatments such as nitrification or acidification. Nitrogen losses during the process are less than 3% for nitrified urine but can be higher for distilled acidified urine (Etter et al., 2015; Tettenborn, 2011; Udert & Wächter, 2012). Phosphorus losses during the process are driven by the pH and the concentration factor, but phosphorus is not likely to precipitate during distillation of acidified urine (pH 4.7 to 7.3, up to

60 g P/L, Tettenborn, 2011). Up to 50% of sodium chloride may be removed during distillation (through sequential distillation) because it is the first salt to precipitate; this reduces the risk of soil salinization after the use of UBFs (Etter et al., 2015). Energy recovery can be performed on the distiller using vapor compression or heat exchange, which reduces the energy consumption by up to 90% (Etter et al., 2015).

The nutrient concentrations of nitrified urine after distillation can reach 4 - 6% N, 0.2 - 0.4% P, and 2 - 4% K in liquid concentrate form (Fumasoli et al., 2016; Martin, 2018, considering a density of 1.14 in the final product, Etter B. [personal communication, November 14, 2018]) and 16 - 24% N, 2% P, and 7% K in dehydrated solid form (Etter et al., 2015; Udert & Wächter, 2012).

2.7.3. Forward osmosis

For forward osmosis, due to osmotic pressure, water is separated from the urine passing through a semipermeable membrane into a highly saline solution such as sea water (Zhang et al., 2014). This process requires a low amount of energy. However, the tested membranes show permeability to urea or NH₃ (Nikiema et al., 2017; Zhang et al., 2014), implying the need to use hydrolyzed acidified urine to obtain a higher retention rate for nitrogen (Volpin et al., 2019). The recovery rates for nitrogen depend on the nitrogen form, e.g. less than 50% for urea and between 31% to 91% for charged ammonium (Zhang et al., 2014). Recovery rates may be higher than 90% for phosphorus and potassium (Nikiema et al., 2017; Zhang et al., 2014). Forward osmosis show lower fouling potential than other membranes processes. Most of experiments use synthetic urine but experiments with real urine are necessary to assess membrane fouling (Volpin et al., 2018; Zhang et al., 2014). Progress is needed to produce membranes with high solute rejection. The volume concentration factor could ranges from 2.5 to 15 depending on how diluted the influent urine is (Zhang et al., 2014).

2.7.4. Reverse osmosis

During reverse osmosis, the water from urine passes through a semipermeable membrane due to the application of a pressure higher than the osmotic pressure. As for forward osmosis, the membranes have the tendency to be permeable to NH₃, so the hydrolyzed urine can be acidified to keep the nitrogen in NH₄⁺ form (Ek et al., 2006). In experiments using hydrolyzed urine, nutrient recovery ranges from 70% to more than 90% (Ek et al., 2006; Maurer et al., 2006). However, scaling problems (salt precipitation on the membrane) were reported (Maurer et al., 2006).

2.7.5. Freezing-thawing

As the solution is slowly frozen, ions and chemical compounds are excluded from the ice crystalline structure. When melted, the ions and compounds are the first to be found in solution and can be separated (Lind, Ban, & Bydén, 2001). Such a treatment enables the concentration of approximately

80% of the nutrients in 25% of the original volume of urine (Lind et al., 2001). In such a case, the volume concentration factor is between 3.2 and 5 (Gulyas et al., 2004; Lind et al., 2001).

2.7.6. Lyophilization

During lyophilization, water is sublimated at very low temperatures (less than -40 °C) and pressures (Udert & Wächter, 2012). Based on total dehydration, the concentration factor for lyophilization is the same as that for evaporation, but the process requires more energy (Udert & Wächter, 2012).

2.7.7. Membrane distillation

During membrane distillation, water is removed through a microporous hydrophobic membrane. Distillation is driven by the vapor pressure gradient induced by the temperature difference on each side of the membrane (e.g. Tun et al., 2016; Volpin et al., 2020). Operational temperature is lower than classic distillation. Then, energy consumption is expected to be lower. However fouling is an important issue and gaseous NH₃ can pass through the membrane (Derese & Verliefde, 2016; Tun et al., 2016). Treatment such as nitrification (Volpin et al., 2020), forward osmosis (Liu et al., 2016), acidification (of hydrolyzed urine) or filtration (Tun et al., 2016) aim to avoid NH₃ permeation or fouling on the membrane. Water removal can be up to 75% - 95% removal (Derese & Verliefde, 2016; Volpin et al., 2020). No energy data were found for membrane distillation applied to urine but energy consumption can vary a lot depending on the different types of technology. Zuo et al.,(2011) mentioned that the energy consumption of a large scale membrane distillation desalination plant can be more than 144 MJ/m³ of water removed (40 kWh/m³), but the environmental impact differs if a natural heating resource is used (e.g. solar, geothermal energy, Cabassud & Wirth, 2003).

3. Comparisons between treatments and resulting fertilizers

3.1. Nutrient concentration

3.1.1. Nitrogen

The UBFs were separated into three ranges of nitrogen concentrations, depending on how far the volume reduction was pushed after source separation of urine, and compared to mineral fertilizers and animal slurries in Figure 2-2.

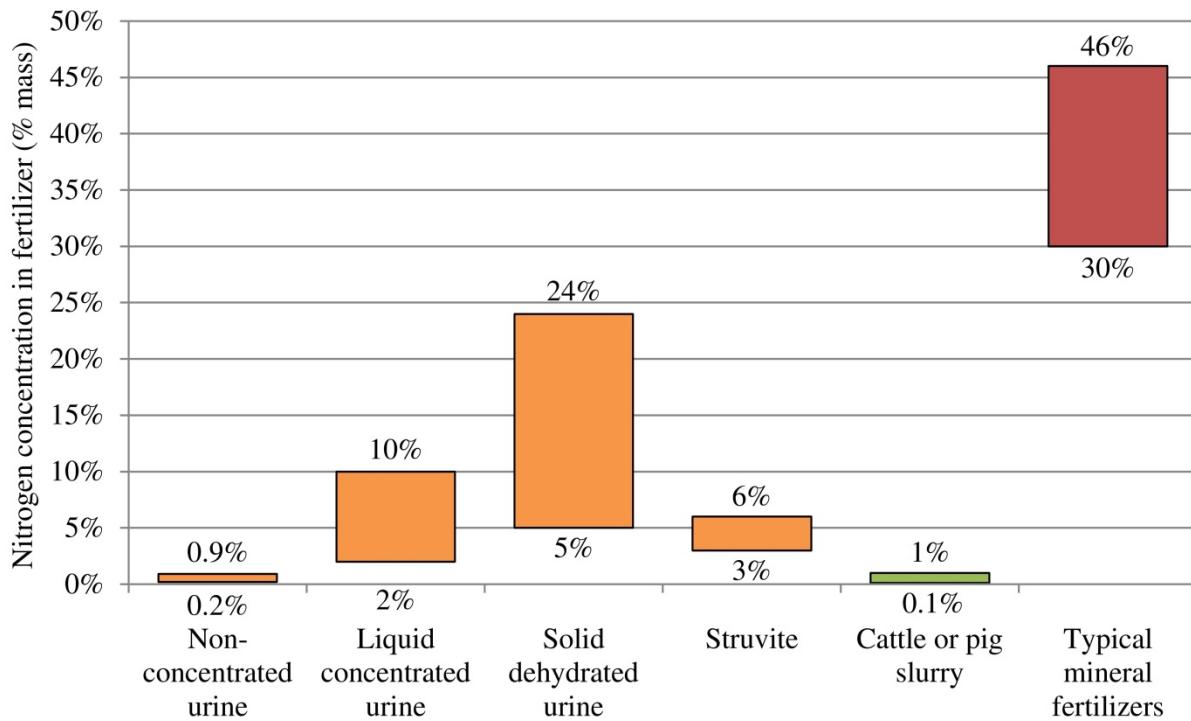


Figure 2-2. Nitrogen concentration in UBFs (in % mass or g N/100 g raw material), compared to nitrogen contents in animal slurries and in the most frequently used mineral fertilizers for grain crops in France : ammonium nitrate, urea ammonium nitrate solution and urea (UNIFA, 2018). Cattle and pig slurry average nitrogen concentration come from Benoît et al., (2014). Non-concentrated urine is based on fresh urine and urine diluted by flushing (Maurer et al., 2006). Liquid concentrated urine is based on reverse osmosis and distilled acidified urine (Maurer et al., 2003), density of 1 is assumed. Solid dehydrated urine is based on acidified dehydrated urine (Antonini, Nguyen et al., 2012) and nitrified dehydrated urine (Udert & Wächter, 2012). Struvite concentrations come from Antonini, Arias et al. (2012).

Non-concentrated urine and animal slurries are in the same ranges, with much lower nitrogen contents than mineral fertilizers. Liquid concentrated urine is approximately 10 times more concentrated but remains below the level of mineral fertilizers. Only dehydrated urine reaches concentrations close to those of mineral fertilizers. Struvite contains nitrogen but is mainly a phosphorus fertilizer. Mixtures of urine and solid organic substrates are not presented, as the nitrogen content varies considerably depending on the mixture. Indeed, the nitrogen forms present in UBFs vary depending on the fertilizer (Figure 2-3).

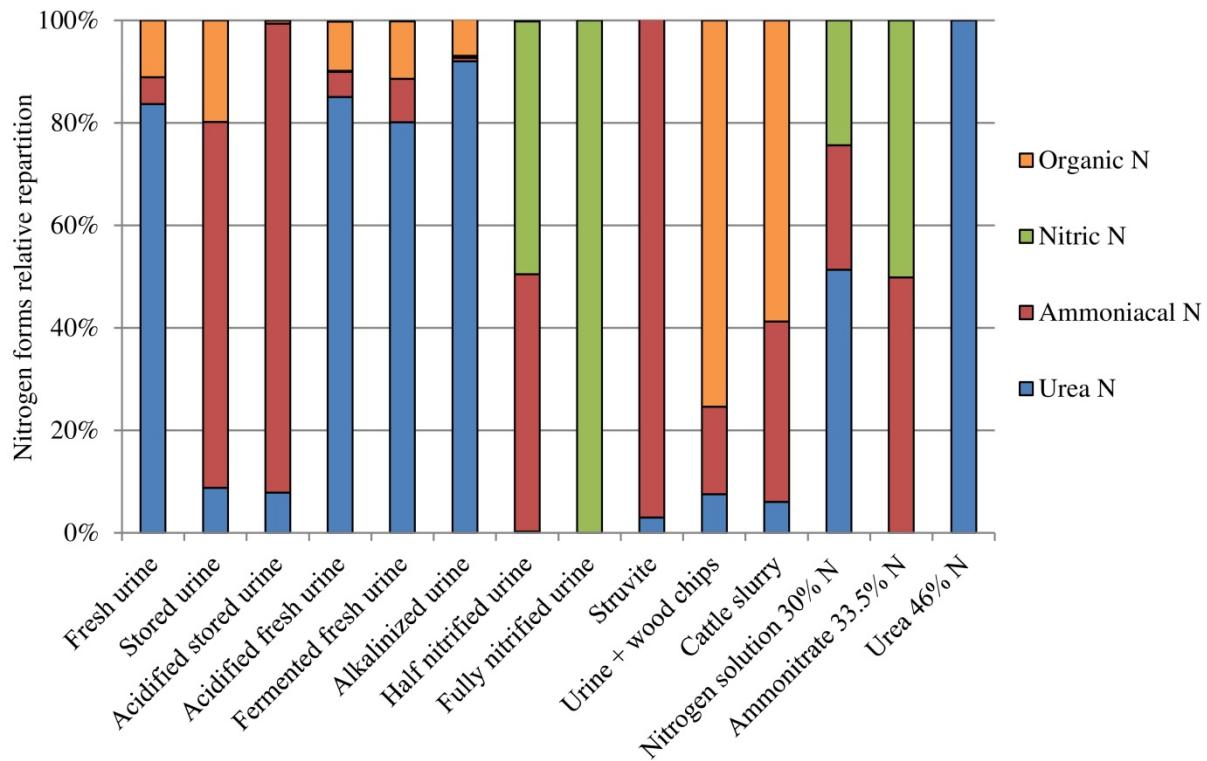


Figure 2-3. Nitrogen forms relative repartition in the different UBFs, cattle slurry and mineral fertilizers. Data for UBFs and cattle slurry are based on Jiang et al. (2011), Martin et al. (2020) and Udert et al. (2006). Urine was acidified using sulfuric acid and alkalinated using lime. Data for mineral fertilizers are based on the 3 most sold nitrogen fertilizers in France for grain crops (UNIFA, 2018).

Except in the urine-organic substrate mixtures, the nitrogen forms are similar to those found in typical mineral fertilizers (urea, ammoniacal and nitrate). Urea is the main form of nitrogen present in fresh urine or in stabilized products after acidification or alkalination of fresh urine. Stored urine, acidified stored urine and struvite are dominated by ammoniacal nitrogen. The pH has a strong influence on the ammoniacal nitrogen form ($pK_a [NH_3 / NH_4^+] = 9.2$). Considering pK_a , NH_3 may represent more than one third of the total nitrogen in stored urine at pH 9.1 and less than 1% of the total nitrogen in acidified urine at pH 6.5. Generally, the organic nitrogen content is lower in UBFs than in slurry, except in mixtures of urine and organic substrates. Chemicals used to stabilize urine can have an impact on the nitrogen forms repartition (e.g. base or acid which contain nitrogen).

3.1.2. Phosphorus and other nutrients

As for nitrogen, the contents and forms of phosphorus differ among UBFs. Considering an N:P ratio of 11.8 (Friedler et al., 2013), the phosphorus concentration is between 0.02 - 0.08% for non-concentrated urine, 0.3 - 0.5% for liquid concentrated urine, and 0.4 - 3.7% for solid dehydrated urine (considering dry solid urine with the upper value of Strauss, 1985). In the case of stored or alkalinated urine, a large part of the phosphorus precipitates and may be managed separately.

Considering a 4.2 N:K ratio (Friedler et al., 2013), the potassium concentration is between 0.05 - 0.2% for non-concentrated urine, 0.7 - 1.4% for liquid concentrated urine, and 1.2 - 5.7% for solid dehydrated urine. Most often, potassium is not affected during the treatments, except in a few cases, e.g. the addition of sulfuric acid followed by distillation may make potassium precipitate as potassium sulfate (Tettenborn, 2011) or precipitation of K-struvite when there is no more nitrogen in solution. Some micronutrients can come from the different chemicals used, such as calcium from lime, phosphorus or potassium from ash or sulfur from sulfuric acid; this may explain the values sometimes higher than those given by Strauss (1985). For pure struvite, the phosphorus concentration is approximately 12.6% (Ahmed et al., 2018), which is in the same range as that in common phosphorus mineral fertilizers (e.g. simple superphosphate, 8% P or 18% P₂O₅, or triple superphosphate 20% P or 45% P₂O₅).

3.2. Fertilizing efficiency and related impacts

3.2.1. Nitrogen and phosphorus use efficiencies

UBFs and especially stored urine were tested on various crops (e.g. Karak & Bhattacharyya, 2011; Pandorf et al., 2019). Table 2-2 provides data about the nutrient use efficiency of different UBFs. Different methods can be used to calculate these efficiencies. When nutrient use efficiency (NUE) is not directly given, it was calculated as the ratio between the additional nutrient uptake by the fertilized crop compared to the uptake by the control crop without fertilization and the nutrient added by the fertilizer (Equation 1):

$$NUE (\%) = \frac{Nutrient\ uptake\ fertilized\ crop - Nutrient\ uptake\ control\ crop}{Nutrient\ added\ by\ fertilizer} \times 100 \quad (1)$$

If no data were available about nutrient exportation, NUE was calculated using the additional yield increase in the fertilized crop compared to the yield of the control crop without fertilization.

The nutrient use efficiency of UBF can be compared to that of mineral fertilizer by calculating the mineral fertilizer equivalence (MFE) as the ratio between the NUE of UBF and that of a reference mineral fertilizer as ammonium nitrate (Equation 2):

$$MFE (\%) = \frac{NUE\ Urine-based\ fertilizer}{NUE\ Mineral\ fertilizer} \times 100 \quad (2)$$

Nitrogen: The nitrogen NUE of UBFs is similar or slightly lower than that of mineral fertilizer and the MFE is high (generally higher than 75% and close to 100%). The differences among acidified, nitrified and stored urine (no nitrogen MFE data for alkalinized urine) are low. However, MFE seems to be slightly higher for nitrified urine or acidified urine compared to stored urine. Alkalized urine was little studied, however tests performed with alkalinized urine have shown positive impacts on crop yield (Filling, 2018). The MFE of urine-organic substrate mixtures may be lower due to the possible

fixation of the nitrogen from urine with the organic substrate (e.g. Martin, 2018). Other mixtures such as compost supplemented with urine have a positive short-term impact on yield (Fatunbi, 2009; Shrestha et al., 2013). The nitrogen efficiency is generally higher than for animal slurries and manure (Martin, 2018; Pradhan et al., 2011).

Phosphorus: Phosphorus availability depends on many parameters. UBFs are generally compared to calcium superphosphate or other soluble phosphate. However, these two mineral fertilizers have different NUE, which can impact the MFE values of UBFs. Because of its high phosphorus content, struvite is mainly used as a phosphorus fertilizer. Different tests were performed with different crops (e.g., Meyer et al., 2018 for dehydrated alkalinized urine). Phosphorous availability also strongly depends on the soil type. In the case of struvite, its efficiency is much higher in acidic soils than in alkaline soils. The MFE varies more for phosphorous than for nitrogen but also remains close to 100% compare to soluble phosphate. Some of the UBFs as struvite may act as a slow-release fertilizer (Everaert et al., 2017).

To summarize, the efficiencies of UBFs are close to those of mineral fertilizers for both phosphorus and nitrogen. However, to allow a clear comparison of the different UBFs, an experiment comparing all UBFs under the same conditions to avoid pedo-climatic and cropping system variations is needed.

Table 2-2. Examples of pot and field agronomical trials using UBFs on various crops. Efficiency is characterized by the nitrogen (N) and phosphorus (P) mineral fertilizer equivalence (MFE). Values recovered from literature are noted with *. Calculated values are noted with **.

| Product | Crop | Type / Element | MFE | Other results and comments | Reference |
|---------------------------------------|---|--------------------------|--------------------------------------|--|--------------------------------|
| Stored urine | Barley | Pot N P | 79%** 128%** | Recalculated from N and P exportations. Compared to K ₂ PO ₄ (soluble phosphate) for phosphorus. Soil pH: 6.2 | Kirchmann and Pettersson, 1995 |
| Stored urine | (1) Winter wheat (2) Oats | Field Yield | 60 - 80%* 50 - 60%* | Organic farming on 4 sites. Calculated as the yield ratio between urine and mineral fertilizer (tested on another conventional site). | Linden, 1997 |
| Stored urine | Spring barley | Field Yield | 80-90%* | MFE using grain yield. 3 years of experiment. | Johansson, 2000 |
| Stored urine | (1) Spring wheat, (2) winter rye, (3) maize. Also tested on oats, oil rape seed and hemp. | Field N | (1) 107%** (2) 86%** (3) 64%** | Calculated using N exportation from all plant part. When different doses of nitrogen are given, the slope of the response curve of N exportation according to N input was use as NUE. | Muskolus, 2007 |
| Stored urine | (1) Maize, (2) tomato. Also tested on carrot and beetroot. | Pot N | (1) 104%** (2) 70%** | Calculated using nutrient exportation from all crop part (e.g. fruit, leave) and the slope of the response curve of N as NUE. | Mnkeni et al., 2008 |
| Stored urine | (1)Winter barley (2) Italian ryegrass | (1) Field N (2) Pot N | (1) 96 - 101%** (2) 91%** | Recalculated using N exportation and N use efficiency (recovered on graph). 2 years of experiments. | Simons, 2008 |
| Stored urine | Spring oats | Field N | 64 - 82%* | 2 years of experiment. | Gómez-Muñoz et al., 2017 |
| Stored urine | English ryegrass | Pot N | 86 - 87%* | 2 doses of nitrogen. | Martin, 2018 |
| Stored urine | English ryegrass | Pot N P | 103%** 74%** | Ratio between nitrogen exportation of urine and mineral fertilizer treatments. Exportation calculated from yield (recovered on graph) and nutrients concentrations in tissues. No ONOP control. Mineral fertilizer: 2:3:2 NPK. Acidic soil (pH 4). | Mchunu et al., 2018 |
| Stored urine | Spring barley | Field Yield | 74 - 114%** | MFE calculated using grain yield (recovered on graph). Mineral fertilizer: N+Mg and 23:3:8 NPK. 2 doses of nitrogen. | Viskari et al., 2018 |
| Acidified urine | Italian ryegrass | Pot N | 102%* | Efficiency higher than stored urine (MFE 91%) | Simons, 2008 |
| Alkalinized urine | Italian ryegrass | Pot P | 83%* | Compared to soluble P: Ca(H ₂ PO ₄) ₂ H ₂ O. Alkaline soil (pH 7.7) | Meyer et al., 2018 |
| Nitrified urine | Italian ryegrass | Pot N | 97%** | Calculated as ratio of N fertilizer recovery in plants from urine and mineral fertilizer. N input not equals. Acidic soil (pH 5.4). | Bonvin et al., 2015 |
| Nitrified urine | English ryegrass | Pot N | 94 - 99%* | 2 doses of nitrogen. | Martin, 2018 |
| Nitrified urine | English ryegrass | Pot N P | 127%** 104%** | Ratio between nitrogen exportation of urine and mineral fertilizer treatments. Exportation calculated from yield (recovered on graph) and nutrients concentrations in tissues. No ONOP control. Mineral fertilizer: 2:3:2 NPK. Acidic soil (pH 4). | Mchunu et al., 2018 |
| Compost impregnated with stored urine | English ryegrass | Pot N | 72 - 73%* | Calculated only with N from urine. Efficiency lower than stored urine alone (MFE 86 - 87%). 2 doses of nitrogen. | Martin, 2018 |
| Struvite | Italian ryegrass | Pot P | 97%* | Compared to soluble P: KH ₂ PO ₄ . Acidic soil (pH 5.4). | Bonvin et al., 2015 |
| Struvite | English ryegrass | Pot P | 90 - 109%* | 9 struvite not from urine. Efficiencies recovered from "apparent effectiveness" compared to Ca(H ₂ PO ₄) ₂ . Soil pH: 6.6 - 7.1. Efficiency better than iron and calcium phosphate from WWTP. | Johnston & Richards, 2003 |
| Struvite | Italian ryegrass | Pot P | (1) 66% - 75%* (2) 104%* | Recovered on graph. Compared to soluble P: Ca(H ₂ PO ₄) ₂ H ₂ O. (1) Alkaline soil (pH 7.7). (2) Acidic soil (pH 5.1). | Meyer et al., 2018 |
| Struvite | (1) Italian ryegrass, (2) red clover. Also tested on grassland. | Pot P | (1) 150 - 175%* (2) 98 - 144%* | Compared to superphosphate. Soil pH: 6. Considering struvite powder from urine. Efficiency generally higher than precipitated P from WWTP. 2 year of experiment. | Simons, 2008 |

3.2.2. Application

Based on the different forms of UBFs (liquid concentrated, solid, etc.), the quantities required to provide 170 kg N/ha are presented in Table 2-3.

Table 2-3. Examples of product application rates to provide 170 kg N/ha. This is the highest dose authorized for an organic fertilizer in the European nitrate directive (Council Directive 91/676/EEC, 1991) and also represents an average nitrogen dose for grain crop in conventional agriculture in temperate climate such as France. Fertilizers concentrations are taken from Figure 2-2. The exact concentrations were added when it was given in the references.

| Fertilizer | Fertilizer nitrogen concentration (% N and g N/L) | | Quantity spread to bring 170 kg N/ha (ton/ha) | | Spreading machinery |
|---------------------------|--|----------------------|---|------|----------------------------------|
| | min | max | min | max | |
| Non-concentrated urine | 0.2% (1.8 g N /L) | 0.9% (8.7 g N /L) | 20 | 94 | Slurry spreader |
| Liquid concentrated urine | 2% (15.5 g N /L) | 10% (96.5 g N /L) | 1.7 | 11 | Liquid sprayer / Slurry spreader |
| Solid dehydrated urine | 5% (54.6 g N/kg) | 24% | 0.7 | 3.1 | Solid spreader |
| Struvite | 3% (29.3 g N/kg) | 6% (60.0 g N/kg) | 2.8 | 5.8 | Solid spreader |
| Cattle slurry | 0.1% (0.9 g N/kg) | 1% (5.4 g N/kg) | 31 | 189 | Slurry spreader |
| Mineral fertilizer | 30% | 46% | 0.37 | 0.57 | Liquid sprayer / Solid spreader |

The quantities range from 0.7 t/ha for dehydrated urine to 94 t/ha for flush-diluted urine. Non-concentrated urine may be spread using a slurry spreader. Liquid concentrated urine has a nitrogen concentration that can be suitable for both liquid mineral fertilizer sprayers and slurry spreaders, depending on the dose needed. Dehydrated products and struvite are more similar to typical mineral fertilizers and may be applied with the same type of equipment if they are transformed into an adapted form (e.g. granular, pellet). It seems possible to replace all mineral fertilizers with dehydrated urine, highly concentrated urine or struvite at similar applied volumes and working times. However, complete fertilization with non-concentrated urine in an intensive cropping system would require approximately 30 to 250 more quantities to be spread compared to the amount of mineral fertilizer required, which represents a considerable amount of time with identical equipment and increased risks of soil compaction.

3.2.3. Gaseous emissions

Ammonia volatilization: Ammonia volatilization causes air pollution (e.g. acidification, particulate matter). Volatilization depends highly on the product characteristics (e.g. pH, NH₃ concentration), application techniques and pedo-climatic conditions (Huijsmans, 2003). For stored urine, using trailing hoses followed by harrowing 4 hours later, ammonia emissions from open soil range from 2% to 10% of the nitrogen applied and only from 0.3% to 1.1% with trailing shoes. Volatilization is very low for both techniques when urine is applied on growing crops (0.2 to 0.4% of nitrogen applied, Rodhe et al., 2004). Ammonia emissions from other products were not studied, and great diversity is expected due to the various characteristics of the products. NH₃ volatilization is expected to be lower for nitrified urine and acidified than for other products due to the lower ammonia concentration and low pH. Alkalized urine may have a volatilization rate as high as that of granular urea since increasing pH near the granule (due to urea hydrolysis) can promote volatilization (Fenn & Hossner, 1985). The other volatile organic compounds (VOCs) were barely studied but may imply some environmental impacts. For example, VOCs from slurry may contribute to the formation of tropospheric ozone (Liu et al., 2018).

N₂O emissions: Nitrous oxide is a greenhouse gas with a strong global warming potential (265 times higher than that of CO₂). N₂O emissions after UBF application were little studied. In an incubation experiment, Gómez-Muñoz et al. (2017) observed significantly lower emissions from stored urine (and cattle slurry) than from mineral fertilizer or other organic products. Simons (2008) measured N₂O emissions ranging from 0.1 to 0.8% of nitrogen applied in a pot experiment. This rate was higher than that for mineral fertilizer (0.1 - 0.3% of nitrogen applied). N₂O emissions were slightly lower for acidified urine than for stored urine in this experiment. Further studies on N₂O field emissions are needed, as well as N₂O emissions during nitrification.

3.2.4. Other impacts linked to field application

Impacts on earthworms: Cattle slurry and animal urine have toxic effects on earthworms because they contain ammonia, benzoic acid and sodium sulfide (Muskolus, 2007). Stored urine has a strong impact on earthworm abundance after application and was qualified as “toxic for earthworms” immediately after application (Muskolus, 2007). The effects decrease with time but can last more than six months under certain conditions. The liquid form of urine infiltrates earthworm holes, and the direct contact with earthworms may induce toxicity. Tillage shortly before or just after application may limit this impact (Muskolus, 2007). The toxicity may also be lower with a solid product. To our knowledge, the impacts of other UBFs on earthworms were not studied.

Seed germination: The application of stored urine may reduce the germination of some plants depending on the experimental conditions, e.g. on hemp in pot conditions or direct application on wheat or sunflower in Petri dishes (Muskolus, 2007; Winkler et al. 2013). No effect was observed in the field, as the urine is buffered by the soil (Viskari et al., 2018; Winkler et al., 2013). The high salt concentration in “pure urine” seems to have a strong negative impact on seed germination (Simons, 2008). Lacto-fermentation of urine before application increases the germination of radish seeds compared to that after the application of stored urine in Petri dish experiments, probably due to the lower ammoniacal nitrogen content in the fermented urine. The pH may also play a role in the germination process (Andreev et al., 2017). No study has shown negative effects on seed germination after struvite application (Gell et al., 2011). The impacts of other UBFs were not studied.

Soil salinization: Fresh urine contains a lot of different salts (Beler-Baykal et al., 2011). The main ions involved in soil salinization are sodium, boron, chloride and sulphates (World Health Organization, 2012). Salinity can affect soil structure and plant growth (Friedler et al., 2013). The concentration in salt and especially in sodium (linked to high levels of table salt in most diets) may be higher than in animal slurry with for example 2.6 g/L in urine (Udert et al., 2006) versus 0.9 g/L in pig slurry (Moral et al., 2008). Salinization generally occurs in semi-arid and arid areas where salts are not lixiviated by the rain. In areas where there are soil salinization problems, caution should be taken before using urine as fertilizer because of its high salt content (Jönsson et al., 2004). High rate of fertilization using urine may imply higher sodium uptake by the crops and may have a negative impact on yield. Then urine may not be used with salt-sensitive crops especially when the soil is sensitive to salinization (Mnkeni et al., 2005, 2008). To our knowledge, no problems were reported in other areas.

Soil acidification: Soil acidification involves complex physico-chemical processes (Bolan & Hedley, 2003). The use of animal urine or mineral nitrogen fertilizers acidifies the soil after application (Black, 1992). Therefore, the application of human urine can be expected to acidify the soil as well. Acid added for the treatments may increase acidification while in areas where the soil is acidic and needs to be limed, the high pH of the alkalinized urine could be beneficial.

More broadly, the long-term impacts of urine application were barely studied. To our knowledge, only one long-term trial is documented (Crucial field test in Denmark, Gómez-Muñoz et al., 2017).

3.3. Contaminants

3.3.1. Trace metals

There is little data on the behavior of trace metals during the treatments. However, trace metal contents are generally low in UBFs, most often lower than in mineral fertilizers such as phosphate rock or cattle slurry, although a fraction of the metals contained in urine may precipitates in struvite (Ronteltap et al., 2007). These contents generally meet regulations for fertilizers or the use of sewage sludge in agriculture (Antonini et al., 2011; Gell et al., 2011, Fumasoli et al., 2016). However, contamination can potentially come from the chemicals used for treatment (e.g. ash for alkalinization, Senecal & Vinnerås, 2017; Simha et al., 2020).

3.3.2. Pharmaceutical residues

The panel of pharmaceutical residues studied is very diverse, including more than 100 substances and metabolites. The effect of storage on pharmaceutical residues is poorly studied. Treatment efficiencies for reducing pharmaceutical residues were classified on a 3-step scale in Table 2-4. Since no reduction guidelines were found, it is difficult to define which concentration reductions are suitable for agricultural use.

All treatments and post-treatments have an impact on pharmaceutical residues, but the impact varies depending on the compound considered (e.g. Schürmann et al., 2012). However, none of the treatments allows high pharmaceutical removal, except specific pharmaceutical post-treatments. Storage is not sufficient to remove pharmaceutical residues and some authors mentioned that the urine of people under medication should not be used in agriculture (Jaatinen et al., 2016; Winkler et al., 2009). The impact of acidification and alkalinization also appears to be small. Nitrification may increase the degradation of pharmaceutical residues compared to storage. The behavior of pharmaceuticals during struvite precipitation is highly dependent on the compound (Kemacheevakul et al., 2012). However, the application of struvite (derived from swine wastewater) can increase the abundance and diversity of antibiotic resistance genes in soil (Chen et al., 2017). We highlight that there is high variability between studies on the same treatment. For example, Gajurel et al. (2007) found no significant reduction in diclofenac during a one-year storage experiment at pH 4, while Butzen et al. (2005) and Schürmann et al. (2012) found over 80% degradation of diclofenac at pH 2 to 3 within 6 to 11 months.

Table 2-4. Efficiency of treatments on pharmaceutical residues. Weak (+): Low impact on pharmaceutical residues. Removal can be high for some compounds and low for other. Moderate (++): Significant removal for most pharmaceutical residues. Strong (+++): Strong impact on short term for most pharmaceutical residues.

| Treatment | Efficiency | Examples | Reference |
|---------------------------------|--------------|--|--|
| Storage | + | - Reduction from 0% (e.g. bezafibrate) to 35% (e.g. tetracycline) of 10 compounds during 11 months storage. | Butzen et al., 2005 |
| | | - Reduction from 79% to 96% of 2 hormones during 6 weeks storage. | Zanchetta et al., 2015 |
| | | - Reduction from 24% (e.g. trimethoprim) to more than 99% (e.g. rifampicin) of 8 compounds during 6 months storage. | Jaatinen et al., 2016 |
| Acidification | + | - Reduction from 0% (e.g. bezafibrate) to 30% (e.g. tetracycline) of 10 compounds during 11 months storage at pH 4. Near 100% reduction (e.g. diclofenac) for some compound at pH 2. | Butzen et al., 2005 |
| | | - No significant reduction of 4 compounds during 1 year storage at pH 4. | Gajurel et al., 2007 |
| | | - Reduction from less than 10% (e.g. tramadol) up to more than 80% (e.g. diclofenac) of 8 compounds during 6 months storage at pH 3. | Schürmann et al., 2012 |
| Alkalization | + | - Reduction from 30% to 42% of 2 hormones during 6 weeks storage at pH 2. | Zanchetta et al., 2015 |
| | | - Reduction from less than 10% (e.g. ibuprofen) up to more than 80% (e.g. sulfadimidine) of 8 compounds during 6 months storage at pH 11. | Schürmann et al., 2012 |
| | | - No significant reduction of 4 pharmaceutical during 1 year storage at pH 10. | Gajurel et al., 2007 |
| Nitrification | ++ | - Estimated half-lives of 10 compounds during nitrification range from 40 min. (e.g. atazanavir) to more than 48 h (e.g. diclofenac). | Etter et al., 2015 |
| Mix with organic substrate | Unknown | - Not studied. | No reference found |
| Precipitation of struvite | ++ | - More than 98% of the 7 compounds studied remained in solution. - From 10 compounds, only two were found in struvite at low concentration (less than 5% of the initial amount) and one at high amount (up to 98% of the initial amount). The 7 other were not found in struvite. | Ronteltap et al., 2007 Kemacheevakul et al., 2012 |
| Volume reduction Post-treatment | Unknown to + | - Reduction from 88% to 97% of 2 hormones during evaporation of stored and acidified urine (pH 2). - No significant reduction (e.g. phenazone) to complete reduction (e.g. β -Sitosterol) for the distillation of acidified urine. - Not studied for other volume reduction treatments on urine. | Zanchetta et al., 2015 Tettenborn, 2011 |
| Pharmaceutical Post-treatment | +++ | - 90% of pharmaceutical residues reduction during batch test using unactivated biochar. - Over 98% pharmaceuticals removal for 11 compounds in nitrified urine using granular activated carbon. | Solanki & Boyer, 2017 Köpping et al., 2020 |

No pharmaceutical residues were detected in crops after the application of unspiked stored urine (Mullen, 2018; Viskari et al., 2018; Winker et al., 2010) and the negative impacts on crop yield or quality are assumed to be negligible by some authors (World Health Organization, 2012). In the example of struvite, the bioaccumulation of pharmaceuticals in tomato was shown to be low and the pharmaceutical contamination of tomato was lower than the acceptable daily intake (de Boer et al., 2018). The comparison with animal manures is difficult because the compounds used in veterinary medicine are not always the same as those used for humans. The use of antibiotics is frequent in animal breeding, but humans may use other impacting substances, such as anticancer drugs. Hammer and Clemens (2007) have calculated that the fluxes of tetracycline (one of the most-sold types of antibiotic for humans and animals) are much lower when a field is fertilized with human urine than with cattle or pig slurry (at least 1 \log_{10} difference). The fluxes of 3 hormones were also lower using stored urine. Post-treatments such as filtration on activated carbon can be performed (e.g. Köpping et al., 2020; Solanki & Boyer, 2017). They are efficient in terms of the reduction of pharmaceutical residues but it could hinder the implementation of urine value chains in some contexts.

Further studies are needed on the behavior of pharmaceuticals in soils after application and their potential ecotoxicological impacts. The management of pharmaceutical residues should be included in a holistic approach. E.g. the “benign by design” approach aims to design “green” biodegradable chemicals (Kümmerer, 2013).

3.3.3. Pathogens

The pathogens contamination of UBFs is much lower than that in wastewater. The effects of treatments on pathogens are more documented than those on pharmaceutical residues. We classified all treatments on a 3-step scale in Table 2-5.

Table 2-5. Efficiency of treatments on pathogenic organisms. Weak (+): Weak inactivation of pathogens. Moderate (++): Partial inactivation or possible under certain conditions (e.g. storage time, NH₃ concentration). Strong (+++): Complete inactivation on short term.

| Treatment | Efficiency | Examples | Reference |
|---------------------------------|-------------------------------|--|--|
| Storage | ++ | - Even at low NH ₃ concentration and 4 °C, the time to inactivate 90% of <i>Salmonella spp.</i> and <i>E. coli</i> is 5 days. <i>Ascaris</i> egg inactivation is more difficult and depends on temperature and NH ₃ concentration. | Nordin, 2010 |
| Acidification | + to ++ | - Strong reductions of bacteria at pH lower than 2. At pH between 2 to 7 (acidified fresh urine), bacteria concentration may be higher compared to non-acidified urine. | Hellström et al., 1999 |
| Alkalization | ++ | - Bacteria and bacteriophages are quickly inactivated in alkalinized dehydrated urine. - Alkaline pH alone did not inactivate <i>Ascaris</i> eggs. | Senecal et al., 2018 Senecal et al., 2020 |
| Nitrification | ++ | - Nitrification causes inactivation of the bacteria <i>Salmonella typhimurium</i> , <i>Enterococcus spp.</i> but has no influence on bacteriophages $\Phi X147$, <i>MS2</i> , <i>Qbeta</i> . | Schertenleib, 2014 |
| Mixture with organic substrate | Unknown for most mixture to + | - Urine may have a positive impact if the organic substrate is contaminated. <i>Ascaris</i> egg inactivation is improved when urine is added to toilet compost. | McKinley et al., 2012 |
| Precipitation of struvite | ++ | - <i>Ascaris</i> eggs and bacteria can accumulate in struvite during filtration. Bacteria, bacteriophage $\Phi X174$ and <i>Ascaris</i> eggs are inactivated at high temperature and low moisture content during drying of struvite. - High salinity after dehydration can also play a role in the inactivation of pathogens. | Decrey et al., 2011; Bischel et al., 2016 |
| Volume reduction Post-treatment | Unknown to +++ | - Dehydration promotes the inactivation of bacteria and bacteriophages in alkalinized urine. <i>Ascaris</i> eggs are resistant to low moisture and temperature is a key factor in their inactivation. - Distillation at 80 °C has a strong impact on pathogens. - Not studied for other processes. | Senecal et al., 2018, 2020 |
| Pathogens Post-treatment | Unknown to +++ | - Pasteurization at a temperature of 70 °C for 30 minutes or 80 °C for 1.2 minutes can be used to sanitize urine. - Oxidation, filtration or ultraviolet processes that are used to remove pathogens in other liquid streams or to treat pharmaceuticals in urine could be efficient on pathogens. | No reference found on urine Noe-Hays et al., n.d. |

Most treatments have a moderate impact on pathogens. During storage, the effect depends on the NH₃ concentration, temperature, and storage time (Nordin, 2010; Decrey & Kohn, 2017). Most pathogenic bacteria studied are generally inactivated within a few days (Höglund et al., 1998; Nordin, 2010), and most viruses and oocysts studied within a few weeks (Chandran et al., 2009; Goetsch et al., 2018). Other pathogens, such as *Ascaris* eggs or Clostridia, can still be present after several months of storage (Höglund et al., 2000; Nordin, 2010). The World Health Organization (WHO) recommends 1 to 6 months of storage (for stored urine) depending on the temperature and the crops on which urine will be applied (World Health Organization, 2012). Urine storage does not lead to complete

sterilization but offers a “selective environment” with a bacterial community dominated by *Lactobacillales* and *Clostridiales*, well adapted to this environment (Lahr et al., 2016). Hydrolysed urine shows faster inactivation of bacteria than non-hydrolysed. In hydrolysed urine, osmotic pressure and concentration factor were found as important factors to reduce *E. Coli* contamination (Oishi et al., 2020). These authors highlighted the necessity that storage recommendations to reduce pathogens should be given according to the concentration factor of the treatment and if urine was hydrolyzed or not. Furthermore, storage period may be lowered for certain storage conditions (Vinnerås et al., 2008) or in some special cases (e.g. in waterless urinal, which prevent fecal cross-contamination). Acidification is little documented but very low pH enhances bacteria inactivation, however acidification to higher pH decreases the inactivation of bacteria compared to that in non-acidified urine (Hellström et al., 1999). This may be due to the lower content of ammonia in solution compared to non-acidified urine. Alkalization permits the inactivation of bacteria and bacteriophages even if the ammonia content is low due to nitrogen stabilization in urea form (Senecal et al., 2018). Nitrification may enhance bacteria inactivation compared to storage but not of bacteriophages (Schertenleib, 2014). Mixtures with organic matter are little documented but high pH and the presence of ammonia from urine may enhance inactivation (McKinley et al., 2012). Dried struvite is in most cases lightly contaminated with pathogens, but the contamination level differs by pathogens. However, the pathogen content in struvite is lower than required in the Dutch fertilizer regulations (Gell et al., 2011). Only some of the volume reduction treatments at high temperatures, such as distillation or post-treatments dedicated to pathogen have a strong impact on pathogens.

There is a need for further research to characterize whether some treatments (e.g. acidification) lead to exposure below the admissible risks proposed by the World Health Organization (2012).

3.4. Treatment inputs

3.4.1. Energy

Energy is particularly needed when volume reduction is conducted or for the transport of urine. The energy consumption greatly varies among the treatments (Table 2-1), ranging from almost no energy to several thousand MJ/kg N. Energy consumption can vary greatly within the same family of treatments depending on the scale of the process and its optimization. The influent nitrogen concentration also varies greatly and has a strong impact on the energy consumption required to reach the same final nitrogen concentration. It also impacts the nitrogen concentration factor. Caution should be taken in the direct comparison of energy consumption per kg of nitrogen, as the concentration factor and final product concentrations also have to be considered. An experiment comparing the energy consumption of processes with the same influent would be interesting and would provide a better comparison.

Most treatments, except some of the osmosis processes and highly optimized distillation processes, require more energy than the production of nitrogen mineral fertilizer (Haber-Bosch process) or treatment in WWTPs. Treatments as nitrification or struvite precipitation require more energy than the other stabilization treatments. However, some treatments can use local renewable energy, such as passive solar stills, which makes direct comparisons of energy consumption less relevant.

Osmosis processes (reverse and forward) do not lead to a high concentration factor (less than 4% of nitrogen in the final product), but they are the least energy-consuming processes. Freeze-thawing increases the concentration factor but the energy consumption is strongly correlated with the scale of the process (a factor of 40 between laboratory scale and large scale). Evaporation is a very heterogeneous category that ranges from solar stills to electrically powered evaporation. If the processes are not optimized, the energy consumption can be very high (Karlsson, 2019) but it can also be easily reduced. Chemicals must be added for nitrogen stabilization before evaporation (e.g. lime, acid), which can reduce the potential nitrogen concentration in the final product. The energy consumption of distillation without energy recovery is high, but the volume reduction can be brought to dehydration. Distillation using energy recovery (e.g. heat exchange, vapour compression) permits a high energy savings of approximately 85 - 90% compared to that required for basic distillation (Etter et al., 2015; Udert & Wächter, 2012). Generally, volume reduction systems are more energy efficient when they have larger infrastructure due to scale effects. However urine is hardly transportable in pipes on long distance (precipitation and the need for a new collection network) preventing the development of centralized infrastructure and many recent processes are in a decentralized perspective (e.g. Randall & Naidoo, 2018). However, for some treatments such as nitrification or reverse osmosis, semi-centralised treatment plant allowing energy saving can be proposed. Nevertheless, urine would have to be transported to the treatment plant causing transport cost and impacts. For distillation processes, the nitrogen concentration of the final product highly varies without clear relationship with energy consumption which depends strongly on optimization and scale of the process.

The relationship between values of primary energy consumption found in the literature and the nitrogen concentration in the final product after the treatment is presented in Figure 2-4.

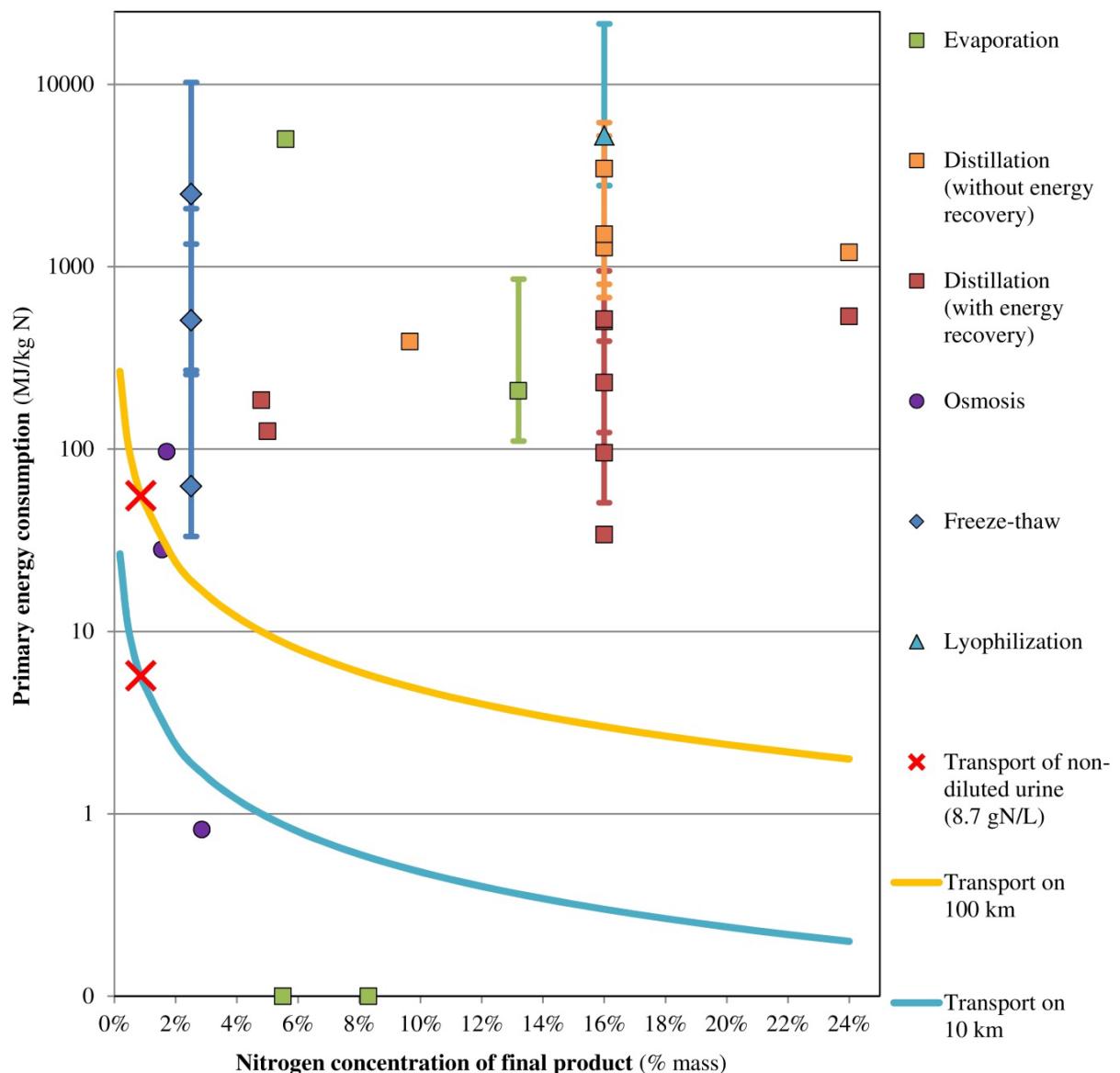


Figure 2-4. Primary energy consumption (MJ/kg N in influent) and nitrogen concentration in end products (in % mass or g N/100 g of raw material) for the volume reduction treatments. The average energy consumption and final product nitrogen concentration were taken from Table 2-1. For processes that reach dehydration, if the final product concentration is not given, a standard value of 16% N was used (mean value of Strauss, 1985). The mean influent nitrogen concentration of all reviewed experiments in Table 2-1 was 5.6 g N/L. This value was used when the influent nitrogen concentration was not given in order to calculate the energy consumption per kg of nitrogen. The least and most concentrated influents in Table 2-1 (1.8 and 11.9 g N/L) were used to plot the uncertainty of the energy consumption when urine at 5.6 g/L was used for calculation. Energy consumption for transport of urine on 10 or 100 km according to its concentration is also represented considering 4.8 MJ/ton/km (Wilsenach & van Loosdrecht, 2006). The nitrogen concentration of non-diluted urine by flushing is assumed to be 8.7 g N/L (Friedler et al., 2013).

Additionally, the energy consumption corresponding to two transportation distances between the toilet and the farm is also considered in Figure 2-4 and Table 2-5. Ten kilometers corresponds to the case of a suburban district near a farm. One hundred kilometers corresponds to the case of urine recovered in the center of a megalopolis. The impacts related to transportation vary greatly due to the different available transport modalities and the transport impact could be reduced by using more efficient way of transportation (e.g. larger capacity truck). Depending of the geographical context, nutrient and urine from cities has to be transported more or less far before to be applied on crops (e.g. Trimmer & Guest, 2018). The energy consumption required for transport is high when urine is not concentrated (up to 267 MJ/kg N for 100 km with urine at 1.8 g N/L), but decreases quickly when urine is concentrated. However, whether urine is concentrated or not, the energy used for transport remains lower than the amount of energy used for treatment. Only osmosis processes permit energy savings in some cases compared to the energy required for the transport of non-concentrated urine. Finally, the energy consumption of non-optimized evaporation, distillation without energy recovery or lyophilization is several orders of magnitude higher than that of the transport of non-concentrated urine.

Calculations were performed to compare energy consumption in simplified scenarios of a value chain that considered only transport and treatment (Table 2-6).

Table 2-6. Primary energy consumption in simplified scenarios of value chains to produce 1 kg of nitrogen fertilizer. Data is extracted from Table 2-1. We consider the use of methanol at the WWTP and 70% of nitrogen treated (Maurer et al. 2003). Energy consumption of the WWTP is considered as avoided. Nitrified concentrated urine scenario is based on Etter et al. (2015). Stored urine at 1.8 g N/L (Etter et al., 2015) and 8.7 g N/L (Friedler et al., 2013) were assumed. To simplify, mineral fertilizer equivalence is 100% for UBFs.

| Scenario | Fertilizer production MJ/kg N | Avoided treatment at WWTP MJ/kg N | Transport on 10 km MJ/kg N | Transport on 100 km MJ/kg N | Total MJ/kg N |
|--|----------------------------------|---|----------------------------------|-----------------------------------|------------------|
| Reference (Mineral fertilizer + WWTP) | 45 | 0 | neglected | neglected | 45 |
| Stored (diluted) urine (1.8 g N/L) | 0 | -76 | 27 | 267 | -49 / 191 |
| Stored urine (8.7 g N/L) | 0 | -76 | 6 | 55 | -21 / -70 |
| Nitrified concentrated urine (50 g N/L) | 839 | -76 | 1 | 10 | 764 / 773 |

We would like to highlight the impact of the volume reduction of stored urine on the energy consumption required for its transport, especially when the urine has to be transported over a long distance. Except in the scenario of dilute stored urine at 1.8 g N/L transported for 100 km, the energy used in the stored urine scenarios was less than in the reference system (mineral fertilizer + WWTP).

However, in the case of nitrified concentrated urine, the treatment has important implications for energy consumption, probably because the process is only at the plant pilot scale and is not fully optimized (diluted urine, etc.). Nevertheless, many other constraints and impacts have to be taken into account (e.g. the high volume of urine at 1.8 g N/L to spread on crops) to evaluate the value chain. The scale and context of implementation are also critical factors: the Haber-Bosch nitrogen plants that produce mineral fertilizers are large-scale installations connected to the worldwide market, whereas the value chains for urine source separation should be smaller-scale and adapted to local contexts.

3.4.2. Chemicals and organic matter inputs

Many different compounds are used during treatments, such as acids, bases or organic substrates. The quantities used vary greatly depending on the treatment, from a few grams per liter (magnesium for struvite precipitation, sulfuric acid to stabilized urine) to larger quantities (addition of organic matter). The characteristics of the compounds are diverse; some are renewable (e.g. wood chips), whereas other chemicals are not (e.g. magnesium from mines). The impacts of chemical use should be taken into account. However, directly comparing their environmental impacts is not easy. Indicators such as the carbon footprint can be used (Table 2-7).

Mineral fertilizers generally have a strong impact on the carbon footprints of farms (e.g. Yan et al., 2015). However, the carbon footprint of chemicals used to treat urine is not always negligible compared to that of mineral fertilizer. For example, adding 10 g of lime per liter of urine to stabilize it represents approximately 14% of the carbon footprint of ammonium nitrate with urine at 8.7 g N/L (the carbon footprint is much higher if 10 g of lime per liter are added to flush diluted urine). As for energy consumption, chemical consumption can potentially be a “hotspot” of environmental concern. All impacts associated with the chemicals have to be assessed, not only those related to climate change (e.g. toxicity to humans).

Table 2-7. Examples of carbon emissions for the production of chemicals compared to mineral fertilizer and transport. We assumed a urine of 8.7 g N/L (Friedler et al., 2013). Data are extracted from Ecoinvent data base version 3 (Wernet et al., 2016). Urine is transported in a 16-32 tons freight lorry. We also assumed a case with non-concentrated urine of 1.8 g N/L (Etter et al., 2015) for transport. Sulfuric acid dose is 60 mEq H⁺/L (Hellström et al., 1999) and 10 g/L for lime (Randall et al., 2016). Ammonium nitrate production (using the Haber-Bosch process) was taken as an example of mineral fertilizers (other fertilizers may have lower carbon footprint). Carbon emissions were assessed with the IPCC (2013) global warming potential values for 100-year time horizon method (IPCC 2013 GWP 100a version 1.0.3) and using the Simapro software version 8.5.4.0 (PRé Consultants, 2017).

| Chemical | Quantity kg/ton | Quantity kg/kg N | Chemical carbon emissions rate kg CO ₂ eq/kg | Carbon emissions kg CO ₂ eq/kg N | Carbon emissions compared to ammonium nitrate | Ecoinvent dataset |
|---|--------------------|---------------------|--|---|---|---|
| Ammonium nitrate | - | - | - | 8.17 | - | Ammonium nitrate, as N {GLO} market for Cut-off, U |
| Lime | 10 | 1.15 | 0.973 | 1.12 | 14% | Lime, hydrated, packed {GLO} market for Cut-off, U |
| Sulfuric acid | 2.94 | 0.34 | 0.163 | 0.06 | 1% | Sulfuric acid {GLO} market for Cut-off, U |
| Transportation of urine on 10 km (8.7 / 1.8 g N/L) | | | | 0.19 / 0.93 | 2% / 11% | Transport, freight, lorry 16-32 metric ton, EUROS |
| Transportation of urine on 100 km (8.7 / 1.8 g N/L) | | | | 1.9 / 9.3 | 24% / 114% | {GLO} market for Cut-off, U |

4. Synthesis and perspectives

Treatments: As summarized in the graphical abstract, the state of knowledge and technology readiness differs among the treatments ranging from laboratory pilot (e.g. acidification of urine or osmosis processes on urine) to already implemented value chains (e.g. neighborhood with source separation and storage). In order to more accurately assess the environmental impacts and the feasibility of value chains, there is a need for implementation at higher scales in order to increase the technology readiness level. Volume reduction post-treatments are less studied and a large number of them were not tested on urine in real conditions.

If urine is source separated, most of the treatments mentioned in this review have good potential for implementation depending on the local resources available (e.g. wood ashes for alkalinization) and the geographical context (e.g. distance between the toilet and the field). Most of them are adapted to different scales of implementation ranging from a single household to neighborhood (with the

exception of some high-tech treatments such as nitrification). Volume reduction post-treatments appear to be necessary for large-scale implementations in dense urban areas and for the use of UBFs in conventional farming systems over a large area. However, their energy consumption is high. Efforts to reduce energy consumption should be a specific focus of research.

Fertilizer efficiency and associated impacts: UBFs have high fertilizer efficiencies, similar to mineral fertilizers, without high variability between them. However, none of the UBFs was largely studied and they need to be tested in different conditions and on different crops. Associated impacts and especially ammonia volatilization after application are expected to vary among UBFs and should to be investigated.

Contaminant removals: The trace metal content of UBFs is low, and the pathogens they contain can be effectively treated. The issue of pharmaceutical residues in UBFs is still under debate, and the health and environmental risks linked with their use in agriculture have to be assessed. Except the treatments specifically dedicated to pharmaceutical residues removal, the other treatments are not able to ensure high removal.

Environmental impacts assessment: All impacts of the value chains must be taken into account. Some environmental impact assessments were carried out with methodologies such as life cycle assessment (e.g. Remy & Jekel, 2008; Spångberg et al., 2014). In general, the environmental impacts of the use of UBFs are better than those of the use of mineral fertilizers and of the treatment of urine at the WWTP (e.g. Spångberg et al., 2014; Tervahauta et al., 2013). A few studies focused on the impacts linked to their use in agriculture (e.g. impact of spreading, ammonia volatilization). The insertion of UBFs in the technical cropping practices is also little studied (e.g. Tidaker et al., 2007). Furthermore, most environmental impact studies focus on stored urine and struvite and other UBFs were barely assessed.

Other issues: Some issues were not considered in this review but remain important for the implementation of value chains. For example, the acceptability of the use of UBFs by farmers or by consumers should be considered (e.g. Brun, 2018; Lienert & Larsen, 2010; Segrè Cohen et al. 2020).

The economic point of view is also important and difficult to take into account. A wide variety of economic system can be considered. An option could be the “waste” approach where the cost of treatment is supported by the “producer” of urine as for the wastewater treatment. Then UBFs may be given free of charge to the farmers such as sewage sludge. A second option is the product approach. Considering that some treatments may permit to reduce the contaminant concentration and guarantee the nutrient content, UBFs may be sold to farmers as a standardized fertilizer.

In conclusion, UBFs are interesting alternative fertilizers derived from a resource that is currently released to the environment. They could help to close the biogeochemical cycles and to reduce the impacts of agricultural systems and human excreta management systems on the environment.

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Supplemental material

SM 2-1. Nutrient extraction and other urine treatments

Examples of the most common treatments are given below without going into detail on the agronomic efficiency of the resulting fertilizer, contaminants removal, or other factors.

Ammonia stripping: After urea hydrolysis, NH₃ can be removed at low pressure and high temperature and recovered in acidic solutions, such as sulfuric acid. Then the acidic solution recovered can be concentrated through evaporation and even dehydrated (Antonini et al., 2011; Jagtap & Boyer, 2018).

Bioelectrochemical systems: These systems were largely studied recently (eg. Jain & He, 2018). In microbial fuel cells (Cid et al., 2018; Kuntke, 2013), bacteria develop on an anode and oxidize the organic matter contained in urine, creating an electric current between an anode and a cathode. Anode and cathode are separated by a membrane, owing movement of ions in solution due to the electric current. Nutrient can be separated by the membrane from the feed solution and then can be recovered in a concentrated stream. In a microbial electrolysis cell, the system is similar (Hou et al., 2018; Zamora et al., 2017), but an external power source is added in addition to the electric potential created by the bacteria, allowing the electrolysis of water and the production of energy as H₂.

Electrochemical system: An electric field provided by a power source drives ions present in the solution through an ion-selective membrane. The ions are recovered in the concentrated stream (De Paepe et al., 2018; Pronk et al. 2006). For electrosorption, a potential is applied between 2 electrodes, and the ions in the solution are caught on electrodes due to the potential. When the potential is equalized, the ions can be released in a highly concentrated stream (e.g. Kuntke, 2013).

Membrane systems: As in stripping, during membrane distillation, nitrogen can be extracted through an ammonia-permeable membrane. In some case, alkalinizing urine and raising temperature are needed for a further recovery (Derese & Verliefde, 2016; Zhao et al., 2013). Osmosis process such as forward osmosis, can be used in a as extractive mode based on the permeability of the membrane to some compound such as urea (e.g. Volpin et al., 2018).

Adsorption processes: Urine passes through a medium with a high ion exchange capacity. This medium can be ion exchange resin (O'Neal & Boyer, 2013; Tarpeh et al., 2018), zeolite (Caspersen & Ganrot, 2018; Ganrot et al., 2007), activated carbon (Pillai et al., 2014; Purnama Putra et al., 2014), biochar (e.g. Xu et al., 2018), or layered double hydroxides (Dox et al., 2019; Everaert et al., 2017). Then, the medium has to be rinsed to recover the nutrient or, in some cases (zeolite, biochar), can be directly applied in the field (Caspersen & Ganrot, 2018; Xu et al., 2018).

Precipitation processes: Some treatments aim to make precipitate nutrients, such as phosphorus with struvite. Potassium can also precipitate and be recovered with the phosphorus as K-struvite

[MgKPO₄·6H₂O] adding magnesium (and phosphorus in some cases) (Gao et al., 2018; Wilsenach et al., 2007). K-struvite precipitates especially when nitrogen was recovered in a previous step and is no more present in the influent (Xu et al., 2011). For nitrogen, urea can precipitate as isobutylaldehyde-di-urea (IBDU) (Behrendt et al., 2002) or methylene urea (Ito et al., 2013).

Precipitation can be done by freeze-concentration. The urine is slowly frozen and ions and chemical compounds are excluded from the ice crystalline structure and remained concentrated in the liquid part. When the temperature goes below the eutectic temperature (for a specific salt), the solution is oversaturated in salt and both pure ice and salt crystalize. As ice is less dense than salt, they can be separated. The different salts present in urine can be recovered according to their respective eutectic temperatures (Randall & Nathoo, 2018; Williams et al., 2015). Freezing processes theoretically consume less energy than heating processes as the heat of fusion is lower than the heat of vaporisation (Randall et al., 2011; Schmidt & Alleman, 2005). Theoretical water removal from urine can reach 85% to 95% (Schmidt & Alleman, 2006).

Other non-extractive processes: Other processes can be applied to stabilize urine or recover nutrients from urine, such as the use of urease inhibitors (e.g. Saggar et al., 2013), storage at high temperatures (e.g. Zhou et al., 2017), microalgae grown in urine (e.g. Piltz & Melkonian, 2018) and other.

SM 2-2. Pharmaceuticals post-treatments

Some “post-treatments” aim specially to treat pharmaceuticals in urine. Examples of the most developed processes used for urine are mentioned below. Their environmental impacts and energy consumption were not yet evaluated.

Pharmaceuticals present in urine can be removed by oxidation processes such as the Fenton reaction (Abdel-Shafy & Mansour, 2013) or ozonation (Dodd et al., 2008; Gulyas et al., 2007; Pronk et al., 2007). As in water treatment, ultraviolet radiation can be used (Gajurel, 2007; Zhang et al., 2015). Biological treatments can also be used (Escher et al., 2006), including algal bioreactors (de Wilt et al., 2016) or membrane bioreactors (Abdel-Shafy & Mansour, 2016). Various filtration processes through membranes with different pore sizes were studied, including microfiltration (Hassan et al., 2016), ultrafiltration (Triger, 2012) or nanofiltration (Escher et al., 2006). Sorption onto ion exchange resins (Landry & Boyer, 2013) or zeolite (Simons, 2008) was studied. Sorption onto activated carbon or biochar can also be a good option for the removal of pharmaceutical residues (Etter et al., 2015; Solanki & Boyer, 2017, 2019). This method was tested on nitrified urine and fresh and stored urine with pharmaceutical removals higher than 90% in most cases (Solanki and Boyer, 2017; Köpping et al., 2020). However, nitrogen and phosphorus can also partly adsorb onto biochar (less than 20% in the experiment of Solanki and Boyer, (2017). Biochar has a lower environmental footprint and is less expensive than activated carbon (Solanki & Boyer, 2019).

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Partie 2

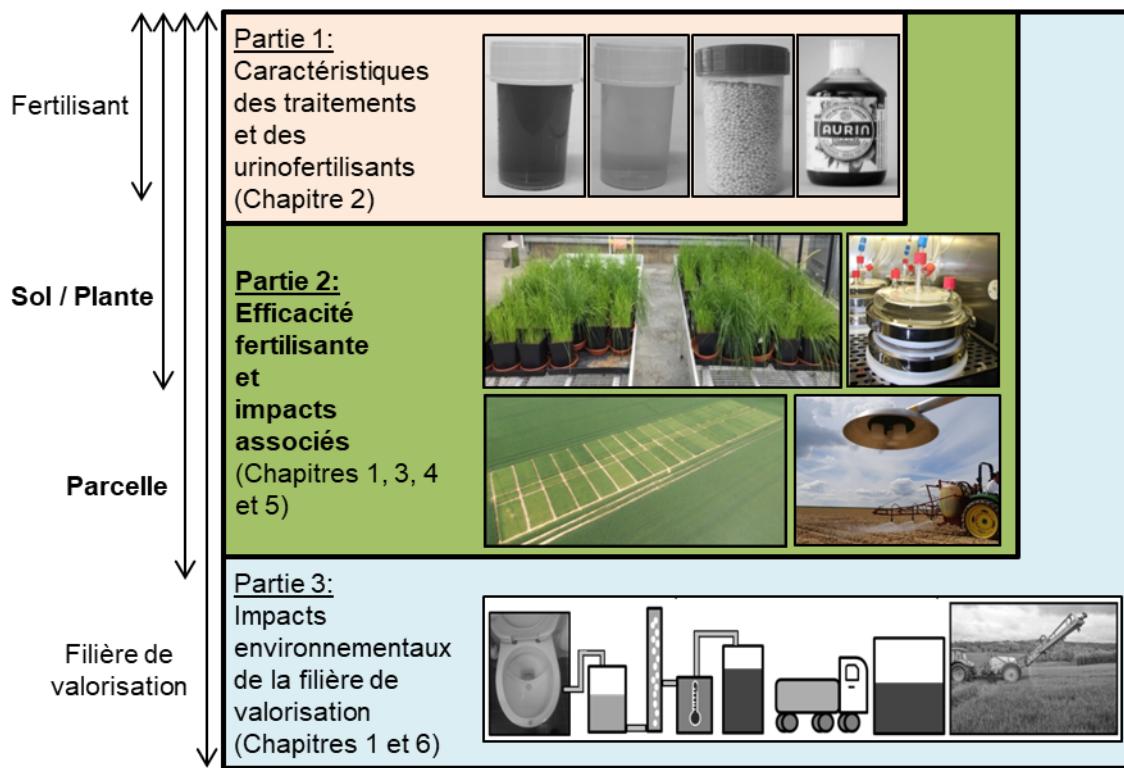
Efficacité fertilisante azotée et impacts associés

Nous avons vu dans la partie 1 que les traitements variés appliqués à l’urine résultent en de nombreux urinofertilisants aux caractéristiques physico-chimiques variées (p. ex., solide, liquide, concentré ou dilué). Ces fertilisants ont une efficacité fertilisante élevée (coefficient d’équivalence engrais généralement au-dessus de 75%). Cependant, excepté pour l’urine stockée, cette efficacité reste très peu étudiée. De plus l’efficacité de différents urinofertilisants n’a pas été comparée dans des conditions expérimentales identiques. La seconde partie de la thèse vise à quantifier l’efficacité fertilisante azotée des urinofertilisants. Comme la volatilisation de l’ammoniac après apport des urinofertilisants au champ constitue un impact environnemental potentiel important, nous nous sommes intéressés également à ce potentiel de volatilisation qui, outre son impact environnemental, peut diminuer fortement la valeur fertilisante azotée des urinofertilisants. Les teneurs des urinofertilisants étudiés en éléments traces et pathogènes¹ ont également été analysées afin de documenter plus largement les impacts associés à leur apport. Cette partie se décompose en trois chapitres.

L’efficacité fertilisante azotée a d’abord été étudiée en conditions contrôlées en serre (chapitre 3) afin de caractériser dans les mêmes conditions expérimentales l’efficacité fertilisante en tant qu’engrais azoté de 9 urinofertilisants aux caractéristiques variées. Dans un second temps (chapitre 4), cette efficacité fertilisante a été mesurée au champ pour 4 urinofertilisants en conditions « réelles » d’application. En serre comme au champ, l’efficacité des urinofertilisants a été comparée à d’autres engrains organiques (P. ex., lisier bovin) et à un engrais minéral.

Les teneurs en éléments traces (chapitre 3) et pathogènes (chapitre 4) ont été analysées dans les urinofertilisants utilisés en serre et au champ. Ces contaminants ne font pas l’objet d’étude approfondie mais les teneurs mesurées seront comparées à celles dans d’autres fertilisants organiques ou à des standards. Nous nous sommes focalisés dans cette thèse au risque de volatilisation de l’ammoniac. La volatilisation ammoniacale a été mesurée en conditions contrôlées pour la plupart des urinofertilisants (chapitre 5) étudiés et au champ pour le produit utilisé le plus couramment (urine stockée, chapitre 4).

¹ Les teneurs en résidus pharmaceutiques font l’objet d’une étude parallèle au sein du programme Agrocapi et ne seront pas abordées ici.



Chapitre 3

Efficacité fertilisante azotée en conditions contrôlées

Mineral fertilizer equivalence of nine different human urine-based fertilizers under greenhouse conditions

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Abstract

Purpose: Most of the nutrients in wastewater come from human urine and their recycling for agricultural purposes is very limited. Conventional agriculture relies on the use of synthetic nitrogen fertilizers, which contribute to the disruption of planetary biogeochemical cycles. After source separation, urine can be post-treated to produce various urine-based fertilizers. This study aims to characterize the nitrogen fertilization efficiency of different urine-based fertilizers.

Methods: Nine urine-based fertilizers were compared together with ammonium nitrate and cattle slurry in a greenhouse pot trial. The biomass production and nitrogen uptake of the plants were measured. The nitrogen use efficiency and the mineral fertilizer equivalent were determined for each fertilizer.

Results: The urine-based fertilizers was classified in four types based on their nitrogen forms (ammonium, nitrate, urea or organic). All presented mineral fertilizer equivalent higher than that of cattle slurry, higher than 85% for most of the urine-based fertilizers and even higher than 100% for nitrified and alkalinized stored urine. The lowest mineral fertilizer equivalent were found for fermented urine and the mixture of urine and woodchips but remained between 65 to 75%. The differences among the urine-based fertilizers and from the cattle slurry were attributed to the fraction of organic nitrogen.

Conclusions: Urine-based fertilizers are efficient fertilizers with low trace element contents issued from a resource that currently is mostly released into the environment.

Key words

Fertilization; Greenhouse trial; Human urine; Nutrient recycling; Source separation; Urine-based fertilizer;

1. Introduction

Wastewater contains large amounts of nutrients whose release in the environment can have undesirable environmental impacts (Sutton et al. 2011). On the one hand, the amount of nutrients recycled from wastewater is low (Esculier et al. 2018). On the other hand, conventional agriculture relies on the use of synthetic nitrogen (N) fertilizers that require a substantial amount of energy for synthesis (Matassa et al. 2015). Most nutrients in wastewater have urine as their source (Friedler et al. 2013) and their recovery could offset a substantial proportion of the mineral fertilizer in agriculture (Trimmer et al. 2019).

Urine can be separated from the other constituents of wastewater by source separation (Rossi et al. 2009). Urine is a diluted solution compared to mineral fertilizers. The concentration of trace elements in urine is low (Ronteltap et al. 2007), and while some pathogens can be present in urine, proper storage inactivates these pathogens to acceptable levels (WHO 2012). In contrast, pharmaceutical residues are of concern, and the need for specific urine treatments to remove them before application is currently a topic of debate (Winker 2009; WHO 2012). Collected urines are often post-treated for: (i) nitrogen stabilization (e.g., by nitrification), to prevent ammonia volatilization and allow volume reduction; (ii) volume reduction, to reduce transport costs and impacts as well as the work required for application; (iii) nutrient extraction to obtain concentrated fertilizers; and (iv) treatment of contaminants to produce safer fertilizers (e.g. urine storage as recommended and frequent treatment for sanitation). All treatments result in different products defined as urine-based fertilizers (UBFs).

The fertilization efficiency of stored urine has been studied on different crops (Pandorf et al. 2019), but other UBFs remain barely studied. As the fertilization efficiency depends on the trial conditions, it is difficult to directly compare studies. This study aimed to characterize the fertilization efficiency of nine UBFs as N fertilizers compared to mineral fertilizer and cattle slurry under the same conditions in a greenhouse trial.

2. Material and methods

2.1. Urine-based fertilizers

Nine UBFs were used either issued from separate collection and treatments that intended to stabilize nitrogen (acidification, alkalinization and nitrification), or from a frequent collecting practice resulting in a mixture of urine with woodchips. Detailed information on the treatments can be found in Martin et al. (in press). Some UBFs were specifically produced for this study.

The stored urine was collected in a university building using a waterless male urinal and stored for six months in an airtight tank. For the acidified stored urine, 31.3 mL of sulfuric acid (96% pure) was

added per liter of stored urine to decrease the pH to 6.5 to reduce ammonia losses ($pK_a \text{ NH}_3/\text{NH}_4^+ = 9.2$).

For acidified and alkalinized fresh urine as well as the mixture with woodchips, urine was collected from approximately 20 donors from the university, stored at 4°C and used within three hours. The objective of acidifying the fresh urine to below pH 4 or alkalinizing the fresh urine to above pH 11 of fresh urine was to prevent urea hydrolysis and stabilize the N. To produce acidified fresh urine, we added 60 mmol H⁺/L to fresh urine (1.61 mL/L of 96% pure sulfuric acid, Hellström et al. 1999). To produce alkalinized fresh urine, we added 10 g Ca(OH)₂/L to fresh urine (Randall et al. 2016). In dry toilets, urine is often mixed with absorbent organic substrates. Thus, a mixture of fresh urine and woodchips was produced one week before the start of the experiment, with 1 kg of woodchips (less than 1 cm pieces) mixed with 286 g of fresh urine (the maximum amount that the woodchips could absorb).

Fermentation and nitrification are treatments that decrease ammonia volatilization risk and make possible further concentration, respectively. The treatment for fermented urine was similar to the one of Andreev et al. (2017). It consisted in the acidification of fresh urine, followed by a long term lactic acid fermentation using malt as inoculum of lactic acid bacteria. This pilot batch was produced by the TOOPI Organics company (www.toopi-organics.com). For the nitrified urine, the biological nitrification of half of the ammoniacal N in the stored urine was followed by volume reduction by distillation (Fumasoli et al. 2016). It was produced by the VUNA company (www.vuna.ch). Alkalized dehydrated urine was provided by the Swedish University of Agricultural Sciences. The urine was alkalinized using two different alkaline media : lime (20.6 g/L urine) and lime (5.1 g/urine) + biochar (15 g/L urine); and the mixtures were dehydrated (Simha et al. 2020).

The cattle slurry was collected in a conventional dairy farm and used as a reference organic fertilizer. Liquid ammonium nitrate was used as a reference mineral fertilizer.

All fertilizers were analyzed for their contents in water, carbon, nutrients, trace elements and the different forms of N (Table 3-1). Information on the used methods is summarized in SM1.

2.2. Greenhouse experiment

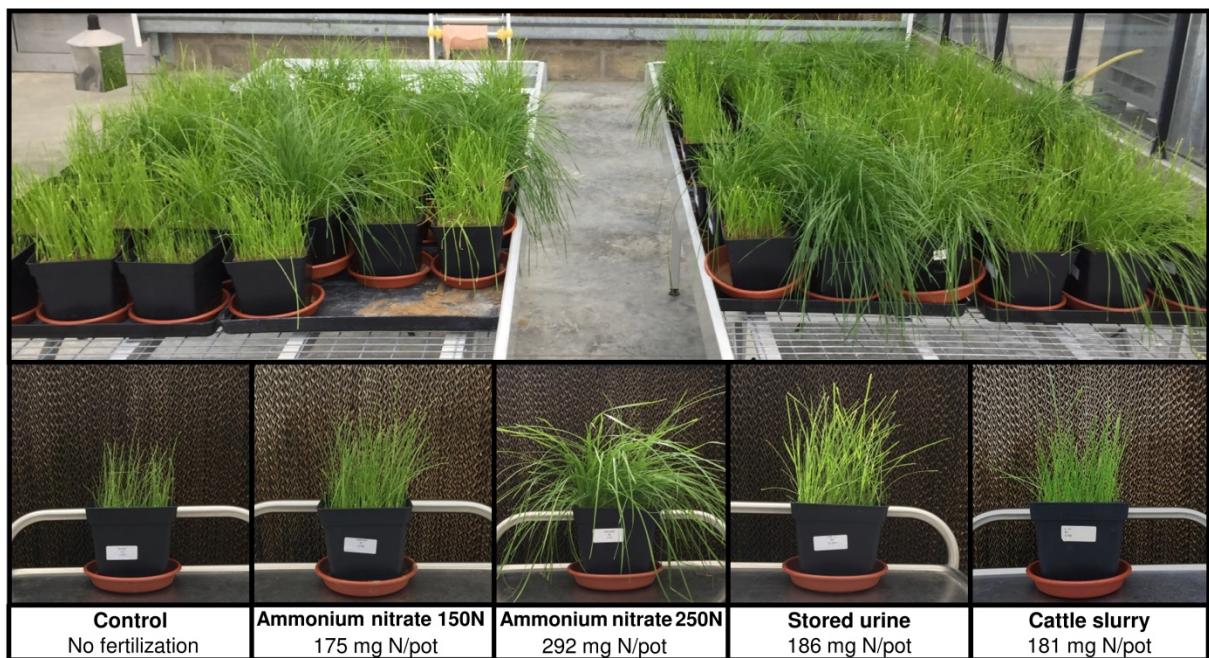


Figure 3-1. Greenhouse trial and biomass in some treatments before the third cut (day 63).

The greenhouse experiment was performed with English ryegrass (*Lolium perenne* L.) sown in a soil sampled from the surface horizon of a silty luvisol (Figure 3-1), sieved at 4 mm and stored at 4°C before the experiment. The soil was lightly carbonated (0.8% CaCO₃) and had a pH (H₂O) of 8.0. The organic matter content was 13.6 g C/kg dry soil, and the initial mineral N content was low (11.9 mg N/kg dry soil). A detailed soil analysis is provided in SM 3-1. The pots were filled with 1.30 kg of fresh soil (equivalent to 1.17 kg of dry soil). In order to ensure that only N would be a limiting nutrient, phosphorus and potassium were added as K₂HPO₄ with 100 mg P/kg dry soil and 250 mg K/kg dry soil. Magnesium was added as MgSO₄ with 40 mg Mg/kg dry soil and iron as FeSO₄.7H₂O with 1 mg Fe/kg dry soil. The sulfur input resulting from the Mg and Fe inputs was 53.9 mg S/kg dry soil.

A control treatment without N addition and 2 ammonium nitrate treatments that received 150 and 250 mg N/kg dry soil were implemented to calculate the response curve of N uptake according to fertilizer input. The target dose for cattle slurry and the UBFs was 150 mg N/kg dry soil (175.4 mg N/pot). Since the nutrient concentrations were not available at the start of the experiment, they were estimated; the actual doses are shown in Table 3-2. All fertilizers were incorporated into the entire soil mass. One gram of ryegrass seeds was sown in each pot. Three replicates were established for each treatment. Water losses were measured by weighing, and the soil moisture was readjusted to 90% of the field capacity (22.5% humidity) three times a week using deionized water. No leachates out of the pot were observed. The positions of the pots in the greenhouse were randomized and moved twice a week to avoid the potential effects of heterogeneity in solar radiation. The grass biomass was

cut 1 cm above the soil surface in each pot on days 22, 42, 63 and 75. Then, it was dried at 50°C for 5 days and powdered, after which the N concentrations were measured. At the end of the trial, the mineral N content in the soils was measured. Nitrogen uptake was calculated using the N concentration in tissues and the biomass of each cut. The analytical methods are detailed in SM 3-2.

2.3. Nitrogen use efficiency and mineral fertilizer equivalent

The fertilization efficiency was estimated using two calculations: the nitrogen use efficiency (NUE) and the mineral fertilizer equivalent (MFE). The NUE of ammonium nitrate corresponded to the slope of the response curve of N uptake by plants according to the amount of N added. In the UBF treatments, the NUE was directly calculated using the following equation (1):

$$NUE (\%) = \frac{Nitrogen\ uptake\ by\ fertilized\ crop - Nitrogen\ uptake\ by\ unfertilized\ control\ crop}{Nitrogen\ added\ by\ fertilizer} \times 100 \quad (1)$$

The nitrogen use efficiency of UBFs can be compared with that of mineral fertilizer by calculating the MFE as the ratio of the NUE of the UBF and the NUE of ammonium nitrate (2):

$$MFE (\%) = \frac{NUE\ Urine-based\ fertilizer}{NUE\ Ammonium\ nitrate} \times 100 \quad (2)$$

The results are expressed as the mean of the 3 replicates with the standard error. Significant differences between treatments were tested using an ANOVA followed by a Tukey HSD post hoc test. Significant differences between UBF and ammonium nitrate were tested using Student's t-test, or a sign test if the distribution was not normal. All tests were performed using R, version 3.3.2 (R Core Team 2016).

3. Results and discussion

3.1. Urine-based fertilizers characteristics

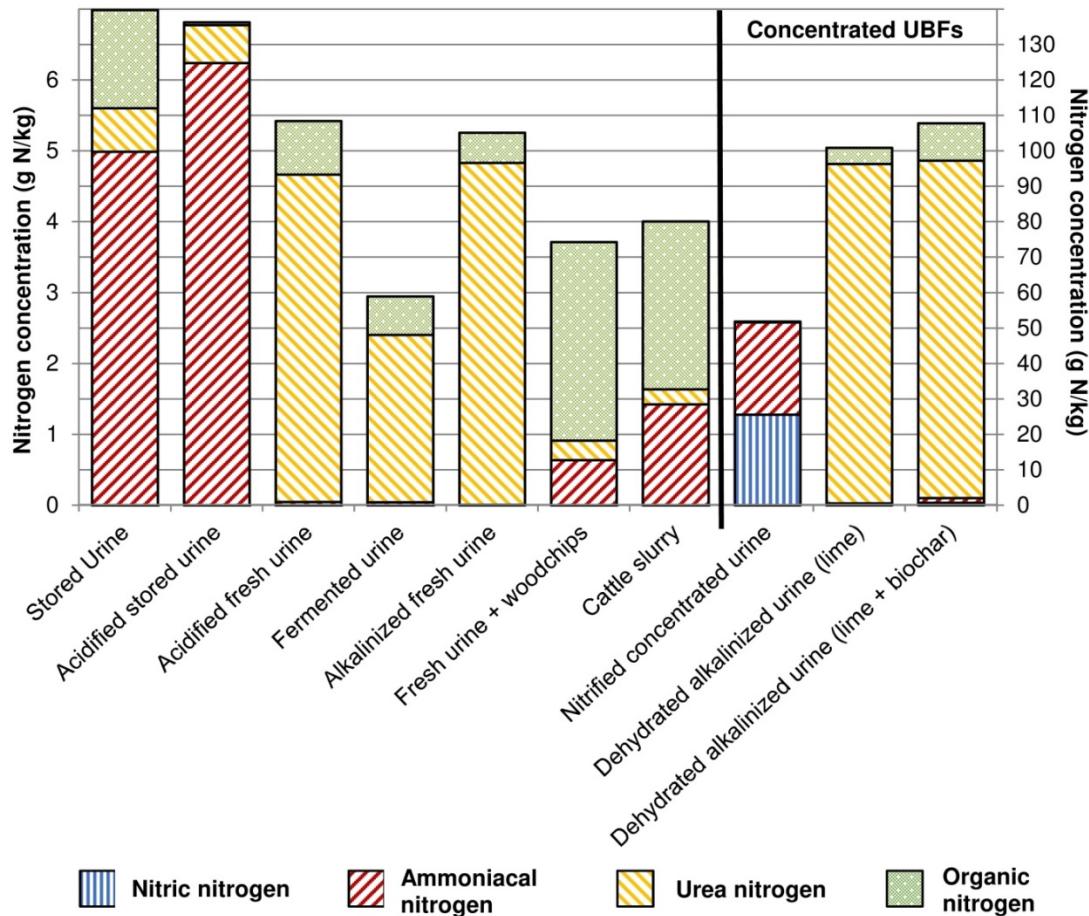


Figure 3-2. Nitrogen concentration (g N/L) and nitrogen forms in the different UBFs and cattle slurry. Concentrated UBFs are plotted on another axis. Colors correspond to the nitrogen form.

There was a strong difference in the N concentrations of the non-concentrated UBFs (below 7 g N/L) and the concentrated UBFs (up to 107 g N/L, Figure 3-2). Most UBFs had N forms similar to those in the typical mineral N fertilizer: urea and ammoniacal or nitric nitrogen (Figure 3-2). Nitrogen is excreted in urine mainly as urea (Udert et al. 2006); however, in stored urine and acidified stored urine, most of the urea is hydrolyzed during storage, and ammoniacal N is the main form of nitrogen. Interestingly, the content of organic N was much lower in the acidified stored urine than in the stored urine (0.04 and 1.4 g N/kg, respectively). The organic N may have been mineralized during acidification (Antonini et al. 2012). In contrast, in fresh urine stabilized by acidification or alkalinization, urea was the main form of N. We did not observe mineralization of organic N in the acidified fresh urine, but 20 times less acid was added to the acidified fresh urine than was added to the acidified stored urine. The N forms in nitrified urine were half nitrate and half ammoniacal N with

a very low content of organic N because the organic N had been mineralized during nitrification (Fumasoli et al. 2016). In the mixture of woodchips and fresh urine, most of the N was under organic form. This may be explained by N immobilization by microorganisms during storage due to the high carbon input from the woodchips (Reichel et al. 2018). The concentration of N in the woodchips was not determined, but, assuming an N content of 0.06%, as was measured in sawdust in Reichel et al. (2018), the expected concentration of the mixture would be 4.3 g N/kg instead of the 3.7 g N/kg measured. This suggests that at least 15% of the urine N may have been volatilized during storage. In the cattle slurry, approximately 60% of the N was under organic form, which is typical for cattle slurry (Benoît et al. 2014).

In addition to the nutrient concentrations, the contents of contaminants (trace elements, pathogens, pharmaceutical residues) must also be considered. Trace element concentrations were low in each UBF and in the cattle slurry (Table 3-1), which was also observed for stored urine by Ronteltap et al. (2007). The fluxes of trace elements in the amounts of fertilizer required to apply 200 kg N/ha would be below the limit of the French standard for the use of sewage sludge compost in agriculture (NF U 44-095).

Table 3-1. Physico-chemical characteristics of fertilizers tested. Three samples of each product were analyzed but as the replicates were homogenous, only the mean value is presented. The density of liquids UBFs was taken as 1 except for nitrified urine for which is 1.14. Dry residue includes crystallized salt. *Not measured because of crystallization. **Value given by the producer (VUNA). Initial urine was the same between stored urine and acidified stored urine and between acidified fresh urine and liquid alkalinized urine. ***As dry matter was performed at 105°C, urea chemical hydrolysis occurs at that temperature. Only trace elements were measured based on dry matter. Then, dry matter and trace elements may have been slightly underestimated.

| Parameters | Unit | Nitrified concentrated urine | Stored urine | Acidified stored urine | Acidified fresh urine | Fermented fresh urine | Alkalinized urine | Dehydrated alkalinized urine (lime) | Dehydrated alkalinized urine (lime + biochar) | Fresh urine + woodchips | Cattle slurry |
|--|-------------------------------|------------------------------|--------------|------------------------|-----------------------|-----------------------|-------------------|-------------------------------------|---|-------------------------|---------------|
| pH | - | 4.0 | 9.2 | 6.5 | 2.1 | 3.5 | 12.3 | 11.2 | 8.9 | 8.2 | 7.3 |
| Conductivity | mS/cm | 43.3 | 39.0 | 49.5 | 16.3 | 6.2 | 18.2 | 16.2 | 18.9 | 0.8 | 3.1 |
| Dry residue (liquid) or dry matter (solid) | g/100g raw material | -* | 1.4 | 3.7 | 1.8 | 0.7 | 2.7 | 84.0*** | 82.0*** | 30.7 | 5.3 |
| Carbon | Tot-C | - | - | - | - | - | - | 129.0 | 331.3 | 153.3 | 22.7 |
| | Organic-C | 2.4 | 3.3 | 2.7 | 5.4 | 8.3 | 5.0 | 82.9 | 321.0 | 153.0 | 22.4 |
| | Inorganic-C | - | - | - | - | - | - | 46.2 | 10.5 | 0.5 | 0.5 |
| Nitrogen | Total-N | 51.8 | 7.0 | 6.8 | 5.4 | 2.9 | 5.3 | 100.6 | 107.0 | 3.7 | 4.0 |
| | NH ₄ -N | 26.1 | 5.0 | 6.2 | 0.1 | 0.04 | 0.01 | 0.3 | 1.3 | 0.6 | 1.4 |
| | NO ₃ -N | 25.6 | < 0.0002 | < 0.0002 | 0.002 | < 0.0002 | 0.002 | 0.3 | 0.7 | <0.01 | 0.002 |
| | Urea-N | 0.1 | 0.6 | 0.5 | 4.6 | 2.4 | 4.8 | 95.8 | 95.2 | 0.27 | 0.21 |
| | Organic-N | 0.1 | 1.4 | 0.04 | 0.8 | 0.5 | 0.4 | 4.5 | 10.5 | 2.8 | 2.4 |
| Other nutrients | P ₂ O ₅ | 8.2 | 0.6 | - | 0.6 | 0.5 | - | 14.6 | 16.4 | 0.7 | 1.4 |
| | K ₂ O | 32.2 | 2.4 | - | 1.7 | 0.6 | - | 51.6 | 50.3 | 1.4 | 3.4 |
| | MgO | 0.1 | < 0.1 | - | < 0.1 | < 0.1 | - | 6.5 | 3.3 | 0.2 | 1.0 |
| | CaO | 0.7 | < 0.1 | - | < 0.1 | < 0.1 | - | 273.7 | 76.8 | 0.5 | 2.7 |
| | SO ₃ | 10.9 | 0.6 | - | 1.5 | 0.2 | - | 14.6 | 14.8 | 0.7 | 0.8 |
| | Na ₂ O | 24.5 | 3.1 | - | 2.3 | 1.0 | - | 49.4 | 50.3 | 1.7 | 1.2 |
| | Cl- | 54.8 | 3.9 | - | 2.8 | 1.3 | - | 24.8 | 24.2 | 0.6 | 1.2 |
| Trace elements | B | 9.5 | 1.1 | - | 1.4 | 0.3 | - | 23.9 | 21.5 | 2.4 | 0.1 |
| | Fe | < 18 | < 20 | - | < 20 | < 20 | - | 712.0 | 728.2 | 31.9 | 95.5 |
| | Cu | 0.34** | < 0.4 | - | < 0.4 | < 0.4 | - | < 2 | < 2 | 0.4 | 0.5 |
| | Mn | 0.4 | < 0.2 | - | < 0.2 | 0.3 | - | 44.7 | 46.4 | 14.2 | 36.4 |
| | Mo | 0.6 | < 0.2 | - | 0.9 | 0.3 | - | < 2 | < 2 | < 0.6 | 0.01 |
| | Zn | 6.5 | 0.1 | - | 1.1 | 1.5 | - | 12.6 | 15.6 | 4.5 | 1.8 |
| | Se | 0.4 | < 0.2 | - | < 0.2 | < 0.2 | - | < 1.3 | < 1.2 | < 0.5 | 0.01 |
| | As | < 0.2 | < 0.2 | - | < 0.2 | < 0.2 | - | 1.0 | 1.0 | < 0.3 | < 0.05 |
| | Cd | 0.03 | < 0.02 | - | 0.05 | 0.06 | - | < 0.1 | 0.07 | 0.11 | 0.04 |
| | Co | < 0.2 | < 0.2 | - | < 0.2 | < 0.2 | - | < 0.8 | < 0.8 | < 0.3 | 0.02 |
| | Cr | < 0.2 | < 0.2 | - | < 0.2 | < 0.2 | - | 6.2 | 2.8 | 0.3 | 0.01 |
| | Hg | < 0.0004 | < 0.0004 | - | < 0.0004 | < 0.0004 | - | < 0.2 | < 0.2 | < 0.1 | < 0.01 |
| | Ni | 0.6 | < 0.2 | - | < 0.2 | < 0.2 | - | 1.4 | < 0.8 | < 0.3 | 0.02 |
| | Pb | < 0.2 | < 0.2 | - | < 0.2 | < 0.2 | - | < 2 | 3.7 | < 0.6 | 0.01 |

3.2. Biomass production and nitrogen uptake

The nitrogen uptake by the above-ground biomass according to the nitrogen input is presented in Figure 3-3. The response curve for ammonium nitrate is linear ($r^2 = 0.99$). Fertilizers above the response curve had higher nitrogen use efficiency than ammonium nitrate and those below the response curve had lower nitrogen use efficiency than ammonium nitrate.

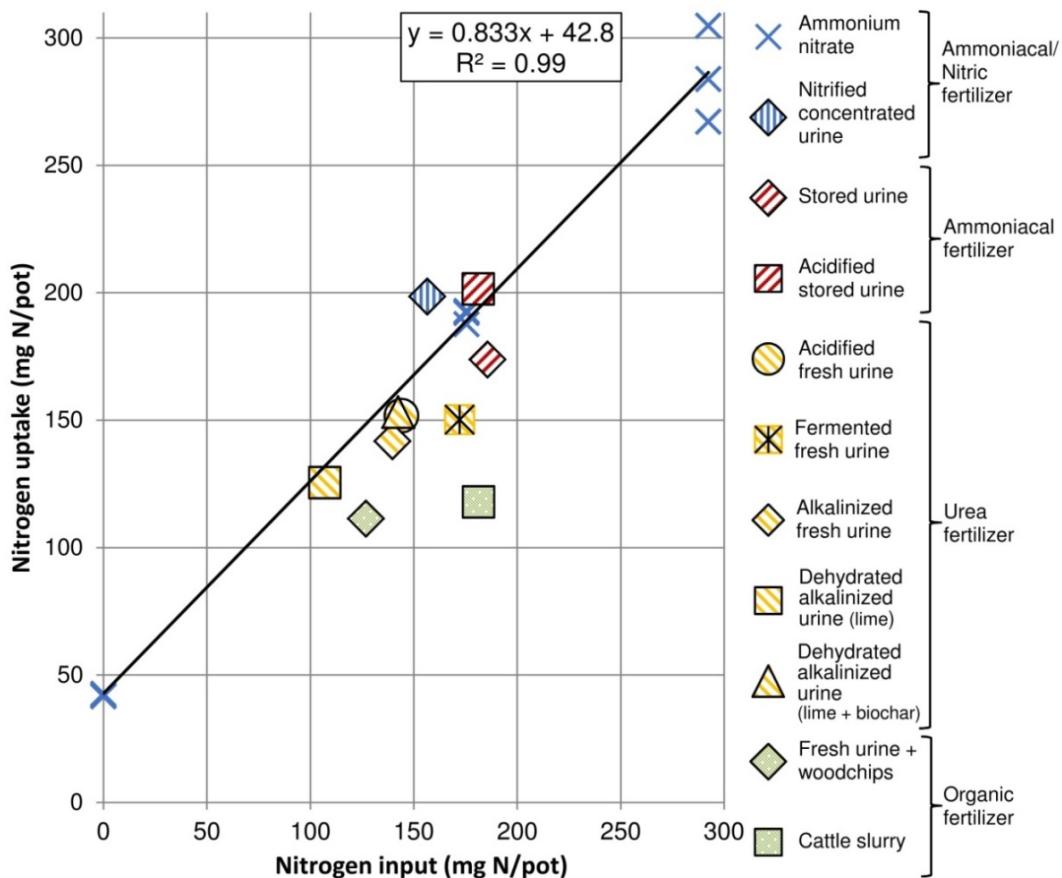


Figure 3-3. Nitrogen uptake by plants according to nitrogen inputs for the different fertilizers.
The linear regression used to compute the nitrogen use efficiency of the reference fertilizer (ammonium nitrate) is represented with a black solid line (with the corresponding equation). Colors correspond to the categories of urine-based fertilizers.

A strong increase in biomass production, biomass N content and N uptake were observed in the fertilized treatments (Table 3-2, Figure 3-3). The NUE of ammonium nitrate was 83% and was similar to those in previous pot trials (Mnkeni et al. 2008). The soil mineral N content was low at the end of the experiment, indicating that most available mineral N was taken up by the ryegrass (SM 3-3).

Table 3-2. Pot experiment: experimental conditions (doses of fertilizers and nitrogen input) and results (plant biomass and nitrogen concentration, nitrogen uptake and nitrogen use efficiency). Statistically significant ($p\text{-value} \leq 0.05$) differences among treatment are represented by letters. Treatments not significantly different from ammonium nitrate are marked with * ($p\text{-value} > 0.05$).

| Treatment | Fertilizer doses (g/pot) | Nitrogen input (mg N/pot) | Dry biomass (g DM/pot) | | Dry biomass nitrogen tissue concentration (mg N/g DM) | | Nitrogen uptake (mg N/pot) | | Nitrogen use efficiency (%) | | Mineral fertilizer equivalent (%) | |
|---|--------------------------|---------------------------|------------------------|------|---|------|----------------------------|-----|-----------------------------|----|-----------------------------------|-----|
| | | | Mean | ± | Mean | ± | Mean | ± | Mean | ± | Mean | ± |
| Control | 0 | 0.0 | 1.63 | 0.02 | 25.9 | 0.5 | 42 | 1 | - | - | - | - |
| Ammonium nitrate | 150N | 3.51 | 175 | 4.48 | 0.03 | 42.6 | 0.4 | 191 | 3 | 83 | 3 | 100 |
| | 250N | 5.85 | 292 | 6.60 | 0.11 | 43.2 | 2.2 | 285 | 19 | | | - |
| Nitrified concentrated urine | 3.02 | 157 | 4.99 | 0.04 | 39.8 | 3.0 | 198 | 13 | 100 ^{e*} | 8 | 120 ^{e*} | 10 |
| Stored urine | 26.57 | 186 | 4.29 | 0.16 | 40.5 | 0.4 | 174 | 6 | 71 ^c | 3 | 86 ^c | 4 |
| Acidified stored urine | 26.57 | 181 | 4.94 | 0.34 | 40.8 | 2.0 | 202 | 20 | 88 ^{de*} | 11 | 106 ^{de*} | 13 |
| Acidified fresh urine | 26.57 | 144 | 4.14 | 0.20 | 36.7 | 0.8 | 152 | 5 | 77 ^{cd*} | 4 | 92 ^{cd*} | 4 |
| Fermented urine | 58.46 | 172 | 4.33 | 0.25 | 34.7 | 2.0 | 150 | 9 | 63 ^{bc} | 5 | 76 ^{bc} | 6 |
| Alkalinized urine | 26.57 | 140 | 3.78 | 0.13 | 37.5 | 0.5 | 142 | 6 | 72 ^c | 4 | 86 ^c | 5 |
| Dehydrated alkalinized urine (lime) | 1.06 | 107 | 3.62 | 0.09 | 34.7 | 1.2 | 126 | 2 | 79 ^{cd} | 2 | 94 ^{cd} | 2 |
| Dehydrated alkalinized urine (lime + biochar) | 1.33 | 142 | 4.08 | 0.26 | 37.6 | 2.1 | 153 | 2 | 79 ^{cd} | 1 | 94 ^{cd} | 1 |
| Fresh urine + woodchips | 34.17 | 127 | 3.35 | 0.46 | 33.6 | 4.3 | 111 | 10 | 55 ^{ab} | 8 | 66 ^{ab} | 9 |
| Cattle slurry | 44.97 | 181 | 3.30 | 0.09 | 35.7 | 0.6 | 118 | 4 | 42 ^a | 2 | 51 ^a | 2 |

3.3. Mineral fertilizer equivalent

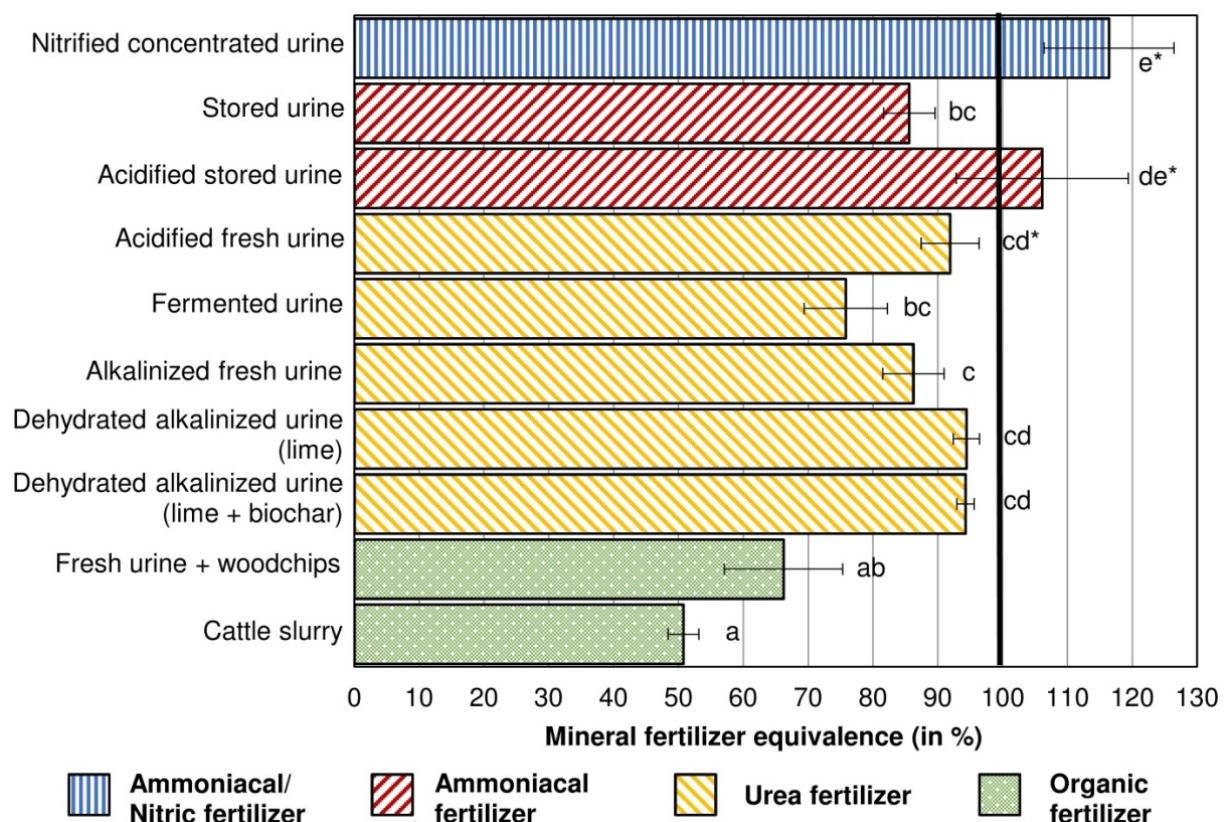


Figure 3-4. Mineral fertilizer equivalent (nitrogen) of the different UBFs and cattle slurry. Statistically significant differences among treatments are represented by letters. Treatments not significantly different from 100% (mineral fertilizer) are marked with *. Colors correspond to the categories of urine-based fertilizers.

Only acidified stored urine and nitrified urine presented MFE values higher than 100% (Figure 3-4), although not significantly different from 100%. The MFE of the nitrified urine was significantly higher than that of all other UBFs, and the MFE of acidified stored urine was significantly higher than that of stored urine. In previous trials, the efficiency of nitrified urine and acidified stored urine was also high (Bonvin et al. 2015). The stored urine, acidified fresh urine, and all three alkalinized urine samples had MFEs between 85% and 95%. Stored urine has been tested on different crops and generally showed similar or slightly lower efficiency values than mineral fertilizer (Kirchmann and Pettersson 1995; Viskari et al. 2018). The other UBFs have been little studied. A lower MFE for stored urine (91%) than for acidified stored urine (102%) has been previously observed in ryegrass (Simons 2008). The fermented urine has not been tested previously and showed a lower MFE than the other treatments. The mixture of fresh urine and woodchips had a significantly lower MFE than most UBFs, as observed for compost impregnated with urine (Martin 2018). The cattle slurry MFE (51%) was significantly lower than those of all UBFs except the mixture with woodchips; this result is consistent

with that in a previous experiment (Gómez-Muñoz et al. 2017). The efficiencies observed in this trial are similar to those observed by Gutser et al. (2005) for animal urine and cattle slurry.

The variation in MFE values could be related to the N form. Both acidified stored urine and nitrified urine presented the highest MFEs; these UBFs contained only mineral N and no organic N. For the other UBFs (except the mixture with woodchips), the percentage of organic N ranged from 4% to 19% of the total N; this may explain the MFE values lower than 100%, because this organic fraction must be mineralized before becoming available to plants. The lower efficiencies observed for the mixture with woodchips and the cattle slurry may be explained by the even higher proportion of organic N (more than 50% of the total N) in these treatments. To a lesser extent, the mineral N forms in fertilizers may impact yield and MFE (Watson 1986, 1987). Even though the soil was supplemented with a mixture of other nutrients, the MFE values higher than 100% may be partly explained by the micronutrient inputs from the UBFs; this is particularly true for the acidified stored urine, which had much higher sulfate content than the other treatments. In parallel with this trial, the phosphorus availability of some of the UBFs was characterized by Dox (2020); the phosphorus availability of these UBFs was not different from that of the mineral fertilizer, which confirmed that the UBFs can supply multiple nutrients. The lower MFE of the fermented urine may be due to the bacteria in the UBF which may have increased N immobilization in the soil. Under the experimental conditions of this study, fertilizers short-term efficiencies were maximized and ammonia volatilization was greatly limited because the fertilizers were incorporated into the whole soil mass. However, under field conditions, substantial differences in ammonia volatilization can be expected due to the various pH values and N forms of the UBFs.

4. Conclusion

The mineral fertilizer equivalent (MFE) of seven out of the nine UBFs were similar and higher than 85%. The main factor explaining the differences in MFE was the proportion of organic nitrogen. Mixing urine with organic matter strongly reduced the MFE. To a lesser extent, fermentation also reduced the MFE. It would be necessary to perform further trials under field conditions to confirm the tendencies. Furthermore, the fertilization efficiency of UBFs may be balanced by other constraints, such as ammonia volatilization, contaminant contents or the volume of UBF that must be applied conditioned by their nitrogen content.

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Supplementary material

SM 3-1. Detailed soil analysis

| Parameter | Unit | Mean | Standard error | Method |
|-------------------------|-------------------------------------|--------|----------------|--|
| Soil texture | Clay | 259 | 3 | |
| | Fine silt | 240 | 6 | |
| | Coarse silt | 407 | 5 | Pipette method with decarbonation |
| | Fine sand | 65.1 | 1.1 | |
| | Coarse sand | 25.7 | 1.0 | |
| General characteristics | pH (water) | - | 8.0 | ISO 10390 |
| | pH CaCl ₂ | - | 6.7 | ISO 10390 |
| | CEC | mEq/kg | 179 | Cobaltihexamine extraction / ISO 23470 |
| | C/N ratio | - | 9.6 | - |
| | Total organic carbon | | 13.6 | ISO 14235 |
| | Organic matter | | 23.5 | ISO 14235 |
| | Total carbonate | | 8 | ISO 10693 |
| | Total nitrogen | | 1.41 | Dumas method / ISO 13878 |
| | NH ₄ | | 0.0003 | KCl extraction (1 M), Berthelot method |
| | NO ₃ | | 0.012 | for NH ₄ . Cd reduction and Griess method for NO ₃ . NH ₄ + NO ₃ for mineral nitrogen. |
| Nitrogen | Mineral nitrogen | g/kg | 0.012 | |
| | P ₂ O ₅ Olsen | | 0.042 | 0 |
| | Sulfur | | 0.26 | ISO 11466 / ICP AES dosage |
| | K ₂ O exchangeable | | 0.41 | 0 |
| | MgO exchangeable | | 0.20 | Cobaltihexamine extraction / |
| | CaO exchangeable | | 4.65 | ISO 23470 |
| | Na ₂ O exchangeable | | 0.013 | 0.002 |
| Other nutrients | Boron | | 0.50 | Boiling water extraction |
| | Copper | | 2.7 | 0.0 |
| | Iron | | 15.3 | 2.4 |
| | Manganese | mg/kg | 8.4 | DTPA extraction / ICP AES dosage |
| | Zinc | | 2.7 | 0.1 |
| | Chloride | | 9.80 | Water extraction & potentiometric dosage |
| | Molybdenum | | <2 | ISO 11466 / ICP-MS dosage (ISO 17294) |

SM 3-2. Analysis method for plant and fertilizers

Fertilizers analysis:

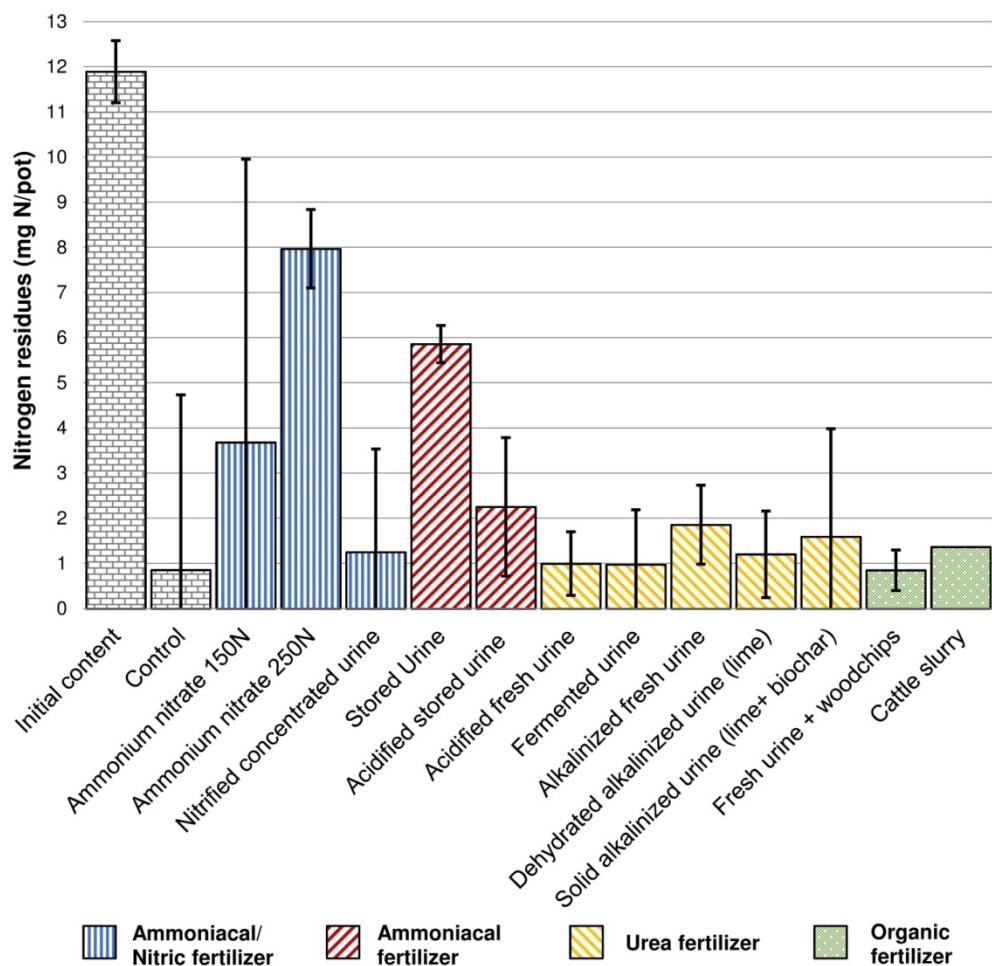
| Parameters | Methods liquid product | Methods for solid product and cattle slurry | | | | | |
|-----------------|---|--|----|----|-------|--|--|
| pH | ISO 10523 | NF EN 13037 | | | | | |
| Conductivity | ISO 7888 | NF EN 13038 | | | | | |
| Dry matter | Determination of dry residue at 105 °C | NF EN 13040 | | | | | |
| Carbon | Tot-C Organic-C Inorganic-C | - NF EN 1484 - | | | | | ISO 10694 ISO 10694 NF U44-001 |
| Nitrogen | Total-N $\text{NH}_4\text{-N}$ $\text{NO}_3\text{-N}$ Urea-N Organic-N | Kjeldahl method + NO_3 Berthelot and Griess method PDAB colorimetry. N total – N mineral – N organic for the mixture with woodchips N total – N mineral | NF | EN | 25663 | Kjeldahl Method + NO_3 (NF EN 13654.-2 for dehydrated alkalinized urine) | |
| Other nutrients | P_2O_5 K_2O MgO CaO Na_2O SO_3 Cl^- B Fe Cu Mn Mo Zn Se As Cd Co Cr Ni Pb | | | | | NF EN 13650 ISO 11885 ISO 10304-1 | ISO 17294 NF EN 13650 ISO 11885 ISO 10304-1 |
| Trace metals | Hg | ISO 1484 | | | | Dry combustion and cold vapor AAS dosage | |

Plant analysis:

The dried samples of each replicate pot and harvest were powdered. Subsamples of 50 mg were digested on 10 mL tubes with 1 mL of 67-70% HNO_3 and allowed to rest overnight prior to boiling for about 4h to almost dryness, then diluted to 10 mL and analyzed for elements by ICP-OES. Internal reference material was used to verify analytical accuracy. Total N was measured by dry combustion with an elemental analyzer (Thermo Scientific, EA1108) via combustion at 900 °C and subsequent analysis of CO_2 and N_2 with gas chromatography.

SM 3-3. Nitrogen residues at the end of the experimentation

An extraction was performed with KCl (1 mol/L). Ammoniacal nitrogen was measured using Berthelot colorimetric method. Nitrite and nitrate were measured using a reduction on a cadmium column and the Griess colorimetric method. Colors correspond to the categories of urine-based fertilizers.



Nitrogen residues were low for each treatment at the end of the experiment indicating a good uptake by crops. They are all below the initial nitrogen content before the launching of the experiment.

Chapitre 4

Efficacité fertilisante azotée et mesures de volatilisation ammoniacale en conditions réelles

Nitrogen fertilizing efficiency of human urine-based fertilizers is similar to mineral fertilizer but with potentially high volatilization of ammonia

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Abstract

Human urine contains large amounts of nutrients today weakly recycled to agriculture. They can be collected through source separation and treated to produce “urine-based fertilizers” with various nutrient contents and physicochemical characteristics. For the first time, we measured the nitrogen fertilizing efficiency of 4 urine-based fertilizers (stored urine from university, stored urine from festival, fermented urine and nitrified urine) compared under field conditions to a mineral fertilizer and 7 other organic fertilizers (bovine slurry, pig slurry compost, bovine manure, horse manure, sewage sludge, bio-waste digestate and dairy manure digestate). The experiments were carried out over 2 years on 6 different sites and on 3 different crops. Additionally, the possibility to completely substitute mineral fertilizer by urine-based fertilizers and the use of urine-based fertilizers in organic farming were studied. The ammonia volatilization of stored urine was also measured in a separate experiment.

Despite high variations, urine-based fertilizers had mineral fertilizer equivalent greater than 70% in most cases and larger than the other organic fertilizers. The differences were mainly explained by the mineral nitrogen content of the fertilizers. The complete substitution of mineral nitrogen by urine-based fertilizers did not lead to yield or grain quality decrease. The experiments with organic farming practices were not concluding due to a poor response to nitrogen input and support a deeper study of the interest of urine-based fertilizers in organic agriculture in interaction with other aspects of the cropping systems. Volatilization of stored urine, which has high ammonia content, was high (34% of the nitrogen applied) compared to ammonium nitrate (1%) due to favorable conditions at spreading. With adequate application practices to limit this volatilization, urine-based fertilizers are promising renewable nitrogen fertilizers. The constraints linked to the field application of large volume of urine-based fertilizers and the fate of urine contaminants call for further investigation.

Key words

Nitrogen; Ammonia volatilization; Fertilization; Human urine; Organic fertilizer; Nutrient recycling;

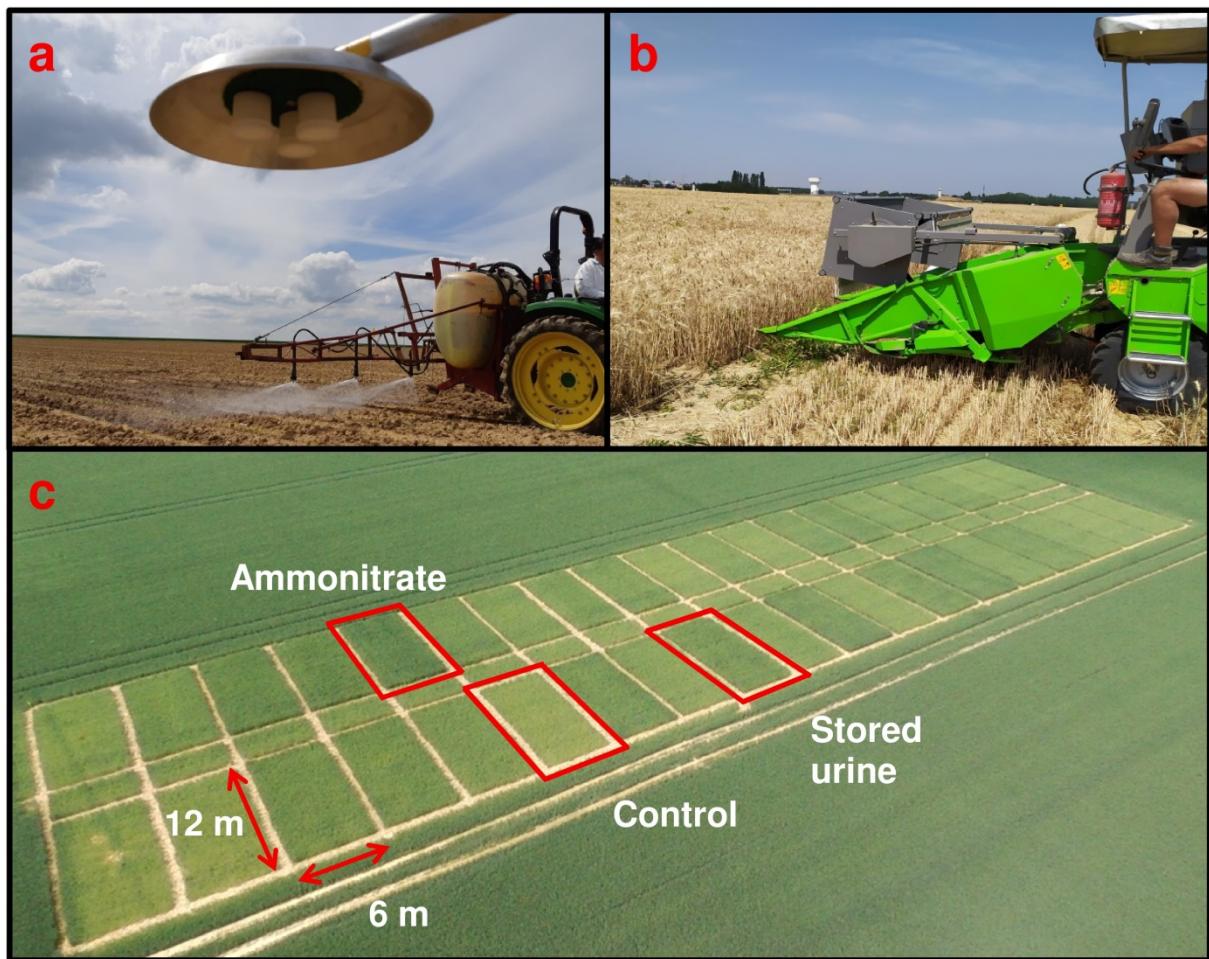


Figure 4-1. Field experiment performed in 2019. (a) Spreading of stored urine in May 2019 (experiment n°6) before maize sowing using an experimental sprayer and the ALPHA badges used to measure ammonia volatilization. (b) Harvest of winter wheat (experiment n°4) in July 2019 using an experimental combine harvester. (c) Aerial view of the mineral fertilizer equivalent and substitution experiments on winter wheat (experiment n°4) in May 2019. Credit: Christophe Dion. One replicate of each treatment of the substitution experiment is encircled in red.

1. Introduction

Conventional agriculture relies on the use of synthetic nitrogen coming from the chemical fixation of atmospheric nitrogen through the Haber-Bosch process. Nitrogen from chemical fixation is now about three times higher than biological fixation and represents 75% of the nitrogen input in agriculture (Matassa et al. 2015). However, it is also an energy intensive process that requires around 1-2% of the current total world energy consumption (Matassa et al. 2015). The use of Haber-Bosch issued nitrogen has permitted a high increase of yield and food production. On the other hand, less than 5% of Paris wastewater nitrogen is currently recycled to agriculture as compared to nearly 50% in the beginning of the 20th century (Esculier et al. 2018). The recovery of all human-derived nitrogen and phosphorus

would permit to offset 16%-21% of inorganic nitrogen use in agriculture (Trimmer et al. 2019), and even more in densely populated areas (e.g., more than 100% in Paris area, calculated from Esculier et al., 2018). Indeed, improving the recycling of these nutrients would make possible to better close the nitrogen biogeochemical cycle (Esculier et al. 2018). Most nutrients present in wastewaters come from urine and in particular around 80% of the nitrogen (Friedler et al. 2013). An improved recycling of human-derived nutrients would thus be facilitated by a separate collection of urine. Source separation of urine can be done using various types of toilets, such as urinals and urine diverting toilets. It avoids nutrient dilution or pathogen contamination by the other components of wastewaters.

The nutrients are excreted in a small volume with about 0.5 m³ of urine per year and per person and diluted in the whole wastewater volume of about 54 m³ per year and per person in high income countries (Friedler et al. 2013). The average nitrogen concentration in fresh urine is around 9 g N/L (Udert et al. 2006; Friedler et al. 2013) with variation with the dietary intake (Trimmer et al. 2019). In fresh urine, about 85% of the nitrogen is excreted as urea, 5% as ammoniacal nitrogen and 10% as organic nitrogen (Udert et al. 2006). During storage of urine and without additional stabilization treatment, urea is rapidly hydrolyzed to ammoniacal nitrogen due to the ubiquitous presence of the urease enzyme secreted by microorganisms (Friedler et al., 2013). Ammoniacal nitrogen is likely to volatilize after application on crops.

Some contaminants may be present in urine. Pathogens mostly come from fecal cross contamination that can occur at the diverting toilet. Most of them can be inactivated under appropriate storage conditions (WHO 2012). Usually, urine concentrations in trace elements are low (Martin et al. in press). The need for specific treatment to remove pharmaceuticals from urine before land application is debated. Indeed, about 70% of pharmaceutical residues excreted by humans are in urine at concentrations from zero to a few mg/L for individuals under medication (Bischel et al. 2015).

After being collected, the urine is treated with different purposes (Martin et al. in press): (i) stabilization of nitrogen, in a form that limits ammonia volatilization, malodor and permits further concentration (e.g., nitrification of ammoniacal nitrogen of stored urine; acidification of fresh urine by lacto-fermentation inhibiting the urea hydrolysis because of the acidification) (ii) volume reduction to reduce storage space, heavy transport over long distance and spreading work, (iii) nutrient extraction to obtain a concentrated final product, (iv) treatments of pharmaceutical residues and/or pathogens to reduce the potential impacts on health and the environment (e.g., storage of urine inactivates most pathogens due to the presence of ammonia).

All these treatments lead to a wide variety of products that can be used in agriculture. The name “urine-based fertilizer” qualifies these products resulting from urine treatments. The characteristics of urine-based fertilizer are diverse, and then the fertilizer efficiency and the related environmental impacts are expected to be different among the end-products.

The storage of urine (stored urine) is the most simple treatment to treat the pathogen contamination (WHO 2012). The nitrogen fertilizing efficiency of stored urine (stored few months before use) has been shown as similar or slightly lower than mineral fertilizers for many crops (Pandorf et al. 2019). The efficiency of other urine-based fertilizers (e.g., nitrified urine and fermented urine) has been little studied. Ammonia volatilization may occur after fertilizer application. It is linked to the ammoniacal content of the fertilizers and is favored by high pH of the fertilizer or the soil. Ammonia is an atmospheric pollutant causing acidification of the environment (e.g., soil, water) and the formation of particulate matter with human health impact. It also represents a loss in the fertilizing efficiency due to losses of nitrogen. It can represent a strong impact from the use of urine-based fertilizers that has been only little studied (Rodhe et al., 2004).

The aim of this study was to measure the nitrogen fertilizing efficiency of different urine-based fertilizers and compare them to other organic fertilizers, in field conditions. A specific experiment was also conducted to measure ammonia volatilization after stored urine application.

2. Material and methods

2.1. Fertilizers tested

Eleven different fertilizers were tested (detailed characteristics in Table 4-3): four urine-based fertilizers (two different stored urines, fermented urine and nitrified urine), seven other organic fertilizers (bovine slurry, bovine solid manure, horse solid manure, compost of the solid part of pig slurry, dehydrated limed sewage sludge, digestate of dairy manure and bio-waste digestate) and one mineral fertilizer (ammonium nitrate).

The stored urine from university used in 2018 and 2019 was collected in a university building using a waterless male urinal. In 2018, urine collection began two years to about six months before the experiment. In 2019, the collection started about six months until one month before the launching of the experiment. Prior to application, the urine was stored in an air-tight tank. Stored urine from festival was collected by a private company at a music festival in August 2018 from dry male urinals. Then it was stored for about 6 months in an airtight tank. The fermented urine was produced by a private company. It was produced as a biostimulant. The treatment was similar to (Andreev et al. 2017) and consisted in the acidification of fresh urine (stabilization of nitrogen as urea) recovered in a festival (different from stored urine festival) with a short storage (two weeks), followed by a long term lactic acid fermentation using malt as inoculum of lactic acid bacteria. This pilot batch was produced by the TOOPI Organics company (www.toopi-organics.com). The nitrified concentrated urine was produced by the company VUNA (www.vuna.ch). The treatment consisted in a biological nitrification

(stabilization of nitrogen as nitrate) of fifty percent of the ammoniacal nitrogen of stored urine followed by a volume reduction by distillation (Fumasoli et al. 2016).

The bovine slurry used in 2018 and 2019 was the liquid phase of the slurry (after phase separation). It was collected a few days before the application, in the storage tank of a conventional dairy farm. The bovine solid manure was collected from the manure pile of the same farm. The horse solid manure was collected from the manure pile of a local horse farm. The composted solid fraction of pig slurry was imported from Brittany (western France) by a local farmer. The dehydrated and limed sewage sludge in 2018 and 2019 were recovered from a local wastewater treatment plant. The digestate of dairy farm manure came from a digester installed in a conventional dairy farm close to the study area (mesophilic anaerobic digestion in an infinitely mixed digester). The main inputs to the digester were bovine slurry, bovine manure, lactoserum and sewage sludge. The bio-waste digestate came from a digester (mesophilic anaerobic digestion in an infinitely mixed digester) located close to the study area fed by food-waste such as supermarket waste, market waste or wastes from the agro-food industry. Both digestates corresponded to the liquid fraction after phase separation.

The following parameters were measured (according to the method used): Dry matter (NF T 90-029), pH (NF ISO 10523), organic carbon (NF EN 1484), total nitrogen (NF EN 25663), C total/N total ratio, ammoniacal nitrogen (NFT 90-015-1 or colorimetry Berthelot), nitric nitrogen (NF EN ISO 15923-1 or colorimetry), urea nitrogen (MA7-15), organic nitrogen (N total – N mineral), total phosphorus (NF EN ISO 17294-2), total potassium (NF EN ISO 17294-2) and total sulfur (NF EN ISO 17294-2). Three samples were analyzed for the urine-based fertilizers but only one for the other organic fertilizers. Solid ammonium nitrate (ammonium nitrate) was used as a reference mineral fertilizer.

Seven pathogen indicators were measured in urine-based fertilizers and bovine slurry (2019): total coliforms (NF V 08-050), *Escherichia coli* (NF EN ISO 16649-2), *Clostridium perfringens*, *Clostridium perfringens* spores, fecal enterococci (NF EN ISO 7899-1), salmonellas (NF EN ISO 6579) and nematode eggs ($MgSO_4$). Three samples were analyzed per fertilizer and the most probable number was calculated (MPN/g raw material)

2.2. General characteristics of the field experiments

The field experiments were carried out on 6 different sites. All sites were located about 15 km to 30 km South-west or west of Paris on the “Saclay plateau” or “Versailles plain”. The climate is an altered oceanic climate with an average temperature of 10.5 °C and approximatively 650 mm of precipitation per year. The region is specialized in arable crops (e.g., winter wheat, rapeseed, barley, maize). All experimental plots were located in the middle of flat and homogeneous agricultural plots (Figure 4-1c). Apart from the nitrogen fertilization and the harvest, the experiments were managed by the farmers with their actual practices (tillage, sowing, weed control, etc.). The main soil

characteristics are presented in Table 4-1. and were measured from 3 different samples randomized in the experiments. For the mineral nitrogen stock, one sample was taken per plot.

Table 4-1. Main soils characteristics for the different experiments. Three samples were analyzed but as the standard error was low, only the mean value is presented. Except the pre-application and post-harvest soil mineral nitrogen stocks, all soil parameters were measured in the first plowed soil horizon (25 cm). CEC stands for cationic exchange capacity. For soil mineral nitrogen stocks, one sample per plot was taken but as they were homogenous, only the mean value of the experiment and the standard error are presented. For each parameter given “per kg”, this means “per kg of dry soil”. **The granulometry could not be measured on this plot but we considered that it was similar to that of experiment 4 as the plots were close to each other.

| Soil parameters | Experiment n° | | | | | | Method | |
|---|----------------------|-----------------------|---------|--------|---------|----------|----------------------------------|--|
| | 1 | 2 | 3 | 4 | 5 | 6 | | |
| Clay (g/kg) | 160 | 160 | 184** | 184 | 197 | 197 | | |
| Fine silt (g/kg) | 250 | 290 | 290** | 290 | 291 | 241 | | |
| Coarse silt (g/kg) | 500 | 480 | 464** | 464 | 456 | 510 | NFX 31-107 with decarbonation | |
| Fine sand (g/kg) | 40 | 50 | 40** | 40 | 39 | 44 | | |
| Coarse sand (g/kg) | 20 | 10 | 20** | 20 | 17 | 6 | | |
| pH water | 7.8 | 5.5 | 6.9 | 7.0 | 6.0 | 6.7 | NF ISO 10 390 | |
| Organic matter (%) or g/100g) | 2.5 | 1.6 | 2.4 | 2.6 | 2.1 | 2.2 | NF ISO 14235 | |
| CEC cobaltihexamine or Metson* (mEq/100 g) | 13.3* | 9.6* | 13.4 | 11.7 | 9.3 | 11.9 | NF ISO 23470 or METSON | |
| Olsen phosphorus (mg P2O5/kg) | 34 | 44 | 59 | 50 | 46 | 93 | NF ISO 11263 | |
| Pre-application soil mineral nitrogen stocks (0-90 cm) (kg N/ha) | 58 ± 8 (2018, 1a) | 46 ± 12 (2019, 1b) | 74 ± 20 | 46 ± 8 | 54 ± 10 | 204 ± 18 | 111 ± 15 | KCl extraction and Berthelot method for NH4. Cd reduction and Griess method for NO3. NH4 + NO3 for mineral nitrogen. |
| Post-harvest soil mineral nitrogen stocks (0-90 cm) (kg N/ha) | 55 ± 7 (2018, 1a) | 68 ± 16 (2019, 1b) | 55 ± 5 | 56 ± 9 | 35 ± 3 | - | - | |

All soils were loamy Luvisol and deep (more than 2 m depth) except for the experiments 2 and 3 for which the depth was about 1 m depth. Such depths ensured a good water availability. The pH ranged from acidic (pH 5.5, experiment 2) to slightly basic (pH 7.8, experiment 1), but most sites had pH between 6 and 7. All soils were decarbonated with less than 0.5% of CaCO₃. The organic matter contents were rather high (2.1% to 2.7%) with regards to the average of the study area (about 1.7 to 1.8%) except experiment 2 for which it was lower (1.6%). No nutrient deficiency was observed, even if the P₂O₅-Olsen was closed to the local threshold for reinforced fertilization: 50 mg P₂O₅/kg for rapeseed and 20 mg P₂O₅/kg for soft wheat and maize. The soil mineral nitrogen stocks (0-90 cm) were measured during the three weeks preceding application and after harvest. They were measured in

each treatment, but they were fairly homogeneous and only the mean value of the experiment with the standard deviation is presented in Table 4-1.

All the field experiments are summarized in Table 4-2. Four types of experiment were performed with different objectives: measurement of nitrogen fertilizer equivalent of the organic fertilizers, maximum substitution of mineral fertilizer (substitution), nitrogen fertilizing efficiency in organic agriculture (organic) and ammonia volatilization. The experiments were carried out during two cultural years from fall 2017 to fall 2019.

Table 4-2. Summary of the experiments carried out.

| Experiment | Type of experiment | Crops | Fertilizers tested | Spreading date | Previous crop |
|------------|-------------------------------|--------------|-----------------------------|------------------|-------------------------------------|
| 1a | Mineral fertilizer equivalent | Winter wheat | Bovine slurry (pre-sowing) | 3 November 2017 | |
| | | | Ammonium | | |
| | | | Stored urine (university) | | |
| | | | Bovine slurry (end winter) | 21 February 2018 | Maize |
| | | | Bio-waste digestate | | |
| 1b | Mineral fertilizer equivalent | Rapeseed | Bovine slurry (pre-sowing) | 27 August 2017 | |
| | | | Ammonium | | |
| | | | Stored urine (festival) | | |
| | | | Bovine slurry (end winter) | 19 February 2019 | Wheat |
| | | | Bio-waste digestate | | |
| 2 | Mineral fertilizer equivalent | Winter wheat | Horse solid manure | | |
| | | | Limed sewage sludge | 3 October 2017 | |
| | | | Ammonium | | |
| | | | Stored urine (university) | 20 February 2018 | Rapeseed |
| 3 | Mineral fertilizer equivalent | Rapeseed | Dairy manure digestate | | |
| | | | Composted solid fraction of | | |
| | | | pig slurry | 29 August 2018 | |
| | | | Limed sewage sludge | | Wheat |
| 4a | Mineral fertilizer equivalent | Winter wheat | Ammonium | | |
| | | | Stored urine (university) | | |
| | | | Stored urine (festival) | | |
| | | | Fermented urine | 19 February 2019 | |
| | | | Nitrified urine | | |
| | | | Bovine slurry | | Sugar beet |
| 4b | Substitution | | | 19 February 2019 | |
| | | | Ammonium | 20 March 2019 | |
| | | | Stored urine (festival) | 17 May 2019 | |
| 5 | Organic | Maize | Bovine slurry + | 5-6 April | Triticale + clover cover crop |
| | | | solid manure | | |
| 6 | Ammonia volatilization | Maize | Stored urine (festival) | 17 May 2019 | |
| | | | Ammonium | | |
| | | | Stored urine (festival) | 22 May 2019 | Wheat |

All experimental plots measured 6 × 12 m, except in the ammonia volatilization experiment (experiment n°6) for which large plots were required (20 × 20 m). In all experiments, three replicate blocks of the different treatments were set up, and all treatments were randomized within each block (only two replicates in the volatilization experiment).

Liquid fertilizers were applied using watering can except stored urine from festival which was applied using an experimental sprayer in experiments 4 and 6 (Figure 4-1a). Solid fertilizers were applied by hand or using an experimental centrifugal broadcaster for ammonium nitrate. The harvest was done using an experimental combine harvester to measure the grain yield (Figure 4-1b). Grains were sampled in each plot to measure the water and nitrogen contents.

2.3. Mineral fertilizer equivalent experiments

The experiments 1a, 1b, 2, 3 and 4a were realized to characterize the nitrogen fertilizing efficiency and mineral nitrogen equivalent of the urine-based fertilizers in conventional agriculture and compare them to other organic fertilizers. The experiment 4 carried out on winter wheat was specifically designed to compare different urine-based fertilizers. In the other experiments (experiments 1, 2 and 3, Table 4-2), a stored urine treatment was compared with local organic fertilizers.

The target doses for urine-based fertilizers were set up to 100 kg N/ha in 2018 and 80 kg N/ha in 2019. The actual nutrient concentrations were measured after the application and the actual applied dose (Table 4-4) varied between 65 kg N/ha and 100 kg N/ha (total nitrogen). The application rates for the other organic fertilizers were in accordance with local farmers practices (Table 4-4), resulting in total nitrogen rates varying from 44 kg N/ha (digestate of dairy manure on winter wheat, experiment 2) to 143 kg N/ha (limed sewage sludge on rapeseed, experiment 3). Besides, one control treatment with no nitrogen fertilization and two mineral N treatments (50 kg N/ha and 100 kg N/ha) were included in all experiments, to design the response curve of nitrogen uptake by grain according to mineral nitrogen application. The slope of this linear relationship was defined as the nitrogen use efficiency of ammonium nitrate. For the urine-based fertilizers and other organic fertilizer treatments, the nitrogen use efficiency was calculated as the ratio between the additional nitrogen uptake by the fertilized plants compared to the plants in the control treatment without fertilization (considered as the supply from the soil) and the nitrogen added by the fertilizer (1):

$$\text{Grain nitrogen use efficiency (\%)} = \frac{N \text{ uptake fertilized grain} - N \text{ uptake control grain}}{N \text{ added by fertilizer}} \times 100 \quad (1)$$

The nitrogen use efficiency of urine-based fertilizers and organic fertilizers was compared with that of mineral fertilizer by calculating the mineral fertilizer equivalent coefficient as the ratio of the nitrogen use efficiency of the urine-based or organic fertilizer and that of the reference mineral fertilizer (2). The mineral fertilizer equivalent determines to which mineral fertilizer dose the urine-based fertilizer (UBF) or organic fertilizer is equivalent (in the conditions of the experiment).

$$\text{Mineral fertilizer equivalent (\%)} = \frac{\text{Nitrogen use efficiency UBF / Organic fertilizer}}{\text{Nitrogen use efficiency mineral fertilizer}} \times 100 \quad (2)$$

For ammonium nitrate, urine-based fertilizers and most of the organic fertilizers, the application was done at the end of winter on wheat at the tillering stage and on rapeseed, which corresponded to

the period of the first mineral fertilizer application by the farmers in the study area. Some organic fertilizers were applied before sowing, in line with farmers practices (and incorporated into the soil) for trafficability or slow mineralization issues: bovine slurry, bovine solid manure, horse manure, limed sewage sludge and compost of the solid part of pig slurry.

2.4. Substitution experiments

The aim of this experiment (experiment 4b) was to study the possibility to completely substitute mineral fertilizers with a urine-based fertilizer to achieve the same yield and the same level of grain quality (e.g., protein content in grain for wheat). The experiment was carried out on winter wheat. They were two different treatments: stored urine (festival) and ammonium nitrate. The inputs were determined based on the farmer practice. The total dose was 190 kg N/ha, divided in three applications: 50 kg N/ha at the tillering stage (19th February), 90 kg N/ha at the ear 1 cm stage (21th March) and 50 kg N/ha at the flowering stage (17th May).

2.5. Nitrogen use efficiency in organic agriculture

The experiment 5 was carried out with practices following the European organic agriculture standard: crop rotations with legumes, no synthetic pesticides, no mineral fertilizers, weeding by tillage, etc. The objective was not to compare to a reference fertilizer but to acquire references on nitrogen use efficiency for little studied organic fertilizers. The target doses for stored urine (festival) was set up to 80 kg N/ha. The bovine slurry + bovine solid manure treatment was the fertilization practice of the farmer and only 2 replicates were set up: 28 m³/ha of bovine slurry and 20 t/ha of bovine solid manure were applied for a total dose of 256 kg N/ha. Bovine slurry was applied on the 5th April using an injector and the bovine solid manure on the 6th April using a manure spreader and incorporated a few hours later. The nitrogen use efficiency was calculated as in the other experiments.

2.6. Ammonia volatilization

The objective was to measure the ammonia volatilization in a maize field (experiment 6) after stored urine application (the most common urine-based fertilizer) relative to ammonium nitrate. Larger plot of 20×20 m were used and 145 kg N/ha of stored urine were brought to the 2-3 leaf stage (22th May). The dose for ammonium nitrate was similar to the farmer's nitrogen dose (156 kg N/ha) but without incorporation into the soil. There were three different treatments: stored urine (festival), ammonium nitrate and a control without fertilization. The application was done using an experimental sprayer for the stored urine (3 m wide, Figure 4-1a.), about 30 cm above ground and without incorporation, and an experimental spreader for ammonium nitrate.

To determine the ammonia fluxes, an atmospheric dispersion model was used. FIDES, is an advection model of gas diffusion in the atmospheric surface layer (Loubet et al. 2018). The inversion

method consists in finding the terms of emission and background concentration of each plot by optimizing the differences between measured and modeled concentrations. The modeling was not a part of the thesis. This model requires air ammonia concentration at the center of the plot (improved accuracy, if the concentration is measured at two heights) and the background concentration measured outside of the experimental plot.

Air ammonia concentrations were measured using ALPHA passive badges (Adapted Low-cost Passive High Absorption, Tang et al. 2001). They consisted of a circular polyethylene envelope with a semi-permeable membrane inside (5 µm porosity) and a filter paper impregnated with citric acid. The membrane allowed the gas diffusion and the acid filter trapped the ammonia (NH_3) contained in the air in the form of ammonium ion (NH_4^+). Two points of measurement to quantify ammonia concentration in the air were installed in the middle of each plot. The heights of the sensors were 0.5 m and 1 m above the ground (not moved during the experimentation). The maize was 5 cm high at the start of the measurement and 30 cm high at the end. Three points of measurement were installed at least 350 m around the experiment plot at 2.2 m above the ground. Three passive sensors were installed per measurement point. After extraction of the acid filters, the ammonium concentration of the extracted solution was determined by conductometry after separation on a semi-permeable membrane (FloRRia, Flow Injection Ammonium Analyzer; EN, Pays-Bas). The concentration of ammonia in the air (in $\mu\text{g}/\text{m}^3$) was calculated according to the exposure time. The measurements were taken from 22th May to 17th June (for 26 days). The passive sensors were changed twice a day at the beginning of the experiment and less frequently at the end (one sampling every three days). Thus, the concentration of ammonia in the air was integrated over a period of a few hours to a few days, to have the dynamic of volatilization.

The climatic conditions were monitored every 30 min during the experimentation using a weather station. During the first 48 hours of the experiment (during which most of the volatilization occur), the air temperature ranged between 6.3 °C and 22.4 °C (mean value 15.4 °C) and the mean wind speed was 1.1 m/s. The solar radiations were high; the maximum value on the first two day after the application was above 900 W/m². There was no rain for the first week after application.

2.7. Statistical analyses

The results are expressed using the mean of the replicates with the standard deviation (std or ±). Significant differences between treatments were tested using an ANOVA test followed by a Tukey HSD post hoc test (p-value ≤ 0.05). All tests were performed using the “R” software, version 3.3.2.

3. Results and discussion

3.1. Physico-chemical characteristics of the fertilizers and pathogens

The nutrient contents and physico-chemical characteristics of the tested fertilizers are presented in Table 4-3. All urine-based fertilizers had much lower contents in nitrogen than ammonium nitrate. Non-concentrated urine-based fertilizers typically had a concentration between 2 and 7 g N/kg and the N content in concentrated nitrified urine was 44 g N/kg. However with other concentration process, it can possibly exceed 100 g N/kg (Simha et al. 2020). Urine collected from festivals (stored urine festival and fermented urine) had lower nitrogen concentration certainly due to large drunk volumes resulting in water excretion in urine and due to the cleaning of the urinals. Non-concentrated urine-based fertilizers (stored and fermented urine) had a nitrogen concentration similar to that of liquid organic fertilizers (slurries and digestates) or solid manure. However, the nitrogen concentration was lower than in other solid organic fertilizers (sewage sludge and pig slurry compost).

Most urine-based fertilizers had nitrogen forms similar to typical mineral nitrogen fertilizer: urea, ammoniacal or nitric nitrogen. Ammoniacal nitrogen was the main form of nitrogen in stored urine. Stored urine (university) collected in 2018 had about 19% of total nitrogen under organic form but organic nitrogen was not present in urine collected at the university in 2019 or from festival. Stored urine (university) collected in 2018 was stored longer time (more than 6 months) than the urine collected in 2019 or urine from festival. Then, the organic fraction of the fresh urine may have been mineralized in “younger” samples but some nitrogen could be “re”-immobilized by microorganisms during the storage of “older” samples. Further analyses are needed to confirm this hypothesis. For the fermented urine about 56% of nitrogen was under ammoniacal form and 38% was stabilized under urea form. Indeed, acidification to a pH below 4 prevents urea hydrolysis during storage and maintains nitrogen as urea (Martin et al. in press). In nitrified urine, approximately 50% of the ammonium content of urine was converted to nitrate. Due to the supply of oxygen and aerobic conditions during nitrification, heterotrophic bacteria degraded approximately 90% of the organic substances contained in urine which explained the lack of organic nitrogen (Fumasoli et al. 2016). In the bovine slurry collected in 2019, about 60% of the nitrogen was under organic form and 40% as ammoniacal nitrogen as classically observed (Houot et al. 2014). Organic nitrogen was the main form of nitrogen in manures, compost, dairy manure digestate and the limed sewage sludge of 2018. The other organic fertilizers (bio-waste digestate, slurries and limed sewage sludge of 2019) were more balanced between organic and ammoniacal nitrogen. The different samples of bovine slurry were similar in terms of characteristics and nutrient concentration. Limed sewage sludge collected in 2018 showed a lower total nitrogen concentration compared to the sample collected in 2019. Organic nitrogen was the main form of nitrogen in 2018, while it was balanced between urea and organic nitrogen in 2019. Bio-waste digestate characteristics were similar in 2018 and 2019.

Table 4-3. Nutrient content and physicochemical characteristics of the mineral fertilizer, urine-based fertilizers and organic fertilizers tested. All the parameters are expressed per kg of raw material. Three samples were analyzed but as the standard error was low, only the mean value is presented. The density of all liquids urine-based fertilizers was taken as 1 except nitrified urine for which it is 1.14. Values replaced by “–“ are not available. Dry matter or dry residues were measured at 105°C.

| Category | Fertilizer | Dry matter (%) | pH | Organic carbon (g C/kg) | Total nitrogen (g N/kg) | C total/ N Total | Ammoniacal nitrogen (g N/kg) | Nitric nitrogen (g N/kg) | Urea nitrogen (g N/kg) | Organic nitrogen (g N/kg) | Total phosphorus (g P ₂ O ₅ /kg) | Total potassium (g K ₂ O/kg) | Total sulfur (g SO ₃ /kg) |
|--------------------------------|---------------------------------|----------------|-----|-------------------------|-------------------------|------------------|------------------------------|--------------------------|------------------------|---------------------------|--|---|--------------------------------------|
| Mineral fertilizer | Ammonium nitrate | - | - | - | 335 | - | 168 | 167 | - | - | - | - | - |
| Urine-based fertilizers | Stored urine (university 2018) | 1.4 | 9.2 | 3.3 | 7.0 | 0.5 | 5.0 | 0 | 0.6 | 1.38 | 0.6 | 2.4 | 0.6 |
| | Stored urine (university 2019) | 1.3 | 9.1 | 2.3 | 6.8 | 0.3 | 6.6 | 0 | 0.3 | 0 | 0.7 | 2.5 | 1.1 |
| | Stored urine (festival) | 0.7 | 7.0 | 4.5 | 3.8 | 1.2 | 3.7 | 0 | 0.3 | 0 | 0.5 | 1.2 | 0.6 |
| | Nitrified urine | - | 4.3 | 0.8 | 44.2 | 0.0 | 21.1 | 23.1 | 0.2 | 0 | 4.9 | 21.6 | 6.7 |
| | Fermented urine | 1.7 | 4.1 | 7.4 | 2.4 | 3.0 | 1.4 | 0 | 0.9 | 0.13 | 0.7 | 0.6 | 0.4 |
| Organic fertilizers | Bovine slurry 2018 (pre-sowing) | 5.4 | - | 18.2 | 3.9 | 4.7 | 1.3 | 0 | 0.2 | 2.4 | 1.3 | 3.1 | - |
| | Bovine slurry 2018 (end winter) | 5.4 | - | 18.4 | 3.9 | 4.7 | 1.3 | 0 | 0.2 | 2.3 | 1.3 | 2.9 | - |
| | Bovine slurry 2019 (pre-sowing) | 4.1 | - | 14.1 | 4.0 | 3.6 | 0.8 | 0 | 0.2 | 2.9 | 1.2 | 3.0 | - |
| | Bovine slurry 2019 (end winter) | 5.9 | 8.1 | 21.6 | 3.7 | 5.8 | 1.6 | 0 | 0.2 | 2.0 | 1.3 | 3.0 | 0.6 |
| | Bovine solid manure | 21.1 | - | 77.1 | 4.1 | 18.7 | 0.1 | 0.6 | 0 | 3.5 | 2.3 | 8.8 | - |
| | Horse solid manure | 37.3 | - | 163 | 4.0 | 40.5 | 0.2 | 0 | 0 | 3.8 | 2.5 | 7.4 | - |
| | Pig solid slurry compost | 48.2 | - | 149 | 17.4 | 8.6 | 0.1 | 0 | 0 | 17.2 | 56.1 | 3.2 | - |
| | Limed sewage sludge 2018 | 32.3 | - | 79.4 | 9.3 | 8.5 | 0.9 | 0 | 0.5 | 8.0 | 18.3 | 0.4 | - |
| | Limed sewage sludge 2019 | 31.9 | - | 93.7 | 11.9 | 7.9 | 0.3 | 0 | 6.2 | 5.5 | 15.0 | 0.6 | - |
| | Dairy manure digestate | 2.8 | - | 9.6 | 1.9 | 5.0 | 0.6 | 0 | 0.1 | 1.3 | 0.7 | 3.3 | - |
| Bio-waste digestate 2018 | Bio-waste digestate 2018 | 1.7 | - | 3.6 | 6.4 | 0.6 | 2.8 | 0 | 0.1 | 3.5 | 0.7 | 1.8 | - |
| | Bio-waste digestate 2019 | 2.1 | 8.5 | 4.6 | 7.3 | 0.6 | 4.0 | 0 | 0.1 | 3.3 | 0.8 | 1.6 | - |

Organic carbon content were low in most urine-based fertilizers except in fermented urine for which it was higher because organic substrate was added during the process. Organic carbon was higher in stored urine from festival but it may be due to a contamination occurring during transport as the truck used usually transports other liquid organic fertilizers and was not completely clean despite tank cleaning.

All urine-based fertilizers and organic fertilizers were multi-nutrient fertilizers. For urine-based fertilizers, phosphorus, potassium and sulfur inputs were 103 to 289 mg P₂O₅, 239 to 490 mg K₂O and 155 to 173 mg SO₃ for one gram of nitrogen applied. The other organic fertilizers also brought significant amounts of other nutrients, often more than with the urine-based fertilizers, e.g., 333 mg P₂O₅ and 797 mg K₂O for one gram of nitrogen applied with bovine slurry 2018 (end winter).

The nine pathogen indicators measured in the urine-based fertilizers were much lower than in bovine slurry and below the detection limit for most of them. Fecal enterococci were quantified in stored urine from festival ($163 \pm 17/g$), stored urine from university ($129 \pm 89/g$) and fermented and nitrified urine ($16 \pm 8/g$). However the contamination was several orders lower than in bovine slurry ($7061 \pm 1982/g$). *Clostridium perfringens* was measured in stored urine from festival ($353 \pm 55/g$) but about ten times lower than in bovine slurry ($3033 \pm 551/g$). Contamination may have occurred during the collection of urine at the festival or during the transportation. Salmonella and helminth eggs were absent in all samples. *Escherichia coli*, total coliforms and *Clostridium perfringens* spores were only quantified in bovine slurry (respectively 333 ± 231 , 587 ± 12 and $217 \pm 151/g$). This agrees with previous studies which found that most pathogen microorganisms can be inactivated under appropriate storage conditions due to high NH₃ concentration and temperature (WHO 2012). As shown by Lahr et al. (2016), *Clostridiales* can survive in stored urine and *C. perfringens* in presumably cross-contaminated festival urine was the only sample and parameter that exceeded the French standard proposed for composted sludges (NFU 44-095) that requires less than 100/g *C. perfringens* for market garden. However, it remained under the limit for other cultures (less than 1000/g). The nitrified urine was distilled at a temperature higher than 80°C and stored urine samples were collected from a dry male urinal (without fecal cross contamination) and stored for at least one month. This can explain the low concentration of pathogens in these urine-based fertilizers.

3.2. Mineral nitrogen equivalent for organic and urine-based fertilizers

The nitrogen input, grain yields, grain nitrogen contents, grain nitrogen uptakes, nitrogen use efficiencies and mineral fertilizer equivalent obtained for all urine-based and other organic fertilizers in the different experiments are presented in Table 4-4.

Table 4-4. Main results of the mineral fertilizer equivalent experiments. Nitrogen use efficiency and mineral fertilizer equivalent are based on grain uptake. Urea nitrogen is considered as mineral nitrogen. “DM” stands for dry matter. For winter wheat in experiment 1a, due to an error at the input, only one replicate was set up for the bovine slurry treatments.

| Experiment | Treatment | Nitrogen Input (kg N/ha) | | | Grain yield (T DM/ha) | | Nitrogen content grain (kg N/T DM) | | Grain nitrogen uptake (kg N/ha) | | Nitrogen use efficiency (%) | | Mineral fertilizer equivalent (%) | |
|-------------------------------------|-----------------------------------|-----------------------------|---------|-------|--------------------------|-----|--|-----|---------------------------------------|-----|-----------------------------------|-----|---|-----|
| | | Mineral | Organic | Total | Mean | std | Mean | std | Mean | std | Mean | std | Mean | std |
| Winter Wheat Experiment n°4a (2019) | Control 0 N | 0 | 0 | 0 | 5.9 | 0.3 | 14 | 1 | 78 | 5 | - | - | - | - |
| | Ammonitrat dose 50 N | 50 | 0 | 50 | 6.9 | 0.4 | 14 | 1 | 95 | 9 | 40 | 6 | - | - |
| | Ammonitrat dose 100 N | 100 | 0 | 100 | 8.3 | 0.4 | 14 | 1 | 118 | 12 | - | - | - | - |
| | Stored urine (university) | 79 | 0 | 79 | 7.5 | 0.3 | 14 | 0 | 108 | 7 | 38 | 9 | 95 | 23 |
| | Stored urine (festival) | 82 | 0 | 82 | 7.6 | 0.3 | 14 | 0 | 104 | 4 | 33 | 5 | 81 | 11 |
| | Fermented urine | 61 | 3 | 65 | 7.1 | 0.1 | 14 | 0 | 99 | 2 | 32 | 3 | 80 | 6 |
| | Nitrified urine | 65 | 0 | 65 | 7.5 | 0.5 | 14 | 1 | 108 | 13 | 47 | 20 | 116 | 51 |
| Winter Wheat Experiment n°2 (2018) | Bovine slurry | 42 | 35 | 78 | 6.5 | 0.5 | 14 | 0 | 90 | 7 | 15 | 9 | 38 | 24 |
| | Control 0 U | 0 | 0 | 0 | 4.8 | 0.8 | 16 | 0 | 78 | 11 | - | - | - | - |
| | Ammonitrat dose 50 N | 50 | 0 | 50 | 6.2 | 0.8 | 18 | 2 | 114 | 28 | 42 | 15 | - | - |
| | Ammonitrat dose 100 N | 100 | 0 | 100 | 6.7 | 0.2 | 18 | 1 | 120 | 5 | - | - | - | - |
| | Stored urine (university) | 80 | 20 | 100 | 6.9 | 0.2 | 18 | 0 | 124 | 2 | 47 | 2 | 111 | 4 |
| | Horse solid manure | 4 | 76 | 80 | 5.0 | 1.2 | 17 | 1 | 87 | 27 | 11 | 34 | 27 | 81 |
| | Limed sewage sludge | 16 | 96 | 112 | 6.0 | 0.6 | 17 | 0 | 100 | 10 | 20 | 9 | 47 | 22 |
| Winter wheat Experiment n°1a (2018) | Dairy manure digestate | 14 | 30 | 44 | 5.4 | 0.8 | 16 | 0 | 86 | 10 | 19 | 23 | 46 | 55 |
| | Control 0 U | 0 | 0 | 0 | 5.0 | 0.5 | 16 | 0 | 95 | 8 | - | - | - | - |
| | Ammonitrat dose 50 N | 50 | 0 | 50 | 6.2 | 0.5 | 16 | 0 | 115 | 8 | 61 | 13 | - | - |
| | Ammonitrat dose 100 N | 100 | 0 | 100 | 7.5 | 0.9 | 18 | 2 | 159 | 34 | - | - | - | - |
| | Stored urine (university) | 80 | 20 | 100 | 7.1 | 0.7 | 17 | 1 | 140 | 24 | 45 | 24 | 73 | 40 |
| | Bovine slurry (pre-sowing) | 38 | 79 | 117 | 5.3 | - | 17 | - | 107 | - | 10 | - | 16 | - |
| | Bovine slurry (end winter) | 35 | 69 | 104 | 5.3 | - | 16 | - | 101 | - | 5 | - | 9 | - |
| Rapeseed Experiment n°3 (2019) | Bio-waste digestate | 43 | 56 | 99 | 6.5 | 0.1 | 16 | 1 | 126 | 5 | 31 | 5 | 50 | 8 |
| | Control 0 U | 0 | 0 | 0 | 1.6 | 0.4 | 31 | 1 | 56 | 13 | - | - | - | - |
| | Ammonitrat dose 50 N | 50 | 0 | 50 | 2.4 | 0.4 | 31 | 1 | 83 | 13 | 27 | 10 | - | - |
| | Ammonitrat dose 100 N | 100 | 0 | 100 | 2.4 | 0.2 | 31 | 2 | 83 | 2 | - | - | - | - |
| | Stored urine (festival) | 82 | 0 | 82 | 2.2 | 0.2 | 31 | 2 | 75 | 10 | 23 | 13 | 88 | 48 |
| | Pig solid slurry compost | 1 | 69 | 70 | 1.7 | 0.3 | 32 | 2 | 62 | 15 | 8 | 22 | 29 | 83 |
| | Limed sewage sludge | 77 | 66 | 143 | 2.2 | 0.5 | 32 | 1 | 77 | 18 | 15 | 12 | 56 | 46 |
| Rapeseed Experiment n°1b (2019) | Control 0 U | 0 | 0 | 0 | 1.9 | 0.2 | 32 | 1 | 59 | 5 | - | - | - | - |
| | Ammonitrat dose 50 N | 50 | 0 | 50 | 2.5 | 0.3 | 31 | 1 | 79 | 7 | 44 | 7 | - | - |
| | Ammonitrat dose 100 N | 100 | 0 | 100 | 3.0 | 0.3 | 34 | 2 | 103 | 12 | - | - | - | - |
| | Stored urine (festival) | 82 | 0 | 82 | 2.5 | 0.1 | 31 | 1 | 78 | 5 | 23 | 6 | 52 | 14 |
| | Bovine slurry (pre-sowing) | 25 | 94 | 119 | 2.4 | 0.6 | 31 | 2 | 76 | 25 | 14 | 21 | 31 | 47 |
| | Bovine slurry (end winter) | 47 | 65 | 112 | 2.3 | 0.2 | 32 | 0 | 74 | 5 | 13 | 4 | 30 | 9 |
| | Bovine solid manure | 20 | 104 | 124 | 2.0 | 0.1 | 32 | 3 | 63 | 6 | 3 | 5 | 7 | 12 |
| | Bio-waste digestate | 49 | 41 | 90 | 2.8 | 0.3 | 32 | 1 | 89 | 8 | 33 | 9 | 75 | 21 |

The crop yields measured in each experiment could not be directly compared because the nitrogen inputs were not the same. However a significant yield and grain increase with nitrogen addition was observed in all experiments making possible the calculation of fertilizer equivalent for the organic fertilizers. In most cases, the nitrogen content in grain was not significantly influenced by the nitrogen input, except for some mineral treatments in experiments 2 and 3.

The nitrogen use efficiency was calculated for the grains and not all above ground biomasses. The nitrogen use efficiency depends on the nitrogen dose, the crop, the climatic conditions and the soil characteristics since high soil mineralization may reduce the nitrogen use efficiency due to the nitrogen available from soil. Nitrogen losses through leaching and gas emissions may also impact the plant nitrogen use. Water or other nutrient deficiencies may also limit the nitrogen absorption by the plant (Krupnik et al. 2004; Semenov et al. 2007). Nitrogen use efficiency for ammonium ranged from 27% for rapeseed (experiment 3) to 61% for winter wheat (experiment 1b).

The nitrogen use efficiency calculated for total above ground biomass of cereals was generally about 60 to 70% (Schjoerring et al. 1995). For wheat in Europe, Krupnik et al. (2004) reported an average nitrogen use efficiency in grain of about 41-43%. In experiments 2 and 4a, the nitrogen use efficiencies of ammonium were 42% and 40%, respectively and highly similar to the values reported by Krupnik et al. (2004). In 2019, the climatic conditions were good and favored high yield for wheat (regional yield of the year of 7.0 t DM per hectare), as measured in the control without fertilization with 6 t DM/ha in experiment 4a. The nitrogen use efficiency of ammonium was even higher in experiment 1a with 61%.

The nitrogen use efficiency calculated for the total above ground biomass of rapeseed was usually lower (50%) than for cereals (60 to 70%). Considering about 80% of the nitrogen uptake in the seeds (Schjoerring et al. 1995), the nitrogen use efficiency for grain should be about 40%. The nitrogen use efficiency of ammonium for rapeseed observed in experiment 1b was highly similar with 44%. However, the response to nitrogen addition by rapeseed was not very good in experiment 3, as there was no difference in yields and nitrogen uptakes between ammonium at doses of 50 and 100 kg N/ha. The nitrogen use efficiency for ammonium was 27% and lower than expected. The lower value may be explained by a crop not very well implanted due to dry conditions at sowing in August 2018. Furthermore, they may have suffered from a lack of water during the growth due to dry conditions in 2019 and the lower water reserve in the soil of this experiment. High temperature may have favor soil organic N mineralization, then decreasing the nitrogen use efficiency.

As shown in Table 4-1, the soil mineral nitrogen stocks before application were low (46 kg N/ha experiment 1b and 3) to medium (74 kg N/ha, experiment 2) and permitted a good valorization of the nitrogen brought by the fertilizers. They were homogeneous among all the different treatments inside each experiment. Post-harvest soil mineral nitrogen stocks were also low to moderate in each

experiments (mean value of the experiments from 35 to 68 kg N/ha). They were homogeneous among all the different treatments indicating a good uptake of available nitrogen from fertilizers by crops.

3.3. Mineral fertilizer equivalents of urine-based fertilizers and comparison to other organic fertilizers

No statistically significant differences were observed among nitrogen use efficiency and mineral fertilizer equivalent within each experiment due to high standard deviation but trends can be interpreted (Figure 4-2). Mineral fertilizer equivalents ranged from 7% (bovine solid manure, rapeseed, experiment 1b) to 116% (nitrified urine, winter wheat, experiment 4a). No significant differences in mineral fertilizer equivalent between wheat and rapeseed were observed.

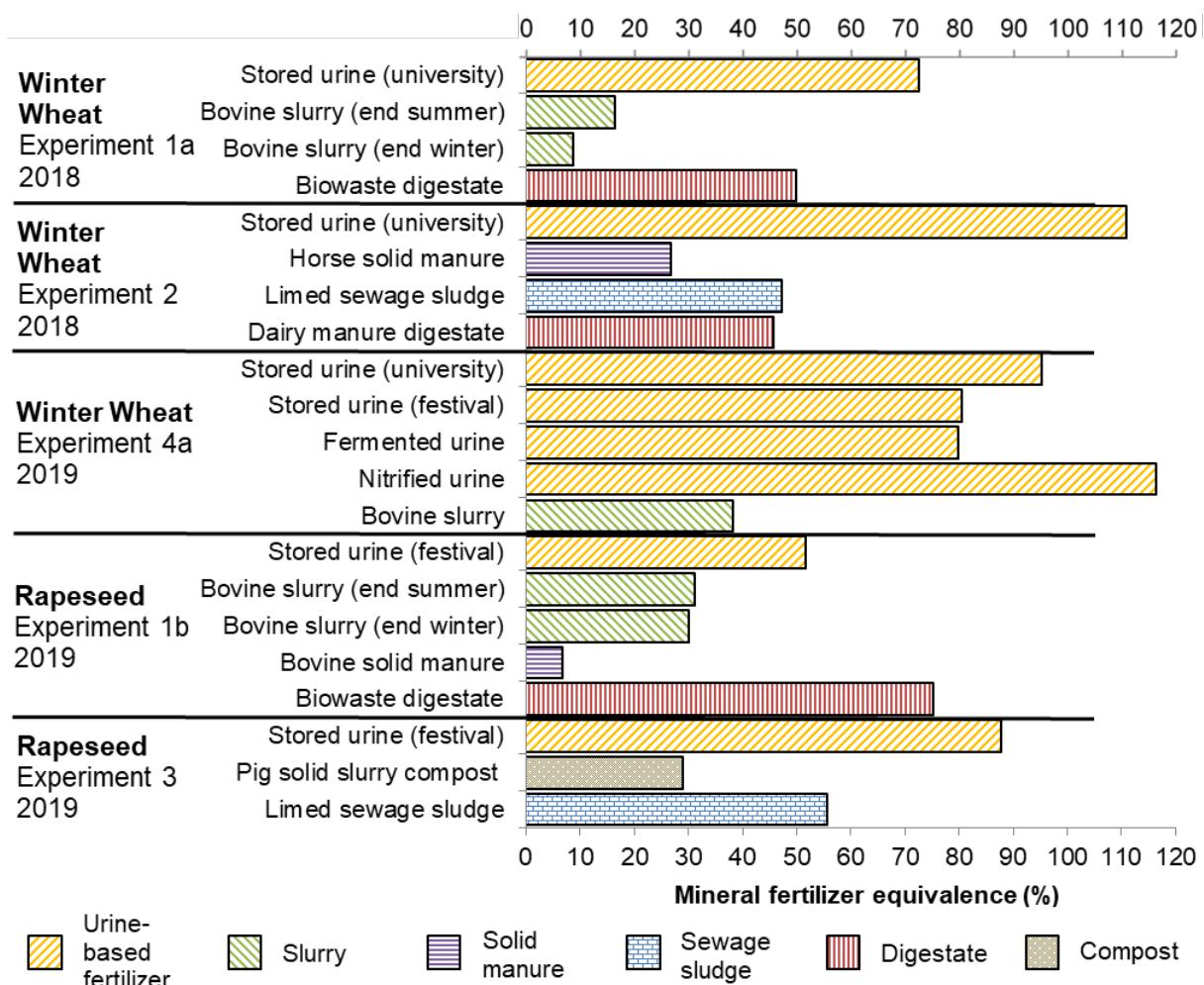


Figure 4-2. Mineral fertilizer equivalent (in %) for the urine-based and other organic fertilizers tested in the different experiments.

The mineral fertilizer equivalent of bovine slurry (experiment 1a, 1b and 4a) varied between 8% (experiment 1a) to 38% (experiment 4a) without clear relationship with the application period before or after winter. They were lower than expected for experiment 1a (Gutser et al. 2005), maybe in

relation to ammonia volatilization. The mineral fertilizer equivalent of sewage sludge in experiments 2 and 3 were similar with respectively 47% and 56%. They were higher than expected for dehydrated sewage sludge despite their high organic nitrogen content (Gutser et al. 2005). Some organic nitrogen mineralization should have occurred during the experimentation. High efficiency in experiment 3 may be linked to the higher urea content in the sample of this year. The two tested digestates showed similar and high mineral fertilizer equivalent ranging from 46 to 75%. They were the most efficient organic fertilizers due to their high ammoniacal nitrogen content (about 32% to 55%). Solid manure (bovine and horse) showed a low mineral fertilizer equivalent ranging from 7% (bovine solid manure, experiment 1b) to 27% (horse solid manure, experiment 2) due to their lower mineral nitrogen content. Finally, mineral fertilizer equivalence of the compost of pig slurry solid phase (experiment 3) was 29% and was higher than expected despite nearly all nitrogen under organic form (Gutser et al. 2005).

Among the different urine-based fertilizers tested, only stored urine (festival) on rapeseed (experiment 1b) had a low mineral fertilizer equivalent of 52%. Compared to the other rapeseed experiment 3, as the crops were more developed when application was done, some of the urine may have remained on the leaves increasing ammonia volatilization. In all other experiments, the urine-based fertilizers showed a high efficiency generally higher than 70% on both wheat and rapeseed indicating a behavior similar to mineral fertilizers. Stored urine from festival and university showed mineral fertilizer equivalence between 73% and 95% (experiment 1a, 3 and 4a). In the experiment 4a, stored urine from university was higher than stored urine from festival (95% versus 81%). Even if the volatilization was expected to be low with the climate conditions (cold temperature) during application, the use of the experimental sprayer for stored urine (festival) may have favored it because of the fine droplets compared to the stored urine (university) applied using a watering can and may explain the slightly lower mineral fertilizer equivalent for stored urine from festival. Additionally, the applied dose was less easy to control with the experimental sprayer. In experiment 2, the mineral fertilizer equivalent of stored urine (university) and nitrified urine were high with 111% and 116%, respectively. The low pH and half of nitrogen under nitrate form can have limited the volatilization and increased the mineral fertilizer equivalent of nitrified urine. Among the urine-based fertilizers tested, fermented urine have a slightly lower mineral fertilizer equivalent compared to stored urine from university. It may be due to nitrogen immobilization in the soil after urea hydrolysis because of the bacteria present in fermented solution.

The mineral fertilizer equivalents were higher for urine-based fertilizer than other organic fertilizers and close to the efficiency of ammonium with a mineral fertilizer equivalent higher than 70% in most cases. The main factor explaining the mineral fertilizer equivalent appears to be the form of nitrogen and especially the mineral nitrogen content of fertilizers (Figure 4-3). Indeed, organic nitrogen must be mineralized before being available to plants. Depending on the stability of the organic matter, only a small part of the organic nitrogen may be mineralized during a cultural year.

Urine-based fertilizer and organic fertilizers may also have a multi-nutrient effect compared to ammonium nitrate and it may explain a mineral fertilizer equivalent above 100% for nitrified urine. In all of these experiments, the volatilization of ammonia should have been in general limited. Indeed, the application was made at the end of winter in February and the temperature was low (lower in 2018 than in 2019). However, it may have participated to lower a bit the mineral fertilizer equivalent of urine-based fertilizers.

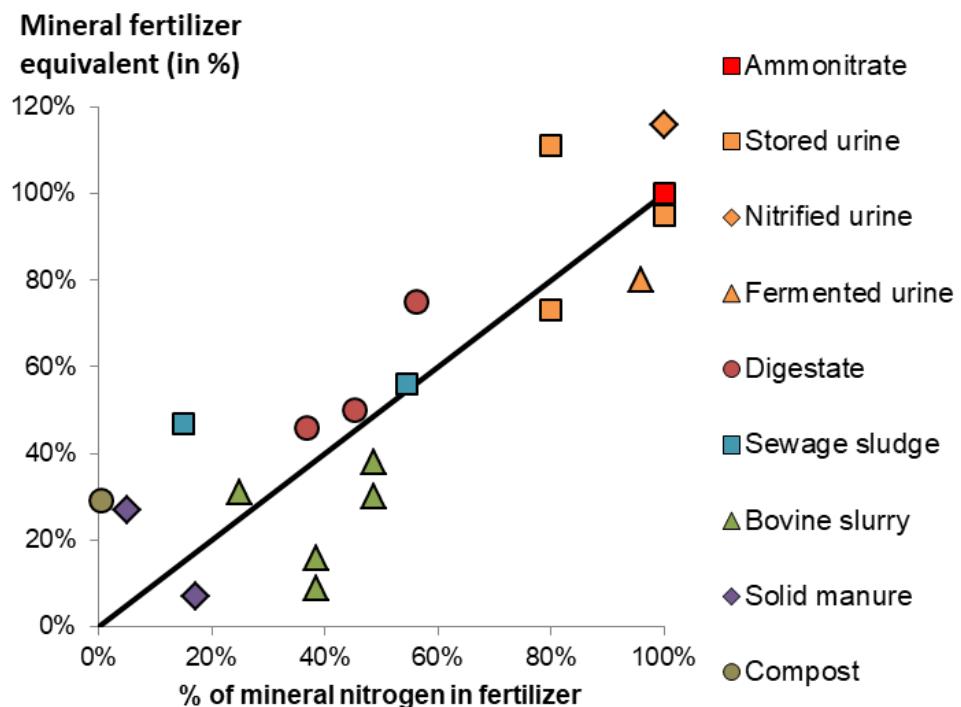


Figure 4-3. Relation between the percentage of mineral nitrogen in the fertilizers (including urea) and the mineral fertilizer equivalent.

Urine-based fertilizers have a high mineral fertilizer equivalent and high mineral nitrogen content. For fertilizers below the black line, losses such as volatilization or immobilization of nitrogen may have occurred. This was the case for a part of the bovine slurry samples where volatilization may have occurred. On the contrary, some of the fertilizers were above the black lines indicating a possible mineralization of the organic fraction of the fertilizer. It is the case for example of one of the sewage sludge sample and for the pig slurry compost.

The mineral fertilizer equivalents obtained for urine-based fertilizers were consistent with the previous experiments. High mineral fertilizer equivalent was measured for nitrified urine (e.g., Bonvin et al. 2015). Stored urine has been tested on different crops and generally showed similar or slightly lower efficiency compared to mineral fertilizers (e.g., Viskari et al. 2018).

Most of these experiments were set up after some requests of the local farmers. It was decided to establish them in their fields to promote the share of knowledge on urine based fertilizers and other organic fertilizers. This implied that we did not control all parameters and that a pre-experiment

homogenization culture was not possible. In addition, many soils in Paris region are rich and deep and are not highly sensitive to deficiencies in a one-year experiment. These elements could explain the high variability of the results and the lack of statistically significant effect for MFE. For further experimentations, a no-fertilized previous crop, larger experimental plots, a higher number of repetitions or a multi-year experiment could increase the significance of the results.

3.4. Fertilization strategies

3.4.1. Complete substitution of mineral fertilizers using urine-based fertilizer is possible

The substitution experiment (experiment 4b) was carried out on winter wheat. The target dose was 190 kg N/ha per hectare and was achieved using ammonium nitrate. However, the stored urine (festival) was a bit less concentrated than expected and the actual dose was 172 kg N/ha. The grain yield and the protein content of the grain are shown in Figure 4-4.

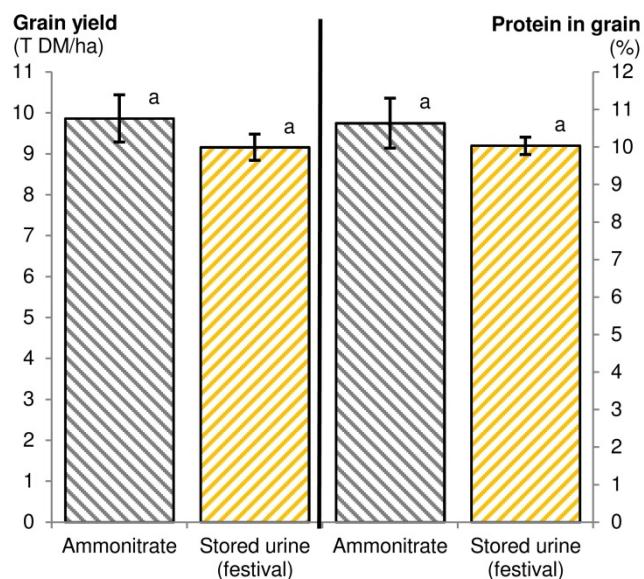


Figure 4-4. Winter wheat grain yield and protein content in the substitution experiment. Nitrogen input was 190 kg N/ha for ammonium nitrate and 172 kg N/ha for stored urine (festival). Statistically significant differences ($p\text{-value} \leq 0.05$) among treatment are represented by letters.

Despite the slightly different N amount brought with stored urine and ammonium nitrate, the yields were very high and not statistically different with 9.9 t DM/ha for ammonium nitrate and 9.2 t DM/ha for stored urine (festival). They were much higher than the regional yield of the year (7.5 t DM/ha). These high yields were due to a very good climatic year for winter wheat. Thus, we observed high crop yield even without mineral fertilizer (5.9 t DM/ha in the control treatment of the 4a experiment). Post-harvest soil mineral nitrogen stocks were low for both fertilizers with 31 kg N/ha for stored urine and 30 kg N/ha for ammonium nitrate indicating a good uptake by crops. The protein contents were not statistically different

between the 2 treatments. However, the protein content was not very high. In general, at least 10.5-11% of protein is expected for uses such as in flour milling. When the yields are high, a dilution of proteins due to high grain production may occur and may explain the lower protein content. Usually, the third input at the flowering stage is sized to produce protein in the grain. The lower protein content for stored urine may be partly due to poor conditions of application for this 3rd input. Indeed, it was done using a watering can but the wheat was already high and it was difficult to apply urine evenly.

This raises the question of the technical constraints of application of urine-based fertilizers. They exhibit high differences in nitrogen concentration leading to very different rates of applications for similar amounts of N. For example, to bring 190 kg N/ha (as in the experiment), 49.6 t/ ha of stored urine (festival) was necessary but only 4.3 t/ ha of nitrified (concentrated) urine and only 0.57 t/ ha of ammonium nitrate. If non-concentrated urine-based fertilizers are applied using heavy machine such as a conventional slurry spreader, it can hardly be done at the end of winter because of their heavy mass, low soil trafficability at this season. In addition, it can damage the crop and cause other impacts such as soil compaction (Arvidsson and Håkansson 1991). For winter wheat example, the first and even the second inputs may be done in non-concentrated liquid form using conventional spreading machinery (e.g., trailing hoses) without important damage to crops before the wheat is too high. However, in the advanced growth stages, a solid urine-based fertilizer or a concentrated liquid urine-based fertilizer such as dehydrated alkalinized urine (Simha et al. 2020) for the last input may be more efficient and less impacting for the crop.

Most nitrogen fertilizers are produced using the Haber-Bosch process and their production request a high energy consumption (Matassa et al. 2015). For cereal production, the use of mineral fertilizers is among the main contributors to the carbon footprint of farms (Autret et al. 2019). With adapted forms of fertilizer, the complete substitution of mineral fertilizer by urine-based fertilizers seems possible and could thus greatly improve the GHG balance of wheat production. However, as the treatments of urine are various (e.g., energy for volume reduction), the entire value chain should be taken into account.

3.4.2. Use of urine-based fertilizers in organic agriculture

In the experiment realized with organic practices (Figure 4-5), the reached yields were high and higher than the regional yield of the year in conventional agriculture (7.0 t DM/ha). The nitrogen grain uptake was 86 kg N/ha in the control, 97 kg N/ha in the stored urine (festival) treatment and 115 kg N/ha in the bovine slurry + solid manure treatment. The yield and nitrogen uptake by the grain were significantly higher for bovine slurry + solid manure than for the control but the nitrogen input was very high in this treatment. Soil mineral nitrogen stocks before application was also very high (204 ± 18 kg N/ha) and may explain that the nitrogen supply from the soil was enough for most of the crop requirements. The cover crop during the intercropping before maize was clover and may have also

increased the nitrogen supply from the soil through the mineralization of the crop residues. Furthermore, climatic conditions in 2019 have favored nitrogen mineralization. The application of bovine slurry and solid manure (5-6th April) was carried out one month and a half before stored urine (17th May) and the organic fraction of bovine slurry and solid manure may have started to mineralize and be available to plants. The nitrogen use efficiencies were low: 18% for stored urine and 12% for bovine slurry + solid manure.

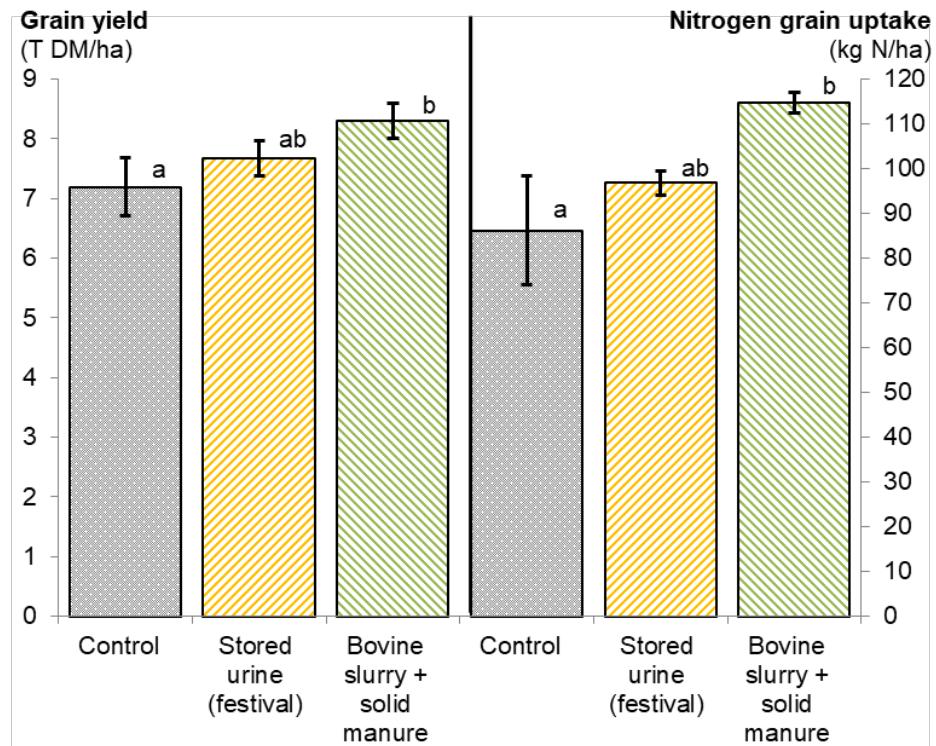


Figure 4-5. Grain yield and nitrogen grain uptake in the different treatments of the organic experiment with maize (experiment 5). Nitrogen input was 72 kg N/ha for stored urine and 256 kg N/ha for bovine slurry + solid manure. Statistically significant differences ($p\text{-value} \leq 0.05$) among treatment are represented by letters.

A renewable and efficient fertilizer such as urine-based fertilizers is of great interest in organic farming. Indeed, they represent a large amount of nutrients in densely populated areas. However, the use of urine-based fertilizers is not allowed in the European Union regulation for organic agriculture. Yields are lower in organic agriculture compared to conventional agriculture with about -40% for wheat and -15% for maize (Seufert et al. 2012). One of the main reasons is nitrogen deficiency (David et al. 2005). For some crops such as winter wheat, needs of nitrogen are early in the season and nitrogen mineralization from soil or organic fertilizers is too late (Berry et al. 2002) which reinforces the interest of urine-based fertilizers with a high proportion of rapidly available nitrogen. Crops such as maize grow later, but they need a lot of nitrogen in a short period of time, and the nitrogen supply

from soil mineralization is also often insufficient (Pang and Letey 2000). Finally, concentrated urine-based fertilizers may also reduce the time spent for fertilization compared to slurry or solid manure while supplying more nitrogen. However, in comparison to urine-based fertilizers, organic fertilizers such as manures also bring organic matter which provides other benefits to soil fertility (e.g., biological activity, limitation of soil sealing).

In regions without animal breeding, the current crop rotation in organic agriculture for field crops contains many legumes which cannot be all valued as human food (e.g., alfalfa). The use of urine-based fertilizers can reduce the dependency on legumes for the nitrogen supply and allow them to be less frequent in crops rotation although they are not only used for the nitrogen supply but also to reduce weed and pest pressure. The effects of urine-based fertilizers in organic agriculture should thus be better characterized in a multi-year experiment to look if these fertilizers would allow decreasing the dependency of organic farming to conventional organic fertilizers and/or to legumes.

3.5. Ammonia volatilization

Table 4-5. Nitrogen input and ammonia volatilization. Statistically significant differences (p-value ≤ 0.05) among treatment are represented by letters.

| Treatment | Nitrogen Input (kg N/ha) | | Volatilization (kg N/ha) | | % of NH ₄ -N applied | | % Total-N applied | |
|------------------------------------|-----------------------------|-------|-----------------------------|-----|---------------------------------|-----|-------------------|-----|
| | NH ₄ -N | N-Tot | Mean | std | Mean | std | Mean | std |
| Control | 0 | 0 | -1.9 | 0 | - | - | - | - |
| Ammonium | 72.5 | 129.9 | 1.4 | 0 | 2 ^a | 0 | 1 ^a | 0 |
| Stored urine (festival) | 125.5 | 145.0 | 43.7 | 4 | 35 ^b | 3 | 34 ^b | 3 |

Similar amounts of nitrogen were applied in the two fertilized treatments but 50% of nitrogen input was under ammoniacal form with ammonium and 87% for stored urine (Table 4-5). Urine was liquid and infiltrates into the soil while ammonium was solid. Ammonium granules dissolve depending on rain and surface moisture and this process took about a week in our experiment. Volatilization flux was close to zero and even negative for the control treatment indicating ammonia deposition from the neighboring plots. It was low for ammonium: less than 1% of total nitrogen, as previously observed for ammonium (e.g., 1.3% for temperate climate, soil pH < 7, EMEP/EEA 2019). However, ammonium is the nitrogen mineral fertilizer with the least volatilization. Mineral fertilizers volatilization could be higher (e.g., 8.2% for nitrogen solution and 13.1% for urea, for temperate climate, soil pH < 7, EMEP/EEA 2019). Volatilization for stored urine represented 34% of total nitrogen applied. About 60% of the volatilization occurred in 24h and 85% in 48h. The remaining 15% was distributed during the 24 following days. Ammonia volatilization depends on the ammoniacal nitrogen content and on the pH of the fertilizer and the soil. The soil pH was 6.7 and may

have lowered a bit the volatilization compared to more alkaline soil. The pH of the stored urine from festival (7.1) was lower than the pH usually observed for the stored urine (e.g., 9.1 in Udert et al. 2006). This value needs to be confirmed by further analyzes, however, it may be due to dilution with water during the collection (urinals cleaning and raining during the event). This may have moderated volatilization compared to usual stored urine.

The main factors favoring ammonia volatilization were the contact surface with air and the duration of the contact (Houot et al. 2014). The weather and application conditions were also favorable to volatilization: sunny (maximum solar radiation above 900 W/m²), high temperature (air temperature between 6.3 °C and 22.4 °C, mean value 15.4 °C), no rain during the days following the application, use of a spreader with fine droplets, no incorporation into the soil, and little development of vegetation cover. Thus, these favorable conditions at the application have probably maximized volatilization which was larger than in previous experiments with stored urine. Rodhe et al. (2004) measured in Sweden (spring or summer) a volatilization of ammonia from 2% to 10% of the total nitrogen for stored urine applied to open soil, using trailing hoses followed by harrowing 4 hours later. In other organic fertilizers, ammonia volatilization can vary from 0-100% of the ammoniacal nitrogen fraction for bovine slurry (40-60% of nitrogen is ammoniacal), 45-100% for bovine solid manure (5-40% of nitrogen is ammoniacal), >10-60% for sewage sludge (10-30% of nitrogen is ammoniacal) (Houot et al. 2014). Since most urine-based fertilizers are liquids they can seep into the soil which should reduce ammonia volatilization compared to more solid organic fertilizers if no soil incorporation is made.

A high ammonia volatilization rate of stored urine will have a negative impact on its mineral fertilizer equivalent. In view of the high mineral fertilizer equivalent observed in our other experiments (section 3.3), volatilization must have been in general limited due to better conditions of application than in that volatilization experiment. However, the high volatilization potential may explain a part of the mineral fertilizer equivalent below 100%. The ammonia volatilization of other urine-based fertilizer has still to be tested.

Finally, different techniques could be used to reduce ammonia volatilization of stored urine. For example, Rodhe et al. (2004) measured only from 0.3% to 1.1% volatilization of total nitrogen when stored urine was incorporated using trailing shoes and 0.2 to 0.4% when urine was applied on growing crops with trailing hoses or trailing shoes (the cover limits the wind at the soil surface, decreases the temperature and may absorb a part of the gaseous ammonia during growth). Houot et al. (2014) reported a reduction in volatilization for slurry, solid manure or sewage sludge of about 25 to 100% with injection, 20 to 100% with rapid incorporation in the soil and about 0 to 75% reduction using trailing hoses. However, these techniques are not always compatible with the technical itinerary, e.g., injection on crop already installed or trailing hoses on crop too high. Some treatment of urine such as acidification, as it is done for slurry, may also limit ammonia volatilization. Other urine treatments,

such as nitrification should also limit volatilization as a part of the nitrogen is under nitrate form and cannot volatilize. It is therefore crucial to take volatilization potential into account and to use adapted spreading methods or application under climatic conditions that limit volatilization (e.g., low wind, rain after the application, evening application).

4. Conclusion

We tested the mineral fertilizer equivalent of four different urine-based fertilizers for the first time together under field conditions and compared them to ammonium nitrate and other organic fertilizers. Despite high variability, we found that urine-based fertilizers have high nitrogen use efficiency and a mineral fertilizer equivalent above 70% in most cases. The urine-based fertilizers had higher mineral fertilizer equivalent than the other organic fertilizers. It was mainly related to the high mineral nitrogen content of urine-based fertilizers. In the conditions of the experiment, the differences among urine-based fertilizers were low. In terms of fertilization management, it seems possible to completely substitute mineral fertilizer by urine-based fertilizer without significant losses of yield or grain quality. However, adapted forms of fertilizers and application techniques must be used. Ammonia volatilization was high (34% of the nitrogen applied) compared to ammonium nitrate (1% of the nitrogen applied) when measured in favorable conditions for volatilization and could thus decrease the nitrogen fertilizing efficiency of urine-based fertilizers applied with volatilization favorable conditions.

Urine-based fertilizers seem relevant to use in agriculture. They could become an important source of renewable nutrients in farming in the future both in conventional and organic agriculture. However, it is necessary to repeat the experiments to acquire new references on different crops. Other environmental impacts and aspects such as soil salinization, N₂O emissions and input of pharmaceutical residues must be considered. The impacts of the upstream part of the value chains (e.g., energy for volume reduction) should also be assessed to allow a clear comparison among urine-based fertilizers. Finally, the acceptability of the use of urine-based fertilizers by consumers and farmers should be further investigated. In line with this last point, the experiments carried out in this study give better knowledge about urine-based fertilizers and contribute to addressing the questions and concerns of farmers about these fertilizers.

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Chapitre 5

Mesures de volatilisation en conditions contrôlées

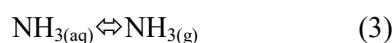
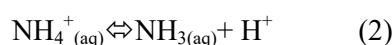
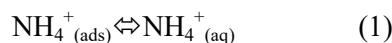
L'acidification et la nitrification de l'urine réduisent efficacement le potentiel de volatilisation ammoniacale des urinofertilisants

1. Introduction

L'ammoniac est un polluant atmosphérique qui provoque à la fois des impacts sur l'environnement (acidification, eutrophisation) et sur la santé, avec la formation de particules fines (Erisman et al. 2008). En France, l'agriculture est responsable de 94% des émissions d'ammoniac vers l'atmosphère (Citepa 2019). Une partie de ces émissions provient de l'élevage, à hauteur d'environ 40% en considérant les bâtiments d'élevage et le stockage des lisiers et fumiers. Cependant, 44% proviennent des apports de fertilisants au champ, en considérant les apports de fertilisants minéraux et les épandages de fumier et de lisier (Citepa 2019). La volatilisation ammoniacale représente également une perte d'efficacité fertilisante en raison des pertes d'azote (Xu et al. 2012). Elle peut être élevée pour certains engrains minéraux (p. ex., de 10% à 31% de l'azote apporté pour l'urée, Jones et al. 2007) ou certains engrains organiques (p. ex., de 36% à 61% de l'azote ammoniacal apporté pour un lisier, Bittman et al. 2005).

La volatilisation ammoniacale se produit généralement au moment de l'apport des fertilisants et dans les jours qui suivent ; c'est un processus physico-chimique qui se décompose en deux étapes :

(i) Passage de l'ammonium (NH_4^+) adsorbé à dissous (1), passage de l'ammonium dissous vers la forme ammoniac (NH_3) dissous (2) et enfin passage de l'ammoniac dissous à gazeux (3).



Ce processus est fonction de la teneur en azote ammoniacal après apport du fertilisant dans le sol et de la température. Il est favorisé par un pH élevé du complexe fertilisant/sol.

(ii) Diffusion de l'ammoniac sous forme gazeuse vers l'atmosphère. Ce processus dépend principalement des conditions micrométéorologiques à la surface du sol elles-mêmes fortement dépendantes des conditions météorologiques (p. ex., température, vent, humidité). C'est un phénomène d'échange de surface qui diminue fortement si les fertilisants sont enfouis.

L'urine peut être séparée des autres constituants des eaux usées par séparation à la source (Rossi et al. 2009). Une fois l'urine séparée, différents traitements peuvent être réalisés en particulier pour stabiliser l'azote dans une forme qui limite les pertes par volatilisation. En effet, l'azote est excréte sous forme d'urée mais l'urée s'hydrolyse rapidement en azote ammoniacal qui est susceptible de se

volatiliser en raison du pH élevé de l'urine (Udert et al. 2006). Ces traitements résultent en différents urinofertilisants aux caractéristiques variées. Ces urinofertilisants ont montré des efficacités fertilisantes élevées lors des essais agronomiques réalisés (chapitres 3 et 4). Cependant, lors des essais en conditions réelles, la plupart des urinofertilisants ont montré une équivalence avec l'engrais minéral inférieure à 100%. Une partie de cette moindre efficacité pourrait être expliquée par la volatilisation de ces produits au champ. La volatilisation ammoniacale a été mesurée pour l'urine stockée après épandage au champ en conditions favorables (p. ex. température assez élevée, voir chapitre 4) ; la perte mesurée était élevée avec 34% de l'azote apporté.

Les urinofertilisants ont des caractéristiques physico-chimiques variées qui pourraient favoriser plus ou moins la volatilisation de leur ammonium après apport au champ (p. ex., pH acide ou alcalin). La volatilisation peut représenter un impact environnemental important de l'utilisation des urinofertilisants qui n'a été que peu étudié. Les mesures de volatilisation en conditions réelles sont très dépendantes des conditions à l'épandage et nécessitent un lourd dispositif expérimental (p. ex., grandes parcelles, quantité de fertilisant importante, voir chapitre 4). Cependant, des dispositifs expérimentaux existent pour mesurer la volatilisation ammoniacale en conditions contrôlées. L'objectif de cette étude était de mesurer le potentiel de volatilisation ammoniacale de 9 urinofertilisants en conditions contrôlées afin de pouvoir les comparer entre eux. Ils ont aussi été comparés à un engrais minéral et à un engrais organique (lisier bovin).

2. Matériel et méthode

2.1. Fertilisants testés

Une grande partie des urinofertilisants étudiés correspondent à ceux testés dans l'expérimentation en serre (chapitre 2). Cependant, les mesures de volatilisation n'ont pas été réalisées en même temps, les échantillons utilisés n'étaient pas les mêmes et les procédures de préparation étaient légèrement différentes. Les caractéristiques des 11 fertilisants utilisés pour la volatilisation sont présentées dans le Tableau 5-1.

Deux séries de mesure ont été faites pour l'**urine stockée (université)**, avec de l'urine collectée en 2017 et 2019 dans un bâtiment de l'université à l'aide d'un urinoir masculin sec. Elles ont été stockées pendant au moins six mois dans un réservoir étanche. Pour l'**urine stockée acidifiée**, 17 ml d'acide sulfurique (pur à 96%) ont été ajoutés par litre d'urine stockée (urine collectée en 2019) pour abaisser le pH à 6,3 afin de réduire les pertes par volatilisation ($pK_a \text{ NH}_3/\text{NH}_4^+ = 9,2$). L'**urine stockée (festival)** a été collectée par une entreprise privée lors d'un festival de musique en août 2018 en utilisant des urinoirs secs. Elle a ensuite été stockée pendant environ 1 an dans une cuve étanche.

Pour l'**urine fraîche acidifiée** et l'**urine fraîche alcalinisée**, l'urine a été recueillie auprès d'une vingtaine de donneurs de l'université. Les mélanges ont été effectués dans les trois heures après collecte et les fertilisants produits ont été stockés à 4 °C entre une à cinq semaines avant le début de l'expérimentation. L'acidification de l'urine fraîche à un pH inférieur à 4 ou l'alcalinisation à un pH supérieur à 11 ont pour objectif d'empêcher l'hydrolyse de l'urée et donc de stabiliser l'azote sous forme uréique qui ne devrait pas se volatiliser avant apport au sol. Pour l'urine fraîche acidifiée, 60 mmol H⁺ ont été ajoutées par litre d'urine fraîche (1,61 mL/L d'acide sulfurique à 96%, Hellström et al. 1999), l'amenant à un pH de 2,1. Pour l'urine fraîche alcalinisée, 10 g de Ca(OH)₂ ont été ajoutés par litre d'urine fraîche (Randall et al. 2016), l'amenant à un pH de 12,3.

L'urine est parfois mélangée à des matières organiques, par exemple dans les toilettes sèches. Pour simuler cette utilisation, un **mélange d'urine fraîche et de copeaux de bois** a été produit une semaine avant le début de l'expérience. La même urine fraîche que dans le cas précédent a été utilisée et 1 kg de copeaux de bois (morceaux avec une taille inférieure à 1 cm) ont été mélangés à 286 g d'urine fraîche, quantité maximale que les copeaux de bois pouvaient absorber. Un autre mélange urine + matière organique a été utilisé pour les mesures de volatilisation. Ce mélange **d'urine stockée et de compost de déchets verts** n'a pas été étudié lors de l'expérimentation en serre présentée au chapitre 3, mais au cours d'une précédente expérimentation (Martin 2018). Avec ce produit, nous souhaitions étudier une possible gestion de l'urine en la mélangeant avec des matières organiques courantes. Le compost de déchets verts a été récupéré sur une plateforme de compostage. Afin de garder le mélange sous forme solide et d'obtenir un produit facilement applicable, ces deux matières ont été mélangées selon un rapport de 16,7 g d'urine stockée (collectée en 2017) pour 20 g de compost.

Le traitement de l'**urine fermentée** consistait en une acidification de l'urine fraîche suivie d'une fermentation lactique. Le traitement est similaire à celui de Andreev et al. (2017). Cet urinofertilisant a été produit par la société TOOPI Organics (www.toopi-organics.com).

Pour l'**urine nitrifiée**, la moitié de l'azote ammoniacal dans l'urine stockée a été nitrifiée (nitrification biologique). La solution a ensuite été concentrée par distillation (Fumasoli et al. 2016). Cet urinofertilisant a été produit par la société VUNA (www.vuna.ch).

Comme pour l'expérimentation en serre, les urinofertilisants ont été comparés au **nitrate d'ammonium** (sous forme liquide) et au lisier bovin. La solution de nitrate d'ammonium a été obtenue en mélangeant 63,7 g de NH₄⁺NO₃⁻ dans 100 ml d'eau.

Le **lisier bovin** a été collecté dans une pré-fosse à lisier sous la stabulation d'une ferme laitière conventionnelle en 2017 et dans la fosse à lisier d'une autre ferme laitière conventionnelle en 2019.

Tableau 5-1. Caractéristiques des fertilisants testés. Les résultats sont exprimés par rapport à la masse de produit frais non séché. MB est pour matière brute et DV pour déchets verts. Le pH du mélange urine fraîche + copeaux n'a pas pu être mesuré.

| Fertilisants | Matière sèche ou résidus sec (% ou g/100g MB) | pH | N-total (g N/kg) | N-NH ₄ (g N/kg) | N-NO ₃ (g N/kg) | N-uréique (g N/kg) | N-organique (g N/kg) |
|--|---|------|------------------|----------------------------|----------------------------|--------------------|----------------------|
| Urine stockée (université) 2017 | 2 | 9,1 | 6,6 | 5,5 | 0 | 0 | 1,2 |
| Urine stockée (université) 2019 | 1 | 9,1 | 6,6 | 6,6 | 0 | 0,3 | 0 |
| Urine stockée (festival) | 1 | 7,1 | 3,8 | 3,7 | 0 | 0,3 | 0 |
| Urine nitrifiée concentrée | -* | 3,9 | 58,1 | 29,6 | 28,5 | 0 | 0 |
| Urine fraîche acidifiée | 2 | 2,1 | 4,5 | 0,0 | 0 | 3,8 | 0,7 |
| Urine stockée acidifiée | 2 | 6,3 | 6,6 | 6,6 | 0 | 0,3 | 0 |
| Urine fermentée | 1 | 2,9 | 2,4 | 0,5 | 0 | 2,0 | 0 |
| Urine fraîche alcalinisée | 3 | 12,3 | 4,5 | 0,0 | 0 | 4,4 | 0,1 |
| Urine fraîche + copeaux de bois | 27 | - | 3,6 | 0,4 | 0 | 3,4 | 0 |
| Urine stockée (2017) + compost DV | 45 | 9,0 | 9,8 | 1,3 | 0 | <1 | 8,1 |
| Lisier bovin 2017 | 12 | 8,0 | 6,0 | 1,4 | 0 | <1 | 4,2 |
| Lisier bovin 2019 | 7 | 7,0 | 3,9 | 1,6 | 0 | 0,5 | 1,8 |
| Nitrate d'ammonium | - | 4,7 | 221,9 | 107,5 | 114,4 | 0 | 0 |

*Non mesurée en raison de la cristallisation.

Les différentes formes de l'azote présentes dans les urino fertisants sont présentées en Figure 5-1. L'azote est excréte dans l'urine principalement sous forme d'urée (Udert et al. 2006). Cependant, dans l'urine stockée et l'urine stockée acidifiée, la plus grande partie de l'urée est hydrolysée pendant le stockage, et l'azote ammoniacal est la principale forme d'azote. Pour les urines fraîches et l'urine fermentée, stabilisées par acidification ou alcalinisation, l'urée est la principale forme d'azote. Comme pour le nitrate d'ammonium, la forme de l'azote dans l'urine nitrifiée concentrée est pour moitié nitrique et pour moitié ammoniacale (Fumasoli et al. 2016). Le lisier bovin prélevé en 2017 étant «frais» (collecté dans une pré-fosse sous la stabulation). Seul 23% de l'azote est sous forme ammoniacale ce qui est dans la fourchette basse de la concentration attendue (Benoît et al. 2014). Environ 40% de l'azote est sous forme ammoniacale dans le lisier bovin prélevé en 2019. Le taux de matière sèche du lisier de 2017 est aussi plus élevé qu'en 2019.

Pour le mélange d'urine fraîche avec des copeaux de bois, la majeure partie de l'azote était sous forme uréique contrairement au mélange testé en serre, pour lequel la majorité de l'azote était sous forme organique. Les transformations de l'azote après le mélange avec les copeaux de bois nécessitent d'être étudiées plus en détail. La concentration en azote dans les copeaux de bois n'a pas été déterminée, mais, en supposant une teneur en azote de 0,06% comme cela a pu être mesuré dans la sciure de bois (Reichel et al., 2018), la concentration prévue du mélange serait de 4,2 g N/kg au lieu des 3,6 g N/kg mesurés. Cela suggère qu'environ 15% de l'azote urinaire aurait pu être hydrolysé puis volatilisé pendant le stockage avant le début de l'expérimentation. Pour le mélange de l'urine stockée avec du compost de déchets verts, la concentration en azote calculée à partir des teneurs en azote du compost et des urines était de 10,7 g N/kg au lieu des 9,7 g N/kg mesurés indiquant une possible volatilisation d'environ 10% de l'azote avant le début de l'expérimentation.

Le pH de l'urine stockée est alcalin (9,1) excepté pour l'urine provenant de festival pour laquelle il est de 7,1. Cela est peut être dû à une dilution avec de l'eau pendant la collecte (nettoyage des urinoirs et pluie pendant l'événement). L'urine alcalinisée a un pH très basique (12,3). Le pH du mélange avec le compost est aussi alcalin (9,0). Les pH de l'urine nitrifiée, de l'urine fermentée et des urines acidifiées sont acides à très acides (2,1 à 6,3).

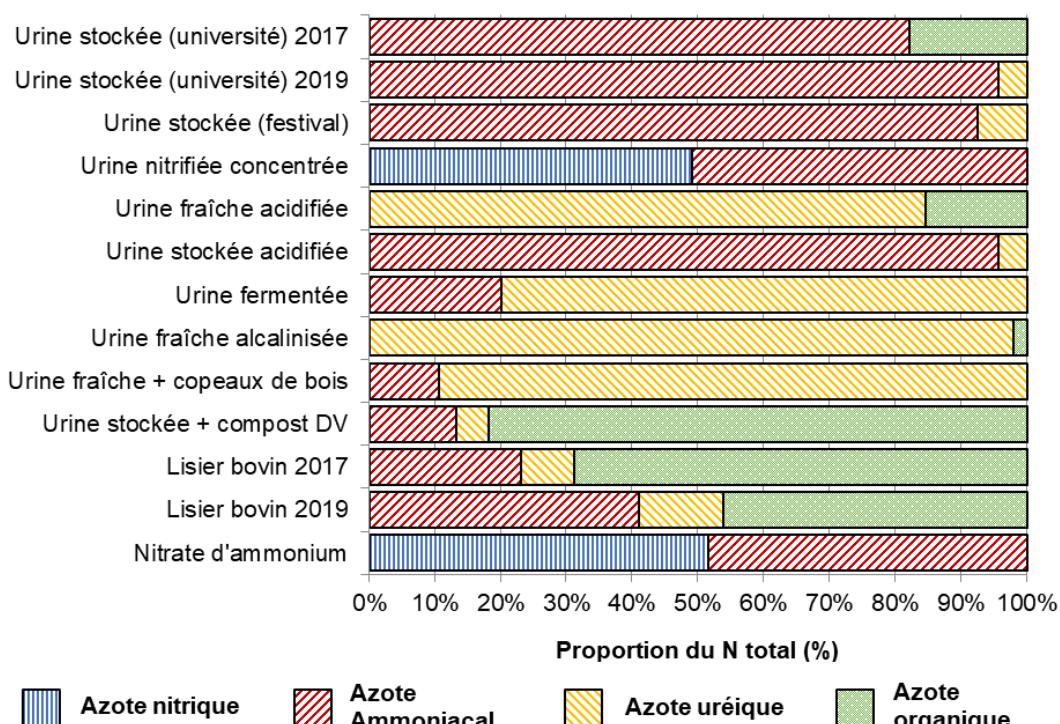


Figure 5-1. Répartition des formes de l'azote dans les urinofertilisants testés. DV est pour déchets verts.

2.1. Dispositif expérimental

Les potentiels de volatilisation se mesurent à l'aide de chambres dynamiques associées à un dispositif de régulation des conditions environnementales (Figure 5-2a., Génermont et al. 2013). Les chambres dynamiques ont une surface de 177 cm² (7,5 cm de rayon). Le sol est contenu dans un cylindre sur une hauteur d'environ 7 cm. Les fertilisants sont épandus en surface du sol (Figure 5-2b.). Une micropipette est utilisée pour apporter les fertilisants liquides. Les fertilisants solides sont apportés par pesée. Une fois les fertilisants apportés, les chambres sont fermées hermétiquement (Figure 5-2c). L'atmosphère des chambres est contrôlée. L'air qui entre dans la chambre est filtré pour ne pas apporter d'ammoniac et est humidifié à une humidité relative constante proche de 95%. Le flux d'air balayant la surface expérimentale est de 3,5 L par minute et la température est maintenue à 15°C. Une fois l'air passé par la chambre, le flux est dirigé vers un barboteur rempli avec une solution d'acide sulfurique. Ce système de piège acide permet de fixer l'ammoniac volatilisé. En effet, le pKa du couple NH₃/NH₄⁺ étant de 9,2, l'ammoniac gazeux (NH₃) est dissous sous forme d'ion ammonium (NH₄⁺) dans la solution d'acide sulfurique après passage par le barboteur. Régulièrement, les barboteurs sont changés et la concentration en azote ammoniacal dans la solution d'acide sulfurique est mesurée par colorimétrie (méthode Berthelot). Puis, les flux de volatilisation sont recalculés à partir de la concentration en ammonium de la solution d'acide sulfurique et du temps de prélèvement. Les prélèvements successifs permettent d'observer la dynamique de volatilisation. Une fois le plateau de volatilisation atteint, l'expérimentation est arrêtée (généralement au bout d'une vingtaine de jours). Pour chaque fertilisant, trois répétitions (trois chambres) ont été mises en place.

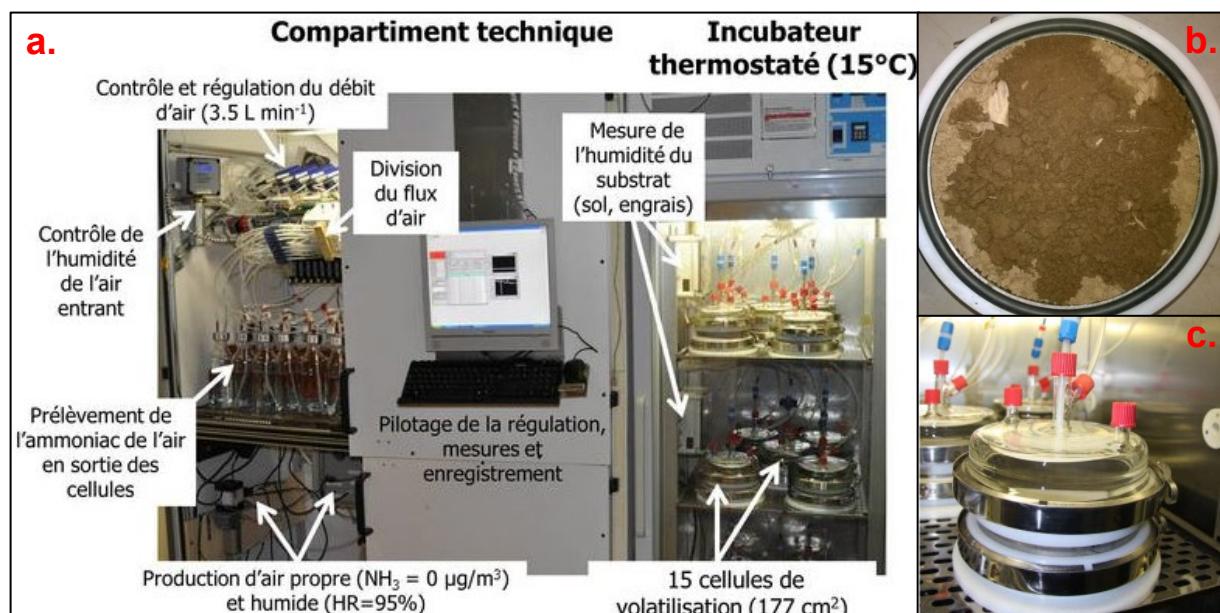


Figure 5-2a. Dispositif expérimental utilisé (INRAE 2020). 5-2b. Cyclindre de sol avec apport d'urine. 5-2c. Chambre fermée pour mesure de flux (INRAE 2020).

2.2. Expérimentations

Au total, 2 séries d'expérimentations différentes ont été menées. Les différentes expérimentations et les principales caractéristiques des sols sont présentées dans le Tableau 5-2.

Tableau 5-2. Les différentes expérimentations menées et les caractéristiques principales de sols utilisés.

| Expérimentation | Date | Fertilisants testés | Traitement du sol | pH (H ₂ O) | Matière organique (% ou g/100 g) | Capacité d'échange cationique (mEq/100 g) |
|-----------------|--|--|-------------------------------------|-----------------------|----------------------------------|---|
| 1a | 04/07/2017 au 24/07/2017 (20 jours) | - Urine stockée (université) - Urine nitrifiée concentrée - Urine stockée + compost de déchets verts - Nitrate d'ammonium | Tamisé | 7,3 | 2,5 | 9,9 |
| 1b | 19/06/2018 au 05/07/2018 (15 jours) | - Urine nitrifiée concentrée - Nitrate d'ammonium - Urine nitrifiée concentrée - Nitrate d'ammonium | | 8,2 | 1,75 | 7,8 |
| 2a | 10/09/2019 au 30/09/2019 (20 jours) | - Urine stockée (université) - Urine stockée (festival) - Urine fermentée - Lisier bovin | Prélevé au champ dans des cylindres | 6,7 | 2,2 | 11,9 |
| 2b | 08/10/2019 au 07/11/2019 (30 jours) | - Urine fraîche acidifiée - Urine stockée acidifiée - Urine fraîche alcalinisée - Urine fraîche + copeaux de bois - Lisier bovin | | | | |

La première expérimentation visait à quantifier la volatilisation ammoniacale de différents urinofertilisants testés dans un précédent essai en serre (Martin 2018). L'urine stockée (université, 2017), l'urine nitrifiée concentrée et le mélange urine stockée + compost de déchets verts ont été testés dans l'expérimentation 1a. Le sol utilisé pour cette expérimentation a été prélevé dans l'horizon de surface d'une parcelle témoin d'un essai agronomique. C'est un sol limoneux ayant un pH de 7,3. Il a été tamisé à 4 mm puis stocké à 4 °C, trois mois avant l'expérimentation. Les cylindres ont été remplis de 7 cm de sol tamisé à une densité de 1,25 kg/L (1,85 kg/sol par cylindre). Le sol a été amené à une humidité pondérale de 25% avant apport des produits.

L'urine nitrifiée concentrée ayant eu une volatilisation très faible au cours de cette expérimentation (Expérimentation 1a), une seconde expérimentation a été réalisée comparant la volatilisation de cette urine nitrifiée dans 2 types de sol (Expérimentation 1b) : (i) avec le sol de l'expérimentation 1a (mais alors stocké 15 mois), (ii) un sol ayant un pH plus alcalin, favorisant la volatilisation (pH 8,2). Le

nitrate d'ammonium a fait office de produit en commun et a été testé dans les différentes configurations afin de s'assurer de la reproductibilité des expérimentations.

La dose d'azote a été fixée à 100 kg N/ha pour les différents fertilisants de l'expérimentation 1a. La dose pour le mélange urine stockée + compost de déchets verts a été calculée en ne prenant en compte que l'azote provenant de l'urine (azote organique très faiblement minéralisable à court terme dans le compost). L'urine nitrifiée a été apportée à une dose de 80 kg N/ha et le nitrate d'ammonium à une dose de 120 kg N/ha dans l'expérimentation 1b. Les concentrations réelles dans les urinofertilisants ayant été mesurées après le début de l'expérimentation, les doses exactes peuvent varier autour de la dose cible et sont présentées dans le Tableau 5-3.

La seconde série d'expérimentations (Expérimentation 2a et 2b) visait à caractériser le potentiel de volatilisation des produits testés lors de l'essai en serre (chapitre 3). Les expérimentations ont été menées en 2019 sur des cylindres de sol de 7 cm de hauteur. Ces cylindres ont été directement prélevés au champ dans l'horizon de surface de la même parcelle où a été réalisé l'essai de volatilisation en conditions réelles (chapitre 4). L'objectif était de perturber le moins possible l'horizon de surface. Ces cylindres de sol ont été stockés à 4°C pendant 1 à 5 semaines avant l'expérimentation. Le sol prélevé est un sol limoneux ayant un pH de 6,7. Il n'a pas été humidifié avant apport et avait une humidité pondérale de 7,7%. Afin de tester les 8 produits (7 urinofertilisants et un lisier bovin), l'expérimentation a été décomposée en deux séries (expérimentations 2a et 2b). Le lisier bovin a servi de produit en commun entre les deux expérimentations afin de s'assurer de la reproductibilité des expérimentations. La dose d'azote visée pour les différents fertilisants a été fixée à la même dose que pour les essais de volatilisation menés aux champs : 145 kg N/ha. En raison d'un volume trop important du mélange urine + copeaux de bois pour rentrer dans la chambre, la dose d'azote pour cet urinofertilisant a été diminuée à environ 60 kg N/ha. Les concentrations réelles ayant été mesurées après le début de l'expérimentation, les doses exactes peuvent varier autour de la dose cible et sont présentées dans le Tableau 5-3.

2.3. Statistiques

Les résultats sont exprimés en utilisant la moyenne des trois répétitions avec l'écart-type. Les différences significatives entre les traitements pour la volatilisation mesurée en fin d'expérimentation ont été étudiées au sein d'une même série d'expérimentation (1a et 1b puis 2a et 2b ensemble). En effet, les sols sont différents entre les deux expérimentations et il n'y a pas de fertilisant en commun permettant de s'assurer de la reproductibilité des résultats. Un test ANOVA suivi d'un test post hoc Tukey HSD ($p\text{-value} \leq 0,05$) ont été réalisés. Tous les tests statistiques ont été effectués à l'aide du logiciel R, version 3.3.2.

3. Résultats et discussion

Les apports de fertilisants et la volatilisation mesurée pour les différents traitements sont présentés dans le Tableau 5-3. La volatilisation pour les différentes expérimentations en pourcentage de l'azote total est représentée sur la Figure 5-3.

La variabilité entre les répétitions est assez élevée pour certains traitements, mais des différences significatives entre les traitements ont pu être calculées. Il est difficile de comparer directement les différentes expérimentations, celles-ci ne s'étant pas déroulées sur le même sol. Par exemple, la capacité d'échange cationique plus élevée, le pH plus faible et la plus faible humidité du sol des expérimentations 2a et 2b ont pu limiter la volatilisation en comparaison des expérimentations 1a et 1b (Sommer and Jacobsen 1999; Fuchs et al. 2014).

En raison de la grande diversité de teneurs en azote, les doses apportées recalculées par hectare varient de 0,4 t/ha (nitrate d'ammonium) à 31,8 t/ha (mélange urine stockée + compost de déchets verts). Les doses d'apport des fertilisants sont proches de la dose cible pour l'expérimentation 1a (100 kg N/ha), entre 89 et 96,6 kg N/ha. La dose totale pour le mélange urine stockée + compost de déchets verts est de 310,9 kg N/ha mais la dose d'azote minéral est proche de la dose cible. Les doses d'apport des fertilisants varient aussi autour de la dose cible dans l'expérimentation 1b (dose cible de 80 kg N/ha pour l'urine nitrifiée concentrée et de 120 kg N/ha pour le nitrate d'ammonium). Les doses apportées en N-NH₄ pour l'expérimentation 1a et 1b vont de 21,4 kg N-NH₄/ha (lisier bovin) à 79,2 kg N/ha (urine stockée université). La volatilisation en pourcentage du N-NH₄ apporté varie de 0% (urine nitrifiée concentrée et nitrate d'ammonium) à 72% (lisier bovin). En pourcentage de l'azote total apporté, la volatilisation varie de 0% (urine nitrifiée concentrée et nitrate d'ammonium) à 17% (lisier bovin).

Pour les expérimentations 2a et 2b, en raison du caractère dilué de la plupart des fertilisants, les doses réellement apportées par hectare sont comprises entre 17 t/ha pour l'urine fraîche + copeaux de bois, et 48 t/ha pour l'urine fermentée. À l'exception du mélange entre l'urine fraîche et les copeaux de bois, les doses d'azote apportées par les fertilisants sont proches de la dose cible (145 kg N/ha) pour les expérimentations 2a et 2b. Les apports en azote ammoniacal vont de 0 kg N-NH₄/ha pour les produits stabilisés sous forme d'urée (urine fraîche acidifiée et alcalinisée) à 139 kg N-NH₄/ha (urine stockée acidifiée). La volatilisation en pourcentage du N-NH₄ apporté est nulle pour les produits stabilisés sous forme d'urée (urine fraîche acidifiée et alcalinisée), en raison de l'absence d'azote ammoniacal dans ces produits mais elle monte jusqu'à 40% pour le mélange urine fraîche + copeaux de bois. En pourcentage de l'azote total apporté, la volatilisation varie de 2% (urine fraîche acidifiée) à 18% (urine fraîche alcalinisée).

Tableau 5-3. Apport des fertilisants en tonne de matière brute (MB), kg N/ha et kg N-NH₄/ha et volatilisation mesurée en fin d'expérimentation en kg N-NH₄/ha, % du N-NH₄ apporté et % du N (total) apporté. La moyenne des 3 répétitions et l'écart-type sont présentés. DV est pour déchets verts.

| Exp | Fertilisant | Apports | | | | | | Volatilisation | | | | | |
|-----|--|-----------------|------------|-------------|------------|-----------------------------|------------|-----------------------------|------------|-------------------------------|------------|---------------|------------|
| | | (t MB brut /ha) | | (kg N / ha) | | (kg N-NH ₄ / ha) | | (kg N-NH ₄ / ha) | | (% N-NH ₄ apporté) | | (% N apporté) | |
| | | Moyenne | Écart-type | Moyenne | Écart-type | Moyenne | Écart-type | Moyenne | Écart-type | Moyenne | Écart-type | Moyenne | Écart-type |
| 1a | Urine stockée (université) | 14,5 | 0,1 | 96 | 0 | 79 | 0 | 16 | 5,4 | 20% | 7% | 16% | 6% |
| | Urine nitrifiée concentrée | 1,5 | 0,0 | 89 | 0 | 45 | 0 | 0,1 | 0,0 | 0% | 0% | 0% | 0% |
| | Urine stockée + compost DV | 31,8 | 0,2 | 311 | 2 | 43 | 0 | 32,5 | 11,2 | 76% | 26% | 10% | 4% |
| | Lisier bovin | 15,7 | 1,0 | 93 | 6 | 21 | 1 | 15,4 | 0,8 | 72% | 8% | 17% | 2% |
| | Nitrate d'ammonium | 0,4 | 0,0 | 97 | 0 | 47 | 0 | 0,1 | 0,0 | 0% | 0% | 0% | 0% |
| 1b | Urine nitrifiée concentrée | 1,4 | 0,1 | 84 | 6 | 43 | 3 | 0,0 | 0,0 | 0% | 0% | 0% | 0% |
| | Nitrate d'ammonium | 0,5 | 0,0 | 121 | 3 | 59 | 1 | 0,0 | 0,0 | 0% | 0% | 0% | 0% |
| | Urine nitrifiée concentrée (sol alcalin) | 1,4 | 0,0 | 80 | 2 | 41 | 1 | 1,9 | 0,5 | 5% | 1% | 2% | 1% |
| | Nitrate d'ammonium (sol alcalin) | 0,6 | 0,0 | 123 | 7 | 60 | 3 | 5,6 | 0,8 | 9% | 1% | 5% | 0% |
| | Urine stockée (université) | 21,0 | 0,1 | 137 | 1 | 138 | 1 | 23,8 | 9,3 | 17% | 7% | 17% | 7% |
| 2a | Urine stockée (festival) | 36,0 | 3,7 | 138 | 14 | 133 | 14 | 19,4 | 6,0 | 15% | 6% | 14% | 6% |
| | Urine fermentée | 48,4 | 0,3 | 116 | 1 | 22 | 0 | 3,1 | 0,5 | 14% | 2% | 3% | 0% |
| | Lisier bovin | 38,8 | 3,9 | 151 | 15 | 62 | 6 | 11,7 | 4,1 | 19% | 7% | 8% | 3% |
| | Urine fraîche acidifiée | 26,2 | 0,1 | 119 | 1 | 0 | 0 | 2,2 | 1,6 | 0% | 0% | 2% | 1% |
| 2b | Urine stockée acidifiée | 21,1 | 0,1 | 138 | 1 | 139 | 1 | 4,2 | 2,9 | 3% | 2% | 3% | 2% |
| | Urine fraîche alcalinisée | 26,5 | 0,2 | 119 | 1 | 0 | 0 | 21,2 | 6,7 | 0% | 0% | 18% | 6% |
| | Urine fraîche + copeaux | 17,1 | 0,5 | 62 | 2 | 7 | 0 | 2,9 | 0,9 | 40% | 12% | 5% | 1% |
| | Lisier bovin | 36,3 | 0,2 | 141 | 1 | 58 | 0 | 15,7 | 1,0 | 27% | 2% | 11% | 1% |

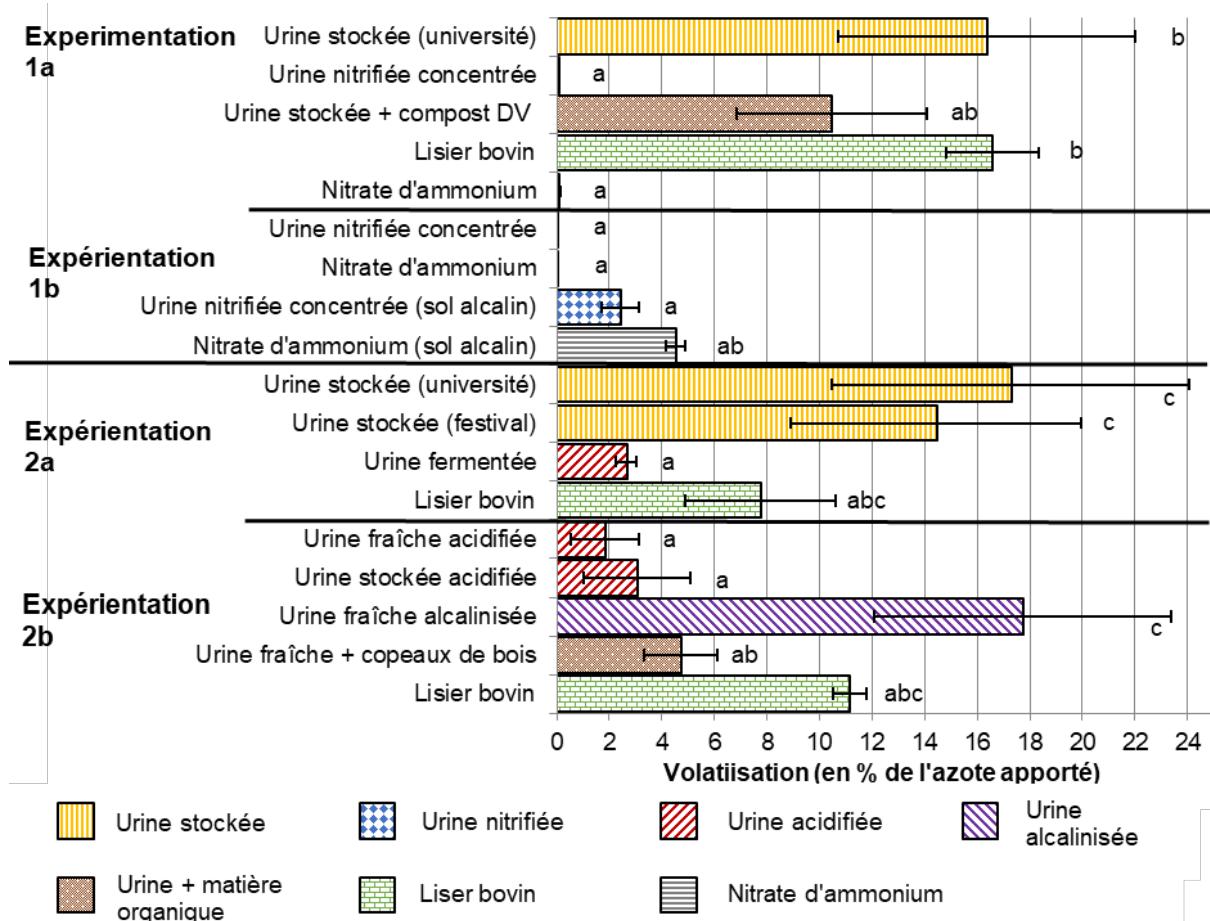


Figure 5-3. Volatilisation mesurée en fin d'expérimentation en pourcentage de l'azote apporté.

Les tests statistiques ont été réalisés au sein d'une même expérience (1a, 1b ensemble et 2a, 2b ensemble). Ces différences significatives ne doivent pas être comparées entre les expérimentations 1 et 2.

Nous pouvons observer qu'il n'y a pas de différence significative pour le nitrate d'ammonium entre les expérimentations 1a et 1b et pour le lisier bovin entre les expérimentations 2a et 2b, indiquant la reproductibilité des résultats entre les différentes expérimentations. Cependant, les différences sont plus grandes pour le lisier en ne considérant que l'azote ammoniacal (19% et 27%).

La première hypothèse permettant d'expliquer la volatilisation ammoniacale est le pH des fertilisants (Génermont and Cellier 1997). Cela est bien visible sur la Figure 5-4,

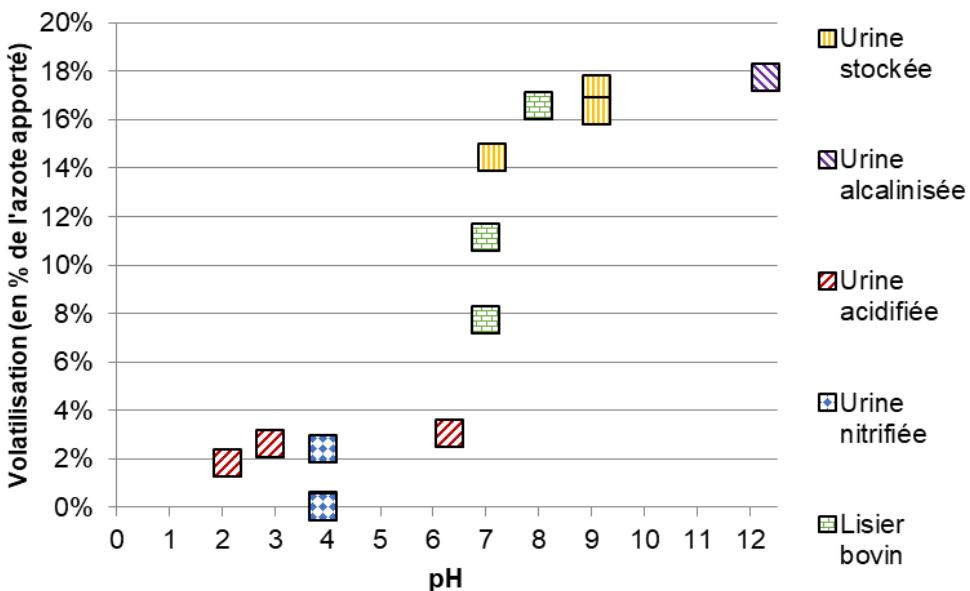


Figure 5-4. Volatilisation ammoniacale (en % de l'azote apporté) en fonction du pH pour les urinofertilisants et le lisier bovin. En raison de leur fort taux de matière organique et de leur forte matière sèche, les mélanges avec les matières organiques n'ont pas été considérés.

En effet, les fertilisants ayant un pH élevé (urine stockée, urine alcalinisée) ont une volatilisation supérieure à 15% (Figure 5-3). Ces fertilisants ont une volatilisation significativement plus élevée en comparaison des autres fertilisants testés à l'exception du lisier bovin et du mélange urine stockée + compost de déchets verts. Cela concerne à la fois les fertilisants ayant une forte teneur en azote ammoniacal (urine stockée), mais aussi les fertilisants ayant une forte teneur en azote uréique. Une fois apporté au sol, cet azote uréique s'hydrolyse rapidement en azote ammoniacal. L'hydrolyse de l'urée entraîne un augmentation du pH du sol et favorise la volatilisation (Overrein and Moe 1967). La dynamique de volatilisation au cours du temps pour l'urine stockée (azote ammoniacal majoritaire) et l'urine fraîche alcalinisée (azote uréique majoritaire) est présentée dans la Figure 5-5. Nous pouvons observer pour l'urine stockée une forte volatilisation dès le premier jour. Cependant, ce flux de volatilisation reste faible pour l'urine fraîche alcalinisée le premier jour puis augmente fortement à partir du deuxième jour pour rejoindre le même plateau que l'urine stockée au bout de 6 à 8 jours. Ce décalage dans la dynamique de volatilisation de l'urine stockée et de l'urine fraîche alcalinisée pourrait être expliqué par le temps nécessaire à l'hydrolyse de l'urée (Overrein and Moe 1967). Une dynamique de volatilisation suivant une courbe sigmoïde est généralement observée pour la volatilisation de l'urée (Ernst and Massey 1960, Zheng et al. 2018). Pour les fertilisants comportant de l'azote majoritairement sous forme ammoniacale, cette courbe est plutôt logarithmique. Nous n'avons pas observé de réduction de la volatilisation de l'urine fraîche alcalinisée par rapport à l'urine stockée malgré l'infiltration dans le sol du fertilisant avant hydrolyse de l'urée.

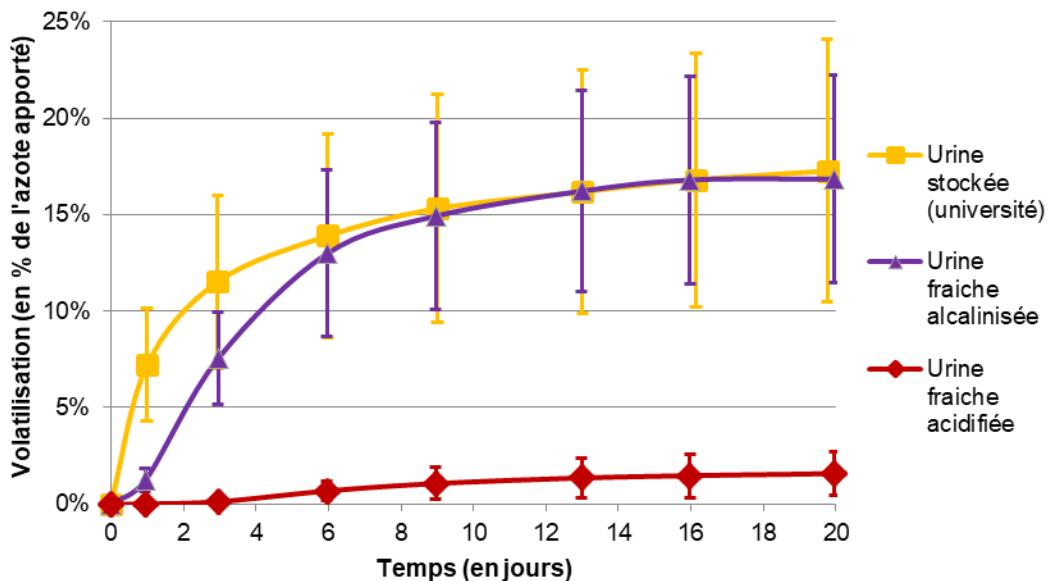


Figure 5-5. Dynamique de la volatilisation pour l'urine stockée, l'urine fraîche alcalinisée et l'urine fraîche acidifiée pendant les 20 premiers jours de l'expérimentation.

Au contraire, nous pouvons observer une faible volatilisation pour les produits acides (urine fraîche acidifiée, urine stockée acidifiée et urine fermentée), peu importe la forme de l'azote majoritaire dans l'urinofertilisant (ammoniacale ou uréique). Cette volatilisation a été mesurée autour de 2 à 3% de l'azote total apporté pour ces urinofertilisants acides et est significativement plus faible que les urinofertilisants alcalins. L'acidification de l'urine stockée a permis de diminuer de 80% la volatilisation (17% pour l'urine stockée contre 3% pour l'urine stockée acidifiée). La stabilisation de l'azote sous forme uréique par acidification permet de fortement limiter la volatilisation au contraire de la stabilisation sous forme uréique par alcalinisation. Comme observé pour les lisiers animaux, l'ajout d'acide pourrait diminuer l'alcalinité résultant de l'hydrolyse de l'urée, limiter le pH du complexe sol/fertilisant et ainsi limiter la volatilisation (Stevens et al. 1992). La faible dose d'acide utilisé pour stabiliser l'urine fraîche (1,6 mL/L urine, pH 2,1) semble aussi efficace pour limiter la volatilisation que l'acidification de l'urine stockée qui nécessite plus d'acide (17,1 mL/L urine pour atteindre un pH de 6,3). Une fois apportée au sol, l'hydrolyse de l'urée à la fois pour l'urine acidifiée et alcalinisée est supposée quasiment complète en raison de la forte efficacité de ces fertilisants mesurés lors de l'essai en serre (chapitre 3). Nous pouvons observer sur la Figure 5-5 que la volatilisation de l'urine fraîche acidifiée est nulle les 3 premiers jours de l'expérimentation et augmente ensuite très faiblement. Cela pourrait être expliqué par le temps nécessaire à l'hydrolyse de l'urée (Overrein and Moe 1967). Il pourrait être intéressant de mesurer le pH du sol après apport des fertilisants afin d'étudier l'impact des urinofertilisants sur celui-ci.

La nitrification de 50% de l'azote ammoniacal de l'urine (urine nitrifiée concentrée) permet de limiter quasiment totalement la volatilisation. Cela est à la fois dû aux 50% d'azote nitrique qui ne peuvent pas se volatiliser et à l'effet acidifiant du traitement (pH 3,9 dans le produit final). Comme

pour le nitrate d'ammonium, la volatilisation est un peu plus élevée sur sol alcalin (expérimentation 1b) mais elle reste faible et en dessous de 5% de l'azote apporté.

Pour le mélange entre l'urine stockée et le compost, la volatilisation en considérant l'azote total du mélange reste modérée en raison de la grande fraction en azote organique apportée par le compost. Cette fraction est peu susceptible de se minéraliser et de se volatiliser au cours des quelques semaines de l'expérimentation. En ne considérant que l'azote ammoniacal qui provient principalement de l'urine stockée, la volatilisation atteint 76% contre 20% pour l'urine stockée seule (expérimentation 1a). Cette augmentation de la volatilisation dans le mélange pourrait être expliquée par le fait que l'urine est retenue en surface dans le mélange au contraire de l'urine stockée liquide qui peut s'infiltrer dans le sol. En effet, un des facteurs les plus importants pour expliquer la volatilisation est le contact entre l'air et le fertilisant (Sommer et al. 2006; Fuchs et al. 2014). Pour le mélange urine + copeaux de bois, la volatilisation de l'azote total apporté est restée faible et a été mesurée autour de 5%. Pour l'échantillon utilisé lors de l'expérimentation serre (chapitre 3), la majorité de l'azote provenant de l'urine avait été organisée. Cette organisation de l'azote n'a pas été observée lors de l'analyse du produit au début de l'expérimentation qui montre une faible concentration en azote organique. Des analyses supplémentaires sont nécessaires pour mieux caractériser ce mélange. Cependant, il est possible que l'organisation de l'azote ait pu se dérouler au cours de l'expérimentation sans pertes majeures par volatilisation. Cette hypothèse pourrait être confirmée par des analyses du mélange au cours du temps. Environ 40% de l'azote ammoniacal du mélange a été volatilisé, ce qui est plus élevé que les urinofertilisants liquides (p. ex., 17% pour l'urine stockée de l'université dans l'expérimentation 2a). Cette forte volatilisation de l'azote ammoniacal pourrait aussi être expliquée par le fait que l'urine a été retenue en surface dans le mélange.

Les différents lisiers bovins ont montré une volatilisation allant de 8% à 17% de l'azote apporté (19% à 72% de l'azote ammoniacal). Les valeurs relevées dans la littérature indiquent que les lisiers ont une gamme de volatilisation très variée en fonction des caractéristiques du lisier, de celles du sol, des méthodes d'épandage ou des conditions météorologiques (de 1% à 100% de l'azote ammoniacal volatilisé, Fuchs et al. 2014). Le facteur d'émission rapporté par l'EMEP/EEA (2009) est de 55% de l'azote ammoniacal. Nous pouvons cependant remarquer que le lisier utilisé en 2017 (expérimentation 1a) a montré une plus forte volatilisation que le lisier utilisé en 2019 (17% contre 8% et 11% du N total). Cela pourrait être dû au plus grand taux de matière sèche dans le lisier de 2017 (12,0% contre 6,6% pour le lisier de 2019). L'infiltration plus lente pour ce lisier moins liquide fait que le lisier reste plus longtemps en surface ce qui permet une plus forte volatilisation malgré une plus faible teneur en azote ammoniacal (Garcia et al. 2012). Le pH plus élevé du lisier de 2017 (8 contre 7 pour le lisier de 2019) a aussi pu favoriser la volatilisation. Environ 76% de l'azote ammoniacal a été volatilisé dans le lisier de 2017 de l'expérimentation 1a contre seulement 19% et 27% pour le lisier de 2019 des expérimentations 2a et 2b. La volatilisation du lisier de l'expérimentation 1a est similaire à celle de

l'urine en considérant l'azote total (16% contre 17%). Cependant, seulement 20% de l'azote ammoniacal a été volatilisé dans l'urine stockée contre 76% pour le lisier, probablement en raison de l'infiltration de l'urine dans le sol.

La volatilisation ammoniacale a été peu étudiée pour les urinofertilisants. Pour de l'urine stockée apportée en utilisant un pendillard suivi d'une incorporation dans le sol 4 heures plus tard, Rodhe et al. (2004) ont mesuré une volatilisation de l'azote apporté variant de 2% à 10%. Cette méthode d'apport permet déjà de réduire la volatilisation ammoniacale, qui est plus faible que les valeurs que nous avons mesurées en chambre. En conditions réelles (chapitre 4), la volatilisation de l'urine stockée (festival) a été mesurée à 34% de l'azote total apporté, soit environ le double de celle mesurée en chambre. Cependant, pour l'expérimentation en conditions réelles, elle a été favorisée par les conditions météorologiques (température élevée). Il reste difficile de comparer les valeurs obtenues au champ ou en chambre aux valeurs de la littérature, car la volatilisation est très différente en fonction des conditions pédoclimatiques. À notre connaissance, la volatilisation ammoniacale des autres urinofertilisants n'a pas été étudiée. Cette expérimentation en conditions contrôlées a permis de hiérarchiser le potentiel de volatilisation des urinofertilisants entre eux et en comparaison d'un lisier bovin et d'un engrais minéral. Les urinofertilisants alcalins ont montré des potentiels de volatilisation plus élevés que les urinofertilisants acides malgré une stabilisation de l'azote sous forme uréique. La volatilisation de l'urine nitrifiée était très faible et similaire à celle de du nitrate d'ammonium (engrais minéral). Cependant, les hypothèses expliquant les différences de volatilisation entre les produits doivent être consolidées. Sur la base de ces résultats en conditions contrôlées, il pourrait être intéressant d'utiliser certains modèles comme le modèle Volt'Air (Garcia et al. 2011) afin de modéliser les émissions de ces fertilisants en fonction de différentes conditions pédoclimatiques au champ et de tester les hypothèses impliquées dans les différences entre les urinofertilisants.

Il existe de nombreuses méthodes d'apport pouvant être utilisées pour réduire la volatilisation (Huijsmans 2003; Webb et al. 2010; Carozzi et al. 2013). Par exemple, Rodhe et al. (2004) n'ont mesuré que 0,3% à 1,1% de volatilisation de l'azote apporté lorsque l'urine stockée était incorporée dans le sol à l'apport à l'aide d'un injecteur. Fuchs et al. (2014) ont rapporté une réduction de la volatilisation en comparaison d'un épandage par buse palette pour le lisier, le fumier solide ou les boues d'épuration d'environ 25 à 100% avec l'injection, de 20 à 100% avec une incorporation rapide dans le sol et d'environ 0 à 75% avec l'utilisation d'un pendillard. Toutefois, ces techniques ne sont pas toujours compatibles avec les itinéraires techniques (p. ex., injection sur des cultures déjà en place). De plus, les méthodes d'apport pour limiter la volatilisation ammoniacale pourraient favoriser d'autres émissions de gaz à effet de serre (p. ex. CH₄, N₂O, Emmerling et al. 2020). Il est donc important de prendre en compte le potentiel de volatilisation des urinofertilisants et d'utiliser des méthodes d'épandage adaptées.

4. Conclusion

Le potentiel de volatilisation ammoniacale de 9 urinofertilisants a été mesuré en conditions contrôlées et a été comparé à celui d'un engrais minéral (nitrate d'ammonium) et d'un lisier bovin. Les urinofertilisants ont montré une volatilisation variable en fonction de leurs caractéristiques physico-chimiques. Les produits alcalins (urine stockée et urine fraîche alcalinisée) ont montré des potentiels de volatilisation plus élevés que les autres urinofertilisants (de 14% à 18% de l'azote apporté). Cette volatilisation est similaire à celle des lisiers bovins étudiés (8% à 17% de l'azote apporté, pH similaire 7 à 8). Les produits acides (urines acidifiées, urine fermentée) au contraire ont montré un faible potentiel de volatilisation, inférieur à 5% de l'azote apporté. La nitrification de l'urine limite très fortement sa volatilisation qui reste faible même sur sol alcalin (moins de 1% à 5% de l'azote apporté). La volatilisation de l'urine nitrifiée concentrée était plus faible en comparaison des autres urinofertilisants et similaire à celle du nitrate d'ammonium. Le mélange entre l'urine stockée et le compost a favorisé la volatilisation de l'azote de l'urine en le retenant à la surface. Le mélange entre l'urine fraîche et les copeaux de bois a montré une faible volatilisation de l'azote total du mélange qui pourrait être due à une organisation de l'azote de l'urine. Cependant, ces hypothèses restent à étudier. L'acidification ou la nitrification apparaissent donc comme des méthodes de stabilisation efficace pour limiter les pertes par volatilisation. L'alcalinisation permet de stabiliser l'azote sous forme uréique, cependant elle ne semble pas limiter la volatilisation une fois le produit apporté au sol. Il serait intéressant de mesurer le potentiel de volatilisation de l'urine alcalinisée déshydratée qui, en raison de sa forme solide et de son pH élevé, pourrait avoir un fort potentiel de volatilisation.

Ces mesures en conditions contrôlées ont permis de hiérarchiser les produits entre eux. Il pourrait être intéressant de tester ces produits en conditions réelles afin de confirmer les résultats observés ou de simuler cette volatilisation à l'aide de modèles dans différentes conditions agropédoclimatiques. La volatilisation peut être limitée par différentes méthodes d'apport. Il est nécessaire de tenir compte du potentiel de volatilisation des différents urinofertilisants et d'utiliser des méthodes d'épandage adaptées ou un apport dans des conditions climatiques qui limitent la volatilisation (p. ex., vent faible, pluie après apport, apport en soirée).

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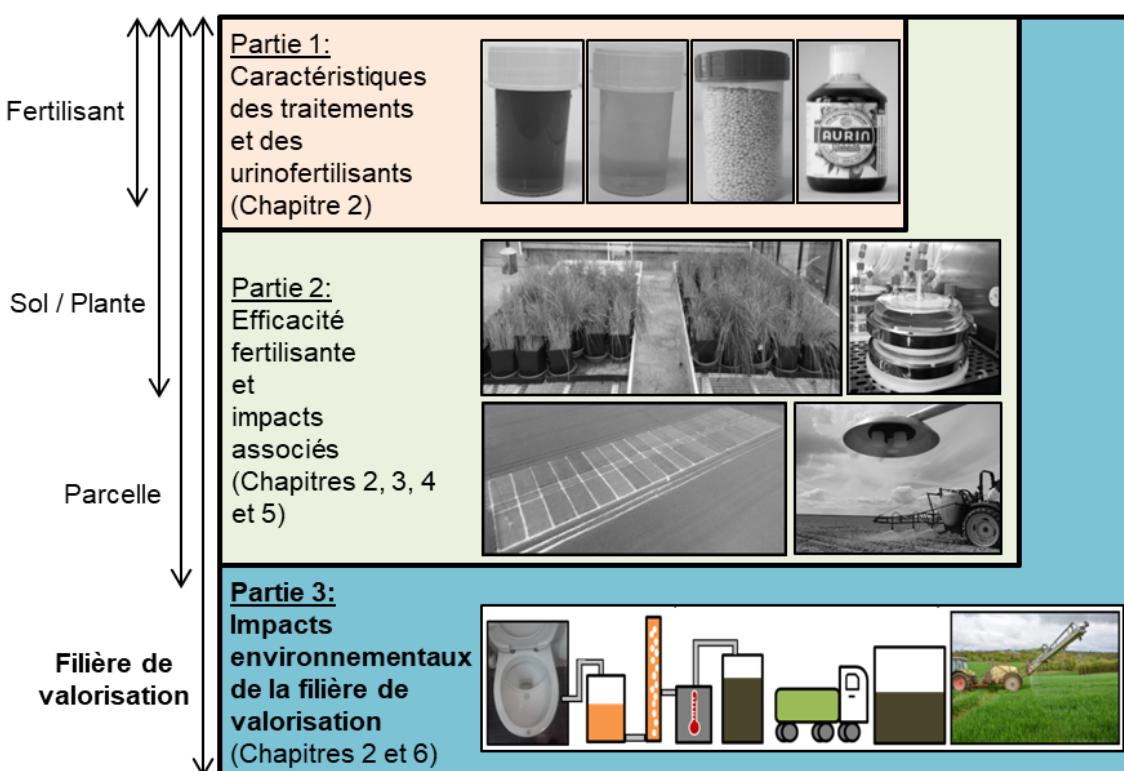
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Partie 3

Impacts des filières de valorisation

Nous avons vu au cours de la partie 1 que les traitements de l'urine sont très variés et qu'il en résulte de nombreux urinofertilisants aux caractéristiques physico-chimiques variées (p. ex., solide, liquide, concentré ou dilué). Ces différents traitements possibles ont aussi des impacts (p. ex. consommation d'énergie, de réactifs). Au cours de la partie 2, nous avons vu que les urinofertilisants ont une efficacité fertilisante azotée élevée et proche des engrains minéraux pour la plupart d'entre eux. Cependant, des impacts associés à leur utilisation (en particulier la volatilisation ammoniacale) peuvent exister en fonction des caractéristiques des urinofertilisants. Afin d'identifier les filières de valorisation les plus prometteuses et les points d'amélioration éventuels, il est donc nécessaire de réaliser un bilan environnemental de l'ensemble de ces filières, depuis la toilette jusqu'à la valorisation de l'urinofertilisant.

Cette troisième partie porte sur l'évaluation des impacts des filières de valorisation de l'urine en agriculture. L'objectif est de comparer différentes filières de valorisation entre elles et aux pratiques actuelles, c'est-à-dire fertilisation classique (minérale ou organique) et traitement de l'urine à la station d'épuration. Pour cela, une évaluation des impacts environnementaux d'une production de céréales fertilisées avec des urinofertilisants ou des engrains minéraux a été réalisée (chapitre 6). Nous avons considéré 3 filières de valorisation de l'urine et deux modes de production agricole (conventionnelle et biologique). Les impacts environnementaux de l'ensemble de la filière ont été pris en compte, allant de la toilette à la production de grains. Cette évaluation a été réalisée en utilisant la méthode de l'analyse de cycle de vie (ACV).



Chapitre 6

**Évaluation des impacts environnementaux de
l'utilisation de trois urinofertilisants en comparaison
des engrains classiques pour la production de céréales
par analyse du cycle de vie**

Comparative study of environmental impacts related to cereal production with human-urine based fertilizers versus classical fertilizers

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Abstract

Most of the fertilizers used in conventional agriculture are derived from non-renewable resources. In organic agriculture, nitrogen is often a limiting factor and organic fertilizers are less efficient in short term and relatively scarce. On the other hand, the nutrients from human urine are weakly recycled to agriculture and may cause various pollutions. Human urine may be collected through source separation and treated to produce “urine-based fertilizers”, resulting in various value chains.

Using the LCA method, we assessed the environmental impacts of producing wheat fertilized with three different urine-based fertilizers (UBF): stored urine, nitrified urine and alkalinized dehydrated urine. We compared them to two reference systems: the use of mineral fertilizers in conventional agriculture and chicken dropping in organic farming. In these scenarios, urine is considered as treated in a wastewater treatment plant.

In most cases, the impacts of all urine-based fertilizer value chains were lower than the reference system for the climate change, eutrophication, fossil resource and water consumption impact categories. These lower impacts were partly related to the reduced impacts from the avoided reference fertilizer production. Moreover, the avoided impacts for the sanitation system (water consumption, greenhouse gas emissions, and nutrients discharge to the river) were significant and permitted to further and greatly reduce the environmental impact of urine-based fertilizer compared to the reference scenarios. Nevertheless, stored and alkalinized dehydrated urine can induce higher ammonia volatilization compared to the reference fertilizers studied resulting in a higher acidification potential and particulate matter emissions. The electricity consumption for nitrified concentrated urine and alkalinized dehydrated urine fertilizers is also higher than the reference system. Thus, ammonia volatilization and energy consumption of the treatment have been identified as the main environmental hotspots of urine-based fertilizer value chains.

In conclusion, urine-based fertilizers contribute to close the biogeochemical cycles and permit to reduce globally the environmental impacts of wheat production.

Key words

Environmental impacts; Human urine; Life cycle assessment; Urine-based fertilizer; Energy; Ammonia volatilization

1. Introduction

About 75% of the nitrogen fertilizers used in conventional agriculture come from the Haber-Bosch process, the 25% left corresponding to biological fixation. However, it is an energy intensive process that requires around 2% of global energy consumption (Matassa et al. 2015). Nitrogen (N) fertilizers represent an important part of the carbon footprints of farms (Yan et al. 2015). Additionally, phosphorus (P) fertilizers mainly come from mines whose reserves are limited and concentrated in few countries (Cordell et al. 2009; U.S. Geological Survey 2019). On the other hand, in organic agriculture the nitrogen supply often remains a limiting factor for the yield (Berry et al. 2002). Today in organic agriculture, nitrogen fertilization is mainly based on biological fixation (legumes) and organic fertilizers (e.g. manure, slurry). Thus, the use of a renewable and efficient fertilizer in organic farming is also of high interest.

In high-income countries, most wastewaters are collected and treated in wastewater treatment plants (WWTP). Sewage sludge issued from the treatments can be recycled towards agriculture (e.g., about 65% in France). However, the proportion of recycled nitrogen is very low since denitrification of nitrogen is considered the best sanitation practice in wastewater treatment and usually allows less than 10% recycling of nitrogen (Esculier et al. 2018). Denitrification is also an energy-intensive treatment (Maurer et al., 2003). In addition, greenhouse gas nitrous oxides can be emitted in high proportions (Kampschreur et al. 2009). The recycling rate can be much higher for phosphorus (typically 80%) but when sewage sludge is incinerated, this leads to a total loss of phosphorus. Finally, a significant proportion of nitrogen and phosphorus remains discharged to surface water. For example, the treatment yield required by European Union regulations in sensitive areas is 70-80% for nitrogen and 80% for phosphorus of the flows entering large WWTPs in usual conditions (Directive 91/271/EEC – urban wastewater treatment), which still leaves more than 20-30% discharge in the receiving waters.

Most of the nutrients in wastewater come from urine with around 80% of the nitrogen and 50% of the phosphorus. When excreted, these nutrients are concentrated in a small volume of about 0.5 m^3 per year and per person (Friedler et al. 2013). Source separation is necessary to separate urine from wastewater and avoid nutrient dilution. It can be performed in various types of toilets and urinals (Rossi et al., 2009).

Nitrogen is excreted in urine mainly as urea (Udert et al. 2006). However, urea is rapidly hydrolyzed into ammoniacal nitrogen during storage. After being collected, urine can be treated, with different objectives (Martin et al. in press): (i) Stabilization of nitrogen (e.g. nitrification, alkalinization), in a form which limits ammonia volatilization, reduce malodor and permit further concentration. (ii) Volume reduction to reduce storage space, heavy transport over long distance and application labor. (iii) Extraction of nitrogen and/or phosphorus to obtain a final product concentrated

in nutrient. (iv) Treatment of pharmaceutical residues and/or pathogens to reduce potential impacts on health and on the environment. All these treatments lead to a wide variety of products with various physico-chemical characteristics (e.g., solid, liquid, basic, acidic) that can be used in agriculture. The name “urine-based fertilizer” (UBF) qualifies these products resulting from urine treatments. Even if the use of UBFs is not explicitly allowed in the European Union regulation for organic agriculture such use of a renewable and efficient fertilizer in organic farming could be of high interest.

Considering their entire value chain from the toilet to the field, including collection, treatment, transport and application on crops, the UBFs can have several environmental impacts such as greenhouse gases emissions due to the consumption of fuel for transport or acidification and eutrophication of the environment due to ammonia volatilization. The various characteristics of the value chains may generate different environmental impacts related with the production of UBFs (e.g. amount of energy and reagents for treatments). Several studies aimed to characterize the environmental impacts of different ways of managing urine using life cycle assessment (LCA). Most studies focused on different ways of managing wastewater (e.g., source separation of urine, all-in-sewer) and the environmental impacts of the sanitation system took into account potential recycling of nutrients from wastewater to agriculture replacing the use of mineral fertilizers (Remy and Jekel 2008a; Tervahauta et al. 2013; Bisinella de Faria et al. 2015). Other studies focused on the production of fertilizer from wastewater and in particular from urine (Spångberg et al. 2014; Malila et al. 2019). However, to our knowledge, only one study focused on applied cases up to agricultural production taking into account the technical constraints for the application of one UBF (stored urine, Tidaker et al. 2007). The studies carried out on urine source separation have generally shown an improved environmental performance (e.g., less eutrophication) for different impact categories compared to the use of mineral fertilizers and the treatment of wastewater in WWTP. Moreover, most studies only concerned stored urine and few other processes such as struvite precipitation. No studies have been carried out on nitrified concentrated urine and alkalinized dehydrated urine, which are emerging treatment process of urine.

The main objective of the study was to assess the environmental impacts of wheat production fertilized with three different UBFs compared to usual fertilizers using the LCA method.

2. Method

2.1. Goal and scope of the study

2.1.1. Goal of the study

The Life Cycle Assessment (LCA) method was used according to the ISO 14000 series standard (ISO 2006a,b). LCA is a tool that allows a global and multi-criteria assessment of the environmental impacts of a production throughout its value chain. LCA can be applied to assess the environmental impacts of agricultural production (van der Werf and Petit 2002; Pradel 2011). Therefore, we decided to conduct a comparative assessment of the environmental impacts of different ways producing wheat using traditional practices of fertilization and the use of various UBFs. We selected three different UBFs: stored urine, nitrified concentrated urine and alkalinized dehydrated urine. They were chosen since they are emerging products on which research is active and because of their various characteristics in terms of treatment, end product and spreading methods (diluted liquid, concentrated liquid, solid, respectively). Their environmental impacts were compared to the currently most frequent system: the use of mineral fertilizer in conventional agriculture and chicken droppings in organic farming and the treatment of urine in WWTP. The technical constraints according to the diversity of fertilized crops (e.g. usable agricultural machinery, doses to be applied) were taken into account to propose technically possible fertilization practices scenarios. Specific agricultural and urban configurations (peri-urban territory) were chosen to reflect at best plausible scenarios of urine recycling chains. Additionally, different scenarios for the use of UBFs in organic agriculture were designed. Thus, eight different scenarios (one reference and 3 UBFs for each agricultural system) were designed.

2.1.2. Functional unit

The functional unit makes it possible to calculate the impacts of the value chain for the same production/function and to compare the different scenarios with each other. The different systems studied included the sanitation sub-system and the agricultural sub-system. In the systems studied the primary functions were: (i) To treat urine and feces, (ii) To produce fertilizers, (iii) To produce crops. The functional unit chosen was **to produce 1 kg of grain** (15% moisture as the agronomical reference to measure the yield) **leaving the farm**. This functional unit was chosen to take into account the impacts at the farm level as they may differ due to various characteristics of UBFs (e.g. amount of fertilizer to be spread, ammonia volatilization).

2.1.3. Boundaries of the system

The different stages of the scenarios and the inputs production that we have fixed are indicated in Figure 6-1.

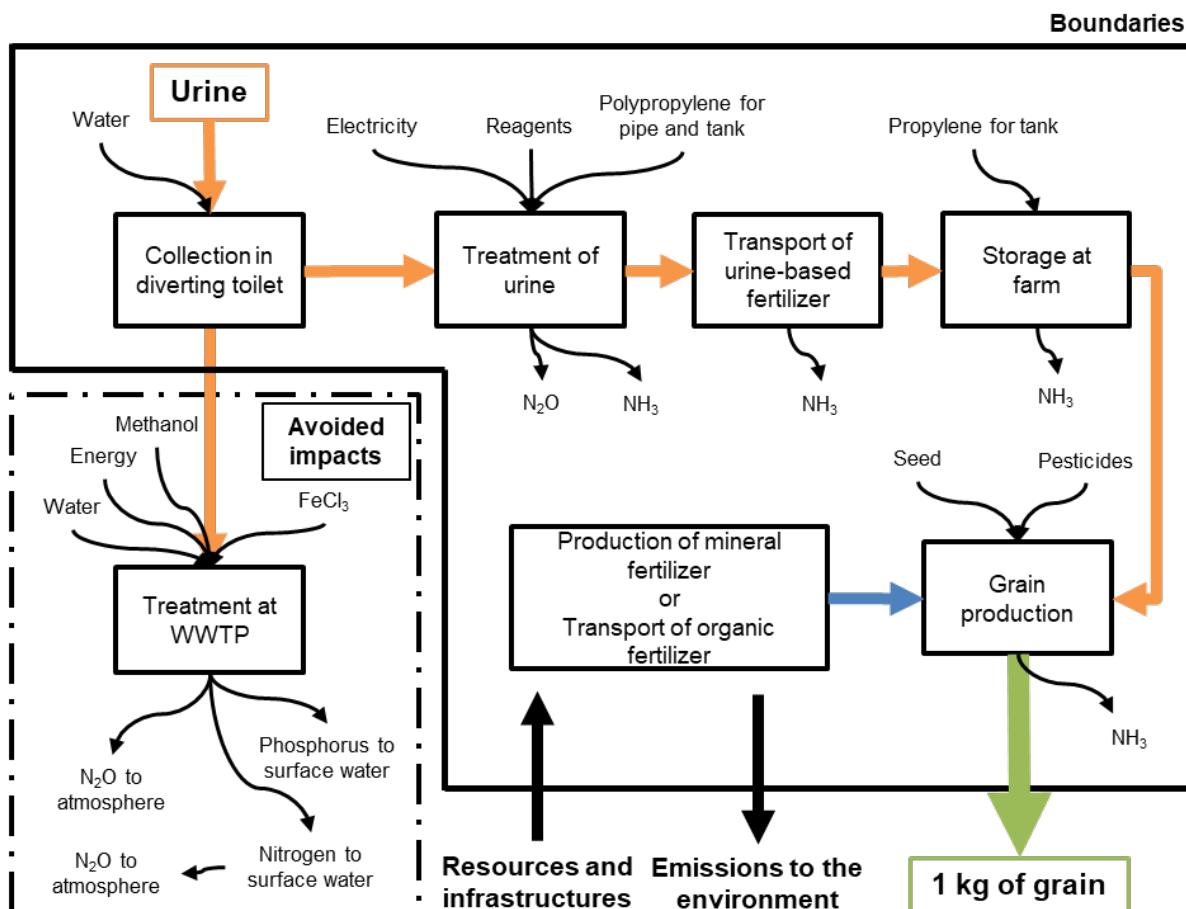


Figure 6-1. Boundaries and foreground data of the systems studied.

For the UBFs value chains, the impacts from the toilet to the farm were taken into account. The study was in a change oriented perspective and corresponded to a conversion from the use of mineral fertilizers (and the associated treatment of urine at the WWTP) to the use of UBFs in agriculture (and the associated treatment of urine to produce UBFs). The proposed value chains could be potentially implemented in the near future in new districts equipped with source separation. The scenarios we considered were “marginal” and with no structural changes.

2.2. Life cycle inventory analysis and description of the scenarios

For the analysis of the inventory, the data used were mainly based on literature review. Four different scenarios were designed and are detailed in the following part: (i) Reference, (ii) Stored urine, (iii) Nitrified concentrated urine (NCU), (iv) Alkalized dehydrated urine (ADU). These scenarios were duplicated on two different farming systems (conventional and organic farming) and on two different crops (soft winter wheat and grain maize). As the results were similar for wheat and maize, only the results for wheat will be presented in details. The main hypothesis taken for the different scenarios are presented in Table 6-1.

Table 6-1. Main data and hypothesis along with the reference used for the different scenarios. The parameters expressed as % concern the gaseous losses or the treatment efficiency occurring at a specific stage of the value chain and not on the initial urine. Tkm stands for the transport of one ton on one km.

| Category | Parameter | Unit | Scenarios | | | Comments and Reference |
|--------------------------------------|---|---|---------------------|------------------------------|---|--|
| | | | Reference | Stored urine | NCU | |
| Urine collection and storage | Volume urine | L/cap/d | 1.27 | 1.27 | 1.27 | |
| | N load in urine | g N/cap/day | 11 | 11 | 11 | 11 |
| | P load in urine | g P/cap/day | 0.93 | 0.93 | 0.93 | 0.93 |
| | Time at home | % | 65 | 65 | 65 | Jönsson (2002) |
| | Number flush at home | p/cap/day | 4.6 | 4.6 | 4.6 | Spångberg et al. (2014) |
| | Separation yield at the toilet | % | 90 | 90 | 90 | Estimated from Gundlach et al. (2021). |
| | Flush for urine diverting toilet | L/flush | | 1.5 | 1.5 | 0.1 L urine bowl and 1.4 L feces bowl. |
| | Flush for urine conventional toilet | L/flush | 3 | - | - | Friedler et al. (2013) |
| Urine treatment | Tank district | m ³ | - | 17 tank of 6 m ³ | Two tanks of 2 m ³ and 10 m ³ | 340 boxes of 0.040 m ³ Polyethylene tank. 1 tank per building for stored urine. 2 per district for nitrified concentrated urine, 1 box per toilet for dehydrated alkalinized urine. |
| | Ammonia volatilization during storage | % of N | - | 0.5 | 0.1 | 0.5 Estimated |
| | Nitrogen concentration factor | - | - | 1 | 10 | 20 Estimated based on Etter et al. (2015) for nitrified urine and Senecal and Vinnerås (2017) for alkalinized dehydrated urine. |
| UBF transport | Electricity consumption | kWh/m ³ urine entering the treatment | - | - | 130 | 533.3 1.92 kWh/toilet/d for alkalinized dehydrated urine, Simha (personal communication). Etter et al. (2015) for nitrified concentrated urine. |
| | NH ₃ volatilization | % of N | - | - | - | 10 Simha (personal communication) |
| | N ₂ O emissions | % of N | - | - | 0.5 | - Estimated |
| | Lime consumption | g Ca(OH) ₂ /L urine | - | - | - | 10 Randall et al. 2016 |
| Storage at farm and grain production | Transport from district to farm | Tkm | - | 30.7 | 3.1 | 1.4 25 km. Freight lorry (Euro 6): 16-32 ton capacity for stored urine, 7.5-16 ton capacity for nitrified concentration urine and 3.5-7.5 ton capacity for alkalinized dehydrated urine. |
| | NH ₃ volatilization | % of N | - | 0.1 | 0 | 0.1 Estimated |
| Treatment at WWTP | Tank | m ³ | Neglected | 2 tank of 250 m ³ | 50 | Neglected 2 Flexible Polyvinyl chloride tank for stored urine and 1 Polyethylene solid tank for nitrified concentrated urine. |
| | Ammonia volatilization at spreading | % of N | - | 7 | 0.5 | 15 See text |
| | Wheat yield | t /ha | 8.2 conv 4.5 org | 8.2 conv 4.5 org | 8.2 conv 4.5 org | 8.2 conv 4.5 org At 15% moisture. Conventional and organic. |
| | N removal efficiency | % of N | 62.3 | - | - | - |
| | P removal efficiency | % of P | 84 | - | - | - |
| | Energy consumption | kWh/kg N removed | 2.78 | - | - | - |
| | Methanol consumption | kg CH ₃ OH/kg N removed | 3.4 | - | - | - |
| | FeCl ₃ consumption | kg FeCl ₃ /kg P removed | 9.15 | - | - | - SIAAP (2020) |
| | N ₂ O emissions to atmosphere | % of N | 2.1 | - | - | - Bollon et al. (2016a, b) |
| | N ₂ O to atmosphere from river | % of N | 0.5 | - | - | - IPCC (2006) |

2.2.1. Common hypothesis and background data

For the WWTP, a significant amount of urine must be diverted to have a significant change on infrastructure and processes (Igos et al. 2017). As our scenario only represents the case of about a thousand inhabitants, the impacts of the sanitation system were considered with an expansion of the system. The avoided impacts of the sanitation system in term of reagent consumption (methanol and FeCl₃), flushing water and energy, because the urine was not sent there but applied in the field, were taken into account. Other impacts on the sanitation system such as infrastructures, change in the quality of sewage sludge or the production of biogas at the WWTP were not taken into account.

For the UBFs scenarios, all relevant activities from the toilet to the farm leaving gate were considered. It included (i) Urine collection (same urine in all scenarios) using diverting toilet, (ii) Urine treatment to produce UBFs, (iii) Transport of UBFs to the farm (iv) Storage of UBFs at the farm level and, (v) All activities associated with grain production. For the reference scenario in conventional agriculture, only mineral fertilizer production and the activities associated to grain production were considered. For the reference scenario in organic agriculture, no impacts were allocated to the production of chicken dropping (except their transport to the farm) as they were considered as waste from chicken breeding (Koch et al. 2015). For the agricultural part, plurennial works (liming, drainage) were not taken into account and only N and P fertilization were considered. To simplify the scenarios no sewage sludge recycling was considered. At the regional level, sewage sludge represents a small amount in the nitrogen and phosphorus balance of farms on average. Soil was considered as part of the system. However, nitrate leaching was considered as an environmental impact. The study timeline was one year from the harvest of the previous crop to the harvest of the crops we studied (Koch et al. 2015). For capital goods, only the additional infrastructure required for the UBFs scenarios were taken into account (e.g. tank for storage at the district and farm level). All other infrastructures (e.g. sewers to WWTP) were excluded from the boundaries of the study. The capital good impact was divided by the estimated service life. Conventional toilets and urine diverting toilets were considered to be identical in terms of impacts and not taken into account. Only the avoided flushing for urine was taken into account for the reference and UBFs scenarios. All aspects relating to disposal, maintenance, impacts linked to the human work (e.g. transport to go to the workplace) were not taken into account. The impacts of the pumping stage before transport were considered as insignificant (Malila et al. 2019). Other aspects such as excavation needed to install tanks were not taken into account.

The scenarios take place in the Paris region (Ile-de-France). The climate is temperate with an average temperature of 10.5 °C and around 650 mm of precipitation per year. The main soil type in the region is silty luvisol. It is an area specialized in arable crops (mainly wheat, maize, rapeseed and barley) using intensive agriculture practices. The electricity mix was considered to be the French mix

with more than two thirds of the electricity produced from nuclear power plants. The different stages of the value chains are hypotheses because none on the valorization chains are already in place on a large scale.

The hypotheses for the upstream part of the UBF scenarios were done to calculate the impacts per kg of UBFs. However the total amount of fertilizer produced was not linked with the farm production. We assumed that urine collection took place in a medium density urban area. A new district of 1020 inhabitants equipped with source separation was considered. The district was made up of 17 buildings. Each building contained 5 floors with 4 apartments per floor (340 apartments in the neighborhood). Three people (2 adults and 1 child) per apartment were considered which represented 60 people per building. The amount of urine excreted was considered to be the same for adults and children and was set at 1.27 L urine/cap/d (Friedler et al. 2013). Assuming that 65% of the time was spent at home (Jönsson 2002), the urine collected was 301.5 L per year per capita. For the UBFs scenarios, the urine separation efficiency was estimated to be 90% (based on the data from Gundlach et al. 2021). It may have been overestimated because the separation efficiency may be lower when toilet is used by children. The ammonia emissions from the toilet were assumed to be insignificant (Malila et al. 2019). The concentrations of nitrogen and phosphorus in fresh urine are 8.7 g N/L and 0.73 g P/L and represent respectively 82% of nitrogen and 46% of phosphorus in domestic wastewater (Friedler et al. 2013). Due to the water balance, the urine in the morning is more concentrated but in order to simplify the calculations, we considered it to be homogenous during the day. The urine flush volume was estimated as 1.5 L in diverting toilet, from which 100 mL go to the urine bowl and dilute urine (Gundlach et al. 2021) and 1.4 L go to the feces bowl. Volumes of 3 L were considered for conventional flushing (Friedler et al. 2013) and 4.6 flushing per capita and per day were considered (Spångberg et al. 2014). The urine dilution factor with the diverting toilet was approximately one third. Taking into account these parameters, in the UBFs scenarios, 59% of the daily urine was source separated representing respectively 48% and 27% of daily nitrogen and phosphorus loads in domestic wastewater. It represents 6.6 kg of N and 0.55 kg of P and 450 m³ collected per day at the district level. For all UBFs, we assumed that the composition of the collected urine was homogeneous in term of nutrient concentration due to the collection of large volumes. The distance between the neighborhood where the urine was collected and the farm was set at 25 km.

The considered WWTP was based on the main WWTP in the Paris region (Achères – Seine-Aval). Operating data are available for this WWTP (SIAAP 2020) but it represents a special case because it is a very large WWTP (5 million inhabitants equivalent, average of 1.5 million m³ treated per day). The treatment of nitrogen consists of nitrification and denitrification. The phosphorus is treated by precipitation using ferric chloride (FeCl₃). The consumption of FeCl₃ was considered according to the amount of phosphorus to be treated. Methanol is used as a carbon source for the denitrification and its consumption is correlated with the quantity of nitrogen to be treated. N₂O is a potent greenhouse gas

with a global warming potential of 265 (100 years, IPCC 2015). Denitrification is carried out on bio-filters with an N₂O emission rate estimated at 2.1% of the N entering the WWTP (Bollon et al. 2016a, b). Energy consumption is related to the amount of nitrogen treated. The N removal efficiency is 62.3% of the N entering the WWTP and the P removal efficiency is 84% of the P entering the WWTP, the rest of nutrients are considered to be released in the river (Esculier 2018) from which 0.5% of the N released to the river is expected to be emitted later as N₂O (IPCC 2006).

For life cycle inventory calculation background data were extracted from Ecoinvent ® data base version 3.4 (Wernet et al. 2016).

2.2.2. Reference scenario of fertilizer production

The reference scenario corresponded to the current dominant system. Fertilization was based on the use of mineral fertilizers in conventional farming. Two nitrogen fertilizers (urea ammonium nitrate solution and ammonium nitrate) were considered. They are produced using the Haber-Bosch process. Phosphorus fertilizers (superphosphate for wheat or 18-46-00 for maize) are derived from fossil-phosphorus from mines. Storage of mineral fertilizers at the farm was neglected due to low quantities occupied implying few infrastructures which already exists. In organic agriculture, the reference fertilizer was chicken droppings. It was chosen as it is an organic fertilizer with a high efficiency (as expected for UBFs) and one of the most used in organic systems in the study region. For the sanitation part, the entire urine collected using conventional toilet in the district was sent to the WWTP for treatment.

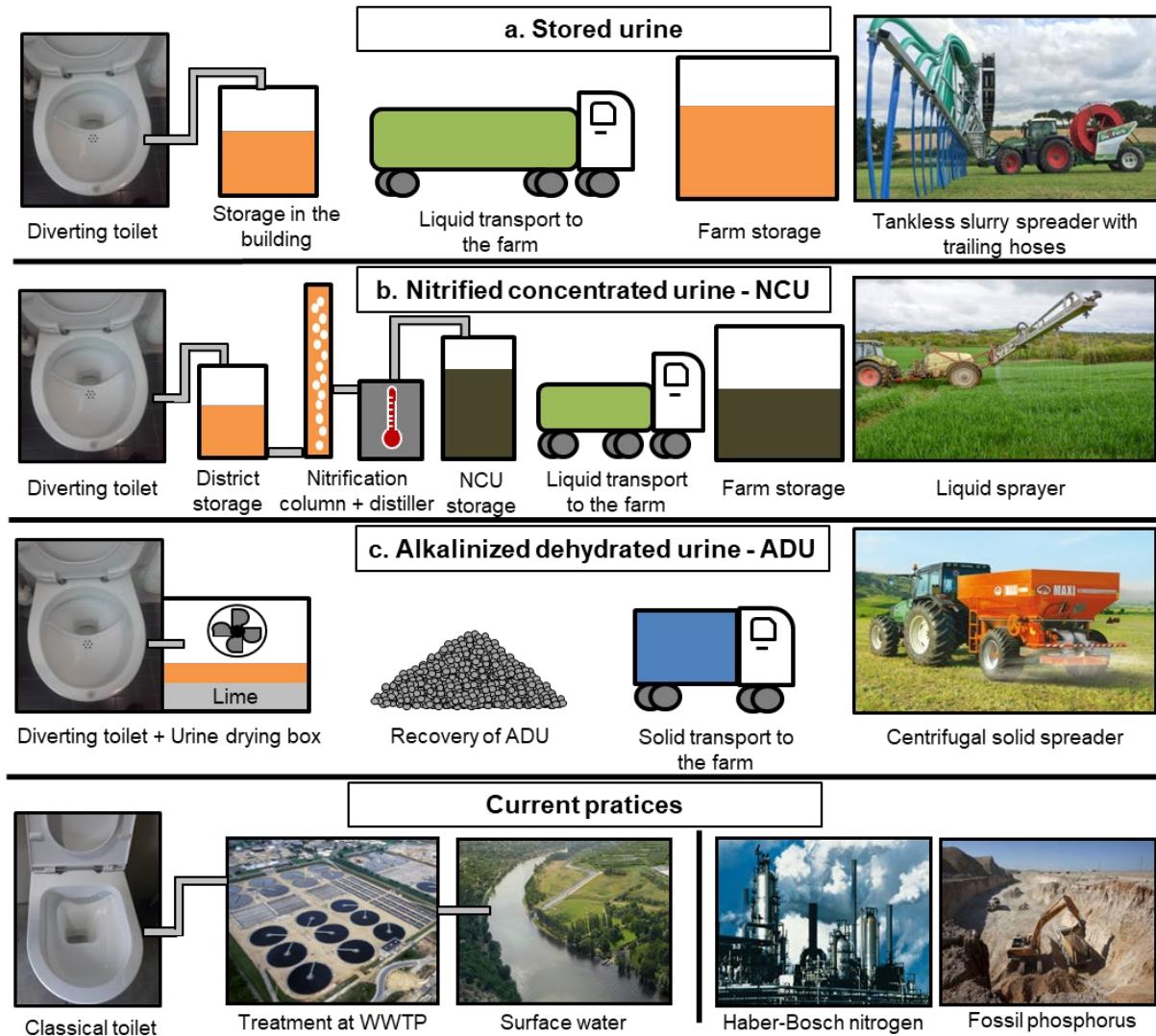


Figure 6-2. Upstream part of the different urine-based fertilizers values chains and current practices. Credits: SIAAP, entraid.com, paysan-breton.fr, osp.frejustoulon.fr, terre-net.fr, GIP Seine-Aval

2.2.3. *Stored urine*

The stored urine value chain is shown in Figure 6-2a. In fresh urine, approximatively 85% of the nitrogen is excreted as urea, 5% as ammoniacal nitrogen and 10% as other organic N (Rose et al. 2015). Phosphorus is mainly present in dissolved form as phosphate (Udert et al. 2006). Storage is considered as a urine treatment because there are changes in the composition of the urine leading to sanitation of urine in terms of potential pathogenic germs (WHO 2012). Indeed, urea is rapidly hydrolyzed into ammoniacal nitrogen due to the ubiquitous presence of the urease enzyme secreted by microorganisms (Friedler et al. 2013). Hydrolysis of urea increases the pH of urine to 9.1 (Udert et al. 2006). Ammonia volatilization can occur during the different steps of the value chain and in particular during the spreading since the pH is close to 9.2, the pKa of the $\text{NH}_4^+ / \text{NH}_3$ pair. Urine collection is organized at the building level. Urine is separated using urine diverting toilets and is directed to a

storage tank in the basement of the building. The storage tank is 6 m³ (polypropylene) and is emptied every 2 months. Ammonia volatilization in pipes and storage tanks is estimated to 0.5% of the N of the collected urine (account for storage at the neighborhood and at the farm). Urine is transported to the farm using a freight lorry (16-32 ton capacity). Ammonia volatilization during transport is 0.1% of the total N entering the truck. About 450 m³ of stored urine is collected per year at the district level. Considering a capacity of 32 tons for the truck, the volume of stored urine recovered is equivalent to 14 trucks. Stored urine is stored at farm level in two different 250 m³ tanks (PVC flexible tank) in order to allow at least 1 month storage to be respected as recommended by the WHO (2012) before application on crops. The increase in pH after urea hydrolysis causes about 30% of the phosphorus to precipitate as struvite and hydroxyapatite forming sludge at the tank bottom or deposits in the urine pipes (Höglund et al. 2000). We considered that the sludge and the deposits were mixed with the liquid part. The nitrogen concentration in the UBF corresponding to stored urine applied is 5.4 g N/L (density is equal to 1).

2.2.4. Nitrified concentrated urine

The nitrified concentrated urine (NCU) value chain is shown in Figure 6-2b. The start of the value chain is the same than for stored urine with source separation using diverting toilet but urine is transported through pipe to a treatment station centralized at the district level located in the central building of the district. Before the treatment, the urine is stored at the treatment station in a 2 m³ tank (polypropylene, 1.2 m³ of flush diluted urine recovered per day in the district). We assumed that urea hydrolysis occurred in the pipe and storage tank before the treatment as it occurs quickly (Udert et al. 2003). Ammonia losses during storage before the treatment were estimated at 0.1% due to the short-term storage. The treatment is continuous and consists of a biological nitrification of half of the ammoniacal nitrogen of the stored urine by adding nitrifying bacteria and supplying air. Nitrification permits to stabilize nitrogen in the form of nitrate and acidifies the solutions, which limits the potential ammonia volatilization and allows volume reduction by evaporation of water. Nitrification is followed by volume reduction by distillation using a vapor compression vacuum distiller (Fumasoli et al. 2016). The entire treatment process is marketed by the company Vuna (www.vuna.ch). The N concentration factor after distillation was set to 10 (53.4 g N/kg in the UBF end product) in accordance with the already existing value chain. Energy consumption was set at 130 kWh/m³ per liter of urine entering the treatment (Etter et al. 2015). As N₂O emissions can occur during nitrification, 0.5% of the N entering the nitrifying column is assumed to be emitted as N₂O. Nitrified concentrated urine is stored in a 10 m³ tank (polypropylene, emptied every 2 months), before being transported to the farm using a freight lorry (6 trucks per year considering an 8 tons capacity truck). Nitrified concentrated urine is stored on the farm in a 50 m³ polyethylene tank (about 45 m³ of nitrified concentrated urine is produced in the district per year, as the density is 1.14, it corresponds to about 51 ton per year).

2.2.5. Alkalinized dehydrated urine

The alkalinized dehydrated urine (ADU) value chain is shown in Figure 6-2c. Alkalinization of fresh urine above pH 10 by adding a base inhibits the urease enzyme and stabilizes the nitrogen in the form of urea (Geinzer 2017). As with the other scenarios, urine is collected in a urine diverting toilet. However, this process is a decentralized treatment at the toilet level. A 20 cm pipe is used to bring urine to the drying box. A short pipe is needed to avoid urea hydrolysis (Senecal and Vinnerås 2017). The drying box consists of a polyethylene box (40 L) next to the toilet. The drying box is filled with hydrated lime at a rate equivalent to 10 g Ca(OH)₂/L of urine. A fan with a power of 80 W operates continuously to evaporate the water from urine. Thus, the energy consumption per toilet is 1.92 kWh/toilet/day or 533 kWh/m³ urine treated (Simha, personal communication). The process has been developed by the Swedish University of agriculture (SLU, Simha et al. 2020). One drying box is installed per toilet per dwelling (340 at the district level). Ammonia losses in the drying box are estimated at 10% because 5% of nitrogen in urine is already excreted as ammonia and volatize due to the high pH of lime, and 5% volatilize during the rest of the treatment. Four times a year, the drying box is emptied (19.8 kg of alkalinized dehydrated urine produces per capita per year) and alkalinized dehydrated urine is transported by truck (4 trucks per year considering a 5 tons capacity truck) to the farm. The nitrogen concentration factor was set at 20 (107 g N/kg in the end product) according to previous studies with this process (Senecal and Vinnerås 2017). Storage of alkalinized dehydrated urine on the farm is neglected due to low space occupied implying few infrastructures and which already exist. (20.2 t produced per year in the district).

2.2.6. Grain production

We considered two averages farm in the Paris region producing field crops, a conventional and an organic farm. In both cases, the farm area was estimated at around 140 ha (calculated from the number of farms in the region and the total agricultural area). We considered that the farm collected all UBFs produced at the district level. All the urine collected in the studied district could cover approximately 10% of the total nitrogen demand of the farm in conventional farming considering the dose applied for wheat (180 kg N/ha, 10.2 to 12.4 ha fertilized with the UBFs collected depending of the scenarios). It represents about one third in organic farming considering the dose applied for wheat (50 kg N/ha, 36.7 to 44.4 ha fertilized with the UBFs collected depending of the scenarios). The different steps of the technical itineraries are presented in Figure 6-3. The straw and other crop residues were considered as incorporated into the soil, no impact were allocated to the straw and crops residues. No impact of the spreading machinery on the yield was considered. The agricultural part of the inventory was carried out using the MEANS-InOut Software version 2.11 (www.inrae.fr/means, Auberger et al. 2018) which is based on the Agribalyse version 1.4 (Koch et al. 2015; Colomb et al. 2015) and Ecoinvent® version 3.4 (Wernet et al. 2016) databases. Except for the ammonia volatilization after UBFs application

which we defined, the other direct emissions at field (NH_3 for mineral fertilizer, N_2O and NO_x) were calculated using the IPCC et al. (2006) and EMEP/EEA (2009, 2016) emissions factors. Losses of N and P to water bodies were calculated using the Arvalis (Tailleur et al. 2012) and SALCA models (Gaillard and Nemecek 2009).

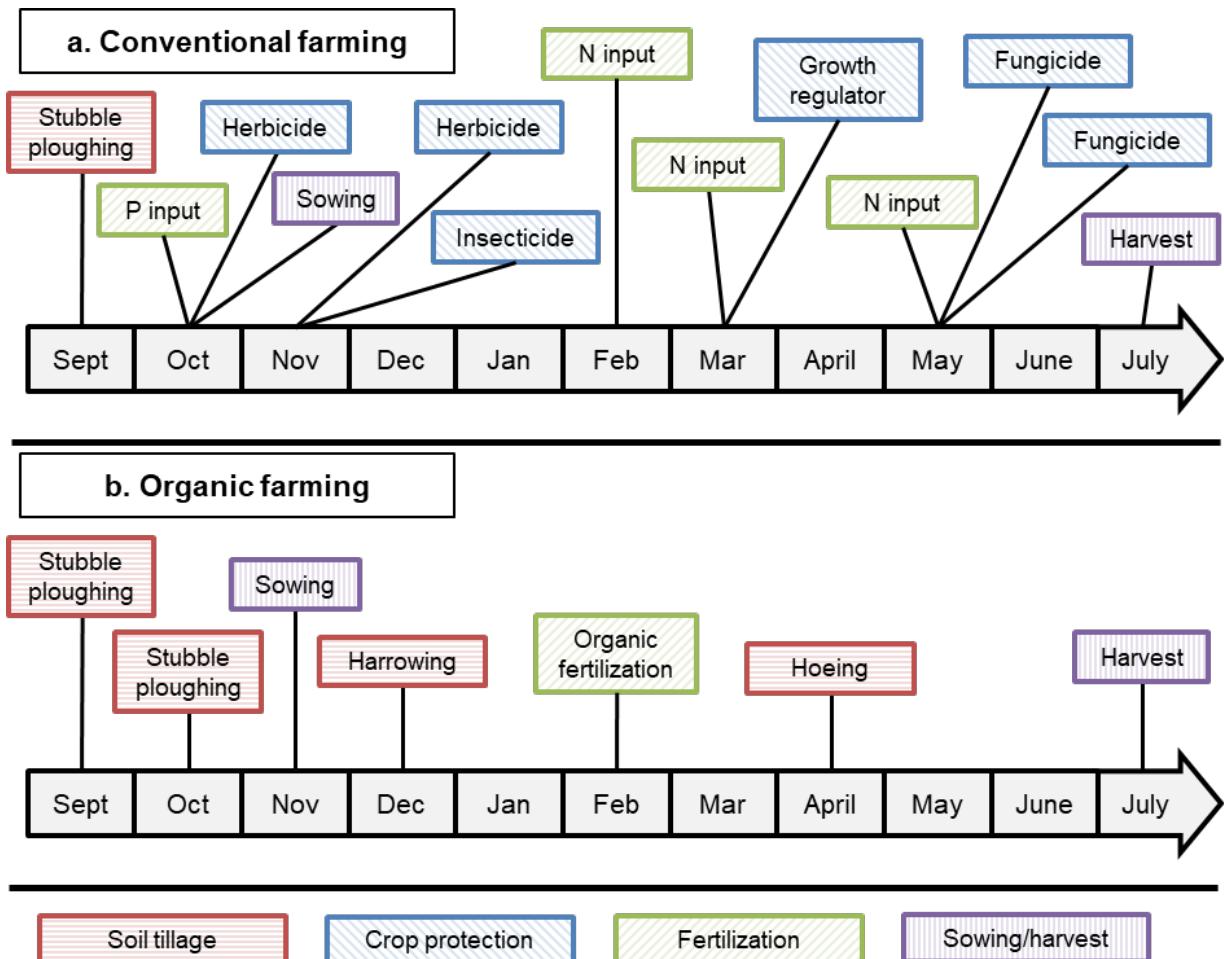


Figure 6-3. Technical itineraries in conventional farming (a.) and organic farming (b.)

The technical itineraries were developed according to local practices and the recommendation of the local farmer organization (Chambre d’Agriculture de région Ile-de-France; Lefèvre 2015). The conventional technical itinerary (Figure 6-3a.) corresponds to intensive agriculture practices. The only soil tillage is stubble ploughing in September in which the straw of the previous harvest (wheat) was incorporated into the soil. Sowing was done at mid-October with 110 kg seed /ha. For crop protection, five treatments including three herbicides, two fungicides and one insecticide were carried out. A growth regulator was applied in March. The grain yield was the same for all scenarios and was set at 8.2 t/ha (at 15% moisture per ha) on the basis of the average yield over 5 years in the region (Chambre d’agriculture de Seine-et-Marne 2017). In the organic farming technical itinerary (Figure 6-3b.), the absence of herbicide was compensated by a more intense tillage (two stubble ploughing, one harrowing, one hoeing). The previous crop was a spring faba bean. Sowing was done in early November with 180 kg seed/ha. The yield was set at 4.5 t/ha (at 15% moisture per ha) in accordance with the observed local yield.

Only the fertilization hypothesis differed between the scenarios for the grain production step (Table 6-2).

Table 6-2. Nitrogen, phosphorus concentration, nitrogen mineral fertilizer equivalent and amount applied for fertilizers tested in conventional and organic agriculture. The nitrogen doses for the different inputs correspond to the available nitrogen considering the mineral fertilizer equivalent. Superphosphate is added to complete the fertilization for a total dose of 50 kg P₂O₅/ha in the conventional farming scenarios. The density of all liquids urine-based fertilizers was taken as 1 except nitrified urine for which it is assumed to be 1.14.

| Fertilizer | Nitrogen concentration (g N/kg) | Phosphorus concentration (g P ₂ O ₅ /kg) | Ammonia volatilization at application (% of total N) | Nitrogen Mineral fertilizer equivalent (%) | Conventional farming | | | | Organic farming | |
|-------------------------------|-------------------------------------|---|---|---|----------------------------------|---------------------------------|---------------------------------|---------------------------------|-----------------------|-----------------------|
| | | | | | Super phosphate input (kg/ha) | 1 st input (t/ha) | 2 nd input (t/ha) | 3 rd input (t/ha) | Total input (t/ha) | Total input (t/ha) |
| Mineral fertilizer | Nitrogen solution | 300 | 0 | 9 | 100 | 111 | 0.20 | 0.27 | - | 0.47 |
| | Ammonitr ate | 335 | 0 | 2 | 100 | | - | - | 0.12 | 0.12 |
| | Stored urine | 5.4 | 1.0 | 7 | 93 | 28 | 12.1 | 16.1 | 8.0 | 36.2 |
| Urine-based fertilizer | Nitrified concentrated urine | 46.9 | 9.1 | 0.5 | 99.5 | 33 | 1.29 | 1.71 | 0.86 | 3.86 |
| | Alkalinized dehydrated urine | 107.0 | 20.7 | 13 | 87 | 20 | 0.64 | 0.86 | 0.43 | 1.93 |
| Organic fertilizer | Chicken droppings | 38.2 | 37.9 | 7 | 45 | - | - | - | - | 2.91 |

The fertilizing efficiency of stored urine (stored few months after source separation and before use) has been studied on many crops (Karak and Bhattacharyya 2011; Pandorf et al. 2019). In general, its fertilizing efficiency is similar or slightly lower than that of mineral fertilizers (Kirchmann and Pettersson 1995; Johansson et al. 2000; Viskari et al. 2018). The fertilizing efficiency of other UBFs is little studied. However, under controlled conditions (greenhouse) and field conditions, the differences between UBFs appear to be limited (Martin et al. in press, in preparation) For the UBFs scenarios, we considered that 100% of the nitrogen fertilization was carried out with UBF. Urine inputs are based on the dose of “available” nitrogen (considering the mineral fertilizer equivalent) as it is the main nutrient for fertilizing field crops. The best available techniques were considered for the application of urine in order to limit ammonia volatilization and soil compaction. However, there are still questions about the technical feasibility and the possibility of applying the last application using heavy machinery like trailing hoses and it needs to be confirmed by experiments. The availability of P was considered to be 100%. Potassium and sulfur (2.1 g K/L and 1.0 g S/L in fresh urine, Friedler et al. 2013) were not considered for fertilization, but the grain export was almost compensated by the UBFs inputs. The mineral fertilizer equivalent characterizes the fertilization efficiency of organic fertilizers. It was taken into account to calculate the UBFs inputs. As mineral fertilizer equivalent of UBFs is high, it was set at 100%, minus the ammonia volatilization that was estimated.

The crop requirement in mineral nitrogen was calculated using the nitrogen balance method of the local farmers organization (Chambre d’agriculture de Seine-et-Marne 2017) and local practices in the Paris region (Lefèvre 2015). For the conventional farming scenario it was set at 180 kg N/ha. Inputs were split into three: 60 kg N/ha at the tillering stage (mid-February), 80 kg N/ha at the 1 cm ear stage (mid-March) and 40 kg N/ha at the flowering stage (early May). P fertilization is done every two years in October with 100 kg P₂O₅/ha using superphosphate. Fertilization of half of the surface (equivalent to one year fertilization) was considered in our scenario resulting in a dose of 50 kg P₂O₅/ha. For the UBFs scenarios, the difference between the P input from the UBFs and the reference dose (50 kg P₂O₅/ha) was completed using superphosphate.

For the reference scenario, solid ammonium nitrate (335 g N/Kg) and solid superphosphate (460 g P₂O₅/kg) were applied using a 2.5 m³ centrifugal solid spreader. The urea ammonium nitrate solution (300 g N/kg, 390 g N/L) was applied using a liquid sprayer (2.5 m³). For nitrogen fertilization, the first two inputs were made using the mineral nitrogen solution and the last input was done using ammonium nitrate according to local practices. The total input of mineral N fertilizer was about 0.6 t/ha.

Stored urine nitrogen concentration is low (5.4 g N/L). It was spread using a tankless slurry spreader fitted with trailing hoses. A tank truck (15 m³) make the way and back from the farm to the plot to supply the tank of the spreader located on the side of the field. The total dose of stored urine

was 36.2 t/ha (2.4 spreader tank capacity per ha, 36 m³ of urine avoided at the WWTP). Ammonia emissions were estimated at 7% of total N based on climatic conditions at the spreading and previous experiments (e.g. Rodhe et al. 2004). Thus, the mineral fertilizer equivalent was set at 93%.

Nitrified concentrated urine (46.9 g N/kg) was spread using a 2.5 m³ liquid sprayer. Spreading using a slurry spreader can be easier in terms of volume to manage but the dose per ha (less than 2 t/ha) is not suitable for this material. The total dose of nitrified urine was 3.86 t/ha (1.5 sprayer capacity per ha, 34 m³ of urine avoided at the WWTP). Due to the low pH and half of the nitrogen as nitrate, the ammonia volatilization was estimated to be very low with 0.5% N of total N. Thus, the mineral fertilizer equivalent was set at 99.5%.

It was assumed that the alkalinized dehydrated urine (107 g N/kg) was applied using a 2.5 m³ centrifugal solid spreader. In a real case, transformation into pellet must be considered because the product recovered is in powder form. The total dose was 1.93 t/ha (0.8 spreader capacity per ha, 43 m³ of urine avoided at the WWTP). Due to the high pH, the potential volatilization was expected to be higher than other UBFs. We estimated it to 15% based on the urea emission factor given by EMEP/EEA (2019, for temperate climate, soil pH < 7). Thus, the mineral fertilizer equivalent was 85%.

For the organic farming scenarios, the total available nitrogen dose was estimated at 50 kg N/ha based on the local practices in the region. For chicken droppings (38.2 g N/kg), the mineral fertilizer equivalent was set at 45% according to local observations and the nitrogen balance method of the local farmers organization (Chambre d'agriculture de Seine-et-Marne 2017). Thus, the total dose for chicken droppings was 2.9 t/ha representing 111 kg N/ha of total N but only 50 kg N/ha of available nitrogen. Chicken droppings were applied using a 10 t capacity manure spreader (0.3 spreader capacity per ha). The entire dose was applied at the end of winter (early March). A transport of 400 km was considered between the laying hen breeding and the farm. Indeed, as there is little chicken breeding in the study region, they are often imported from other region (e.g. Britanny, Belgium). In view of the lower yield, the export of P by grain is compensated by the input from chicken droppings or UBF. The inputs were made using the same agricultural machinery as for the conventional farming scenarios. The doses were 10.1 t/ha for stored urine (0.7 spreader capacity per ha, 10 m³ of urine avoided at the WWTP), 1.07 t/ha for nitrified concentrated urine (0.4 spreader capacity per ha, 9.5 m³ of urine avoided at the WWTP) and 0.55 t/ha for alkalinized dehydrated urine (0.2 spreader capacity per ha, 12 m³ of urine avoided at the WWTP).

To study the differences between different crops, scenarios were also considered for grain maize production (same functional unit). The technical itineraries are presented in Figure SM-6-1 and the total fertilizer doses for the different scenarios are presented in Table SM-6-1. The main differences were more intense tillage, less use of pesticides and a lower available nitrogen dose for the

conventional farming scenarios (160 kg N/ha). In the reference scenario, phosphorus was brought as a starter fertilizer (18-46-00, 32 kg P₂O₅/ha). The amount of phosphorus brought with UBFs was about the same and no other P fertilization was considered. Fertilizers were incorporated into the soil, which greatly limits ammonia volatilization and increase the mineral fertilizer equivalent. A volatilization reduction of 66% was considered due to incorporation of fertilizers into the soil (incorporation using tine machine (Webb et al. 2010). The spreading machinery was the same than for wheat except stored urine which was applied using a 15 m³ slurry injector.

2.2.7. Sensitivity analysis

After the study of the base scenarios described previously, a sensitivity analysis was carried out to study the impacts of variations of two parameters for the different scenarios.

- (i) Transport distance of UBFs could have a strong impact on energy consumption and contribution to climate change through the consumption of fossil fuels. One of the main differences between the different UBFs is nutrient concentration. For concentrated UBFs, impacts should be lower for transport (but higher during treatment). The basic assumption was 25 km between the district and the farm. Two other transport distance scenarios were studied. They consist respectively of a “peri-urban” case where the distance between the neighborhood and the farm was set at 10 km and a “center of the metropolis” case where the distance was set at 100 km.
- (ii) The treatment for alkalinized dehydrated urine has high energy consumption but the process in its present form is not optimized. The basic assumption was one drying unit per toilet. However, we could assume that the design of the apartments on the same floor of buildings in the neighborhood could be done in such a way that the toilets of the apartments (4 apartments per floor) are close to each other. Then all four toilets can be connected to the same drying unit which is already sized in its current form to handle that amount of urine. The impact of only a single drying unit per floor (1 unit for 4 toilets) was tested.

2.3. Life cycle impact assessment

For the life cycle impact assessment, the aim is to calculate impact indicators to quantify the environmental impacts. The impacts indicators calculated and the different methods used are presented in Table 6-3. The main method used was ILCD 2011 Midpoint+ which has been developed by the joint research center of the European commission (European Commission et al. 2012). It is considered as a reference method in European Union. For eutrophication, we used the CML method as it permits to combine the impacts of both N and P. Mid-point indicators assess the impacts in the middle of the

causality chain (e.g. potential acidification of ecosystems) which is less aggregated and more near to the physical and biological mechanisms (e.g. biodiversity losses due to acidification). The calculations were carried out using Simapro software version 8.5.4.0 (PRé Consultants 2017).

Table 6-3. Impacts categories calculated according to the method used.

| Category | Impact indicator | Unit | Method | Version | Reference |
|---|--|-------------------------------------|--------------------------|---------|--|
| Environmental impact at mid-point level | Climate change | kg CO ₂ eq | ILCD 2011 Midpoint+ | 1.10 | IPCC (2007) |
| | Acidification | mol H ⁺ eq | ILCD 2011 Midpoint+ | 1.10 | Seppälä et al. (2006); Posch et al. (2008) |
| | Particulate matter | kg PM 2.5 eq | ILCD 2011 Midpoint+ | 1.10 | Rabl & Spadaro (2004) |
| | Eutrophication | kg PO ₄ ³⁻ eq | CML IA baseline | 3.05 | Heijungs et al. (1992) |
| Resource and energy consumption | Water resource depletion | m ³ water eq | ILCD 2011 Midpoint+ | 1.10 | Frischknecht et al. (2006) |
| | Mineral, fossil and renewable resource depletion | kg Sb eq | ILCD 2011 Midpoint+ | 1.10 | van Oers et al. (2002) |
| | Cumulative energy demand | MJ | Cumulative energy demand | 1.10 | Frischknecht et al. (2007) |

(i) **Climate change** impact category permits to calculate the radiative forcing over a time horizon of 100 years through the emissions of greenhouse gases into the atmosphere. For agricultural systems, it mainly takes into account the emissions of carbon dioxide (CO₂), methane (CH₄) and nitrous oxide (N₂O).

(ii) **Acidification** impact category permits to quantify the acidification of the environment caused by gases emissions such as ammonia (NH₃), nitrous oxides (N₂O) and sulfur oxides (SO_x). These gases can deposit as dust or in dissolved form in rain (acid rain) and cause acidification of soil and surface water.

(iii) **Particulate matter** impact category permits to quantify the air pollution: the impact on premature deaths or disability by emissions of primary particulate matter (PM 2.5 and 10) and secondary particulate matter through the emissions of nitrogen oxides (NO_x), sulfur dioxide (SO₂), ammonia (NH₃), and carbon monoxide (CO).

(iv) **Eutrophication** impact category provides information on the pollution of water bodies by excess supply of nitrogen and phosphorus to water bodies, which can lead to excess growth of primary

production inducing an oxygen deficiency. Phosphorus is generally the limiting nutrient in fresh water and nitrogen in sea water.

(v) **Water resource depletion** quantifies the water consumption along the value chain (e.g. flushing toilets, manufacturing of mineral fertilizers).

(vi) **Mineral, fossil and renewable resources depletion** permits to quantify the resource consumption of the value chain. It takes mainly into account the consumption of electricity, fossil fuels and rare earths. The impact is correlated with the scarcity.

(vii) **Cumulative energy demand** quantifies the primary energy needed by the value chains. Both fossil-based and renewable energy consumptions are taken into account.

Other impacts categories such as ecotoxicological impacts may be of interest because of the possible presence of pharmaceutical residues and pathogens in UBFs. However, there is a lack of data and method to quantify these impacts finely. We thus did not include them in our study.

The results are presented by impact categories. For a clearer analysis, the impacts have been classified into six domains:

(i) **Reference fertilizers (Ref) production and fertilization** includes all impacts related to mineral fertilizers and chicken droppings: their production, their transport to the farm and their application. As in most cases their nutrient concentration is high and application impacts are low, we have grouped fertilizer production and application unlike for UBFs. Reference fertilizers are mainly used in the reference scenarios except for P fertilization for which mineral fertilizers is also used in the conventional UBF scenarios.

(ii) **UBF production** takes into account all the impacts from the toilet to the farm entrance gate: urine storage, treatment and transport.

(iii) **UBF fertilization** takes into account all the impact of UBFs at farm level: storage and application.

(iv) **Direct field emissions** include emissions to the atmosphere of nitrous oxide (N_2O), nitrogen oxides (NO_x) and ammonia (NH_3), from all the components of the systems (soil, mineral fertilizers, UBFs). It also includes emissions of nitrogen and phosphorus in water bodies.

(v) **Other field operations** group together the impacts linked to crop protection (pesticides and their application), tillage, sowing and harvesting.

(vi) **Avoided impacts for the sanitation system** represent the consumption and impact avoided because the urine was not treated in the WWTP but spread on the crops. It includes the consumption of water, energy, chemical (methanol and $FeCl_3$) and emissions to the atmosphere (NH_3 , N_2O) and to water bodies (N and P).

All results are expressed using the functional unit: the production of 1 kg of grain (at 15% moisture).

3. Results and discussion

3.1. Results by impact categories

3.1.1. Climate change

The impacts of the different scenarios for the climate change category are shown in Figure 6-4.

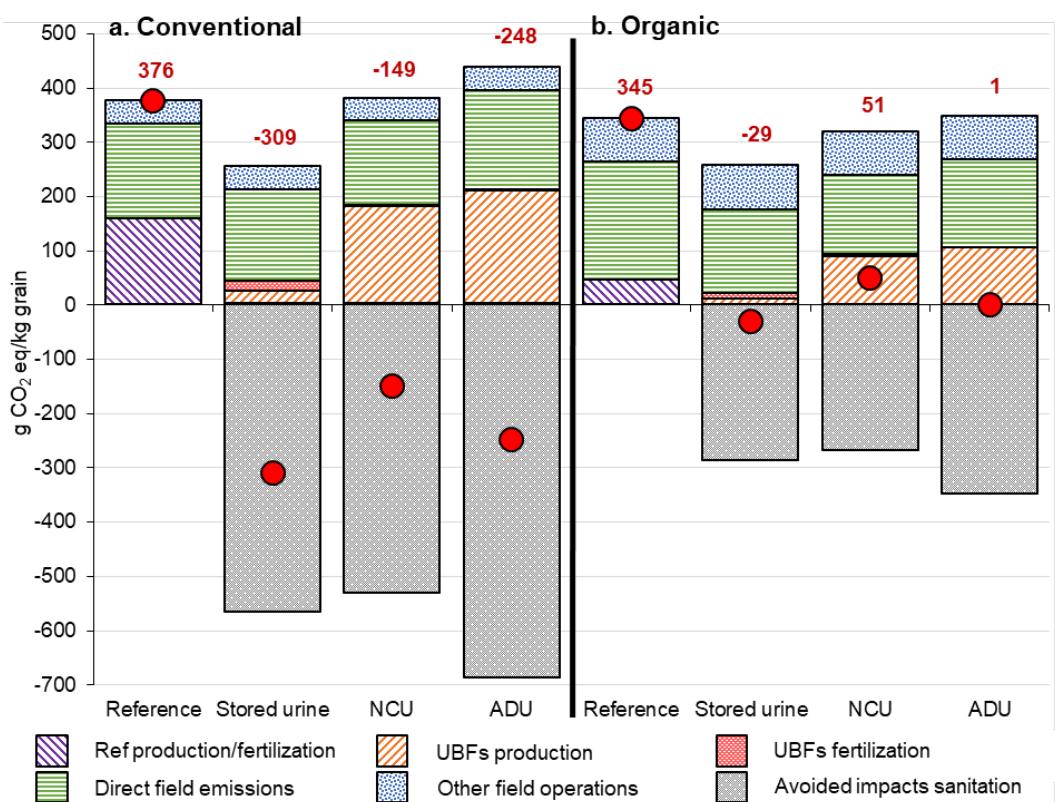


Figure 6-4. Climate change impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the number above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

In conventional agriculture, the climate change impact is 376 g CO₂ eq/kg grain in the reference scenario and from -309 to 149 g CO₂ eq/kg grain for UBFs value chains. For the reference scenario, the production of mineral fertilizers is the main contributor. The impact of the treatments for nitrified concentrated urine (NCU) and alkalinized dehydrated urine (ADU) is somewhat higher than the production of mineral fertilizer due to the high consumption of electricity and to a lesser extent due to

N_2O emissions during the treatment for NCU and lime consumption for the ADU. For stored urine, despite high transport needs, the impact is minor as well as the impact related to its application. The impacts of transport and application are negligible for NCU and ADU. Direct field emissions (mainly N_2O emissions) contribute for a large part of climate change impact and the other field operations have a minor contribution. The avoided impacts at the WWTP represent a significant part in the total/final impact indicator value due to the avoided high N_2O emissions, and offset the impacts of UBF value chains. Thus, the balance of the UBF scenarios is negative and lower than in the reference scenario.

In organic agriculture, the climate change impact of the reference scenario is 345 g CO_2 eq/kg grain and range from -29 to 51 g CO_2 eq/kg grain for UBFs value chains. The impact of chicken dropping transport is about the same order of magnitude that the treatment of NCU and ADU. The impacts of other field operations cannot be considered as negligible due to the increased tillage and associated diesel consumption. The avoided impacts are lower than in conventional agriculture due to a lower application rate, resulting in a positive balance for NCU and ADU. However, the impacts of organic UBFs scenarios remain lower than in the reference organic scenario.

3.1.2. Acidification

The impacts of the different scenarios for the acidification category are presented in Figure 6-5. In conventional agriculture, the impacts of the reference scenario are 7.4 mmol H^+ /kg grain and range from 1.5 to 24.5 mmol H^+ /kg grain for the UBF value chains. The main contributors are the direct field emissions and especially the ammonia volatilization (15% at the field level for the ADU). The impacts of the fertilizers production are negligible for stored urine and NCU (low NH_3 losses during the treatment) and low for the manufacture of mineral fertilizers. The production of ADU is a very important contributor due to the volatilization of ammonia during the treatment (10% of total N). The avoided impacts at the WWTP are low. The impacts related to the application and other field operations are minor. Compared to the reference scenario, the balance for stored urine is similar and about 5 times lower for NCU. However, the impact is approximately 3.6 times higher for ADU compared to the reference scenario.

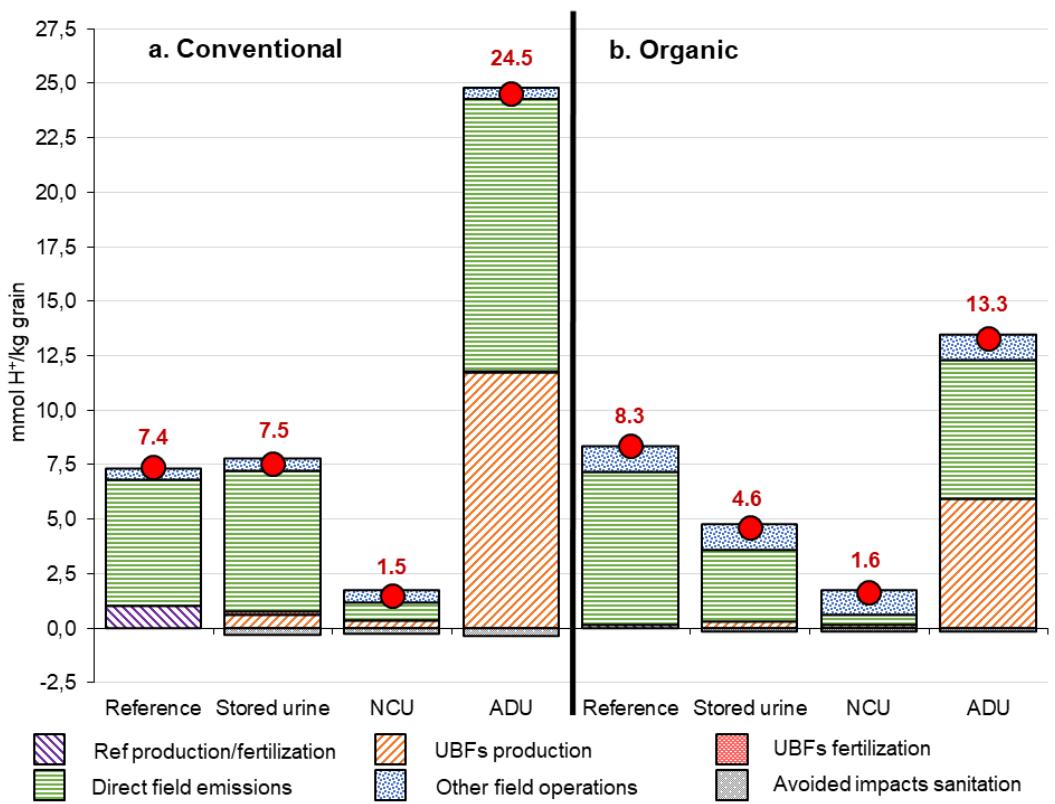


Figure 6-5. Acidification impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

For organic farming scenarios, the impacts in the reference scenario are 8.3 mmol H⁺/kg grain and range from 1.6 to 13.3 mmol H⁺/kg grain for the UBF value chains. The impact of the reference scenario is higher than for stored urine and NCU because the ammonia volatilization rate of chicken dropping is higher compared to stored urine and NCU. However, it is lower compared to ADU.

3.2.3. Particulate matter

The impacts of the different scenarios for the particulate matter category are shown in Figure 6-6.

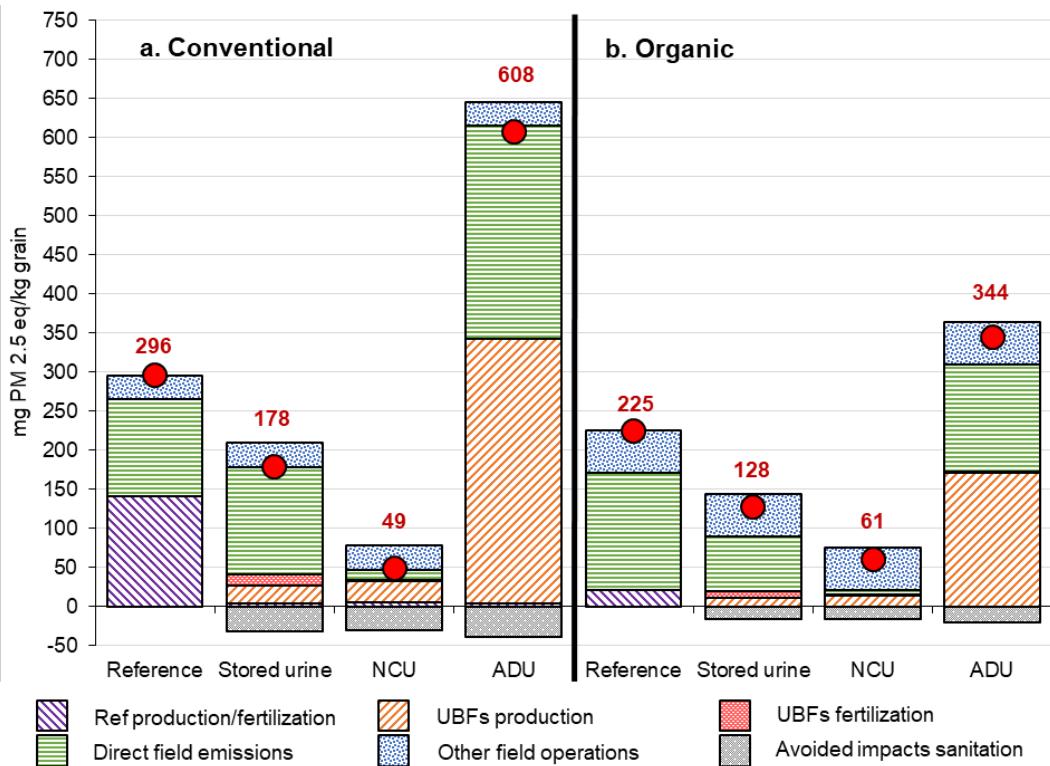


Figure 6-6. Particulate matter impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

In conventional farming, the impacts of the reference scenario are 296 mg PM 2.5 eq/kg grain and range from 49 to 608 mg PM 2.5 eq/kg grain for the UBF value chains. As for acidification, the impacts are mainly attributable to the volatilization of ammonia (secondary particles). However, in the reference scenarios, the production of mineral fertilizers represents about half of the impacts (mainly due to the chemical and the energy needed for their manufacture). In the stored urine scenario, the impact of the application is small but not negligible. The avoided impacts at the WWTP are low. The main contributor is the avoided production of FeCl₃. For stored urine, the transport to the farm and the ammonia emissions at the neighborhood level account for most of the impacts of the UBF production category. Most of the particles for NCU production come from the electricity consumed during processing. In the ADU scenario, the high impacts are related to UBF production (electricity but also NH₃ volatilization during treatment) and direct field emission.

In the organic farming scenarios, the impacts in the reference scenario are 255 mg PM 2.5 eq/kg grain and ranged from 61 to 344 mg PM 2.5 eq/kg grain for UBF value chains. A significant part of the impacts in the reference scenario is due to the transport to the farm of chicken dropping.

In both farming systems, the balance for stored urine and NCU is lower than the reference scenario but higher for ADU.

3.2.4. Eutrophication

The impacts of the different scenarios for the eutrophication category are presented in Figure 6-7.

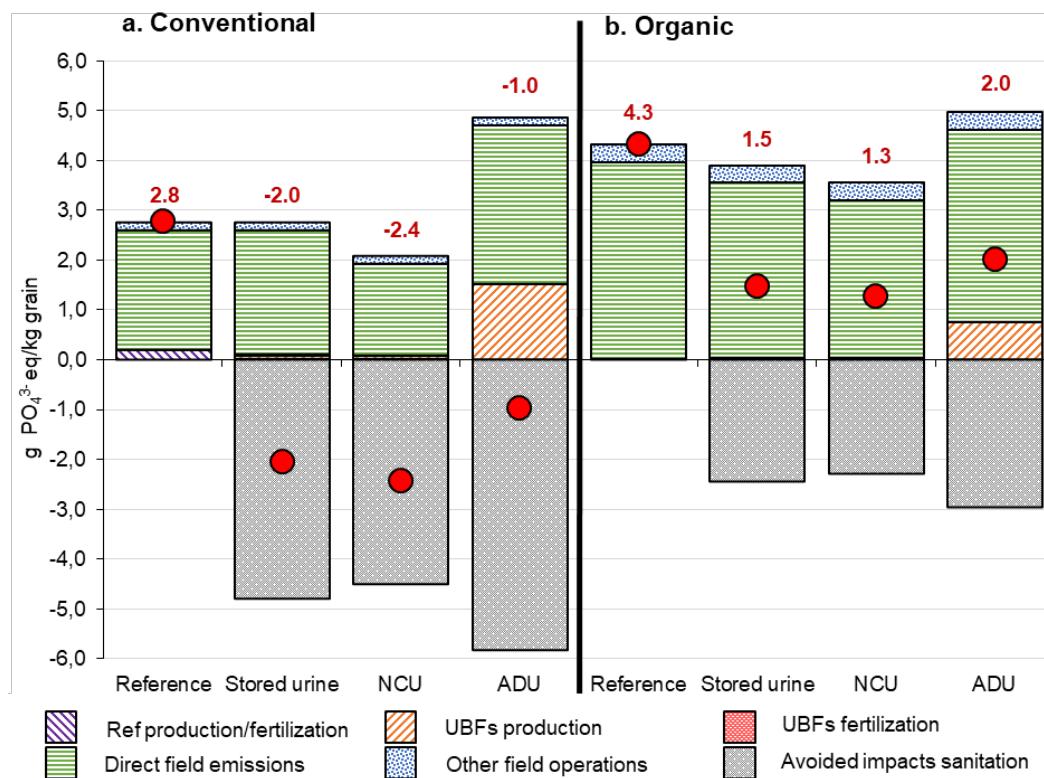


Figure 6-7. Eutrophication impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

In conventional agriculture, the impacts are 2.8 g PO₄³⁻ eq/kg grain for the reference scenarios and range from -2.0 to 1.0 g PO₄³⁻ eq/kg grain for UBFs value chains. In the organic farming scenarios, the impacts are 4.3 g PO₄³⁻ eq/kg grain in the reference scenario with chicken dropping and 1.3 to 2.0 g PO₄³⁻ eq/kg grain in the scenarios with UBF value chains. Eutrophication is mainly due to field direct emissions. The models used to simulate nutrient emissions to water bodies do not depend on the amount of nitrogen applied but are based on the time between sowing and harvesting and soil cover in winter. These parameters being equal in all scenarios, direct field emissions linked to emissions of N and P to water bodies are equal in all scenarios for organic and conventional farming systems. Part of the direct emissions is however related to ammonia volatilization, so the impact is greater for ADU and the chicken dropping. The impacts of the treatments are low except for ADU. The N and P avoided emissions at the WWTP are high and make it possible to offset the impacts of the value chain. Thus, the impacts of all UBF scenarios are lower than in the reference scenario and are negative for the conventional systems with UBFs. Indeed, the separate collection of urine avoids the impacts

towards fresh water in the WWTP (904 kg of N and 32 kg of P per year, respectively about 0.9 kg N and 32 g P/cap/y). The impacts from other sources are minor. The balance remains positive in organic farming due to the lower yield (4.5 t/ha vs 8.2 t/ha) and the lower dose applied (50 kg N/ha in conventional vs 180 kg N/ha). It is also due to the limit of the model used for nitrite leaching which may overestimate leaching in organic agriculture.

3.2.5. Water resource depletion

The impacts of the different scenarios for the water resource depletion category are shown in Figure 6-8.

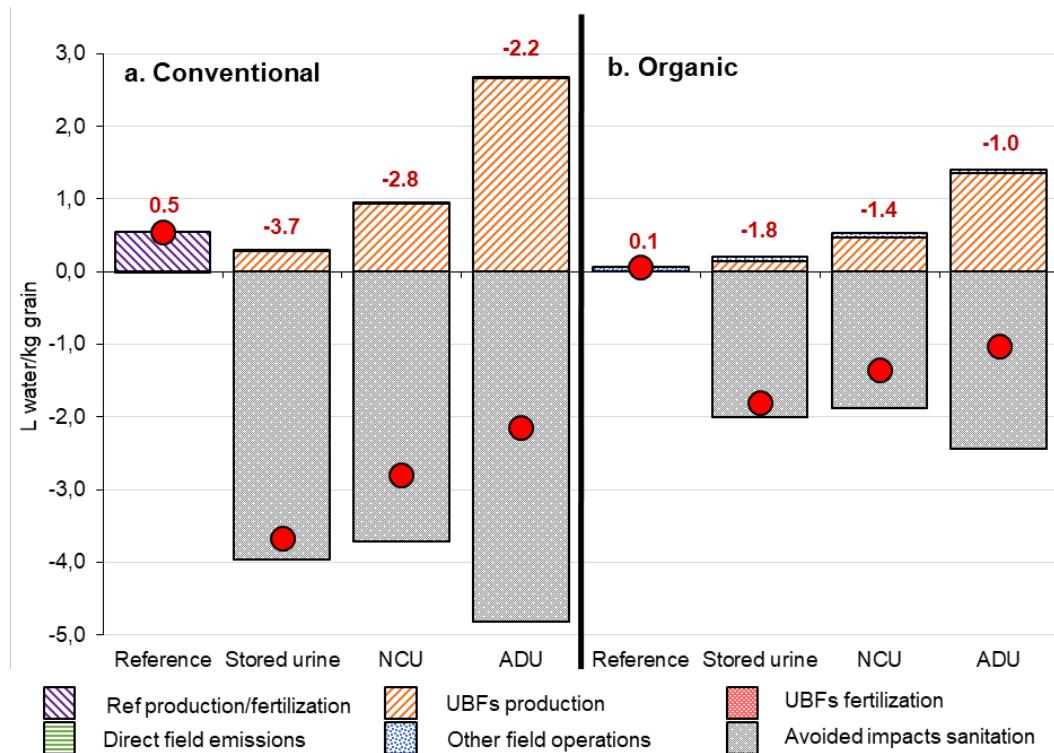


Figure 6-8. Water resource depletion impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between water consumed by the system due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine at WWTP. ADU: alkalinized dehydrated urine.

In conventional farming, the water consumption for the reference scenario is 0.5 L/kg grain and ranges from -3.7 to -2.2 L/kg grain for UBF value chains. In organic agriculture, the water consumption is 0.1 L/kg grain in the reference scenario with chicken dropping and from -1.8 to -1.0 L/kg grain for UBFs value chains. Some water is used for the production of mineral fertilizers and source separation at the toilet level (0.1 L per flushing). The differences between UBFs value chains are mainly due to the different amount of fertilizers used (due to their different mineral fertilizer equivalents etc.) and the electricity consumption for the treatment. However, the avoided impacts for

the sanitation system largely outweigh the positive consumption of the value chains. Thus, all UBFs scenarios have negative balance and save water compared to the reference scenarios. Indeed, with our hypothesis (1.5 L for flushing in diverting toilet versus 3 L in conventional toilets), approximately 2570 m³ per year are saved at the district level (about 2.5 m³ per year and per person and 8.4 L of water per L of urine collected).

3.5.6. Mineral, fossil and renewable resources depletion

The impacts of the different scenarios for the mineral, fossil and renewable resources depletion category are presented in Figure 6-9.

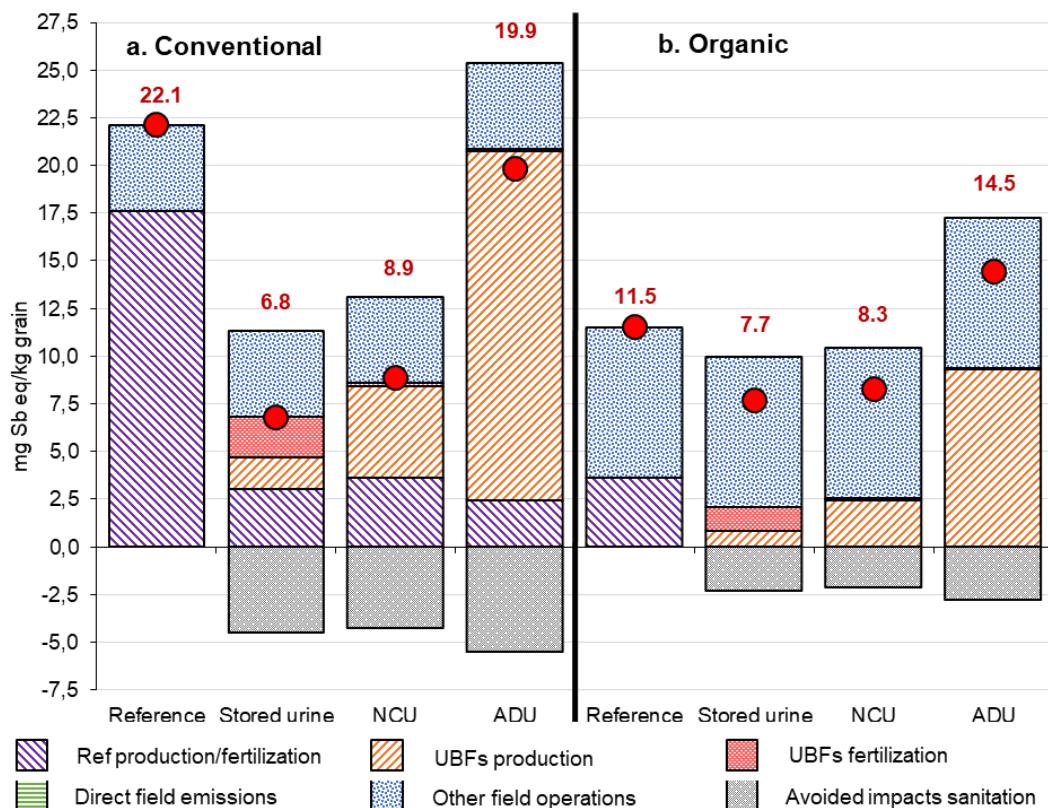


Figure 6-9. Resource depletion impact for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

In conventional agriculture, the resource consumption for the reference scenario is 22.1 mg Sb eq/kg grain and ranges from 6.8 to 19.9 mg Sb eq/kg grain for UBFs value chains. The use of phosphate fertilizer (in the fertilizer production category) is the main contributor (about two thirds) in the reference scenario, the remainder representing the production of mineral nitrogen fertilizers. P mineral fertilizers impact is also significant for the UBF scenarios. The impact of the production of stored urine is mainly due to its transport from the district to the farm. Most impacts for NCU and

ADU production relate to the electricity consumption during the treatment. The impact of UBF application is significant only for stored urine (diesel consumption by slurry spreader). For other field operations, most of the impacts are related to the materials used in the agricultural machinery (e.g. tractor, combine harvester) and some of the pesticides used. The avoided impacts for the sanitation system are mainly linked to the avoided consumption of iron chloride and to a lesser extent of methanol and water. The consumptions of the stored urine and NCU scenarios are about 3 times lower than in the reference scenario. The impacts of ADU remained higher compared to the reference scenario.

In organic agriculture, the consumption in the reference scenario is 11.5 mg Sb eq/kg grain and ranges from 7.7 to 14.5 mg Sb eq/kg grain for UBF value chains. The results are similar to those of conventional farming except that there is neither mineral P fertilization nor use of pesticides. The resource consumption of the reference scenario is due to the transport of the breeding of laying hens to the farm. The other field operation impacts are higher due to the increased use of agricultural machinery for tillage. All UBFs scenarios are lower than the reference system except ADU. However, the reference scenario in organic farming is much lower than that of conventional farming.

3.5.7. Cumulative energy demand

The impacts of the different scenarios for the cumulative energy demand are shown in Figure 6-10.

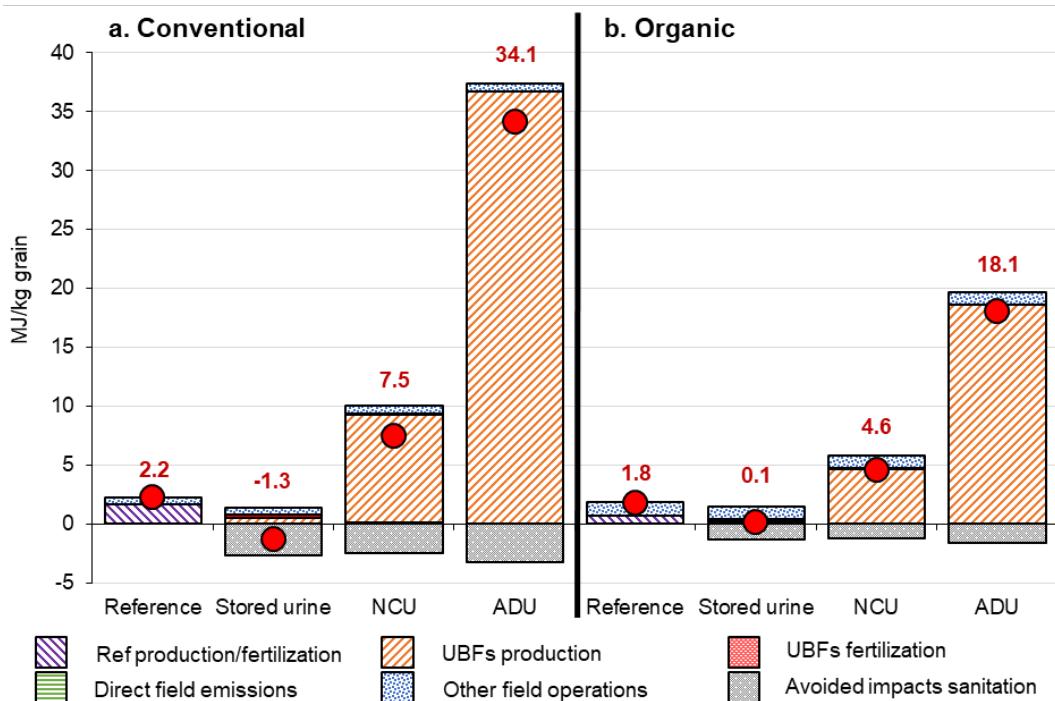


Figure 6-10. Cumulative energy demand for the different scenarios in conventional (a) and organic (b) agriculture. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

For conventional farming scenarios, the cumulative energy demand is 2.2 MJ/kg grain in the reference scenario and ranges from -1.3 to 34.1 MJ/kg grain for UBF value chains. For the reference scenario, energy consumption is mainly linked to the production of mineral fertilizers. The energy consumption for the transport of fertilizers and their application and for the other field operations is very low. The energy consumption of the UBF value chains is mainly driven by the electricity consumption of the treatment and is high for NCU and ADU. The avoided impacts of the WWTP are low. Most of the avoided impacts are related to the avoided production of methanol. With the exception of stored urine for which no energy is required for the treatment, the energy consumption of the other UBFs value chains are above the reference scenario. The energy balance of the stored urine scenario is negative because of the avoided impacts at the WWTP.

In organic farming, the cumulative energy demand is 1.8 MJ/kg grain for the reference scenario and ranges from 0.1 to 18.1 MJ/kg grain for UBF value chains. The results are similar to those of conventional agriculture. The energy consumption for chicken dropping is linked to their transport to the farm.

3.2. Maize production

The results for the different impact categories are shown in Figures SM-2 through SM-8. In the conventional scenarios, the impacts linked to the fertilizer dose are lower because the nitrogen input is 160 kg N/ha instead of 180 kg N/ha for wheat production. In addition, as the yield of maize is higher than that of wheat (9.9 T/ha for the conventional farming scenarios and 7.0 T/ha for the organic farming scenarios), the impacts expressed in kg of grain are lower. For some of the impact categories (e.g. cumulative energy demand), the balance is more favorable for maize production than for wheat production because of these two factors (e.g. -25% for NCU or -33% for ADU for the cumulative energy demand in conventional farming).

One of the main differences with the wheat production is that all fertilizers can be applied before sowing. Thus, it is much easier to manage the volatilization of ammonia at the field level by incorporation into the soil. All impact categories strongly linked to ammonia volatilization (e.g. acidification, particulate matter) have a better balance compared to wheat (-68% reduction of the acidification impact on maize compare to wheat for stored urine). For the specific case of ADU, the main contributor becomes the volatilization occurring during the treatment.

As no P fertilization is added for the maize production (except in the reference scenarios in conventional farming), impact categories such as mineral, fossil and resource depletion are lower than for wheat. More frequent tillage occurs in the maize scenario and involves greater use of agricultural machinery and increase diesel consumption. Thus, in most cases, the other field operations impacts are higher compare to the wheat scenarios (despite a lower use of pesticides). The production of maize

seeds intended for sowing has a significant impact on the water resource depletion. The impacts of the injection of stored urine increase for different impact categories (e.g. climate change, particulate matter) due to higher diesel consumption and greater use of agricultural machinery. For the mineral, fossil and renewable resources consumption, impact of stored urine is higher than for NCU unlike wheat due to the use of the injector (e.g. for stored urine, +234% on maize compare to wheat for the UBFs fertilization category for the mineral, fossil and renewable resources consumption).

As the production time of maize is longer than for wheat and as maize is a plant with little ground cover, there is more erosion of P and more eutrophication impact. There is also more N leaching for organic maize due to the absence of intercropping culture after maize harvest. In addition, as the fertilizer dose is lower than for wheat, the avoided impacts for the sanitation system are lower compared to wheat and may result in a less favorable balance (e.g. eutrophication, climate change).

3.3. Sensitivity analyzes

The impacts of the sensitivity analyzes for the different impact categories are presented in Figure 6-11.

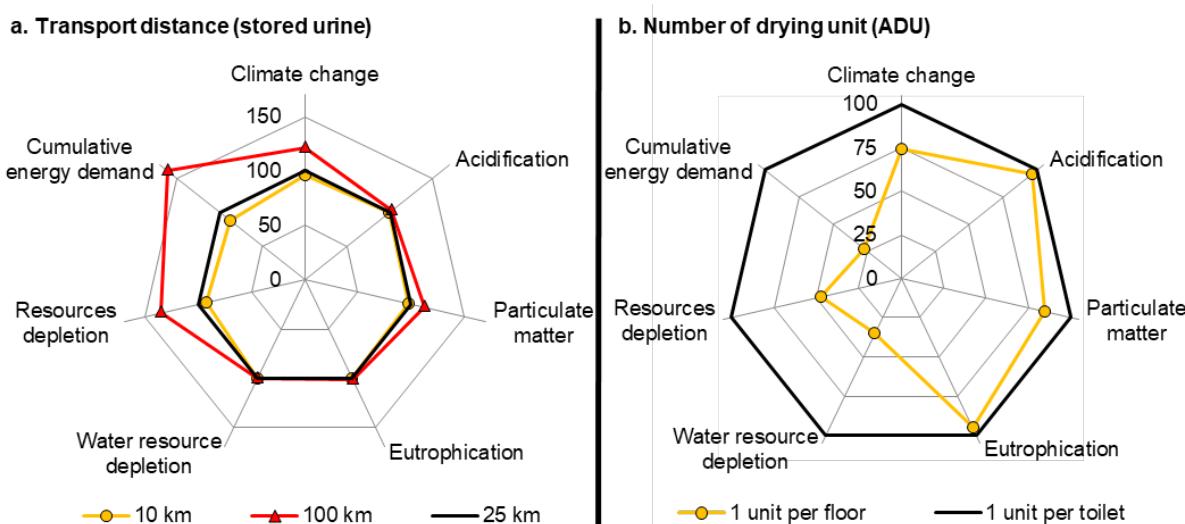


Figure 6-11. Results for the sensitivity analyzes carried out: a. Relative impact compared to the main scenario (25 km, base 100) for the transport of stored urine; b. Relative impact of the number of drying units for ADU (alkalinized dehydrated urine).compared to the main scenario (1 drying unit per toilet, base 100). Only positive impacts (without the avoided impact of sanitation) were taken into account for the calculations.

3.3.1. Transport distance

Among the different UBFs, the effect of the transport distance is high only for stored urine (Figure 6-11). In addition, the differences are especially significant for the 100 km transport on the following impact categories: cumulative energy demand (+64%), mineral, fossil and renewable resources depletion (+37%), climate change (21%) and to a lesser extent particulate matter (+12%). The impact

on acidification, eutrophication and particulate matter are very low as they are not linked to the transport. For the 10 km scenario, the relative impact compared to the reference scenario decreases by 13% for the cumulative energy demand but decreases by less than 10% for the other impact categories. Even if the impact for climate change, for example, is higher of 21% for the 100 km scenario, the overall impacts considering the entire value chain are limited as shown in Figure SM-6-9. For this impact category, the direct field emissions remain the main contributor. For NCU and ADU, the impact of the transport is very limited and not significant. The decrease from 25 to 10 km reduces the impact to a maximum of 1%, while the 100 km scenario increases the mineral, fossil and renewable resource depletion impact category to a maximum of 7% for NCU (5% for ADU).

3.3.2. Number of drying units

The energy consumption for ADU is high, but the process is not fully optimized, and the energy consumption can be decreased at the level of the residential building. The relative impact of the number of drying units is illustrated in Figure SM-6-3 and SM-6-4. The drying unit is already dimensioned to treat 4 times more urine than in the basic scenario. Thus, the installation of a single drying unit per floor (instead of 4) makes it possible to reduce about 66% of the water resource and mineral, fossil and renewable resources depletion and 75% of the cumulative energy demand (from 38 to 10 MJ/kg grain). The impact on climate change is also significant but not major with a reduction of 25% as most of the electricity comes from nuclear plant. The reduction of the impact is significant as shown for the mineral, fossil and renewable resources depletion in Figure SM-6-3 for which ADU with only one drying unit per floor is the least impacting UBFs scenario. The other impact categories depend mainly on volatilization and are little affected by the reduction in the number of drying units. As shown for the cumulative energy demand in Figure SM-6-4., by using only one drying unit per floor, it can reduce energy consumption up to the same amount as the NCU. However, energy consumption is still higher than the reference or stored urine scenarios.

3.4. Discussion

Comparison between UBFs value chains: Some of the mid-point environmental impacts are directly related to the volatilization of ammonia: acidification, particulate matter and eutrophication. For these impact categories, the impact of the NCU value chain is lower than that of other UBFs due to the low volatilization potential. Thus, stored urine and in particular ADU have higher impacts due to higher volatilization at the field level and also at the treatment level for ADU.

For the climate change impact category, the direct field emissions are a major contributor and are similar for UBFs value chains. However, part of the impact on climate change is mainly related to the treatment and is higher for NCU and ADU than for stored urine due to a high consumption of electricity. Finally, for the impact categories related to the consumption of resources (water, mineral,

fossil and renewable, and energy demand), stored urine is also the value chain with the least impact compared to the NCU and especially the ADU because of their high energy consumption.

Comparison with reference scenarios: The impacts of all UBFs value chains are much lower than the reference scenarios for climate change, eutrophication and water resource depletion due to the high avoided impacts for the sanitation system. For mineral, fossil and renewable resource consumption, the impact of stored urine and NCU value chains is lower than the reference scenarios. However, the impact is greater for ADU due to the high electricity consumption. This is also the case for impact categories related to ammonia volatilization (in particular acidification and particulate matter) for which the impact of stored urine and NCU value chains are similar or lower to the reference scenarios but higher for the ADU value chain. Finally, for the cumulative energy demand, only the impact of the stored urine scenario is lower than the reference scenarios, the others are higher due to the high electricity consumption for the treatment. Even without taking into account the avoided impacts WWTP, in most cases, the impacts for stored urine or NCU are similar or lower than the reference scenario.

Organic farming: It is difficult to directly compare the conventional or organic system because LCA hardly takes into account certain positive aspects of organic agriculture such as the impact on biodiversity (van der Werf et al. 2020). In our scenarios, the hierarchy between the UBF impacts is essentially the same as that in conventional agriculture. However, as the yield is lower, the impacts not directly related to the fertilizer dose (e.g. eutrophication) are greater than in conventional agriculture. The impacts associated to ammonia volatilization or energy consumption are lower compared to conventional farming due to the lower fertilizer dose. We can highlight that due to the lower fertilizer dose, the avoided impacts for the sanitation system are much lower. For most of the impact categories, UBF value chain impacts were lower or similar compared to the reference scenario. However, UBFs are interesting fertilizers that could potentially be used in organic farming and can be an important source of nitrogen and phosphorus for organic systems compared.

Environmental hotspots: Most of the mid-point environmental impacts from value chains are related to lower field direct emissions and to a lesser extent to lower impacts from treatment in the case of high energy consumption or ammonia volatilization during the process. However, for the impact categories based on the resource consumption, the main contributors are for most of them at the treatment level. Our study highlights the sanitation system as a hotspot. The use of UBF in agricultural systems helps to reduce their impacts. For UBFs value chains, the ammonia volatilization (during the treatment or at the field level) and the energy consumption during the treatment appear to be very decisive for the environmental impacts of value chains. These two points should be given special attention for the development of future UBF value chains. The impacts related to the transport of UBFs, their application or the necessary infrastructures such as storage tanks seem to have a limited

environmental impact. However, for stored urine, since energy consumption of the treatment is limited, their impact may be in proportion more significant at the level of the value chain.

LCA makes it possible to assess environmental impacts based on current research knowledge. However, the strong assumptions with a global change are more difficult to model and the comparison of the new value chains is made with large scale and optimized reference systems. As UBFs value chains are not already developed, the assumption and data used may differ in the implementation of real case. In addition, the treatments studied are still evolving and not fully optimized in their current state of development and their environmental impacts may be more or less easily reduced. For example, other setups for the treatment can be designed such as transporting stored urine in a nitrification plant which can save energy due to a scaling effect. Thus, the aim of this study was not to perform a detailed LCA of these value chains but to assess the global trends and the environmental hotspots of these UBFs value chains. Furthermore, some of the mentioned issues such as eutrophication or volatilization can be managed independently of UBF value chains at the field level (e.g. nitrogen catch crops, spreading techniques).

The comparison with the literature is not always easy because the system boundaries are not completely the same as well as the used method to assess the environmental impacts. For stored urine, the results of our study are in line with previous studies. With similar boundaries, Tidaker et al. (2007), Malila et al. (2019) and Medeiros et al. (2020) observed a reduction in eutrophication but an increased acidification potential due to ammonia volatilization compared to the reference scenarios studied (mineral fertilizers and WWTP or skeptic tank). In other studies, the impact of stored urine value chains versus mineral fertilizer use on climate change was not so important because the hypothesis on greenhouse gases emissions at the WWTP was lower or not taken into account (Tidaker et al. 2007; Spångberg et al. 2014). N₂O emissions at the WWTP level are an environmental hotspot to be taken into account for the assessment of new value chains (Bisinella de Faria et al. 2015). Water saving at the collection level has also been highlighted by Jönsson (2002). The energy consumption of value chains strongly depends on the boundaries of the study. The stored urine value chain usually shows lower energy consumption than using mineral fertilizer and urine treatment in the WWTP (Jönsson 2002; Tidaker et al. 2007; Remy and Jekel 2008).

Nitrogen fertilizers represent an important part of the carbon footprints of farms (Yan et al. 2015). The impact on climate change for grain production that we observed were similar to previous studies with a high impact of the direct field emissions (Autret et al. 2019). Field emissions have been shown to be a determining factor in the environmental impact of the use of organic residues in agriculture (Brockmann et al. 2014). Compared to the use of sewage sludge in agriculture, the source separation of urine appears to have a better environmental balance. In addition to the limited recovery of N and P, sewage sludge has a high impact on human toxicity and eco-toxicity impact categories (Yoshida et al. 2018). We did not consider this impact but UBFs concentration in trace metal elements is low (Martin

et al. in preparation; Ronteltap et al. 2007). Most pathogens comes from fecal cross-contamination that can occur in diverting toilet (Höglund et al. 1998) but proper storage inactivates them to acceptable levels (WHO 2012). Besson (2020) observed that urine source separation and its agricultural valorization showed a lower impact than the recovery of nutrient in a highly optimized WWTP. For other organic waste treatments such as pig manure treatment, the electricity consumption of the treatment also appears as an hotspot (Corbala-Robles et al. 2018).

The LCA methodology is based on technical criteria and focuses only on the environmental impacts. However, many other criteria must be taken into account for the implementation of UBFs value chains. For example, stored urine appears to be the least impacting value chain for many impact categories. However, since it is a non-concentrated UBF, the volume to applied is substantial compared to concentrated UBFs (e.g. 36.2 t/ha vs 1.98 t/ha for ADU). Thus, it involves much more work for the fertilization step at farm level and it may not be suitable for the cultural systems currently in place in the study region as well as the collection in densely populated areas due to lack of space. Other aspects such as the odors of UBFs, the acceptability of their use by farmers and consumers (e.g. Lienert and Larsen 2010; Segré Cohen et al. 2020) or the contaminant content are also important to consider. For example, pharmaceutical residues are of concern with concentrations in urine from zero to a few mg/L for individuals under medication (Lienert et al. 2007; Bischel et al. 2015). The need for urine specific treatment to remove pharmaceuticals prior to application is debated (Winkler et al. 2009; WHO 2012). However, adapted indicators are needed to quantify the risk.

The recovery of all the nitrogen and phosphorus of human origin currently not recycled would make it possible to offset respectively 16%-21% and 9-12% of global inorganic inputs of nitrogen and phosphorus in agriculture (Trimmer et al. 2019). However, it depends of the geographical conditions and of the farming system. For example, in the Paris region (12 million inhabitants, 569 000 ha of arable lands cultivated mainly with an intensive cropping system), the nutrients present in the urine of the inhabitants of the metropolis could cover the nitrogen and phosphorus currently applied in fields (calculated from Esculier et al. 2018 and UNIFA 2018). In less intensive systems such as organic agriculture, coverage rates may be higher even in less densely populated areas because the required fertilization inputs are lower.

Finally, other UBFs treatment such as urine acidification (Hellström et al. 1999) should be investigated as they may have a different impact at the treatment level or at the field level (e.g. less ammonia volatilization).

4. Conclusion

We assessed the environmental impacts of producing wheat with three different urine-based fertilizers using the LCA methodology compared to the current system (use of mineral fertilizers in conventional agriculture or chicken dropping in organic agriculture and treatment of urine in WWTP). The impacts of all urine-based fertilizer value chains were lower than the reference system for the climate change, eutrophication, fossil resource and water consumption impact categories. Besides the avoided emissions linked to the avoided fertilizers, we underlined that the avoided impacts for the sanitation system (water consumption, greenhouse gas emissions, and nutrient discharge to the river) were significant and permitted to greatly reduce the environmental impacts of urine-based fertilizer compared to the reference scenarios. On the contrary, some impacts were higher for some UBF value chains as for acidification, particulate matter emissions and the cumulative energy demand. Acidification and particulate matter emissions are directly related to ammonia volatilization which may be higher than with the reference fertilizers for stored and alkalinized dehydrated urine. Electricity consumption for nitrified concentrated urine and alkalinized dehydrated urine is high. Thus, ammonia volatilization and energy consumption of the treatment have been identified as the main environmental hot spots of urine-based fertilizer value chains.

Urine-based fertilizers are efficient fertilizers derived from a resource currently largely released into the environment. They contribute to close the biogeochemical cycles and they permit to reduce the impacts of agricultural and sanitation systems on the environment. More research is needed to reduce the energy consumption of the treatments and the potential ammonia volatilization of urine-based fertilizers. In addition to environmental impacts, other aspects such as the organization of value chains, technical constraints linked to the urban context and the agricultural system, or the acceptability of the use of urine-based fertilizers by farmers and consumers must be taken into account.

5. Perspectives

To complete the study, a sensitivity analysis extended to several high-impact parameters seems to be of interest. The first is the type of WWTP for the avoided impacts during the sanitation system. Indeed, the parameters of WWTP chosen in this study had a significant impact, in particular on climate change due to the high rate of N₂O emissions. Thus, in the case of a smaller WWTP with activated sludge treatment should be studied with N₂O emissions rate that could be reduced up to 20 times less than that used in this study (0.1% N₂O emissions instead of 2.1%). Another interesting parameter to look at is the electricity production mix. Since the French energy mix is mainly based on nuclear energy, the impact of electricity consumption on climate change is limited. The use of the

average European mix for electricity production could be interesting to open our study on a more general case.

Some parameters and hypothesis can be improved to have a clearer vision of the impacts of the different value chains. Ammonia volatilization from UBFs may be updated using modelling and considering different application techniques. Other aspects may be of interest to take into account such as the impacts on yield or soil compaction due to heavy spreading machinery. For the reference scenario in organic farming, certain impacts directly linked to the agricultural valorization of chicken dropping, such as the drying of chicken dropping, must be taken into account.

The weight of the impacts and the uncertainties of the different parameters used in this study may be further studied with a Monte Carlo simulation. Finally, a step of normalization of the results could be interesting. The designed scenarios are marginal, but the hypothesis can be generalized to a systemic system to compare the impact of UBFs value chains to regional or national emissions for the different impact categories. For example, does a higher energy consumption of the UBFs value chains significantly impact the global energy consumption at the regional scale ?

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Supplementary Material

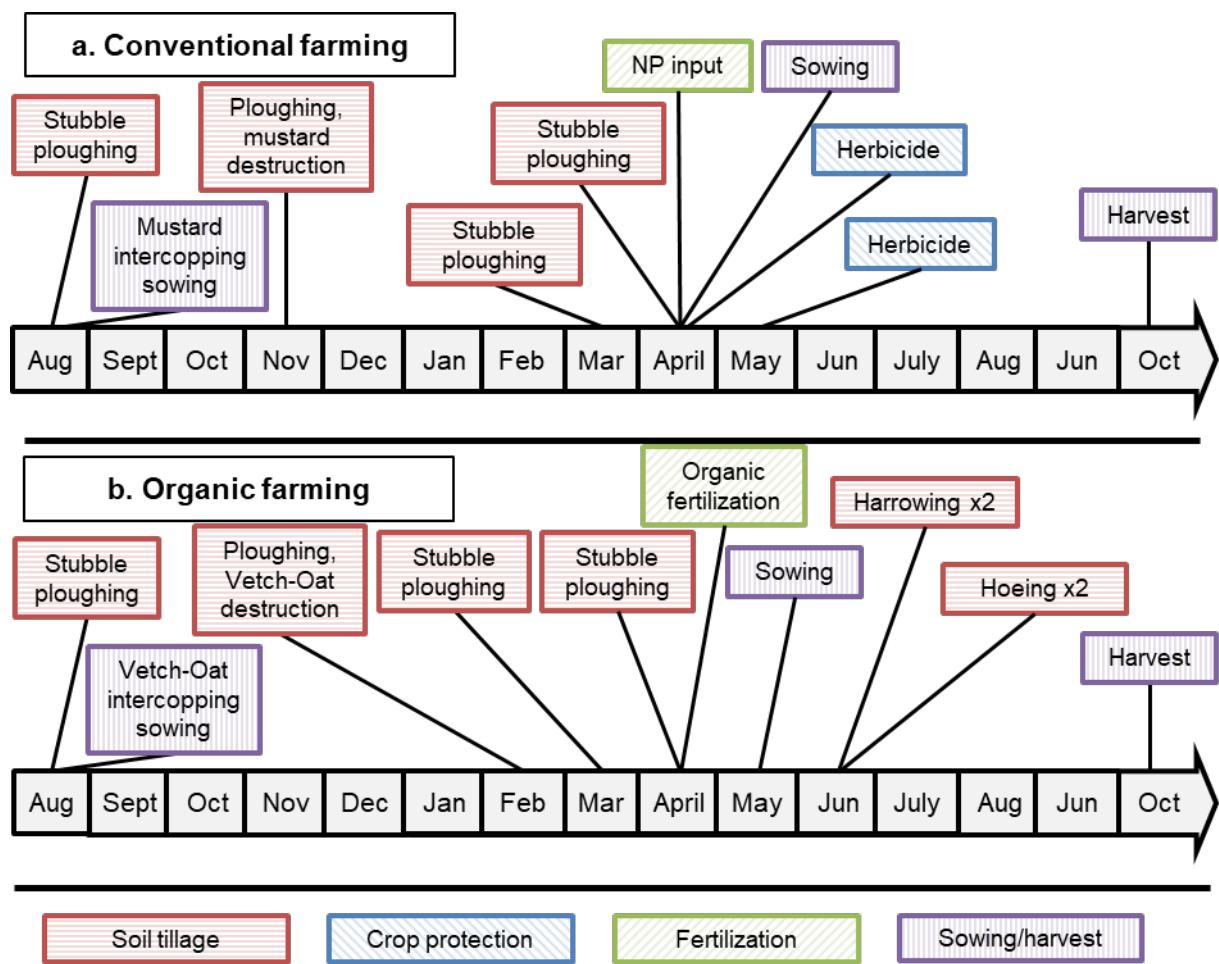


Figure SM-6-1. Technical itineraries for maize in conventional farming (a.) and organic farming (b.)

The yield was set at 9.9 t /ha (99 qt/ha at 15% moisture) in conventional farming and 7.0 t DM/ha (70 qt/ha at 15% moisture) in organic farming. The previous crop was wheat and an intercropping was sown between wheat and maize: Mustard in conventional farming and Vetch-Oat in organic farming.

Table SI-6-1. Nitrogen, phosphorus concentration, nitrogen mineral fertilizer equivalent and quantity applied for the fertilizers tested in conventional and organic farming for the maize production. Nitrogen doses for the different input correspond to the available nitrogen considering the mineral fertilizer equivalent.

| Fertilizer | Nitrogen concentration (g N/kg) | Phosphorus concentration (g P ₂ O ₅ /kg) | Ammonia volatilization at application (% of total N) | Nitrogen Mineral fertilizer equivalent (%) | Conventional farming | Organic farming |
|--|---|--|---|--|--------------------------------------|-------------------------------------|
| | | | | | Total input 160 kg N/ha (t/ha) | Total input 50 kg N/ha (t/ha) |
| Mineral fertilizer | 18-46-00 | 180 | 460 | 0 | 100 | 0.07 |
| | Nitrogen solution | 300 | 0 | 3 | 100 | 0.49 |
| Urine- based fertilizer | Stored urine | 5.35 | 1.0 | 2 | 98 | 30.5 |
| | Nitrified concentrated urine | 3.97 | 9.1 | 0.1 | 99.9 | 3.4 |
| Organic fertilizer | Alkalinized dehydrated urine | 106.99 | 20.7 | 4 | 96 | 1.56 |
| | Chicken droppings | 38.2 | 37.9 | 2 | 50 | 2.62 |

Since fertilizers were incorporated into the soil, ammonia volatilization was lower and the mineral fertilizer equivalents were higher than for wheat. With the exception of the stored urine applied using a 15 m³ slurry injector, the spreading machinery was the same as for wheat. 66% volatilization reduction was considered for the reference scenarios due to incorporation of fertilizers in soil.

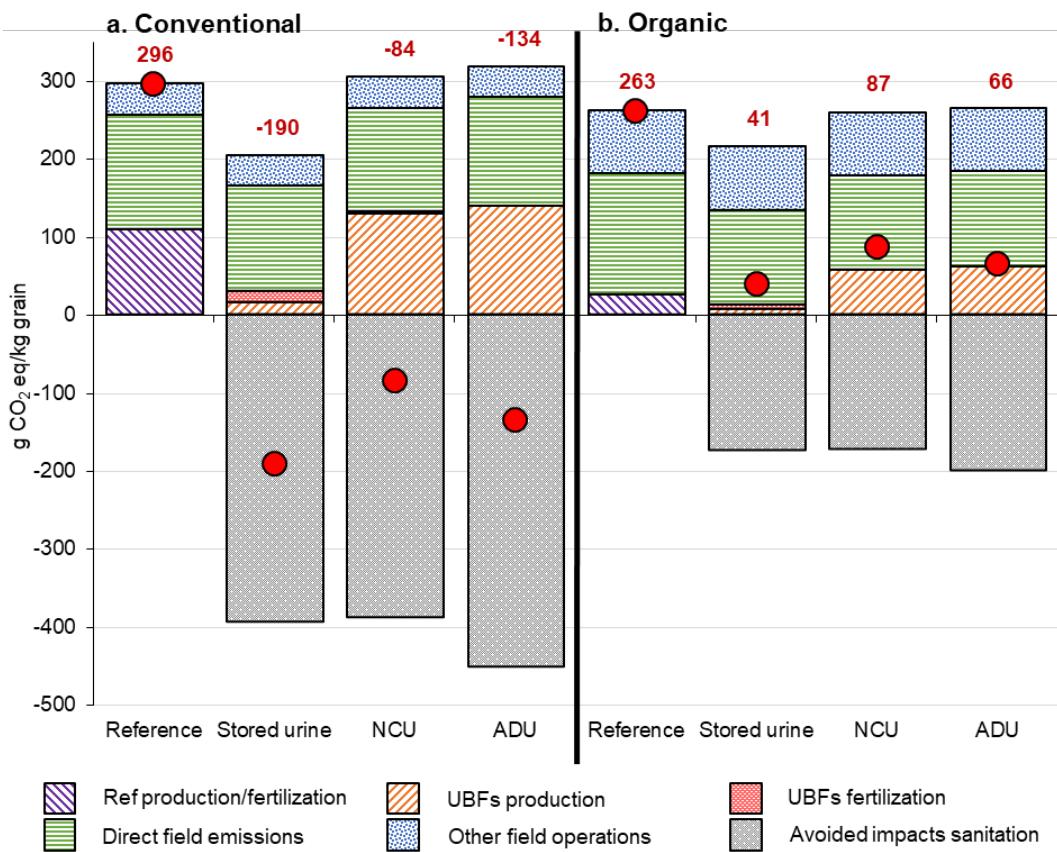


Figure SM-6-2. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the climate change impact category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

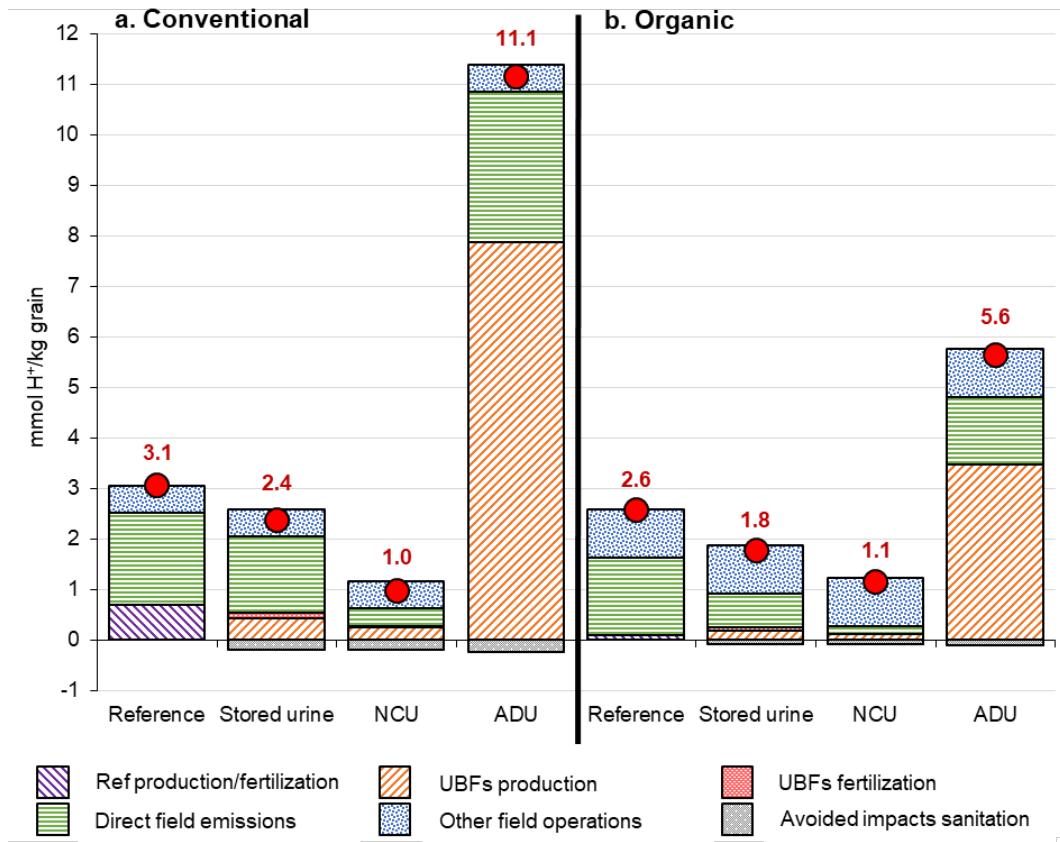


Figure SM-6-3. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the acidification impact category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

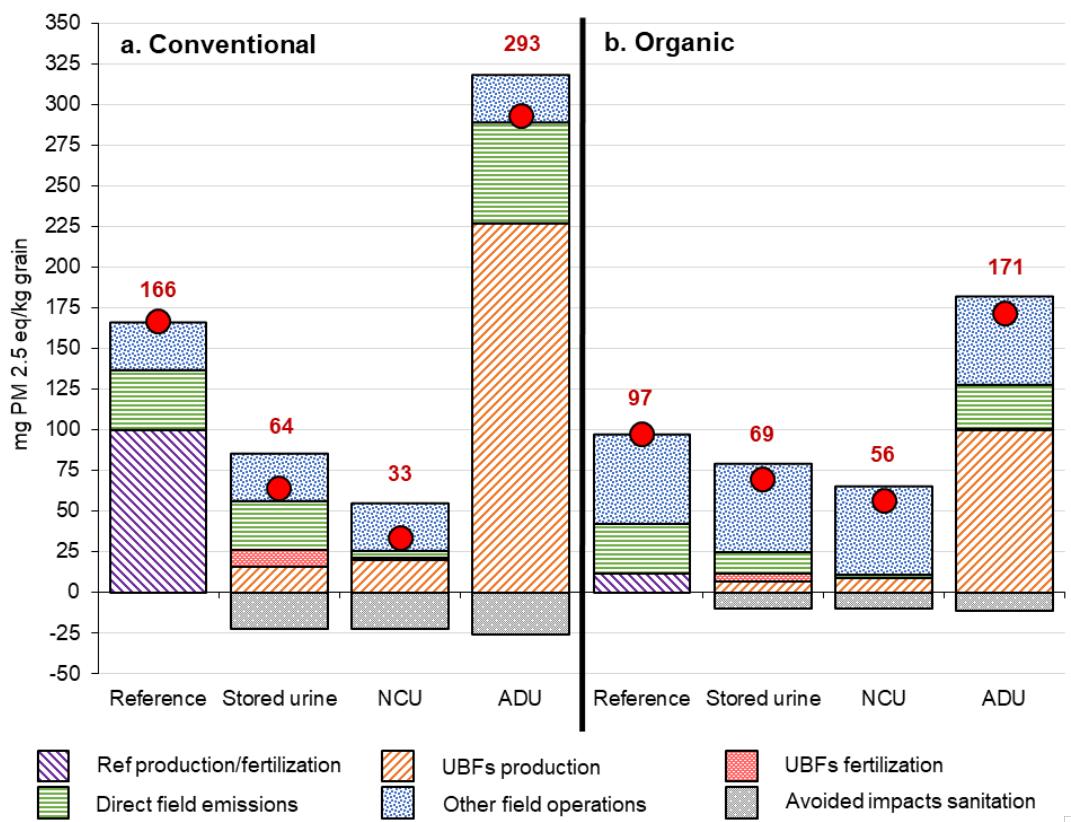


Figure SM-6-4. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the particulate matter impact category. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

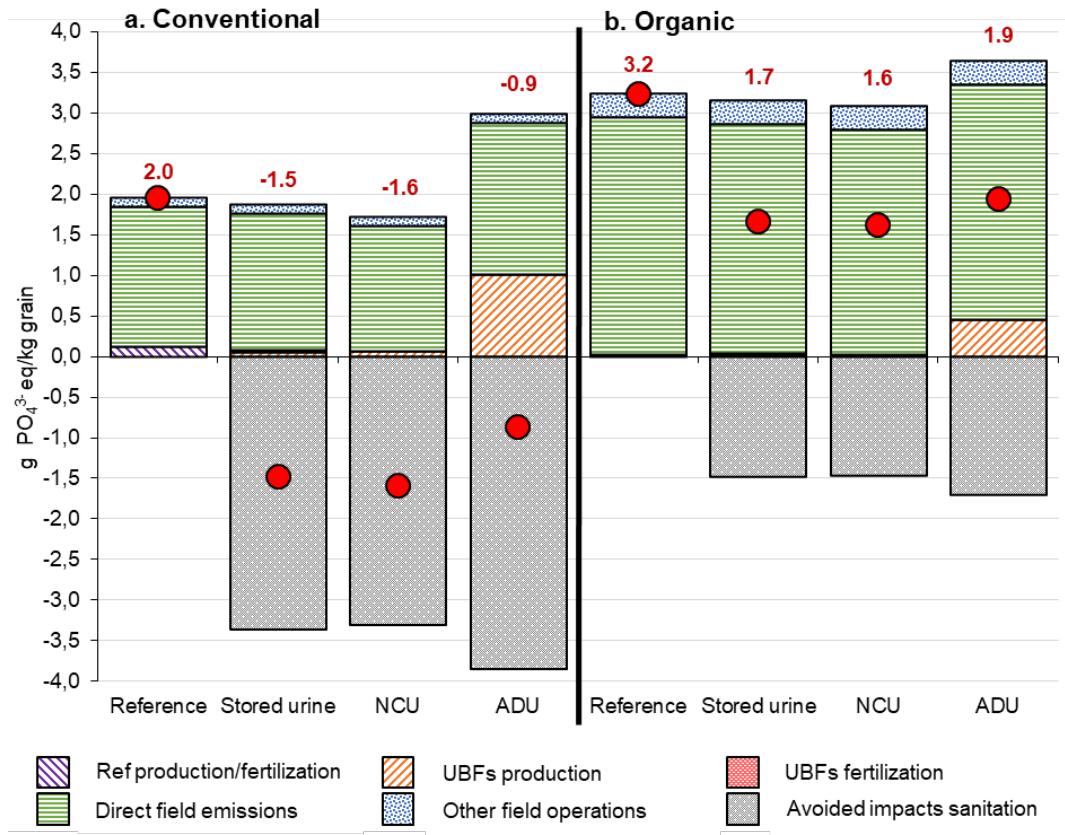


Figure SM-6-5. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the eutrophication impact category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

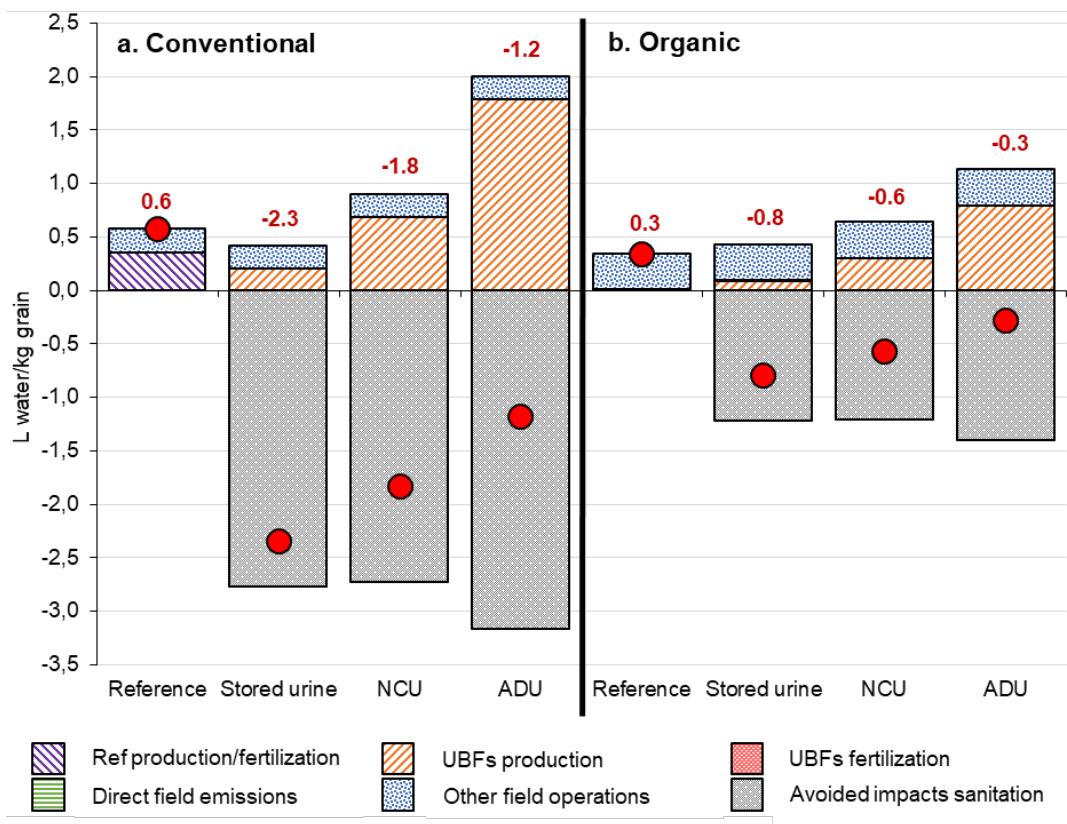


Figure SM-6-6. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the water resource depletion impact category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

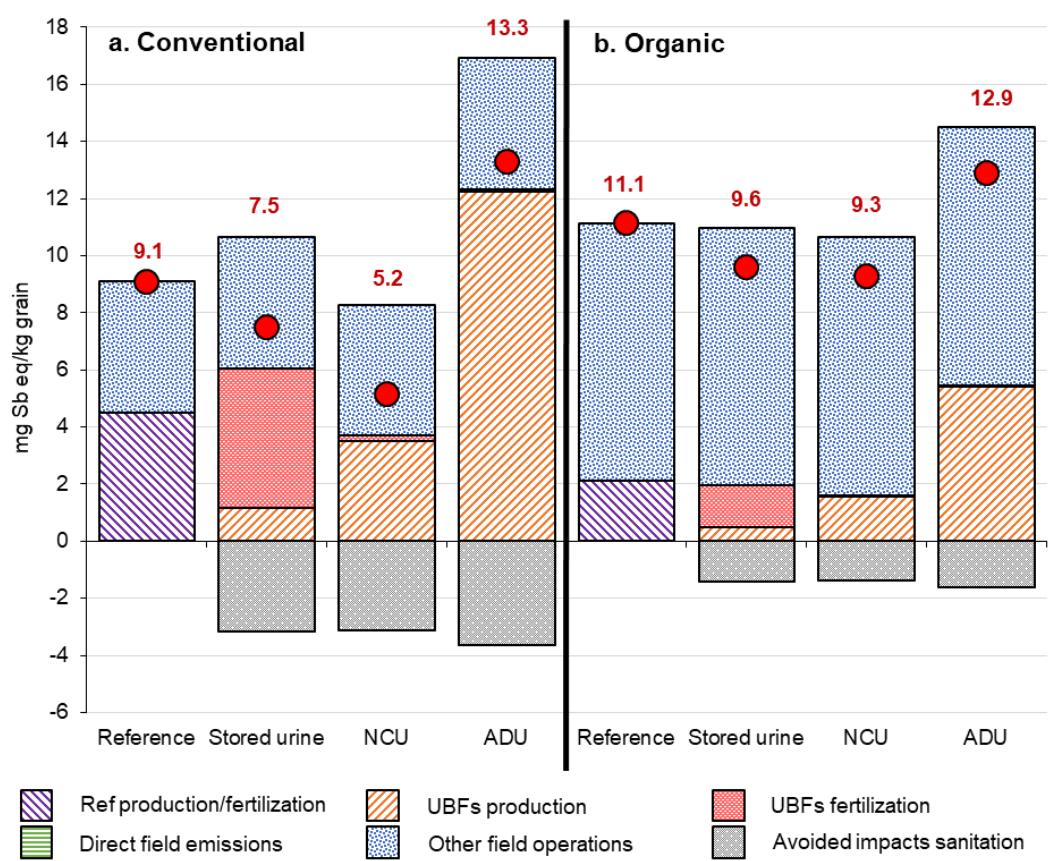


Figure SM-6-7. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the resource depletion impact category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

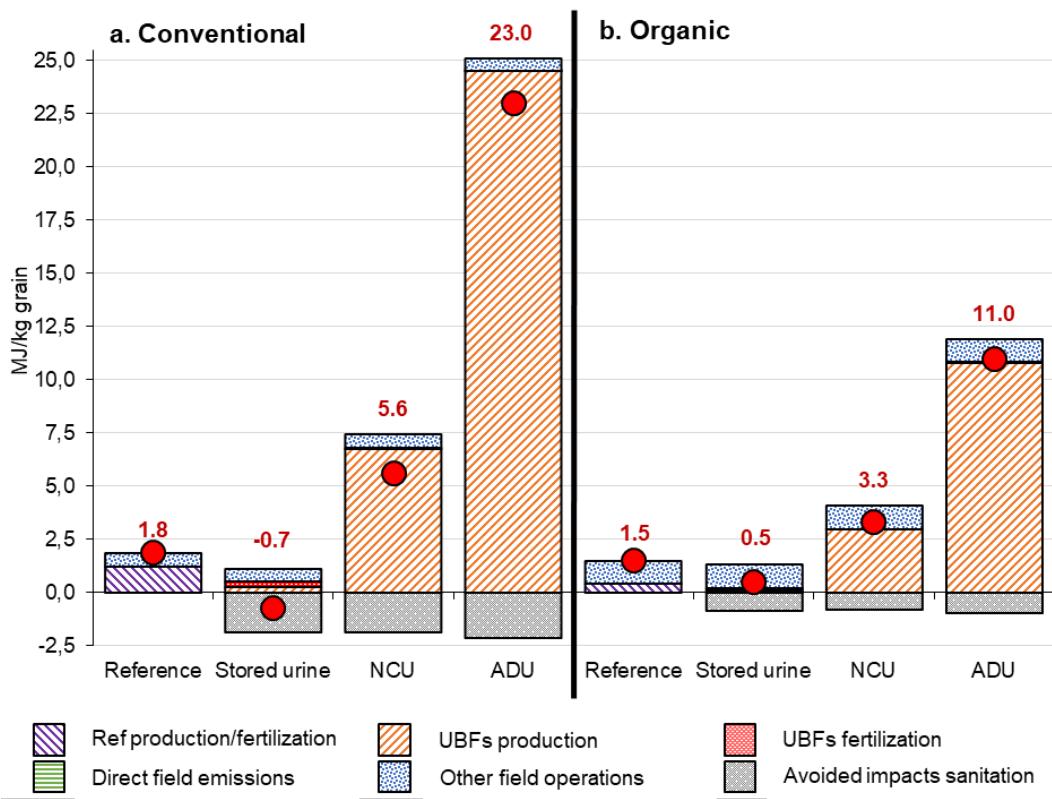


Figure SM-6-8. Results of the different scenarios in conventional (a.) and organic (b.) agriculture for the cumulative energy demand category for the production of maize. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

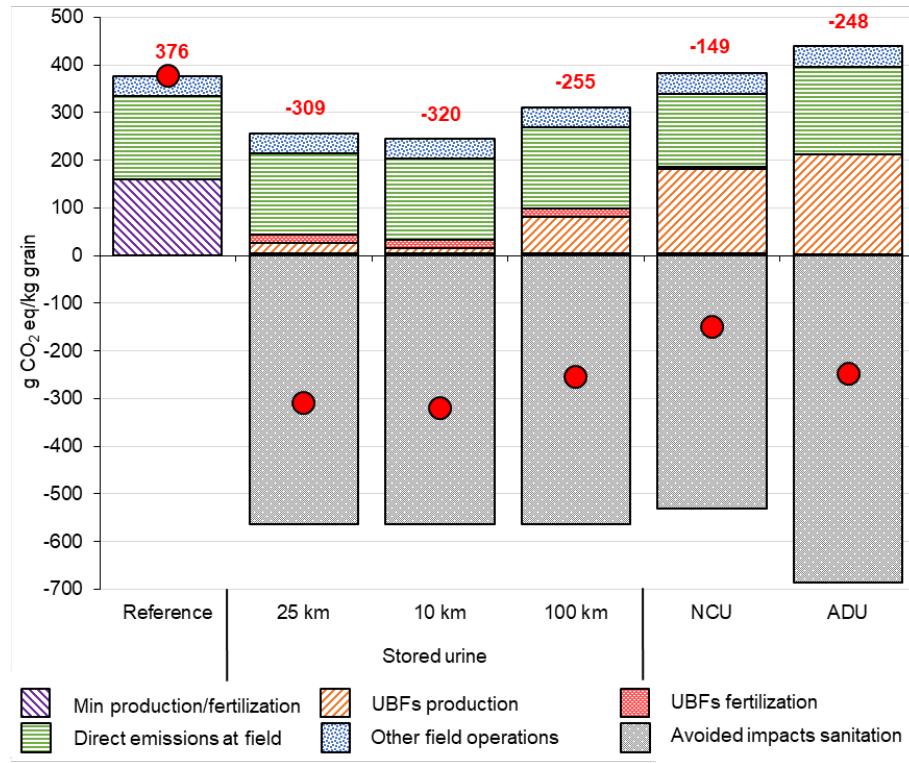


Figure SM-6-9. Results of the different scenarios in conventional agriculture for the climate change impact category including the impacts of the sensitivity analysis for stored urine. The red dot and the number above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. For stored urine, are also shown 25 km correspond to the main scenarios. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

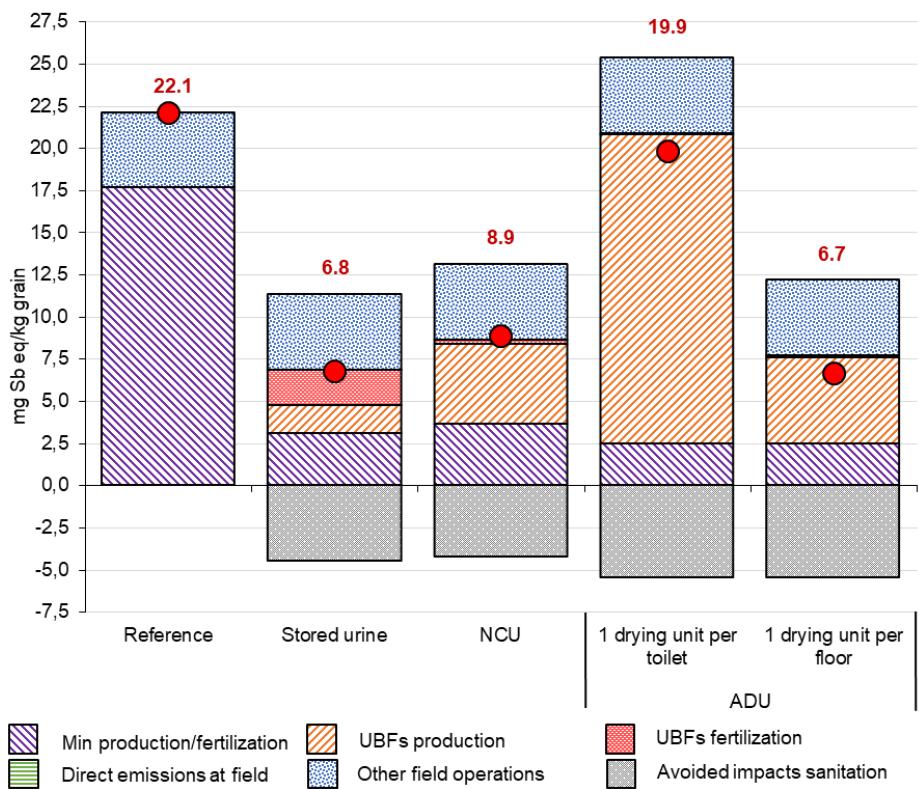


Figure SM-6-10. Results of the different scenarios in conventional agriculture for the resource depletion impact category including the impacts of the sensitivity analysis for ADU. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. 1 drying unit per toilet corresponds to the main scenario. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

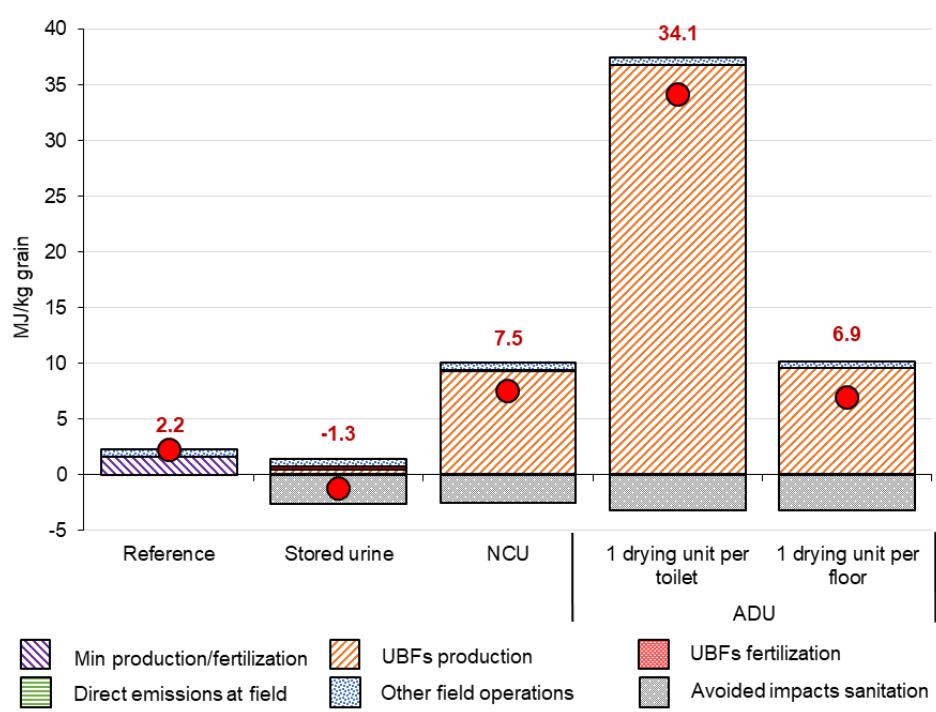


Figure SM-6-11. Results of the different scenarios in conventional agriculture for the cumulative energy demand category including the impacts of the sensitivity analysis for ADU. The red dot and the numbers above the bars indicate the balance between impacts due to wheat production and urine treatment and avoided impacts at WWTP. 1 drying unit per toilet corresponds to the main scenario. NCU: nitrified concentrated urine. ADU: alkalinized dehydrated urine.

Chapitre 7

Conclusion générale

1. Synthèse des résultats

1.1. L'urine humaine en agriculture

Les plantes ont besoin d'éléments nutritifs pour leur croissance et la fertilisation des cultures est aujourd'hui majoritairement conduite à l'aide d'engrais minéraux : azote issu du procédé Haber-Bosch ou phosphore issu de mines. De par leur caractère fossile, la consommation de ressources nécessaires à leur production et leurs impacts environnementaux, ces engrains ne sont pas durables du point de vue environnemental. Quelles que soient les chaînes alimentaires considérées, une partie de ces nutriments est absorbée par les êtres humains. Ces nutriments sont ensuite excrétés majoritairement dans l'urine qui représente environ 90% de l'azote et 60% du phosphore excrétés (Friedler et al. 2013). L'urine humaine qui représente un faible volume est mélangée avec les autres composantes des eaux usées : la chasse d'eau, les matières fécales et les eaux grises. Les eaux usées urbaines sont normalement traitées à la station d'épuration, mais ces traitements ne permettent qu'un très faible recyclage des nutriments (p. ex., 4% de l'azote dans le cas de l'agglomération parisienne, Esculier et al. 2018). Ces nutriments sont pour une grande partie renvoyés vers l'environnement, ce qui résulte en un système de gestion des nutriments linéaire et polluant. L'urine humaine peut être séparée à la source des autres composantes des eaux usées pour éviter la dilution ou le mélange avec les matières fécales. Sa valorisation en tant qu'engrais en agriculture pourrait participer au bouclage des cycles biogéochimiques. L'azote est excrété majoritairement sous forme d'urée dans l'urine et l'urée s'hydrolyse rapidement en azote ammoniacal qui est susceptible de se volatiliser. Ainsi, de nombreux traitements de l'urine sont possibles pour stabiliser l'azote sous une forme limitant la volatilisation et/ou réduire le volume. Par ailleurs des traitements supplémentaires peuvent être ajoutés pour réduire la contamination en pathogènes ou micropolluants organiques pouvant être présents dans l'urine. De ces traitements résultent de nombreux urinofertilisants différents et différentes filières de valorisation de l'urine en agriculture sont donc possibles. L'objectif de ce travail était de caractériser les filières envisageables de valorisation de l'urine humaine en agriculture au niveau agronomique et leurs impacts environnementaux.

1.2. Quels sont les traitements possibles de l'urine et quelles sont les caractéristiques des urinofertilisants résultant de ces traitements ?

La revue de la littérature réalisée dans la première partie de la thèse a fait la synthèse des différents traitements possibles de l'urine et des urinofertilisants résultant de ces traitements. Elle a mis en évidence une grande diversité de traitements ayant différents objectifs : (i) réduction des pertes azotées tout au long de la filière de valorisation en stabilisant l'azote dans une forme les limitant. La question de l'odeur des urinofertilisants est aussi importante et peut faire l'objet d'un traitement ad hoc.

L'odeur étant en partie liée à l'ammoniac gazeux (Hashemi and Han 2017), les traitements de stabilisation de l'azote permettent généralement de réduire l'odeur des urinofertilisants (ii) réduction du volume dans le but d'augmenter la concentration en nutriments dans les urinofertilisants et de réduire l'espace nécessaire au stockage, les besoins en transport ainsi que le travail lors de l'épandage (iii) réduction de la contamination en pathogènes et en micropolluants (dont résidus de pharmaceutiques) pour réduire les risques sur la santé ou l'environnement (iv) extraction d'un ou plusieurs nutriments de l'urine pour obtenir un fertilisant concentré.

Ainsi, il existe deux optiques de traitement : l'extraction des nutriments de l'urine (procédés extractifs), ou la conservation de la plupart des nutriments de l'urine, extrayant l'eau de l'urine et non les nutriments lorsqu'une réduction du volume est réalisée (procédés conservatifs). Considérant qu'il est pertinent de retourner aux sols agricoles non seulement l'azote et le phosphore mais également la majorité de la matière organique de l'urine, le potassium, les autres macronutriments (soufre, magnésium, calcium) et la majorité – voire l'intégralité – des micronutriments, nous nous sommes principalement intéressés aux traitements conservatifs. Les traitements étudiés ont aussi été choisis parmi les plus développés. Ainsi, les traitements de l'urine ayant été étudiés au cours de la revue sont les suivants : stockage, acidification, alcalinisation, nitrification, mélange avec des matières organiques et précipitation du phosphore (procédé extractif parmi les plus développés). Les traitements de réduction du volume, conservant les nutriments, ont aussi été considérés (Figure 7-1).

Cependant, de nombreuses autres combinaisons de traitements sont possibles (Figure 7-1). En effet, différents niveaux de traitements sont possibles en combinant réduction des pertes azotées, réduction du volume et réduction des pathogènes et des résidus de pharmaceutiques. Ces différentes filières peuvent être adaptées en fonction des contraintes des différentes étapes des filières, y compris l'étape d'épandage au champ (p. ex., sols à pH élevé favorisant la volatilisation, sols sensibles à la compaction limitant la possibilité d'épandre des fertilisants peu concentrés). La compatibilité entre les différents maillons des filières de traitements est à vérifier.

Les trois filières étudiées en détail au cours de l'analyse du cycle de vie ont été choisies car elles diffèrent dans les étapes et urinofertilisants produits tout en ayant des intérêts fertilisants élevés et un caractère minéral. Elles sont représentatives de filières existantes. L'une de ces filières correspond au stockage, qui est le traitement le plus simple de l'urine. Les deux autres filières sont l'urine nitrifiée et concentrée et l'urine alcalinisée déshydratée, qui sont respectivement à un stade de commercialisation et de pré-commercialisation.

D'autres filières ont été considérées. Les urinofertilisants produits ont été inclus dans le panel de produits dont on a étudié l'intérêt fertilisant azoté. En France, le mélange entre l'urine et les matières organiques est la pratique la plus répandue actuellement à travers l'utilisation de toilettes sèches unitaires. Cependant, ce produit se rapproche plus d'un amendement sur certains aspects (p. ex., taux

élevé de matière organique) que d'un fertilisant. En France également, la fermentation de l'urine se développe dans une optique de culture de bactéries et de production de biostimulants. Nous avons inclus ce produit dans notre étude en l'analysant comme un engrais (et non comme un biostimulant).

La revue de la littérature et l'analyse du cycle de vie ont mis en évidence le fait que ces traitements peuvent avoir des consommations d'énergie et de réactifs élevées qui auront un poids important dans le bilan environnemental des filières.

Les urinofertilisants passés en revue résultant de ces combinaisons de traitements ont des caractéristiques très variées. Tout d'abord, au niveau des formes de l'azote, nous pouvons observer une grande diversité en fonction des traitements de stabilisation de l'azote effectués : azote uréique, ammoniacal, nitrique ou organique. Les formes physiques des fertilisants (solide, liquide) et la concentration en nutriments sont aussi très variables. Par exemple, la concentration en azote des fertilisants testés au cours de ce travail varie entre 2 g N/kg et 107 g N/kg. Le pH des urinofertilisants est aussi très variable avec une gamme allant de 2 à 11. Enfin, les urinofertilisants ne comportent pas seulement de l'azote mais aussi du phosphore, du potassium et de nombreux autres nutriments (soufre, calcium, etc.).

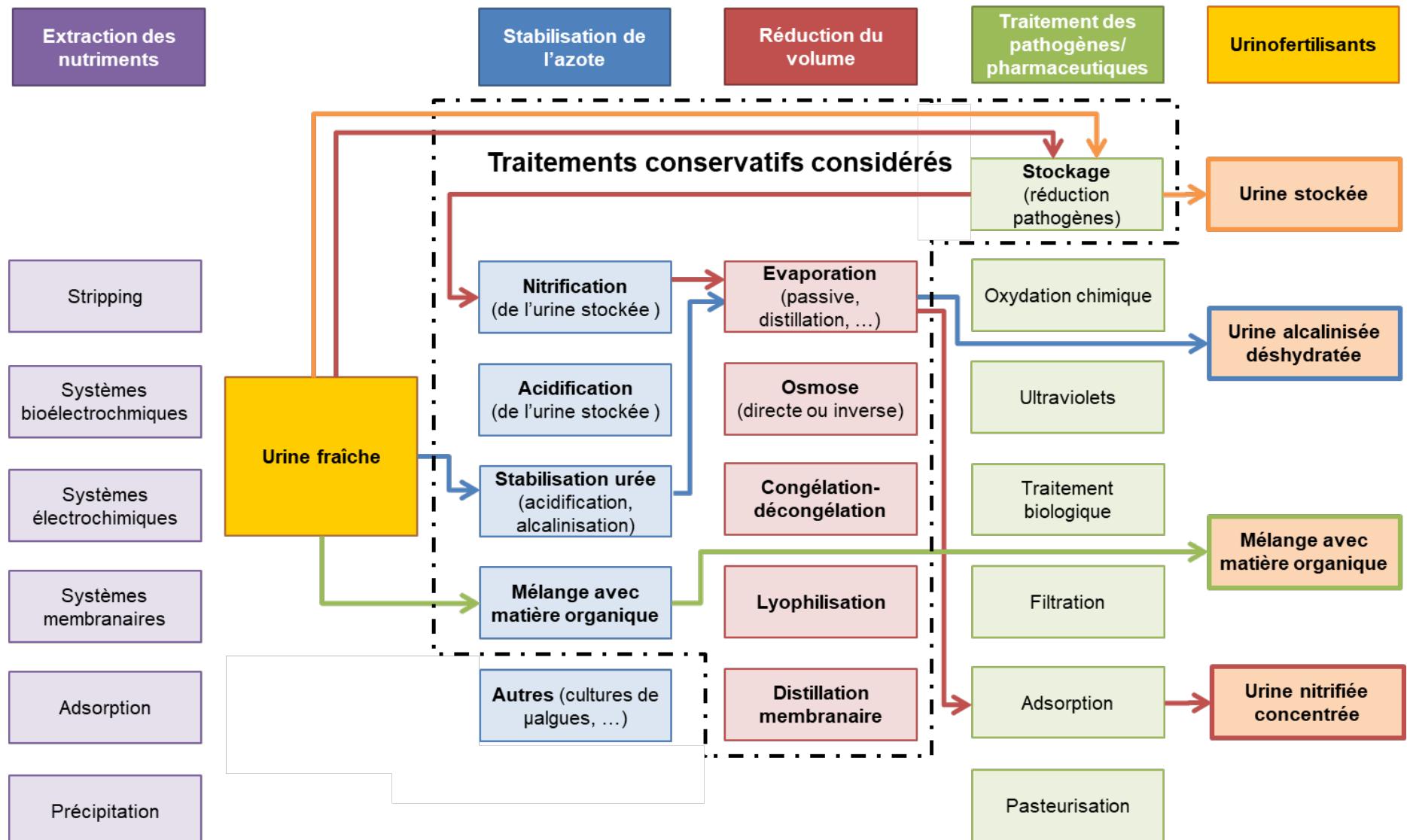


Figure 7-1. Les différents traitements possibles et souvent combinables. Les étapes successives de quatre traitements parmi les plus développés sont représentées par les flèches colorées. Le stockage est considéré comme un traitement des pathogènes. Les pointillés délimitent les traitements étudiés.

1.3. Quelle est l'efficacité fertilisante azotée des urinofertilisants en tant qu'engrais azotés ?

La revue de la littérature réalisée au chapitre 1 a mis en évidence une efficacité élevée des urinofertilisants. Cependant, excepté pour l'urine stockée, cette efficacité reste très peu étudiée pour les autres urinofertilisants. Ces résultats ont été confirmés par les essais réalisés en serre (chapitre 3) et au champ (chapitre 5) au cours desquels la plupart des urinofertilisants ont montré une efficacité élevée. L'efficacité fertilisante est ici principalement caractérisée par le coefficient d'équivalence engrais, qui correspond à l'efficacité relative d'un fertilisant par rapport à celle d'un engrais minéral en termes de quantité d'azote absorbé par la plante sur un cycle de culture. Pour les 9 urinofertilisants testés en conditions contrôlées (en serre), les coefficients d'équivalence engrais mesurés allaient de 66% à 120%, toujours supérieurs à celui mesuré pour le lisier de bovins dont le coefficient équivalent engrais était de 51%. Sept urinofertilisants avaient même un coefficient d'équivalence engrais supérieur à 85% et donc une efficacité proche de celle de l'engrais minéral. En conditions réelles (au champ), nous avons observé pour la plupart des urinofertilisants un coefficient d'équivalence engrais supérieur à 70% (allant de 52% à 116%). L'efficacité des urinofertilisants était plus élevée que celle des autres engrains organiques testés, tels que les digestats et les lisiers bovins, pour lesquels le coefficient d'équivalence engrais variait entre 50% et 75% pour les digestats et entre 9% et 38% pour les lisiers bovins. L'efficacité azote était plus faible en comparaison de celle mesurée en serre. Ces différences peuvent s'expliquer par les conditions expérimentales. En particulier, la volatilisation ammoniacale a été très fortement limitée en serre, ce qui a maximisé l'azote utilisable par la plante et peut expliquer des coefficients d'équivalence engrais plus élevés.

Cette efficacité est corrélée avec la forte teneur en azote minéral de la majorité des urinofertilisants. En effet, la fraction organique est généralement comprise entre 0% et 20% de l'azote total (en considérant l'urée comme faisant partie de la fraction minérale). Cet azote organique doit d'abord être minéralisé avant de pouvoir être absorbé par les cultures. C'est une des hypothèses qui peut expliquer des coefficients d'équivalence engrais inférieurs à 100% pour la plupart des urinofertilisants (exceptée l'urine nitrifiée qui ne contient pas d'azote organique). L'organisation de l'azote de l'urine qui se produit en mélangeant l'urine avec des matières organiques entraîne une décroissance de l'efficacité azote à court terme. L'efficacité de l'azote de l'urine fermentée est également un peu plus faible, ce qui pourrait être dû à l'organisation de l'azote par la microflore se développant pendant la fermentation. Dans les expérimentations au champ, les urinofertilisants ont une forte efficacité azote, ce qui signifierait que la volatilisation ammoniacale est restée limitée en raison des conditions climatiques à l'épandage (températures basses à l'épandage en sortie d'hiver).

Même si nous n'avons étudié que l'azote, l'efficacité fertilisante en tant qu'engrais azoté pourrait aussi être accrue par les apports d'autres nutriments (p. ex., P, K, S) contenus dans les

urinofertilisants. Les hypothèses sur les différences d'efficacité entre les urinofertilisants (en particulier pour l'urine fermentée et le mélange avec les matières organiques) pourraient être confirmées par la mise en place d'une expérimentation de suivi de l'azote après apport au sol afin d'étudier les différentes transformations de l'azote (p. ex., nitrification, organisation). Les essais menés au cours de ce travail ont été effectués sur des céréales et du colza. L'azote des urinofertilisants est disponible quasi immédiatement après l'apport et une variabilité des coefficients d'équivalence selon les cultures ayant des dynamiques d'absorption d'azote différentes, est peu probable. Cependant, il serait intéressant de conforter l'efficacité fertilisante des urinofertilisants pour d'autres cultures (p. ex., maraîchage).

Les essais menés au champ ont également montré qu'une substitution complète des engrains minéraux par des urinofertilisants semble possible sans perte de rendement ou de qualité de la récolte. Dans ce cas, des formes concentrées de fertilisants semblent plus aisées à utiliser afin d'éviter les problèmes de compaction liés aux passages répétés d'engins et de dégâts aux cultures. Un fertilisant d'origine renouvelable et ayant une efficacité élevée serait d'un grand intérêt en agriculture biologique pour pallier les carences en azote qui limitent parfois le rendement en comparaison de l'agriculture conventionnelle. Cependant, l'insertion des urinofertilisants dans les systèmes biologiques n'est pas explicitement autorisée dans la réglementation, est très peu étudiée et nécessite d'être creusée, en interaction avec les autres sources alternatives d'azote (légumineuses, autres PRO).

1.4. Quels sont les impacts associés à l'apport d'urinofertilisants au niveau de la parcelle ?

Les impacts associés aux apports d'urinofertilisants considérés sont principalement les contaminants apportés par ces urinofertilisants et la volatilisation ammoniacale. Une première synthèse sur ces impacts a été réalisée dans la revue bibliographique. Les trois principaux types de contaminants considérés dans les urinofertilisants sont les éléments traces, les pathogènes et les micropolluants organiques (principalement les résidus de pharmaceutiques). Les urinofertilisants sont faiblement contaminés en éléments traces, mais certains métaux peuvent être apportés par les réactifs utilisés dans certains traitements (p. ex., cendre, Simha et al. 2020). Généralement, les flux de métaux liés à l'utilisation d'urinofertilisants sont inférieurs à ceux provenant de l'utilisation d'engrais phosphatés ou de certains engrains organiques tels que les lisiers de bovin. Pour tous les urinofertilisants testés en serre, les flux de métaux liés à leur apport à une dose agronomique sont inférieurs au seuil de la norme NF U 44-095, pour l'utilisation du compost de boues d'épuration en agriculture. Outre les éléments traces, les urinofertilisants contiennent différents sels (lié à la forte consommation de sel de table en moyenne) qui peuvent impacter négativement les cultures ou la structure du sol.

Certains pathogènes peuvent être excrétés dans l'urine mais la plupart des contaminations s'effectuent au niveau de la toilette à séparation, où des matières fécales peuvent contaminer la partie

urine des toilettes. La contamination reste cependant limitée avec l'utilisation de certains types de toilettes tels que des urinoirs secs. Toutefois, en raison de la présence d'ammoniac et de pH acide ou basique dans de nombreux urinofertilisants, la contamination en organismes pathogènes est généralement traitée efficacement. Le traitement le plus simple consiste en un stockage de l'urine en milieu clos pour une durée allant de 1 à 6 mois (WHO 2012). Les pathogènes peuvent aussi être traités lors de la réduction du volume (distillation à haute température) ou par un traitement spécifique (p. ex., traitement à l'ozone).

La contamination en sept pathogènes indicateurs a été mesurée sur certains des produits testés. Cette contamination était, pour la majorité des indicateurs, plus faible que celle du lisier bovin et en dessous des valeurs seuils requises par la norme NF U 44-095 pour l'utilisation du compost de boues d'épuration en agriculture.

Parmi les différents contaminants trouvés dans l'urine, les résidus pharmaceutiques semblent être les plus préoccupants en raison de leur potentiel éco-toxicologique et de leur concentration relativement élevée par rapport à d'autres composés. Environ deux tiers des résidus pharmaceutiques sont excrétés dans l'urine (Lienert et al. 2007). Ils sont présents dans "l'urine moyenne" à des concentrations allant jusqu'à plusieurs mg/L (Winkler et al. 2008; Bischel et al. 2015). La nécessité de traitements spécifiques de ces résidus pharmaceutiques avant l'apport de l'urine est actuellement débattue et cette question nécessite d'être approfondie (Winkler et al. 2009; WHO 2012). L'adsorption des résidus pharmaceutiques sur charbon actif est un traitement qui semble prometteur pour assurer cette réduction efficace tout en conservant la grande majorité des nutriments (Köpping et al. 2020). Les impacts liés aux apports de ces contaminants doivent être étudiés au champ après apport des urinofertilisants (p. ex., impact sur les communautés microbiennes du sol, antibiorésistance).

Les impacts liés aux apports des urinofertilisants et aux émissions d'azote après apport sont importants à maîtriser. Ainsi, les volumes à apporter varient considérablement entre les urinofertilisants et peuvent être importants. Par exemple, pour apporter 170 kg N/ha (limite pour les fertilisants organiques dans la directive nitrate), ces volumes vont de 94 t/ha (urine diluée par chasse d'eau) à 0,7 t/ha (urine déshydratée en considérant 24% N dans la matière sèche de l'urine, Simha et al. 2020). Épandus avec une tonne à lisier, ces volumes importants peuvent engendrer une compaction du sol (Lamandé and Schjønning 2018) et potentiellement des pertes de rendement. L'usage de matériel d'épandage sans tonne pourrait limiter ces problèmes mais peut complexifier les chantiers d'épandage (Johansson et al. 2000).

La volatilisation ammoniacale apparaît comme un impact potentiel important au niveau de la parcelle. La volatilisation ammoniacale cause des impacts à la fois du point de vue agronomique (perte de nutriments), sur l'environnement (eutrophisation et acidification) et la santé (formation de particules fines). Une première expérimentation a été réalisée en conditions contrôlées (chapitre 3) afin

de mesurer le potentiel de volatilisation de 9 urinofertilisants en comparaison d'un engrais minéral (nitrate d'ammonium) et d'un engrais organique (lisier bovin). Les urinofertilisants ont montré des potentiels de volatilisation différents en fonction de leurs caractéristiques physico-chimiques. La plus forte volatilisation a été mesurée pour les urinofertilisants alcalins (urine stockée et urine stabilisée par alcalinisation, entre 14% et 18% de l'azote apporté). Cette volatilisation est similaire à celle du lisier bovin. En revanche, une faible volatilisation a été mesurée pour les urinofertilisants acides (urine stabilisée par acidification, urine fermentée, moins de 5% de l'azote apporté). Lorsque l'urine est mélangée avec certaines matières organiques, le mélange étant solide, elle peut être retenue en surface et entraîner une plus forte volatilisation (p. ex., 76% de l'azote ammoniacal pour le mélange urine stockée + compost de déchets verts). Au contraire, le mélange avec d'autres matières organiques peut peut-être limiter la volatilisation en stabilisant l'azote (p. ex., 5% de l'azote apporté volatilisé pour le mélange urine fraîche + copeaux de bois). La nitrification de l'urine limite presque totalement la volatilisation qui est alors similaire à celle de l'engrais minéral testé (nitrate d'ammonium, moins de 1% de l'azote apporté).

Au champ, seule la volatilisation ammoniacale de l'urine stockée a été comparée à celle de l'ammonitrat (chapitre 5). Cette mesure s'est effectuée en conditions favorables à la volatilisation (température élevée et vent). Pour l'urine stockée, 34% de l'azote apporté a été volatilisé contre 1% pour l'engrais minéral. Les risques de pertes par volatilisation ammoniacale doivent donc être pris en compte au moment de l'apport des urinofertilisants. Cependant, celle-ci pourra être grandement limitée par différentes méthodes d'apports comme l'injection des produits liquides ou l'enfouissement dans le sol avant le semis. La volatilisation ammoniacale étant fortement dépendante des conditions météorologiques et des caractéristiques des sols, elle doit être étudiée en conditions réelles pour les autres urinofertilisants et dans différents contextes pédoclimatiques. Même si l'impact de différentes méthodes d'apport a été étudié pour d'autres fertilisants (p. ex., Webb et al. 2010), il doit être confirmé pour les urinofertilisants.

1.5. Quels sont les impacts environnementaux des différentes filières de valorisation de l'urine ?

La revue de la littérature (chapitre 1) a mis en évidence une grande diversité dans les consommations d'énergie des différents traitements. Elle est particulièrement liée aux traitements de réduction du volume, où elle peut être plus élevée que celle pour la fabrication des engrais minéraux. Certains traitements comme l'osmose semblent avoir une consommation d'énergie modérée, mais un facteur de concentration limité de 3 à 5. D'autres traitements permettent une déshydratation de l'urine, au prix cependant d'une forte consommation d'énergie. Certains traitements nécessitent aussi l'utilisation de réactifs d'origine fossile (p. ex., utilisation de magnésium pour la précipitation du phosphore).

Toutefois, l'impact des filières de valorisation et des traitements de l'urine reste peu étudié. La revue de la littérature a mis en évidence le besoin de filières pilotes pour mieux évaluer leurs impacts.

Une analyse du cycle de vie de la production de céréales a été conduite en considérant trois filières de valorisation de l'urine (urine stockée, urine nitrifiée concentrée et urine alcalinisée déshydratée, chapitre 6). Cette étude a mis en évidence un meilleur bilan environnemental de ces 3 filières en comparaison des pratiques actuelles (utilisation d'engrais minéraux et traitement de l'urine à la station d'épuration) pour une grande partie des indicateurs d'impacts. L'utilisation d'urinofertilisants permet notamment de réduire les impacts sur le changement climatique, l'eutrophisation, l'utilisation de ressources fossiles et la consommation d'eau au niveau des toilettes. Ces impacts sont meilleurs en grande partie grâce à la non consommation d'engrais minéraux ainsi qu'aux impacts évités à la station d'épuration. Cependant, les indicateurs d'impacts liés à la volatilisation – comme l'acidification de l'environnement ou la formation de particules fines – peuvent être élevés, en comparaison des pratiques actuelles, au niveau du traitement et/ou de l'épandage (urine stockée et urine alcalinisée déshydratée). Parmi les scénarios étudiés, la consommation d'énergie pour réduire le volume peut être plus élevée que les pratiques actuelles (urine nitrifiée concentrée et urine alcalinisée déshydratée). Les impacts dus au transport, même de volumes importants, semblent limités. La volatilisation ammoniacale et la consommation d'énergie ont été identifiées comme les deux éléments les plus sensibles du bilan environnemental des filières de valorisation et doivent être surveillées lors de la future mise en place des filières.

En conclusion, les urinofertilisants sont des engrains dérivés d'une ressource qui est actuellement très peu valorisée et en partie rejetée dans l'environnement. Ils ont montré pour la plupart d'entre eux une efficacité fertilisante élevée et un meilleur bilan environnemental que les pratiques actuelles. Cependant, certains impacts liés à leur filière de valorisation (volatilisation ammoniacale, consommation d'énergie des traitements, contaminants) sont à surveiller. Les urinofertilisants peuvent contribuer à boucler les cycles biogéochimiques et à réduire les impacts des systèmes agricoles et des systèmes de gestion des excréments humains sur l'environnement. Cependant, l'utilisation d'urinofertilisants n'est qu'un des leviers pour améliorer le bilan environnemental de notre alimentation. Celui-ci peut en effet être réduit par un changement de régime alimentaire (p. ex., moindre consommation de produits animaux) ou bien encore par la mise en place de bonnes pratiques agronomiques (p. ex., cultures pièges à nitrate).

1.6. Synthèse de quatre filières parmi les plus développées

Un résumé de 4 filières de valorisation parmi les plus développées aujourd'hui est proposé en Tableau 7-1.

Tableau 7-1. Résumé des caractéristiques de quatre filières de valorisation d'urinofertilisants en agriculture parmi les plus développées.

| Urinofertilisant | Caractéristiques du fertilisant | Efficacité fertilisante (Coefficient d'équivalence engrais) | Potentiel de volatilisation (% N apporté) | Quantité à épandre (pour apporter 170 kg N/ha) | Impacts de la filière amont |
|---|---|--|---|---|---|
| Urine stockée | - Azote ammoniacal majoritaire (71% à 97%) - pH basique (7 à 9) - Liquide - Urinofertilisant peu concentré (3 à 7 g N/kg) | - Élevé si volatilisation limitée - 86% (serre) - 52% à 111% (champ) | - Élevé en raison du pH élevé et de la forte proportion en N ammoniacal - 14 à 17% (chambre) - 34% (champ) | - Élevée - 24 à 57 t/ha | - Faibles impacts |
| Urine nitrifiée concentrée | - Azote à 50% ammoniacal et 50% nitrique - pH acide (4) - Liquide - Urinofertilisant concentré (44 à 52 g N/kg) | - Très élevé en raison de la faible volatilisation - 120% (serre) - 116% (champ) | - Très faible en raison du pH acide et de la forte proportion en N nitrique - < 2% (chambre) | - Faible à moyenne - 3 à 4 t/ha | - Forte consommation d'énergie (130 kWh/m ³) - Autres impacts faibles |
| Urine alcalinisée déshydratée | - Azote uréique majoritaire (89% à 95%). - pH basique (9 à 11) - Solide - Urinofertilisant concentré (101 à 107 g N/kg) | - Élevé si volatilisation limitée - 86% (serre) | - Élevé en raison du pH élevé et de la forme solide qui reste en surface - 18% (chambre, mesuré sur urine alcalinisée non déshydratée) | - Faible - 1,7 à 1,8 t/ha | - Forte consommation d'énergie (533 kWh/m ³) - Volatilisation pendant le traitement (10% N tot) - Autres impact faibles |
| Mélange avec matières organiques | - Généralement, forte proportion d'azote organique (76% à 83%) - pH généralement basique (8 à 9) - Solide - Généralement moins concentré que les autres urinofertilisants (3 à 4 g N/kg, en ne considérant que l'azote provenant de l'urine) | - Généralement plus faible que les autres urinofertilisants en raison de la forte proportion en N organique - 66% (serre) | - Variable en fonction de la concentration du mélange - 5% à 10% (N total) - 40% à 76% (N-NH ₄) (chambre) | - Potentiellement élevée en raison de la dilution - 43 à 57 t/ha | - Non étudiés |

L’urine stockée est la filière de traitement la plus simple et est déjà mise en œuvre à l’échelle de quartiers essentiellement à l’étranger (p. ex., Suède, Johansson et al. 2000). L’azote est majoritairement sous forme ammoniacale ce qui permet une efficacité fertilisante élevée. Le pH et l’azote ammoniacal peuvent cependant entraîner une forte volatilisation au champ. L’urinofertilisant étant faiblement concentré, les volumes à apporter au champ sont importants. Les impacts de la filière amont sont faibles.

L’urine nitrifiée concentrée est un fertilisant ne comportant pas d’azote organique, expliquant une très forte efficacité fertilisante. De plus, il est acide et son apport entraîne une très faible volatilisation ammoniacale. Les nutriments ayant été concentrés par distillation, les volumes à apporter sont limités. Cependant, la consommation d’énergie du traitement (stabilisation de l’azote par nitrification puis distillation) est élevée.

L’urine alcalinisée déshydratée correspond à une stabilisation de l’azote sous forme d’urée, l’urine est ensuite déshydratée par évaporation. C’est l’urinofertilisant le plus concentré. Cependant, le pH élevé entraîne une volatilisation potentiellement élevée au niveau du traitement et au moment de l’apport. Si la volatilisation est limitée, l’efficacité fertilisante est élevée. Cependant, le procédé n’est pas encore optimisé et la consommation d’énergie pour la déshydratation est élevée.

Pour le mélange de l’urine avec des matières organiques, une forte volatilisation ammoniacale est possible pendant le traitement (p. ex., estimée à 15% pour le mélange urine fraîche + copeaux de bois stocké dans un contenant scellé). La majorité de l’azote est généralement sous forme organique dans le produit final. Cette forte proportion d’azote organique diminue l’efficacité fertilisante à court terme en comparaison des autres urinofertilisants. Si une partie de l’azote reste sous forme ammoniacale et que le mélange solide n’est pas incorporé dans le sol, la volatilisation peut potentiellement être élevée. Le produit final est généralement peu concentré en azote minéral, les quantités à apporter au champ sont donc élevées.

Enfin, la production de biostimulant à partir d’urine est actuellement en cours de développement en France. L’étude de biostimulants n’a toutefois pas été inclue dans cette thèse et la réalisation de protocoles d’étude adaptés est nécessaire pour permettre leur évaluation.

Proposition de filière de valorisation :

Au regard des deux points sensibles que représentent la volatilisation ammoniacale et la consommation d’énergie des traitements, il pourrait être intéressant d’aller vers un traitement stabilisant l’azote par acidification, avant hydrolyse de l’urée ce qui nécessite une quantité d’acide plus faible que pour l’acidification de l’urine stockée. En effet, l’acidification de l’urine fraîche semble efficace pour limiter la volatilisation. Cette stabilisation de l’azote pourrait également être combinée à une concentration de l’urine réalisée à l’aide d’une source d’énergie renouvelable (p. ex., séchage solaire, Antonini et al. 2012) ou en utilisant de la chaleur fatale (réfrigérateur, eaux

ménagères, etc.). De telles filières pourraient alors correspondre à une performance élevée sur les quatre paramètres du Tableau 7-1 à savoir une efficacité fertilisante élevée, un potentiel de volatilisation faible, une quantité à épandre faible et des impacts de la filière amont réduits.

2. Perspectives

2.1. Impacts agronomiques

Les effets considérés au cours de ce travail opèrent majoritairement à court terme (p. ex., efficacité fertilisante à court terme, volatilisation ammoniacale après apport). Cependant, de nombreux impacts intervenant à plus long terme doivent être pris en compte et pourraient être étudiés à travers un essai pluriannuel. À notre connaissance, le seul essai de longue durée actuellement en cours est l'essai Crucial au Danemark (López-Rayó et al. 2016). Depuis 2003, une partie des parcelles est fertilisée avec de l'urine stockée récupérée d'un quartier d'habitations équipées de toilettes à séparation à la source. Les effets de l'urine stockée sont comparés à ceux d'un engrais minéral et d'autres fertilisants organiques. Parmi les résultats obtenus, l'évolution de la teneur en matière organique du sol des parcelles fertilisées avec de l'urine stockée est très similaire à celle de l'engrais minéral en raison de la faible teneur en matière organique de l'urine. Cette teneur en matière organique dans les sols fertilisés avec de l'urine est plus faible que celle des parcelles où a été apporté du compost par exemple, mais reste similaire à celles où ont été apportés des boues d'épuration ou du lisier bovin. En raison de la grande quantité en sodium des régimes alimentaires occidentaux standards, l'usage d'urinofertilisant peut présenter un risque de salinisation des sols. L'épandage systématique d'urinofertilisant pourrait dégrader la structure du sol et augmenter l'énergie nécessaire au travail du sol (Peltre et al. 2015). Les indicateurs d'activité microbienne du sol des parcelles fertilisées avec l'urine stockée sont similaires à celles fertilisées avec l'engrais minéral, mais inférieurs à certaines parcelles fertilisées avec d'autres engrains organiques (Poulsen et al. 2013b). Aucun changement majeur dans les communautés d'organismes procaryotes du sol n'a été observé (Poulsen et al. 2013a). Le pH après 10 ans d'apport d'urine (6,1) est similaire au traitement engrais minéral (6,4) et à celui du traitement sans fertilisation et des autres engrains organiques (Gómez-Muñoz et al. 2017). Ces résultats soulignent que l'urine n'exclut pas l'usage d'autres PRO et qu'une complémentarité entre l'urine pour les apports de nutriments rapidement disponibles et les autres PRO pour la fertilité du sol à long terme peut exister.

Un autre point important à étudier est la présence de micropolluants organiques, et en particulier de résidus de produits pharmaceutiques, présents dans les urinofertilisants et les risques liés à leur utilisation. En effet, la majorité des traitements ne permet pas leur dégradation (excepté certains traitements spécifiquement dédiés à leur diminution, p. ex., adsorption sur charbon actif). La concentration de 59 résidus pharmaceutiques et micropolluants (p. ex., antibiotiques, anti-inflammatoires, produits de soin personnels) est actuellement étudiée au sein du projet Agrocapi

(Goulas et al. 2020). Le devenir de ces micropolluants dans les sols après épandage est aussi considéré. L'antibiorésistance est une problématique de santé et correspond à la capacité pour les bactéries à devenir résistantes aux antibiotiques. Elle se développe lorsque celles-ci sont en contact avec des antibiotiques (p. ex. dans la cuve de stockage avant apport ou dans le sol après apport d'un fertilisant contaminé avec des antibiotiques). Certains gènes de résistance aux antibiotiques sont actuellement recherchés dans les produits épandus lors des essais au champ du projet Agrocapi ainsi que dans les sols sur lesquels ces produits ont été épandus.

Les émissions de gaz à effet de serre au niveau de la parcelle (principalement N₂O) ont été identifiées comme étant un contributeur majeur à l'impact « changement climatique » dans le cas de la production de céréales lors de l'analyse du cycle de vie réalisée. Les émissions de N₂O se produisent lors des transformations de l'azote dans le sol (nitrification et dénitritation). Elles sont liées aux apports d'azote par les fertilisants. Les émissions de N₂O après apport de différents urinofertilisants (urine stockée, urine nitrifiée) ont été suivies en comparaison d'un engrais minéral sur un des essais agronomiques menés au cours de ce travail. Cependant, ces résultats n'ont pas pu être intégrés dans cette thèse. Les méthodes d'apport pour limiter la volatilisation ammoniacale pourraient favoriser d'autres émissions gazeuses (p. ex., N₂O, Emmerling et al. 2020). Il reste donc nécessaire d'étudier si les émissions de N₂O suite à un apport d'urine sont similaires à celles après un apport d'engrais minéral, la forme de l'azote pouvant avoir un impact sur ces émissions (Hénault et al. 2012).

2.2. Freins et leviers sociotechniques (en France)

En France, la déconnexion actuelle entre la gestion de l'alimentation humaine et la gestion de l'excrétion humaine résulte d'un processus conjoint d'urbanisation, d'industrialisation et de développement de l'hygiénisme qui date de plus d'un siècle (Esculier & Barles, 2019). L'utilisation des engrais minéraux et la collecte de l'urine par l'égout sont en place depuis de nombreuses décennies. Ce sont des systèmes fortement verrouillés et il est difficile d'y faire émerger des innovations. Les freins et leviers liés à la mise en place de filières de valorisation agricole de l'urine en France ont été étudiés dans le cadre du projet Agrocapi par Brun (2018). Différents freins et verrous sociotechniques ont déjà été identifiés (Brun 2018; Esculier 2018). En ce qui concerne la communauté agricole, nous retrouvons :

(i) La méconnaissance des effets fertilisants de l'urine ("urine blindness", Drangert 1998) et l'absence de discours institutionnel sur ce sujet (p. ex., syndicats agricoles, ministère). Les travaux de recherche menés en France depuis 5 ans sur le sujet ainsi que la dynamique citoyenne et entrepreneuriale actuelle esquiscent potentiellement un changement vers une meilleure (re-)connaissance de l'urine (« urine awareness », Esculier 2018)

(ii) Le manque de connaissance sur les différents impacts négatifs liés à l'utilisation d'urinofertilisants et en particulier sur les micropolluants organiques. Une partie de ces freins peuvent être levés par des projets de recherche.

(iii) L'insertion des urinofertilisants dans les itinéraires techniques et la possibilité d'épandre des volumes importants en terme d'organisation et de matériel disponible. Dans le cas particulier de l'Ile-de-France, l'élevage étant peu présent, peu de machines permettent actuellement d'épandre des urinofertilisants non concentrés sur les cultures. Dans le cadre de la mise en place de filières de valorisation, des subventions à l'équipement pourraient être proposées. Brun (2018) note que les urinofertilisants liquides semblent moins appréciés en raison des contraintes de stockage et des volumes importants à apporter.

(iv) Le nombre élevé d'acteurs impliqués dans les filières de valorisation entre la ville et l'agriculture (Meynard et al. 2013; Crolais et al. 2016). Une démarche de co-conception avec ces différents acteurs (p. ex., syndicats d'assainissement, agriculteur) pourrait être mise en place (Prost et al. 2017, Réseau IDEAS).

(v) Une problématique d'échelle et un effet de seuil pour le lancement des filières. Par exemple, un quartier de 1000 habitants permet seulement de substituer une petite partie de l'azote minéral sur une ferme céréalière typique conventionnelle en Ile-de-France (voir chapitre 6).

(vi) L'absence de prise en compte claire des urinofertilisants dans la réglementation. Brun (2018) a relevé l'importance des statuts réglementaires des urinofertilisants dans leur acceptabilité par les agriculteurs. Différentes options peuvent être envisagées. Dans l'approche "déchets", le coût du traitement est supporté par le "producteur" des urinofertilisants comme pour le traitement des eaux usées. Comme pour les boues d'épuration, les urinofertilisants pourraient alors être épandus en agriculture aux frais de la collectivité (zéro euro rendu racine) en contrepartie du service rendu par l'agriculture. Une deuxième option est l'approche « produit ». Étant donné que certains traitements permettent de réduire la concentration des contaminants et de garantir une teneur en éléments nutritifs, les urinofertilisants pourraient être vendus aux agriculteurs en tant que fertilisants normalisés. L'urine nitrifiée et concentrée sous le nom « aurin » a ainsi obtenu une autorisation de mise sur le marché en tant que produit en Suisse (www.vuna.ch/). Les urinofertilisants ne font pas explicitement partie des intrants autorisés en agriculture biologique (Union européenne 2018, règlement 2018/248). Leur certification permettrait un gage de qualité sur leur innocuité et l'introduction d'une source de phosphore d'origine organique, l'agriculture biologique reposant significativement sur du phosphore d'origine conventionnelle (Nowak et al. 2013).

(vii) L'absence de traduction économique des externalités des modes actuels de fertilisation azotée et de gestion de l'urine humaine. Les modèles économiques qui permettraient la mise en œuvre de filières de valorisation d'urinofertilisants sont possibles mais délicats à bâtir dans le contexte actuel

(Crolais et al. 2016). Alors que l'analyse de cycle de vie réalisée montre un bilan environnemental globalement meilleur avec l'utilisation d'urinofertilisants, ces bénéfices ne sont pas nécessairement intégrés dans le modèle économique actuel. Les filières peuvent toutefois se mettre en œuvre sur des niches économiques ou avec l'appui d'acteurs engagés.

De nombreuses autres études ont été conduites sur l'acceptabilité d'utiliser l'urine en tant que fertilisant par les agriculteurs et consommateurs dans différents contextes (Lienert et Larsen 2010; Simha et al. 2017; Simha et al. 2018; Wilde et al. 2019; Segrè Cohen et al. 2020). Ces études ont mis en évidence une acceptabilité plutôt élevée à la fois de la part des agriculteurs et des consommateurs. Le retour au sol de l'engrais humain reste toutefois majoritairement étudié *a priori*. Le dégoût, ou *a minima* l'impensé actuel lié à l'urine, n'est pas anodin dans la mise en place de filières. Les modalités de (ré-)appropriation de cette rétro-innovation restent encore largement à explorer. Selon le contexte, les savoirs et les imaginaires des communautés concernées et les voies de développement de l'usage des urinofertilisants peuvent être extrêmement variées (Legrand 2020). La question des odeurs, aux différents maillons des filières de valorisation, est un point de vigilance à ne pas négliger non plus.

Enfin, nous pouvons observer une forte dynamique en région parisienne ces dernières années sur la mise en place de filières de valorisation de l'urine en agriculture. Cette dynamique inclut de nombreux acteurs : organismes de recherche, bureaux d'étude, municipalités, chambre d'agriculture, etc. L'Agence de l'Eau Seine-Normandie, en particulier, a intégré dans son programme d'intervention la possibilité de subventionner jusqu'à 80% les projets de séparation à la source de l'urine. Plusieurs projets de quartiers ou d'immeubles équipés de toilettes à séparation à la source de l'urine sont actuellement étudiés. Le développement de cas pilotes pourrait œuvrer à lever les verrous sociotechniques identifiés.

2.3. Passage à l'échelle territoriale

L'analyse du cycle de vie conduite a permis d'évaluer les impacts de la production de céréales en utilisant différents urinofertilisants. Cependant, afin d'avoir une vue plus globale d'une possible valorisation de l'urine, des scénarios de territorialisation seraient intéressants pour quantifier les ressources disponibles sur un territoire et pour étudier leurs insertions dans les pratiques culturales du territoire. Plusieurs travaux proposent déjà des scénarios territoriaux permettant de boucler une partie des cycles biogéochimiques.

La première perspective est une perspective de substitution aux engrains minéraux. Au niveau mondial, Trimmer et al. (2019) indiquent que l'azote contenu dans les excrétats humains (urine + matières fécales) permettrait de remplacer entre 16 et 21% des engrains azotés et 9 à 12% des engrains phosphatés. À l'échelle de la Suède, Akram et al. (2019) indiquent que le recyclage des excrétats humains pourrait remplacer 21% des engrains azotés et 16% des engrains phosphatés. À un niveau plus local, par exemple au niveau de l'Ile-de-France, les flux d'azote et de phosphore contenus dans les

excrétas calculés par Esculier et al. (2018) représentent respectivement 140% et 75% des livraisons d’engrais azotés et phosphatés de la région (UNIFA 2018). Cette forte couverture des besoins est due à une forte concentration humaine (métropole de 12 millions d’habitants) et une surface agricole proportionnellement limitée. Ainsi, de fortes différences entre les zones rurales et urbaines existent. Les régions urbaines étant excédentaires en nutriments, des transferts vers des régions moins peuplées ou plus demandeuses d’engrais sont nécessaires pour être à l’équilibre (Akram et al. 2019). Les exemples passés de valorisation de l’urine humaine montrent toutefois une co-évolution des territoires urbains et agricoles : ceinture maraîchère de Paris (Philipponneau 1956), ceintures maraîchères en Chine (King 1911), appropriation spécifique par l’agriculture flamande au XIX^{ème} siècle (Mathey 2020), etc.

La simple substitution d’une fraction des engrais minéraux par des urinofertilisants est toutefois problématique à de nombreux égards. En effet, la majorité des systèmes alimentaires actuels présentent une très forte empreinte environnementale, les rendant non soutenables, et la simple substitution partielle par des urinofertilisants ne permettrait pas de réduire cette empreinte (émissions de gaz à effet de serre, contamination des milieux aquatiques et des nappes en nutriments et en phytosanitaires, etc.). Différents scénarios de changement des systèmes agricoles et alimentaires ont été proposés et comprennent notamment une réduction importante ou l’arrêt de l’utilisation des engrais minéraux et des changements dans le régime alimentaire (Billen et al. 2012; Solagro 2016; Poux et Aubert 2018). Cependant, ces scénarios n’ont pas considéré une possible utilisation de l’urine humaine en tant que fertilisant. Des scénarios incluant, de façon systémique, une co-évolution des pratiques agricoles, des régimes alimentaires et des modalités de gestion de l’urine humaine ont été réalisés à l’échelle de l’Île-de-France (Esculier 2018) ou à l’échelle du plateau de Saclay (Verger et al. 2016). Ils montrent la possibilité de réduire très sensiblement l’empreinte environnementale des systèmes alimentation/excrétion actuels.

Dans la suite de ce travail et toujours dans le cadre du projet Agrocapi, des scénarios territoriaux de gestion des nutriments doivent être menés. L’objectif est à la fois d’approfondir les scénarios au niveau de l’exploitation agricole pour l’insertion des urinofertilisants dans les itinéraires techniques, le matériel agricole utilisé ou le temps de travail nécessaire selon les différents types de production agricole et les ressources en urinofertilisants potentiellement disponibles, le tout en fonction de la zone géographique considérée.

Finalement, la mise en place de filières de valorisation de l’urine humaine en agriculture peut participer d’une réinterrogation en profondeur du métabolisme des territoires urbains et ruraux. Dans un contexte de crise environnementale marquée par le dépassement de nombreuses limites planétaires, des transformations substantielles du mode de fonctionnement de nos sociétés apparaissent souhaitables et nécessaires. Mettre en œuvre une circularité matérielle, permettant de retourner aux sols agricoles les nutriments contenus dans l’urine humaine, semble en être une contribution

pertinente. Au-delà, il convient toutefois de s'interroger sur les évolutions systémiques dans lesquelles la valorisation d'urinofertilisants peut s'inscrire. Dans les habitats, la gestion de l'urine doit être pensée en cohérence avec la gestion des matières fécales, des biodéchets, de l'approvisionnement en eau et du devenir des eaux ménagères, etc. : aux champs, l'intégration d'urinofertilisants doit être pensée en cohérence avec la gestion de l'eau, du sol, de la biodiversité, etc. Des champs aux habitats et des habitats aux champs, il s'agit d'interroger les modalités matérielles, sociales et économiques de ces échanges, la forme et l'organisation des territoires. En recréant un mutualisme entre territoires urbains et ruraux, la valorisation de l'urine humaine en agriculture peut finalement constituer un maillon d'une authentique transition socio-écologique.

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Titre : L'urine humaine en agriculture : des filières variées pour contribuer à une fertilisation azotée durable

Mots clés : Urine humaine ; Séparation à la source ; Fertilisant ; Filière de valorisation ; Essais agronomiques ; Impacts environnementaux

Résumé : Pour nourrir les humains, l'agriculture actuelle est fortement tributaire de l'utilisation de fertilisants issus de ressources fossiles. Or la majorité des nutriments de l'alimentation est ensuite excrétée dans les urines. Celles-ci sont usuellement mélangées aux eaux usées dont la gestion ne permet qu'un faible recyclage de ces nutriments et entraîne de nombreux impacts environnementaux. L'objectif de cette thèse est de caractériser les filières envisageables de valorisation de l'urine humaine en agriculture au niveau agronomique et de leurs impacts environnementaux. Une revue de la littérature des différents traitements de l'urine et des urinofertilisants obtenus montre que : (i) l'efficacité agronomique de la plupart des urinofertilisants est haute et nécessite d'être davantage étudiée ; (ii) la majorité des pathogènes peuvent être facilement inactivés, les résidus de pharmaceutiques sont plus difficilement dégradés ; (iii) la consommation d'énergie et de réactifs des traitements peut être élevée. L'efficacité fertilisante d'une dizaine d'urinofertilisants a ensuite été mesurée au champ et en serre. Elle est élevée pour la majorité et proche de celle

des engrains minéraux (équivalence engrais de 52% à 120%). Elle est liée à une forte teneur en azote minéral dans la majorité des urinofertilisants. La volatilisation ammoniacale peut potentiellement être importante (e.g. 34% de l'azote en conditions propices), le pH élevé et la teneur en azote ammoniacal, selon les urinofertilisants, étant des facteurs de risque importants. Enfin, une évaluation par analyse du cycle de vie des impacts environnementaux associés à la production de céréales a été réalisée selon le mode de fertilisation : biologique et conventionnelle versus trois urinofertilisants. Les impacts sont plus faibles pour la majorité des indicateurs en comparaison aux pratiques actuelles, en grande partie grâce aux impacts évités de l'épuration des eaux usées et de la production d'engrais minéraux. La volatilisation ammoniacale et la consommation d'énergie des traitements sont les deux éléments les plus sensibles du bilan environnemental. Ces résultats montrent que le déploiement de filières de valorisation de l'urine humaine peut contribuer à une transition vers une gestion systémique et soutenable des nutriments.

Title : Human urine in agriculture : various management options contributing to sustainable nitrogen fertilization

Keywords : Human urine ; Source separation ; Fertilizer ; Management option ; Agronomic trials ; Environmental impacts

Abstract: To feed humans, agriculture mostly relies on the use of fertilizers derived from fossil resources. Yet, most nutrients from food are excreted in urines and mixed in wastewaters. Wastewater treatment allows only a weak recycling of the nutrients and has many environmental impacts. The objective of this thesis is to characterize the possible management options for the use of human urine in agriculture considering their fertilizing efficiency and their environmental impacts. A literature review of the various urine treatments and urine-based fertilizers shows that: (i) the fertilizing efficiency of most urine-based fertilizers is high but needs to be further studied; (ii) most pathogens in urine can be easily inactivated but pharmaceutical residues are more difficult to degrade; (iii) the energy and chemical consumption of treatments can be high. The fertilizing efficiency of ten urine-based fertilizers has been measured under greenhouse and field conditions. It is high for most of the urine-based fertilizers and close to that of mineral fertilizers (equivalent ranging

from 52% to 120%). It is linked to high mineral nitrogen content in the majority of urine-based fertilizers. Ammonia volatilization after field application can potentially be high (e.g. 34% of total nitrogen in favorable conditions). High pH and ammoniacal nitrogen content according to the urine-based fertilizers are important risk factors. Finally, a life cycle assessment of the environmental impacts for cereal production was carried out considering three urine-based fertilizers and two agricultural systems (conventional and organic). The impacts are lower for the majority of the indicators compared to the current practices. It is mainly due to the avoided impacts due to wastewater treatment and mineral fertilizers synthesis. Ammonia volatilization and the energy consumption of the treatments appear as the main environmental hotspots. These results show that the implementation of human urine management options can contribute to a transition towards a more sustainable and systemic management of nutrients.