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Swedish University of Agricultural Sciences

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Fertiliser derived from human urine: Novel media for alkaline urine dehydration

– Humanurinbaserad gödsel: Nya innovativa bäddmaterial för alkalisk urintorkning

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Abstract

Every year, each human excretes approximately 4 kg of nitrogen, 1 kg of potassium and 0.3 kg of phosphorus through their urine. In conventional wastewater treatment, these nutrients are usually not recovered for agricultural production, although they can enhance plant growth. Dehydration technologies decrease the volume of source-separated urine and increase its nutrient density, facilitating the use of the dried product as fertiliser. This study investigated the suitability of various alkaline dehydration media for urine dehydration. Magnesium oxide and calcium hydroxide were used as alkalising agents, and biochar and wheat bran acted as co-substrates. Pure magnesium oxide and mixtures between magnesium oxide, biochar, wheat bran and calcium hydroxide were subjected to an average dehydration temperature of 48.3 °C (SD 2.8 °C) during the dehydration process. In this dehydration setup, all dehydration media reduced the mass of urine by > 90 % and dehydration rates of > 19 kg m^{-2} d^{-1} were observed. Magnesium oxide showed an N-recovery of 66.8 % (±1.2), while the other four dehydration media showed an N-recovery rate of > 74.5 %. A large amount of the ammonia that could not be recovered was plausibly lost due to ammonia stripping, as > 30 % of the urea in the urine used for this experiment was already hydrolysed. These promising dehydration and N-recovery rates contribute to the research on new on-site dehydration systems that sustainably produce fertiliser out of human urine.

Keywords: Nutrient recovery, Fertiliser, Urine drying, Source separation, Magnesium oxide, Calcium hydroxide

Popular Scientific Summary

If human urine is dried in alkaline dehydration media at about 50 °C, plant nutrients that otherwise would get lost can be retained.

Urine contains urea, a substance rich in nitrogen. Nitrogen is the main component of air and crucial for the growth, health, and well-being of plants, as it is an essential element of protein. Urea is rapidly broken down when it gets into contact with an enzyme called urease. Bacteria produce enzymes and use them as their tools to transform one substance into another to access nutrients. Urease is very abundant in nature, and when the enzyme urease gets a hold of urea, it turns it into another nitrogen-rich substance called ammonium. Ammonium can become gaseous, and if that happens, it can get lost to the air and cannot be accessed by most of the plants anymore. In urine-collecting sanitation systems, the urease enzyme, therefore, needs to be deactivated to avoid losing nitrogen to the atmosphere. The urine can be made acidic or alkaline to deactivate urease. If the pH or the temperature is too high or too low, urea gets destroyed, and the plant-available nitrogen gets lost, even though urease might not be active anymore. Therefore, our research group tried to find a combination of temperature, pH and material, which works best to inactivate the urease and conserve the urea of the urine. In this study, we used magnesium oxide and calcium hydroxide (also called “slacked lime”), to make the urine alkaline. We mixed these substances with wheat bran and biochar because they help with the drying of urine and wheat bran already contains some nitrogen. We also raised the air temperature to about 50 °C because previous studies indicated that this temperature could provide accelerated drying, while still keeping thermal urea degradation at a minimum. According to our results, less than 1 m² of material would be needed to dehydrate the urine of four people, if each person excretes 1.5 L urine per day. This means that a family with four family members could easily fit the dehydration unit into their existing bathroom. The family would require about 15 kg of magnesium oxide for urine dehydration per year, which equates to approximately 5 USD annual costs. As a minimum, 150 L of urine can be dehydrated per kg of magnesium oxide. A dehydration unit of 1 m² and a depth of 1 cm for a family of four, needs to be changed about 5 times a year, so about every 2.5 months. The dried fertiliser could be collected, and the new dehydration medium could be provided via municipal solid waste collection, minimising the administrative and logistical burden.

Using alkaline dehydration, we recovered more than 67 % of the nitrogen in the urine. The other essential nutrients plants needed in larger quantities (so-called “macronutrients”), like phosphorus and potassium, are also retained in this dry fertiliser because drying at this temperature does not affect them, so they stay in the substrate.

On a personal, societal and global level, this means that with urine separation and subsequent dehydration with one of the dehydration media that we developed, we can:

1. save resources and money by cutting down on the use and production of artificial fertiliser
2. reduce the pollution of water bodies and the need for wastewater treatment because the input of nutrients into the water streams is minimised
3. reduce the costs for the transport of urine because of the volume reduction
4. produce a dry fertiliser that farmers might be willing to use as it is easy to handle with already existing infrastructure and equipment

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Abbreviations

In alphabetical order; conversion factor to SI base units in parentheses

A	Ampere
ANOVA	Analysis of Variance
Bran	Wheat Bran (aleurone and pericarp of wheat)
°C	Degree Celsius (0 °C = 273.15 K)
$Ca(OH)_2$	Calcium hydroxide (IUPAC name) (see also “Lime”)
$CaCO_3$	Calcium carbonate (IUPAC name)
Char	Biochar
cm	Centimetre (1×10^{-2} m)
$CO(NH_2)_2$	Urea / Carbonic diamide (IUPAC name)
d	Day (86400 s)
d	only if mentioned in context with a scale: Readability (resolution)
dr	Dehydration run
dm	Dehydration medium
EPS	Expanded polystyrene
FAO	Food and Agriculture Organization of the United Nations
IUPAC	International Union of Pure and Applied Chemistry
g	Gram (1×10^{-3} kg)
h	Hour (3600 s)
ICP	Inductively coupled plasma
K	Kelvin
kg	Kilogram
L	Litre (1×10^{-3} m^3)
Lime	Calcium hydroxide $Ca(OH)_2$
m	Metre
max.	Maximum
MgO	Magnesium oxide (IUPAC name)
min	Minute (60 s)

mL	Millilitre (1×10^{-6} m³)
mm	Millimetre (1×10^{-3} m)
Mt	Megatonne (1×10^{9} kg)
N	Nitrogen (IUPAC name)
NH_3	Ammonia / Azane (IUPAC name)
NH_4^+	Ammonium / Azanium (IUPAC name)
pH	Potential of hydrogen
PP	Polypropylene / Poly(propene) (IUPAC name)
s	Second
SD	Standard deviation
SDGs	Sustainable Development Goals
SLU	Swedish University of Agricultural Sciences
t	Tonne / metric ton (10^3 kg)
Tg	Teragram (10^9 kg)
tot	Total
Tot-C	Total carbon
Tot-N	Total nitrogen
Tot-P	Total phosphorus
TS	Total solids
UDDT	Urine-Diverting Dry Toilet
UDFT	Urine-Diverting Flush Toilet
UDT	Urine-Diversion Toilet
UN	United Nations
UNICEF	United Nations Children's Fund
USA	United States of America
VS	Volatile solids
W	Watt (m^2 kg s^{-3})
WHO	World Health Organization
µm	Micrometre (1×10^{-6} m)

1. Introduction

By the year 2050, there will be an estimated 9.7 billion people living on earth (United Nations, Department of Economic and Social Affairs, Population Division 2017). An increase in population inevitably comes with an increased demand for food and agricultural products. In conventional agriculture, fertiliser containing N (nitrogen), P (phosphorus) and K (potassium), a so-called "NPK-fertiliser", is often used to fertilise agricultural soils and to increase crop yields. Such artificial fertiliser is produced by using finite resources like potash and phosphate rock, and energy-intense processes are needed to make nitrogen derived from the atmosphere plant-available. Rockström et al. (2009) established planetary boundaries for the exploitation of these finite resources, and Steffen et al. (2015) showed that humanity has, in regards to the biochemical flows of nitrogen and phosphorus, exceeded the planetary boundaries for safe development. Steffen et al. (2015) determined that the planetary boundary for the flow of P from freshwater systems to the ocean lies at approximately 11 Tg P $year^{-1}$, while the current flow lies at 22 Tg P yr^{-1}, so at about double of the amount of the planetary boundary. They further state that the planetary boundary for the intentional and industrial biological fixation of N is at 62 Tg N yr^{-1}, while it is currently approximately 150 Tg N yr^{-1}, which is more than double of the limit. This means that humanity needs to cut down on introducing reactive N and P into water bodies, as the current conditions are far beyond sustainable. According to the Food and Agriculture Organization of the United Nations (2019), the world demand for fertiliser nutrient use of N, P_2O_5 and K_2O is projected to increase to 201 Mt in 2022 though. This represents an increase of over 7.8 % over the demand of 2016. To satisfy the increase in fertiliser demand, and to be able to feed an additional 2 billion people by 2050, humanity needs to either increase the production of artificial fertiliser or ramp up its efforts to find other strategies to supply crops with nutrients.

One of these strategies is the recycling of plant nutrients that are excreted through urine and faeces. Presently human excreta are often seen and treated as waste products and sometimes even discarded without treatment, although they could be safely collected, treated and recycled. This attitude can lead to environmental problems like eutrophication of ponds and lakes and the development of hypoxic zones in coastal areas (Lienert & Larsen 2010; National Oceanic and Atmospheric Administration 2019). Akram et al. (2019) on the other

hand showed that if Sweden would recycle all human excreta (based on data from 2007), instead of treating them as waste products, it could theoretically meet 167 % of its crop potassium, 81 % of its crop phosphorus and 75 % of its crop nitrogen needs. This shows that there is the potential that excreta recycling can decrease the demand for artificial NPK-fertiliser not only in Sweden but worldwide. Excreta are the sum of urine and faeces, which both contain valuable plant nutrients. These plant nutrients are not equally distributed between urine and faeces though, with urine containing 50–80 % of the phosphorus, 80–90 % of nitrogen and 80–90 % of the potassium of the amounts consumed through food (Kirchmann & Pettersson 1994; Jönsson 2001; Vinnerås 2001). Faeces also contain bacteria that are pathogenic for humans, while human urine is usually free of pathogens (Willey et al. 2014). Because of their inherently different properties, it is beneficial to collect and store urine and faeces separately for further treatment and usage (Larsen et al. 2013).

In recent decades, the beneficial properties of urine have raised interest in using human urine or products derived from it, as a fertiliser (Kirchmann & Pettersson 1994; Larsen & Gujer 1996; Jönsson et al. 1997; Stintzing et al. 2004; Vinnerås et al. 2008; Winker et al. 2009; Karak & Bhattacharyya 2011; Senecal & Vinnerås 2017; Harder et al. 2019; Simha et al. 2020b). The simplest way of separately collecting urine and faeces is by using urine separating toilets and urinals, where separation occurs directly after excretion. Stored urine could be applied to agricultural fields directly (Vinnerås et al. 2008), but its high water content of 97 % (Putnam 1971) can cause problems with logistics and application, as high volumes have to be transported over long distances to be applied to agricultural fields (Trimmer & Guest 2018; Chipako & Randall 2020). This is why different strategies for nutrient extraction and nutrient concentration from urine have emerged in recent years (Maurer et al. 2006; Harder et al. 2019). Some technologies like membrane distillation (Tun et al. 2016; McCartney et al. 2020), passive evaporation (Bethune et al. 2014) and nitrification-distillation (Udert & Wächter 2012; Fumasoli et al. 2016), focus on volume reduction, which increases the nutrient concentration in the product. Other technologies, for instance, ion-exchange (Tarpeh et al. 2017), precipitation (Lind et al. 2000; Etter et al. 2011) and stripping (Başakçilardan-Kabakci et al. 2007; Pradhan et al. 2017) on the other hand focus on the extraction of selected nutrients from urine. While volume reduction can be very advantageous regarding logistics, most of the mentioned technologies do not capture all plant nutrients. For instance, during passive evaporation, nitrogen is lost to the atmosphere because of urea hydrolysis (Bethune et al. 2014). Therefore, before the volume of urine is reduced, it needs to be biochemically stabilised either by acidification or alkalinisation to inhibit urea hydrolysis by urease (Randall et al. 2016; Saetta & Boyer 2017). The stabilised urine can then be dehydrated to produce a dry, powder-like product, which can easily be transported and spread on

agricultural soil. Alkaline urine dehydration technologies have successfully been tested in the past and show promising results regarding volume reduction and nutrient recovery (Dutta & Vinnerås 2016; Senecal & Vinnerås 2017; Simha 2020; Simha et al. 2020b). The closer the dehydration of urine happens to the point of excretion; the faster urea hydrolysis can be inhibited, and the lesser liquid urine volume has to be transported through pipes or by motorised transport. If dehydration happens in the toilet itself, no additional pipes have to be installed, and the moist air can exit the building through the buildings ventilation system. The dry nutrient-rich powder could be collected by municipal waste collection systems, which regularly visit the households. Therefore, on-site dehydration technologies hold the potential for the simple production of nutrient-rich fertiliser. Past studies regarding alkaline dehydration have used different alkalising substances like wood ash, alkalised biochar or calcium hydroxide to stabilise urine (Dutta & Vinnerås 2016; Senecal & Vinnerås 2017; Simha et al. 2018, 2020b). Magnesium oxide though, a substance able to raise the pH of urine to pH 9.9 (± 0.2) and with low solubility in urine, that is regularly used in struvite production from urine (Maurer et al. 2006), has never before been tested as an alkaline dehydration medium, despite its promising properties.

2. Objective

The two main objectives of this experiment were to:

1. evaluate if magnesium oxide is suitable for alkaline urine dehydration, as its low solubility in urine promises benefits regarding cost savings and user-friendliness.
2. compare five dehydration media consisting of varying proportions of magnesium oxide, calcium hydroxide, wheat bran and biochar regarding their dehydration rate, N-recovery, mass reduction and physicochemical properties

3. Literature

3.1. Current practices

Sanitation in countries in Europe and North America heavily relies on sewered sanitation systems. These sanitation systems use drinking water as a transport medium to carry human excreta from their source to a wastewater treatment plant or directly into the environment. Sewered sanitation relies on a costly network of sewage- and water supply pipes, water- and wastewater treatment plants and regular operation and maintenance (Langergraber & Muellegger 2005). In conventional wastewater treatment plants, the focus lies on removing organic matter and plant nutrients like phosphorus or nitrogen from the wastewater. When none of the products of this sanitation system are reused as plant nutrients, it can be described as a linear sanitation system, in comparison to circular sanitation, where plant nutrients are fed back to the agricultural system. The plant nutrients that are entering a linear sanitation system and that are not reused in agriculture, can, therefore, be seen as lost for the agricultural system. The objective of installing such wastewater treatment plants in the first place was to keep plant nutrients and organic matter from entering the water bodies to prevent eutrophication and to prevent the spread of diseases (Orhon 2015).

3.2. Reuse approaches

In recent years, realising the upcoming shortage of non-renewable resources like phosphorus and nitrogen (Rockström et al. 2009; Ashley et al. 2011; Steffen et al. 2015), the topic of plant nutrient recycling is coming back to the agenda of politics and society. Reuse approaches include the direct use of sewage sludge or different methods for phosphorus-recovery from the sewage sludge. The use of sewage sludge as a fertiliser has often been restricted or banned though, or restrictions for its use are being discussed (Regeringskansliet 2018; Umweltbundesamt 2018; VN 2019). In Austria, restrictions were put in place because it has been found that sewage sludge can contain different contaminants like the plasticiser

Bis(2-ethylhexyl) phthalate or the flame retardant Hexabromocyclododecane (Bundesministeriums für Land- und Forstwirtschaft, Umwelt und Wasserwirtschaft (BMLFUW) 2016). These and other contaminants are often introduced into the wastewater stream by mixing blackwater[1] with wastewater from industries, greywater[2] from households and rainwater runoff from streets in combined sewers or at the wastewater treatment plants. This cross-contamination of faeces and urine with contaminants from other sources during its transport and treatment is another reason why alternatives for conventional sewered sanitation systems have gotten more attention since the 1990s. It has been suggested many times that source separation of urine and faeces could be beneficial for plant nutrient recycling (Harder et al. 2019). It was also hypothesised that source separation and decentralised treatment technologies could provide means to tackle the increasing need for sanitation services in urban contexts that are projected to increase due to population growth (Larsen et al. 2013). Human urine attracted particular attention because it contributes a large part of the plant nutrients and volume of human excreta, while at the same time, urine only forms a small part of the total wastewater, less than 1% (Kirchmann & Pettersson 1994; Larsen & Gujer 1996; Jönsson et al. 1997).

3.3. Characteristics and hygienic quality of human excreta

The most obvious difference between human faeces and human urine is their water content and therefore, their state. Human faeces are composed of about 74 % water (Rose et al. 2015) and semi-solid, while urine is liquid and consists of around 97 % water (Senecal & Vinnerås 2017). Besides these visible differences, also the hygienic quality of faeces and urine differs significantly. Faeces can contain a multitude of potentially pathogenic bacteria, viruses, protozoa and helminths (Feachem et al. 1983), while urine, if excreted by healthy mammals, is usually free of pathogens, but not sterile (Hilt et al. 2014; Willey et al. 2014). This is because there is an inhospitable environment in the bladder, kidneys, and ureter produced by metabolic end products like enzymes, fatty acids, mucin, uric acid and a low pH (Willey et al. 2014). If a person is infected with *Leptospira interrogans, Salmonella typhi, Schistosoma haematobium* or *Salmonella paratyphi*, or certain kinds of helminths though, their urine can contain these specific pathogenic microorganisms (Feachem et al. 1983; Heinonen-Tanski & van Wijk-Sijbesma 2005). Separating urine and faeces from the point of excretion onwards provides the benefit of avoiding contact between urine and faeces and therefore, the introduction of large

[1] The mixture of faeces and urine with flush water and anal cleansing material or anal cleansing water.
[2] Water that was used for bathing, washing of clothes, food, and dishware.

numbers of pathogens into the urine. Even though urine separating interfaces separate the urine from faeces, faecal cross-contamination cannot entirely be ruled out with today's technologies. Studies showed that urine in collection tanks had a mean faecal contamination ranging from non-detection up to 13.3 mg L_{urine}^{-1} (Höglund et al. 1998) and 9.1 mg L_{urine}^{-1} (± 5.6 mg) respectively (Schönning et al. 2002).

3.4. Human urine as fertiliser

Although plant nutrients can be found in human faeces as well, urine is the primary source of plant nutrients in human excreta (Table 1) (Vinnerås et al. 2006).

Table 1. Proposed default values for excreted mass and nutrients in kg person^{-1} year^{-1} (Vinnerås et al. 2006)

Parameter	Urine (kg person^{-1} year^{-1})	Faeces (kg person^{-1} year^{-1})
Wet mass	550	51
Dry mass	21	11
Nitrogen	4	0.55
Phosphorus	0.365	0.183
Potassium	1	0.365

About 85 % of the N in urine is present in the form of non-volatile urea (Kirchmann & Pettersson 1994). If the urine gets into contact with urease, an extracellular enzyme with a high abundance in the environment (Mobley & Hausinger 1989; Krajewska 2009), the urea is hydrolysed to the volatile form ammonia (NH_3) and carbonic acid. The half-life of urea, when subjected to urease from jack beans (*Canavalia ensiformis*), is 0.02 s at 25 °C (Callahan et al. 2005). Urea hydrolysis also increases the pH and produces bicarbonate ions (Hellström et al. 1999). After urea is hydrolysed, about 90 % of the total nitrogen occurs as NH_4^+ and NH_3 (Udert et al. 2006).

Equation 1. Urea hydrolysis via urease enzyme

$$CO(NH_2)_2 + 3H_2O \xrightarrow{urease} 2NH_4^+ + HCO_3^- + OH^-$$

Equation 2. Ammonium and dissolved ammonia are in equilibrium

$$NH_4^+ + OH^- \leftrightarrow NH_3(aq) + H_2O$$

Equation 3. Gaseous ammonia is in equilibrium with dissolved ammonia

$$NH_3(g) \leftrightarrow NH_3(aq)$$

Because the urine-pH and the concentration of ammoniacal nitrogen are rising during urea decomposition, there is a risk of ammonia evaporation, which leads to a loss of nitrogen to the surrounding air. Ammonia has a Henry's constant of 62 mol L^{-1} atm^{-1}, making it a very volatile substance (Larsen et al. 2013), so the partial pressure of ammonia in the layer of air on the surface of the urine strongly influences the evaporation of ammonia, and ventilation, for instance through wind or forced ventilation by fans, will increase the ammonia-evaporation (Hellström et al. 1999). Since the urease enzyme is ubiquitous, urea hydrolysis will occur, increasing pH. The increased pH causes precipitation of struvite, calcite and hydroxyapatite in sewage and urine pipes and storage tanks (Udert et al. 2003; Larsen et al. 2013) and also occurs when urine is applied directly to agricultural fields. The ammonia in urine that degases into the surrounding air can be seen as "lost" for the agricultural system, as most plants cannot fix nitrogen from the air. This ammonia-loss is not only an odour nuisance but also contributes to environmental pollution and a decline in plant biodiversity (Guthrie et al. 2018). Urease activity and enzymatic ureolysis need to be inhibited to minimise these nitrogen losses due to urea hydrolysation. This can be done by stabilising the urine through acidification (Hellström et al. 1999; Saetta & Boyer 2017) or alkalinisation (Randall et al. 2016; Senecal & Vinnerås 2017). As urease is ubiquitous in toilet bowls, sewage pipes and storage tanks and since the half-life of urea can be as short as 0.02 s, inhibition of ureolysis needs to happen as close to the source of excretion as possible. If nitrogen recovery in the form of urea is to be maximised, long transport of untreated urine through pipes should be avoided. This speaks for decentralised sanitation systems, where urine separation and treatment happen at the source.

3.5. Urine-diverting user interfaces

To collect urine and faeces separately, they need to be kept separate from excretion onwards. There are several user interfaces on the market, which facilitate this separation. So-called "Urine-Diversion Toilets" (UDT) keep urine and faeces separate at the source. There are two different varieties: "Urine-Diverting Dry Toilets" (UDDT) and "Urine-Diverting Flush Toilets" (UDFT). In UDDTs, the faeces are collected without the addition of flush water (and with the design shown in Figure 1, also without anal cleansing water), and in UDFTs the faeces get flushed away using water (Figure 2). Urinals, as seen in Figure 3, facilitate the collection of urine with or without flush water, but do not offer a possibility for the collection of faeces.

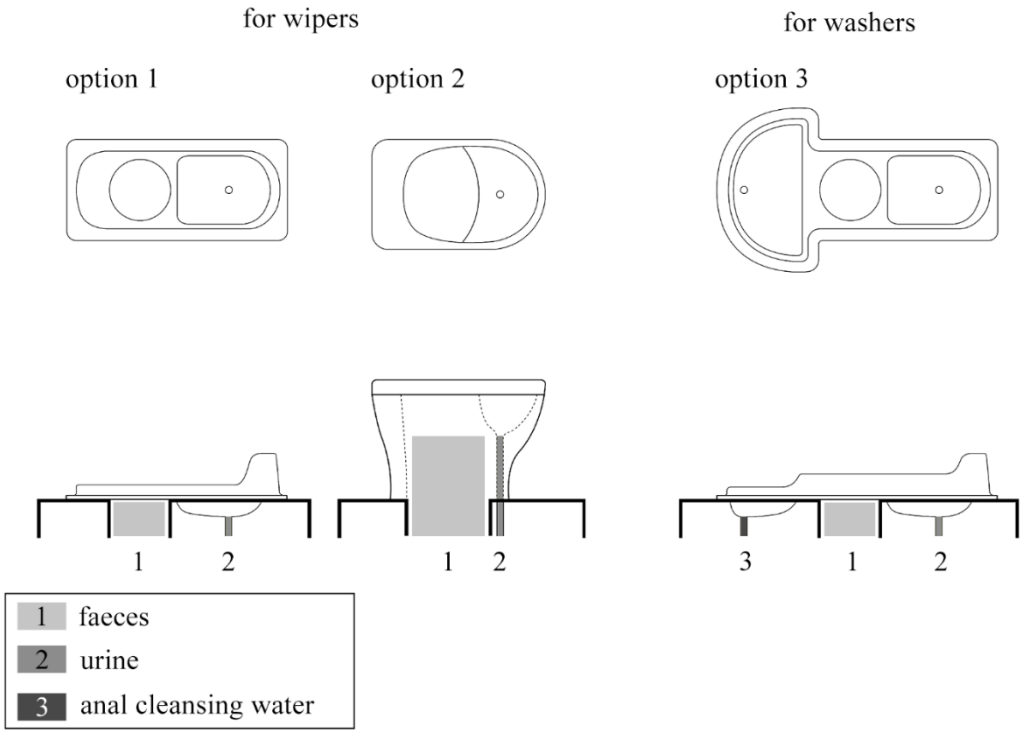

Figure 1. Urine-Diverting Dry Toilets (UDDTs), with the design for people using toilet paper on the left and the design for people using water for anal cleansing on the right side (amended from Tilley et al., 2014)

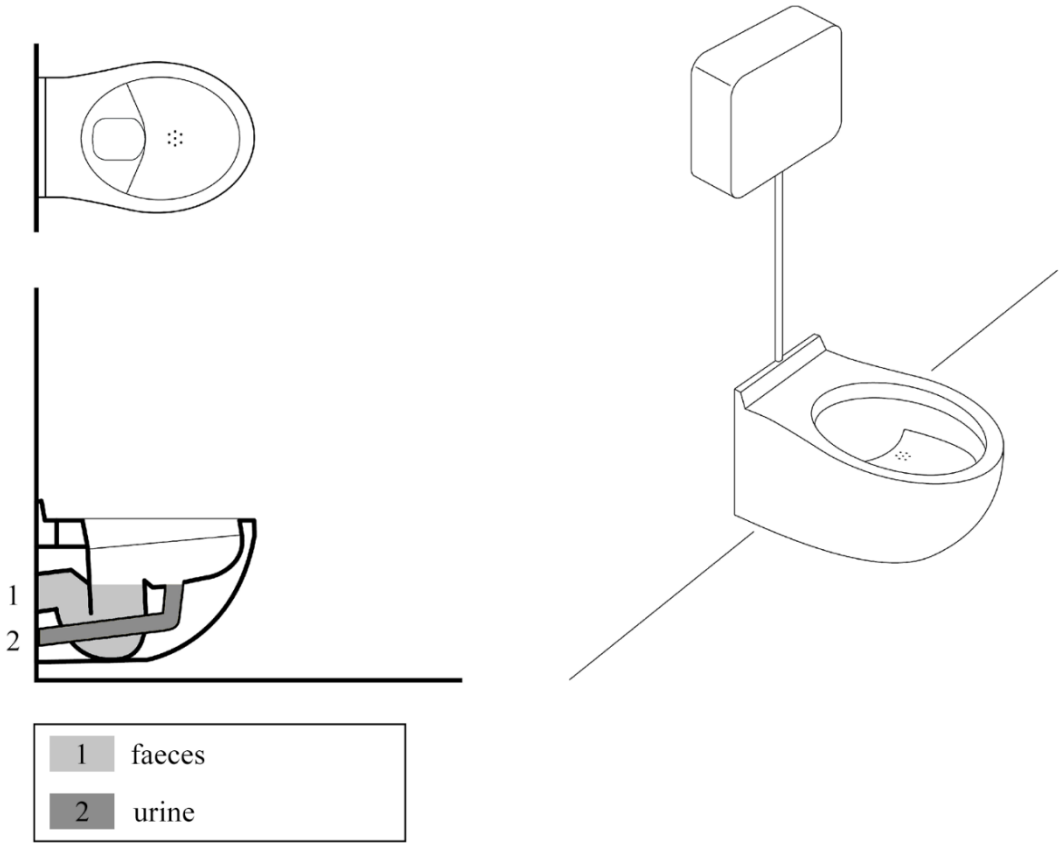

Figure 2. Urine-Diverting Flush Toilet (UDFT) (amended from Tilley et al., 2014)

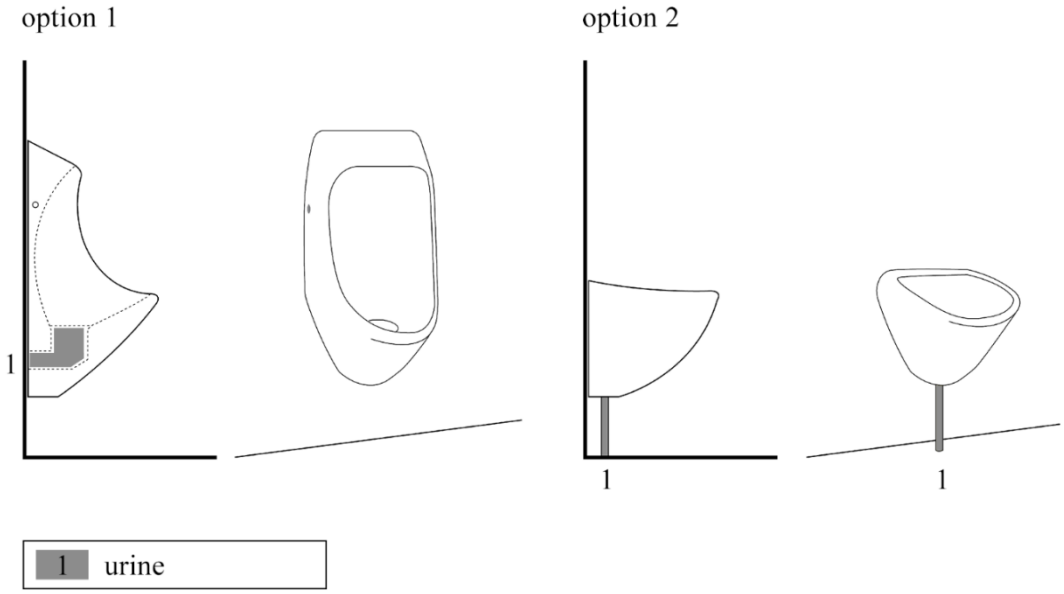

Figure 3. Different versions of urinals (amended from Tilley et al., 2014)

3.6. Urine treatment technologies

Due to the different scenarios where a need for urine treatment and urine-diversion might arise, like in a rural area versus a city, with centralised versus decentralised treatment solutions and because of the different aims of urine treatment, various urine treatment technologies have emerged. Maurer et al. (2006) classified urine treatment technologies into seven categories: stabilisation, volume reduction, hygienisation, N-recovery, micropollution removal, P-recovery and nutrient removal. Harder et al. (2019) then classified treatment technologies for urine and yellow water (a mixture of urine and flush water) into four categories: stabilisation, contaminant reduction, water extraction and nutrient extraction. These two classifications are mostly overlapping, and as the focus of this study lies in the recovery of N from urine, an overview of the most relevant technologies in the area of nutrient recovery is provided:

1. Struvite crystallisation

   Struvite, which is magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$), can be produced by adding magnesium to urine, and it precipitates in a pH range of about pH 7 to pH 11. Usually, $MgCl_2$, $Mg(OH)_2$, or MgO are used as a magnesium source (Maurer et al. 2006). When the urea in urine is hydrolysed, it increases the alkalinity of urine by about factor 20 (Udert et al. 2006), which further aids struvite precipitation (J. R. Buchanan et al. 1994). More than 90 % of phosphate and some N can be recovered by struvite precipitation, and which can then be used as a slow-release fertiliser (Bridger et al. 1962; Lind et al. 2000; Ban & Dave

2004; Ronteltap et al. 2007, 2010; Wilsenach et al. 2007; Etter et al. 2011; Udert et al. 2016; Harder et al. 2019).

2. Ion Exchange

   Substances with a high affinity for ammonium like naturally occurring zeolite (for instance clinoptilolite or wollastonite) have been tested for removal of ammonia from urine and the treatment of wastewater (Ban & Dave 2004; Tarpeh et al. 2017). Ion exchange combined with struvite precipitation recovered 65–80 % of N from urine (Lind et al. 2000).

3. NH_3-stripping

   Ammonia gas can be stripped of urine using air as a carrier and water or H_2SO_4 as a receiving medium. Behrendt et al. (2002) showed that ammonia extraction from stored urine under vacuum (0.4 bar pressure) at 40 °C, and absorption of the gas in water under 5 bar at 20 °C, resulted in a 10 % ammonia solution. Pradhan et al. (2017) used a combination of struvite production with $Ca(OH)_2$ and ammonia stripping with H_2SO_4 and recovered 99 % of P and 85–99 % of N (w/w) from urine in 28 h at 40 °C.

4. Nitrification-distillation

   This system combines biological nitrification by nitrite-oxidising bacteria and ammonia-oxidising bacteria for nutrient recovery with distillation for nutrient concentration. This combination of processes reduces the water content of the urine by 95–97 % and recovers all nutrients contained in urine in a concentrated nutrient solution or a dry solid, although about 3 % of the total nitrogen of the urine is lost during distillation. The process produces distilled water and sludge as a by-product and uses about 140 Wh L^{-1} energy, which is mainly used for water evaporation (Udert & Wächter 2012; Fumasoli et al. 2016; Udert et al. 2016).

5. Membrane distillation

   In conventional, direct contact membrane distillation, a temperature difference from one side of a hydrophobic, microporous membrane to the other creates a vapour pressure difference, which leads to a dewatering process. This concentrates the nitrate and ammonia on the membrane's feed side (Tun et al. 2016). Other technologies like isothermal membrane distillation with an acidic collector have a lower energy demand than conventional membrane distillation, do not suffer from poor selectivity in ammonia transport which dilutes the ammonia concentration of the product and achieve about 60 % ammonia recovery from urine (McCartney et al. 2020).

6. Dehydration

   Urine consists of approximately 97 % of water (Senecal & Vinnerås 2017), which is why dehydration can drastically reduce the volume of urine. All nutrients found in urine can be fully recovered through dehydration. However, if the input urine is not stabilised by acidification or alkalinisation, most of the nitrogen degases to the surrounding air in the form of NH_3, due to the hydrolysis of urea in urine. In the passive and open evaporation system that Bethune et al. tested in 2014, about 90 % of the NH_3/NH_4 of the input-urine got lost to the atmosphere. Measures to prevent hydrolysis of urea, which drastically increase the N-recovery in the dehydration step include acidification (Antonini et al. 2012; Saetta & Boyer 2017) and alkalinisation of the urine (Dutta & Vinnerås 2016; Simha et al. 2018, 2020b). If the urine is alkalised, all P and more than 90 % of N can be recovered from the urine (Simha et al. 2020b).

Besides dehydration and nitrification-distillation, all these technologies leave behind a liquid residue containing most of the other constituents of urine besides the recovered nutrient(s). This presents a problem, as this residue has to be dealt with and presents a new waste stream (Udert et al. 2016). Dehydration elegantly avoids creating this additional waste stream by evaporating the water and only leaving behind a solid residue, which can then be used as a dry fertiliser in agriculture.

3.7. Dehydration

The purpose of dehydration is “to remove bound water or hydrogen and oxygen from (a chemical compound) in the proportion in which they form water” (*Definition of DEHYDRATE* 2018 by Merriam-Webster). This study used convective cross-flow air drying in a batch mode to remove water from the dehydration medium by evaporation, to reduce the volume and the weight of the product. This dehydration process involves two steps that happen simultaneously – heat transfer and the resulting mass transfer. Heat energy is transferred to the dehydration medium by the heated air. The water vapour that forms above the dehydration medium's surface due to evaporation is transported away from the substance (confer Figure 4). Factors that influence the dehydration process are the velocity, temperature and humidity of the air. As long as the evaporation rate limits the dehydration rate, increasing the air velocity will increase the heat and mass transfer and the dehydration rate. Increasing the temperature and decreasing the humidity of the air will also increase the dehydration rate in the onset, as long as case hardening (the formation of a moisture-impermeable crust on top of the

dehydration medium) is avoided (Berk 2009). If moisture removal rates are improved by one of the factors mentioned above, the area required for dehydrating a particular volume of water or urine could be reduced, hence improving the area footprint of the urine dehydrating technology (Simha et al. 2020b).

3.8. Alkaline dehydration of urine

Urea hydrolysis is pH-dependent, and Kabdaşlı et al. (2006) reported that no biological or chemical urea hydrolysis is detectable at pH 10 or higher at a temperature of 20 °C (± 1 °C). However, Geinzer (2017) found that urease enzymes can be reactivated after the pH drops below pH 10 again. Bethune et al. (2014) showed that about 90 % of the NH_3/NH_4 from the input-urine got lost in their open passive evaporation system that did not inhibit urease. This significant loss of nitrogen indicates that passive evaporation without inhibition of urease activity is not an efficient way to recover nitrogen from urine.

Studies showed that it is possible to recover > 90 % of the N from urine by alkaline dehydration, using different dehydration media and temperatures (Simha et al. 2020b). The dehydration media and temperatures used, and the N-retention attained in earlier studies were:

1. a mixture of calcium hydroxide and wood ash, with a dehydration temperature of 20 °C, 35 °C and 60 °C, leading to an N-recovery of up to 74 % at 35 °C (Dutta & Vinnerås 2016),
2. wood ash, at 35 °C and 60 °C, leading to an N-recovery of up to 90 % at 35 °C (Senecal & Vinnerås 2017),
3. wood ash or alkalised biochar, at 40 °C, 45 °C and 50 °C, leading to an N-recovery of >70 % at all temperatures (Simha et al. 2018),
4. biochar, wheat bran, desert soil, wood ash and calcium hydroxide, alone or in combination at a temperature of 50 °C and 60 °C, leading to an N-recovery of > 90 % at both temperatures (Simha et al. 2020b),
5. a mixture of calcium hydroxide and wood ash, used in two pilot plants, at dehydration temperatures varying from 40 °C (± 27 °C) to 66 °C (± 15 °C) and from 36 °C (± 20 °C) to 64 °C (± 14 °C) respectively, lead to a recovery of 30 % (± 6 %) N (Simha et al. 2020a).

The concept of alkaline dehydration is illustrated in Figure 4, exemplified by the setup used for this study.

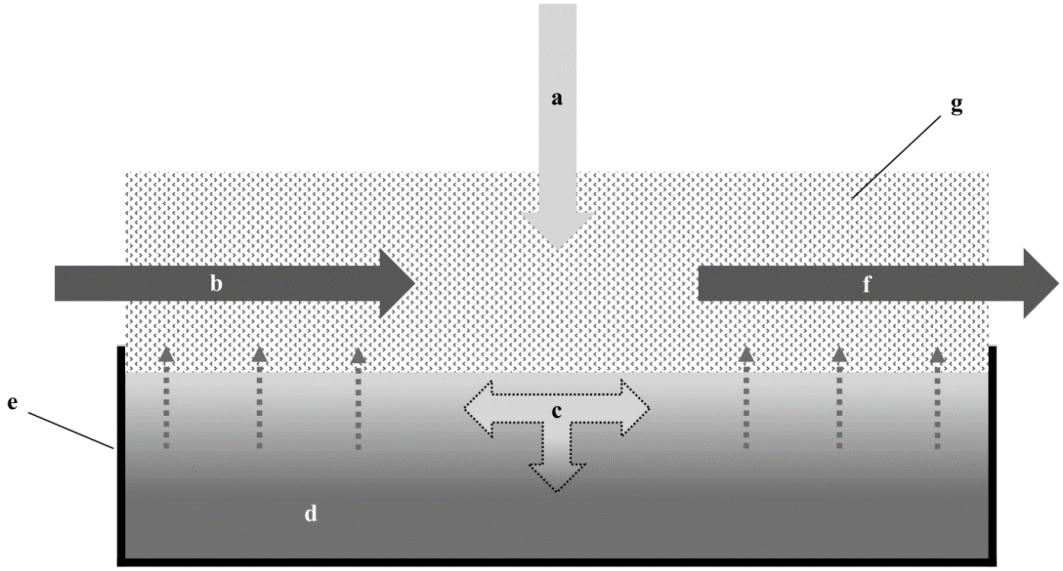

Figure 4. Alkaline dehydration of urine exemplified by the setup used in this study; a. fresh urine; b. heated air; c. urine infiltrating in and being absorbed by the dehydration medium; d. alkaline dehydration medium (e.g. magnesium oxide as alkalising agent and biochar as co-substrate); e. Petri dish; f. moisture-laden air; g. evaporation (own work, adapted from Simha et al. (2018))

3.8.1. Limiting factors for alkaline dehydration of urine

Temperature

In previous studies at SLU (Dutta & Vinnerås 2016), it was found that lower dehydration temperatures (for example 35 °C) lead to higher nitrogen retention if urine is dehydrated on a mixture of ash and lime. This gain in N-retention comes at the cost of longer dehydration times. Randall et al. (2016) tentatively suggested an upper limit for dehydration temperature at 40 °C to avoid excessive chemical urea degradation, which he suggested should be explored in future studies.

pH

The pH of the mixture of urine and dehydration medium gradually declines during convective dehydration, mainly because CO_2 is being absorbed, and CO_3^{2-} and HCO_3^- is formed (Simha et al. 2018). This means that either enough alkalising medium to always keep the pH >10 needs to be provided initially or additional alkalising medium needs to be continuously resupplied, risking N-loss if urea is reactivated when the pH falls below 10, as shown by Geinzer (2017). Chemical urea hydrolysis limits the scope of both urine acidification and alkalinisation technologies, as it significantly decreases the urea half-life from more than a year at around 20 °C down to days at pH <2 and pH >12 at temperatures of >60 °C (Randall et al. 2016).

3.8.2. Alkalising media used in previous studies

The studies mentioned above that were carried out at SLU looked at wood ash, alkalised biochar and calcium hydroxide as alkalising media.

Wood ash

Wood ash is a waste product readily available in regions where wood is used for cooking or heating purposes. It has a high initial pH of >12.5 and a high surface area (Senecal & Vinnerås 2017).

Alkalised biochar

The pH of biochar depends on the feedstock from which is it derived and the conditions during pyrolysis. Zhang et al. (2019) found the biochar used during their experiments to have a slightly alkaline pH of around 8.76 (Zhang et al. 2019). Since urea hydrolysis is only inhibited by a pH >10 (Geinzer 2017), it needs to be alkalised to be used as an alkalising agent. This can be done, for instance, by a method described by Simha et al. (2018) where biochar is mixed with KOH pellets and deionised water, to reach a pH >12.5.

Calcium hydroxide ($Ca(OH)_2$)

Calcium hydroxide is well known for its alkalising properties and used in agriculture to treat acidified soils. Randall et al. (2016) showed that the saturation-pH of calcium hydroxide in urine is pH 12.5, its solubility in urine 3.5–5 g L^{-1} and that the addition of 4.3–5.8 g $Ca(OH)_2$ $L^{-1}_{\text{fresh urine}}$ at 25 °C prevented urea hydrolysis by urease. They also suggest adding 10 g $L^{-1}_{\text{fresh urine}}$ to ensure sufficient $Ca(OH)_2$ is always available for urease inhibition. It is a low-cost, broadly available bulk chemical (US$ 0.08 kg^{-1}) (Muster et al. 2013), which is already extensively used in agriculture to combat acidification of soils (Haynes & Naidu 1998).

3.9. Motivation for dehydration media selection and system setup

Two alkalising media were chosen for this study: magnesium oxide and calcium hydroxide. Two co-substrates were chosen for this study: wheat bran and biochar.

3.9.1. Magnesium oxide (MgO)

The motivation for including MgO in this study was threefold. Firstly, it was to be tested, if the saturation-pH of magnesium oxide of pH 9.9 (SD 0.2) in urine (Simha et al. in preparation) would be sufficient for sustained inhibition of urea hydrolysis, as Geinzer (2017) found that urea hydrolysis was only inhibited at pH >10.

Secondly, it was assumed that its low solubility in unhydrolysed urine of <1.5 g L_{urine}^{-1} (Simha et al. in preparation) could provide benefits over other alkalising agents like $Ca(OH)_2$, whose solubility is more than double. A dehydration medium containing MgO that is used in a dehydrating toilet would have to be changed less frequently than a dehydration medium containing $Ca(OH)_2$, which would increase the user-friendliness of the toilet. Thirdly, the ability of MgO to capture the ammonium present in urine that was shown in earlier studies (Wilsenach et al. 2007) might further increase N-recovery. Because of its novel use as an alkalising agent for alkaline urine dehydration, it was also of the highest interest to test the use of pure magnesium oxide as a dehydration medium.

3.9.2. Calcium hydroxide ($Ca(OH)_2$)

Calcium hydroxide was selected for this experiment because of its beneficial properties like high alkalinity, low price, broad availability, and its widespread use in agriculture. To establish if there were differences in the pH of the dehydration media where calcium hydroxide is used in combination with magnesium oxide, calcium hydroxide was mixed into two out of five dehydration media used in this study. For alkalinisation of fresh urine, Randall et al. (2016) suggest using 10 g $Ca(OH)_2$ L_{urine}^{-1}, to have solid $Ca(OH)_2$ present in the urine at all times to ensure a high pH. Studies about the sole use of calcium hydroxide as dehydration medium already exist (Simha et al. 2020b), so for this study, calcium hydroxide was mixed with magnesium oxide, which itself is also able to raise the pH to about pH 10, so it was chosen to use less than 10 g $Ca(OH)_2$ L_{urine}^{-1}.

3.9.3. Wheat bran

Wheat bran, which forms about 14–16 % of the grain, is produced during the milling process when the endosperm is separated from the germ and bran fraction of wheat. It is made up of the aleurone layers, testa, pericarp and hyaline (Stevenson et al. 2012). When wheat bran was compared against sawdust as a potential candidate for the use as a part of the dehydration media, it seemed to have less of a market for reuse, besides being used as an ingredient of fodder and for few baking goods (Reisinger et al. 2013). So, using wheat bran for urine dehydration could establish new reuse options and open new markets for it. Wheat bran also introduces some nitrogen into the dehydration medium, making the final fertiliser product an even more valuable fertiliser.

3.9.4. Biochar

Biochar is widely used as an agricultural soil amendment (confer “Terra preta”) (De la Rosa 2020). It can be produced by a range of processes like hydrothermal conversion, pyrolysis or gasification, where biomass is heated without a supply of

oxygen (Zheng et al. 2010). Biochar is able to adsorb NH_4^+ and PO_4^{3-} (Cai et al. 2016; Takaya et al. 2016; Trazzi et al. 2016), and ammonia adsorbed by biochar was shown to be bioavailable for plants (Taghizadeh-Toosi et al. 2012). Urea intercalated biochar showed potential as a slow-release fertiliser (Manikandan 2013). Trial runs established that biochar could break up the lipid-film that can accumulate at the surface of urine during dehydration, a property, which could prove beneficial during dehydration (unpublished data). Biochar is widely available, but prices vary significantly from 80 USD to > 13 000 USD t^{-1} depending on, e.g. the quality of the biochar (Campbell et al. 2018). As biochar has so many beneficial properties, it was included in four out of five dehydration media of this study. The percentage of biochar varied from 33 % to 75 % of the mass of the dehydration medium.

3.9.5. Airflow and dehydration temperature considerations

Choosing an airflow rate of around 5 L_{air} min^{-1} and a dehydration temperature of 50 °C was based on experiences made during previous studies. For instance, Dutta and Vinnerås (2016) aimed at inhibiting urea hydrolysis by urease by using a mixture of $Ca(OH)_2$ and wood ash as dehydration medium. In their study, the highest nitrogen retention of 74 % was achieved at a dehydration temperature of 35 °C. Dutta and Vinnerås stated that further research was needed to find an optimal combination of a dehydration temperature between 35–60 °C and airflow of 1–5 L min^{-1} in the dehydration setup.

4. Materials and Methods

4.1. Urine collection

Approximately twenty volunteers (male and female) of around 25–65 years and with different diets and lifestyles anonymously donated urine at the Department of Energy and Technology, SLU at different times during the day. Sterile, high-density polyethene bottles with a screw cap, a separate seal and a volume of 500 mL (VWR International, Gosselin, France) were used to collect the urine. The filled bottles were collected after each working day, so within less than 12 h and stored at 3 °C (± 1 °C) for less than 14 days before the urine was used for the experiments. The collected urine had an average pH of 6.6 (SD 0.2) and an average EC of 11.7 mS/cm (SD 1.2).

Figure 5. Urine collection in sterile bottles in the toilets at the department of Energy and Technology, SLU (own work)

Figure 6. Urine yield of one workday (approximately 9 L) (own work)

4.2. Experimental procedure

4.2.1. Urine preparation

To prepare a uniform urine mixture for the dehydration runs, about 5 L of urine was taken from the refrigerator and mixed in a sterile, 5 L glass Florence flask. The mixed urine was then poured back into the 500 mL HDPE-bottles (Gosselin, France) in which the urine was collected and stored before mixing. 450 mL of fresh urine was needed for each run of the experiment (30 mL of urine were applied to each of the 15 samples of the dehydration media), so 450 mL of urine was put aside for start of the experiment, and all remaining bottles were refrigerated again for future dehydration runs.

4.2.2. Media preparation

Each dehydration medium had a mass of 30 g at the start of the experiment (composition confer Table 2). For detailed descriptions about sources of the materials used for the dehydration media, see Table 6 in the Appendix. Magnesium oxide (MgO) (Acros Organics, Belgium; laboratory-grade) in powder form (d_{99} < 150 µm), non-activated biochar (Vindelkol AB, Sweden; grain size < 1 mm), wheat bran (Kungsörnen, Sweden; food-grade) and calcium hydroxide ($Ca(OH)_2$) (Nordkalk Corporation, Sweden; technical grade, powder-form) were used as is, and not pre-treated.

Table 2. Mass composition of the dehydration media, values of ingredients given in % of total mass, rounded to one decimal place. The mass of each dehydration medium was 30 g

Dehydration media name	MgO (% of total) (MgO)	Biochar (% of total) (Char)	Wheat Bran (% of total) (Bran)	$Ca(OH)_2$ (% of total) (Lime)
MgO	100.0			
MgO-Char	25.0	75.0		
MgO-Char-Lime	11.7	66.7		21.7
MgO-Char-Bran	25.0	37.5	37.5	
MgO-Char-Bran-Lime	11.7	33.3	21.7	33.3

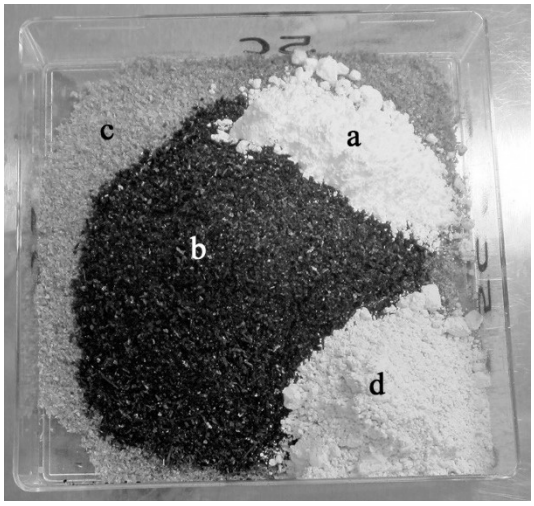

Figure 7. Dehydration medium with MgO (a), biochar (b), wheat bran (c) and $Ca(OH)_2$ (d) during the weighting and before the mixing process (own work)

30 g of dehydration medium (composition in Table 2) was placed into square polystyrene Petri dishes with an edge length of 100 mm and a height of 20 mm (Sarstedt, Germany). The substances were weighed into the same Petri dish and mixed using a stainless-steel spatula (confer Figure 7). The experiment was done in triplicate, meaning that three Petri dishes were prepared with each dehydration medium.

4.2.3. Application of urine

Before application to the dehydration media, the urine was heated to 37 °C (± 2 °C) in a water bath for about 5 min, to mimic the temperature of urine at excretion. The urine temperature was repeatedly measured with a thermometer to avoid overheating (FLUKE 52 k/J, John Fluke MFG.CO., INC). Before each dehydration run, 30 mL of urine was uniformly applied to each dehydration medium, using a pipette (Eppendorf Research 10 mL Pipette, Eppendorf AG). The weight of the added urine was weighed (Adventurer Pro AV2102, OHAUS Corporation;

d = 0.01 g), and an average urine density of 1.04 g mL^{-1} (SD 0.01) was calculated. Therefore, the 90 mL of urine that were applied during the first run equated to an average of 94.90 g (SD 0.61) and the 30 mL of urine that were added for each other experimental run, equated to an average of 31.33 g (SD 0.74). In total, an average of 1159.99 g (SD 1.97) of urine was added to each dehydration medium throughout the experiment. Unpublished trial runs (Simha and Friedrich) showed that biochar is very electrostatic for about the first 12 hours of dehydration time, leading to a loss of biochar as particles get displaced out of the Petri Dishes. It was also noted that biochar gets displaced out of the Petri Dishes if the fans' air velocity inside the dehydration setup is too high. Based on the experience from these trial runs, it was decided to:

1. apply a larger amount of urine (90 mL instead of 30 mL) to each dehydration medium before the very first dehydration run
2. reduce the initial fan speed

4.2.4. Dehydration time and temperature

Dehydration happened at a mean temperature of 48.3 °C (SD 2.8 °C) (confer Dehydration temperature in Results). Based on experiences made during a previous study by Simha et al. (2020) an estimated dehydration rate of 7 min mL^{-1} was assumed, and each dehydration run was set to last for 3.5 h (0.15 d). Due to the addition of 90 mL of urine in the first dehydration run, a dehydration time of 10.5 h (0.44 d) for this dehydration run was chosen. Every sixth experimental run, the dehydration media were dried for 4.5 h (0.19 d), to avoid the accumulation of excess urine. After each dehydration run, the samples were removed from the oven and weighed (Adventurer Pro AV2102, OHAUS Corporation; d = 0.01 g).

4.2.5. Dehydration setup

Fresh urine was dehydrated in a modified benchtop oven (Electrolux, Sweden) with inner dimensions of 42 cm width by 35 cm depth by 32 cm height. The oven was equipped with eight computer fans (Spire Corp, The Netherlands) with a dimension of 60 mm edge length by 15 mm height, rated at an airflow of 464 L min^{-1} at 12 V to allow for better air distribution inside the oven. Four fans (two fans on each side) were installed for each of the two layers of drying racks in the oven (Figure 8, number 2). The front of the fans was facing the dehydration media, and the fans were pointed towards each other, providing an airflow parallel to the surface of the dehydration media. The fans had an adjustable voltage which allowed for regulation of the fan speed. Four holes were drilled into the top of the oven to insert plastic tubing for forced aeration of the oven with four aquarium pumps (“Rena 301”) (Rena Aquatic Supply, USA), each rated at 5–5.5 L_{air} min^{-1} and 30 kPa (Figure 8,

number 1). Plastic T-piece adapters were installed at the end of the aquarium pump tubing to ensure distribution of the inlet air parallel to the surface of the dehydration media (Figure 8, number 5).

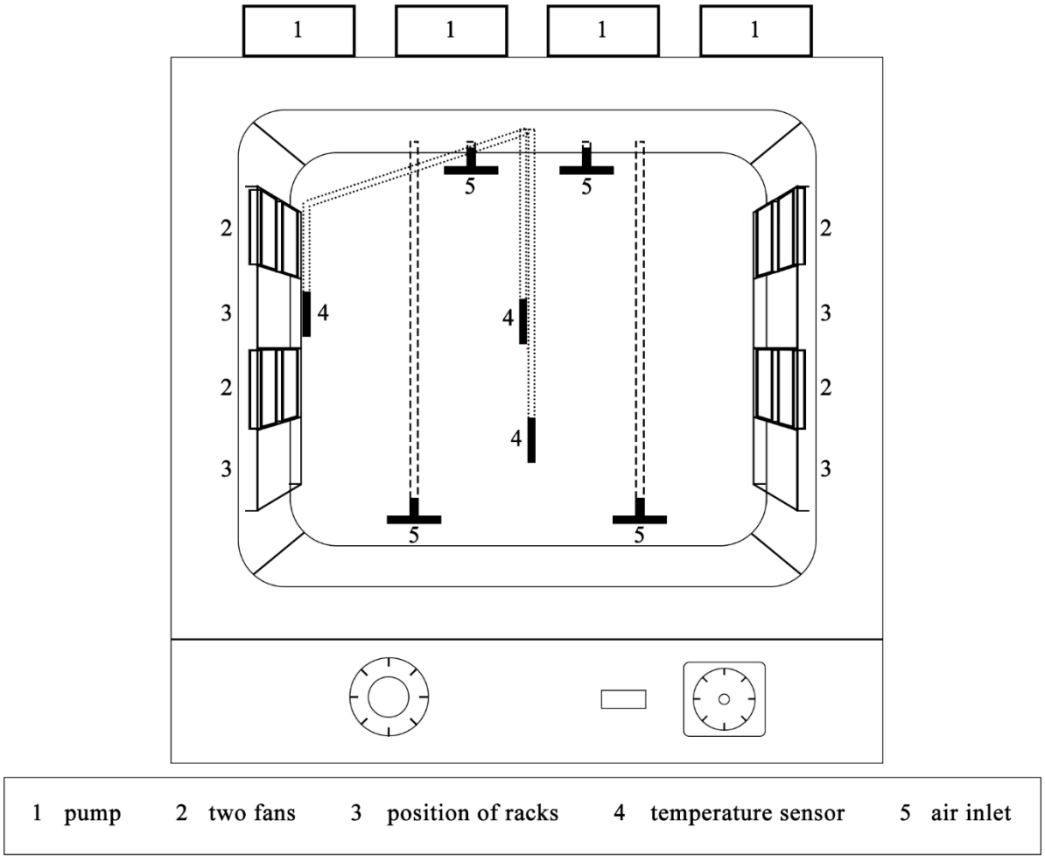

Figure 8. Schematic drawing of modified benchtop oven used as dehydration setup with inner dimensions of 42 cm width by 35 cm depth by 32 cm height (own work)

Two standard oven racks were used to ensure unrestricted airflow. The benchtop oven's door was kept open at a width of 4 cm during the whole experiment to avoid overpressure and let moist air escape the dehydration system.

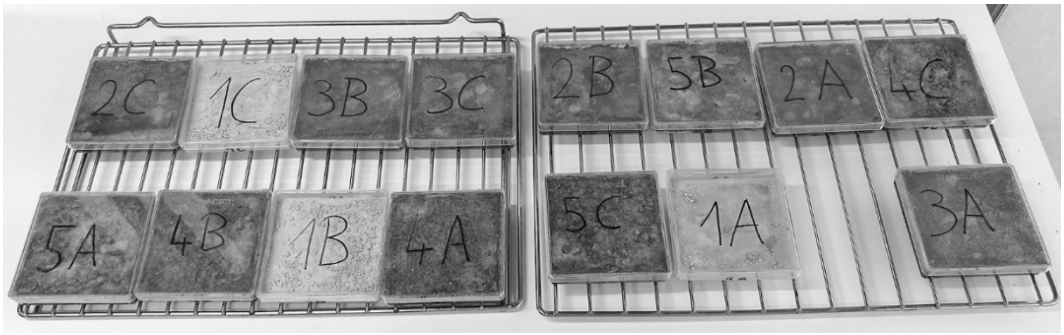

Figure 9. Oven racks used in the dehydration setup with five dehydration media in triplicates (own work)

4.2.6. Shifting of the samples in the dehydration setup

The position of the samples in the oven was changed before each dehydration run. During trial runs (unpublished data), the samples positioned next to the fans or on the lower oven rack (closest to the heating elements in the floor of the oven), dried best. After each run, priority for the places with better drying capacity was given to samples that had pooling on top of the substrate. This was done to be able to add urine to this dehydration medium in the next experimental run again because too much pooling would have prohibited further addition of urine. Priority was also given to never have all triplicates of one dehydration medium on the same oven rack.

4.2.7. Mixing of dehydration media during the experiment

After the first and the second dehydration runs, the dehydration media were manually mixed with a spatula within their own Petri dish before urine was added for the next dehydration run. After discussion with Vinnerås, it was decided to stop mixing the dehydration media after each dehydration run, to limit the time the samples are out of the dehydration setup and to avoid any material loss by spilling of material during the mixing process.

4.2.8. Storage of the dehydration media between dehydration runs

Due to the duration of the dehydration runs, the experiment lasted from 26.06.2017 to 20.07.2017. Between the dehydration runs, the samples were stored with a Petri dish lid, at ambient temperature in the laboratory, which was 21 °C (± 2 °C). The samples were stored following the dehydration runs 1–7, 9, 10, 12, 14, 16, 19, 27, 30 and 31.

4.3. Physicochemical analysis

4.3.1. Procedure

Each working day, after all dehydration runs were performed, a sample of 10 mL of the mixed urine that was used for the dehydration runs, was collected and stored at -20 °C for further analysis. This urine was then analysed for electrical conductivity (EC), pH, volatile solids (VS), NH_4-N, total solids (TS), Tot-P and Tot-N.

4.3.2. pH and EC

The pH of the urine and the dehydration media was measured with a pH-meter (PHM210, RADIOMETER ANALYTICAL S.A., France) and a pH-probe (Red Rod Combined pH electrode, RADIOMETER ANALYTICAL S.A., France). An EC-meter (Cond 340i, WTW, Germany) and an EC-probe (TetraCon 325, WTW, Germany) were used to measure electrical conductivity. The dehydration media's pH and EC were measured twice in total, once at the beginning of the experiment and once at the end of the experiment. The urine pH and EC were measured for each batch of urine after it was mixed in a sterile, 5 L Florence flask (see Materials and Methods).

For the preparation of the dehydration media samples, 5 g of each dehydration medium was mixed with 25 mL of urine in conical 50 mL polypropylene Falcon™ centrifuge tubes. It was mixed using an analogous vortex mixer (VWR, USA) and then left to rest for one hour. Then the pH and the EC of the supernatant was measured. Urine was used instead of distilled water to measure the pH and EC because it was of interest for this experiment, which pH and EC the dehydration media reach when they react with the urine.

4.3.3. Mass measurements

For measuring the mass during this study, two different scales have been used. For the measurement of mass during total solids and volatile solids determination of the urine, a digital scale with four decimal places of accuracy (OHAUS Corporation, USA) was used. A digital scale with two decimal places of accuracy (OHAUS Corporation, USA) was used for all other measurements.

4.3.4. Total solids (TS) and adjustment for loss of urea

The total solids of the dehydration media were measured by subjecting them to a temperature of approximately 110 °C for 14 h. When setting the oven to 110 °C, the analogous temperature control of the benchtop oven sometimes showed temperatures of up to 120 °C. Since the temperature was not measured in any other way, the temperature is given as an approximation. If the analogous measurement showed higher readings than 110 °C, the temperature was adjusted back to 110 °C as soon as it was noticed.

To account for the urea-loss due to heat, the urine-TS were adjusted by calculating the urea concentration in the urine.

The NH_4-N concentration in urine was 1.7 g L_{urine}^{-1} and the concentration of Tot-N 5.73 g L_{urine}^{-1}. Assuming that NH_4-N and urea-N are the only forms of N in urine, NH_4-N accounts for approximately 30 % of Tot-N, and urea-N accounts for the remaining 70 % of N, therefore equating to 4.03 g L_{urine}^{-1}.

4.3.5. Volatile solids (VS)

The samples and the urine that were heated for TS-determination were then subjected to 550 °C for a duration of 6 h in a muffle oven to determine the volatile solids content of the dehydration medium and the urine.

4.3.6. Nitrogen, Phosphorus, Potassium and Carbon

The initial P-, and K-content of the dehydration media was calculated using results of the analysis of biochar and wheat bran which were analysed using emission spectrophotometry with inductively coupled plasma (ICP) (Optima 7300 DV Coupled Plasma Optical Emission Spectrophotometry (ICP-OES), PerkinElmer Inc., USA). It was assumed that $Ca(OH)_2$ and MgO did not contain any P or K. Therefore, pure $Ca(OH)_2$ and pure MgO were not analysed for their P- or K-content.

The dehydration media's final N- and C-content was measured using Dumas dry combustion method (LECO Corporation, USA). It was assumed that $Ca(OH)_2$ and MgO do not add a significant amount of N or C to the dehydration media. Therefore, pure $Ca(OH)_2$ and pure MgO were not analysed for their N- or C-content.

After the experiment, samples of all dehydration media were analysed for their final P- or K-content using emission spectrophotometry with inductively coupled plasma ICP (Avio 200 ICP Optical Emission Spectrometer, PerkinElmer Inc., USA).

The Tot-N of the urine was analysed using "Spectroquant Crack Set 20" (Merck KGaA, Germany) and "Spectroquant Nitrate test NO_3^- test kit" (Merck KGaA, Germany). The test kit has a measuring range of 1–25 mg L^{-1} NO_3-N. The urine was diluted 1000-fold with deionised water for the analysis.

The NH_4-N-content of the urine was analysed using "Spectroquant Ammonium test" (Merck KGaA, Germany). The test has a measuring range of 5–150 mg L^{-1} NH_4-N. The urine was diluted 100-fold with deionised water for the analysis.

The P-content of the urine was analysed using "Spectroquant Crack Set 10" (Merck KGaA, Germany) and "Spectroquant Phosphate test" (Merck KGaA, Germany). The test kit has a measuring range of 0.05–5 mg/L PO_4-P. The urine was diluted 1000-fold with deionised water for the analysis.

After preparation with the test kits, a photometer (NOVA 60 A Spectroquant®, Merck KGaA, Germany) was used to take N and P readings.

4.3.7. Calculations

The dehydration media were evaluated for their effectiveness to reduce urine mass (on wet basis). For evaluating the mass reduction, we used Equation 4, for the mass concentration factor Equation 5 and for the average dehydration rate Equation 6. The mass of the dehydration medium at the beginning of the experiment was labelled as m_{media}, the total mass of urine added to in the experiment m_{urine}, and

the mass of the product at the end $m_{end-product}$. The surface area in m² was labelled A, dehydration time was labelled t, the Petri dish weight after the addition of urine to the dehydration medium was labelled w_i, the Petri dish weight after the dehydration was labelled w_{i+1} and the number of dehydration runs was labelled n.

Equation 4. Mass reduction (% wet basis)

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

Equation 5. Mass concentration factor

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

Equation 6. Average dehydration rate

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

To estimate the N-recovery, a mass balance was carried out on a Tot-N basis. The mass balance was compared against the potential urea-recovery at 48.1 °C (±1.5 °C), the average temperature inside the dehydration setup. Using an equation by Simha et al. (2020b), a urea half-life of 133 days was calculated. The estimation for theoretical urea-N-recovery was based on the urea-N content, which was estimated to be 70 % of Tot-N in the input-urine. This was done by assuming that NH_4-N and urea-N are the only forms of N in the input-urine and since the concentration of NH_4-N was 1.7 g L_{urine}^{-1}, so about 30 % of the concentration of Tot-N 5.73 g L_{urine}^{-1}.

4.3.8. Statistical analysis

Normality of the data was tested using a Shapiro-Wilk test, and the equal variance between the different dehydration media data was verified using the Brown-Forsythe-Levene test. We performed an analysis of variance (ANOVA) followed by a Tukey's Honest Significant Difference Test (HSD) to reveal significant differences ($p = 0.05$) between the dehydration media. For comparison of the dehydration media's physicochemical properties at the beginning and at the end of the experiment, unpaired two-sample Student's t-tests were executed. The software used for all statistical analyses was RStudio version 1.2.5042 and R version 4.0.0 (RStudio Team 2016), with R-packages "agricolae" by de Mendiburu (2020) and "car" by Fox & Weisberg (2019).

4.3.9. Exclusion of Outlier

One of the triplicates of MgO-Char had to be excluded from the calculations because it was categorised as an outlier after discussion with Vinnerås. This was concluded after reviewing the results of the analysis of the N-content which were determined by burning in a "TruMac"-furnace. Two of the triplicates showed a very similar nitrogen content of 4.12 % and 4.39 %, while the third sample showed 0.71 %. The sample was analysed again, and the second analysis confirmed the significantly lower nitrogen content. This was the only case where an outlier was detected, and the only case where a triplicate was excluded from further calculations. It is believed that this outlier was due to contamination of the sample with urease, which was incompletely inactivated because this triplicate showed the lowest final pH (pH 9.71) of all dehydration media.

4.3.10. Handling of errors

Handling of the few errors that happened during the experiments:

1. Dehydration time too long or too short: Time was added to or deducted from the next experimental run. All dehydration media had the same total dehydration time.
2. If too much urine was added (40 mL instead of 30 mL), which happened once for one of the triplicates of MgO, in the next run, less urine (20 mL instead of 30 mL) was added. All dehydration media had the same total amount of urine added to them.

5. Results

5.1. Mass balance

MgO

Input			Input				Output			Output	
Urine in (g)			**Medium in (g)**				**Product out (g)**			**Gases out (g)**	
Mass	1162.00		Mass	30.00			Mass	84.60		Mass	1104.90
N	6.36		N	0.00			N	4.25		N	2.11
P	0.68	+	P	0.00	→	=	P	0.32	+	P	-
K	2.12		K	0.00			K	2.12		K	-
TS	13.50		TS	30.00			TS	55.90		TS	-
TSadj	23.50		VS	-			VS	65.00		TSadj	-

MgO-Char

Input			Input				Output			Output	
Urine in (g)			**Medium in (g)**				**Product out (g)**			**Gases out (g)**	
Mass	1160.10		Mass	30.30			Mass	113.00		Mass	1073.40
N	6.36		N	0.02			N	4.80		N	1.58
P	0.68	+	P	0.00	→	=	P	0.38	+	P	-
K	2.45		K	0.03			K	2.48		K	-
TS	13.50		TS	30.10			TS	54.60		TS	-
TSadj	23.50		VS	-			VS	64.90		TSadj	-

MgO-Char-Lime

Input			Input				Output			Output	
Urine in (g)			**Medium in (g)**				**Product out (g)**			**Gases out (g)**	
Mass	1160.40		Mass	30.30			Mass	112.60		Mass	1072.10
N	6.36		N	0.02			N	4.74		N	1.64
P	0.68	+	P	0.00	→	=	P	0.37	+	P	-
K	2.34		K	0.03			K	2.37		K	-
TS	13.50		TS	30.00			TS	54.00		TS	-
TSadj	23.50		VS	-			VS	64.20		TSadj	-

MgO-Char-Bran

Input			Input				Output			Output	
Urine in (g)			**Medium in (g)**				**Product out (g)**			**Gases out (g)**	
Mass	1159.00		Mass	30.10			Mass	111.90		Mass	1072.30
N	6.36		N	0.30			N	5.07		N	1.60
P	0.68	+	P	0.11	→	=	P	0.46	+	P	-
K	2.34		K	0.15			K	2.49		K	-
TS	13.50		TS	29.00			TS	54.10		TS	-
TSadj	23.50		VS	-			VS	64.90		TSadj	-

MgO-Char-Bran-Lime

Input			Input				Output			Output	
Urine in (g)			**Medium in (g)**				**Product out (g)**			**Gases out (g)**	
Mass	1158.50		Mass	30.20			Mass	113.00		Mass	1070.80
N	6.36		N	0.27			N	4.94		N	1.69
P	0.68	+	P	0.10	→	=	P	0.43	+	P	-
K	2.37		K	0.13			K	2.51		K	-
TS	13.50		TS	29.10			TS	52.90		TS	-
TSadj	23.50		VS	-			VS	63.50		TSadj	-

Figure 10. Mass balance of all dehydration media, displaying average values, n = 3 (only for MgO-Char n = 2, as one triplicate was excluded from the calculations since it was identified as an outlier)

Mass balances were carried out for all dehydration media (Figure 10). All dehydration media managed to reduce the mass of urine by > 90 %, with pure MgO being significantly more effective ($p > 0.001$) and weighing around 25 % less than the other products at the end of the experiment. When MgO was mixed with other substrates, the mass concentration factor reduced (Table 3). There were no significant differences between the average dehydration rates of the different dehydration media ($p > 0.05$), which varied from 19.1 kg m^{-2} d^{-1} (± 4.3) to 19.7 kg m^{-2} d^{-1} (± 4.5) for MgO. The average dehydration rate of each dehydration medium (Table 3) varied over time due to material properties as well as the position of the dehydration medium on the oven racks.

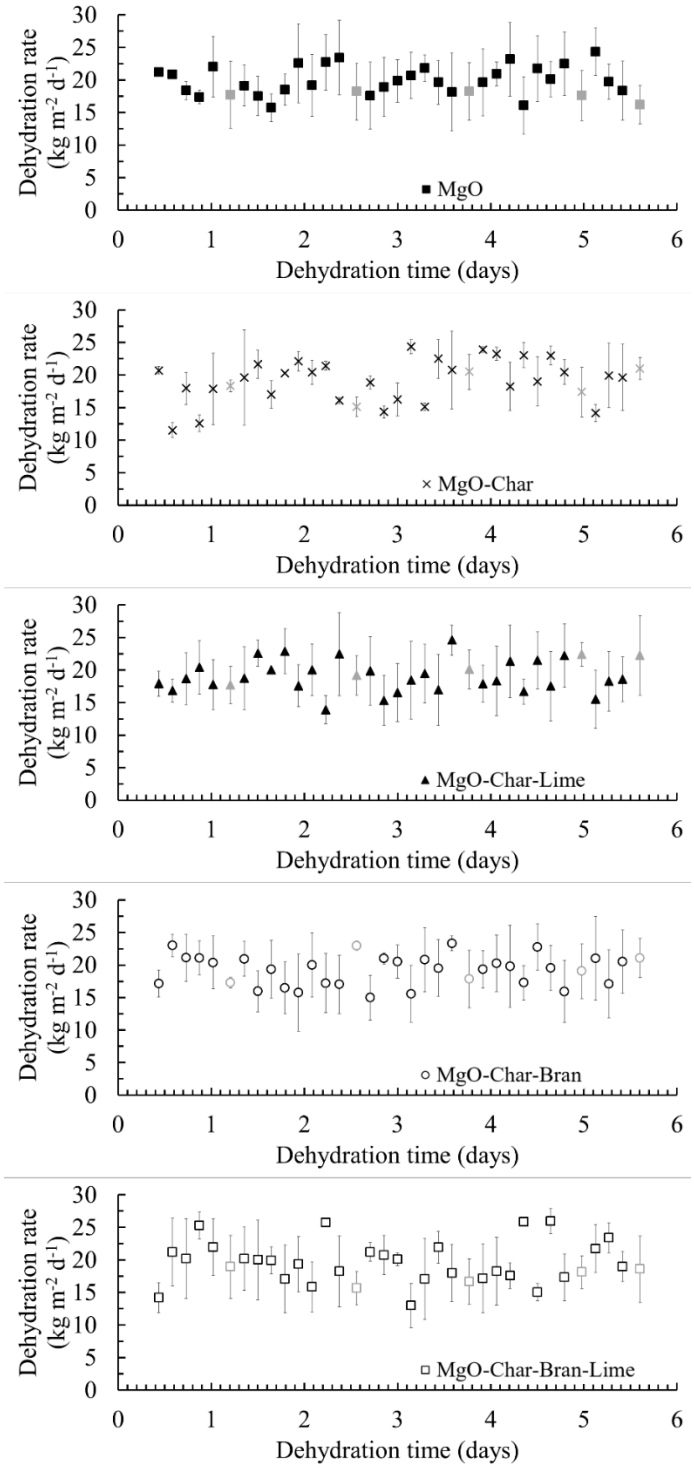

Figure 11. Dehydration rate of the dehydration media in kg m^{-2} d^{-1} over the course of the total dehydration time of 5.6 days. n = 3 (only for MgO-Char n = 2, as one triplicate was excluded from the calculations since it was identified as an outlier); the first experimental run lasted 10.5 h (0.44 d) (90 mL urine addition), "regular" dehydration runs lasted 3.5 h (0.15 d) (30 mL urine addition), and "dry runs", which were done to avoid excess accumulation of urine, lasted 4.5 h (0.19 d) (indicated by grey symbols)

N-recovery was estimated by performing the mass balances (Figure 10) and showed that pure MgO had the lowest N-recovery of about 67 %. The highest N-recovery was achieved in the dehydration medium that consisted of a mixture of MgO with biochar and wheat bran (for media composition see Table 2). When $Ca(OH)_2$ was part of the media, the N-recovery decreased compared to the other dehydration media mixtures (Table 3), although the difference was not always statistically significant.

Table 3. Means and standard deviation of mass reduction on wet basis ($mass.red_{WB}$), mass concentration factor on wet basis ($mass.cf_{WB}$), dehydration rate on wet basis ($dry.rate_{WB}$) and N-recovery from urine for all dehydration media; n = 3, significant difference is illustrated by different superscript letters within the same row (α = 0.05)

Properties	MgO	MgO-Char[a]	MgO-Char-Lime	MgO-Char-Bran	MgO-Char-Bran-Lime
mass.red$_{WB}$ (%)	92.9 (0.1)^{A}	90.5 (0.3)^{B}	90.5 (0.2)^{B}	90.6 (0.5)^{B}	90.5 (0.4)^{B}
mass.cf$_{WB}$	14.1 (0.2)^{A}	10.6 (0.4)^{B}	10.6 (0.2)^{B}	10.7 (0.6)^{B}	10.5 (0.6)^{B}
dry.rate$_{WB}$ (kg d^{-1} m^{-2})	19.7 (4.5)	19.1 (4.3)	19.2 (4.7)	19.2 (4.6)	19.5 (5.0)
N-recovery (%)	66.8 (1.2)^{D}	75.5 (0.4)^{BC}	74.5 (0.9)^{C}	79.7 (1.1)^{A}	77.6 (0.7)^{AB}

[a] n = 2, because one triplicate was identified as an outlier and excluded from the calculation

The dehydration process (at approximately pH 10 and 50 °C) resulted in a loss of > 20 % nitrogen. About 70 % of the Tot-N in urine was assumed to be present as urea-N (confer Materials and Methods), so according to Warner (1942), under the prevailing conditions during dehydration, chemical urea hydrolysis should only account for 1.2 % of urea loss, which means that urea was also lost due to other factors. All media besides pure MgO managed to recover more than 74 % of the input Tot-N, so the other media did most probably recover around 15–30 % of the ammonium.

5.1.1. Elemental composition

All elemental compositions were calculated on TS-basis (see Table 4). Using pure MgO as a dehydration medium, resulted in an NPK-content of 6.5 % (N), 0.5 % (P) and 3.3 % (K). All other dehydration media, where MgO was mixed with other substances (composition see Table 2), achieved a higher NPK-content at the end of the experiment. The dehydration media with the highest NPK-content were MgO-Char-Bran and MgO-Char-Bran-Lime, both of which achieved on average about 7.8 % (N), 0.7 % (P), 3.9 % (K), their NPK-content not significantly being influenced by calcium hydroxide ($p < 0.05$). The final C-content of the dehydration media was lowest in MgO with 6.8 % and highest in MgO-Char with 30.9 %.

Table 4. Mean elemental composition displayed in % of total solids of the dehydration media at the beginning and end of the experiment, n = 3, standard deviation in parentheses; within rows, same superscript letters illustrate no significant difference (α = 0.05)

Properties	MgO	MgO-Char[a]	MgO-Char-Lime	MgO-Char-Bran	MgO-Char-Bran-Lime
N					
initial	0.0 (0.00)^{D}	0.07 (0.01)^{C}	0.07 (0.01)^{C}	1.01 (0.01)^{A}	0.9 (0.00)^{B}
final	6.5 (0.12)^{C}	7.4 (0.04)^{B}	7.4 (0.09)^{B}	7.8 (0.11)^{A}	7.8 (0.07)^{A}
P					
initial	0.0 (0.00)^{C}	0.01 (0.00)^{C}	0.01 (0.00)^{C}	0.38 (0.01)^{A}	0.3 (0.01)^{B}
final	0.5 (0.03)^{C}	0.6 (0.00)^{B}	0.6 (0.02)^{B}	0.7 (0.02)^{A}	0.7 (0.01)^{A}
K					
initial	0.0 (0.00)^{D}	0.09 (0.01)^{C}	0.08 (0.01)^{C}	0.49 (0.02)^{A}	0.4 (0.01)^{B}
final	3.3 (0.22)^{B}	3.8 (0.01)^{A}	3.7 (0.09)^{AB}	3.8 (0.21)^{A}	3.9 (0.23)^{A}
C					
initial	0.0 (0.00)^{E}	54.2 (1.75)^{A}	48.2 (1.56)^{B}	43.5 (0.88)^{C}	38.6 (0.78)^{D}
final	6.8 (0.04)^{D}	30.9 (0.4)^{A}	29.8 (0.7)^{A}	27.7 (0.8)^{B}	24.8 (0.3)^{C}

[a] n = 2, because one triplicate was identified as an outlier and excluded from the calculation

5.1.2. pH and EC

The initial pH of all dehydration media was ≥ 9.8, with media with only MgO as an alkalising substance having a pH of about 10, reflecting the saturation-pH of $MgO/Mg(OH)_2$ in urine established by Simha et al. (in preparation). Randall et al. (2016) established a saturation-pH of $Ca(OH)_2$ in urine of pH 12.5, which is well reflected by both media containing $Ca(OH)_2$, having an initial pH of ≥12.6. There was no significant difference in the final pH of the dehydration media ($p > 0.05$), with all media dropping to a pH of about pH 10. The urine that was added to the dehydration media had an average EC of 11.7 mS cm^{-1} (± 1.2) and increased the final EC of all dehydration media (besides pure MgO) to around 25 mS cm^{-1}.

Table 5. Initial and final mean pH and EC of the dehydration media measured in 1:5 (w/v) media:urine suspension at 21 °C (± 2 °C); n = 3, standard deviation in parentheses; within rows, same superscript letters indicate no significant difference (α = 0.05)

Properties	MgO	MgO-Char[c]	MgO-Char-Lime	MgO-Char-Bran	MgO-Char-Bran-Lime
pH					
initial[a]	10.3 (0.0)^{C}	10.2 (0.1)^{C}	12.7 (0.0)^{A}	9.8 (0.0)^{D}	12.6 (0.0)^{B}
final[b]	10.1 (0.0)^{A}	9.9 (0.0)^{A}	10.0 (0.1)^{A}	9.9 (0.0)^{A}	10.0 (0.3)^{A}
EC (mS cm^{-1})					
initial[a]	9.2 (0.0)^{AB}	8.7 (0.0)^{AB}	12.8 (3.9)^{A}	6.6 (1.2)^{B}	13.1 (2.9)^{A}
final[b]	29.7 (0.6)^{A}	25.4 (0.5)^{B}	25.2 (0.3)^{B}	25.8 (0.9)^{B}	25.4 (0.5)^{B}

[a] the urine used for the analysis of the initial pH and EC of the dehydration media had a pH of 6.7 and an EC of 9.4 mS cm^{-1}

[b] the urine used for the analysis of the final pH and EC of the dehydration media had a pH of 6.9 and an EC of 10.1 mS cm^{-1}

[c] n = 2, because one triplicate was identified as an outlier and excluded from the calculation

5.2. Dehydration temperature

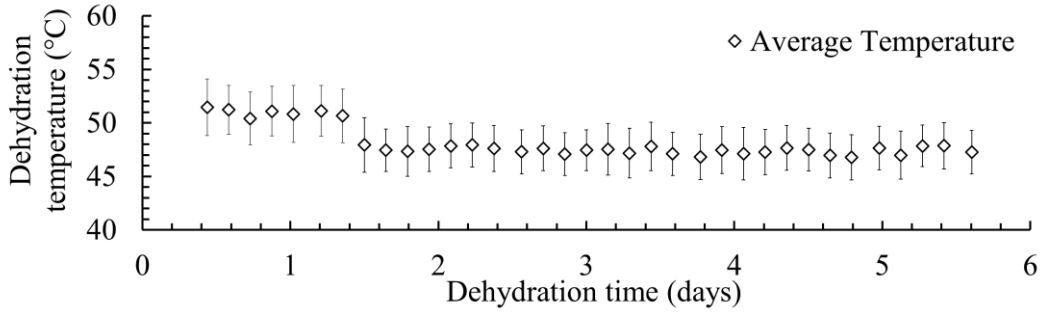

Figure 12. Average temperature and standard deviation (°C) inside of the dehydration setup, measured at three different positions (see Figure 8, number 4)

A drop in average dehydration temperature from 51.08 °C (SD 2.53 °C) to 47.42 °C (SD 2.16 °C) after 1.35 days of dehydration time for the rest of the remaining dehydration time was recorded (see Figure 12). The change in dehydration temperature can be attributed to the increase in fan speed due to the change in voltage from 7.5 V to 9 V. The voltage was increased because pooling of urine could be seen on the dehydration media at the end of the dehydration runs. As described in Materials and Methods, the lower voltage of 7.5 V was initially chosen to avoid displacing biochar out of the Petri dishes by too high air velocity.

6. Discussion

6.1. Ammonia concentration in the input-urine

Thirty per cent of the total nitrogen in the input-urine was in the form of ammonia, which is considerably higher than the ammonia concentration of < 5 % that is to be expected in freshly excreted urine. The high ammonia concentration in the input-urine shows that the urea in the input-urine partially hydrolysed during collection and storage before its application to the dehydration media, although utmost care was taken during urine collection and storage. This means that urease might have been introduced to the dehydration media through the urine, hydrolysing a part of the urea that was added throughout the experiment. Further N-losses were probably counteracted by the ability of MgO to form struvite, magnesium ammonium phosphate, ($NH_4MgPO_4 \cdot 6H_2O$) with NH_4^+ in the urine. This property distinguishes MgO from other alkalising media like $Ca(OH)_2$ or wood ash.

6.2. Nutrient recovery

This study aimed to test magnesium oxide's suitability to recover nutrients from human urine by alkaline urine dehydration. Earlier studies that looked into alkaline urine dehydration managed to recover up to 90 % of input urine-N in wood ash (Senecal & Vinnerås 2017) but assumed the urea-N in the input-urine to be 5 % based on Udert et al. (2006). Using only $Ca(OH)_2$ as a dehydration medium, Simha et al. (2020a) managed to achieve an N-recovery of about 78 % at 50 °C. However, for this study, fresh urine was used, and it was assumed that the urine was unhydrolysed. In contrast to these previous studies, using MgO as a sole alkalising and dehydration medium only achieved an N-recovery of about 67 %. In the aforementioned study by (Senecal & Vinnerås 2017) the urea-concentration in the input-urine was estimated to be 95 % of Tot-N, and the recovery of input-urine Tot-N was 90 %, so wood ash managed to recover 95 % of the urea-N. In our study, because the urea-concentration in the input-urine was estimated to be 70 % of Tot-N, and the recovery of Tot-N was 67 %, pure MgO managed to recover 96 % of the

urea from the input-urine. This shows that pure MgO at 50 °C can achieve urea-N-recovery rates comparable to wood ash at 30 °C. Adding co-substrates to the dehydration medium increased the N-recovery to > 74 %, which shows that, compared to pure MgO, the other dehydration media used during this study were also capable of recovering NH_4-N, which is further discussed under "The effects of adding co-substrates".

Regarding the recovery of Tot-N, three factors might help to explain the lower N-retention experienced during this study compared to other studies: the inverse solubility of MgO in urine, the partially hydrolysed input-urine and ammonia-stripping.

6.3. MgO solubility

The solubility and the pH of MgO in urine are temperature-dependent, meaning that the solubility of MgO decreases as the temperature increases (Rocha et al. 2004). The final pH of all media was about pH 10, regardless of whether only MgO or MgO combined with $Ca(OH)_2$ was used as alkalising agent(s). The inverse solubility of MgO and its temperature-dependent pH means that the pH of the media that only used MgO as an alkalising agent potentially dropped to around pH 8.8 at 50 °C during the experiment (Simha et al. in preparation). As Geinzer (2017) showed, urease gets deactivated if the pH increases to > 10, but it can become reactivated if the pH falls below pH 10 again. As the chemical urea degradation under the prevailing conditions was calculated to only account for around 1.2 % urea-loss, the observed nitrogen loss may be accredited to enzymatic urea hydrolysis by urease that seems to have occurred during the experiment. Since the N-recovery was 67 %, when pure MgO was used as dehydrating medium, and only 70 % of Tot-N was in the form of urea enzymatic ureolysis seems to have been at least partially inhibited. Further studies, perhaps with urine with very little or no NH_4-N, are needed to establish a clear understanding of the activity of urease at the given treatment conditions.

6.4. Ammonia stripping

The high nitrogen losses that occurred during this experiment might be explained by ammonia stripping (Başakçilardan-Kabakci et al. 2007). Ammonia-nitrogen exists as both NH_4^+ as well as free ammonia/dissolved ammonia gas in urine. The equilibrium depends on the pH, the temperature and the ionic strength of the urine. In freshly excreted, untreated urine with a pH < 7, NH_3-N is nearly exclusively present in the form of ammonium (NH_4^+). When the temperature and pH increase and urine is concentrated during dehydration, the equilibrium shifts towards

ammonia (NH_3). This means that at 50 °C and pH >10, more than 80 % of the NH_3-N is in the form of ammonia (NH_3). The surface area available for dehydration was the same for all dehydration media (100 cm^2). As ammonia has a Henry's constant of 62 mol L^{-1} atm^{-1} in water solution (Larsen et al. 2013), the partial pressure of ammonia in the layer of air on the surface of the urine strongly influences ammonia evaporation. This means that high nitrogen losses in this experiment could have resulted from the interfacial transfer of gaseous ammonia due to ventilation.

6.5. The effects of adding co-substrates

Adding other substrates like wheat bran, biochar or calcium hydroxide to MgO, increased the N-retention to > 74 % (Table 3). This was possibly due to pooling of urine on top of the substrate, which might increase the transfer of gaseous NH_3 to the air, was less frequent when MgO was mixed with co-substrates. Because MgO has a higher density than urine (3.58 g m^{-3} versus approximately 1.05 g cm^{-3}), and because of the low solubility of MgO in urine, there was significant pooling visible on top of MgO after each application of urine, as MgO settled on the bottom of the Petri dish. When co-substrates were added to pure MgO, less pooling of urine could be seen on top of the substrate, as wheat bran and biochar have a high water holding capacity. The dehydration process sometimes made the particles reversibly hydrophobic though, meaning that pooling was sometimes visible during the first few minutes after urine application. Adding wheat bran and biochar seems to have helped break up the lipid layer that forms on the surface of urine during dehydration. The co-substrates could have restricted the diffusion of aqueous ammonia, thereby lowering its transfer to the gaseous state, and increasing NH_4-N recovery.

If the amount of MgO was decreased and $Ca(OH)_2$ was added as an additional alkalising agent, as in MgO-Char-Lime and MgO-Char-Bran-Lime, the initial pH increased to > 12.5. The N-recovery of these two media decreased compared to the media with only MgO as an alkalising agent, although the differences were not significant ($p < 0.05$). The lower N-recovery might be attributed to a higher rate of chemical urea hydrolysis in the dehydration media containing $Ca(OH)_2$, as urea half-life decreases to a few days at a pH > 12, or the decreased ability of the mixture with $Ca(OH)_2$ to form struvite with ammonia that was present in the urine.

6.6. Fan speed

Trial runs showed that untreated biochar gets displaced from the Petri dishes if the fan speed is too high. Therefore, the fan speed at the beginning of the experiment was adjusted to a speed where no biochar was displaced. After seven dehydration runs, partial crust formation and solidification of the dehydration media were

observed, making the dehydration media less susceptible to changes in air velocity. The fan speed was then increased, which lowered the temperature in the dehydration system from 51.08 °C (SD 2.53 °C) to 47.42 °C (SD 2.16 °C). This change in temperature probably resulted from the higher air exchange with the air surrounding the dehydration setup (through the oven door gap) due to higher turbulence within the dehydration setup. Following the change in air velocity, less pooling was observed on top of the dehydration media, but this did not significantly affect the dehydration rate (Figure 11 and Figure 12).

6.7. Mass balance

The mass balances, which were carried out for all dehydration media (Figure 10), showed that the gaseous N-losses were highest (2.11 g) when pure MgO was used as a dehydration medium, and lowest when MgO was mixed with biochar (1.58 g). This might be because the co-substrates that were added to the other dehydration media hinder the diffusion of $NH_{3(aq)}$, as discussed earlier. The highest N-content in the products was measured in the dehydration media that contained wheat bran, with MgO-Char-Bran containing 5.07 g and MgO-Char-Bran-Lime with 4.94 g. Wheat bran contains nitrogen, thereby introducing some N to the dehydration medium at the start of the experiment. The mixture of wheat bran, biochar and MgO reached the highest N-content of any dehydration medium (5.07 g). When calcium hydroxide was added to the mix (in MgO-Char-Bran-Lime), the initial and final N-content decreased. This was probably because the wheat bran content had to be lowered in comparison to MgO-Char-Bran, to allow for the addition of calcium hydroxide (Table 2), and because calcium hydroxide caused a significantly higher pH at the start of the experiment than just MgO (pH 12.6, confer Table 5), which might have increased both gaseous losses of ammonia and chemical urea degradation. The highest P-content in any product could be observed in MgO-Char-Bran (0.46 g), and the lowest in pure MgO (0.32 g), highlighting the added value that wheat bran can bring to the product, as it already contains P. The highest K-content with 2.49 g can be observed in MgO-Char-Bran while using MgO as the sole dehydration medium results in the lowest K-value of 2.12 g. As the mass balance shows, this can again be attributed to the dehydration medium's wheat bran content, adding 0.15 g K to the dehydration medium at the start. All dehydration media had 30 g at the start of the experiment and had about 1160 g of urine added to them. However, only four out of five dehydration media reached a comparably similar end weight of around 113 g, with MgO ending up at a significantly lower 84.6 g. This is reflected in the higher mass of gases that left the system when only MgO was used as a dehydration medium (1104.9 g) and the higher mass reduction and mass concentration factor (Table 3) in comparison to the other dehydration media.

6.8. Application scenario

Magnesium oxide, as the sole dehydration medium showed an average dehydration rate of 19.7 kg d^{-1} m^{-2}. According to Vinnerås et al. (2006), each person excretes around 550 kg of urine per year, which means that for a family of four around 2200 kg of urine per year should be accounted for. If the aim were to dehydrate 6 kg of urine daily for a family of four, 1 m^2 of dehydration medium would provide more than enough surface to dehydrate all the urine. During this experiment, we dehydrated 1.16 kg of urine in 30 g of dehydration medium. Given that the dehydration medium's final pH at the end of the experiment did not fall below pH 10, more urine could potentially have been dehydrated in the medium until the pH falls below 10 and more alkalising medium needs to be added, or the dehydration medium exchanged. This would probably even have increased the fertiliser value of the product. Our experiments showed that approximately 37 kg of urine could be dehydrated per kg dehydration medium. This means that less than 60 kg of dehydration medium would be enough to dehydrate all the urine of a family of four excreted over a year. With a bulk price of 0.3 USD kg^{-1} (Bray & Ghalayin 2020), the costs for one year worth of magnesium oxide as sole dehydration medium for a family of four would roughly amount to 18 USD. The dry fertilizer could be collected through the already existing solid waste collection system, thereby minimizing additional costs for logistics of collection and transport. The fertiliser could then further be processed in a central facility, to, for instance, produce pellets out of the dry fertiliser powder. Having the fertiliser in the form of pellets will make it possible to use existing fertiliser application equipment and infrastructure in agriculture. In countries where a large part of the population might still have land plots for agricultural production available to themselves, the dry fertiliser could be applied to the field directly after the dehydration process is finished. This would eliminate collection and transport of the fertiliser product and the added yield due to the fertiliser and well as possibly selling excess fertiliser could potentially create an additional source of income for the people.

6.9. Fertiliser mass and nutrient concentration

A family of four produces around 2200 L of urine per year. If the family used pure MgO as a dehydration medium, the total mass of fertiliser acquired by the end of the year would amount to 86.8 kg (based on the urine density and TS-values measured during this experiment). Based on the NPK-values of the fertiliser acquired using pure MgO as a dehydration medium during the study (6.5 % N, 0.5 % P and 3.3 % K based on TS-values), a family of four could, within a year, produce a fertiliser that would contain around 5.6 kg N, 0.4 kg P and 2.9 kg K, which could then be used as a sustainable fertiliser for agricultural crops.

6.10. Outlook

This study showed that it is possible to recover up to 80 % N of a partially hydrolysed urine (70 % of Tot-N was urea) in a dehydration medium consisting of magnesium oxide, biochar and wheat bran at a dehydration temperature of 50 °C. These results and previous studies by our group suggest that if conditions would further be optimised for achieving low ammonia losses, and by using unhydrolysed urine as a source, N-recovery could be significantly improved. For instance, this could be achieved by utilising lower dehydration temperatures of < 40 °C and direct excretion of fresh urine onto the substrate. Utilising wheat bran and biochar as co-substrates showed potential to break up the lipid film that would otherwise build up on pooling urine on the dehydration medium's surface, which beneficially influenced the N-recovery. Since biochar is already used as a soil amendment (confer “Terra Preta”), and wheat bran adds additional N, P and K to the final fertiliser, the addition of both co-substrates holds the potential to further increase the value the fertiliser provides for agricultural use.

6.11. Recommendations

The author suggests further investigation into the potential of a combination of MgO, wheat bran and biochar as dehydration medium, at dehydration temperatures lower than 40 °C. This takes into consideration the value biochar, and wheat bran add to the product, as well as the solubility of MgO in urine, which decreases at higher temperatures, which in turn decreases the urine pH. The availability of magnesium, being the world’s 8th most abundant element (United States Geological Survey) is essentially unlimited, eliminating any concerns about future shortages in supply. Using MgO instead of $Ca(OH)_2$ provides the benefit of lower solubility of MgO, making less frequent changing intervals of the dehydration medium possible. In comparison to $Ca(OH)_2$, magnesium oxide also provides the added benefit of eliminating the Mg-deficiency as a limiting factor for struvite precipitation, therefore bearing the potential for precipitation of NH_4^+ in the urine in the form of struvite. The lower saturation-pH of MgO compared to $Ca(OH)_2$ in urine also minimises N-losses by chemical urea degradation that might occur at a pH >12. Using lower dehydration temperatures and further optimising the dehydration setup might increase the N-retention and increase the product's fertiliser value.

6.12. Limitations and Disclaimer

In a parallel study done by Prithvi Simha, Cecilia Lalander, Annika Nordin and Björn Vinnerås from SLU (unpublished results) it was discovered that during the

mixing of the dehydration media that was done at the end of the experiment to be able to take representative samples of the dehydration media, a certain amount of water that initially remained in the sample, was lost. This was due to the low humidity level (approximately 20 % humidity) in the air, the frequent air exchange in the laboratory and the long mixing and storage time (about 1 to 2 days).

As described in Materials and Methods, biochar gets displaced from the Petri Dishes quite easily at the beginning of the experiment, due to its electrostatic behaviour and the high air velocity in the dehydration setup. To avoid this, in this experiment, a higher loading rate of urine was chosen for the first urine addition (90 mL instead of 30 mL). In future studies, it should be evaluated, if compaction of the biochar to a more solid form (e.g. pellets that fall apart when coming into contact with a liquid), could solve the problem of displacement of the biochar.

In this study, the temperature within the media was not measured. In future studies, it would be interesting to look closer at the temperature within the media, and how this affects the dehydration. This is especially interesting, if the depth of the material is increased or if the samples, like in this study, are heated intermittently. In this study, the dehydration media had a maximum depth of about 20 mm at the end of the experiment and were removed from the oven frequently to either add fresh urine for the next experimental run or to store them until the next dehydration run. When the samples were stored, it could be assumed that they cooled down to room temperature, which was approximately 21 °C (± 2 °C).

The greatest difficulty the study setup presented, was the accumulation of urine over time, which leads to the Petri dishes becoming full. This entailed the risk of spillage of urine and dehydration media. For this reason, if the same experimental setup is chosen in future studies, Petri dishes with higher sidewalls would be beneficial. These higher sidewalls could change the drying pattern by restricting the airflow over the dehydration media though.

Data acquired during this thesis will be used in a publication which the author of this thesis will be co-authoring.

7. Conclusions

This study's objective was to investigate the use of MgO as an alkalising agent and sole dehydration medium for alkaline urine dehydration. Our results showed that pure MgO recovered 66.8 % (SD 1.2) of nitrogen from partially hydrolysed urine (30 % of Tot-N being NH_4-N). When MgO was mixed with biochar and wheat bran, an N-retention of 79.7 % (SD 1.1) was achieved. The temperature dependency of the pH and the solubility of MgO showed a risk of reactivation of urease, which increases N-losses, as urea gets hydrolysed. All dehydration media that contained MgO had an initial and final pH of around 10, which suggests increased suitability of MgO as a dehydration medium at lower temperatures (< 40 °C), where the temperature dependency of the pH and solubility is not as prominent as in these experiments. The experiments showed that if partially hydrolysed urine is dehydrated, stripping of ammonia can increase N-losses. The products obtained by alkaline urine dehydration during this study had high NPK values, which makes them suitable as crop fertilisers.

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I want to dedicate my thesis to my grandparents Maria and Manfred and our friend Sigrid. They did not live to see this thesis to be finished but would have been delighted to see it in its entirety.

9. Appendix

Table 6. Materials and equipment, their producers, model and article numbers

Product	Identification	Producer	Additional information
Benchtop oven for dehydration system	Model number 2177 970 80 11	Electrolux, Sweden	Output of 1000 W, max. temperature 200 °C
Biochar		Vindelkol AB, Sweden	Grain size: 1mm
Computer fan	Model number SP06015S1M3	Spire Corp, The Netherlands	60 mm edge length by 15 mm height, 12 V, Sleeve bearing, operating temperature range 30–70°C
Conical Centrifuge Tubes, 50 mL	VWR article number 734-0448	Corning Incorporated, USA	Poly(propene), Falcon™
Digital scale, two decimal places	Adventurer Pro AV2102	OHAUS Corporation, USA	d = 0.01 g
Digital scale, four decimal places	PA114C	OHAUS Corporation, USA	d = 0.0001 g
Dry combustion	TruMac	LECO Corporation, USA	
EC-meter	Cond 340i	WTW, Germany	Ser.-Nr.: 05120030
EC-probe	TetraCon 325	WTW, Germany	
ICP	Optima 7300 DV Coupled Plasma Optical Emission (ICP-OES)	PerkinElmer Inc., USA	
ICP	Avio 200 ICP Optical Emission Spectrometer	PerkinElmer Inc., USA	

Magnesium oxide MgO	Code: 263835000; Lot: A0377994; CAS: 1309-48-4	Acros Organics, Belgium	98 %, extra pure, powder, particle size 99 % <150 µm, 500 g
Nordkalk SL $Ca(OH)_2$		Nordkalk Corporation, Sweden	Technical grade
Photometer	NOVA 60 A Spectroquant	Merck KGaA, Germany	
pH-meter	PHM210 651R032N020	RADIOMETER ANALYTICAL S.A., France	
pH-probe	"Red Rod" Combined pH electrode	RADIOMETER ANALYTICAL S.A., France	
Pipet tips 5–10 mL	Article number 8987-532	VWR International	
Pipette 1–10 mL	Eppendorf Research 10 mL System nr 041-3474	Eppendorf AG, Germany	
Rotary switch adapter	Article number 44710	Kjell & Co Elektronik AB, Sweden	max. output current 2.25 A, max. output voltage of 27 VA
R	version 4.0.0	RStudio Team 2016	
RStudio	version 1.2.5042	RStudio Team 2016	
R-package *car*		Fox & Weisberg 2019	
R-package *agricolae*		de Mendiburu 2020	
Statistical software	Minitab, Version 18.1	Minitab, Inc., USA	
Spectroquant Ammonium test	1.00683.0001	Merck KGaA, Germany	Used for analysis of NH_4-N
Spectroquant Nitrate test NO_3^- test kit	1.09713.0001 and 1.09713.0002	Merck KGaA, Germany	Used for analysis of Tot-N
Spectroquant Crack Set 20	1.14963.0001	Merck KGaA, Germany	Used to transform all Nitrogen in the sample into Nitrate

Spectroquant Crack Set 10	1.14687.0001	Merck KGaA, Germany	
Spectroquant Phosphate test	1.14848.0001	Merck KGaA, Germany	
Wheat Bran	Batch number 1702230016, Package numbers 022691 and 022692	Kungsörnen, Lantmännen Cerealia, Sweden	Purchased at a local supermarket

Figure 13. One of the triplicates of the dehydration medium MgO after the end of the experiment, after 1.16 kg of human urine was dehydrated in this Petri dish (own work)

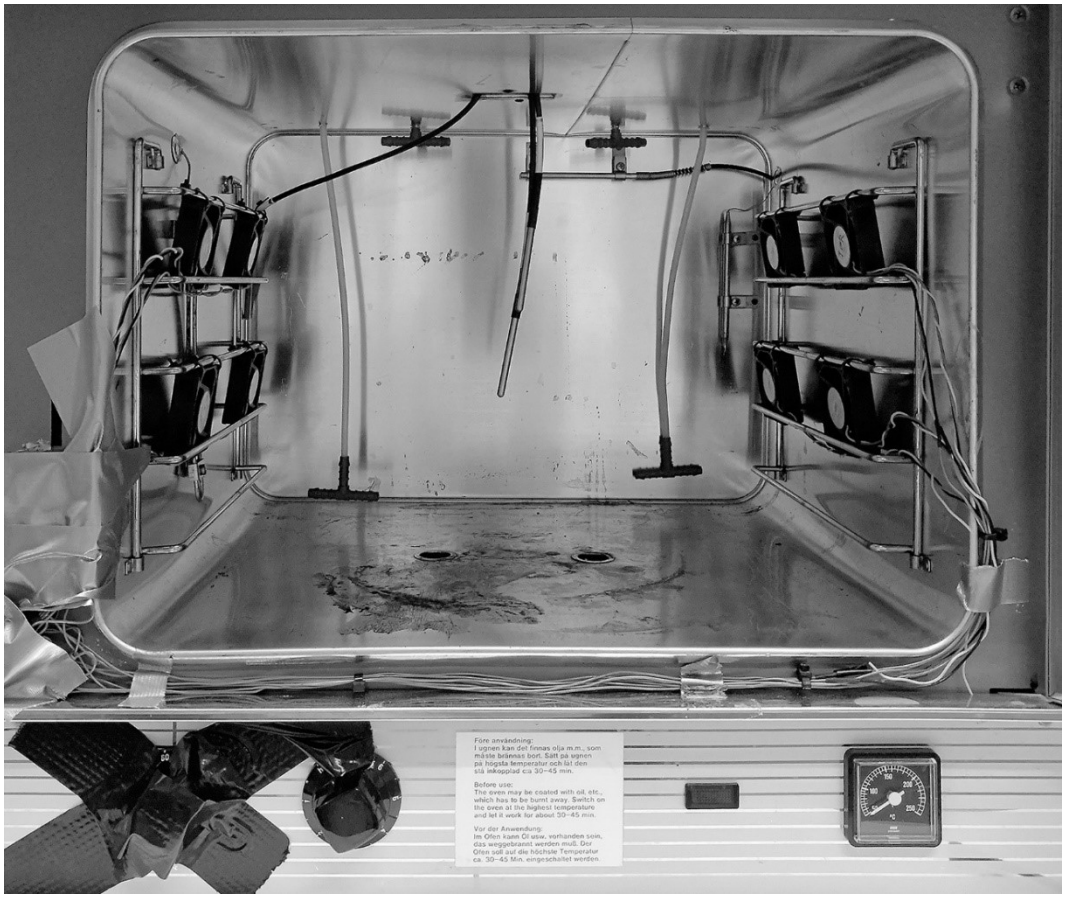

Figure 14. Dehydration setup used during the experiment (own work)

9.1. Excluded dehydration media

9.1.1. Wheat bran and $Ca(OH)_2$

The mixture of wheat bran with $Ca(OH)_2$ 3:1 by weight, formed a surface layer that was impermeable for urine even after a prolonged contact time.

9.1.2. Coffee grounds

First, used coffee grounds were autoclaved at 121 °C for 20 min. The material was moist and absorbed water very well. It was mixed with $Ca(OH)_2$ 3:1 by weight. After the mixing, the mixture became hydrophobic, which made it unsuitable for the experiments.

9.1.3. Hermetia illucens larvae shells

The shells were sieved and then sorted by hand to remove debris and impurities. They were dried at 105 °C for approximately 48 h and then ground using a standard coffee grinder. The ground larvae shells were mixed with $Ca(OH)_2$ at a ratio of 1:1

by weight. This mixture was hydrophobic, which made it unsuitable for the experiments.

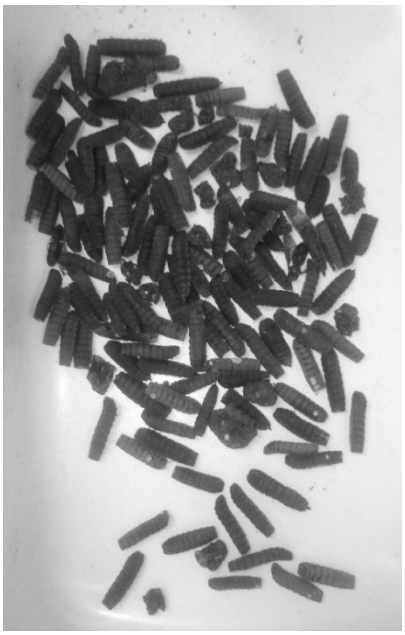

Figure 15. Hermetia illucens larvae shells with impurities (own work)

Figure 16. Hermetia illucens larvae shells after sieving and manual selection (own work)

9.1.4. $CaCO_3$

Calcium carbonate was not used in the final selection of the experiment because the density of calcium carbonate (2.8 g cm^{-3}) (ICSC 1193 - CALCIUM CARBONATE) was considerably higher than the density of other dehydration media used in the experiment, like wheat bran (0.17–0.25 g cm^{-3}) (Food and Agriculture Organization of the United Nations). Because the weight of the dehydration media was fixed to 30 g, and the amount of urine that was applied was fixed to 30 mL, there would have been pooling of urine on top of the calcium

carbonate, while there would at the same time be no pooling on top of other dehydration media.

9.1.5. Sawdust

It was decided to exclude sawdust from the final dehydration media selection because the chemical properties of sawdust and wheat bran were too much alike to advocate for the use of both.

9.2. Trial runs

On the following pages, photos of the many trial runs conducted, and the plethora of tested ideas are shown. They are accompanied by qualitative descriptions to why these ideas were not included in the final experimental design. The photos were taken by the author, but credits for the ideas also go to Björn Vinnerås and Prithvi Simha.

Figure 17. Dehydration media consisting of ash and glass beads – this idea was excluded because the addition of glass beads did not lead to expected higher dehydration rate (own work)

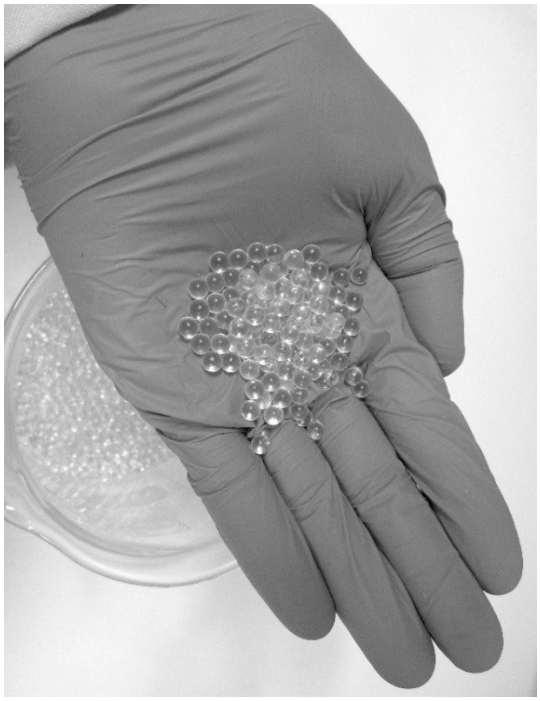

Figure 18. Glass beads that were used for the experiment in Figure 17 (own work)

Figure 19. Two different dehydration setups tested during the trial runs, both were loaded with sawdust and water was applied; the air was pumped out of these setups at the top during the dehydration. These dehydration setups were not chosen because the moist air condensed in the air pumps and destroyed them. Also, as can be seen in Figure 21, with the chosen filling height and loading rate, the sawdust expanded and was blocking the holes on the side of the setup and falling out of them (own work)

Figure 20. Filling of the dehydration setup seen on the left in Figure 19 and in Figure 21 with dry sawdust (own work)

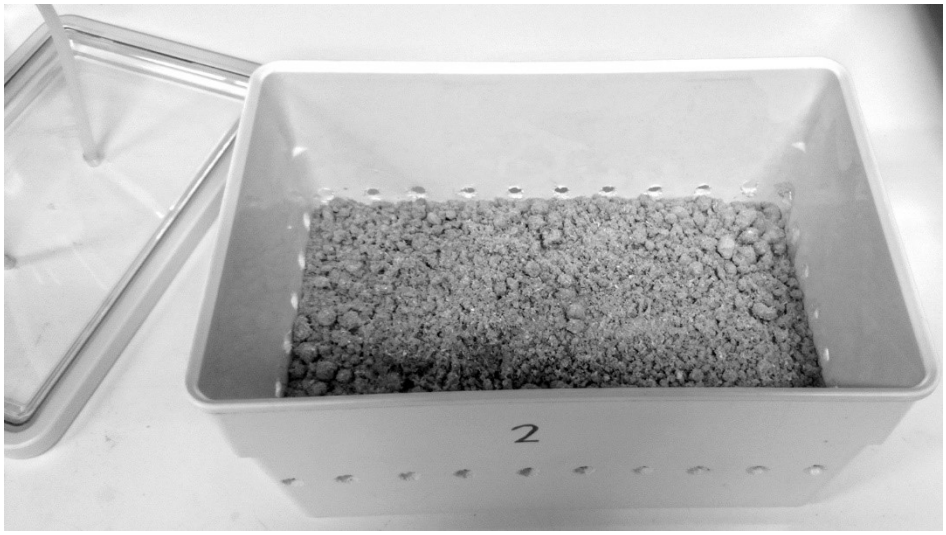

Figure 21. Setup of Figure 19 (on the left side) after the dehydration of water in the dehydration medium sawdust. As can be seen here, the sawdust got very close to the ventilation holes in the walls as compared to the initial filling height seen in Figure 20 (own work)

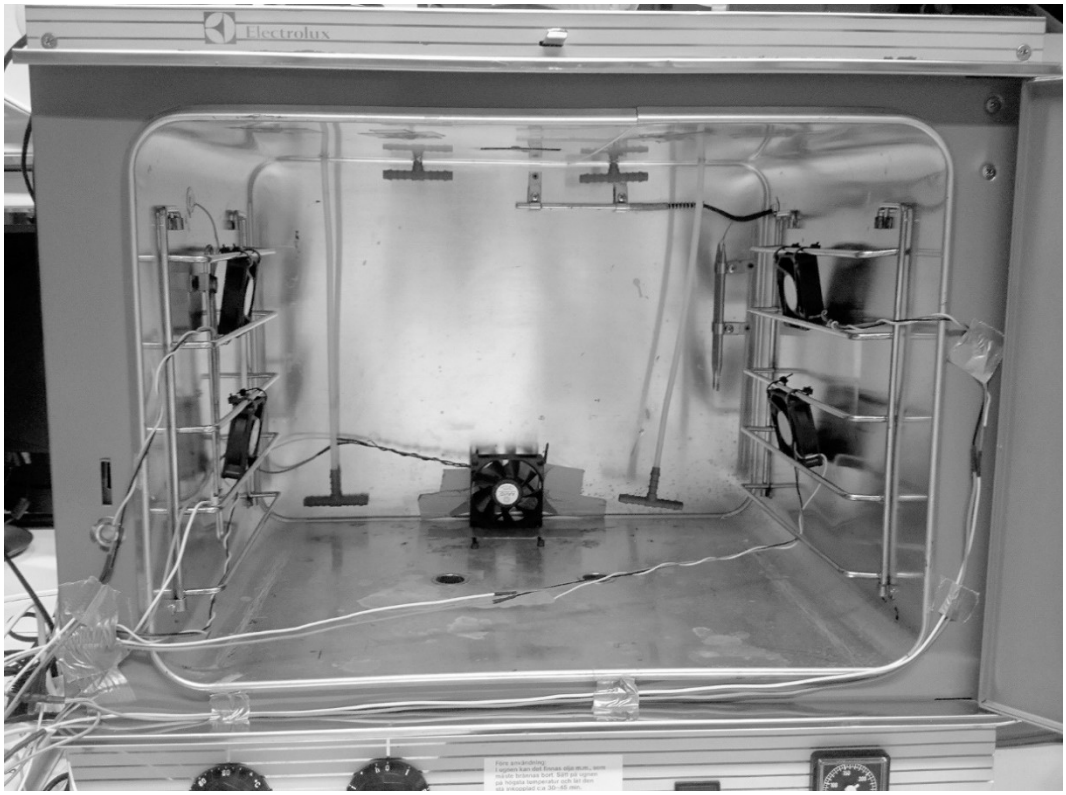

Figure 22. Test of a different setup and a lower number of fans for ventilation in the trial runs, this setup was not used, because the dehydration rate in the dehydration system proved to be too low (own work)

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