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Alkaline Urine Dehydration

How to dry source-separated human urine and recover nutrients?

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Cover: A urine-diverting toilet integrated with an alkaline urine dehydrator.
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Abstract

Human urine is a renewable resource from which water, nutrients and energy can be recovered and safely recycled. This thesis presents a novel on-site technology, called alkaline dehydration, for recovering nutrients from source-separated urine.

To recover urea (the major nitrogenous compound in fresh urine) and prevent its urease enzyme-catalysed hydrolysis to ammonia, fresh urine was alkalised to pH ≥10 by anion exchange or use of alkaline substrates. To reduce the volume and concentrate the nutrients, the alkalised urine was dried in different substrates at temperatures of 40-60 °C. To evaluate alkaline urine dehydration at pilot-scale, a prototype dryer with capacity to treat 30 L urine d^{-1} was built and field-tested for three months at a military camp in Finland.

More than 90% of the urine mass was reduced and dehydration rates of up to 40 L urine d^{-1} m^{-2} were obtained. Up to 98% of the nitrogen (N) and 100% of the phosphorus (P) and potassium (K) in urine were recovered. A dry solid containing 10% N, 1.5% P and 6.0% K, with similar salt and heavy metal content to synthetic fertilisers available in Sweden was produced.

At least 12.5 g $Ca(OH)_2$ or 6.8 g MgO per litre of fresh urine was required to buffer the pH of urine against absorption of CO_2 during dehydration. The energy demand for evaporating urine was similar to that of incineration toilets, but could be reduced if heat energy from the dryer's exhaust air were recovered.

Overall, this thesis shows that a new sanitation system that safely collects, treats, transports and applies urine as fertiliser could be created by integrating alkaline urine dehydration with existing infrastructure. If implemented globally, this system could recycle 31 Tg N y^{-1} and 2.8 Tg P y^{-1}, which would reduce the transgression of the planetary boundary for N and P by 35% and 25%, respectively.

Keywords: Circular Bioeconomy; Fertiliser; Resource recovery; Safe Nutrient Recycling; Sanitation; Source separation; Urine diversion; Wastewater treatment

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Alkalisk urinuttorkning

Sammanfattning

Humanurin är en förnybar resurs ur vilken vatten, näringsämnen och energi kan återvinnas. I denna avhandling presenteras en ny teknik, alkalisk urintorkning, för utvinning av näringsämnen från källsorterad urin.

För att återvinna urea (den huvudsakliga kväveföreningen i färsk urin), och förhindra ureasenzymkatalyserad hydrolys till ammoniak, höjdes pH i färsk urin till pH >10 genom anjonbyte eller tillsats av alkaliska substrat. För att minska volymen och koncentrera näringsämnena torkades alkaliserad urin i olika substrat vid 40–60 °C. En prototyp med kapacitet att behandla 30 liter urin d^{-1} byggdes för att utvärdera systemet i större skala, vilket gjordes under tre månader vid en militäranläggning.

Urinmassan minskade med över 90% och torkningshastigheter på upp till 40 liter urin d^{-1} m^{-2} påvisades. Upp till 98% av kväve (N) och 100% av fosfor (P) och kalium (K) från urinen återfanns in det torkade materialet, som innehöll 10% N, 1,5% P och 6,0% K, samt salt och tungmetallhalter jämförbara med de i konstgödsel som används i Sverige idag. Minst 12,5 g $Ca(OH)_2$ eller 6,8 g MgO L^{-1} färsk urin krävdes för att buffra pH i urinen tillräckligt för att motverka absorptionen av CO_2 under torkningsprocessen. Urintorkningen krävde lika mycket energi per kg urin som en förbränningstoalett, men energibehovet skulle kunna minskas med en värmeväxlare som tog tillvara värmeenergin i frånluften.

Sammantaget visar resultaten att ett nytt sanitetssystem för återvinning av resurser i urin skulle kunna utformas genom att integrera alkalisk urintorkning med befintlig infrastruktur. Om detta system skulle implementeras globalt skulle 31 Tg N $år^{-1}$ och 2.8 Tg P $år^{-1}$ återvinnas och det skulle minska dagens överskridning av de planetära gränserna för N och P med 35%, respektive 25%.

Keywords: cirkulär bioekonomi; gödselmedel; resursåtervinning; säker återföring av näringsämnen; sanitet; källsortering; urinsortering; avloppsrening

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Contents

List of publications

This thesis is based on the work contained in the following papers, referred to by Roman numerals in the text:

I. **Simha, P.**, Senecal, J., Nordin, A., Lalander, C. & Vinnerås, B. (2018). Alkaline dehydration of anion–exchanged human urine: Volume reduction, nutrient recovery and process optimisation. *Water Research* 142, 325-336.

II. **Simha, P.**, Lalander, C., Nordin, A. & Vinnerås, B. (2020). Alkaline dehydration of source-separated fresh human urine: Preliminary insights into using different dehydration temperature and media. *Science of the Total Environment* 733, 139313.

III. **Simha, P.**, Friedrich, C., Randall, D.G. & Vinnerås, B. (2021). Alkaline dehydration of human urine collected in source-separated sanitation systems using Magnesium Oxide. *Frontiers in Environmental Science* 8, 619901.

IV. **Simha, P.**, Karlsson, C., Viskari, E.-L., Malila, R. & Vinnerås, B. (2020). Field testing a pilot-scale system for alkaline dehydration of source-separated human urine: a case study in Finland. *Frontiers in Environmental Science* 8, 570637.

V. **Simha, P.**, Senecal, J., Gustavsson, D.J. & Vinnerås, B. (2020). Resource recovery from wastewater: a new approach with alkaline dehydration of urine at source. In *Current Developments in Biotechnology and Bioengineering,* 205-221. Elsevier, Netherlands.

Papers I-V are reproduced with the permission of the publishers.
The contribution of Prithvi Simha to the papers was as follows:

I. Simha and Vinnerås planned the study. Simha performed the laboratory work. Senecal performed pre-trials with the anion-exchanger. Simha carried out the formal data analysis with inputs from Lalander and Nordin. Simha wrote the paper, with revisions by the co-authors.

II. Simha and Vinnerås planned the study. Simha performed the laboratory work. Simha and Lalander carried out the formal data analysis. Simha wrote the paper with revisions by the co-authors.

III. Simha, Friedrich, and Vinnerås planned the study. Friedrich and Simha performed the laboratory work. Randall performed the chemical speciation modelling. Simha and Friedrich did the formal data analysis. Simha wrote the paper with revisions by the co-authors.

IV. Simha, Viskari, and Vinnerås planned the study. Simha and Karlsson built and installed the pilot-scale dehydrator. Simha, Karlsson and Viskari performed the monitoring, data collection and formal analysis, with inputs from Vinnerås and Malila. Simha wrote the paper, with revisions by the co-authors.

V. Simha, Senecal, and Vinnerås conceptualised the book chapter. Simha led the writing and produced the first draft. Senecal wrote the section on hygiene, Gustavsson wrote the section on wastewater treatment plants, and Vinnerås wrote the section on micropollutants.

Papers produced but not included in this thesis:

VI. **Simha, P.,** Lalander, C., Vinnerås, B. & Ganesapillai, M. (2017). Farmer attitudes and perceptions to the re–use of fertiliser products from resource–oriented sanitation systems–The case of Vellore, South India. *Science of the Total Environment* 581, 885-896**.**

VII. **Simha, P.,** Lalander, C., Ramanathan, A., Vijayalakshmi, C., McConville, J.R., Vinnerås, B. & Ganesapillai, M. (2018). What do consumers think about recycling human urine as fertiliser? Perceptions and attitudes of a university community in South India. *Water Research* 143, 527-538.

VIII. Senecal, J., Nordin, A., **Simha, P.** & Vinnerås, B. (2018). Hygiene aspect of treating human urine by alkaline dehydration. *Water Research* 144, 474-481.

IX. **Simha, P.,** Barton, M.A, Perez-Mercado, L.F., McConville, J.R. … & Vinnerås, B. (2021). Willingness among food consumers to recycle human urine as crop fertiliser: Evidence from a multinational survey. *Science of the Total Environment* 765, 144438.

X. Barton, M.A, **Simha, P.**, Magri, M.E., Dutta, S. … & Vinnerås, B. (2021). Attitudes of food consumers at universities towards recycling human urine as crop fertiliser: A multinational survey dataset. *Data in Brief* 35, 106794.

Popular science articles based on this thesis:

XI. **Simha, P.,** Vinnerås, B. & Senecal J. We found a way to turn urine into solid fertiliser – it could make farming more sustainable. *The Conversation UK. November 6, 2020.*

XII. **Simha, P.,** Buckley, C. & Senecal, J. We developed a simple process to recycle urine. Here’s how it’s done. *The Conversation Africa. November 18, 2020.*

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1. Introduction

In September 2000, the leaders of 189 countries signed the United Nations (UN) Millennium Declaration, in which they committed to a new global partnership to achieve a set of eight measurable and universally agreed goals – popularly known as the MDGs (UNGA, 2000). Among these was the target to halve, by 2015, the proportion of people without access to basic sanitation. However, more than half of all the low- and middle-income countries for which data are available missed this target. In 2015, the MDGs were superseded by the Sustainable Development Goals (SDGs), with SDG 6 setting the target of achieving, by 2030, "clean water and sanitation for all". In addition, the goal seeks to "halve the proportion of untreated wastewater and substantially increase recycling and safe reuse globally". However, according to the latest report from the UN, the global progress on SDG 6 is alarmingly off track – in 2017, 2.2 billion people still lacked safely managed drinking water and 4.2 billion lacked safely managed sanitation, of which 2 billion were without basic sanitation and 673 million practiced open defecation (UN, 2020). If matters continue as usual, then the SDG 6 targets are likely to be missed, just like the MDGs before them.

Many experts are of the opinion that the slow progress on the sustainability goals is partly because of the old engineering conventions that have dominated the water and wastewater management industry for the past 100 years (Hoffmann *et al.*, 2020; McConville *et al.*, 2017b; Larsen *et al.*, 2013; Guest *et al.*, 2009; Larsen *et al.*, 2009; Langergraber & Muellegger, 2005; Jönsson *et al.*, 2004). Under these conventions, domestic wastewater is removed from households and sent to a centralised treatment plant, where it can be treated to remove pollutants and then discharged to the environment (Daigger, 2009). However, even the most advanced treatment plants in the world today cannot remove all pollutants, especially nitrogen. Nitrogen (N) is in fact a plant-essential nutrient that accumulates in water bodies, where it can contribute to hypoxia and eutrophication (Conley *et al.*, 2009). Technologies that can reduce the concentration of nutrients (nitrogen and phosphorus) in wastewater are expensive and beyond the reach of low- and middle-income countries (Larsen, 2020; Sato *et al.*, 2013), and are projected to remain so for the next few decades (van Puijenbroek *et al.*, 2018).

A different approach to wastewater management considers wastewater as a renewable resource (Guest *et al.*, 2009), from which water (Reynaert *et al.*, 2020), nutrients (Winker *et al.*, 2009), and energy (Hao *et al.*, 2019) can be recovered and recycled. It is true that many wastewater treatment plants, especially in high-income countries, are increasingly integrating processes that can recover nutrients, *e.g.* chemical phosphorus (P) precipitation (Perera *et al.*, 2019). However, the recovery rate of all other nutrients is low, because centralised treatment plants were originally designed to promote urban hygiene (Wilsenach *et al.*, 2003).

In the early 1990s, in line with growing concerns over environmental sustainability, parallel work began in Sweden and Switzerland on the concept of source separation of wastewater (Jönsson *et al.*, 1997; Jönsson *et al.*, 1996; Larsen & Gujer, 1996). The underlying principle is that, different fractions of wastewater, each with its own distinct composition are produced separately at source (household), but are mixed before treatment (Friedler *et al.*, 2013; Vinnerås *et al.*, 2006). If these fractions could be collected separately, then they could be treated differently (Larsen *et al.*, 2013). Ultimately, source separation would open up wastewater treatment to a new suite of technologies that could help recover and recycle resources (Guest *et al.*, 2009; Vinnerås & Jönsson, 2002).

One fraction of wastewater that has received much research attention since the very beginning of research in source separation is human urine (Jönsson *et al.*, 1997; Larsen & Gujer, 1996). This is because human urine contributes just 1% of the volume but 80% of the nitrogen and 50% of the phosphorus in domestic wastewater (Vinnerås *et al.*, 2006). In the three decades since source separation of urine was first proposed (1990-2020), research on different aspects of recycling urine has been performed. Large transdisciplinary projects like EcoSanRes (Esrey *et al.*, 1998) and Novaquatis (Larsen & Lienert, 2007) have helped to develop innovations like urine-diverting toilets and guidelines on how urine should be collected, stored, and sanitised, for safe use as fertiliser (Jönsson *et al.*, 2004). As a result, urine recycling has become popular at eco-villages and summerhouses (McConville *et al.*, 2017a), and has even been adopted by some municipalities, such as Tanum in Sweden (Kvarnström *et al.*, 2006) and Durban in South Africa (Mkhize *et al.*, 2017). However, it has not yet been mainstreamed and has failed to achieve large-scale implementation.

One reason why recycling urine has not become more popular is that realising it in practice means that large volumes of urine have to be collected and stored at households (550 kg per capita and year according to Vinnerås *et al.* (2006)), and eventually transported and applied to farmland (Larsen & Gujer, 2013). However, as urine consists mostly of water (Friedler *et al.*, 2013), to apply 90 kg N ha^{-1}, around 15 000 kg of urine must be applied, whereas only 200 kg of synthetic fertiliser (*e.g.* urea) are required. Therefore, research in the past 10 years has attempted to develop technologies that can either reduce the volume of urine or recover the valuable resources it contains. Excellent overviews of these technologies can be found in Harder *et al.* (2019) and Martin *et al.* (2020). However, most of these technologies have failed to emerge from the laboratory, perhaps because implementing them in practice and integrating them with existing infrastructure has proven difficult. This thesis aimed at addressing this gap by developing an on-site urine treatment technology that could make recycling of urine a more attractive proposition in practice.

2. Aims and structure

2.1 Overall aim

The overall aim of this thesis was to investigate the processes involved in treating source-separated human urine by alkaline dehydration.
Specific objectives were to –

- Develop methods to prevent hydrolysis of urea in fresh urine and alkalised urine, and during urine dehydration (Papers I-III)
- Optimise recovery of nitrogen from alkalised human urine during the dehydration treatment (Paper II)
- Determine the fate of different elements and the composition of the dry fertiliser produced by dehydrating urine (Papers I-IV)
- Analyse the factors influencing dehydration rate and volume reduction of urine (Papers I-IV)
- Evaluate aspects affecting the scalability and implementation of alkaline urine dehydration technology in practice (Papers IV & V)

2.2 Thesis structure

Of the five papers on which this thesis is based, Papers I-IV are experimental studies investigating alkaline dehydration at laboratory scale (Papers I-III) and pilot scale (Paper IV), while Paper V describes the overall concept of the technology and offers arguments in support of integrating it with existing infrastructure. Paper V also suggests possible ways of safely collecting, drying, transporting and applying human urine as fertiliser.

Overall, two main research topics were investigated in this thesis. The first topic was *alkalisation,* in which the pH of fresh human urine (which is usually below 7) was increased to ≥10 to inactivate the enzyme urease and thus prevent urea hydrolysis. To study this, urine was either passed through an anion-exchanger (Paper I) or added to different alkaline substrates (Paper II-IV). The second topic was *dehydration*, in which nutrients present in alkalised human urine were concentrated by evaporating water, ultimately producing a solid fertiliser. To study this, setups for drying urine were designed, built and tested at different scales.

In all experiments, the primary focus was to recover nutrients from urine, especially nitrogen and, to monitor changes in the physicochemical properties of urine and the drying substrates. In Papers II-IV, the products obtained by drying urine were also characterised in terms of their macronutrient, micronutrient, and heavy metal content. In Papers IV and V, the practical considerations in implementing alkaline urine dehydration, including the energy demand for treating urine, were assessed. Those papers include a brief discussion on substances that could pose concerns as regards human and environmental health, such as heavy metals, micropollutants and salts, but they were not the main focus of the work.

3. Background

3.1 Food security

Food security has been one of humanity's age-old preoccupations. However, in the 21st century, the threats to the global food system are very different from those in the past. According to projections made by the United Nations (UN, 2019), the global population will surpass 11 billion people by the year 2100. A clear implication of this is that, to meet the demands of a growing population, production and supply of food will have to increase over the coming decades (FAO, 2017a). However, this is an immense challenge because more than 820 million people worldwide were still hungry and undernourished in 2018 (FAO, 2019). At the same time, soil productivity is declining across the world (UNCCD, 2017) and a changing climate is expected to reduce crop yields and increase the price of key food crops (Nelson, 2010).

Unlike in previous times, it is no longer possible to increase food production simply by bringing more land under cultivation, because changing land use severely degrades environmental conditions and ecosystem services (Foley *et al.*, 2005). In fact, the global food system is already the single largest contributor to anthropogenic transgression of several of the planetary boundaries (Gladek *et al.*, 2017), including those on biogeochemical flows of nitrogen and phosphorus. Agriculture disrupts the natural cycles of nitrogen and phosphorus (Campbell *et al.*, 2017), as they are chemically synthesised or extracted from reserves to produce fertilisers. Since plant growth is most limited by the concentrations of these two elements in soil, applying them as fertiliser increases crop yield. It is estimated that synthetic nitrogen fertilisers produced by the Haber-Bosch process support nearly half of the world's population today (Erisman *et al.*, 2008). However, the Haber-Bosch process, in which atmospheric nitrogen gas (N_2) is converted to ammonia (NH_3), is energy-intensive (primary energy demand 43.7 MJ kg_N^{-1} according to Maurer *et al.* (2003)) and accounts for 1-2% of the world's energy consumption (IFIA, 2014). Another issues is that, the phosphate rock required to produce phosphorus fertilisers, is a non-renewable resource that is concentrated to a few countries (Van Vuuren *et al.*, 2010).

The Haber-Bosch process has been optimised over the years, but the subsequent use of nitrogen fertilisers in agriculture involves large losses. Through the so-called nitrogen cascade effect (volatilisation, leaching, surface and subsurface runoff) and owing to food losses during harvesting, transport and consumption, only 4-14% of reactive nitrogen (NH_3/NH_4^+, NO_3^-, N_2O) used in agriculture ends up in the human body as consumed protein (Pikaar *et al.*, 2017). Almost half of the nitrogen is lost during fertiliser application, which can have negative effects on human and environmental health by contributing to eutrophication of waters (Diaz & Rosenberg, 2008), acidification of soils and waters (Guo *et al.*, 2010) and, as a consequence, loss of biodiversity (Campbell *et al.*, 2017). According to Steffen *et al.* (2015), the current flux of nitrogen and phosphorus to surface water already greatly exceeds its so-called planetary-level safe space. This means that if humankind continues down the same path, there is high risk of functional collapse of key Earth system processes (Rockström *et al.*, 2009). According to projections made by FAO (2017a), however, there will still be a 50% increase in demand for nitrogen fertiliser by 2050. Therefore, humanity faces one of its greatest ever food security challenges – matching the increasing food demands of a growing world population while reshaping the nitrogen and phosphorus cycles to operate within the planetary-level safe space that existed prior to introduction of the Haber-Bosch process.

3.2 Sanitation systems

Water is indispensable for life on earth. At the household level, when clean water is mixed with pollutants, wastewater is produced. Wastewater can be generally classified as blackwater, which is toilet wastewater, *i.e.* urine, faeces, flush water and/or toilet paper, and greywater, which is untreated household wastewater from baths, sinks, laundry, and kitchen appliances that has not been contaminated by toilet waste (WHO, 2006). In an urban residential setting, greywater accounts for the majority of the volume of wastewater produced (Oteng-Peprah *et al.*, 2018), with blackwater making up less than one-third of the total volume. Blackwater and greywater are usually collected together, either in sewers that transport the mixed wastewater to a communal treatment plant or in septic tanks or pit latrines. It is estimated that 80% of the wastewater produced globally receives no treatment (UNESCO, 2017). If wastewater ends up at a treatment plant, it is converted into an effluent that can be discharged into water bodies after removing suspended solids, oxygen-consuming substances and, to a certain extent, nutrients like nitrogen and phosphorus.

Historically, wastewater treatment in cities was introduced in a stepwise manner, starting with primary treatment (removal of suspended solids by sedimentation), followed by secondary treatment (degradation of organic matter and oxidation of ammonia to nitrate by aerobic biological processes), and finally, tertiary treatment (biological removal of nitrogen and biological or chemical removal of phosphorus) (Naturvårdsverket, 2018). At present, tertiary wastewater treatment is carried out only in high-income countries (Sato *et al.*, 2013), and a dramatic increase in global tertiary treatment capacity is highly unlikely (UNESCO, 2017). According to Eurostat (2020), only 54% of the population in the European Union in 2017 were connected to wastewater treatment plants with tertiary treatment.

The overall trend in wastewater management is still to increase sewage connection rates and primary/secondary treatment capacities in low- and middle-income countries, while upgrading to tertiary treatment in high-income countries. According to estimates by van Puijenbroek *et al.* (2018), in the coming three decades the number of people connected to sewage systems will increase by 2-4 billion, accompanied by a 10-40% increase in global wastewater treatment capacity. If that is the case, the targets set out in Sustainable Development Goal 6.3 will only be met by 2050. Moreover, equipping the entire world population with a sewer system connected to advanced wastewater treatment plants would incur costs of the magnitude of trillion US$/year, and this development would therefore be prohibitively expensive (Larsen, 2020).

3.3 Source separation

In the early 1990s, many experts started to question the conventional concept of wastewater treatment and began exploring the idea of mainstreaming source separation in urban wastewater management. The overall concept of source separation is simple – various wastewater fractions have different compositions, (Vinnerås *et al.*, 2006) and different potential benefits and risks (Winker *et al.*, 2009), so if they could be collected separately, they could be treated differently (Larsen *et al.*, 2013; Vinnerås & Jönsson, 2002). For instance, about 40 L blackwater p^{-1} d^{-1} containing on average 600-1000 mg N L^{-1} and 90-140 mg P L^{-1} is produced (Friedler *et al.*, 2013; Kujawa-Roeleveld & Zeeman, 2006). In contrast, 300 L $p^{-1}d^{-1}$ of mixed wastewater entered Swedish municipal wastewater treatment plants in 2018, with an average concentration of 40 mg N L^{-1} and < 5 mg P L^{-1} (Statistics Sweden, 2020). While treatment plants in Sweden removed 95% of the incoming phosphorus (Naturvårdsverket, 2018), they removed only 60% of the nitrogen (40% of which was converted to N_2) and just 6% of the potassium (K) (Dagerskog & Olsson, 2020). Around 15 400 tonnes of nitrogen and 240 tonnes of phosphorus were released from Swedish treatment plants to water recipients in 2016 (Statistics Sweden, 2018).

Proponents of source separation argue that the opportunities to reuse domestic wastewater are limited by the current infrastructure, which dilutes and removes nutrients (Guest *et al.*, 2009; Larsen *et al.*, 2009; Vinnerås & Jönsson, 2002). If different fractions of wastewater could be collected separately, then a different set of treatment technologies could be used to recover the nutrients. A growing body of evidence suggests that source separation and decentralised treatment can have several benefits (Larsen *et al.*, 2013). For instance, separately collecting the urine fraction alone can improve the performance and carbon footprint of existing centralised wastewater treatment plants (Wilsenach & Loosdrecht, 2006), reduce freshwater consumption (Mbaya *et al.*, 2017), energy demand (Maurer *et al.*, 2003), and emissions of greenhouse gases (McNicol *et al.*, 2020), offset the need for producing synthetic fertilisers (Hilton *et al.*, 2021; Trimmer *et al.*, 2017), and improve the environmental footprint of food production (Lam *et al.*, 2015; Tidåker *et al.*, 2007).

According to van Puijenbroek *et al.* (2018), around 4 billion people will be connected to new sewage systems in the next 30 years. If these people were to practise urine diversion, up to 15 Tg N y^{-1} and 1.2 Tg P y^{-1} could be recycled, which would reduce the flux of nitrogen and phosphorus in the environment. In Northern Europe, many cities are currently piloting (or are planning) blackwater source separation at neighbourhood scale (>1000 inhabitants) (Skambraks *et al.*, 2017). One reason why blackwater has received much attention is that the technologies for treating it are proven and have already been implemented and demonstrated in different settings. However, the same cannot be said for urine recycling. It has been shown that urine can be collected, stored and used as a liquid fertiliser (Jönsson *et al.*, 2004), but this system has drawbacks (see Section 3.5). Therefore, research has focused on developing technologies to produce urine-based fertilisers (Harder *et al.*, 2019), but these technologies are not yet mature and often fail to emerge from the laboratories where they are conceived (Martin *et al.*, 2020; McConville *et al.*, 2017b).

3.4 Why and how to separate urine at source

Among all the fractions of wastewater that can be collected separately at source, human urine contains the majority of the nutrients. An average adult excretes 550 L urine y^{-1}, containing about 4000 g N, 1000 g K and 365 g P (Vinnerås *et al.*, 2006). From a recycling perspective, urine is attractive as it contributes less than 1% to the total volume of wastewater produced by households, but contains about 80% of the N, 50% of the P and 70% of the K (Friedler *et al.*, 2013). If all the urine produced in the world were to be recycled, around 25% of nitrogen and phosphorus currently used in agriculture as fertiliser could be replaced (Figure 1).

Urine also contains lower concentrations of heavy metals and hormones than found in livestock manure (Jönsson & Vinnerås, 2013). Very few pathogens are excreted via urine but faecal cross-contamination during urine collection can increase the risk of transmission of diseases (Höglund, 2001). A conventional urine-diverting toilet (Tilley *et al.*, 2014) with two separate bowls, one for urine and the other for faeces, can be used for collecting urine. New pedestal designs can even separate urine by using surface tension and a trap that is invisible to the toilet user, making the source-separating toilet appear just like a conventional flushed toilet (Gundlach *et al.*, 2021).

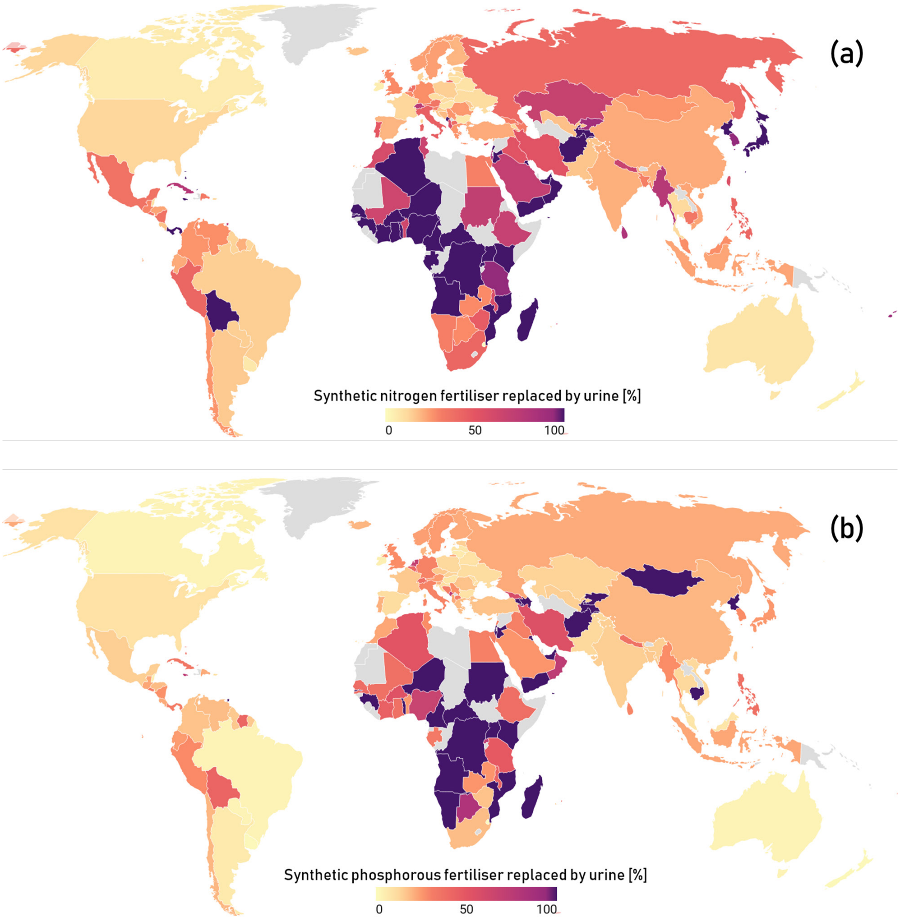

Figure 1. Potential of human urine to replace all (a) nitrogen and (b) phosphorus used in agriculture in different countries. Nitrogen and phosphorus content of urine was calculated for each country by the approach of Jönsson and Vinnerås (2004), using data from FAO (2017b) on new food balances. Population data were sourced from UN (2019) and country-wise data for fertiliser consumption in 2018 from FAOSTAT (FAO, 2017b). The replacement potential is very high in some countries with low fertiliser consumption and high population (*e.g.* 800% for nitrogen in Uganda). For these countries, the values are truncated to 100% for graphical purposes. All data and calculations are shown in Simha (2021).

3.5 Treating source-separated urine

If urine is collected from healthy people, it does not contain any pathogens. However, in source-separating sanitation systems, urine is usually cross-contaminated with faeces (Schönning *et al.*, 2002), which increases the potential risk for transmitting diseases. Urine can be sanitized, for example by storage (Vinneras *et al.*, 2008), after which it can be applied as a liquid fertiliser (Jönsson *et al.*, 2004). However, the water content of urine is very high, 95% according to Friedler *et al.* (2013). For this reason, households that try to recycle urine as fertiliser often report that storing, transporting and spreading it is a complex process (Martin *et al.*, 2020). Transporting urine can also be prohibitively expensive, especially when the distance between cities (where wastewater is produced) and cropland (where nutrients are applied) is large (Trimmer & Guest, 2018). This is a major reason why recycling human urine has not been more popular (Larsen & Gujer, 2013). To overcome this, recent research has focused on developing technologies that either extract nutrients present in urine or reduce its volume, both of which reduce the mass of the recycled products that need to be transported.

3.5.1 Ureolysis

In fresh urine, nitrogen is excreted as urea (82%) and total ammonia nitrogen (TAN = NH_3+NH_4^+; about 7%), phosphorus is excreted as inorganic phosphate (>95%), while potassium is excreted as ions (Friedler *et al.*, 2013; Udert, 2002). Shortly after urine is collected, urea is decomposed to ammonia and bicarbonate (Eq. 1) (Krajewska, 2009), because of the catalytic action of the enzyme urease (urea amidohydrolase, EC 3.5.1.5) . The true product of the enzymatic reaction is ammonium carbamate, which decomposes to ammonia and carbonic acid, independent of the presence of the enzyme (Eq. 2) (Jespersen, 1975). At physiological pH, the carbonic acid proton dissociates (Eq. 3), while ammonia equilibrates with water and becomes protonated, resulting in a net increase in pH (Eq. 4) (Qin & Cabral, 2009).

$$\mathrm{H_2NCONH_2 + H_2O \xrightarrow{\mathit{urease}} H_2NCOOH + \ NH_3} \tag{1}$$

$$\mathrm{H_2NCOOH + H_2O \rightarrow H_2CO_3 + NH_3} \tag{2}$$

$$\mathrm{CO_2 + H_2O \leftrightharpoons H_2CO_3 \leftrightharpoons H^+ + HCO_3^-} \tag{3}$$

$$\mathrm{NH_{3(g)} \leftrightharpoons NH_{3(aq)} + H_2O \leftrightharpoons NH_4^+ + OH^-} \tag{4}$$

Urease is produced in nature by bacteria, fungi, and plants (Mobley & Hausinger, 1989). Irrespective of the source, the overall enzyme structures are widely similar, containing one bi-nickel (Ni(II)) centre per active site, which is always located on the α subunit of the enzyme (Mazzei *et al.*, 2020). While plant and fungal ureases are made of identical subunits assembled as trimers (α_3) or hexamers (α_6), bacterial ureases have three distinct subunits, commonly forming trimers ($(\alpha\beta\gamma)_3$) (Krajewska, 2009). The two Ni(II) ions perform key roles, binding and activating urea and the nucleophile water (Ciurli, 1999). For most ureases, the reaction rate is optimal at pH 7 and temperature 60 °C (Mobley & Hausinger, 1989). The hydrolysis of urea by the enzyme is well described by the Micahelis-Menten equation, with K_M values for bacterial urease ranging from 0.2 to 32 mM urea (Qin & Cabral, 2009). In the absence of urease, urea in a solution at room temperature has a half-life of 3.6 years (Shaw & Bordeaux, 1955), whereas, in its presence, the half-life can be short as 200 μs (Dixon, 1980).

Urease-positive bacteria are widespread in the environment (Qin & Cabral, 2009; Mobley & Hausinger, 1989), so their colonisation of toilets, odour traps and pipes cannot be prevented. Thus, in urine-collecting sanitation systems, enzymatic hydrolysis of urea is also inevitable. Hydrolysis increases the TAN concentration of urine, which shifts the pH from near neutrality to around pH 9 (Udert *et al.*, 2003a). This in turn can induce supersaturation of certain minerals like struvite, calcite, and hydroxylapatite in urine and trigger their precipitation, which causes blockages of traps and pipes (Udert *et al.*, 2003b). The majority of the hydrolysis can be traced back to urease-producing bacteria, which are present in biofilms in pipes and the plumbing system. However, when these ureolysing bacteria are flushed into collection tanks, free urease is released following cell lysis, and urea can continue to be hydrolysed in the collection tank (Udert, 2002).

3.5.2 Speciation of ammonia nitrogen in urine

In a solution in general, ammonia nitrogen exists as dissolved gas (NH_3) and ammonium ion (NH_4^+). In fresh or non-hydrolysed human urine, these two ammonia species usually make up less than 10% of total nitrogen (N_{tot}) and both are detected when urine is analysed for TAN. When urea hydrolysis is complete, TAN accounts for almost all the N_{tot} in urine

(Udert, 2002). As NH_3 is a gas, its aqueous concentration in solution depends on the partial pressure of $NH_{3(g)}$ above the solution (Huang & Shang, 2006), which is why storing urine in closed tanks minimises ammonia volatilisation and the losses of nitrogen (Johansson, 1999). The equilibrium (Eq. 4) between NH_3 and NH_4^+ in urine strongly depends on both pH and temperature (*e.g.* Figure 2).

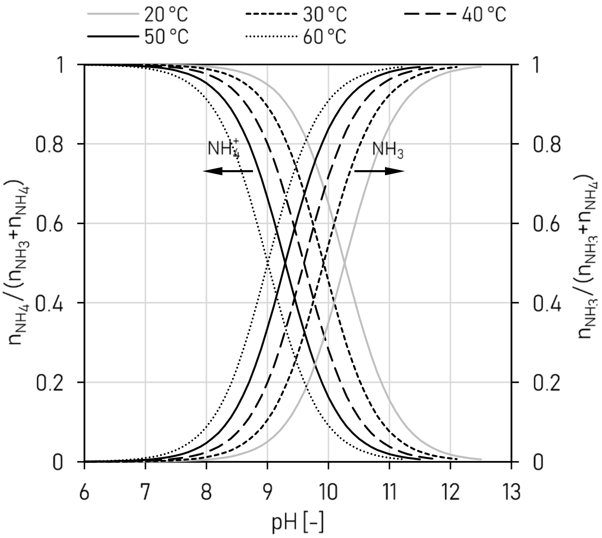

Figure 2. Distribution of ammonium NH_4^+ and ammonia ($NH_{3(aq)}$) in human urine saturated with magnesium hydroxide ($Mg(OH)_2$), followed by addition of calcium hydroxide ($Ca(OH)_2$) to obtain a pH range from 6 to 13 (Paper III).

3.5.3 Treating hydrolysed urine

Since urine naturally undergoes hydrolysis, most technologies proposed in the literature have tried to produce urine-based fertilisers from hydrolysed urine. These technologies have been reviewed by Maurer *et al.* (2006), and more recently by Harder *et al.* (2019) and Martin *et al.* (2020), and are therefore not presented in detail in this thesis. Overall, two strategies for treating urine can be adopted. In the first, water is removed from urine to concentrate its nutrients. This results in production of urine-based fertilisers in the form of concentrated liquids, slurries, or dry powders. Examples include passive evaporation (Pahore *et al.*, 2010), nitrification-distillation (Udert & Wachter, 2012), membrane distillation (Tun *et al.*, 2016) and forward osmosis (Volpin *et al.*, 2019). Apart from nitrification-distillation, most technologies do not recover all the nutrients present in urine, *e.g.* the recovery of nitrogen is usually poor. The second strategy involves selectively recovering nutrients excreted in urine. This can be done by precipitation (Le *et al.*, 2020; Etter *et al.*, 2011), adsorption (Pillai *et al.*, 2014; Ganrot *et al.*, 2007), stripping (Başakçilardan-Kabakci *et al.*, 2007) or ion-exchange (Tarpeh *et al.*, 2017). These treatments also yield products (*e.g.* struvite) with nutrient concentrations higher than those originally present in fresh urine. However, they also generate wastewater streams that need to be treated (before they can be discharged) and do not recover all the nutrients excreted in urine.

3.5.4 Preventing enzymatic urea hydrolysis

Of all the nutrients excreted in urine, recovering nitrogen is the most difficult, especially when the objective of the treatment also includes reducing the volume of urine. In fresh urine, urea undergoes urease-catalysed hydrolytic degradation to ammonia. The high pH and high TAN concentration of urine when it is hydrolysed prevents the recovery of nitrogen when it is dried, since most of the nitrogen can be volatilised as $NH_{3(g)}$ (Bethune *et al.*, 2014; Antonini *et al.*, 2012). One approach to stabilise urine after it has been hydrolysed is partial nitrification, where half of the TAN is converted to non-volatile nitrate, which reduces the pH and stabilises the other half as NH_4^+ (Udert & Wachter, 2012). The nitrified urine can then be concentrated by distillation. However, nitrification is a biological process and is sensitive to changes in pH, especially during start-up of the reactor (Fumasoli *et al.*, 2016). Acidification can be another way to stabilise urine after urea has already degraded, but the large amount of acid required (600 meq L^{-1}) makes this approach impractical (Hellström *et al.*, 1999).

A completely different approach for stabilising urine involves inactivating urease, thus preserving the majority of the nitrogen in urine as urea. Since microbial ureolysis can have undesirable consequences (see Section 3.4.1), stopping it could be the “ultimate stabilisation process” according to Maurer *et al.* (2006). A wide range of compounds can inhibit urease, many of which have been developed for preventing losses of nitrogen during application of urea fertiliser in agriculture or for treating infections caused by ureolytic bacteria. Broadly, these inhibitors can act in three ways (Svane *et al.*, 2020): (i) by binding to the Ni(II) active site (*e.g.*, hydroxyurea); (ii) by locking the mobile "flap" of the active site in a configuration that leads to loss of activity (*e.g.*, quinones); and (iii) by chelating Ni(II) ions (*e.g.*, EDTA). As urine is intended for use in agriculture, urease-inhibiting compounds that are pharmaceutically active should be avoided. On the other hand, most anti-ureolytic compounds that are used in agriculture would pose a health hazard if they were used for household applications. The compound N-(n-butyl) Thiophosphoric triamide (NBPT), is the most commonly used urease inhibitor (Zanin *et al.*, 2016). It is generally formulated into granular urea fertilisers, but is considered a hazardous chemical according to the OSHA (2012) Hazard Communication Standard 29 CFR 1910.1200.

Shifting the pH of urine can be a better approach to prevent ureolysis. There is evidence that, microbial urease in human urine is inactive at pH values below 5 (Ray *et al.*, 2018; Hellström *et al.*, 1999) and above 10 (Geinzer, 2017). This is in accordance with the enzyme's pH profile, which shows decreasing affinity for urea at a pH lower than 5.3 and higher than 9.1 (Krajewska & Ciurli, 2005). In urease, the active site features two nickel ions that are held together by a carbamylated lysine and a hydroxide ion (Jabri *et al.*, 1995). Each active site is also capped by a mobile "flap" that regulates entry and exit of the substrate and the catalysis products (Mazzei *et al.*, 2020; Roberts *et al.*, 2012). The flap consists of amino acid residues, several of which are ionisable. At least three ionisable groups with pK_a values of 5.3, 6.6, and 9.0 have been identified. Krajewska and Ciurli (2005) attribute the pK_as values of 5.3 and 6.6 to carboxyl and imidazole groups, and the pK_a value of 9.1 to the hydroxide. The Ni-bridging hydroxide group is a nucleophile that participates in the catalysis reaction only in its protonated state, by transferring a proton to the amino group in urea, thereby forming ammonia (Benini *et al.*, 1999). Consequently, increasing the pH above 9.1 would deprotonate this group, affecting catalysis and functioning of the flap, and would ultimately result in loss of enzymatic activity. Acidification has the same effect but on the imidazole functional group of a histidine residue.

3.5.5 Drying human urine

Water is the largest component in urine (Friedler *et al.*, 2013). There are several ways to remove water and reduce the volume of urine, including distillation, osmosis, evaporation, freeze-thaw and filtration, and some have already been tested for treating urine (Section 3.4.3). Drying (or dehydration) is one relatively simple option to concentrate urine that can be applied almost anywhere in the world.

Water can exist as a solid, liquid or gas/vapour. Water molecules are polar and hydrogen bonds form between them. Breaking these bonds require energy, about 23 kJ mol^{-1} for liquid water. Hence, to increase the temperature of water, a large amount of energy is necessary (specific heat: 4.18 kJ K^{-1} kg^{-1} at 20 °C). An even larger amount of energy is required to change its physical state. The latent heat of vaporisation (ΔH_v) is about 2360 kJ kg^{-1} and can be calculated at different temperatures (T in °C) as ΔH_v=347.57×(374-T)$^{0.33333}$ (Liley, 1999). However, some water is less mobile or more "bound" structurally, chemically or biologically than other water. Unbound water exerts an equilibrium vapour pressure equal to that of pure water, and is easier to remove than bound water.

Drying involves removing water vapour from the surface of a material and into the surrounding space, and can generally be described by the following vapour flux equation (Chen, 2008).

$$N_v'' = h_m \cdot (\rho_{v,s} - \rho_{v,\infty}) \tag{5}$$

where N_v'' is the vapour flux (kg m^{-2} s^{-1}) and h_m is the mass transfer coefficient (m s^{-1}) (Eq. 5). When the vapour pressure or the concentration difference between the interface of the moist material ($\rho_{v,s}$) and the surrounding space ($\rho_{v,\infty}$) is positive, vapour leaves the surface, thereby drying the material. To improve the vapour flux, either the vapour concentration of the air supplied to the dryer must be reduced or the generation of water vapour at the material's interface must be increased by overcoming the latent heat. Increasing the temperature of the air supplied increases its moisture-carrying capacity, while also supplying sensible heat convectively to the material being dried.

To show the changes that occur during a drying cycle and to identify different drying regimes, the drying rate can be plotted against the moisture content (Kerr, 2013) (Figure 3). When a moist solid is dried, there can be a short initial phase (A to B) where the rate increases or decreases. After this, the rate remains constant (B to C in Figure 3) as the drying interface is always saturated with unbound water and remains approximately at the wet bulb temperature. Here, the temperature difference between the supplied air and the wet bulb temperature provides the driving force for heat transfer, whereas the difference in vapour concentration drives the mass transfer. Once the critical moisture content (X_C) is reached, the drying rate starts falling (phase C-D-E in Figure 3) as the rate of moisture removal becomes higher than the rate at which it is replaced from inside the material being dried. In that case, the drying rate is controlled by the movement of moisture within the solid matrix, through capillary action, void spaces or intercellular pockets. In the second falling rate period (D to E), the top surface is completely dry and the liquid-vapour interface recedes within the product, making vapour diffusion the rate-controlling mechanism.

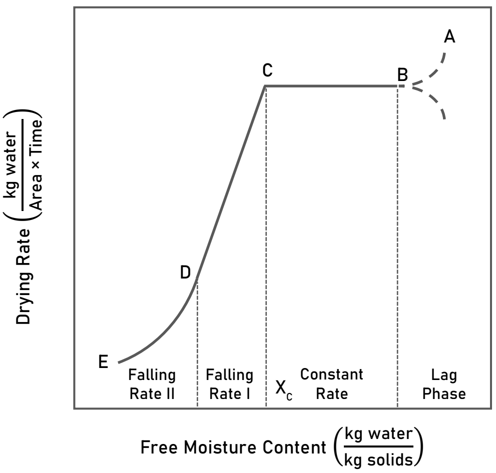

Figure 3. Drying rate as a function of moisture content in the material during a typical drying cycle under different drying regimes (A to E).

Drying human urine is not necessarily a simple process. Human urine is a complex biofluid and typically contains hundreds of metabolic breakdown products (Bouatra *et al.*, 2013). It also has high concentrations of inorganic salts (>14 g L^{-1}) and therefore high ionic strength. According to Putnam (1971), the specific conductivity of urine can vary between 16 and 22 mS cm^{-1}. Due to the large amount of organic compounds present, urine cannot be evaporated freely, as water removal is quickly inhibited by a thin oily film that forms over its surface (Tettenborn et al., 2007). To overcome this, urine can be added to a solid substrate, but then the properties of the substrate can significantly affect the drying rate. In either case, if urease-catalysed urea hydrolysis is not prevented, there can be large nitrogen losses when urine is dehydrated, as seen in previous studies by Antonini *et al.* (2012), Pahore *et al.* (2012), and Bethune *et al.* (2014).

4. Methodology

4.1 Alkalising fresh human urine

To prevent urease-catalysed hydrolysis of urea, the pH of fresh human urine was increased in two different ways in experiments described in this thesis. First, the anions normally present in fresh human urine (Cl^-, PO_4^{3-}, SO_4^{2-}) were exchanged with hydroxyl (OH^-) ions using a strong base anion resin (Amberlite™ IRA410 type-2, Merck, Germany) with exchange capacity 1.25 eq L^{-1} (Paper I). Based on pre-trials and resin exchange capacity, the resin to urine volume ratio was fixed at 0.2. Use of the ion exchanger simulated a situation where urine is alkalised immediately after excretion, for instance by placing the resin inside the toilet bowl, so that afterwards the urine can be transported via pipes without losing nitrogen. Not all urine may pass through the ion exchanger, so the minimum volume of urine that needed to be ion exchanged to increase the pH of the composite mixture of ion exchanged and fresh urine to >10 was investigated (Paper I).

In the second approach, fresh urine was added to different alkaline substrates (or alkaline media, as they are referred to in Papers I, II and IV) such as alkali metal hydroxides (*e.g.*, KOH), alkaline earth metal compounds (*e.g.*, MgO or $Ca(OH)_2$), or alkaline wastes (*e.g.*, wood ash). After addition to the substrate, the urine was dehydrated to reduce its volume. This was done to simulate a situation where urine is stabilised and treated on-site, with minimal or no transport via pipes (Papers II-IV).

4.2 Dehydrating alkalised human urine

In the laboratory-based experiments (Papers I-III), alkalised human urine was dehydrated in modified benchtop incubators, equipped with pumps for introducing air and computer fans for distributing air within the incubator cavity. Urine was added to different alkalising agents and blends with co-substrates, and thus, to different alkaline substrates, and dehydrated at temperatures between 40 and 60 °C (Table 1). In Paper IV, a pilot-scale alkaline dehydrator with capacity to treat 30 L urine per day was built, integrated within an existing portable dry sanitation system, and field-tested at a military training camp in Finland.

At the start of the laboratory experiments, the substrates were placed in containers, a given volume of urine was added and the contents were dried for a fixed duration of time and at fixed drying conditions. The containers were then withdrawn from the incubator and weighed, marking the end of what we called a drying cycle. To start a new cycle, urine was again added to the containers and the containers were returned to the incubator. The drying was done in cycles to represent real-life implementation, where the surface area available for drying urine inside bathrooms is usually small and drying would have to be performed after every urination event.

In the pilot study, drying was performed within three interconnected boxes placed in a series configuration, with each box containing a mixture of 6.5 kg wood ash and 1.5 kg $Ca(OH)_2$. The boxes were covered with acrylic reinforced lids and each lid housed two heater fans (40-137, Biltema, Sweden) that provided airflow directly onto the surface of the drying substrate. The boxes were placed in the immediate vicinity of a urinal and a urine-diverting toilet and connected to the toilets using short pipes (length <50 cm). Fresh urine from the toilets flowed directly into the boxes and was dried continuously.

4.3 Physical and chemical analyses

Fresh urine, the substrates used for drying urine and the products collected when the urine had dried in the substrates were analysed for total solids (TS), volatile solids (VS), pH, electrical conductivity (EC) and elemental composition. Samples were dried at 105 °C for 12 h to determine TS and combusted at 550 °C for 6 h to determine VS. The pH and EC of fresh urine and anion-exchanged urine were measured without dilution, whereas for the substrates and the products, they were measured in 1:5 suspensions of deionised water (Papers I & IV) or fresh urine (Papers II & III). Spectroquant® test kits were used according to the instructions provided by the manufacturer (Merck KGaA, Germany) to measure the concentrations of TAN, N_{tot} and P_{tot} in fresh urine. The elemental composition of the solids, before and after the dehydration treatments, was measured using the Dumas dry combustion method and inductively coupled plasma optical emission spectroscopy (ICP-OES).

4.4 Calculations

To evaluate the effectiveness of the dehydration treatment in reducing the mass of urine, the following parameters were calculated, on a wet basis (WB) – overall mass reduction ($mass.red_{WB}$), mass concentration factor ($mass.cf_{WB}$), and average dehydration rate ($\overline{dry.rate_{WB}}$). For these calculations, the weights of the urine added ($mass_{urine}$), and of the drying substrates or media ($mass_{media}$) and the products collected at the end of the experiments ($mass_{end-product}$) was monitored, including the weight lost between drying cycles ($w_i - w_{i+1}$). In these equations, t is the drying time, A is the surface area available, and T is the drying temperature.

$$mass.red_{WB} = \left(\frac{m_{media} + m_{urine} - m_{end-product}}{m_{media} + m_{urine}}\right) \times 100 \tag{6}$$

$$mass.cf_{WB} = \left(\frac{m_{media} + m_{urine}}{m_{end-product}}\right) \tag{7}$$

$$\overline{dry.rate_{WB}} = \frac{1}{n}\sum_{i=1}^{n}\left(\frac{w_i - w_{i+1}}{t \times A}\right) \times 100 \tag{8}$$

$$h_{1/2} = 1.352 \times 10^6 \times e^{-(0.1257T)} \tag{9}$$

$$urea.rec_i = (urea.add_i + urea.rec_{i-1}) \times \left(1 - \frac{t}{2 \times h_{1/2}}\right) \tag{10}$$

To determine the recovery of nutrients, mass balance calculations were performed using the experimental data collected during the physicochemical analyses. The focus was largely on recovery of nitrogen since, apart from sulphur (S), all the essential mineral elements in urine are non-volatile and therefore completely recovered in the treatment. The experimental nitrogen recovery was compared against the theoretical recovery, which was calculated using the following procedure. First, it was assumed that the concentration of urea-N in urine was equal to the difference between the concentration of N_{tot} and TAN. Secondly, the half-life ($h_{1/2}$) of urea was calculated at a given drying temperature using Eq. 9, which was derived using data collected by Warner (1942). Lastly, in the pH range 2-12, the amount of urea recovered was calculated using Eq. 10, where $urea.add_i$ is the amount of urea added in time period i, $urea.add_{i-1}$ is amount of urea recovered in the previous time period, and t is the time difference between the two periods.

4.5 Statistical analyses

To identify significant differences between the various alkaline substrates and the products obtained in terms of recovery of nutrients, physicochemical properties and elemental composition, one-way analysis of variance (ANOVA) was performed at 95% confidence interval. Where a significant difference was found, the mean values were compared using Tukey's Honest Significant Difference test. The results from the Tukey's multiple comparison test were summarised using compact letter displays, so that mean values that were not significantly different (at α=0.05) were followed by a common superscript letter. Prior to these analyses, the Shapiro-Wilk test for normality was applied and the homogeneity of variance was checked using the Brown-Forsythe-Levene test. All these analyses were performed using R version 4.0.0 and RStudio version 1.2.5042 (RStudio Team, 2016).

5. Results

5.1 Alkalising fresh human urine

The first approach for alkalising urine involved using a resin to exchange anions (Cl^-, PO_4^{3-}, SO_4^{2-},etc.) present in urine with OH^- ions. The increase in the pH of urine because of anion exchange was found to correlate well with the volume of urine that passed through the resin (R^2>0.95, p<0.001). When at least 75% of the urine had been subjected to anion exchange, the pH of the composite mixture of ion-exchanged urine and the residual fresh urine increased to >10 (Paper I).

In experiments where urine was added directly to an alkaline substrate (Papers II-IV), wood ash was the first substrate studied. It was found to increase the pH of urine to between 12.8 and 13.3. According to the ICP-OES measurements, the ash contained 17-34% Ca, 7-10% K and 3% Mg. The pH of urine added to biochar alkalised with KOH was >12. When urine was added to the poorly soluble alkaline earth metal compounds $Ca(OH)_2$ (Paper II) and MgO (Paper III), a saturated solution was formed. The pH of urine saturated with $Ca(OH)_2$ was between 12.6 and 12.8, while the pH of urine saturated with MgO was between 9.8 and 10.3. Since the composition of urine varied between the studies, there were slight differences in the pH of urine at saturation.

5.2 Dehydrating alkalised human urine

In laboratory experiments, the mass of urine was reduced by over 92% on average (Table 1). Since most of the water present in urine was removed, the products obtained after treatment had low residual moisture content.

The weight reduction in urine also resulted in products that weighed 1/10th to 1/17th of the total mass of urine and drying substrate added as input to the process. In general, high drying rates were obtained in all studies, with the rate varying due to differences in the material properties of the substrate and the drying conditions (*e.g.* temperature, airflow), but also due to the configuration of the drying set-ups (Table 1). In the laboratory-scale studies, the average drying rate was 6.6 L $d^{-1}m^{-2}$ at 40 °C, 19.3 L $d^{-1}m^{-2}$ at 50 °C, and 27.1 L $d^{-1}m^{-2}$ at 60 °C (Papers I-III). In the pilot-scale field study, at 60 °C, the drying rate was 40 L $d^{-1}m^{-2}$ (Paper IV).

5.3 Drying capacity of the alkaline substrates

The pH of fresh urine increased to above 10 when it was added to the alkaline substrates investigated. However, the pH of urine did not remain above 10 in all the substrates upon dehydration. To compare the substrates, the drying capacity, *i.e.* the volume of urine treated by a substrate before the pH of urine dropped to <10, was assessed. For every kg of wood ash, <16 L urine could be treated before the pH dropped below 10 (Paper I). When the ash was mixed with wheat bran (1:1 ratio, w/w), the drying capacity was reduced to <10 L kg^{-1} ash (Paper II). In alkalised biochar (1:4 KOH:biochar, w/w), 11.3 L urine kg^{-1} substrate could be dried (Paper I). Of all the substrates tested, only MgO and $Ca(OH)_2$ were able to maintain their alkalinity for the entire duration of the experiment (Figure 4). At least 50 L of urine could be treated per kg $Ca(OH)_2$ with a negligible drop in pH (<0.05 units). In fact, $Ca(OH)_2$ mixed with biochar at a ratio of 1:4 (w/w) was able to dry 80 L urine kg^{-1} $Ca(OH)_2$ at a drying temperature of 50 °C and 90 L urine kg^{-1} $Ca(OH)_2$ at 60 °C, before the pH dropped to <10 (Paper II). When $Ca(OH)_2$ was mixed with MgO (Paper III), the pH of the urine decreased from 12.7 ± 0.1 at the start of the drying treatment to 10 ± 0.2 at the end (total drying time 5.6 days), indicating that the dosage of 6 g $Ca(OH)_2$ L^{-1} was insufficient to keep the pH>12. When pure MgO was used, at least 150 L urine kg^{-1} could be dried with little change (<0.2 units) in the pH. Overall, the results of these studies suggested that at least 12.5 g $Ca(OH)_2$ L^{-1} or 6.8 g MgO L^{-1} is required to dry fresh human urine at a rate of 19 L d^{-1} m^{-2}.

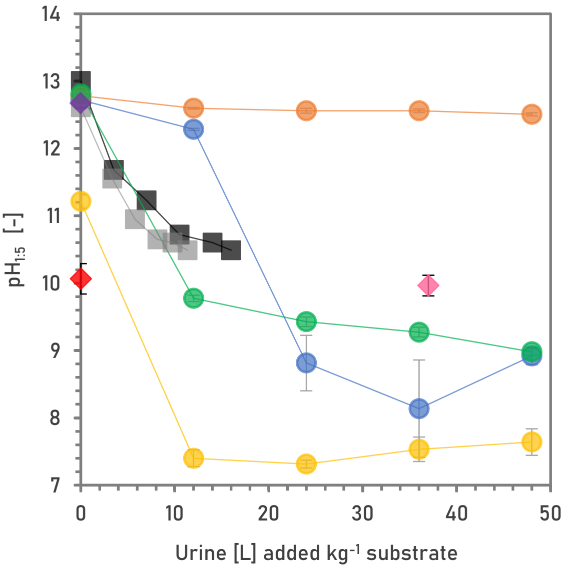

Figure 4. Change in pH measured as 1:5 suspensions (media/urine, w/v) in Paper I (squares), Paper II (circles), and Paper III (diamonds). In Paper I, wood ash (■) and KOH-biochar (■) were used. In Paper II, $Ca(OH)_2$ (●), bran-ash (●), biochar-lime (●), and pure wood ash (●) were used. In Paper III, MgO and $Ca(OH)_2$ were used but the pH was measured only at the start for MgO (◆) and MgO+$Ca(OH)_2$ (◆) and at the end (◆) of the study. Average values (*n*=3) are plotted with error bars showing the standard deviation.

5.4 Recovery of nitrogen

In the laboratory experiments, between 70 and 98% of N_{tot} in urine was recovered (Table 1). In the study using anion-exchange to alkalise the urine (Paper I), TAN present in fresh urine was not recovered by the drying substrate (wood ash or KOH-biochar), as it precipitated as struvite over the anion-exchange resin beads. However, nearly 80% of the nitrogen present in the anion-exchanged urine was recovered when it was dried at a temperature between 40 and 50 °C. When fresh urine was dried in ash-based and $Ca(OH)_2$-based alkaline substrates at 50 and 60 °C (Paper II), more than 90% of N_{tot} was recovered. The highest nitrogen recovery (98%) was achieved when urine was added to either pure wood ash, pure $Ca(OH)_2$ or $Ca(OH)_2$ mixed with co-substrates (biochar or soil). When partially hydrolysed urine (with nearly 30% of N_{tot} in the form of TAN) was dried at 50 °C using pure MgO as the alkaline substrate (Paper III), only 67% of

N_{tot} was recovered. However, when urine was dried using MgO (25% w/w) mixed with equal parts biochar and wheat bran, the N_{tot} recovery improved to 80%. Based on the mass balance calculations, the pilot-scale urine drying system recovered 30 ± 6% of nitrogen present in the urine (Paper IV), although the theoretical nitrogen recovery for the system, if operated at an average drying temperature of 60 °C, was expected to be 66%.

5.5 End-product elemental composition

The fresh urine used in the experiments contained, on average, 0.5% N, 0.2% K and <0.06% P. When the urine was dehydrated, products with element concentrations significantly higher than those originally present in urine were obtained (Table 2). For instance, drying using pure $Ca(OH)_2$ at 60 °C resulted in a 20-fold increase in the macronutrient (N, P, K) content in the products compared with that in the fresh urine. On a TS basis, the product contained 9.3% N, 0.7% P and 3.9% K (Paper II).

The composition of the products reflected the composition of the urine treated, but also the initial composition of the drying substrates. For the same drying substrate, the choice of drying temperature affected the nitrogen content of the products, and it was found that nitrogen recovery was higher at 60 °C than at 50 °C (Paper II). Drying partially hydrolysed urine (Paper III), as opposed to drying fresh urine (Paper II), resulted in higher nitrogen losses (Table 1), which lowered the nitrogen content of the end-products. The pilot-scale system dried <10% of the urine volume that it was designed to treat (30 L d^{-1}), and lost >60% of the nitrogen due to high drying temperatures (up to 90 °C). Because of this, the end-products contained only 1.4% N, 0.9% P and 8.3% K (Paper IV). However, according to the mass balance calculations, if the pilot system dried 30 L urine d^{-1} at 60 °C using a mixture of $Ca(OH)_2$ and wood ash, a fertiliser containing 13.2% N, 2.3% P and 6.0% K could have been produced.

Table 1. Type and amount of alkalising agent(s), co-substrate(s), and urine used in Papers I-IV and the drying temperatures tested. Mass reduction ($mass.red_{WB}$), concentration factor ($mass.cf_{WB}$) and drying rate ($dry.rate_{WB}$) on wet basis (WB) and percentage nitrogen (N) are also shown.

Paper	Alkalising agent(s)	Co-substrate(s)	$Substrate_{in}$	$Urine_{in}$	T	$mass.red_{WB}$	$dry.rate_{WB}$	$mass.cf_{WB}$	N recovery
			[g]	[L]	[°C]	[%]	[kg d^{-1}m^{-2}]	[–]	[%]
I	Ash	–	175	1.80	40	90.4	6.6	10.4	77
	Ash	–	100	1.55	50	92.8	11.9	13.9	74
	KOH	Biochar	125	1.42	45	85.9	6.4	7.1	72
II	Ash	Bran	30	1.44	50	91.9	19 ± 4.7^{a}	12 ± 0.3^{cd}	75 ± 3^{b}
	Ash	Bran	30	1.44	60	93.7	27 ± 5.5^{a}	16 ± 0.5^{b}	95 ± 7^{a}
	$Ca(OH)_2$	Biochar	30	1.44	50	92.4	19 ± 4.8^{a}	13 ± 0.4^{c}	11 ± 1^{c}
	$Ca(OH)_2$	Biochar	30	1.44	60	93.1	27 ± 5.1^{a}	14 ± 0.2^{c}	98 ± 2^{a}
	$Ca(OH)_2$	Soil	60	1.44	50	91.6	19 ± 4.0^{a}	12 ± 0.4^{d}	88 ± 5^{a}
	$Ca(OH)_2$	Soil	60	1.44	60	92.4	27 ± 4.6^{a}	13 ± 0.7^{d}	93 ± 9^{a}
	$Ca(OH)_2$	–	30	1.44	50	93.1	19 ± 4.3^{a}	14 ± 0.7^{b}	78 ± 2^{b}
	$Ca(OH)_2$	–	30	1.44	60	94.1	27 ± 5.2^{a}	17 ± 0.4^{ab}	96 ± 2^{a}
	Ash	–	30	1.44	50	93.9	19 ± 3.9^{a}	16 ± 0.4^{a}	95 ± 5^{a}
	Ash	–	30	1.44	60	94.2	27 ± 4.6^{a}	17 ± 0.2^{a}	98 ± 1^{a}
III	MgO	–	30	1.10	50	92.9	20 ± 4.5^{a}	14 ± 0.2^{a}	69 ± 1.2^{a}
	MgO	Biochar	30	1.10	50	90.5	19 ± 4.3^{a}	11 ± 0.4^{b}	76 ± 0.4^{bc}
	MgO	Biochar+Bran	30	1.10	50	90.6	19 ± 4.6^{a}	11 ± 0.6^{b}	80 ± 1.1^{a}
	$MgO+Ca(OH)_2$	Biochar	30	1.10	50	90.5	19 ± 4.7^{a}	11 ± 0.2^{b}	75 ± 0.9^{c}
	$MgO+Ca(OH)_2$	Biochar+Bran	30	1.10	50	90.5	19 ± 5.0^{a}	10 ± 0.6^{b}	78 ± 0.7^{ab}
IV	$Ash+Ca(OH)_2$	–	16000	35	60	62.7	–	2.7	29
	$Ash+Ca(OH)_2$	–	16000	62	60	70.6	–	3.4	31
	$Ash+Ca(OH)_2$	–	16000	90	60	72.0	–	3.6	38

Where experiments were performed in triplicate, the results are shown as mean ± standard deviation.
For each study, values within columns marked with different letters are significantly different at α=0.05.

Table 2. Elemental composition (% of total solids, TS) of the products obtained by drying urine in various substrates at 50 and 60 °C.

Paper	*T*	Substrate(s)	N	P	K	C	S	Ca	Mg	Na	Fe	Mn	Al
II	50 °C	Bran-Ash	$8.5^{a,***}$	$1.7^{a,*}$	6.9^{a}	$14.9^{b,*}$	0.5^{bc}	5.2^{c}	$0.6^{b,*}$	$2.7^{bc,*}$	$<0.005^{b}$	0.070^{b}	0.046^{c}
		Char-Lime	$1.5^{c,***}$	$1.3^{b,*}$	4.3^{b}	$26.9^{a,*}$	0.6^{ab}	6.3^{bc}	0.2^{d}	3.8^{a}	0.097^{b}	0.009^{d}	0.086^{bc}
		Soil-Lime	7.2^{b}	0.6^{c}	2.8^{c}	8.0^{d}	0.4^{c}	8.8^{bc}	0.7^{b}	2.2^{c}	1.360^{a}	0.031^{c}	0.356^{a}
		Lime	$7.2^{b,*}$	0.8^{c}	4.2^{b}	12.0^{c}	0.5^{abc}	22.1^{a}	0.3^{c}	$3.7^{a,*}$	0.076^{b}	$<0.005^{d}$	$0.129^{b,*}$
		Ash	9.6^{a}	1.7^{a}	7.7^{a}	11.7^{c}	0.7^{a}	15.6^{ab}	1.1^{a}	3.6^{ab}	0.061^{b}	0.148^{a}	0.082^{bc}
II	60 °C	Bran-Ash	$9.9^{a,***}$	$1.4^{a,*}$	6.1^{b}	$17.9^{b,*}$	0.6^{ab}	5.8^{d}	$0.6^{b,*}$	$3.1^{a,*}$	$<0.005^{b}$	0.072^{b}	0.046^{d}
		Char-Lime	$9.7^{a,***}$	$0.9^{b,*}$	3.6^{cd}	$26.5^{a,*}$	0.5^{bc}	4.8^{e}	0.2^{d}	3.0^{a}	0.062^{b}	0.007^{d}	0.072^{cd}
		Soil-Lime	6.8^{b}	0.5^{c}	2.9^{d}	7.6^{d}	0.4^{c}	7.6^{c}	0.7^{b}	2.4^{b}	1.256^{a}	0.029^{c}	0.342^{a}
		Lime	$9.3^{a,*}$	0.7^{bc}	3.9^{c}	13.3^{c}	0.6^{abc}	16.4^{a}	0.3^{c}	$3.1^{a,*}$	0.081^{b}	$<0.005^{d}$	$0.140^{b,*}$
		Ash	9.7^{a}	1.5^{a}	8.4^{a}	11.9^{c}	0.7^{a}	11.4^{b}	1.0^{a}	3.3^{a}	0.053^{b}	0.138^{a}	0.088^{cd}
III	50 °C	MgO	6.5^{c}	0.5^{c}	3.3^{b}	6.8^{d}	0.6^{a}	0.1^{b}	27.7^{a}	2.9^{b}	$<0.005^{b}$	$<0.005^{c}$	$<0.005^{d}$
		MgO-Char	7.4^{b}	0.6^{b}	3.8^{a}	30.9^{a}	0.6^{a}	0.2^{b}	7.4^{b}	3.5^{a}	0.09^{a}	0.01^{a}	0.05^{b}
		MgO-Char-Bran	7.8^{a}	0.7^{a}	3.8^{a}	27.7^{b}	0.6^{a}	0.2^{b}	7.4^{b}	3.3^{a}	$<0.005^{b}$	0.008^{b}	0.02^{c}
		MgO-Char-Lime	7.4^{b}	0.6^{b}	3.7^{ab}	29.8^{a}	0.6^{a}	6.6^{a}	3.9^{c}	3.4^{a}	0.1^{a}	0.01^{a}	0.02^{c}
		MgO-Char-Bran-Lime	7.8^{a}	0.7^{a}	3.9^{a}	24.8^{c}	0.6^{a}	6.7^{a}	4.2^{c}	3.4^{a}	0.01^{b}	0.008^{b}	0.08^{a}
IV	60 °C	Ash-Lime (March)	0.5^{c}	0.7^{b}	6.1^{a}	–	0.3^{c}	19.2^{a}	2.9^{a}	0.6^{c}	–	0.9^{b}	–
		Ash-Lime (April)	0.8^{b}	0.8^{ab}	6.8^{a}	–	0.5^{a}	17.3^{b}	2.6^{b}	1.2^{a}	–	0.8^{c}	–
		Ash-Lime (May)	1.4^{a}	0.9^{a}	7.4^{a}	–	0.4^{b}	15.0^{c}	3.0^{a}	0.9^{b}	–	1.0^{a}	–
		Ash-Lime (Simulated)	9.9	1.5	6.0		0.8	10.2	1.6	4.6		0.5	

Within each column (and paper), values marked with different letters are significantly different (at α=0.05) at that temperature.
For each drying substrate, significant differences between the two temperatures are indicated as *$p < 0.05$, **$p < 0.01$ and ***$p < 0.001$.

When urine was dried in pure MgO or $Ca(OH)_2$, all the nitrogen, phosphorus and potassium in the end-product originated from urine. No drying substrate other than wheat bran contained nitrogen, which contributed to <8% of the N_{tot} content of the product obtained by drying urine using Bran-Ash (Table 2). Pure wood ash contributed nearly 50% of the phosphorus and potassium found in the end-products. Other plant-essential nutrients (Ca, Mg, S) and micronutrients (copper (Cu), iron (Fe), chlorine (Cl), Sodium (Na), manganese (Mn), zinc (Zn)) were also retained in the end-product. Among these, almost all the sodium and chlorine, and the majority of the sulphur, originated from the urine, while the rest of the elements primarily originated from the drying substrates. Use of MgO, $Ca(OH)_2$ or wood ash resulted in high calcium and magnesium content in the products. Addition of biochar or wheat bran as co-substrates resulted in carbon-rich products (C content >25%).

Table 3. Copper (Cu) and zinc (Zn) content (mg kg^{-1} recovered P) in the products obtained by drying urine in different alkaline substrates.

Product	**Cu**	**Zn**
Bran-Ash [a]	2025	42101
Char-Lime [a]	10	1209
Soil-Lime [a]	500	3002
Lime [a]	13	145
Ash [a]	3999	82112
MgO [b]	n.d.	468
MgO-Char [b]	n.d.	3540
MgO-Char-Bran [b]	144	4528
MgO-Char-Lime [b]	n.d.	2885
MgO-Char-Bran-Lime [b]	n.d.	4352
Ash-Lime (March) [c]	13178	27130
Ash-Lime (April) [c]	12162	24560
Ash-Lime (May) [c]	11976	23950
Ash-Lime (Simulated) [d]	2480	4960

[a] Average (*n*=6) metal content of urine dried at 50 and 60 °C taken from Paper II
[b] Average (*n*=3) metal content of urine dried at 50 °C taken from Paper IV
[c] Average (*n*=3) metal content of dried urine produced during technology piloting
[d] Simulation assumes that 900 L urine $month^{-1}$ is treated; n.d. = not detected.

The choice and amount of substrate(s) used influenced the copper and zinc content of the products (Table 3). Drying urine in pure MgO or pure $Ca(OH)_2$ resulted in the lowest amount of copper (<13 mg Cu kg^{-1} P) and zinc (<500 mg Zn kg^{-1} P). When urine was dried in wood ash or wood ash-based substrates, the zinc content was high, 82 000 mg Zn kg^{-1} P or around 0.13% of TS (Papers II and IV). The cadmium (Cd) content of the products was measured only during the pilot study (Paper IV), in which urine was dried in a mixture of wood ash and calcium hydroxide, and was found to be 529 ± 76 mg Cd kg^{-1} P. If the pilot system had been operated at design capacity, then the products would have contained <370 mg Cd kg^{-1} P.

5.6 Energy demand

The energy required to dehydrate urine was monitored only in the pilot-scale study (Paper IV). The average amount of electrical energy needed to operate the pilot system was 1800 ± 573 kWh $month^{-1}$ or 24.5 ± 13 kWh kg^{-1} urine. Therefore, the primary energy demand for dehydrating urine was between 21 and 76 MJ kg^{-1} urine, assuming a power conversion factor of 2.1 for the average European electricity mix (EC, 2018). If the system had operated at its design capacity (drying 30 L urine d^{-1}), then the primary energy demand would have been between 10 and 12 MJ kg^{-1} urine.

6. Discussion

To treat human urine by alkaline dehydration, fresh human urine first needs to be collected, which can be done using a urine-diverting toilet or a urinal. To prevent enzyme-catalysed hydrolytic degradation of urea (Krajewska, 2009; Udert *et al.*, 2003a) and to recover urea when the urine is dehydrated, the collected urine must be immediately alkalised to pH >10. However, the inhibition of enzymatic activity is reversible (Geinzer, 2017). If the pH of the alkalised urine drops below 10, the risk of reactivating urease, growth of ureolytic bacteria and production of new enzyme all increase (Papers II-IV). Hence, it is essential to maintain the pH above 10 throughout the collection and treatment of urine by alkaline dehydration (Paper V). After the dehydration of urine is complete, the pH of the product obtained will remain high, which can help provide long-term inhibition against contamination by urease and ureolytic bacteria. In the following sections, the different aspects of dehydrating urine are discussed, from the prevention of enzymatic ureolysis by alkalisation of urine to the use of dehydrated urine as a fertiliser in agriculture.

6.1 How to prevent the enzymatic degradation of urea?

The enzyme urease can be inactivated simply by increasing the pH of fresh human urine to a value of ≥10, preventing hydrolysis of urea (Geinzer, 2017; Randall *et al.*, 2016). In this thesis, different methods to alkalise urine were explored. The first method tested, anion exchange (Paper I), offered an interesting approach to increase the concentration of hydroxyl ions [OH^-] in urine. The working hypothesis was that the resin beads could be held in a perforated pouch and simply placed inside the urine-diverting bowl, just like a urinal deodoriser block. Alkalising fresh urine in the toilet could allow it to be transported via pipes (*e.g.* to the basement of a building where it could be dried). However, the results indicated that the resin requirement in relation to the volume of urine was too large (20% v/v). This is because the concentration of anions in urine was 0.22 eq L^{-1} and the resin's maximum exchange capacity was 1.25 eq L^{-1}. Therefore, for a family of four people, about 1.2 L resin d^{-1} would be required. As the resin costs US$ 140 L^{-1}, it would not be economically feasible to integrate it into the design of a urine-diverting sanitation system.

In the second approach to prevent ureolysis (Papers II-IV), fresh urine was added to alkaline substrates. Here, the working concept was that the alkaline substrate would be placed inside a urine dryer, which would be placed either next to or below the toilet (*e.g.* Paper IV) to alkalise the urine immediately. An alkaline waste (wood ash), an alkali metal hydroxide (KOH) and two alkaline earth compounds (MgO and $Ca(OH)_2$) were tested. The approach of using alkaline earth metal compounds was most favourable, for the following reasons. First, these compounds are sparingly soluble in urine (Randall *et al.*, 2016) (Paper III). This allows urine to be dosed passively, for instance, when urine is added to a dryer filled with excess $Ca(OH)_2$, only an amount equivalent to its solubility is dissolved, and thus used (Flanagan & Randall, 2018). Second, urine can be easily saturated with these compounds to reach pH values that do not differ significantly for different urine compositions (Randall *et al.*, 2016). Third, the pH does not change when urine is dosed in excess of that required for achieving saturation (Randall et al., 2016; Simha et al., 2020b). In contrast, when urine is alkalised with alkali metal hydroxides (*e.g.* KOH), the volume of urine determines the dosage required and subsequently the pH (Paper I).

$$CO_2 + H_2O \leftrightarrow H_2CO_3 \leftrightarrow HCO_3^- + H^+ \leftrightarrow CO_3^{2-} + 2H^+ \qquad (11)$$

According to literature, 1.6 g KOH L^{-1} (Vasiljev, 2020), <5 g $Ca(OH)_2$ L^{-1} (Randall *et al.*, 2016) or <2 g MgO L^{-1} (Paper III) is required to alkalise fresh urine and increase its pH to above 10. However, based on the results of the laboratory experiments in Papers I-III, a relatively greater addition of these alkaline materials was required when they were used as substrates for drying urine, *i.e.* 60-100 g wood ash L^{-1}, 6 g KOH L^{-1}, 12.5 g $Ca(OH)_2$ L^{-1}, and <7 g MgO L^{-1}. This was because, during convective dehydration, the alkalised urine absorbed carbon dioxide (CO_2) present in air (about 412 ppm), which increased its H^+ ion concentration (see Eq. 3) and gradually decreased its pH.

Absorption of CO_2 reverted hydroxides to their carbonate forms, via a recarbonation reaction that is part of natural cycles (Figure 5). In alkalised urine these carbonates precipitated, which promoted more CO_2 absorption and H^+ production, decreasing the pH further (Eq. 11). Similarly, any degradation of urea also increased the carbonate concentration in urine and contributed to lowering the pH.

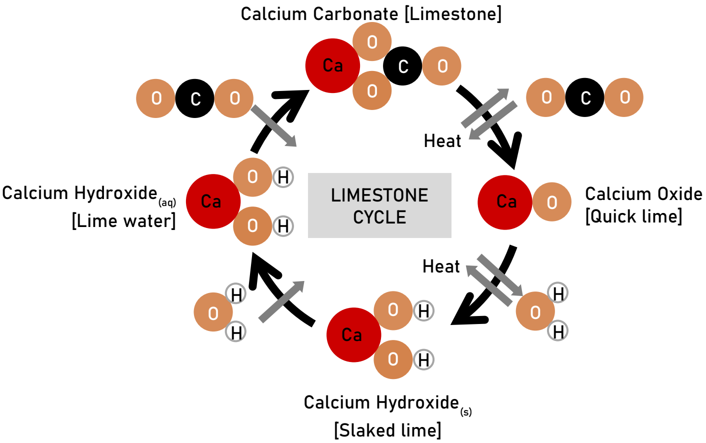

Figure 5. The calcium carbonate ($CaCO_3$) cycle. When incinerated, $CaCO_3$ is converted to calcium oxide (CaO), which hydrates to produce calcium hydroxide ($Ca(OH)_2$). The gradual re-absorption of carbon dioxide (CO_2) from air (*i.e.* recarbonation) converts $Ca(OH)_2$ back to $CaCO_3$.

The inactivation of urease achieved by alkalising fresh urine is reversible (Geinzer, 2017). Results obtained in this thesis suggest 15 g $Ca(OH)_2$ L^{-1} urine or 8 g MgO L^{-1} should be used to ensure that the pH remains above 10 and that urine can be dried without loss of urea to enzymatic hydrolysis. Alternatively, the following actions can be taken to prolong the use of an alkaline substrate and keep the pH >10: (i) minimising the air flow rate to limit the mass transfer of CO_2 (Han & Kim, 2014); (ii) pre-treating the air to remove CO_2 (Andersson & Nordberg, 2017); (iii) pre-treating the urine, *e.g.* by anion exchange (Paper I); and (iv) using substrates with lower saturation pH in urine, *e.g.* MgO *vs*. $Ca(OH)_2$, since the solubility of CO_2 increases with increasing pH (Paper III).

When urine is treated by alkaline dehydration, there may be mechanisms other than the high pH that can inhibit urease. First, the conformation of enzymes and enzymatic reactions depend on the amount of water present, and tend to proceed at a slower rate at low moisture levels (Kerr, 2013), such as towards the end of a drying cycle. For instance, (Sahrawat, 1984) found that urease activity decreased with decrease in moisture content of soils, with no detectable activity in air dried soils (moisture content <6%). Second, urease can be deactivated through substrate inhibition at urea concentrations higher than 30 g L^{-1} (Qin & Cabral, 2009). The urea concentration in freshly excreted urine varies between 9 and 22.8 g L^{-1} (Putnam, 1971). Third, at pH values higher than pH 7, urease activity is strongly influenced by the presence of electrolytes. For instance, the activity of soybean urease is reduced by half in solution containing 29 g NaCl L^{-1} (Kumar & Kayastha, 2010). According to Putnam (1971), human urine contains 8 g NaCl L^{-1}, 14.1 g L^{-1} of inorganic salts and 4 g L^{-1} ammonium salts. Therefore, further studies are needed to understand the influence of these factors, alone and in combination, on the activity of urease. It is known for certain that, to recover urea from urine by alkaline dehydration, enzymatic ureolysis has to be prevented by alkalising fresh urine to pH >10 (Geinzer, 2017). However, it is unclear whether this threshold pH value also applies when urine is dried and concentrated.

6.2 How to recover nitrogen?

To recover all the nitrogen present in fresh urine, urea, TAN and nitrogen-containing organic compounds (other than urea) must be recovered. All the organic nitrogenous compounds are quite stable (Putnam, 1971), so it can be assumed that they are not degraded during alkaline dehydration. To prevent enzyme-catalysed degradation of urea, fresh urine was alkalised in Papers I-IV in this thesis. Dehydrating alkalised urine at the rate of 27 L d^{-1} m^{-2} and 60 °C resulted in more than 90% N_{tot} recovery, irrespective of the substrate used (Paper II). However, nitrogen could not be completely recovered because increasing the pH of urine and its temperature during dehydration promoted two undesirable reactions that resulted in loss of nitrogen as NH_3.

$$\mathrm{NH_2(CO)NH_2 + OH^- \leftrightarrow NH_2(CO)NH^- + H_2O} \qquad (12a)$$

$$\mathrm{NH_2(CO)NH^- + H_2O \leftrightarrow CHNO + NH_3 + OH^-} \qquad (12b)$$

The first undesirable effect of alkalising urine was chemical hydrolysis of urea, which is catalysed by OH^- and occurs at pH values above 12, forming cyanic acid (CHNO) and ammonia (Eq. 12) (Blakeley *et al.*, 1982). About 1-5% of the N_{tot} loss observed in Papers I-III could be explained by this. However, in Paper II, contrary to the understanding that chemical urea hydrolysis increased with temperature, the recovery of nitrogen was higher at the higher drying temperature tested. The difference in drying time, 5.3 d at 60 °C and 7.3 d at 50 °C, was suggested as one reason for this anomaly. However, recent modelling work on the chemical speciation in alkalised urine suggests that $Ca(OH)_2$ and MgO display inverse solubility in urine, *i.e.* their solubility decreases with temperature (Simha *et al.*, 2021; Randall *et al.*, 2016). At 60 °C, the pH of urine saturated with $Ca(OH)_2$ is below 12, and therefore drying would result in negligible loss of urea to chemical hydrolysis (Warner, 1942).

Chemical hydrolysis of urea was also hypothesised as being the primary reason for the losses of nitrogen recorded during field-testing of the technology (Paper IV). In the pilot-scale setup, only 30 ± 6% of N_{tot} was recovered, although it was expected that 66% of N_{tot} could be recovered by drying 30-40 L urine d^{-1} m^{-2} at an average temperature of 60 °C. This discrepancy in nitrogen recovery was explained by the fact that the pilot system received less than 10% (<3 L d^{-1}) of the urine that it was designed to treat. This reduced the evaporative cooling effect that would otherwise have occurred (see Section 3.4.5), and there was temperature overshoot in the dryer of up to 90 °C. Since the pH was >12, urea was likely lost due to chemical hydrolysis (Eq. 12) (Blakeley *et al.*, 1982).

The second undesirable effect of alkalising urine was a shift in the ammonia equilibrium from NH_4^+ (in fresh urine) to $NH_{3(aq)}$ (in alkalised urine) (Figure 2), which volatilised during ventilation and could not be captured in the substrate. Ammonia volatilisation was identified as the main reason for the loss of nitrogen in Paper III, because the urine used in the study was partially hydrolysed during collection and/or storage, and contained 30% of its N_{tot} in the form of TAN. Paper III also highlighted the importance of alkalising and dehydrating urine at the point where it is collected (toilet).

When treating urine by alkaline dehydration, it may be possible to recover all the nitrogen excreted in urine by stabilising the urea (Paper II) and by precipitating the TAN (Paper III). To accomplish this, fresh urine would first have to be alkalised at room temperature to attain a pH between 10 and 12, as this would prevent enzymatic and chemical hydrolysis of the urea. To capture TAN in the form of struvite, magnesium ammonium phosphate

($NH_4MgPO_4 \cdot 6H_2O$), the alkalised urine would then have to be dried in a substrate containing available Mg^{2+}. This is because the concentration of Mg in fresh urine is typically low and the molar ratio of [Mg]:[NH_4]:[PO_4] is about 0.1:1:0.98 (Rose *et al.*, 2015), whereas a molar ratio of 1:1:1 is needed to form struvite (Ronteltap *et al.*, 2007). According to stoichiometric calculations, all the TAN can be captured if the drying substrate contains at least 0.7 g MgO L^{-1}, 3.7 g $MgCl_2.6H_2O$ L^{-1} or 2.2 g $MgSO_4$ L^{-1} urine. However, the substrate would need to be free of Ca, as in its presence phosphate excreted in urine precipitates as hydroxylapatite instead of struvite (Randall *et al.*, 2016). The drying treatment would also have to take place at temperatures below 30 °C to limit volatilisation of ammonia.

6.3 How to choose the drying conditions?

Flow rate, humidity and temperature of air, choice of alkaline drying substrate(s) and the surface area available for drying were all factors that were observed to influence the drying rate and the recovery of nutrients from urine (Tables 1 and 2). Increasing the airflow rate resulted in increasing drying rate (Paper I). However, this likely also promoted absorption of CO_2 from air (Han & Kim, 2014), and thus recarbonation of the substrates, which ultimately decreased the pH. Increasing the drying temperature from 40 to 60 °C increased the dehydration rate from 6.6 to 40 L $d^{-1}m^{-2}$ (Table 1). This implies that, to dehydrate the daily urine produced by a family of four (6 L according to Vinnerås *et al.* (2006)), the drying surface area required is 0.9 m^2 at 40 °C, but as little as 0.15 m^2 at 60 °C. Therefore, a urine dryer could be installed in existing bathrooms, by either making use of dead spaces (*e.g.* below the water cistern or behind the toilet) or by integrating the dryer into the design of future urinals and urine-diverting toilets (Papers II, IV & V).

The dehydration rate in combination with the temperature was found to significantly affect nitrogen recovery (Figure 6). At a given temperature, drying urine at a higher dehydration rate resulted in higher nitrogen recovery, since this reduced the total dehydration time and thus also the time available for degradation of urea by chemical hydrolysis. To help tentatively guide the design of a urine dryer, a chart developed in Paper II can be used to identify the dehydration rate that needs to be achieved at different temperatures to recover >90% of all nitrogen (Figure 6). The pH and the solubility of substrates such as $Ca(OH)_2$ depend on the temperature (Randall *et al.*, 2016).

Hence, the drying temperature must be selected depending on the substrate used. For instance, if wood ash or $Ca(OH)_2$ are used, the drying temperature may be increased up to 60 °C as the pH is still >11 at this temperature (Paper IV). However, when using MgO, it is tentatively recommended to keep the temperature below 40 °C, since at higher temperatures the saturation pH of urine drops below 10 (Paper III).

Ultimately, selecting the conditions for drying urine in real-life settings will most likely be a multi-criteria optimisation problem. The ideal is to maximise the recovery of nitrogen and the drying rate, while minimising the airflow rate, energy demand, and amount of space and the substrate required for treating urine. In reality, however, drying urine will also be a sustainability problem that would involve balancing the trade-offs between the three pillars of sustainability (economic, social, environmental). Preliminary discussions with toilet producers, renters and service providers suggest that they place higher value on the drying rate, as it determines toilet usage rates, servicing times and logistics, and because the cost recovered from the sales of the fertiliser produced will likely be low. However, policymakers, environmentally aware toilet users, and forward-thinking municipalities may place higher emphasis on the recovery of nutrients.

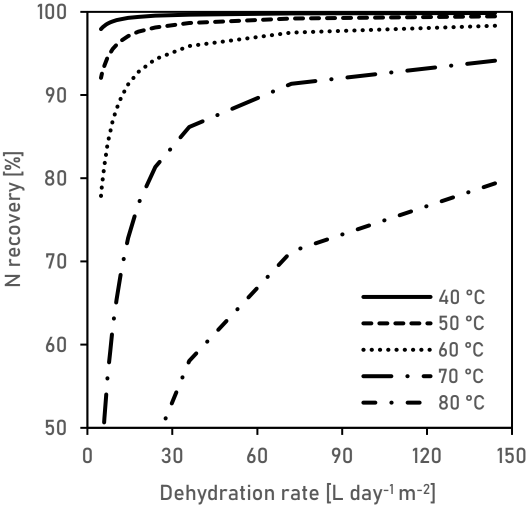

Figure 6. Theoretical nitrogen (N) recovery (%) as a function of urine dehydration rate (L day^{-1} m^{-2}) at different temperatures. Theoretical recovery was based on chemical urea hydrolysis, calculated using the approach suggested by Warner (1942), where the half-life of urea in the pH range 2-12 is estimated from Eq. 4 and by using the dehydration time at different dehydration rates. The simulation assumed that fresh urine was added cumulatively and dried 48 times within the same alkaline substrate.

6.4 How to select the drying substrate(s)?

Results from the laboratory experiments in Papers I-III suggested that, to dry the urine excreted by a person in a year (550 kg according to Vinnerås *et al.* (2006)), approximately 3.8 kg MgO, 7 kg $Ca(OH)_2$ or 35-50 kg wood ash would be required. Wood ash is a waste material, but its nutrient value can be improved by using it to dry urine, although there are concerns about its heavy metal content if applied to agricultural fields (see Section 6.6.2). The other two substrates are commodity chemicals, with virtually unlimited global reserves. The bulk price of MgO is US$ 0.3 kg^{-1} (Bray & Ghalayin, 2020), while that of $Ca(OH)_2$ is US$ 0.08 kg^{-1} (Muster *et al.*, 2013). Therefore, using either substrate to dry urine holds much promise.

However, the findings in this thesis and in previous studies by others suggest that the choice between Ca or Mg hydroxide can significantly influence the nitrogen recovery and the chemical speciation of elements excreted in urine. First, according to Randall *et al.* (2016), adding $Ca(OH)_2$ would precipitate all of the phosphate excreted in fresh urine as amorphous $Ca_3(PO_4)_2$, which at thermodynamic equilibrium would be converted to $Ca_{10}(PO_4)_6(OH)_2$ (hydroxylapatite). No struvite would be formed, as the magnesium naturally present in urine would precipitate as $Mg(OH)_2$ (Randall *et al.*, 2016). In contrast, adding MgO would precipitate most of the excreted phosphate in urine as struvite (Paper III). Therefore, Mg-based substrates could potentially recover TAN in the form of struvite. Second, at temperatures higher than 40 °C, the pH of urine saturated with MgO is <9, which increases the risk of microbial ureolysis (Paper III), whereas $Ca(OH)_2$-saturated urine at these temperatures has a pH of >11 (Randall *et al.*, 2016). Therefore, in contexts where high drying rates are essential and the drying temperature must be set >40 °C, $Ca(OH)_2$ rather than $Mg(OH)_2$ should be used to minimise the risk of ureolysis. Third, the CO_2 mass transfer coefficient and CO_2 absorption potential of MgO/$Mg(OH)_2$ is less than that of $Ca(OH)_2$ and far lower than that of KOH (Jung, 2004), since it a function of pH. Fourth, the time required to alkalise fresh urine (or achieve 95% saturation) is 15-30 min for MgO (unpublished data), but is less than 1 min for $Ca(OH)_2$ (Randall *et al.*, 2016).

In addition to increasing the pH of urine, a substrate or a co-substrate can provide additional benefits. For instance, adding a co-substrate with high water absorption capacity to sparingly soluble substrates like MgO can help limit the loss of TAN when urine is dried (Paper III). Applying urine dried in carbon-rich substrates like biochar can help amend soils, improve soil fertility and sequester carbon (Jeffery *et al.*, 2015). Using agricultural residues like bran or husk as substrates can open a new pathway for recycling materials usually discarded as waste (Paper II). The co-substrate used will also affect the elemental composition of the fertiliser produced; *e.g.* when biochar is used as the co-substrate, a carbon-rich fertiliser is produced (Table 2). Therefore, by drying urine in different mixtures of substrates, it would be possible to produce a variety of fertilisers with different composition. This can be advantageous, because fertilisers are usually applied in relation to the nutrient requirements of the crops grown, which vary between crops (cereals, grass, vegetables or fruits) (Papers II and IV).

Overall, three factors could be important to consider when selecting a drying substrate(s). First, the substrate or the substrate mixture must be sufficiently alkaline to prevent enzymatic hydrolysis of urea. Second, it must be recognised that adding substrate(s) to fresh urine, either to alkalise it or for any of the above-mentioned purposes, will increase the total solids content of the fertiliser produced. Thus, the amount of the fertiliser that has to be collected, transported and applied will also increase, which may have a bearing on the overall cost of recycling urine. Lastly, the substrates could be selected in relation to the end-use of the fertiliser. Depending on whether the fertiliser is destined for application in agroforestry or agriculture, or for fertilising edible or non-edible crops, different substrates could be used.

6.5 How to reduce the energy demand for drying urine?

The energy demand for drying urine was monitored only in the pilot-scale field study, and was found to vary between 21 and 76 MJ kg^{-1} urine. The energy demand was far more than expected, as operating the pilot at its design capacity of 900 L urine per month would require 10-12 MJ kg^{-1} urine. Even at its design capacity, the pilot system would have consumed 10 times more energy than the conventional scenario, where the urine is sent to a wastewater treatment plant (0.9 MJ kg^{-1} urine) and mineral fertiliser is produced equivalent to the amount of N, P, and K excreted in human urine (0.4 MJ kg^{-1} urine).

The pilot system was not optimised for energy consumption. Despite this, its energy demand was comparable to, or less than, the energy demand for treating urine in incineration toilets, which are popular in houses with on-site sanitation across Scandinavia; e.g. the Cinderella® Classic model uses 0.8-2 kWh $flush^{-1}$ and Separett's CINDI® toilets require 0.4-1.7 kWh $flush^{-1}$. The minimum primary energy demand for evaporating urine at a temperature of 60 °C is approximately 5.3 MJ kg^{-1} (170 kJ kg^{-1} to increase the temperature from 20 to 60 °C and 2360 kJ kg^{-1} for phase transition). If 85% of this energy can be recovered, *e.g.* with a heat pump, the energy demand can potentially be reduced to 0.8 MJ kg^{-1}.

The energy demand for drying urine can be reduced in many ways. First, the heater fans used in the pilot set-up had a power rating of 700/1200 W, but could be replaced with devices with a lower rating, *e.g.* the fans used in food dryers (MediaMarkt, 2021). Second, the heat energy in the exhaust air from the system could be recovered (60-95%) using air-to-air heat exchangers or heat pumps, both widely used in building ventilation systems (Mardiana-Idayu & Riffat, 2012) and in laundry dryers (International Energy Agency, 2012). Third, collecting urine without flush water during the whole day, including the morning urine (which the pilot set-up did not receive), would reduce the energy demand per kg nitrogen recovered. The air humidity and temperature could be monitored by sensors and used as feedback to decide the duration of the drying cycles, and to match the energy input with the moisture content of the substrates. This would also prevent unnecessary drying (Paper IV).

By separately collecting and drying urine, the amount of toilet flush water (Ishii & Boyer, 2015) and the energy required to heat water at home and to heat wastewater in sewage pipes (Hao *et al.*, 2019) could be reduced. Energy would also be saved by replacing synthetic fertilisers with dried urine and by having to treat smaller quantities of nitrogen at wastewater treatment plants (Kjerstadius *et al.*, 2017). However, to perform alkaline urine dehydration, in addition to the energy required to evaporate water, energy would be needed to produce substrates like calcium hydroxide, to collect dried urine from toilets, and to manufacture the fertiliser pellets. A full life cycle analysis can help understand the energetic aspects of treating urine by alkaline dehydration.

6.6 How to manage substances of concern?

6.6.1 Salts

As salts are excreted in urine, they also gradually accumulate in the substrate when urine is dried. This can be problematic from a treatment perspective, as salt accumulation results in boiling point elevation, which in turn increases the energy needed for evaporating urine (Udert & Wachter, 2012). However, salts could be a concern from an end-use perspective, as applying urine may lead to soil salinisation (Mnkeni *et al.*, 2008). Sodium chloride is the most abundant salt excreted in urine (Putnam, 1971). In the laboratory experiments (Papers II and III), the maximum sodium and chlorine content of the fertiliser products on a dry matter basis was 3.8% and 4.8%, respectively. The simulated mass balance for the pilot-scale system at its design capacity suggested that the products would contain <5% Na and about 7% Cl (Paper IV). The chlorine content of dried urine is similar to that of blended mineral fertilisers used in Finland and Sweden (*e.g.* Yara Mila 21-4-7), which also contain 7% Cl by weight (Yara, 2020). However, the sodium content of dried urine is relatively high, although this may not be an issue for two reasons: (i) all plants have evolved mechanisms to tolerate, to varying extents, accumulation of NaCl in soils, as it is widespread in the environment (Munns & Tester, 2008); and (ii) salts can normally be flushed out of soil during irrigation and rainfall events, or leached by drainage (Guizani *et al.*, 2016). However, in arid regions, the salt content of the dried urine fertiliser needs to be considered, especially if reclaimed water is used for irrigation and if there is no appropriate soil drainage (Rebhun, 2004). In such cases, urinary salts would have to be removed prior to the drying treatment (*e.g.* by ion exchange).

6.6.2 Heavy metals

In domestic wastewater, greywater contributes the most to the heavy metal load. According to Friedler *et al.* (2013), about 90% of copper and zinc can be traced back to this fraction. As urine contains products of human metabolism, the concentration of heavy metals in urine is very low (Jönsson *et al.*, 2004). Copper (Cu), chromium (Cr), nickel (Ni) and zinc (Zn) are biologically essential heavy metals, of which copper and zinc are plant-essential micronutrients that are excreted in urine. Therefore, applying urine can reduce the risk of soil micronutrient deficiency (Jönsson *et al.*, 2004).

The substrates used for drying urine in this thesis also contained heavy metals (Table 4). When urine was dried in either pure $Ca(OH)_2$ or pure MgO, the heavy metal content of the fertiliser produced was lower than that of mineral phosphorus fertilisers, and much lower than the level typically present in sewage sludge and farmyard manure (Papers II and III). The same applied when these two substrates were mixed with biochar, bran or soil. Drying urine in wood ash or ash-containing substrates produced fertilisers with a relatively high content of heavy metals, especially zinc, lead (Pb) and cadmium (Cd), although their metal to phosphorus ratio was still lower than that of other household wastewater fractions like greywater.

Table 4. Simulated heavy metal content (mg kg^{-1} P) of urine dehydrated in different substrates calculated based on a mass balance ($metal_{substrate} + metal_{urine} = metal_{dried\ urine}$). The composition of fresh urine used in these calculations was taken from Vinnerås *et al.* (2006) and the metal content of the untreated substrates were obtained from the following sources: Głodowska (2015) for biochar, SLU (2020) for wood ash, Peters *et al.* (2008) for soil, Jorhem (2013) for wheat bran, Amlinger (2004) for $Ca(OH)_2$, and VWR (2021) for MgO. It was assumed that 1.5 L fresh urine was dried in 30 g of substrate. The heavy metal content (mg kg^{-1} P) of fresh urine (Vinnerås *et al.*, 2006), greywater (Vinnerås *et al.*, 2006), sewage sludge produced at municipal WWTPs in Sweden (Statistics Sweden, 2018), farmyard manure used in organic cattle farms in Sweden (Jönsson *et al.*, 2004), and mineral P fertiliser (Nziguheba & Smolders, 2008) is also presented.

Product	Cu	Zn	Cr	Ni	Pb	Hg	Cd
Dried urine							
Bran-Ash	1112	6125	813	332	436	1	86
Char-Lime	410	3316	253	264	239	2	2
Soil-Lime	1254	2265	290	2563	178	4	6
Lime	436	521	589	188	86	7	4
Ash	1746	9796	1460	587	786	2	152
MgO	403	196	10	7	303	1	1
MgO-Char	402	1211	43	78	300	1	1
MgO-Char-Bran	364	2141	54	106	167	1	2
MgO-Char-Lime	409	2969	223	234	248	2	2
MgO-Char-Bran-Lime	375	2009	163	131	138	2	2
Fresh urine	101	45	10	7	2	1	0.7
Greywater	13158	19211	721	2368	1842	8	63
Sewage sludge	12764	21298	860	614	599	19	30
Farmyard manure	3537	18049	463	427	124	-	16
Mineral P fertiliser	-	2290	1100	190	55	-	83

(-) not available

Cadmium is a particular concern to human health, as it can accumulate in the body and cause renal damage, and plants can take up cadmium if it is present in soil (Roberts, 2014). The ash-based fertilisers produced in the laboratory-scale studies contained <60 mg Cd kg_P^{-1} or <0.9 mg Cd kg_TS^{-1} (Table 3). The cadmium content of urine dehydrated in ash was higher than that of animal manure but within the European limits (EU, 2019) for solid organic fertilisers (1.5 mg Cd kg_TS^{-1}) and organo-mineral fertilisers containing less than 5% P_2O_5 (3 mg Cd kg_TS^{-1}). Nevertheless, given the concerns about cadmium, it is recommended to use urine dried in wood ash either in forestry, where cadmium originates from and is not a major concern (Pitman, 2006), or in places where the limit values are not exceeded.

The overall risk to human health from heavy metals excreted in urine or those accumulated in dried urine fertiliser is minimal. However, it is important to recognise that the drying substrate can also contain heavy metals, which can potentially contaminate the urine. Therefore, care must be taken when selecting the alkaline substrate(s).

6.6.3 Micropollutants

To obtain preliminary insights into the fate of micropollutants during alkaline urine dehydration, they were monitored in the pilot study (Paper IV). In the untreated urine, eight pharmaceuticals (out of 126 analysed) and four hormones were detected. After dehydration, some of the pharmaceuticals were detected in the solid fertiliser, but none of the hormones was detected. In the dehumidifier condensate, only caffeine was detected, which suggests that micropollutants primarily partition to the solid phase. However, the results in Paper IV must be viewed with caution for the following reasons. First, the urine fed to the urine dryer came from healthy young adults undergoing military training. Second, the urine composition was not be monitored throughout the study, which explains the discrepancies in the mass balance. Third, the calculations indicated that some pharmaceuticals were reduced by the treatment, but it is unclear whether this was because they could not be extracted or detected or because they were in fact degraded.

In human urine, it is normal for micropollutants such as residues, metabolites and transformation products of pharmaceuticals and natural and synthetic hormones to be excreted (Lienert *et al.*, 2007). Since it is possible for micropollutants to accumulate in soil and be taken up by plants, there is concern surrounding agricultural use of urine and urine-based fertilisers (Winker, 2009). However, many experts believe that the risk from micropollutants excreted in urine is small, far smaller than the risks associated with the current food and water systems (Jönsson *et al.*, 2004). Micropollutants have been detected for years in the drinking water reservoirs of many cities, which suggests that wastewater treatment plants can, at best, only partially eliminate these substances (Levén *et al.*, 2016). The likelihood of micropollutants being degraded is much higher if they are applied on arable land (Viskari *et al.*, 2018), as they can be retained in the topsoil for months and the microbial community in soils is just as diverse and active as that found in biological wastewater treatment plants (Jönsson *et al.*, 2004).

It is reasonable to expect that most micropollutants will not be degraded by the alkaline dehydration treatment and that they will accumulate in the drying substrate. While many of these compounds are likely to degrade when dried urine is applied to soil as fertiliser, they could also be removed so that they do not enter the food chain. Micropollutants can be removed by treating the urine before it is dried, for instance by ultra-violet irradiation (Zhang *et al.*, 2015), adsorption (Kopping *et al.*, 2020; Solanki & Boyer, 2017) or nanofiltration (Pronk *et al.*, 2006). Alternatively, the drying substrate could be designed to adsorb micropollutants, so that they are not released when the fertiliser is applied to soils. For instance, biochar, which is widely used for adsorbing pharmaceuticals from wastewater (de Andrade *et al.*, 2018), is also a good co-substrate for drying urine. According to Kopping *et al.* (2020), about 60 times less granular activated carbon is required for removing 90% of the pharmaceuticals if urine is treated on-site (with nitrification) than if the pharmaceuticals are removed from wastewater treatment plant effluent.

6.7 How to integrate urine drying with existing systems?

As demonstrated in this thesis, before treating urine by alkaline dehydration it must first be collected separately from the faeces, to prevent enzymatic ureolysis. For the same reason, urine must be alkalised immediately after it is collected. Therefore, to recycle urine using this approach, users would have to install a urinal or a urine-diverting toilet that is connected to a urine dryer placed in the immediate vicinity of the toilet. In addition, the dryer would need to be filled with the alkaline substrate, operated and eventually emptied when the substrate pH drops below 10 (see Section 6.1). Finally, the dried urine collected would have to be stored or applied as fertiliser.

At a small scale and in settings such as rural areas, holiday homes and remote locations with dry toilets (*e.g.* national parks, tourist attractions, or military training sites), it would be possible for individuals or groups of individuals to perform all the above-mentioned steps. For instance, off-grid houses usually have on-site sanitation and are equipped with urine-diverting toilets. In Sweden, it is estimated that there are 135,000 urine-diverting toilets, installed mostly at holiday homes (Kvarnström *et al.*, 2006). As these households are already in the habit of collecting and handling urine, they can be one market for introducing the urine drying technology.

To dry urine at a larger scale, such as in a multi-storey building or for an entire neighbourhood, a new semi-decentralised sanitation system could be established. Such a system was proposed in Paper V. In this system, individual households would still need to use a urine-diverting toilet attached to a urine dryer, but would be connected to a service chain that supplies them with the alkaline substrate and collects their dried urine (Figure 7). There can be several advantages of creating a service chain. First, it could encourage people to recycle urine, as they would not have to perform all the steps involved in recycling urine themselves. Second, it could help ensure that the treatment is done properly, *e.g.* by regularly supplying new substrate, a service provider can make sure that households do not use their substrate beyond its intended duration (before its pH drops to <10). Third, it could help standardise the system. For instance, a service provider could supply all the households with the same substrate and even provide the urine dryer for a monthly fee. The dried urine collected from the households could be processed to create a homogeneous, pelleted and safe fertiliser that can be supplied to farmers. Fourth, as toilets can be dry or water-flushed, the same service could collect dried faeces and the products of faecal treatment.

Drying urine at a large scale would affect how existing wastewater treatment plants operate (Paper V), because collecting urine locally would reduce the nitrogen inflow to the plant and increase the biological oxygen demand:nitrogen (BOD:N) ratio from 5 to >20 (Larsen, 2015). This would decrease the need for energy-demanding nitrification and organics-demanding denitrification processes (Larsen, 2015; Kampschreur *et al.*, 2009; Wilsenach & Loosdrecht, 2006). The amount of coagulants required for precipitating phosphorus and the volume of the chemical sludge produced at the wastewater treatment plant would also be reduced (Larsen, 2015). According to Wilsenach and Loosdrecht (2006), diverting 85% of the urine away from a simple wastewater treatment plant with a solid retention time of just one day would produce effluent of the same quality as that from the most advanced wastewater treatment system in the world today, in which mixed sewage is treated with a retention time of 12 days and with addition of methanol as an external source of carbon. Hence, by diverting and locally treating the urine produced in new neighbourhoods, existing wastewater treatment plants in rapidly urbanising areas of the world would not have to increase their treatment capacity. This could extend plant lifetime, which could be an advantage for cities like Paris and Malmö that are currently considering alternative ways to manage the wastewater that will be generated by new inhabitants. Wastewater treatment plants and municipalities can therefore be key stakeholders and beneficiaries of a new sanitation system based on diverting and dehydrating urine.

However, to create this new sanitation system, new actors will be needed *e.g.* to manufacture the urine dryer, to produce the alkaline substrate(s) and the fertiliser, and to perform servicing, maintenance and repairs. Depending on local factors and the context, the service chain could also be organised in different ways. For instance, a forward-looking municipality might wish to supply households with drying substrate, collect their dried urine and even use the fertiliser products in municipal parks/gardens. Municipalities already have systems in place for collecting different fractions of solid wastes from households, so dried urine can simply be another fraction that they collect.

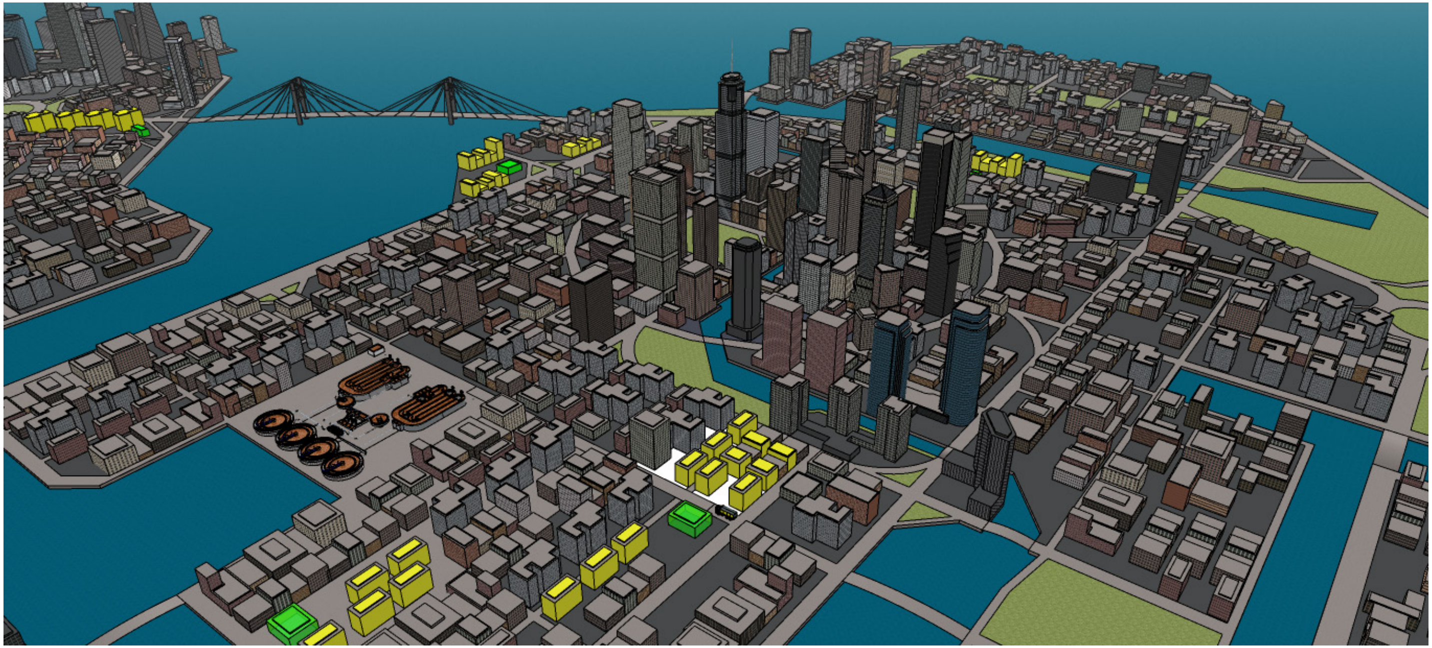

Figure 7. As cities have grown, old wastewater treatment plants (WWTPs) located on the city outskirts now occupy prime real estate. By collecting/treating urine from newly built neighbourhoods or buildings undergoing renovations, these WWTPs could be made more energy- and space-efficient, so they can avoid expanding their treatment capacity. The illustration above shows how this could be done. Brown- and grey-water produced in the new buildings (depicted in yellow) could flow to the existing WWTP, but urine could be treated on-site. The dried urine could either be moved periodically to a collection and transfer station (depicted in green) and/or collected by multi-compartment trucks that are already used for collecting different fractions of municipal solid wastes.

6.8 How to use dried urine as fertiliser?

The dried urine produced in the laboratory in this thesis was a solid fertiliser containing 9.9% N, 1.5% P and 6.0% K on a dry matter basis. If the pilot-scale urine dryer had been operated at the design capacity, a fertiliser containing 13.2% N, 2.3% P and 6.0% K could have been produced. In theory, if urine from an average person (Vinnerås *et al.*, 2006) were to be dehydrated without using any substrate, then a fertiliser containing 19% N, 1.7% P and 4.8% K on a dry matter basis could be obtained. The fertiliser value (N-P-K as % of TS) of the dried urine obtained in this thesis was much higher than that of fresh human urine (0.7-0.06-0.2 according to Vinnerås *et al.* (2006)) and liquid dairy manure (0.4-0.1-0.25 according to Brown (2008)). However, it was lower than that of synthetic fertilisers like ammonium nitrate (33-0-0), potassium chloride (0-0-52) and diammonium phosphate (18-23.5-0) (Maguire *et al.*, 2009), and of blended fertilisers used in Sweden (21-4-7 for cereal crops and 11-4.6-17.6 for vegetables, fruits and berries) (Granngården, 2019). One possible way of improving the fertiliser value of dried urine is by pre-blending the alkaline substrate with co-substrates like agricultural residues or post-blending the dried urine with other waste-derived compounds like struvite, so that its nutrient content corresponds to the nutrient requirements of specific crops.

In contrast to fresh/stored human urine, dried urine has much lower mass and much higher nutrient content, which makes it easier to collect, transport, store and apply as fertiliser. To fertilise a hectare of land, a farmer would need to apply 900 kg of dried urine instead of 15 000 kg of liquid urine (assuming cereal crops need 90 kg N ha^{-1} and dried urine contains 10% N). Once it has been collected from households, it may be possible to convert dried urine into pellets, so that it can be applied using conventional farm machinery. This would mean that farmers would not have to do anything different other than to buy dried urine that contains recycled nutrients instead of synthetic fertilisers produced using fossil energy and virgin materials.

The dried urine produced in cities could be used locally to replace mineral fertilisers used in urban agriculture, thereby improving the resilience of local food systems. According to Nierenberg *et al.* (2011), urban farms contribute nearly 20% of the food produced worldwide. The dried urine could also be used to fertilise rural cropland that would otherwise be inaccessible, because of the high costs involved in transporting urban wastes with low nutrient content (*e.g.* stored human urine collected in cities) (Trimmer & Guest, 2018). In addition, it could be used as a raw material by the fertiliser industry. If the urine produced worldwide were to be recycled as dried fertiliser, then around 31 Tg N y^{-1} and 2.8 Tg P y^{-1} could be recycled. This would reduce the current anthropogenic transgression of the nitrogen and phosphorus planetary boundary by 35% and 25%, respectively.

7. Conclusions

In this thesis, a new, simple and effective on-site sanitation technology for treating human urine, alkaline dehydration, was developed and evaluated. By alkalising source-separated fresh human urine, enzyme-catalysed hydrolytic degradation of urea was prevented and by drying the alkalised urine, a solid fertiliser was produced. The combination of these two treatments resulted in near complete recovery of the nutrients, including up to 98% of the nitrogen, and a product containing 10% N, 1.5% P and 6.0% K.

To alkalise urine, an anion-exchanger was initially used, but abandoned in favour of adding source-separated urine directly to an alkaline substrate, as the latter proved to be more practically feasible. When fresh urine was added to any of the alkaline substrates tested, its pH was found to increase to values above 10 and microbial ureolysis was prevented. However, when alkalised urine was dehydrated, it absorbed carbon dioxide from air and its pH was found to decrease gradually. It was concluded that 15 g $Ca(OH)_2$ or 8 g MgO L^{-1} fresh urine should be added to maintain a high pH and to minimise the risk of enzymatic urea hydrolysis during alkaline dehydration treatment.

The recovery of nitrogen from urine was found to be affected by several factors, including the type and amount of drying substrate and co-substrate(s), dryer design, drying rate and drying conditions (temperature, airflow, relative humidity). According to mass balance calculations, most of the substrates recovered urea but not TAN. As recovering all TAN would mean complete nitrogen recovery, this could be one aspect for future studies to explore. The set-ups used to dry urine, both in the laboratory and in the field, were able to achieve drying rates of up to 40 L urine $d^{-1}m^{-2}$. This indicates that a urine dryer could easily be placed inside bathrooms or even integrated into future toilets.

By developing and implementing a pilot-scale urine dryer in the field, this thesis demonstrated that alkaline urine dehydration can work in real-life settings and that it can be scaled up. However, the energy demand for evaporating urine in the pilot-scale system was estimated to be 10 times that of conventional systems (municipal wastewater treatment and synthetic fertiliser production), although similar to that of incineration toilets. Future studies could look into ways of reducing the energy consumption of the dryer.

Elemental analysis of the solid product obtained by dehydrating urine showed that one tonne of dried urine contained 95 kg N, 15 kg P and 80 kg K, which would be enough NPK per hectare for cultivating cereal crops. The analysis also indicated that the salt and heavy metal content of dried urine was similar to that of synthetic fertilisers used in Sweden. Micropollutants such as pharmaceutical residues were detected in the urine sampled from the field experiment, and were found to accumulate in the solid fertiliser. The risk to human and environmental health from micropollutants in dried urine is likely to be minimal, but may have to be addressed if future research points to it being the barrier to consumer acceptance of urine recycling.

To achieve the Sustainable Development Goal of “securing sustainable sanitation for all” (SDG6) by 2030, in the next 10 years a large number of new sanitation systems will have to be installed and old systems will have to be converted across the world. This presents an opportunity to introduce a more circular economy approach to the water and wastewater management sector. One way to manage human excreta differently is to collect and treat different fractions like human urine separately. To make recycling of source-separated urine a more attractive proposition, this thesis developed a simple and effective on-site treatment technology—alkaline dehydration.

8. Future Research

The following research questions may be of interest for future studies:

Urease: It is likely that the pH of urine is not the only parameter affecting the activity of urease, especially when urine is concentrated. Further research is needed to understand the effect of substrate (urea) inhibition, increasing ionic strength and water activity on the rate of enzymatic urea degradation.

On-site stabilisation and off-site drying? In multi-storey buildings, it may be possible to transport the urine collected from all toilets to a centralised alkaline dryer without losing nitrogen to microbial ureolysis. Such a system could be established if urine-separating toilets could be equipped with urine-alkalising reactors that dose urine with substrates such as calcium hydroxide or magnesium oxide.

Drying kinetics and CO_2 absorption: Studies that investigate and model the kinetics of water removal and the rate of mass transfer of CO_2 from air to the different alkaline substrates may also be of value. Identifying substrates and drying conditions that reduce the chemical consumption for alkalising urine can also help reduce the frequency of servicing the dryer with fresh substrate.

Energy demand: The current prototypes of the urine dryer have an energy demand of 600-1200 W L^{-1}. However, it is possible to recover heat energy from the dryer's exhaust air using heat exchangers and heat pumps. The dryer could also be taken off the grid by designing it as a passive (solar/wind) system. In future prototyping, reducing the energy demand must be a central objective since the dryer's electricity consumption is likely to determine its user acceptance and suitability in different contexts.

Clean water from urine: The exhaust air from the dryer can be condensed to recover water, as demonstrated during field testing of the technology in

Finland (Paper IV). This may be of great interest in arid regions of the world. There is preliminary evidence that this water can contain pollutants like ammonia and caffeine. Further research is needed to evaluate the quality of the water produced, the treatment(s) necessary so that it can be reused (for handwashing, toilet flushing, or drinking) and user preferences in such recycling.

Micropollutants: to remove or not to remove? Current understanding of the fate of different micropollutants during alkaline dehydration is poor. It is likely that most micropollutants such as pharmaceutical compounds will not be degraded simply by alkaline dehydration, but little is known about the fate of such substances when dried urine is applied to soils as fertiliser. If future studies show a potential risk to human and environmental health from such recycling, then an additional treatment step that involves removal of micropollutants would have to be integrated with the drying process.

A digital urine-dehydrating toilet for smart homes: Experience from piloting the technology in Sweden and Finland suggests that digitising the dryer can help reduce the energy demand and improve operation of the dryer, recovery of nitrogen and the logistics of collection of dried urine. By leveraging megatrends like the Internet of Things and Machine Learning, it may be possible to develop a smart urine-dehydrating toilet that can be integrated with new smart homes.

Dried urine in agriculture: The output of a urine dryer is a solid powder with a high content of plant-essential nutrients. Trials under greenhouse conditions have shown that dried urine works as well as mineral fertiliser (Martin *et al.*, 2020). However, use of dried urine produced using different substrates still needs to be evaluated at different scales and growing conditions. For large-scale use, dried urine would also have to be homogenised and pelleted.

Willingness to recycle urine: Studies by myself (Simha *et al.*, 2020; Simha *et al.*, 2018; Simha *et al.*, 2017) and others (Segre Cohen *et al.*, 2020; Lienert & Larsen, 2009) have shown that there is broad interest in the idea of recycling urine among farmers, consumers and toilet users. In reality, however, few people actually recycle urine. It is easier to simply flush urine down the toilet and let someone else (municipality) be responsible for its treatment. A sanitation system based on dehydrating and recycling urine, as proposed in this thesis, would have to compete with this status quo.

Therefore, such new systems must provide the same level of comfort and satisfaction as the current gold standard (water-flushed toilets connected to a sewage treatment plant). In many contexts, it is also probable that acceptance of urine recycling will be limited by internal actors in the sanitation sector (*e.g.* utilities, city planners, architects, policymakers, food and fertiliser industries *etc.*), who are reluctant to change for a variety of reasons. Future social research may thus benefit from focusing on other stakeholders in the sanitation chain, especially in contexts where consumer acceptance of the concepts is already high.

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Popular science summary

Access to safe water and sanitation is a basic human right recognised by UN Sustainable Development Goal (SDG) 6. However, global progress towards SDG6 is alarmingly off track. In 2017, 80% of the wastewater produced globally received no treatment and 4 billion people lacked safely managed sanitation. A business-as-usual approach will likely result in the world failing to meet its SDG6 and Agenda 2030 targets.

Human urine contributes 80% of the nitrogen and more than 50% of the phosphorus and potassium in household wastewater, but only 1% of the volume, making it the most nutrient-rich fraction. However, opportunities to recycle urine are limited since it is usually mixed with other household wastewater and sent to treatment plants, which try to remove the nutrients. In NoMix or urine-diverting toilets, urine can be collected separately at source and used as liquid fertiliser. Such recycling is mostly performed today in eco-villages and off-grid homes that have on-site sanitation, since storing, transporting and spreading urine is laborious because the average individual excretes 550 kg urine per year, which by composition is 95% water.

This thesis presents a simple new on-site technology called 'alkaline urine dehydration' that can capture the nutrients in urine without its water. It works by first alkalising freshly excreted human urine to pH values above 10, as this prevents urea, the major nitrogen compound in urine, being broken down by enzymes. If urine is not alkalised, then urea is converted to ammonia by urease-producing bacteria that are ubiquitous in the biofilms of the piping system. When the alkalised urine is dehydrated to evaporate water, its volume is reduced and its nutrients are concentrated. Alkalisation and drying are both done in a "urine dryer" beside the toilet. The dryer is a small box (60×40×20 cm) that contains an alkaline substrate and a fan that provides ventilation and occasionally, heating. Because the water is evaporated, less than 40 kg dried urine per person and year needs to be managed, instead of 550 kg liquid urine. The output of the dryer is a solid fertiliser containing 10% N, 1.5% P and 6.0% K, which can be converted into pellets and spread using conventional farm machinery, so farmers would not have to do anything differently. The salt and heavy metal content of dried urine is also similar to that of synthetic fertilisers used in Sweden.

During drying, more than 95% of the nitrogen and all the potassium and phosphorus excreted in urine are recovered. To capture all the nitrogen, it is important to (a) alkalise fresh urine as soon as it is collected, as this inhibits urease-producing bacteria and the enzyme; (b) choose drying conditions (temperature, substrate) that minimise chemical urea hydrolysis; and (c) capture ammonia in the substrate (*e.g.* as struvite).

The pH of urine gradually drops during dehydration, since urine absorbs CO_2 present in air (about 412 ppm), which converts hydroxides used to alkalise urine to their carbonate forms. Because of this, the dryer needs to be refilled with fresh substrate from time to time, but around 2 kg of magnesium oxide or 8 kg of calcium hydroxide is sufficient to treat all the urine excreted by a person in a year, at a cost of less than US$ 1 per person and year.

Drying is a simple operation that can be performed everywhere. It does not need sophisticated equipment and can be scaled up simply by increasing the supply of air and chemicals and the drying area. However, drying urine also requires almost as much energy as an incineration toilet. There are many ways to reduce the dryer's energy demand, and future work on this will likely ensure wider acceptance and suitability of the technology in different contexts.

Populärvetenskaplig sammanfattning

Tillgång till rent vatten och sanitet är en grundläggande mänsklig rättighet som framhålls i FN:s 6:e Globala mål för hållbar utveckling (SDG 6). Dock, så är progressen mot att uppfylla Mål 6 oroväckande långsam. År 2017 behandlades inte 80% av globalt producerat avloppsvatten och 4 miljarder människor saknade sanitetslösningar som hanterar avfallet säkert. Med denna utvecklingstakt kommer målen relaterade till sanitet i Agenda 2030 inte att uppnås.

Urinen står för 80% av N och >50% av P och K, men utgör endast 1% av volymen av det totala avloppsvattnet från hushåll, vilket gör det till den näringsrikaste avloppsfraktionen. Möjligheterna att återvinna urin är dock begränsade eftersom urinen vanligtvis blandas med resten av hushållens avloppsvatten och skickas till reningsverk som försöker ta bort näringsämnena. Genom att använda urinsorterande toaletter kan urinen samlas in separat i bostaden och användas som flytande gödselmedel. Sådan återvinning utförs idag, mestadels i ekobyar och hushåll med enskilda avlopp, eftersom lagring, transport och spridning av urin är krävande. Detta beror på att vi utsöndrar 550 kg urin per person och år, urin som till 95 % består av vatten.

Denna avhandling har utvecklat en enkelteknik för enskilda avlopp som benämns "alkalisk urintorkning" vilken koncentrerar näringsämnena i urinen genom att ta bort vattnet. Metoden fungerar genom att först alkalisera nyligen utsöndrad humanurin till ett pH över 10, eftersom det alkaliska pH:t förhindrar enzymatisk nedbrytning av urea, den huvudsakliga kväveföreningen i urin. Om urinen inte är alkaliserad omvandlas urea till ammoniak av ureasproducerande bakterier som finns överallt i rörsystemens biofilmer. När den alkaliserade urinen torkas avdunstar vattnet och volymen minskar samtidigt som näringsämnena koncentreras. Både alkaliseringen och torkningen sker på toaletten i en ”urintork” - en liten låda (60 × 40 × 20 cm) som innehåller ett alkaliskt material och en värmefläkt för ventilation och eventuellt uppvärming. Efter att vattnet avdunstas väger den torkade urinen från en person under ett år mindre än 40 kg, detta jämfört med de 550 kg flytande urin som en person utsöndrar per år. Produkten från urintorkningen är ett fast gödselmedel som innehåller 10 % N, 1,5 % P och 6,0 % K, ett material som kan vidareförädlas till pellets och då spridas med konventionella lantbruksmaskiner, så att jordbrukarna kan använda sitt vanliga gödslingsförfarande. Halterna av salt och tungmetaller i torkad urin liknar halterna hos de syntetiska gödningsmedel som används i Sverige.

Under torkbehandlingen bevaras >95 % av kvävet och allt kalium och fosfor som utsöndras i urinen. För att behålla allt kväve är det viktigt att: (a) alkalisera färsk urin så snart den uppsamlas eftersom detta hämmar ureasproducerande bakterier och enzymet; (b) välja torkningsförhållanden (temperatur, substrat) som minimerar kemisk nedbrytning av urea; (c) välja material som kan fånga även ammoniak (t.ex. i form av struvit).

Urinens pH sjunker gradvis under torkningen då koldioxid från luften reagerar med de hydroxider som används för att alkalisera urinen. På grund av detta måste det alkaliska materialet i torken ersättas då och då. Cirka 2 kg magnesiumoxid eller 8 kg kalciumhydroxid räcker dock för att behandla all urin som utsöndras av en person under ett år och kostar mindre än 10 kr per person och år.

Torkning av urin är relativt enkelt och kan utföras överallt. Det behövs ingen sofistikerad utrustning och metoden kan enkelt skalas upp genom att öka tillförseln av luft, kemikalier och torkbäddens yta. Torkningen kräver dock energi, nästan lika mycket som en förbränningstoalett. Det finns många sätt att minska torkens energiförbrukning och framtida studier av detta kommer sannolikt att avgöra teknikens bredare acceptans och lämplighet i olika sammanhang.

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Human urine is the most nutrient-rich fraction of household wastewater but it contains 95% water by composition. This thesis presents a novel technology, called alkaline urine dehydration, which captures nutrients in urine without its water. By drying alkalised urine, a solid fertiliser containing 10% N, 1.5% P, and 6.0% K is produced. About 31 Tg N y^{-1} and 2.8 Tg P y^{-1} could be recycled if it is implemented worldwide.

Prithvi Simha received his PhD education at the Department of Energy and Technology at the Swedish University of Agricultural Sciences. He holds a Joint MSc degree in Environmental Sciences, Policy and Management from the University of Manchester, Lund University, and Central European University and a BTech degree in Chemical Process Engineering from VIT University.

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