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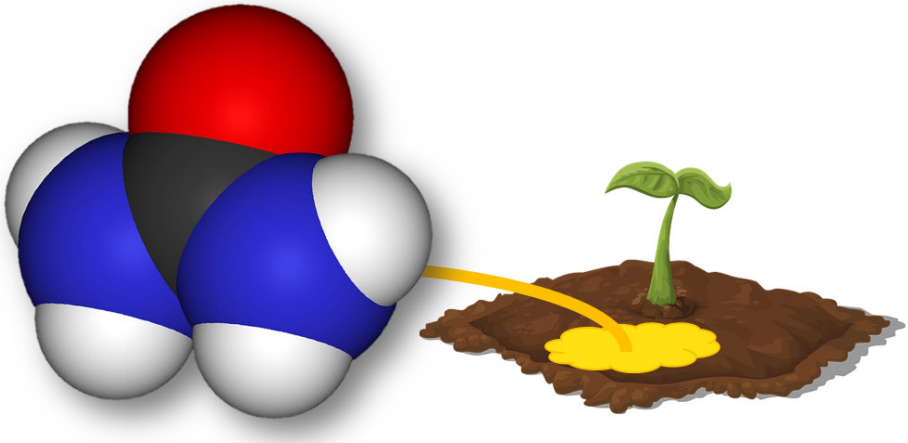
System and Hygiene Evaluation of Alkaline Urine Dehydration

Jenna Senecal



Safe Nutrient Recovery from Human Urine

System and Hygiene Evaluation of
Alkaline Urine Dehydration

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Safe Nutrient Recovery from Human Urine. System and Hygiene Evaluation of Alkaline Urine Dehydration

Abstract

Only 7% of the world's wastewater receives tertiary treatment, while the rest is causing eutrophication, hypoxia and climate change through the plant nutrients found in excreta, particularly urine. If managed adequately, urine can be used as a fertiliser because it contains the same nutrients as the fertilisers used to produce food world-wide. To replace the nutrients removed from fields during harvesting, vast amounts of fertiliser are manufactured and applied, and ultimately more plant nutrients are being released into the environment.

Use of human urine as a fertiliser is limited by its low nutrient concentration compared with commercial fertilisers. This thesis describes efforts to increase the nitrogen (N) concentration from 0.6% to >6% through dehydration to produce a dry urine-based fertiliser, so that no liquid urine disposal from toilets is required. The overall aim of the work was to develop and evaluate alkaline dehydration as a nutrient recycling urine treatment technology. Fresh human urine was added at various intervals to wood ash and biochar to alkalise the material and thus inhibit the enzyme urease, which catalyses hydrolysis of urea. The urine was then dehydrated at temperatures between 35 and 65 °C. A hygiene evaluation was undertaken to monitor inactivation of five microorganisms during and after alkaline urine dehydration.

Urine mass was reduced by 95% during dehydration, while preserving up to 90% of nitrogen and all phosphorus (P) and potassium (K). Inactivation data for a persistent pathogen, Ascaris eggs, were fitted to a non-linear regression model, which estimated that 325 days of open storage would be required for a 3 $\log_{10}$ reduction at 20 °C, compared with 58-110 days in a sealed container. Bacteria and bacteriophages showed a 6 $\log_{10}$ reduction within four days at 20 °C. Simply keeping urine separate from faeces will result in a 4.3 $\log_{10}$ lower pathogen concentration than collecting urine together with faeces.

A truly innovative finding was the final product, a dry powder with 7.8% N, 2.5% P and 10.9% K by dry weight, *i.e.* equivalent to commercial fertiliser. After only four days of storage, the dehydrated medium met world guidelines for unrestricted fertiliser use on non-processed crops in areas not prone to soil-transmitted helminths. By connecting the alkaline dehydration system to new or existing urine-diverting toilets, urine drying can be performed in-house, minimising handling of liquid urine and potentially decreasing the transportation costs of urine-based fertiliser.

Keywords: Alkaline, ash, dehydration, hygiene, pathogen, sanitation, source separation, urease enzyme, urine diversion, volume reduction.

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Säker återhämtning av näringsämnen från mänsklig urin. System- och hygienutvärdering av alkalisk urinuttorkning.

Abstrakt

För nuvarande uppskattas endast 7% av världens avloppsvatten behandlas med tertiär rening, medan resterande avloppsvatten orsakar övergödning, syrebrist och klimatförändringar. Effekterna orsakas av näringsämnen som främst kommer från urinen. Urin kan användas som gödningsmedel eftersom den innehåller samma växtnäringsämnen som det konstgödsel som används för att producera mat. För att ersätta de näringsämnen som tas bort från åkrarna med skörden, tillförs åkern mer gödningsmedel som i slutändan leder till ökat läckage till miljön.

Användningen av humanurin som gödselmedel begränsas av dess låga näringskoncentration jämfört med kommersiellt gödselmedel. Denna studie ämnade att genom torkning öka kvävehalten (N) från 0,6% till > 6% för att producera ett torrt, urinbaserat gödningsmedel och därmed minska det flytande avfallet från toaletten. Det övergripande syftet med avhandlingen var att testa och utveckla alkalisk torkning som urinbehandlingsteknik för återföring av näringsämnen. Färsk humanurin tillsattes med olika intervall till träaska och biokol för att först alkalisera och hämma enzymet ureas som katalyserar ureahydrolysen. Urinen torkades sedan vid temperaturer mellan 35 och 65 °C. En hygienutvärdering genomfördes för att studera inaktiveringen av fem mikroorganismer under och i slutet av den alkaliska torkningsprocessen.

Urinvikten reducerades med 95% under torkningen, medan upp till 90% av N och all P och K bevarades. Inaktiveringsdata för Ascaris ägg anpassades till en icke-linjär regressionsmodell, som uppskattade att 325 dagars öppen lagring skulle krävas för en 3$\log_{10}$-reduktion vid 20 °C, jämfört med 58 till 110 dagars försluten lagring. Bakterierna och bakteriofagerna visade en 6 $\log_{10}$ reduktion efter fyra dagar vid 20 °C. Bara att hålla urinen åtskild från fekalier resulterar i en 4,3 $\log_{10}$ lägre patogenkoncentration än om urinen samlas upp tillsammans med fekalierna.

Den verkligt innovativa funktionen är slutprodukten, ett torrt pulver med 7,8% N, 2,5% P och 10,9% K på torrviktsbasis, dvs. motsvarande kommersiella gödningsmedel. Efter bara fyra dagars lagring skulle det torkade mediet uppfylla WHOs (2006) och USEPAs (1994) riktlinjer för obegränsad gödsling, så länge förekomsten av mag-tarm parasiter är låg.. När ett sådant alkaliskt torkningssystem ansluts till nya eller redan existerande urinseparerande toaletter kan hanteringen av flytande urin minimeras och transportkostnaderna för urinbaserat gödningsmedel potentiellt minskas.

Keywords: Alkalisk, aska, torkning, hygienisering, patogen, källseparation.

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Dedication

To my kids, Gui André and Felicity Anne – may you too follow your dreams.

How wonderful it is that nobody need wait a single moment before starting to improve the world.

Anne Frank

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.**, Gustavsson, D.J.I., Vinnerås, B. 2020. Resource recovery from wastewater: a new approach with alkaline dehydration of urine at source. In: *Current Developments in Biotechnology and Bioengineering: Sustainable Bioresources for Emerging Bioeconomy* (Eds. A. Pandey, R. Kataki, D. Pant, S. Khanal), Elsevier Publications, The Netherlands. (*Accepted, in press*)

II **Senecal, J.** and Vinnerås, B. 2017. Urea stabilisation and concentration for urine-diverting dry toilets: Urine dehydration in ash. *Science of The Total Environment* 586: 650–657.

III 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.

IV **Senecal, J.**, Nordin, A. & Vinnerås, B. 2020. Fate of Ascaris at various pH, temperature and moisture levels. *Journal of Water & Health. (Accepted, in press).*

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

VI **Senecal, J.**, Nordin, A., and Vinnerås, B. 2020. Fate of Ascaris eggs in calcium hydroxide-treated urine in relation to temperature and moisture content. (*Manuscript*).

Papers I & IV are in the process of being published. Papers II, III and V are reproduced with the permission of the publishers.

The contribution by Jenna Senecal to the papers included in this thesis was as follows

I Worked on the initial drafts with all co-authors, with Simha leading writing process. Focused specifically on hygiene and nitrogen loss sections.

II Planned the study with Vinnerås and performed the laboratory work. Wrote the paper together with Vinnerås.

III Designed and performed preliminary trials with the ion-exchange system, with support by Vinnerås. Provided input and revisions with other co-authors to a paper written by Simha.

IV Designed the experiment with support from Vinnerås and performed the experiment. Analysed the results together with Nordin. Wrote the paper with revisions by the co-authors.

V Designed the experiment with support from Vinnerås and performed the experiment. Wrote the paper together with Nordin, with revisions by the co-authors.

VI Designed the experiment with supervision from Vinnerås and performed the experiment. Analysed the results together with Nordin. Wrote the paper with revisions by the co-authors.

Abbreviations

dw	Dry weight (g)
MC	Moisture content (%)
NPK	Elemental nitrogen, phosphorus and potassium in a fertiliser product (%)
RH	Relative humidity (%)
TS	Total solids (g)
VS	Volatile solids (g)
WWTP	Wastewater treatment plant
ww	Weight wet (g)
Compounds	
H_2CO_3	Carbonic acid
H_2N-COOH	Carbamate
$Ca(OH)_2$	Calcium hydroxide or lime
$Ca_{10}(PO_4)_6(OH)_2$	Hydroxyapatite
Cd	Cadmium
Cl	Chloride
$CO(NH_2)_2$	Urea
K_2SO_4	Potassium sulphate anhydrous
KCl	Potassium chloride
KOH	Potassium hydroxide
$Mg(OH)_2$	Magnesium hydroxide
$MgNH_4PO_4 \cdot 6H_2O$	Struvite
$MgSO_4$	Magnesium sulphate anhydrous
N_2	Dinitrogen (nitrogen gas)
NH_3	Ammonia
NH_4^+	Ammonium
NO_3^-	Nitrate
PO_4^{3-}	Phosphate

1 Introduction

Globally, excreta from over 4.4 billion people are not safely managed before entering the environment (WHO & UNICEF, 2017), causing the spread of disease and triggering eutrophication, hypoxia and climate change (Sutton *et al*., 2013). To effectively remove reactive nitrogen from wastewater, advanced nitrogen treatment is required. According to current estimates, only 7% of the world's wastewater receives such tertiary treatment (Paper I). Even with tertiary treatment, there are still reactive nitrogen losses, for example in the form of the potent greenhouse gas nitrous oxide, N_2O (Baresel *et al*., 2016). A wastewater treatment plant (WWTP) removes the plant nutrients found in wastewater by releasing nitrogen back into the atmosphere and precipitating out the phosphorus, which is typically landfilled (Gendebien, 2009), neither of which is 100% efficeint in their removal of nutrients (SEPA, 2016).

The global negligence regarding human excreta management is inexplicable, since it is known that human excreta contain the same nitrogen, phosphorus and potassium (NPK) minerals as the plant fertilisers used to grow the food consumed by humans (Winker *et al*., 2009). In fact, humans readily use animal manure, but generally not human excreta. For centuries, human excreta were highly sought after, particularly in China, Korea and Japan (King, 1911). For example, in 1908 the city of Shanghai sold a contract, at the time worth $31 000 in gold, for the rights to collect, remove and re-sell 71 000 metric tonnes of human excrement. During the same period, the USA and Europe annually poured roughly 4 million kg of nitrogen, 1 million kg of phosphorus and 2 million kg of potassium per million adults into lakes, rivers and seas (King, 1911).

Previously positive attitudes to the use of human excrement started to change towards the end of the 19th century, due to three developments. The first was a growing awareness of the connection between bacteria and the spread of disease *via* excreta (Spink, 1979). The second was the introduction of water closets that transported excreta away *via* pipes in flows of water that were directly

discharged into surrounding surface waters, and thus no longer accessible to be used as fertiliser (SEPA, 2016). The third was the production and accessibility of mineral and synthetic fertilisers (Mårald, 2000; King, 1911). Such fertilisers are easier to transport and apply, as they are more concentrated, and more pleasant to handle, as they smell less. These developments transformed what was previously a circular economy into the linear system that currently dominates around the world. This linear system contributes to environmental change at global level (Steffen *et al*., 2015; Smil, 2002).

Developing a circular economy focused on recycling plant nutrients in the form of human excreta back to agricultural fields would reduce the current dependence on fertilisers derived from non-renewable resources (Ramírez & Worrell, 2006). It could also improve crop yields, for example in sub-Saharan Africa where fertiliser application is low (FAO, 2015), and protect marine ecosystems in many places by limiting the flow of excess nutrients to surface waters (Steffen *et al*., 2015).

Urine, rather than faeces, contains the majority of the NPK excreted. The average person in Sweden excretes in their urine: 80-90% of the total 4 kg of N excreted, 50-80% of the 0.4 kg of P and 80-90% of the 1 kg of K (Vinnerås *et al*., 2006). The main limitation with using urine as a fertiliser is that it is mostly water (97%), meaning that its concentration of plant nutrients is low. For example, the nitrogen concentration in urine (without flush water) is about 0.6% (Vinnerås *et al*., 2006), whereas that of the manufactured fertiliser ammonium nitrate (NH_4NO_3) is 36% N. The lower nutrient concentrations therefore require larger quantities of urine to be applied per hectare. For example, 15 000 L urine ha^{-1} is required, compared with 265 kg NH_4NO_3 ha^{-1}, for a 90 kg N ha^{-1} application rate. Managing such quantities of urine is a logistical problem in terms of storage (since approximately 500 L of urine are produced per person per year) and increases the costs of transportation and application. Hence urine, as it is excreted, is not a competitive fertiliser compared with synthetic fertilisers, which have higher NKP concentrations.

Several excreta treatment technologies under development aim to use the treated urine as a fertiliser (Harder *et al*., 2019; Randall & Naidoo, 2018; Tilmans *et al*., 2015; Udert *et al*., 2015; WHO, 2014; Antonini *et al*., 2012; Vinnerås, 2002). However, such treatment technologies have typically remained difficult to implement, often failing to move beyond the laboratory to the market (McConville *et al*., 2017; Larsen *et al*., 2009; Maurer et al., 2006). Reuse of human excreta as fertiliser, if not properly treated, carries a risk of contaminating the produce by recirculating not only the plant nutrients but also the pathogens

in the excreta (Yajima *et al*., 2009). Adequate sanitisation of the excreta is an essential step to minimise the risk of spreading pathogens (WHO, 2014).

As more people become aware of the environmental impacts of their everyday habits, there is growing demand for a circular economy. Nobody wants to revert back to collecting excreta in buckets in order to recycle the plant nutrients from human excreta back to the fields. Therefore this thesis looked to revolutionise the way in which modern societies handle human excreta.

2 Aims and structure

2.1 Overall aim

The overall aim of this thesis was develop and evaluate alkaline urine dehydration as a safe recycling technology for plant nutrients. Specific objectives were to determine whether alkaline urine dehydration could:

- Function in a circular economy, in terms of what the service chain would comprise in order to treat, collect and convert human urine into dry fertiliser in an urban context (Paper I)

- Retain >80% of the total nitrogen excreted in urine, while increasing the concentration of nitrogen from 0.6% to >6% during dehydration (Papers II & III)

- Inactivate pathogens during and after the dehydration process, to produce a fertiliser that meets the WHO and USEPA guidelines for excreta re-use on non-processed crops (Papers IV, V & VI)

2.2 Structure of the thesis

This thesis is based on the work described in Papers I-VI (Figure 1). Paper I is a book chapter that gives an overview of how the nutrient recycling technology alkaline urine dehydration could function in an urban society. Papers II and III evaluated the effectiveness of preventing biochemical degradation of urea in urine by alkaline treatment, to enable urine dehydration while retaining specifically the nitrogen, but also other plant nutrients present in human urine. In urine-diverting toilets, there is a hygiene risk, mainly from faecal cross-

contamination, so Papers IV, V and VI evaluated whether pathogens are inactivated during and after alkaline urine dehydration.

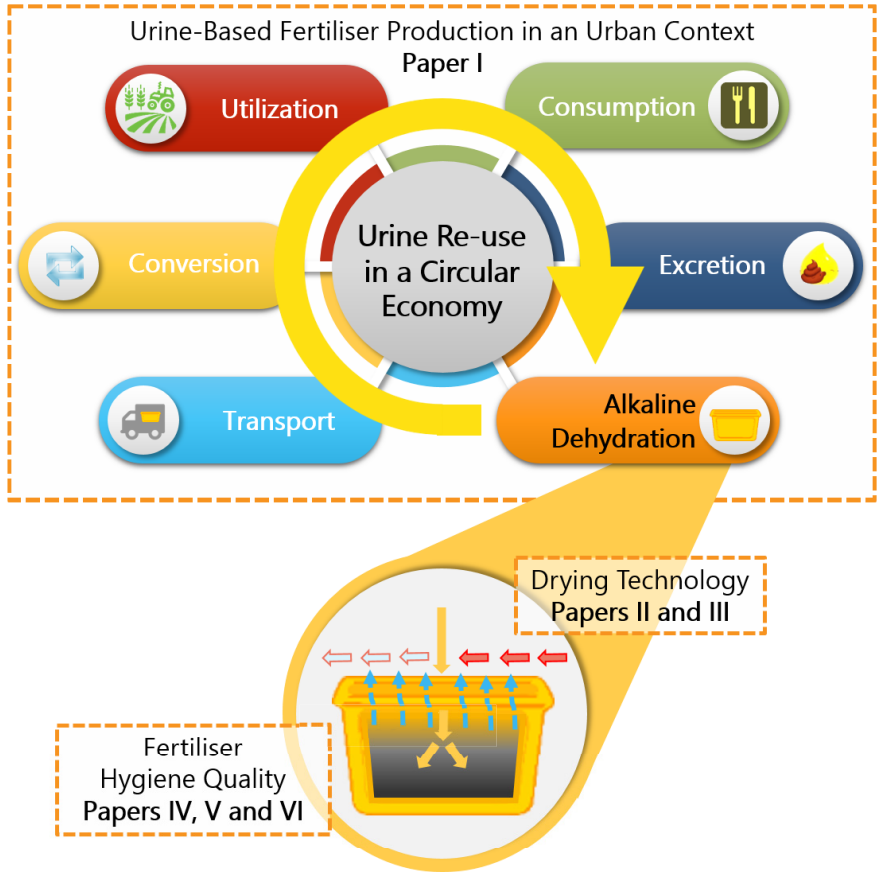

Figure 1. For the alkaline urine dehydration technology to fit in a circular economy, several factors need to be evaluated and services implemented to produce a urine-based fertiliser in an urban context (Paper I). The technology uses urine-diverting toilets, where the urine is led to an alkaline dehydration medium. Alkaline conditions block biochemical degradation of urea and enable evaporation of water through convective dehydration with minimal nitrogen loss (Papers II & III). Inactivation rates of model microorganisms were studied during and after alkaline urine dehydration (Papers IV-VI). (Source: diagram created by modifying images from PresentationGO.com, Annika Nordin and Prithvi Simha.)

3 Background

3.1 Human urine in a circular economy

3.1.1 Motives for source separation

Recycling plant nutrients and water from wastewater is of great significance because, according to Steffen *et al*. (2015), we have already transgressed and distorted our planet's biogeochemical flows of nitrogen and phosphorus and are operating beyond its safe limit. The ambition in this thesis was to shift the mentality from removing the plant nutrients found in wastewater to reusing these plant nutrients (Figure 2).

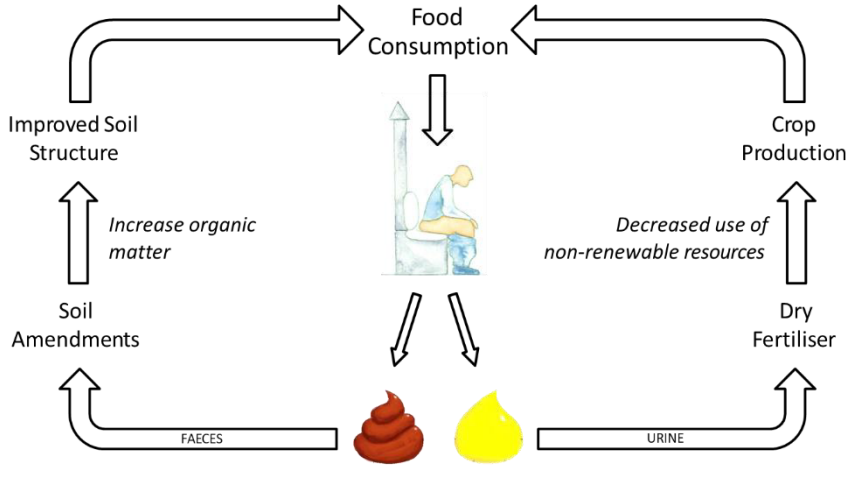

Figure 2. Circulating plant nutrients from the toilet back to fields to grow new food to be consumed and excreted once again. (Source: images of person on toilet, urine and faeces by Donna-Lee Smith)

To enable this shift, an increasing body of research suggests that we need to transition away from the conventional mixed wastewater management towards on-site source separation (Kärrman *et al*., 2017; Larsen *et al*., 2016; Larsen *et al*., 2009; Vinnerås, 2002). The concept involves separate collection of the toilet fractions (urine and faeces) from the other household water fractions (referred to as greywater and includes water from, for example, the shower, sinks and laundry). Mixing the toilet fractions with the other water fractions dilutes the plant nutrients and mixes potential pathogens from the faeces into a greater volume of water to treat.

Collecting the urine separate from faeces and flush water could enable better nutrient and energy recovery from the fractions (Larsen *et al*., 2016), as the composition and characteristics of urine and faeces are quite different. Human urine contains 50-90% of the nitrogen, phosphorus and potassium present in wastewater, while contributing <1% of its volumetric flow (Vinnerås, 2002). Faeces contain more organic carbon (good as a soil conditioner) than urine and are the main contributor of pathogens to wastewater (Feachem *et al*., 1983). Jönsson (2011) suggested that, in Sweden, the economic value of plant nutrients available in urine is at least three-fold higher than that of wastewater sludge. Because of their different characteristics, separate collection, management and end-use of urine and faeces would be desirable. Urine-diverting toilets make it possible to collect human urine and faeces separately at source. These toilets are commercially available and are being used in several parts of the world. However, managing source-separated excreta and safely recycling the streams from households to farmlands is challenging. Typically, the management of on-site treatment systems is left to the household, in contrast to a centralised treatment system where the municipality manages the excreta.

3.1.2 Nitrogen's boundaries

Nitrogen is required by plants more than any other nutrient (based on the number of atoms, excluding carbon from carbon dioxide (CO_2) and hydrogen from water (H_2O)), and is often a limiting nutrient for non-leguminous plants (Camberato, 2011; Jönsson *et al*., 2004). An increase in nitrogen fertilisation levels often results in more plant growth than an increase in any other nutrient (Camberato, 2011), and hence nitrogen in various forms is the most widely produced and used fertiliser worldwide (FAO, 2015). Nitrogen is the most abundant element in the atmosphere, but 99% of it is in inert gaseous form, as dinitrogen (N_2) (Camberato, 2011). Plants are unable to utilise this form of nitrogen and instead mainly use two inorganic forms, ammonium (NH_4^+) and nitrate (NO_3^-), the latter being favoured by most crops (Jönsson *et al*., 2004; Lasa *et al*., 2000).

There are three dominant pathways to fix atmospheric N_2 into a form that is more usable in the biosphere. These are: lightning (5 x 10^9 kg N $year^{-1}$), biological N_2 fixation by an enzyme in prokaryotic microbes called nitrogenase (1.0-1.4 x 10^{11} kg N $year^{-1}$) and the Haber-Bosch N_2 fixation process (1.1 x 10^{11} kg N $year^{-1}$) (Camberato, 2011). Fixation by lightning is too uncontrollable to be used as a regular production method and there are only a few bacteria and algae that utilise the N_2-fixing enzyme (Prosser, 2011). Thus the Haber-Bosh process has become the dominant pathway for fixing atmospheric N_2 and converting it into a reactive plant-available form.

The mass of reactive nitrogen in the biospheric cycle has doubled since the pre-industrial era, causing aquatic, terrestrial and atmospheric changes at local and global level (Cassou, 2018; Steffen *et al*., 2015; Smil, 2002). The global capacity to deal with reactive nitrogen has been exceeded more than two-fold (Steffen *et al*., 2015). The proposed global capacity (or boundary) for reactive nitrogen is to limit industrial and agricultural fixation of N_2 to 62 x 10^9 kg N $year^{-1}$ (or approximately 41% of N_2 fixed in the terrestrial ecosystem per year) (Steffen *et al*., 2015).

The reactive nitrogen produced is used inefficiently today. Nitrogen fertiliser is manufactured and applied to fields, from where 50% of the nitrogen applied commonly enters the environment, largely as reactive nitrogen, through leaching, volatilisation and as N_2 and N_2O through nitrification and denitrification processes in the soil (Cassou, 2018; Smil, 2002). The harvested crops are used for human and animal consumption. The nitrogen from the food consumed, particularly by adults, is subsequently excreted, predominantly in urine (Jönsson *et al*., 2005). An estimated 56% of human excreta worldwide are not treated at all before entering the environment (WHO & UNICEF, 2017), and thus most of the nitrogen flows into aquatic ecosystems, where it is either denitrified into N_2 (desired) or contaminates the aquifer with high nitrate levels, causing eutrophication (not desired) (Smil, 2002). The nitrogen lost from the fields through the harvested crop, leaching *etc.* is then replaced with newly manufactured mineral nitrogen fertiliser, further pushing the nitrogen planetary balance beyond its threshold as more reactive nitrogen is released into the environment (Rockström *et al*., 2009). The amount of reactive nitrogen released into the environment could be decreased with more efficient nutrient use and management, such as recycling human excreta as fertiliser (Rockström *et al*., 2009; Steffen *et al*., 2015; Smil, 2002).

3.2 Dehydrating urine

3.2.1 Urine chemistry

The nitrogen in freshly excreted urine is predominantly found as urea (~85%), ammonium and uric acid, which together account for 90-95% of total nitrogen in urine (Kirchmann & Pettersson, 1994). The urea in fresh human urine is stable in the absence of active urease enzymes (urea amidohydrolase, EC 3.5.1.5) (Feng *et al*., 2008). Urease enzymes are most commonly known for their role in converting urea into ammonia (NH_3), for example in soils fertilised with urea. These enzymes are found in most environments and, unknown to most toilet users, human faeces contain large amounts of urease-forming bacteria (Wozny *et al*., 1977). Urease enzymes are found in toilet piping systems due to cross-contamination with faeces and due to biofilm formation on pipe surfaces, both of which can cause rapid hydrolysis of urea, even in urine-diverting dry toilets (Vinnerås & Jönsson, 2002).

Once urine is excreted into the toilet, the urease enzymes present in the toilet quickly hydrolyse urea ($CO(NH_2)_2$) into carbamate (H_2NCOOH), volatile ammonia and water (Equation 1). The carbamate then spontaneously hydrolyses into carbonic acid (H_2CO_3) and releases a second ammonia molecule (Krajewska, 2009). Ammonia gas is highly soluble in water, but the concentration is dependent on temperature and pH. The amount of $NH_{3(aq)}$ lost to the air above the water is proportional to the partial pressure of $NH_{3(g)}$ above the solution (Zumdahl & Zumdahl, 2000). Hence, in a urine dehydration system with a large air flow, the NH_3 would largely be lost with the water during the process. It is difficult to prevent the urea hydrolysis reaction (Equation 1), since urease enzymes are found essentially everywhere.

$$CO(NH_2)_2 + 2H_2O \xrightarrow{Urease} H_2NCOOH + NH_3 + H_2O \longrightarrow H_2CO_3 + 2NH_3 \quad \textit{Equation 1}$$

Ureases are a group of highly efficient natural enzymes used by plants, algae, fungi and several microorganisms to catalyse the hydrolysis of urea to extract the nitrogen for use in the synthesis of proteins, amino acids, DNA and RNA (Jørgensen, 2009; Ciurli *et al*., 1996). Urease is an extracellular enzyme that can be immobilised on particles, where it continues to degrade urea (Ciurli *et al*., 1996). All urease enzymes have two nickel (Ni^{+2}) ions located on an active site (Blakeley *et al*., 1982). The size of the enzyme varies with the different urease-forming bacteria (enzyme molar mass ranges from 190-300 kDa), as does the speed of hydrolysis and the optimal pH conditions (Krajewska, 2009).

To limit the urease enzymes in agricultural soils, inhibitors such as N-(n-butyl) thiophosphoric triamide have been developed to slow down the release of nitrogen from urea fertiliser, in order to decrease the amount of soluble nitrogen in the soil at a given time and thus decrease the amount lost due to leaching and volatilisation (Parker *et al*., 2012). However, due to the potential risks to human and environmental health (Ciurli *et al*., 1999), such inhibitors cannot be considered a viable option for blocking the enzymes in urine at household level. The urease enzymes can also be inhibited by pH (>10) and temperature (>80 °C) (Geinzer, 2017; Hotta & Funamizu, 2008; Kabdaşlı *et al*., 2006; Huang & Chen, 1991; Sizer, 1940).

At elevated pH (>10) urease enzyme activity may be inhibited, but urea can start to degrade at pH ≥12 by an elimination reaction with a hydroxide ion (OH^-) (p*K*a′ of urea ≈ 14) as the base catalyst (Equations 2 and 3) (Zerner, 1991; Blakeley *et al*., 1982). With regard to temperature, urea is very stable at 25 °C in neutral pH with a half-life of 40 years, but as the temperature increases the half-life decreases exponentially (Table 1). However, the elimination reaction and the thermal degradation of urea take $>10^7$ times longer than enzyme-catalysed hydrolysis (Table 1). Hence the priority in this thesis was to limit the urease enzyme by elevating the pH to enable dehydration of the urine with minimal nitrogen loss.

$$H_2NCONH_2 + OH^- \rightleftharpoons HNCONH_2 + H_2O \qquad \textit{Equation 2}$$

$$HNCONH_2 + H_2O \rightarrow HNCO + NH_3 + OH^- \qquad \textit{Equation 3}$$

Table 1. Examples of the half-life of urea (t½) depending on the temperature, pH and catalyst

Conditions	t½
Uncatalysed	
25 °C (neutral pH)[z]	40 y
38 °C (2< pH <12)[y]	3.6 y
65 °C (2< pH <12)[x]	15.3 days
Base catalysis	
20 °C (pH >10) [w]	Negligible for 32 days
65 °C (pH >12.5)[x]	14.1 days
Enzymatic catalysis	
Jack-bean (neutral pH, 25 °C)[z]	0.02 s

[z](Callahan *et al*., 2005).
[y](Zerner, 1991).
[x]Derived from Warner (1942).
[w](Kabdaşlı *et al*., 2006).

3.2.2 Emerging technologies for recirculating human urine

Over the past two decades, considerable amounts of research have been performed to develop technologies for treating mainly hydrolysed source-separated human urine. According to Harder *et al*. (2019), two broad strategies exist for treating human urine. The first selectively extracts plant nutrients present in human urine, *e.g*. phosphorus by precipitation (Etter *et al*., 2011), urea by adsorption (Ganesapillai *et al*., 2015), ammonia (NH_3) by stripping (Tarpeh *et al*., 2018) or ammonium (NH_4^+) by ion-exchange (Tarpeh *et al*., 2017). The second strategy removes water from urine, thereby concentrating its plant nutrients, and produces concentrated liquids, slurries or dry solids. The processes used include nitrification-distillation (Udert *et al*., 2015; Feng *et al*., 2008) and forward osmosis (Volpin *et al*., 2019; Liu *et al*., 2016). The implementation and acceptance of such technologies have been limited thus far, due to the technologies still being in the development phase and lack of an organised system for reusing the urine (McConville *et al*., 2017).

The strategy of removing water from urine is attractive, as potentially all the plant nutrients can be retained. However, nitrogen can be a challenge to retain during dehydration if the urine is hydrolysed (urea degraded to ammonia). A pre-treatment that limits urease activity to prevent the hydrolysis of urea in the urine can enable dehydration with limited nitrogen loss. Examples of stabilisation techniques include acidification (Hellström *et al*., 1999), alkalisation (Dutta & Vinnerås, 2016; Randall *et al*., 2016) and salinisation (Pahore *et al*., 2010). When the urease enzyme activity is limited by elevated pH and/or elevated temperature, urea degradation still occurs, but at a much slower rate (Jespersen, 1975) (Table 1).

Alkalisation of urine is an attractive option as there are several sources of strong bases available. Slaked lime ($Ca(OH)_2$) has been demonstrated to effectively increase the pH of urine to 12.46 and to inhibit urease activity in a chemical reactor prior to drying in a separate system (Randall *et al*., 2016). Other alkali salts such as magnesium hydroxide ($Mg(OH)_2$), potassium hydroxide (KOH) and sodium hydroxide (NaOH) are available, and could potentially be used to inhibit urease activity. The end-product (fertiliser) would still be alkaline, which could be beneficial as alkalisation is often needed due to soil acidification from fertiliser application (in particular urea) (Goulding, 2016).

Wood ash is also alkaline (pH 9 to 13.5) and is a common by-product in Sweden (Karltun, 2008; Etiégni & Campbell, 1991). The major components in ash are calcium (Ca), potassium, magnesium (Mg), silicon (Si) and phosphorus, with OH^- being the main source (92%) of the soluble alkalinity (Etiégni & Campbell, 1991). During combustion, the biomass is oxidised and the inorganic species are freed and transformed into oxides, hydroxides, chlorides, sulphates,

carbonates (such as $CaCO_3$) and crystalline potassium salts (such as K_2SO_4, KCl and $K_2Ca(CO_3)_2$) (Steenari, 1998). The pH and the relative element abundance in wood ash depend on the source of the wood, but also on the temperature and method of combustion (Etiégni & Campbell, 1991).

The pH of urine can also be increased by anion exchange. Ion exchange is a method commonly used to soften water or sometimes in wastewater treatment plants to remove nitrogen, sulphates and heavy metals (Tchobanoglous *et al*., 2003). Anions present in urine can be exchanged for OH^- when passed through a resin, such as the strong basic resin Amberlite™ IRA 410 type 2 (dimethyl ethanol ammonium). Amberlite has a molar equivalent of 1.25 eq L^{-1} where hydroxide anions are exchanged with the anions in the urine, increasing the pH. In human urine, the anions present, in order of strength of charge, are $HCO_3^- < Cl^- < CO_3^{2-} < SO_4^{2-} < PO_4^{3-}$ (Ferslew *et al*., 2001; Putnam, 1971). The anion present in the highest concentration in urine is Cl^-. Theoretically, the average exchange capacity of urine is 0.23 eq L^{-1} (calculated based on the ionic mass of urine measured in Ferslew *et al*. (2001) and Putnam (1971)), giving a potential volume ratio (urine to resin) of 5.4:1.

3.2.3 Dehydration from a porous medium

Dehydration is the simultaneous process of transferring heat and mass (Anderson, 2014; Gavrila *et al*., 2008). Heat enters a product, while mass (as moisture) is removed by evaporation into an unsaturated gas (Harrison *et al*., 2015; Kutz, 2013). In this thesis, air convection was the main mechanism used to transfer heat to the urine contained in and around the dehydration medium. Below the surface, conduction is another mechanism for heat transfer. In such a system there are several different heat and mass transfer processes happening at the same time, such as heat and moisture exchange between the medium and the air, absorption and desorption rates of heat in the medium and the psychometric properties of the air (Gavrila *et al*., 2008).

The depth to which the medium is dehydrated is dependent on the diffusion and transportation of moisture in the medium. Diffusion of moisture is caused by gradients in moisture, in partial vapour pressure and in temperature within the medium (Gavrila *et al*., 2008). For a liquid with high ionic strength, such as urine during dehydration (fresh urine 19.5 mS cm^{-1}; Putnam, 1971), osmotic forces could also be a mass transfer mechanism. Ash and biochar (alkaline media used for urine dehydration in this thesis) are porous materials, where capillary flow and diffusion would be mass transfer mechanisms of moisture below the surface (Harrison *et al*., 2015; Gavrila *et al*., 2008).

Converting liquid water to vapour requires a large amount of energy, 2451 $kJ\,kg^{-1}$ at 20 °C or 586 times more energy than required to raise the temperature of an equal quantity of water by 1 °C (Tsotsas *et al*., 2011). Urine contains non-volatile solutes (such as Na^{+}), which would elevate the boiling point compared with that of distilled water and would thus require more energy per kg of urine. This difference is referred to as the boiling point elevation (BPE) of the solution (Harrison *et al*., 2015). In convective dehydration, the heat provided has been found to be consumed by evaporation (20-60%), heating the medium (5-25%), heat loss with the air exhaust (15-40%) and heat loss within the dryer (through the wall to the atmosphere) (3-10%) (Strumiłło *et al*., 2014).

The rate of drying changes during the course of dehydration and the changes can be represented in four stages (Kutz, 2013) (Figure 3). Stage 1 of dehydration is the transient period or lag phase, *i.e.* the period of time required for the medium to absorb energy (heat) and reach the wet-bulb temperature (the lowest temperature to which air can be cooled by evaporation of water into the air at a constant pressure (Razak, 2007)). This is dependent on the rate of heat transfer, in the case considered in this thesis from the warm air to the wet medium.

Stage 2 occurs when the evaporation rate is constant. During this stage, the surface of the medium is saturated and the temperature remains close to the wet-bulb temperature (heat transfer) and water vapour forming at the surface is taken up by the large warm mass of air passing over (mass transfer) (Figure 3). The greater the difference between the air temperature and medium surface (wet-bulb temperature), the greater the potential heat transfer (Kutz, 2013). The mass transfer rate is the gaseous diffusion of the liquid from the surface into the surrounding air (Harrison *et al*., 2015). The diffusion at the surface is limited by the difference in relative humidity in the air and at the surface of the medium, and is dependent on the rate at which air is exchanged (Kutz, 2013). The dehydration during stage 2 removes unbound water that is freestanding on the surface (the liquid is not adsorbed to the medium, also known as bulk water). By raising the incoming air temperature, the holding capacity of the incoming air will be increased, as will the heat transfer, which in most cases will increase the evaporation rate.

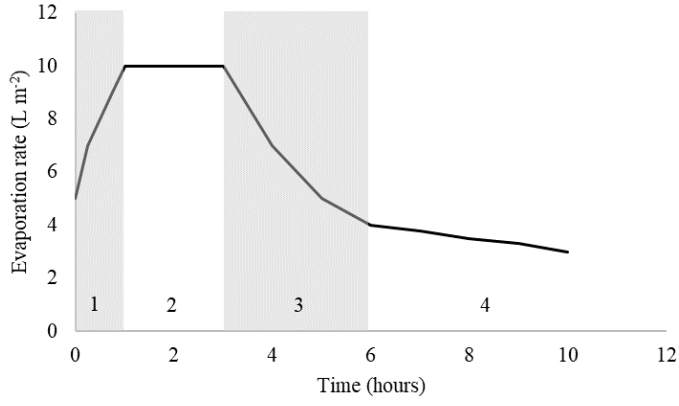

Figure 3. Example of the four stages of dehydration of liquid from an initially cold and saturated porous medium with the evaporation rate changing with time and decreasing moisture content (not shown). (Source: based on Kutz (2013))

The dehydration shifts from Stage 2 to 3 (the falling rate period) when the critical moisture content is reached. This is the point at which surface water content decreases to a level where the hydraulic capacity (mechanisms such as capillary action or liquid/gas diffusion) of the medium is unable to supply enough liquid to the surface to maintain saturation, and therefore the dehydration rate decreases (Jensen & Allen, 2016; Kutz, 2013). The remaining liquid is either bound or below the surface (Kutz, 2013) and the diffusion and transport of the liquid to the surface lags the evaporative capacity (Jensen & Allen, 2016). In porous material, a final dehydration, Stage 4, can occur (Figure 3). It is referred to as the second falling rate period, in which the dehydration rate decreases even further as only bound water remains (Kutz, 2013).

The evaporation rate in Stages 3 and 4 is limited by: (1) conduction of sensible heat (heat transfer) to the lower layers of the medium and (2) diffusion/transportation of vapour and liquid from the lower layers to the surface (Jensen & Allen, 2016). The energy efficiency decreases in Stage 3, and even more in Stage 4, because then the heat is being used to further heat the now dry surface and the medium below, to reach a high enough temperature for the removal of the bound water (Strumiłło *et al*., 2014).

Bound water occurs in several forms. In porous material, the water can be bound in small pores – the smaller the pore size, the larger the curvature of the meniscus of the water in the pore and the stronger the capillary strength (Harrison *et al*., 2015). Water can be bound due to osmotic pressure (by electrostatic and/or hydrogen bonds) and in either physical or chemical combination with the solid (Harrison *et al*., 2015). As urine is concentrated, the

salts bind the water in the urine, which may further limit the evaporation rate. Typically, the actual evaporation rate is much lower than the potential evaporation rate due to the additional energy required to free the molecules of water bound to salt or by capillary action and due to the heat loss in the system (such as through the walls) (Tsotsas *et al*., 2011).

3.3 Pathogens

3.3.1 Pathogens in excreta

Urine in the human bladder is typically pathogen-free in healthy individuals (Willey *et al*., 2009). Even in infected individuals, only a few pathogens are excreted *via* the urine, compared with faeces (Aw, 2018), and are rarely abundant or widespread enough to be a significant public health problem (Höglund, 2001). Thus the risk related to the reuse of human urine is often considered insignificant or limited, particularly in temperate climates (Aw, 2018; WHO, 2006; Höglund, 2001; Feachem *et al*., 1983).

Although the pathogens excreted in urine are regarded as a limited risk, there is a risk of cross-contamination from the faeces to the urine during excretion and collection in urine-diverting toilets (Höglund *et al*., 2000). Faeces can potentially contain many pathogenic viruses, bacteria, protozoa and helminths, many of which are immediately infectious upon excretion (Aw, 2018). Just one gram of faeces can contain 10^9 infectious virus particles without the human host necessarily exhibiting clinical signs (Feachem *et al*., 1983). A cross-contamination rate of 9.1 mg of faeces per litre of urine has been estimated for urine-diverting toilets (Schönning *et al*., 2002) – a new estimation is required for the new urine-diverting toilets. By reusing the urine in the proposed system, in parallel to an existing system, an additional pathway for pathogen exposure is created. This is a risk that has to be considered. Hence hygiene evaluations on urine treatment technologies are important, as there is a potential health risk to the people handling and/or using diverted urine as a fertiliser (WHO, 2006).

3.3.2 Model organisms

When assessing the effectiveness of a sanitisation treatment, model organisms are typically used. The ideal model organism has a similar or greater survival rate than the enteric pathogen it represents, while not multiplying in the environment (Copper & Olivieri, 1998). Defining a reliable model is difficult as, for example, four bacterial strains studied in a biological wastewater treatment

behaved differently (Wéry *et al*., 2008), meaning that just one bacterial strain will not represent all bacteria. However, it is too time-consuming and costly to have models for all relevant pathogens. To compensate for this diversity in behaviour between strains and organisms, the thesis used five model organisms that were selected based on their persistence and history of being used as model organisms.

Viruses

Human virus surrogates (such as bacteriophages) are commonly used to represent enteric viruses (Decrey & Kohn, 2017; Jebri *et al*., 2017; Decrey *et al*., 2016; Magri *et al*., 2015; Bertrand *et al*., 2012;). Two bacteriophages were used in the studies included in this thesis, MS2 and ΦX 174. MS2 is a linear ssRNA, non-enveloped, bacteriophage that has been shown to be less sensitive than human adenovirus in urine (Decrey, 2015). ΦX 174 is a circular ssDNA, non-enveloped, bacteriophage that is highly persistent within several different media and temperatures, making it a useful indicator for virus inactivation studies (Bertrand *et al*., 2012).

Bacteria

Two bacterial model organisms were studied, *Salmonella enterica* and *Enterococcus faecalis*. *Salmonella enterica* subspecies *enterica* serovar Typhimurium (hereafter referred to as *S*. Typhimurium) was used instead of other gram negative bacteria because *Salmonella* spp. have high survival rates (Hasan *et al*., 2019). *Enterococcus faecalis* is a commonly-used conservative indicator bacteria (Momba *et al*., 2019) which can be more resilient than *S*. Typhimurium in dry alkaline treatments (Magri *et al*., 2013).

Parasites

Eggs of *Ascaris* (roundworm) spp. are often used as a conservative indicator, because they are one of the most persistent human enteric pathogens (Maya *et al*., 2010; Pecson *et al*., 2007; Jiménez, 2006; Feachem *et al*., 1983). Compared with other parasites, such as *Cryptosporidium* spp. oocysts and *Giardia lamblia* cysts, *Ascaris* spp. eggs are more resilient to alkaline and elevated temperature (Capizzi-Banas *et al*., 2004; Kato *et al*., 2003). *Ascaris lumbricoides* is the species that infects humans (Pawlowski, 1982), but for practicality, *Ascaris suum*, the species that infects pigs, is typically used for studying Ascaris inactivation (Johnson *et al*., 1998; Feachem *et al*., 1983). *Ascaris suum* eggs are easier to acquire and they tend to behave similarly or more conservatively than *A*. *lumbricoides* eggs in treatments (Pecson *et al*., 2007; Ghiglietti *et al*., 1995).

4 Materials and methods

4.1 Urine in a circular economy (Paper I)

Alkaline dehydration of urine is a new type of sanitation technology focused on resource recovery rather than pollution management. However, the way in which this technology could fit into a circular economy with new sanitation systems or existing infrastructure has not been described previously in terms of urine management. An attempt was made to address this gap in Paper I, using results from different experiments (such as those described in Papers II, III and V), experiences from pilot tests on the dehydration technology in Sweden, France and Finland, and data in the literature concerning decentralised sanitation systems (for full reference list, see Paper I).

4.2 Urine and faeces collection (Papers II-VI)

Human urine and faeces (Paper II-VI) were collected anonymously from between five to 10 people aged between 24 and 65. Upon excretion, the urine and faeces were collected and stored in sterile containers at 3 and -18 °C, respectively, until use.

4.3 Dehydration (Papers II & III)

Dehydration of urine was assessed at four temperatures, 35, 40, 50 and 65 °C, with an air flow of 750 L min^{-1}. Three dehydration media were used: ash from wood pellets (Paper II), ash from birch trees (Papers II & III) and biochar from chopped willow trees (Paper III). Wood ash had a high initial pH (>12), whereas the pH of biochar (initially <9) was increased to >12.5 with potassium hydroxide (KOH). In three of the trials (Table 2), the urine was pre-treated with an anion

exchanger, Amberlite™ IRA410 type-2 resin, to increase the pH of urine to ≥10 before being added to the medium, to assess whether this could help maintain elevated pH in the dehydration media over a longer period.

Two dosing methods were tested: Static loading, where all dehydration medium was added at the beginning, and dose loading, where the medium was added with each urine application at 5% (w/w) ash/urine. In the 35, 40 and 50 °C trials, the nitrogen concentration was measured as total dry combustion of nitrogen (LECO TruMac® CN, USA) at the end of the experiment. In the 65 °C trial, the nitrogen concentration in the ash treatments was measured three times (on days 20, 33 and 41). The ash was first incubated with urease enzymes to convert the urea to NH_4^+, and then nitrogen was measured as NH_3-N by an ammonia electrode probe (Metrohm AG, Switzerland). The initial and final phosphorus (as phosphate-P, PO_4-P) and potassium concentrations were analysed using ICP (ICP Optima 7300 DV Swedish Standard: SS 02 83 11).

Table 2. *Treatments applied to dehydration media after all were thermally pre-treated. In addition to thermal treatment, biochar was dosed with KOH to increase the initial pH to >12.5. In three trials, the urine was pre-treated by anion exchanger to increase the pH to >10. Temp. is the temperature during dehydration. In static loading, all dehydration medium was added at the beginning. In dose loading, the dehydration medium was added with each urine application at 20:1 (urine:ash, w/w)*

Medium	Pre-treatment		Temp. (°C)	Loading method
	Media	Urine		
Birch ash	65°C (24 h)	-	35	Static Dose
	500°C (5 h)	Ion-exchange	40 50	Static
	65°C (24 h)	-	65	Static Dose
Wood pellet ash	65°C (24 h)	-	65	Static Dose
Biochar	KOH/110°C (12 h)	Ion-exchange	45	Static

4.4 Hygiene evaluation (Papers IV, V & VI)

Inactivation rates of microorganisms were studied in two alkaline media used for alkaline urine dehydration, ash and lime, and various controls to isolate the effect of temperature, moisture content and pH (Table 3). At the time of the experiments, the design of the alkaline urine treatment system was not finalised,

so inactivation of the model pathogens was assessed in three treatment situations. In the first situation, the urine/medium solution was dried and then placed in sealed containers for storage at 20 and 35 °C. In the second situation, the urine/medium solution was directly stored at 20 and 35 °C in either sealed or open containers. In the third situation (thermal treatment), the urine/medium solution was stored at 42 and 50 °C in either sealed or open containers. The intention was to use the measured inactivation rates in the various situations to provide handling recommendations, in terms of hygiene, for the urine/medium solution to produce a urine-based fertiliser that would meet the WHO (2006) and USEPA (1994) guidelines for unrestricted fertiliser use.

To prepare the ash medium, urine (without model organisms) was dehydrated in ash until the pH reached 10.5 (as described in Paper III), after which the five indicator organisms were pre-mixed with faeces added to the urine/ash (Paper V). Organisms were inoculated into faeces because faeces material has been shown to have a protective effect (Capizzi-Banas *et al*., 2004; Ghiglietti *et al*., 1997; Eriksen *et al*., 1995). The urine/ash samples were left open to the air for the duration of the treatment at two temperatures: at 20 °C, to simulate ash being stockpiled until its use as a fertiliser (once or twice a year), and at 42 °C, to simulate a final dehydration step.

One organism (*A. suum*) was evaluated in lime (Paper VI). To prepare the lime medium, 10 g of $Ca(OH)_2$ were added per L of urine and mixed for 10 minutes (pH 12.5). From this mixture, three fractions were formed, mixed, supernate and precipitates. The mixed fraction was the whole agitated mixture, representing all the solution being treated and dried together. The supernate and precipitate fractions were made by decanting the solution after standing for 3 hours. These represented a situation where, after the urine was stabilised with $Ca(OH)_2$, the top liquid (supernate) was pumped out of the reactor for drying, while the solids (precipitates) were removed and dried separately. The model pathogens were pre-mixed into faeces before being added to the specific treatment.

Table 3. *Treatments studied for inactivation of* Ascaris suum *eggs in various media (urine/ash, urine/lime, urine, buffer and soil), at various temperatures (20 to 50 °C), in three moisture conditions (wet >97%; partially wet 60-82%; dried <33%) and pH (7.2 to 12.5). * indicates inactivation of* Salmonella *Typhimurium,* Enterococcus faecalis *and the indicator organisms MS2 and ϕx174, which were assessed in the same conditions*

Treatment	Storage Temperature	Media	Fraction	Conditions	Drying (days)	Storage (days)	# samples	pH	Paper
Drying (open) followed by storage (closed)	20 °C	Urine/lime	Supernate	Partially wet	8	91	7	12.5	VI
				Dried	2	91	9		
			Mixed	Partially wet	9	90	9		
				Dried	2	126	6		
			Precipitates	Partially wet	8	91	10		
				Dried	2	91	6		
	35 °C		Supernate	Partially wet	8	77	6		
				Dried	4	63	5		
			Mixed	Partially wet	8	77	6		
				Dried	4	91	6		
			Precipitates	Partially wet	8	77	6		
				Dried	2	79	7		

Storage	20 °C	Urine/lime	Supernate	Wet	-	126	10	12.5	VI
			Mixed				4		
			Precipitates				9		
		Urine/ash	-	Partially wet	346	-	27	> 10	V*
		Fresh Urine	-	Wet	-	116	5	8.5	
		Buffer	-	Wet	-	94	5	7.2	IV/V*
						126	7	10.5	
						126	7	12.5	IV
		Soil	-	Partially wet Dried	-	98 94	5 9	7.2	IV
	35 °C	Urine/lime	Supernate	Wet	-	49	4	12.5	VI
			Mixed			126	4		
			Precipitates			77	5		
		Buffer	-	Wet	-	93	5	7.2	IV
						126	8	10.5	
						96	6	12.5	
		Soil	-	Partially wet Dried	-	98 94	5 6	7.2	
Thermal Treatment	42 °C	Urine/lime	Supernate	Wet	-	16	4	12.5	VI
		Urine/ash	-	Partially wet	14	-	8	> 10	V
		Buffer	-	Wet	-	16	4	7.2	IV
						50	5	10.5	
						16	5	12.5	
	50 °C	Urine/lime	Supernate	Wet	-	32 h	3	12.5	VI
		Buffer	-	Wet	-	34 h	4	7.2	IV
							4	12.5	

5 Results

5.1 Urine in a circular economy (Paper I)

Alkaline urine dehydration, as the name suggests, involves alkalising human urine and dehydrating the urine into a dry end-product. This is a two-step, on-site treatment technology. First, fresh human urine collected from a urine-diverting toilet or a urinal is added to an alkaline substrate (*e.g.* lime, wood ash) (Figure 4). This addition increases the pH of fresh urine to >10, which is necessary to biochemically stabilise urine as it otherwise undergoes a natural hydrolysis reaction (see section 3.2.1). The urine added to the alkaline substrate is then dehydrated using forced ventilation, to yield a dry, nutrient-rich solid fertiliser (also referred to as 'Granurine'). Since by composition urine is 95% water, dehydration can reduce the volume of urine by >90%.

To implement urine dehydration technology, a semi-decentralised sanitation system is proposed in this thesis, where source-separated urine is treated in-house and separately from the faeces. The faeces, wiping material, anal cleansing water and/or flush water are treated by other technology, *e.g.* utilising waste water treatment plants if already in place (Figure 4). For the alkaline urine dehydration system to work, the urine needs to be collected separately during excretion to minimise cross-contamination with the faeces and have minimal pipe transport to minimise time for urea hydrolysis. To minimise pipe transport, the alkaline urine dehydration system should be ideally located in the toilet cubicle and directly connected to the urine-diverting toilet. No alteration to the house's piping network would be required, and thereby the system can be an option for new buildings and for retrofitting existing toilets with a urine-diverting system.

The pH of the alkaline substrate used to dehydrate the urine decreases over time, and once the pH is <10.5 the urine containment unit (also referred to as a

cassette) needs to be replaced with a new one. The amount of alkaline medium present in the unit, the dehydration conditions (mass of air flow, temperature and humidity) and the number and frequency of toilet users will determine how long the containment unit can stay in operation (see sections 6.1 and 6.2). This thesis focuses on the urine fraction, as it contains the majority of the plant nutrients found in wastewater.

Collection and transfer (Figure 4) of the used cassettes can be performed in a system similar to that used for recycling in Sweden, with local stations (*e.g.* outside private apartment blocks or outside grocery stores) for collection of cassettes before efficient transport to the conversion plant. At the conversion plant, Granurine could be pelleted and bagged to be sold as a urine-based fertiliser with NPK concentrations comparable to those in conventional fertilisers. Implementation of this alkaline system is further discussed in section 6.1.

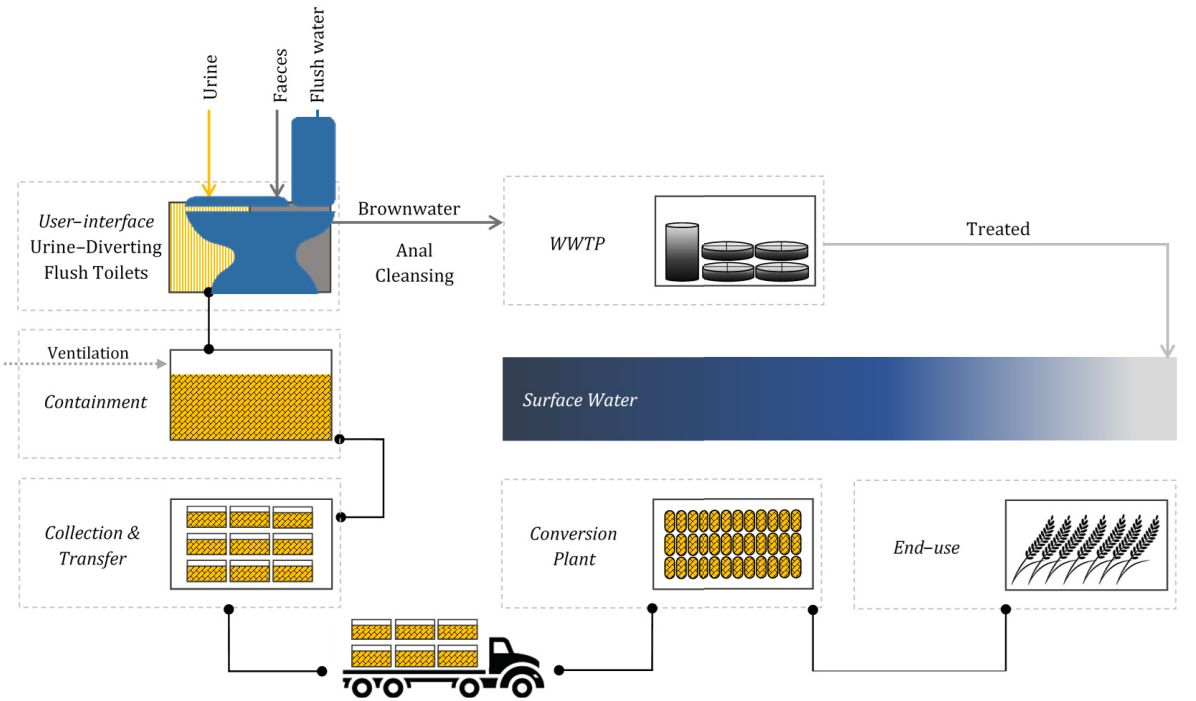

Figure 4. Schematic representation of proposed alkaline urine treatment sanitation system using a urine-diverting-flush toilet. Open arrow indicates input of resources and lines indicates interconnection between units within the chain. (Source: original diagram by Prithvi Simha.)

5.2 Dehydration (Papers II & III)

With hydrolysis of the urea prevented by high pH, the water in urine could be removed by dehydration. Dehydrating urine in the alkaline medium achieved a dry (5-7% moisture), powdery end-product while retaining 64 to 90% of the nitrogen (Table 4). The lower the operating temperature, the greater the nitrogen retention. The loading approach used for the urine to be alkalised, *i.e.* static loading (all dehydration medium added at the start of the experiment) or dose loading (medium added with each urine addition, urine:medium was 20:1), had little effect on the amount of nitrogen retained (2-8% difference) (Table 4).

During the dehydration process, water from the urine was removed by heat and mass transfer. Overall, the volume of urine was reduced by >90% in all alkaline urine dehydration treatments (Papers II & III). Figure 5 shows an example of the measured mass balance of the dehydration process. The initial concentration of nitrogen in the two ashes was <0.05%. The nitrogen loss shown in Figure 5 is included in the total volatile solids (VS) loss. The experiment was terminated when the pH of the media decreased to <10.5, at which point the medium is referred to as saturated.

Increasing the pH of the incoming urine (pH >10), using an anion exchanger, did not have a noticeable effect on increasing the capacity of the ash (the amount of urine dehydrated before the pH of the ash reached <10.5) or on the nitrogen retention.

The output of the process (Granurine) was a dry urine-based fertiliser. The urine/ash fertiliser had an NKP concentration of 7.8:2.5:10.9 (dry weight), with urine contributing 100% of the nitrogen, 28% of the phosphorus and 13% of the potassium. As expected, some nitrogen was lost due to the initial high pH of the media (>12.8) and the elevated temperature (35-65 °C) (Table 4). There was complete recovery of phosphorus and potassium within the media, since these elements are non-volatile at the temperatures studied.

Table 4. *Dehydration temperature applied to the different dehydration media, loading method and rate, final ratio of urine to dehydration medium, and percentage of nitrogen (N) retained*

Medium	Temp. (°C)	Loading method	Loading rate (L m^{-2} d^{-1})	Ratio[z]	N retained
Birch ash	35	Static	3.8	7.5	90%
Birch ash	35	Dose	3.8	20	82%
Birch ash[y]	40	Static	7.3	10	76%
Birch ash[y]	50	Static	12.2	16	74%
Birch ash	65	Static	5.1	16	64%
Birch ash	65	Dose	5.1	20	66%
Wood pellet ash	65	Static	5.1	16	64%
Wood pellet ash	65	Dose	5.1	20	66%
Biochar[y]	45	Static	6.4	11	72%

[z]Urine:medium, ww/dw.
[y]Urine was pre-treated by anion exchange.

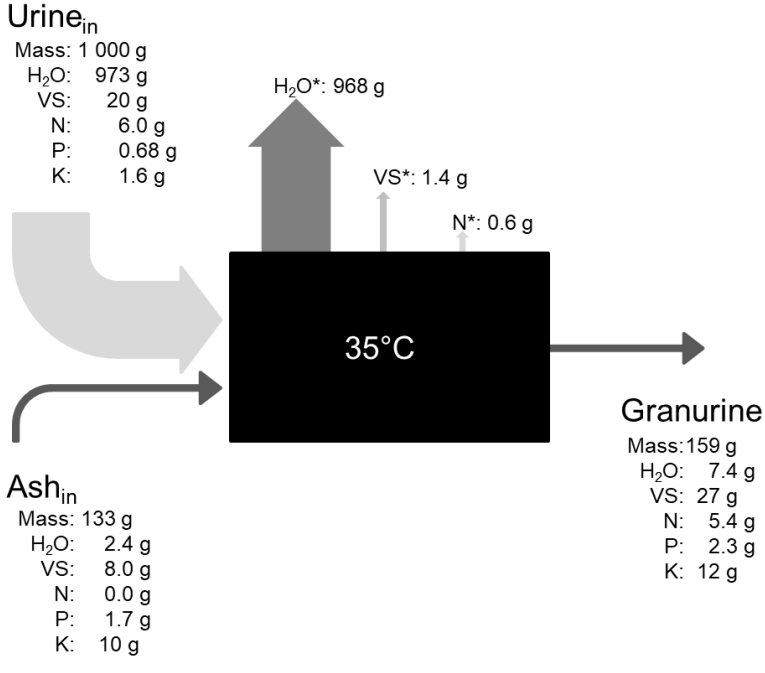

Figure 5. Measured mass balance for dehydration of 1 kg of urine in 133 g of ash at 35 °C in the static dehydration bed, where all dehydration medium was added at the beginning and urine was added at regular intervals. * indicates calculated values. Overall mass reduction was 86%, including the drying medium in the balance.

5.3 Hygiene evaluation (Papers IV, V & VI)

The Ascaris eggs persisted for a much longer time than the other organisms tested and showed a pattern of biphasic inactivation. The times for a 3 $\log_{10}$ reduction were calculated (Table 5) based on the derived lag phase and inactivation rate constant (k) (Papers IV, V and VI). In urine/lime medium, where the tubes were sealed, there was a faster reduction in egg viability than in the urine/ash which was open to the air. The pH 10.5 control showed slower inactivation, indicating that pH was not the only factor influencing the speed of Ascaris egg inactivation. The inactivation of bacteria and phages followed a log-linear trend and inactivation rate constants were obtained and the time required for a 6 $\log_{10}$ reduction was assessed (Paper V). In the urine/ash medium, the bacteria and phages at 20 °C showed a 6 $\log_{10}$ reduction within four days.

For Ascaris, a minimum of 100 eggs per sample were checked under the microscope directly at sampling (prior to incubation for viability analyses). In urine/ash stored open at 20 °C, pre-larval development was found to be occurring within the medium after 46 days, while after 74 days 7% of the eggs (9 out of 124) had developed to the larval stage. At the last observation (day 346), 9% of the eggs (97 out of 1090) had developed into larvae during storage, but had then died (visual defects included bubbles inside the larvae and no movement). At the last day of observation, only 1538 eggs were recovered, compared with 3000-5000 eggs recovered earlier during the experiment. Egg loss was also observed during the thermal treatment of urine/ash at 42 °C, where on day 14 (data not shown) only 426 eggs were found out of >2500 initially added, but six eggs had developed into larvae during incubation. In the thermal treatment at 42 °C, >1000 eggs were extracted from the buffer (pH = 7.2) after 14 days (data not shown) with no disintegration observed and no eggs were found to be viable after incubation. Egg recovery was >1000 eggs in the urine/lime medium, except in two of the dried precipitate fractions, from temperatures 20 and 35 °C, where 632 and 540 eggs were recovered, respectively, out of the approximately 10 000 eggs added.

Table 5. *Time required for a three-log$_{10}$ reduction in* Ascaris suum *eggs in three treatments. When no reduction in viability was observed (NR), duration of the viability study is given. V* indicates that a 6 log$_{10}$ reduction in* Salmonella *Typhimurium,* Enterococcus faecalis *and the bacteriophages MS2 and ϕx174 occurred within 4 days in the same conditions. For specific details of the lag phase and inactivation constant, see Papers IV-VI. All inactivation was studied in sealed containers except for urine/ash, which was exposed to ambient air. Initial moisture content (MC) in urine/ash was 43%, but decreased to 1% by day 102 at 20 °C.*

Treatment	Storage Temp.	Media	Fraction	Conditions	pH	3 $\log_{10}$ (days)	MC %	Paper
Drying (open) followed by storage (closed)	20 °C	Urine/lime	Supernate	Partially wet	12.5	111	78	VI
				Dried		53	13	
			Mixed	Partially wet		95	82	
				Dried		58	14	
			Precipitates	Partially wet		97	81	
				Dried		73	16	
	35 °C		Supernate	Partially wet		16	73	
				Dried		12	18	
			Mixed	Partially wet		23	78	
				Dried		18	24	
			Precipitates	Partially wet		59	77	
				Dried		79	33	
Storage	20 °C	Urine/lime	Supernate	Wet	12.5	98	97	VI
			Mixed			102		
			Precipitates			60		
		Urine/ash	-	Partially wet	> 10	325	1	V*
		Fresh Urine	-	Wet	8.5	90	97	V
		Buffer	-	Wet	7.2	NR, 94	> 90	IV/V
					10.5	NR, 126		
					12.5	NR, 126		IV
		Soil	-	Partially wet	7.2	NR, 98	60	IV
				Dried		NR, 94	< 20	
	35 °C	Urine/lime	Supernate	Wet	12.5	15	98	VI
			Mixed			48	97	
			Precipitates			53	92	
		Buffer	-	Wet	7.2	85	> 90	IV
					10.5	63		
					12.5	23		
		Soil	-	Partially wet	7.2	79	60	
				Dried		63	< 20	
Thermal Treatment	42 °C	Urine/lime	Supernate	Wet	12.5	4	97	VI
		Urine/ash	-	Partially wet	> 10	9	43	V
		Buffer	-	Wet	7.2	8	> 90	IV
					10.5			
					12.5			
	50 °C	Urine/lime	Supernate	Wet	12.5	<9 h	97	VI
		Buffer	-	Wet	7.2	<9 h	> 90	IV
					10.5			
					12.5			

6 Discussion

6.1 Alkaline urine treatment service chain

The pH of alkaline dehydration media will decrease over time and the cassette would need to be changed on a regular basis. In the current pilot system, a standardised box with a surface area of 0.24 m^2 (60 cm by 40 cm) and containing 6 kg of alkaline dehydration medium, with the capacity for treating 6-8 L of urine per day, is used as the cassette. This could serve a family of four for one month, producing approximately 14 kg of Granurine containing >7% N and >2% P per month (Figure 6). Once the dehydration medium is saturated (pH <10.5), the used cassette would be removed and replaced with a new one.

Connecting toilet users to a sanitation supply chain is crucial in order to provide a sustainable service (Verhagen & Carrasco, 2013). A supply chain helps formalise and regulate decentralised sanitation systems and ensures that toilets users are not left to manage their waste themselves. In the context of the alkaline dehydration technology, sanitation servicing is needed to supply users with fresh cassettes of alkaline substrate and to establish a collection service for the used cassettes.

6.1.1 Collection and transport

The used cassette could be managed in one of two ways. (1) It could be used on-site as a fertiliser or (2) transported to a conversion plant. In on-site use, the medium in the cassette could be used directly as a fertiliser, *e.g.* in home gardens, and new cassettes could be purchased online or at a local store. For transport of cassettes to a conversion plant, a collection service would need to be established. In a Swedish context, the collection could closely resemble the existing system for municipal solid waste. The quantity of saturated medium produced per person and year would be approximately 40 kg, which constitutes about 10% of

the current estimated urban solid waste production rate of 440 kg person^{-1} yr^{-1} (Hoornweg & Bhada-Tata, 2012), or 19% of the food waste collected in Sweden (EuroParl, 2017). Collection and transportation of wastes can be expensive, *e.g.* in municipal solid waste management it can account for as much as 66% of the annual operational costs in the West Bengal State in India (Ghose *et al*., 2006). However, compared with transporting liquid urine as fertiliser, Granurine is 12-fold lighter and has a comparable mass to synthetic fertiliser with similar NKP concentrations (Figure 6). Optimising collection times, delivery and pick-up routes for the cassettes could help minimise the fuel and labour costs. The collection and transportation of the cassettes could occur in the same way as municipal solid wastes.

There are a few established urine-diverting, container-based sanitation providers in low- to middle-income countries and all those established to date struggle with management of the collected urine. In several cases, the urine is not collected and it is up to the household to dispose of it (Sanivation, 2020; SOIL, 2019). The simplest option is to percolate the urine into the ground, which can lead to groundwater contamination (Xu & Usher, 2006). Having a dried product coming from the toilet system would enable better urine management practices, e.g. this could enable urine collection frequency to be reduced from weekly to monthly collection. By having a concentrated, solid product, storage of urine to be used as fertiliser is much simpler than storage of liquid urine, which requires six months of storage in a sealed container for hygiene treatment of urine (WHO, 2006) and 20- to 50-fold the storage volume compared with a solid product.

6.1.2 Conversion plant

Granurine can be used as-is (directly from the toilet) in areas where soil-transmitted helminths do not occur, or in home gardens (WHO, 2006) (see section 6.3). If not used at the household level, Granurine could be taken to a conversion plant (transported inside the cassette) for post-processing to transform it into a commercial product.

Granurine would be collected, mixed and potentially further dried. Pelleting Granurine would enable its distribution to soil by common granular fertiliser spreaders. Further research is needed on the final stages of the process, such as identifying binding material to be used during pelleting. Packaging the processed Granurine fertiliser product in a sealed bag would be ideal, as some hydrolysis of urea into ammonia can be expected during storage. Paper II showed that up to 13% of nitrogen could be hydrolysed during storage at 35 °C. The actual magnitude of the loss will depend on the storage conditions. Storing Granurine

in a sealed bag and in cool conditions will minimise nitrogen losses. However, the formation of ammonia would improve the hygienic quality of the material (see section 6.3.4).

When wood ash, at a ratio of 20:1 (urine:ash), was used in the alkaline urine dehydration process, the Granurine had a final NPK concentration of 7.4:2.3:10.3 (% ww) and 5% moisture content (Paper II), with 0% of N, 72% of P and 87% of K coming from the ash. The NPK value in fresh urine, 0.6:0.07:0.16, was concentrated to 7.4:0.8:2.0 (% ww). This compares well with another urine-based fertiliser that is commercially available, Aurine by Vuna, which contains 4.2:0.17:1.5 NPK (% ww) (Vuna, 2020). Since Paper III was published, further studies have been performed by the research team,with the aim of improving the nitrogen concentration in Granurine by manipulating the alkaline dehydration media. The nitrogen concentration has now reached >10% (unpublished data). The highest theoretical percentage of $N_{dry\text{-}weight}$ in urine-based fertiliser is 36.7% without any drying medium. This is based on the total solids (TS) content in the fresh urine (lower range 3% TS) and the initial nitrogen concentration (upper range 11 g N L^{-1}).

Depending on the end-user's needs, the dehydration medium can be altered to provide a tailored Granurine product with specific NPK concentrations. Different alkaline dehydration media will have different ratios of plant nutrients and can be manipulated based on plant requirements, such as addition of sulphur. The specific forms in which phosphorus and potassium are retained in Granurine were not investigated in this thesis. Phosphorus from the urine was likely to be present mainly in the form of precipitated metal phosphates such as struvite ($MgNH_4PO_4 \cdot 6H_2O$) and hydroxyapatite (HAP, $Ca_{10}(PO_4)_6(OH)_2$) due to the high pH (Udert *et al*., 2003) and to the added magnesium and calcium from the ash and biochar. However, it is not known whether the magnesium and calcium are readily bio-available, as they could have low solubility (Etiégni & Campbell, 1991). The potassium from the urine can be assumed to remain mainly in its original salt forms, such as KCl, K_2SO_4, $KHCO_3$ and K_3PO_4 (Putnam, 1971). By manipulating the medium, phosphorus and potassium can be steered towards the desired forms, *e.g*. by adding Mg to precipitate struvite or using an ion exchanger to remove chloride ions (Cl^-) (Paper III). Ion exchange was shown to be impractical, but is still a good example of how nutrient ratios in Granurine can be manipulated.

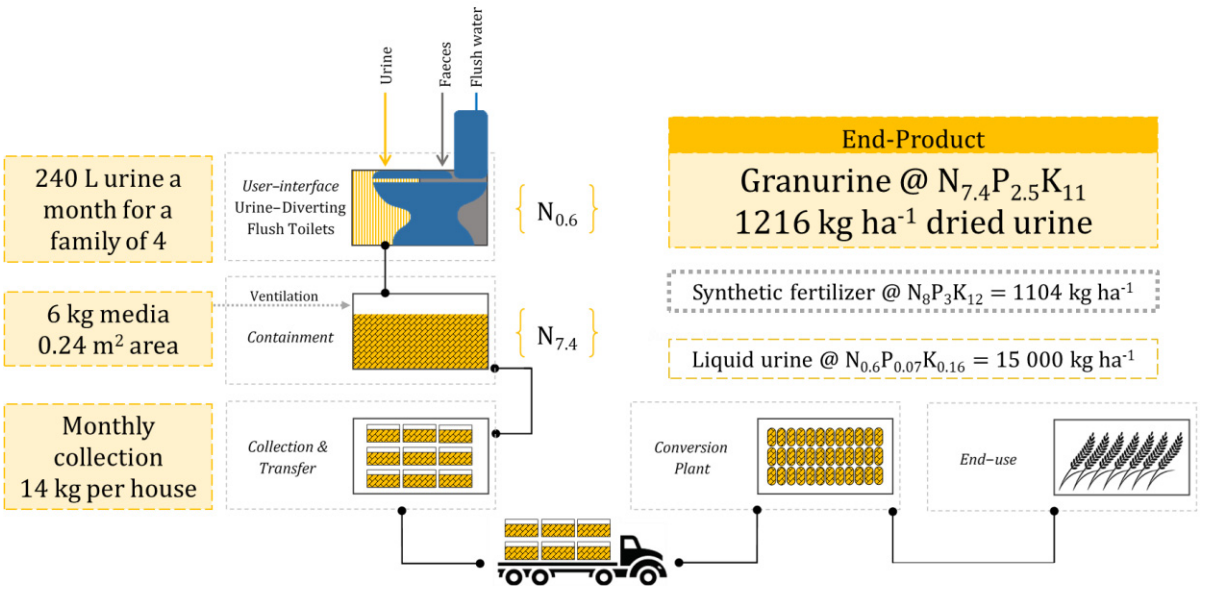

Figure 6. Schematic representation of a proposed alkaline urine treatment sanitation system using urine-diverting-flush toilets with the incoming mass of urine (240 L month^{-1}) reduced to 14 kg, including dehydration medium. The end-product is compared to synthetic fertiliser and liquid urine for a 90 kg N ha^{-1} application rate. (Source: original image by Prithvi Simha.)

6.2 Alkaline dehydration technological performance

6.2.1 Nitrogen retention

The initial pH (>12) of the dehydration media was high enough to prevent biochemical degradation of urea in the urine during dehydration. This supports findings by Geinzer (2017) that urease enzymes are inactivated at pH >10. Nitrogen losses occurred, although at lower rates than expected, during the dehydration process. Thermal and chemical degradation of urea should have been minimal at 35 °C. Thermal degradation at 38 °C is minimal (Table 1) and the pH decreased to <12 within the first 12 days of the experiment. Increasing the temperature from 38 to 65 °C will decrease the half-life of urea from 3.6 years to 15.3 days for pH <12, while increasing the pH to >12 at 65 °C will decrease the half-life of urea by one more day, to 14.1 days (Table 1). At 65 °C, thermal degradation will become a driving factor for nitrogen loss. The dehydration medium exposed to an elevated temperature (65 °C) for 42 days lost 35% of N, but a predicted loss of >50% was expected (Equation 4 in section 6.2.2 and Table 1). This difference can probably be explained by the average medium temperature being lower than the air temperature. The wet surface of the medium would be cooler than the flowing air due to evaporative cooling (wet-bulb temperature, energy adsorbed by the water to convert from liquid to gaseous state). The temperature of the wet medium could be as low as 37 °C during the dehydration process, depending on the saturation level on the surface (calculated using psychometric charts with incoming air at 65 °C and 30% relative humidity). In addition, every time urine was added the medium would be cooled and would need to heat up again, resulting in a lower average temperature than the incoming air temperature.

Nitrogen retention during dehydration (64-90%; Table 4) was similar to, or higher than, that in other urine treatments such as solar drying (68%; Antonini *et al*., 2012), struvite precipitation (3%; Udert *et al*., 2015), ammonia adsorption on zeolite and wollastonite (65-80%; Lind *et al*., 2000) and evaporation on gauze sheeting with high salt concentrations (85%; Pahore *et al*., 2011). The alkaline urine dehydration treatment system has no liquid to be disposed of and all the other plant nutrients, in addition to nitrogen, are retained. This is an advantage over other urine treatments, which produce a liquid effluent that needs further treatment before disposal as it still contains plant nutrients. Treatments include struvite precipitation (<2% volume reduction), ion exchange for nitrogen removal (0% volume reduction) and ammonia stripping (0% volume reduction) (Udert *et al*., 2015; Maurer *et al*., 2006).

Advantages of using high pH for inhibiting urease activity are the long-term effect on microbes and on soils. The remaining elevated pH would continue to inhibit contamination or regrowth of pathogens, including inhibition of urease-producing bacteria (Nyberg *et al*., 2011). In other treatments, such as oxidation, that have good initial inhibition, there is no protection against regrowth of urease-producing bacteria or pathogens post-oxidation, which leads to hydrolysis of urea and/or increased risk of disease transmission (Zhang *et al*., 2013). Acidification of soil in the humid northern temperate and humid tropic zones is a problem commonly alleviated by adding alkaline medium, such as lime or ash, to the soil (FAO, 1986).

6.2.2 Calculating urea loss

During the alkaline urine dehydration process, it was expected that some urea would be lost due to degradation influenced by the elevated pH and temperature. To estimate the theoretical amount of urea contained in the end-product, Equation 4 was derived:

$$U = \sum_{i=0}^{n-1} x_i \left(\frac{1}{2}\right)^{\frac{(t-t_i)}{t^{1/2}_{T,pH}}}$$

Equation 4

where U is total mass of urea retained, X_i is mass of urea added in addition i, t is total storage time since first addition, t_i is time at which urea addition i was performed (as time since first addition) and $t^{1/2}_{T,pH}$ is half-life of urea at the treatment temperature and pH of the medium (values given in Table 1). This equation is an improvement on the former version (Equation 3 in Paper II) since urea degradation is calculated based on the mass of urea remaining in the dehydration medium, whereas the equation in Paper II calculated urea as a loss.

6.2.3 Anion exchanger

The pH of the incoming urine in the dehydration trials at 40, 45 and 50 °C was increased by an anion exchanger to >10. The anion exchanger adsorbed anions from the urine (such as Cl^-) and, in exchange, released OH^-. The reasons for using the anion exchanger (resin) were twofold: (i) to increase the pH of the urine within the toilet, which would enable pipe transport with minimal risk of enzymatic hydrolysis of urea, and (ii) to increase the capacity of the dehydration bed. The resin functioned to increase the initial pH of urine (<7) to >10 (Paper III), which is high enough to keep the urease enzyme inactive (Geinzer, 2017). However, increasing the pH of the urine through anion exchange did not result

in higher pH in the dehydration medium over time and did not increase the urine:medium ratio (Table 4).

The factor influencing the capacity of the anion exchanger was the initial concentration of the anions in the urine. The concentration of anions in human urine can vary greatly on a daily basis, for example Cl^- has been measured to vary between 750 and 11 456 mg/L (Ferslew *et al*., 2001), making it difficult to predict the pH that the urine will reach after passing through the anion exchanger. The urine used in the preliminary anion-exchanger experiments had a lower and narrower range, 626 to 2224 mg Cl^- L^{-1} urine (data not published).

In practical application, using the anion exchanger (Amberlite resin) to increase the pH of urine was less effective than hoped. The mass of resin required in a toilet to stabilise the urea in urine before pipe transport is considerable: 150 to 200 mL for every litre of urine (urine:resine ratio ranged from 5 to 6.7:1, which was similar to the initial estimate of 5.4:1). The resin would need to be frequently changed or regenerated. One person can produce 0.8 to 3 L urine day^{-1}, meaning 5.6 to 17 L of resin would be needed per week for a family of four (calculated based on all toilet visits being at home). To the best of my knowledge, anion-exchange resins have not used previously used to alkalise human urine, but cation exchangers have been tested to capture the ammonium in hydrolysed urine (Tarpeh *et al*., 2017). In that study, the volume of resin needed for a family of four on a weekly basis was similar to that identified in this thesis, 5.3-19 L resin $week^{-1}$. In addition to the large mass of resin required, there were also challenges with precipitates forming as the pH of the urine increased. When using a column (Figure 7; data not shown), the flow-through rate decreased from 20 mL min^{-1} to <5 mL hr^{-1} after a urine:resin ratio of 6:1 was passed through the column, making it difficult to regenerated the resin in-situ.

Figure 7. Resin in a column with white precipitates from the urine on top.

With all the complications that arose when using the anion exchanger, *i.e.* high consumption of resin, blockages due to precipitation, no lasting alkalising effect in the medium and risk of bacterial growth in the resin, it was concluded that it would not be convenient to have an anion exchanger in a household or public toilet for preventing urea degradation in the

urine. Bed clogging and costly regeneration of the ion-exchange process are common problems that have limited implementation of such processes at WWTPs, however advancements are being made to address these problems (Huang et al., 2020).

6.2.4 CO_2 limiting pH stability

The theoretical ratio of urine to ash before pH dropped below 10.5, based on separately measured buffering capacity of urine and ash, was initially calculated to be 33:1 (data not shown). However, in the experiments (Papers II and III), the ratio ranged from 8 to 20:1 before the pH dropped to <10.5. The saturation of the ash bed (pH of the ash <10.5) was not limited by the mass of urine added or by the initial pH of the urine, but rather by the mass of carbon dioxide being absorbed from the air.

Carbon dioxide and moisture can spontaneously react with ash or biochar and form carbonates (CO_3^{2-}), bicarbonates (HCO_3^-) and calcium carbonate ($CaCO_3$) (Andersson & Nordberg, 2017; Etiégni & Campbell, 1991). When urine is added, more carbon dioxide can be absorbed as the urine acts as a solvent. The highly ventilated and wet surface of the ash enables good carbon dioxide absorption conditions (Mott, 2013). The pH decreases since, during the formation of HCO_3^- and CO_3^{2-}, OH^- is consumed and/or H^+ is released (Andersson & Nordberg, 2017; Smit *et al*., 2003). Wood ash can have a potential absorption capacity of 0.01 to 0.20 g CO_2 g^{-1} dry ash, depending on the physical and chemical properties of the ash and surrounding air (Andersson & Nordberg, 2017). The potassium present in the ash could also have a similar affect to the calcium (Zhao *et al*., 2014).

The pH decreased in the ash at the same rate regardless of whether urine or distilled water was added (see 'controls' in Figure 1 in Paper II). The similarity in pH decline highlights that potential sources of acids from urine, *e.g*. urea hydrolysis releasing carbonic acid or any oxidation of organic compounds in urine which would release acids (Putnam, 1971), did not have a measureable effect on the pH. In addition to the water control in the dehydration experiment, an open container of ash (30 g) was placed in the oven and not wetted. The pH of this dry control also decreased to <10.5 (data not shown) over time, but not as quickly as in the wetted ash.

6.2.5 Factors limiting evaporation

The evaporation rate of 782 L urine (no media added) was observed to decrease by 78% over 81 days in one previous study (Bethune, 2015). A decrease in the

evaporation rate over time is anticipated as water molecules are polar, which makes them attracted to the accumulating salt ions (thus elevating the boiling point) and more energy will be required to break the bond to evaporate the water (Harrison *et al*., 2015). Pahore *et al*. (2011) observed that the evaporation rate was not hindered by accumulating salts in synthetic urine, however salt concentration was increased 8-fold, while in this thesis the concentration was increased 8 to 20-fold. The hindrance may be thus still be from the salt. Another hindrance could be the visible film that forms over time on the surface of urine and medium. This layer is probably composed of amino acids (such as glycine) that are gradually released from the organic compounds (606 mg L^{-1}; Putnam, 1971) in the urine. The use of dehydration medium limited the time for which urine pooled on the surface (compared with not using medium) during dehydration, but a yellowish layer still formed on the surface of dehydration media, which could decrease the evaporation rate over time. The evaporation rate of distilled water was faster than that of urine (data not shown).

The specific surface area of ash ranges from 4200 to 100 600 m^2 kg^{-1} (Grau *et al*., 2015), which is >15 000 times greater than having no dehydration medium in a 0.24 m^2 box. However, as explained in section 3.2.3, the evaporation rate can be limited by the hydraulic capacity of the ash and the heat transfer to the lower layers. The capillary action of the porous media can keep the water bound, as was seen in the water/ash controls (data not shown) where the moisture content was 2% after 51 days of drying at 35 °C. The corresponding urine/ash samples had a higher moisture content (4.6-5.1%), probably due to the salts and organic matter binding the water.

6.2.6 Energy consumption

Convection was the driving force for heat and mass transfer of water vapour in the alkaline urine dehydration system described in this thesis. Increasing the temperature allows for more rapid removal of moisture from the surface, which decreases the dehydration time (Menzies & O'Callaghan, 1971). As the air temperature is increased, the water-holding capacity of the air increases exponentially, *e.g.* from 40 g kg^{-1} dry air at 35 °C to 200 g kg^{-1} dry air at 65 °C (calculated with psychometric charts). Increasing the temperature of the incoming air thus decreases the volume of air that needs to be exchanged to dehydrate a daily volume of 6 L of urine from approximately 400 m^3 d^{-1} to 60 m^3 d^{-1}.

The current alkaline dehydration process uses 1.2-3.2 kWh L^{-1} urine (based on laboratory and field trials, data not published). Warming the inlet air by solar energy would improve the dehydration rate while reducing primary energy use

(Bethune, 2015; Antonini *et al*., 2012). Alternatively, in a Swedish context, the exhaust air from the bathroom (already warm) could potentially be used, as it is often exchanged 4-8 times per hour.

The energy consumption per litre will decrease when the system is automated (see section 8.2 for details). The two following situations exemplify how automating the system could decrease the energy consumption. Stage 2 of the dehydration curve has the highest evaporation rate, so during this stage the airflow rate and temperature could be increased to maximise the heat and mass transfer rate. As the surface of the medium dries and reaches Stage 3, the air exchange rate is no longer a limiting factor and the airflow rate can be decreased or stopped to conserve energy (Strumiłło *et al*., 2014). The mass of liquid remaining in the medium at Stage 3 will depend on the hydraulic characteristics of the medium, *i.e.* a less porous medium will have less water remaining in the medium at Stage 3. While the cassette of the alkaline urine dehydration system is in use, there is no need to dry the medium beyond Stage 2.

A final dehydration step before the cassette is changed or at the conversion plant will be necessary and would occur during Stage 3. During Stage 3, the air exchange rate is no longer the limiting factor, but rather heat and mass diffusion through the media. Thus the temperature could be increased while decreasing the air flow. By controlling when and at which power the fans and heaters are operating, energy consumption per litre of urine could be decreased.

The current energy requirement of the alkaline urine dehydration technology to treat one litre of urine is more than double that of a conventional wastewater treatment plant (WWTP), including the energy required to produce the triple superphosphate and ammonium nitrate to replace the fertiliser gained from recycling the urine (Spångberg *et al*., 2014). However, these are very different technologies: WWTPs use mature technology (highly optimised) and even so, these advanced systems have large emission of nitrous oxides (1-2.4% of the total nitrogen load; Gruber *et al*., 2020), while alkaline urine dehydration is still in development.

A toilet currently on the market with similar handling to the alkaline urine treatment (in that it requires no liquid disposal and needs addition of some media) is the incineration toilet produced by Separett AB. The energy needed to treat one litre of urine and to manufacture the equivalent amount of fertiliser as gained from alkaline urine dehydration was estimated to be 2-8.5 kWh L^{-1} (based on the toilet utilising 0.4 to 1.7 kWh per flush (Separett AB) and energy requirements for fertiliser production based on Spångberg *et al*. (2014)).

These energy calculations are rough and only include the energy needed to treat the urine and not the energy required for manufacturing the systems and

media, or the transportation. Energy demand for any sanitation treatment technology is important, as societies should all be aiming to decrease consumption globally, but energy consumption should not be a prohibiting factor, at least not in the early development stage in which the alkaline dehydration system is at present. The additional benefits, such as effectively preventing nitrogen and phosphorus from entering surface waters and instead recycling them to productive land, as well as the flexibility of the system, also need to be taken into consideration when comparing the technologies. See section 6.4 for more potential advantages of implementing such a system.

6.3 Hygiene

6.3.1 Effect of diverting and concentrating urine

Diverting and collecting urine separately from faeces is a good way to retrieve a large fraction of the plant nutrients from excreta while excluding the majority of the pathogens. Excretion by mass of faeces (200 g d^{-1}) is less than one-tenth that of urine (1-3 L d^{-1}), so keeping the urine separate from faeces results in a 4.3 $\log_{10}$ lower pathogen concentration than when the urine is collected together with the faeces. This is based on an estimated 9.1 mg cross-contamination of faeces per litre of urine (Schönning *et al*., 2002). When concentrating the urine during dehydration, any pathogens present would also be concentrated unless they are inactivated during the dehydration process.

The inactivation of pathogens in Paper V mimicked faecal contamination of the cassette at the last moment before the cassette had to be exchanged for a new one. Thus Paper V did not represent what will happen to pathogens from faecal contamination during the alkaline urine dehydration process. The operating conditions in alkaline urine dehydration will be similar to those in the urine/lime treatment, where Ascaris egg inactivation was assessed during the dehydration process (Paper VI). Papers IV and V showed that alkali and drying had an effect on pathogen inactivation, and Paper VI indicated that ammonia may have also contributed to the inactivation of Ascaris eggs.

6.3.2 Bacteria and virus inactivation

After four days of storage of Granurine (the dry product), the model bacteria and viruses in the urine/ash were reduced by 6 $\log_{10}$. This reduction would meet the WHO (2006) and USEPA (1994) guidelines for safe reuse on non-processed food crops (<1000 *E. coli* or faecal coliforms per g TS) with great margins. The

results in Paper V confirm previous findings of rapid inactivation of indicator bacteria (Nyberg *et al*., 2011) and viruses (Decrey *et al*., 2016) in alkaline medium. As any ammonia formed was not accumulated in the open air urine/ash treatment, the main factors influencing the rapid inactivation of bacteria and viruses were potentially the pH (see the controls), the lowered moisture content (Ward & Ashley, 1977) and raising the osmotic pressure (Oishi *et al*., 2017).

6.3.3 Ascaris inactivation

Ascaris eggs were able to withstand the stresses from the alkaline treatment better than the bacteria and phages (Paper V). The results showed that exposure of Ascaris eggs to elevated pH (10.5-12.5) at temperatures ≤27.5 °C for >70 days had no effect on egg viability (Paper IV). The eggs used in this thesis were dormant (pre-embryo development), a stage at which they are most resilient as the eggshell is only permeable to organic solvents, lipid-permeable vapours (such as ammonia; Weiner & Hamm, 2007) and respiratory gases (Clarke & Perry, 1980; Barrett, 1976).

Ascaris eggs are able to withstand extreme conditions because they are well-protected by a four-layer shell. This protective shell enables Ascaris eggs to be (i) resilient, *i.e*. able to withstand harsh treatment conditions (Naidoo *et al*., 2016; Lalander *et al*., 2013; Papajova *et al*., 2008; Pecson *et al*., 2007; Gaasenbeek & Borgsteede, 1998; Eriksen *et al*., 1995) and (ii) persistent, *i.e*. remain viable in soil for several years, even in anhydrous and anaerobic environments (Clarke & Perry, 1988; Feachem *et al*., 1983; Pawlowski, 1982; Wharton, 1980). However, as the temperature increases, the ability of the egg to restrict water loss through the shell decreases exponentially (Wharton, 1979). Compounding effects of alkaline pH (≥10.5) or low moisture content (<20%) were observed at 35 °C, with pH having more of an effect than low moisture content (Paper IV). There was higher survival at elevated pH (10.5) with low moisture content (43% MC initially) conditions in the urine/ash treatment compared to the control with higher moisture content (pH 7.2; >90% MC) at 42 °C (Paper V). This difference could be due to the thermal conductivity of the buffer (liquid) being a magnitude higher than that of air (Çengel, 2008). The time for a 3 $\log_{10}$ reduction in viable eggs at 42 °C was faster compared to other studies (Koné et al., 2007; Pecson et al., 2007). This difference could be due to the small size of the samples in this thesis which enabled faster and thorough temperature increase compared to larger-scale treatments.

While alkaline pH alone did not inactivate the eggs at temperatures ≤27.5 °C, it may enhance the effect of ammonia, which is likely to be present in organic

wastes. For organic waste streams treated with lime ($Ca(OH)_2$), the inactivation effect on Ascaris eggs may mainly derive from ammonia present in the material, formation of which increases with alkaline pH (Pecson *et al*., 2007; Pecson & Nelson, 2005; Gaasenbeek & Borgsteede, 1998), rather than because of the pH reached (Ghiglietti *et al*., 1997). Ammonia is effective at inactivating Ascaris eggs because gaseous ammonia is uncharged, in contrast to the hydroxide ion (OH^-), and is a smaller molecule than oxygen gas (O_2). The uncharged molecule enables ammonia to diffuse passively through the Ascaris eggshell. The shell aids in restricting water loss while still allowing adequate oxygen gas diffusion (Wharton, 1980; Hinton, 1969). As a consequence of this, the eggshell is not only permeable to water vapour (kinetic diameter 265 pm), but also to ammonia (kinetic diameter 260 pm), which is a smaller molecule than O_2 (kinetic diameter 346 pm) (Breck, 1974; Hinton, 1969).

In the urine/ash treatments, the ammonia would be have been minimal as the containers were open to the atmosphere (Paper V), while the urine/lime treatments were sealed (Paper VI). The initial total ammonia nitrogen (TAN) concentration in the urine/lime was believed to be negligible (<10 mM) when setting up the experimental designs (Paper VI). Such low levels were believed to have no effect on Ascaris during the experiment, as ammonia concentrations around 20 mM NH_3 are not sufficient to give inactivation at 24 °C (Nordin *et al*., 2009). Fidjeland *et al.* (2016) observed no inactivation of Ascaris at 28 °C with 12 mM NH_3 after 48 days, while with a 39 mM NH_3 concentration a 4 $\log_{10}$ reduction occurred within 45 days. The TAN concentrations in lime-treated urine kept in closed containers have been shown to be stable over 27 days at 25 °C (Randall *et al*., 2016). However, the accumulated ammonia may still have had an effect on the inactivation times in Paper VI (Table 5). Ascaris eggs in the urine/ash (open) required 325 days at 20 °C for a 3 $\log_{10}$ reduction in viable eggs, compared with 111 days (for the slowest inactivation) in urine/lime (sealed container). Even if NH_3 concentrations were low, ammonia were present in combination with a pH much higher compared to pH 8.8 to 10 in other studies (Fidjeland *et al*., 2016; Nordin *et al*., 2009). Effects on Ascaris eggs may start at lower concentrations at the alkaline pH reached during urine dehydration.

The Ascaris inactivation studies could have been strengthened with controls studying the drying media without urine, to assess whether the medium itself had an effect. Formation and accumulation of ammonia could have been monitored in the different fractions and at different moisture conditions to gain a better understanding of the results (Paper VI).

6.3.4 Safe nutrient recycling

During the dehydration process itself, there would be no hygiene risk to the user. In handling the cassette, such as when exchanging the old cassette for a new one, there is a risk of exposure. However, the risk may be comparable to that posed by cleaning a toilet and is probably lower than when cleaning a child's bottom – yet to be assessed – and the risk could be minimized with a when designing the cassette. By collecting and treating the urine separately from the faeces, a post-treatment of the dried alkaline urine could be simply storage for four days at 20 °C in geographical areas where soil-transmitted helminths are not prevalent (Figure 8). For areas where soil-transmitted helminths are prevalent, the Ascaris eggs would accumulate as a result of faecal contamination since the results indicate that inactivation is slow during the dehydration process. Granurine could thus pose a risk to transmit helminths in areas where soil-transmitted helminths are endemic, such as to farmers using Granurine as a fertilizer and/or to people consuming food grown with Granuine (Haas et al., 2014).

In areas where Ascaris is endemic, post-treatment of Granurine at a conversion plant is recommended to ensure safe use by decreasing the concentration to <1 egg per 1 g Granurine, in order to meet the WHO (2006) and USEPA (1994) guidelines for unrestricted fertiliser use (crops consumed raw). Two potential post-treatments are thermal treatment and/or storage. A final thermal treatment, potentially during the final drying of Granurine, could be used to ensure inactivation of soil-transmitted helminths. A thermal treatment would be more effective than decreasing the moisture content (Paper IV). For a 3 $\log_{10}$ reduction in viable eggs 9 days at 42 °C was required as a thermal treatment.

Storing the dried urine in a sealed bag would retain any ammonia formed, which could result in further inactivation of microbial contaminants, especially *Ascaris* spp. Tests on control samples demonstrated that neither high pH (12.5) nor dryness had an effect on the inactivation rate of Ascaris at ≤27 °C for >70 days. Based on the measured inactivation times, a minimum storage time of 111 days at 20 °C and 79 days at 35 °C in sealed containers could lead to a 3 $\log_{10}$ reduction in any Ascaris present, ensuring a hygiene safety level meeting the international guidelines for unrestricted fertiliser use. Storage of Granurine is an ideal option, as storage would happen regardless if a post-treatment is required or not as fertilizer is only applied one or twice a growing season.

Ascaris eggs withstood the alkaline environment better than the bacteria and phage model organisms assessed in this thesis (Paper V). If Ascaris eggs are inactivated by alkaline treatment, then other relevant pathogens, such as *Salmonella* spp. (Fidjeland *et al*., 2016), are also inactivated. The suggested post-treatment durations will need to be confirmed at a larger-scale.

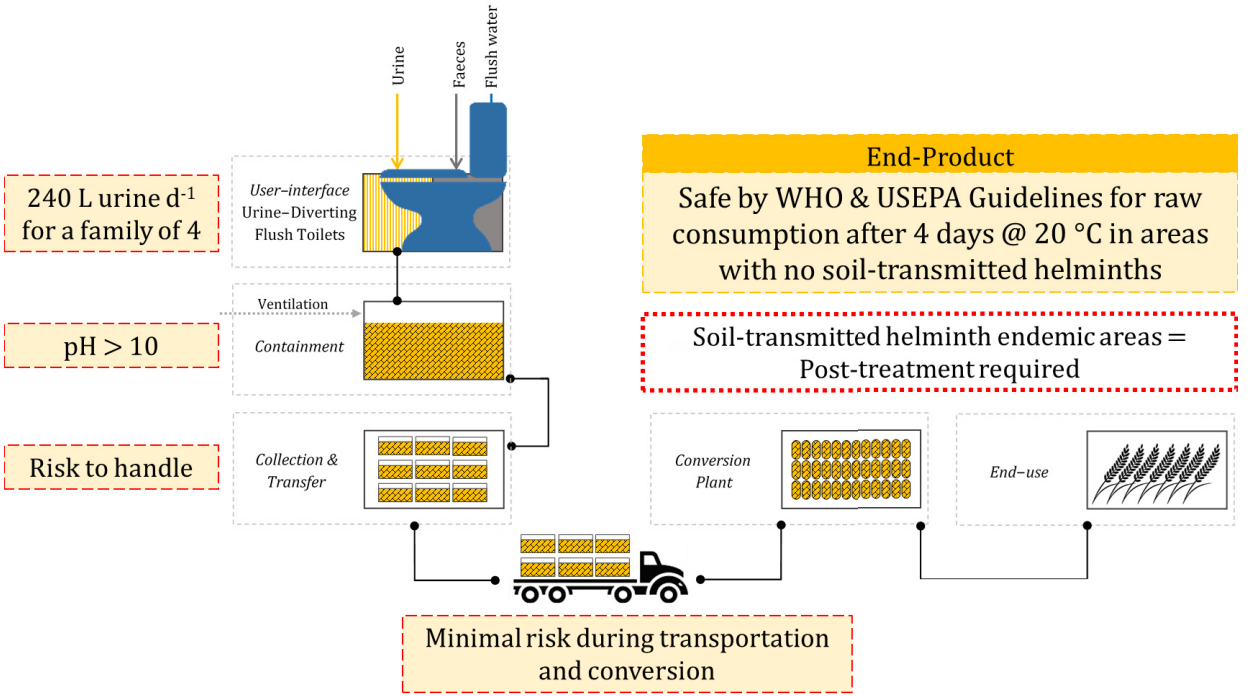

Figure 8. Schematic representation of a proposed alkaline urine treatment sanitation system using urine-diverting-flush toilets, showing the risks associated with potential cross-contamination from faeces. After four days of storage of Granurine (the dry product) at 20 °C, the model bacteria and viruses were reduced by 6 $\log_{10}$, while helminth eggs were not. (Source: original image by Prithvi Simha.)

6.4 Alkaline urine dehydration – considerations

6.4.1 Advantages of urine-based fertilisers

Only 7% of the world's wastewater receives tertiary treatment (such as advanced treatment to remove nitrogen) at a WWTP, meaning that at least 93% is released still containing nitrogen and phosphorous, which is potentially causing eutrophication, hypoxia and climate change (Paper I). Diversion of the plant nutrients from the wastewater stream back to agricultural fields can potentially reduce the flows of reactive nitrogen to the environment, thus limiting acidification, eutrophication, global warming and destruction of stratospheric ozone (Sutton *et al*., 2013).

A decentralised sanitation system that collects and dehydrates urine will influence household wastewater composition. With urine-diverting toilets, potentially 80% of the plant nutrients would be kept out of the wastewater. This could improve on-site wastewater-treatment systems, as they are not always reliable to sufficiently remove nitrogen and phosphorus (Heinonen-Tanski & Matikka, 2017). WWTPs' N_2O emissions could be minimized by keeping urine out of the wastewater (Gruber *et al*., 2020). The capacity of a WWTP to treat raw and settled wastewater could be also increased by 20% and 60%, respectively (Wilsenach & van Loosdrecht, 2004). By diverting all excreta back to agricultural fields, the greenhouse gas emissions from the production of commercial fertilisers used in Sweden could be reduced by 20% (Jönsson, 2011). As more stick effluent qualities are implemented, an increase in CO_2 emissions are expected due to increase in energy consumption and emissions from additional processes from installing/upgrading wastewater treatment plants to meet the quality standards (Bajón Fernández *et al*., 2017). Urine-diverting collection and treatment systems could be more efficient than tertiary WWTPs due to the savings in resources (Wilsenach & Loosdrecht, 2006) and potential reduction in emissions.

The collected urine contains the same nutrients as the plants (and animals) extracted when growing and the amount of plant nutrients in human excreta essentially equals that consumed (Jönsson *et al*., 2004). The ratio of plant nutrients can be modified to meet users' needs during pelleting of the end-product (section 6.1.2). By pelleting the dried urine, the urine-based fertiliser can be applied to soil with the same machinery that farmers use for mineral and synthetic fertilisers, including precision nutrient application. Urine-based fertiliser is a renewable resource, which could minimize CO_2 emissions both from fertiliser production and from wastewater treatment.

6.4.2 Heavy metals

Jönsson (2011) suggests that the fertiliser product produced from human urine can contain less heavy metals than mineral fertilisers. Heavy metal concentrations found in human urine are far below the Swedish limit for land application (Jönsson *et al*., 1997) and well below the levels in other biologically derived fertilisers (Hammer & Clemens, 2007; Jönsson *et al*., 2004). In this thesis work, the easiest-to-access alkaline medium to use in urine dehydration was wood ash, a common by-product in Sweden, but there is concern about the presence of heavy metals in the ash. Initial analyses showed that Granurine had heavy metal levels below the European Union limits for fertiliser use (data not published). However, the levels of heavy metals in wood ash vary considerably (Reimann *et al*., 2008). Depending on the source of calcium hydroxide (also referred to as hydrated lime), there may be issues of contaminating the soils with cadmium (Cd) (Spångberg, 2014). Using a cleaner medium to begin with would then require less monitoring of the heavy metals. The resulting alkaline Granurine product could potentially add less cadmium (Cd) to the soil than the commercial lime-containing mineral fertilisers applied in Sweden. An alternative industrial by-product for use as an alkaline medium that is available globally and which has a composition beneficial for arable soil has yet to be identified.

6.4.3 Pharmaceutical residues

The health risk from pharmaceutical residues is frequently discussed when considering urine-based fertilisers, but are already a problem with lack of adequate sanitation and with conventional WWTPs. Pharmaceutical residues can occur in very small quantities in wastewater and they are difficult to detect and remove at WWTPs (Luo *et al*., 2014), which are usually designed to treat bulk substances (Larsen *et al*., 2004). After passing through the WWTP, pharmaceutical residues remain biochemically active in water and can alter the behaviour of aquatic animals (Van Donk *et al*., 2016; Brodin *et al*., 2013).

Active ingredients in pharmaceutical are excreted *via* the urine. Lienert *et al*. (2007) analysed the excretion pathways of 212 pharmaceutical active ingredients (encompassing 1409 products) and found that, on average, approximately 64% (±27%) of each active ingredient was excreted *via* the urine. However, they also observed extreme variability, except for X-ray contrast medium, of which 94% (±4%) was excreted *via* urine (Lienert *et al*., 2007). Pharmaceutical residues and hormones have been detected in the drinking water of many cities (Mompelat *et al*., 2009). Levén *et al.* (2016) found in a simulation study that intake of pharmaceutical residues would be at least 100-fold higher

through drinking tap water taken from a reservoir (for Stockholm) containing approximately 5% treated wastewater effluent than through eating carrots fertilised with treated blackwater. Contrary to popular belief, human urine contains lower concentration of hormones and antibiotics than animal manure, which is widely used as a fertiliser in agriculture (Hammer & Clemens, 2007).

Removing the urine from the wastewater stream and utilising it as fertiliser could have three potential benefits. First, some degradation could occur in the field due to exposure to ultraviolet light from the sun (Kim *et al*., 2009). Second, microbiological activity in the soil will probably lead to degradation of pharmaceutical active ingredients (Salvia *et al*., 2014; Yu *et al.*, 2013). Many hormonal pharmaceuticals are similar to naturally occurring substances for which degradation routes exist in nature (Jönsson *et al*., 2004). Third, the collection process for Granurine can be digitised, where a household utilising specifically hazardous compounds (such as X-ray medium) could have a special (anonymised) barcode on their cassettes so that these could be handled separately at the conversion plant. Utilising such a digitised urine treatment system in hospitals and care homes could help divert sources of micropollutants away from the wastewater treatment plants (Michael et al., 2013). With alkaline urine dehydration treatment, there is potential to better manage the hotspot sources of micropollutants.

One can question the risk from pharmaceutical active ingredients and residues potentially entering the food system compared with the risk of consuming pesticides. Pharmaceuticals are approved for human consumption, so the risks of bioaccumulation and chronic exposure have been assessed (FASS, 2012). Pesticides, which are often detected in food (He *et al*., 2015; NSF *et al*., 2015), are less regulated and pose risks to human health (Li, 2018). The risk of potentially having some pharmaceutical residues present in fertiliser may be less than the risk of consuming food treated with pesticides.

7 Conclusions

- *Urine in a circular economy*: Diversion of plant nutrients from the wastewater stream back to agricultural fields can potentially reduce the flow of reactive nitrogen to the environment, thus limiting acidification, eutrophication, climate change and destruction of stratospheric ozone. To enable large-scale on-site nutrient recycling from urine, a service chain is needed to support the on-site treatment technology, especially in an urban context where urine-based fertiliser might not be used directly.

- *Technological performance*: Urine dehydrated in alkaline medium produced a dried end-product, nitrogen retention ranged from 64 to 90% and there was complete recovery of phosphorus and potassium. The liquid mass was reduced by 95%. Including the medium, the mass reduction ranged from 75 to 90%, depending on the ratio of dehydration medium used. The concentration (w/w %) of NPK in urine increased from 0.6:0.07:0.16 upto 7.4:0.8:2.0.

- *Technical limitations*: Maintaining the alkalinity is necessary to ensure effective retention of nitrogen during the evaporation process. The decrease in pH is due to absorption of carbon dioxide, which is the limiting factor for the dehydration medium. As the dehydration process becomes more efficient, the salt and organics concentration may become the limiting factor.

- *Hygiene performance*: In terms of existing guidelines for excreta reuse on non-processed food crops, the alkaline dehydration system produces a safe, dry fertiliser within four days of storage at 20 °C, except in areas with soil-transmitted helminths (Ascaris). At temperatures ≤27.5 °C, elevated pH (10.5-12.5) has no effect in decreasing Ascaris egg viability within 70 days. In areas prone to soil-transmitted helminths, post-treatment of the urine-based fertiliser is recommended before use.

8 Future research & development

8.1 Future research

- *Pipe transport*: Pipe transport would increase the flexibility of the system, as urine dehydration could then occur in a basement or outside the building with minimal nitrogen loss. Initial ideas are to use chemical dosing or an ion-exchange system (both of which would still require in-house maintenance). Preventing biofilm growth by using pipes coated with antimicrobial coatings or a super-hydrophobic surface may be an option in the future. The medical field is developing such surfaces to prevent urinary tract infections in patients with catheters (Al-Qahtani *et al*., 2019; Santiago-Rodriguez *et al*., 2015)

- *Using the water vapour*: Heat exchangers could be used to extract heat from the moistened warm air exiting the dehydration system and recirculate it back into the system. Condensed liquid could be used as flush water or hand-washing water. Initial trials indicate that the condensate would need to be treated before use, as it has an odour and contains some ammonia. Due to the ammonia, a filter on the outgoing air may be required.

- *Fertiliser potential*: Preliminary crop trials have indicated promising results and positive comments from those spreading the fertiliser (easier to use and less odorous than liquid-based urine fertilisers). The next step is to identify techniques for pelleting the Granurine and assessing the fate of pharmaceutical residues.

- *Field testing*: In coming years, field testing will be prioritised. The performance of the system in different geographic areas needs to be evaluated, specifically in terms of increasing nitrogen concentration (aiming for >15% N in the end-product), finding alternative alkaline media, evaporation efficiency and confirming pathogen inactivation.

- *Standards*: A daily urine loading cycle that can be used for comparison of different urine treatment technology performances needs to be defined. Detailed studies on the composition of urine in different geographical regions could help to determine how the composition of urine-based fertilisers might vary geographically. Certification protocol ISO 30500:2018 for non-sewered sanitation systems has a test regime that could be applicable when scaling up the alkaline urine dehydration treatment system.

8.2 Digitisation

With the current pilot systems, there is no way of knowing how much urine has entered the system or if it is about to reach capacity and overflow. Such aspects can be monitored manually, which is time-consuming and associated with recording errors and overflow of the system. Digitising the system with sensors that can monitor urine level, carbon dioxide level, weight, temperature, electric conductivity and/or relative humidity, and relay the data, would provide good knowledge of the state of the system. Ventilation and temperature could be altered remotely. These parameters could eventually be used to regulate the drying process in a fully automated system. Automation would save energy, as the system settings would adjust to the current urine load. A message could be sent to a private phone when the dehydration cassette needs to be changed or to a service provider who could optimise delivery and collection schedules for door-to-door service, especially for businesses using the dehydration system. However, there are potential risks associated with digitising appliances, especially regarding privacy, network security, and regulation and use of collected data (Nique & Smertnik, 2015; Perera *et al*., 2014; Gubbi *et al*., 2013). Our research group is currently assessing how to move forward with this.

8.3 Large-scale implementation

As society is becoming more aware of its direct impacts on the environment, there is growing demand for source-separating technologies and municipalities, such as Malmö, Stockholm, Helsingborg and Gotland in Sweden are investing in such technologies. However, wastewater professionals are hesitant to promote and adopt source-separating technologies, as these are still considered immature and risky (McConville *et al*., 2017). The current extensive sewer networks connecting households to municipal sewage treatment plants have been researched and developed over decades with heavy investment, while source-separating technologies have received only limited attention and resources (Larsen *et al*., 2009). However, this is improving due to the growing concern

about future fertiliser availability (Harder *et al*., 2019). The two most mature nutrient-recovery technologies are struvite crystallisation from anaerobic digester supernatant and incineration of sewage sludge followed by phosphorus retrieval from the ash (Harder *et al*., 2019), neither of which recirculate all of the plant nutrients found in excreta.

Implementing source-separating technologies on a large scale, such as an alkaline urine dehydration system that retains potentially >90% of the macro- and micro-nutrients found in urine, has true potential to shift society towards a circular economy. Large-scale implementation of alkaline urine treatment technology could replace 19% of the nitrogen and 21% of the phosphorus used each year in commercial fertilisers in Sweden (Jönsson, 2011). For this to happen, there are several barriers that need to be overcome, not just the maturation of the technologies, but also establishing guidelines, norms and standards for reuse, as many stakeholders see a grey area of responsibility for these source-separated waste fractions (McConville *et al*., 2017). To enable implementation, technologies need to move beyond the laboratory. Spin-off companies from universities that are commercialising source-separating technologies, such as Sanitation360 from SLU and Vuna from EAWAG, will play an important role in this shift and in helping wastewater professionals change their perception.

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Popular science summary

Only 7% of the world's wastewater receives advanced wastewater treatment, and the release of wastewater still containing nitrogen and phosphorous is resulting in nutrient overload and dead zones in aquatic ecosystems and contributing to climate change. The global negligence regarding human excreta management is odd, as they contain the same plant nutrients as the fertilisers used to grow the food we consume. We readily use animal manure, so why not human excreta?

Urine, rather than faeces, contains the majority of the plant nutrients excreted. The main limitation with using urine as a fertiliser is that it is mostly water (97%), meaning that its concentration of plant nutrients is low. The lower nutrient concentrations require larger quantities of urine to be applied per hectare as fertiliser, which creates logistical problems in terms of storage and increases the costs of transportation and application. Hence the objective of this thesis was to help develop a system to concentrate the plant nutrients in urine and produce a dry fertiliser.

In the work, a two-step process was developed to convert liquid urine into a dry fertiliser. Step 1 was to stabilise the urine to minimise nutrient losses, which was done by increasing the pH of the urine to above 10 using wood ash or calcium hydroxide. Step 2 was to remove the water in the urine, which was done using convective air-drying. The concept involves installing the system directly inside the toilet cubicle and connecting it to a urine-diverting toilet, where 80% of the plant nutrients are kept out of the wastewater. The advantage with the proposed system is that the liquid management of urine is solved and there is no need to change the piping in the building to install the system. The truly innovative feature is the final product, a dry powder with equivalent plant nutrient concentrations to commercial fertilisers. By converting the urine into dry-based fertiliser, the flows of polluting plant nutrients to the environment can be minimised, which in turn will help to limit nutrient overload and dead zones in aquatic ecosystem and, potentially, lower the contribution to climate change.

Populärvetenskaplig sammanfattning

Endast 7% av världens avloppsvatten genomgår avancerad rening för avloppsvatten och frisläppandet av obehandlade mänskliga utsöndringar resulterar i överbelastning av näringsämnen, döda zoner i akvatiska ekosystem och bidrar till klimatförändringar. Den globala vårdslösheten när det gäller hantering av mänskliga utsöndringar är udda, eftersom de innehåller samma växtnäringsämnen som gödselmedel som används för att odla maten vi konsumerar. Vi använder gärna djurgödsel, så varför inte mänskliga utsöndringar?

Urin, snarare än avföring, innehåller huvuddelen av de växtnäringsämnen som utsöndras. Den huvudsakliga begränsningen med att använda urin som gödningsmedel är att den mestadels innehåller vatten (97%), vilket innebär att dess koncentration av växtnäringsämnen är låg. De lägre näringskoncentrationerna kräver att större mängder urin appliceras per hektar som gödselmedel, vilket skapar logistiska problem vad gäller lagring och ökar kostnaderna för transport och applicering. Därför var syftet med denna avhandling att hjälpa till att utveckla ett system för att koncentrera växtnäringsämnena i urin och producera ett torrt gödningsmedel.

I arbetet utvecklades en tvåstegsprocess för att omvandla flytande urin till ett torrt gödningsmedel. Steg 1 var att stabilisera urinen för att minimera näringsförluster, vilket gjordes genom att höja pH i urinen till över 10 med träaska eller kalciumhydroxid. Steg 2 var att ta bort vattnet i urinen, vilket gjordes med konvektiv lufttorkning. Konceptet handlar om att installera systemet direkt i toalettfacket och ansluta det till en urinsorterande toalett, där 80% av växtnäringsämnena separeras från avloppsvattnet. Fördelen med det föreslagna systemet är att vätskehanteringen av urin löses och det inte finns något behov av att ändra rörledningarna i byggnaden för att installera systemet. Den verkligt utmärkande egenskapen är slutprodukten, ett torrt pulver med motsvarande koncentrationer av växtnäringsämnen som kommersiella gödningsmedel. Genom att omvandla urinen till torrbaserat gödselmedel kan flödet av förorenande växtnäringsämnen till miljön minimeras, vilket i sin tur kommer att bidra till att begränsa överbelastningen av näringsämnen, döda zoner i vattenlevande ekosystem och eventuellt sänka bidraget till klimatförändringarna.

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Human urine can be used as a fertiliser as it contains the same plant nutrients as the fertilisers used to produce the food that we eat. The limitation of human urine as a fertiliser is the low nutrient concentration (0.6% N) compared with commercial fertilisers (36% N in ammonium-nitrate). The innovative feature of this study was the final product: a dry powder fertilizer with >7% N that was safe for unrestricted fertiliser use after just four days of storage.

Jenna Senecal received her graduate education from the Department of Energy and Technology at the Swedish University of Agricultural Sciences (SLU). She previously received her Master of Science degree from the Department of Bioresource Engineering at McGill University.

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