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# Alkaline dehydration of source-separated fresh human urine: Preliminary insights into using different dehydration temperature and media

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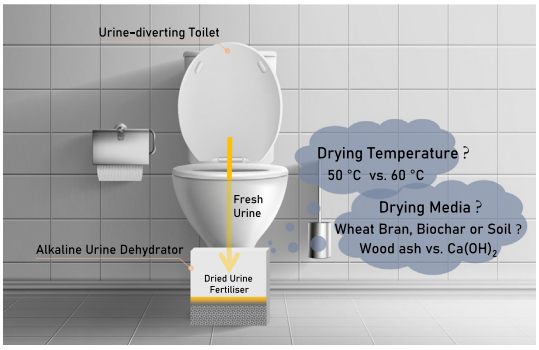
## HIGHLIGHTS

- Innovative sanitation technology developed to convert urine to nutrient-rich solid.
- Urine-based fertiliser produced with 10% N, 1% P & 4% K on dry matter basis.
- >90% N recovered despite drying urine at high temperature (60 °C) & pH (>12).
- Provided insights into selection of alkaline substrates for drying human urine.
- Proposed design chart to estimate N recovery at different drying temperatures.

## GRAPHICAL ABSTRACT

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## ABSTRACT

For sanitation systems aiming at recycling nutrients, separately collecting urine at source is desirable as urine contains most of the nutrients in wastewater. However, reducing the volume of the collected urine and recovering majority of its nutrients is necessary, as this improves the transportability and the end-application of urine-based fertilisers. In this study, we present an innovative method, alkaline dehydration, for treating fresh human urine into a nutrient-rich dry solid. Our aim was to investigate whether fresh urine (pH < 7) added to five different alkaline media (pH > 11) could be dehydrated at elevated temperatures (50 and 60 °C) with minimal loss of urea, urine's principal nitrogen compound. We found that it was possible to concentrate urine 48 times, yielding dry end-products with high fertiliser value: approximately, 10% N, 1% P, and 4% K. We monitored the physico-chemical properties and the composition of various dehydration media to provide useful insights into their suitability for dehydrating urine. We demonstrated that it is possible to recover >90% nitrogen when treating fresh urine by alkaline dehydration by inhibiting the enzymatic hydrolysis of urea at elevated pH and minimising the chemical hydrolysis of urea with high urine dehydration rates.


## 1. Introduction

A growing body of research on new sanitation systems suggests that different household wastewater fractions have different characteristics, different potential benefits as well as risks (Vinneras et al., 2008a;

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Winker et al., 2009), and hence they should be managed separately (Guest et al., 2009; Larsen et al., 2013; Skambraks et al., 2017). In particular, the source-separation of human urine using urine-diverting toilets and the recycling of urine as fertiliser has received significant attention (Harder et al., 2019; Kvarnström et al., 2006; Winker et al., 2009). Collecting urine at source, separate from the rest of the wastewater can, among other things, improve the performance of existing centralised wastewater treatment plants (Wilsenach and Loosdrecht, 2006), reduce freshwater consumption (Mbaya et al., 2017), energy use (Maurer et al., 2003; Tervahauta et al., 2013) and reliance on synthetic fertilisers (Trimmer et al., 2017), as well as mitigate environmental pollution (Lam et al., 2015; Tidåker et al., 2007). However, several aspects constrain the recycling of urine as fertiliser – the composition of urine (mostly water, low nutrient concentration (Putnam, 1971)), long transport distances between urban nutrients and cropland (Trimmer and Guest, 2018), pre-existing wastewater infrastructure (Särkilahti et al., 2017), local policies, legislation, and decision-making (McConville et al., 2017), socio-cultural taboos (Simha et al., 2018a), *et-cetera*. Moreover, separating and recycling urine at the building-scale in urban areas has its own set of challenges, as implemented systems need to be robust, easy to use, operate and maintain (Boyer and Saetta, 2019).

The collected urine can be applied directly as liquid fertiliser after storage (Pradhan et al., 2009; Vinneras et al., 2008b) or used to produce urine-based fertiliser products (Harder et al., 2019; Maurer et al., 2006; Winker et al., 2009). Harder et al. (2019) suggest that there are two broad strategies for treating urine. The first, where water removal from urine concentrates nutrients, producing urine-based fertilisers that are either concentrated liquids, slurries or dry powders; *e.g.* passive evaporation (Pahore et al., 2010), nitrification-distillation (Udert and Wachter, 2012), membrane distillation (Tun et al., 2016) and forward osmosis (Volpin et al., 2018). Yet, apart from nitrification-distillation, most technologies do not recover all the nutrients excreted in urine, especially nitrogen. The second strategy applies treatment processes which selectively extract nutrients present in urine; *e.g.* by precipitation (Etter et al., 2011; Le et al., 2020), adsorption (Pillai et al., 2014), stripping (Başakçilardan-Kabakci et al., 2007) and ion-exchange (Tarpeh et al., 2017). These treatments also concentrate nutrients present in urine, but leave system users with wastewater streams (*e.g.* reject water following struvite precipitation or ion-exchange) that either need to be managed or require further treatment before they can be discharged into the environment.

In order to improve the transportability and the end-use of urine-based fertilisers, it is essential to both reduce the volume of urine and to maximise nutrient recovery during urine treatment. To achieve both these treatment objectives, we have recently suggested alkaline dehydration as a urine treatment technology (Dutta and Vinneras, 2016; Senecal and Vinneras, 2017; Simha et al., 2018b). In alkaline dehydration, source-separated urine is alkalised (pH > 10) and concentrated by dehydration to reduce its volume.

Increasing the pH of fresh urine prevents the enzymatic hydrolysis of urea, urine's major N compound (Lentner, 1981). Hydrolysis of urea to ammonia occurs due to the catalytic action of a urea-specific enzyme, urease (urea amidohydrolase, EC 3.5.1.5). As the bacteria producing the enzymes are ubiqutous, their presence in urine collecting systems is inevitable. However, urease activity and enzymatic ureolysis can be inhibited by alkalisation (Randall et al., 2016; Senecal and Vinneras, 2017; Simha et al., 2018b) or acidification (Saetta and Boyer, 2017). Urea can also undergo thermal degradation (Chin and Kroontje, 1963) and chemical hydrolysis (Blakeley et al., 1982). Yet, at low temperatures (20–40 °C), the rate of enzymatic urea hydrolysis is much greater in relation to the other mechanisms of degradation (Senecal and Vinneras, 2017). Thus, when treating urine, inhibiting urease activity is essential to recover N as urea.

In past studies (Senecal and Vinneras, 2017; Simha et al., 2018b), we have demonstrated that 70–90% of urine's N can be recovered by designing setups that dry between 4 and 12 L urine $d^{-1}$ $m^{-2}$ and which operate in the temperature range 35–50 °C. Yet, improving the dehydration rate of urine can be beneficial as higher moisture removal rates could mean smaller surface area requirements and shorter drying times. One way of doing this is by increasing the air temperature as this increases the moisture carrying capacity of air (Mwithiga and Olwal, 2005). However, increasing the temperature during alkaline urine dehydration can also promote the chemical hydrolysis of urea (Warner, 1942). Hence, our intention in this study was to investigate whether fresh human urine can be treated by alkaline dehydration at two temperatures (50 and 60 °C) with minimal loss of urea. The temperatures were selected because urban areas often have waste heat sources which can potentially be used locally as heat supply for dehydrating urine (Loibl et al., 2017). Moreover, in an earlier study, Randall et al. (2016) pointed out that a conservative upper temperature limit for stabilising urea at high pH is 40 °C, but stated that more research is needed on this end, which we explore in this study.

A system was built to treat urine, where fresh urine was added and concentrated by dehydration in five different alkaline media. Two alkalising agents, wood ash and calcium hydroxide ($Ca(OH)_2$) were used alone, or mixed with one of the three substrates (wheat bran, biochar, and a desert soil). The objective of the study was to test the use of $Ca(OH)_2$ for alkalising and dehydrating urine. Hence, it was used alone or mixed with biochar and soil. However, it was difficult to dry urine in wheat bran mixed with $Ca(OH)_2$ (due to surface hardening), so bran was mixed with wood ash instead. Wood ash was used as a reference media, to compare results with our earlier studies and because it is a waste by-product, widely available in Scandinavia and North America (Pitman, 2006). Lime was used since it is a low-cost alkalising agent (US $ 0.08 $kg^{-1}$) (Muster et al., 2013) that is routinely applied to acidic agricultural soil (Haynes and Naidu, 1998). The choice of the substrates was motivated by their availability or their properties. Soil is available everywhere whereas wheat bran is an agro-industrial residue produced in large quantities and mainly used as a low-value ingredient in animal feed (Reisinger et al., 2013). For biochar, there is increasing advocacy to apply it to soil for improving soil fertility, nutrient-use efficiency and to sequester carbon (Jeffery et al., 2015).

The overall aim in this study was to assess the possibility of treating fresh urine by alkaline dehydration at high temperatures (50 and 60 °C). The specific objectives of the study were to evaluate the: (i) recovery of N; (ii) changes in physicochemical properties of the dehydration media; (iii) composition of the end-products.

## 2. Methodology

### 2.1. Materials

#### 2.1.1. Urine

Fresh human urine was collected from 15 donors (eight females and seven males, 24–65 years old) using 500 mL polypropylene flasks. Donations were made at different times of the day but the flasks were collected at the end of each working day (in <8 h) and stored at 3 ± 1 °C for at the most one week. Prior to use, urine samples from at least five donations were pooled and heated to 37 ± 2 °C using a water bath, simulating urine temperature at excretion.

#### 2.1.2. Alkaline media

Wheat bran, biochar, desert soil, calcium hydroxide, and wood ash, either alone or as combinations, were used to prepare the drying media. Non-activated biochar was manufactured by pyrolysis of willow (*Salix*) at 450 °C and sieved to <Ø 1 mm. The desert soil (Martian Garden, Texas, U.S.A.), was pre-treated by wet autoclaving at 135 °C for 100 min followed by oven drying at 105 °C for 12 h. Wood ash was collected from stoves burning wood for domestic heating at households in Uppsala, sieved (<Ø 1 mm) and pre-treated in a furnace (LH30/12, Nabertherm GmbH, Germany) for 6 h at 550 °C to convert metal carbonates present in ash to metal oxides, and outgas $CO_2$ into air. Calcium

hydroxide ($Ca(OH)_2$) (Nordkalk Corporation, Sweden) and food grade wheat bran (Kungsörnen, Sweden) were used without any pre-treatment.

The mass composition of the alkaline media used in this study were as follows: Bran-Ash (50% wheat bran and 50% wood ash); Char-Lime (75% biochar and 25% calcium hydroxide); Soil-Lime (75% desert soil and 25% calcium hydroxide); Lime (100% calcium hydroxide); and Ash (100% wood ash) (Table 1). The lime content of the media was based on its solubility in urine, following Randall et al. (2016).

### 2.2. Experiment

#### 2.2.1. Setup

To perform the dehydration experiments, two conventional benchtop incubators (Electrolux, Sweden) were modified (Fig. 1). The incubators had an inbuilt cavity of 470 × 330 × 580 mm and adjustable temperature setting of 50–200 °C. Air was introduced at two different inlets within the cavity using four pumps, each supplying 5 L air $min^{-1}$. Two stainless steel grates were placed and eight computer fans (Spire Corp, The Netherlands) were installed to distribute air parallel to the surface of the grates. The temperature was recorded using three 1-wire digital temperature sensors (DS18B20, Embedded Data Systems LLC, USA).

#### 2.2.2. Procedure

Two identical urine dehydration experiments were performed, each using the five different alkaline media and at the two temperatures. In *Experiment I*, the objective was to monitor the change in physicochemical properties of the alkaline media during urine dehydration, whereas in *Experiment II* the nutrient recovery potential of the treatment and end-product composition was evaluated.

At the beginning of each experiment, 30 g triplicate samples of the alkaline drying media were placed in square polystyrene Petri dishes (Sarstedt, Germany) with dimensions 100 × 100 × 20 mm. However, for Soil-Lime, because of its relatively high bulk density, 60 g alkaline media was placed in the Petri dish. Subsequently, 30 mL fresh urine (preheated to 37 ± 2 °C) was added to each media and the Petri dishes were placed over metal grates in the two incubators (50 and 60 °C). The incubators were operated for fixed time durations – 3.5 h at 50 °C and 2.5 h at 60 °C, respectively. After this duration, the Petri dishes were removed, weighed, and another 30 mL fresh urine was added to each dish before they were returned to the incubators. In *Experiment I*, 30 mL urine was added 36 times to the same media, while in *Experiment II*, 30 mL urine was added 48 times. Over the course of the experiments, urine was observed to pool over the media surface. Hence, for certain drying cycles, the drying time was prolonged by operating the incubators for an additional hour. (Fig. 2).

Before performing the actual experiments, pre-trials were carried out in which deionised water was dehydrated in wood ash placed in Petri dishes. Based on these pre-trials, it was decided to randomise the position of the Petri dishes in the oven after every addition of urine. This was because the dishes placed near the computer fans dried out faster than the rest.

**Table 1**
The average physicochemical properties and the elemental composition (as % of TS) of all the drying media ($n = 3$) and the urine ($n = 163$) at the start of the experiment is presented.

| | Bran-Ash | Char-Lime | Soil-Lime | Lime | Ash | Urine |
|---|---|---|---|---|---|---|
| pH [−][a] | 11.2 | 12.7 | 12.8 | 12.8 | 12.8 | 6.6 |
| EC [mS $cm^{-1}$][a] | 18.6 | 13.5 | 14.1 | 15.5 | 39.5 | 14.4 |
| Total solids, TS [%] | 92 | 99.4 | 96.7 | 100 | 99.2 | 1.9[c] |
| Elemental composition [% of TS] | | | | | | |
| N | 1.3 | 0.1 | 0.0 | – | 0.0 | 24.3 |
| C | 26.0 | 55.9 | 0.6 | ≤1.09[b] | 8.2 | – |
| P | 1.5 | 0.0 | 0.1 | – | 1.9 | 2.3 |
| Ca | 17.0 | 0.1 | 1.0 | >60.7[b] | 33.9 | – |
| Mg | 1.7 | 0.1 | 1.0 | ≤3.0[b] | 3.1 | – |
| Fe | 0.1 | 0.1 | 2.4 | – | 0.2 | – |
| K | 5.6 | 0.1 | 0.2 | – | 10.1 | – |
| Na | 0.1 | 0.0 | 0.1 | – | 0.1 | – |
| Mn | 0.2 | 0.0 | 0.0 | – | 0.4 | – |
| Al | 0.1 | 0.1 | 0.7 | – | 0.2 | – |
| Cu | 0.0 | 0.0 | 0.0 | – | 0.0 | – |
| Zn | 0.2 | 0.0 | 0.0 | – | 0.4 | – |
| S | 0.4 | 0.0 | 0.0 | ≤0.80[b] | 0.6 | – |

[a] pH and EC were measured in 1:5 (media:urine) suspensions at start of the experiment.
[b] Data available from producer.
[c] Not adjusted for loss of urea; (−) not measured.

#### 2.2.3. Sampling and physicochemical analysis

All the Petri dishes were weighed (Kern KB 2000–2NM, Germany; 0.01 g precision) after every addition of urine and after they were removed from the incubator to monitor the weight loss. The $pH_{1:5}$ and Electrical Conductivity ($EC_{1:5}$) of the added urine was monitored throughout both experiments. The $pH_{1:5}$ and $EC_{1:5}$ of the drying media was analysed before the start of the experiments, after the dehydration of every 360 mL of urine in *Experiment I*, and at the end of *Experiment II* (Fig. 2). For these analyses, 5 g of thoroughly mixed material from the Petri dishes were diluted with 25 mL fresh urine and allowed to rest for 1 h in closed tubes prior to measurement. The measurements were done using a pH meter (PHM210, Radiometer Analytical SAS, France) and a handheld EC meter (Cond 340i, WTW, Germany). Following the measurement, the samples and the urine were returned to the Petri dish. Urine was used to dilute the samples in order to represent real-life situation, where it is the pH of urine following its addition to the alkaline media which is of interest.

The urine was analysed for total N, total P and ammonium-N using Spectroquant® test kits as per the instructions provided by the manufacturer (Merck KGaA, Darmstadt, Germany). For total N analysis, urine was diluted 1000-fold, digested using Spectroquant® Crack-Set 20 (114963), and its nitrate concentration determined using Spectroquant® nitrate test kit (109713) with concentration range 1–25 mg $L^{-1}$. For total P analysis, urine was diluted 1000-fold, digested using Spectroquant® Crack-Set 10 (114687), and its phosphate concentration determined using Spectroquant® phosphate test kit (100798) with concentration range 1–100 mg $L^{-1}$. To measure $NH_4$-N, urine was diluted 10-fold and analysed using Spectroquant® ammonium test kit (109713) with concentration range 5–150 mg $L^{-1}$.

The elemental composition of the drying media was analysed before and after *Experiment II*. The total N and total C content was analysed by Dumas combustion method using an elemental analyser (LECO TruMac® CN, USA). ICP-OES measurements were performed with an Optima Avio 200 optical emission spectrometer (PerkinElmer, USA) to analyse metals (K, Ca, Mg, Fe, Na, Mn, Al, Cu, and Zn) and non-metals (P and S) in the drying media.

Substrates and urine used in the experiments were analysed for Total Solids (TS) and ash content by drying at 105 °C for 12 h, followed by combustion in a furnace (LH30/12, Nabertherm GmbH, Germany) at 550 °C for 6 h. The TS content was adjusted to account for the loss of urea. The urea-N content was estimated to be 88% of the total-N content of urine (4.87 g $L^{-1}$) since the $NH_4$-N content of urine was 0.59 g $L^{-1}$.

### 2.3. Calculations

The concentration of urine due to dehydration was calculated on wet basis as the mass concentration factor ($mass.cf_{WB}$):

$$\text{mass}.cf_{WB} = \left(\frac{m_{\text{media}} + m_{\text{urine}}}{m_{end-\text{product}}}\right), \quad (1)$$

where $m_{media}$, $m_{urine}$, and $m_{end\text{-}product}$ are the weight of the drying media at the start of the experiment, the total urine added in the

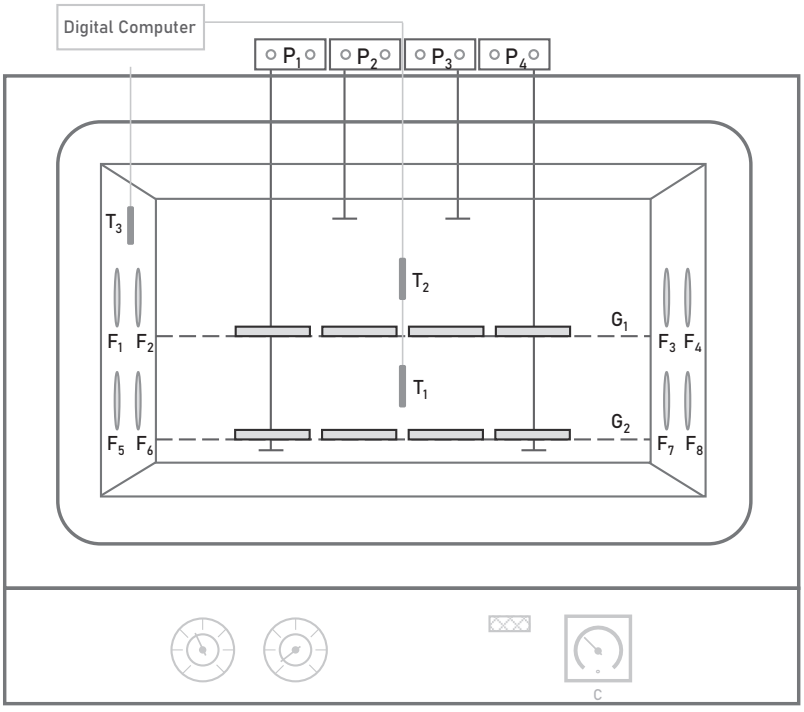


**Fig. 1.** Schematic of the study's urine dehydrator setup. Pumps ($P_1$-$P_4$) introduced air into the incubator cavity and computer fans ($F_1$-$F_8$) distributed it across the surface of the grates ($G_1$ and $G_2$). Petri dishes (grey rectangles) containing the drying media and urine were placed on the grates and dried for fixed time durations. Temperature ($T_1$-$T_3$) was monitored at different positions in the incubator using digital sensors connected to a computer.

experiment, and the end-product, respectively (Eq. (1)). The urine dehydration rate, $dry.rate_{WB}$ (kg urine day$^{-1}$ m$^{-2}$) was calculated as:

$$dry.\text{rate}_{WB} = \left(\frac{w_i - w_{i+1}}{t \times A}\right) \times 100, \quad (2)$$

where $w_i$ and $w_{i+1}$ are the weight of the Petri dish measured after fresh urine was added to the media, and after the urine had dried in the incubator; $t$ is the dehydration time and $A$ is the surface area available for dehydration (Eq. (2)).

The theoretical amount of urea recovered by dehydrating urine was calculated as:

$$\text{urea}.rec_i = (\text{urea}.add_i + \text{urea}.rec_{i-1}) \times \left(1 - \frac{t}{2 \times h_{\frac{1}{2}}}\right), \quad (3)$$

where $urea.add_i$ is amount of urea added in time period $i$, $urea.rec_{i-1}$ is amount of urea recovered in the previous time period, $t$ is the time difference between the two periods, and $h_{1/2}$ is the half-life of urea (Eq. (3)). For the pH range 2–12, the half-life of urea (in hours) at temperature $T$ (in °C) was estimated using Eq. (4), which was derived using

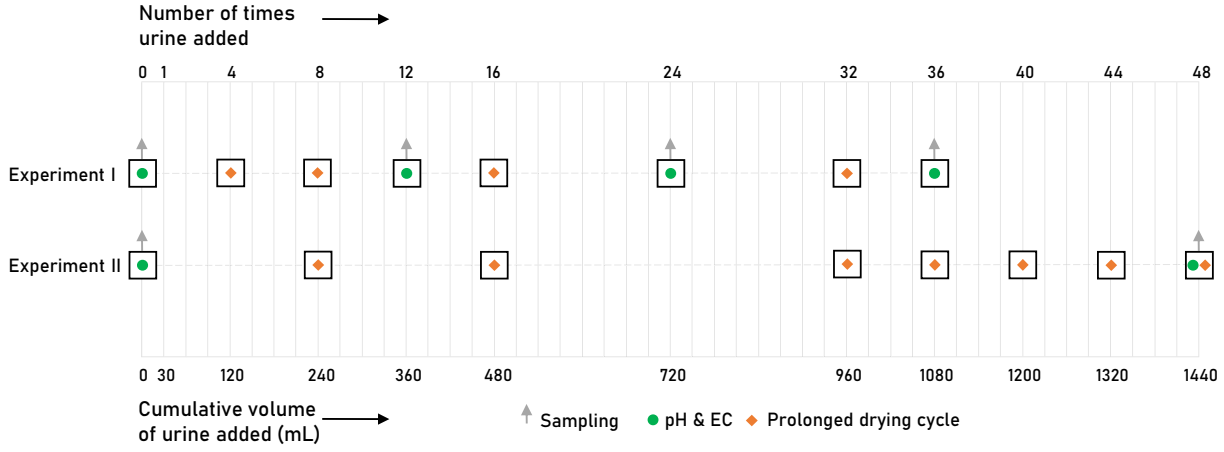


**Fig. 2.** Schematic representation of the experimental procedure and sampling of the dehydration media used in this study. In *Experiment I*, 30 mL fresh urine was added to the alkaline media 36 times and dehydrated, while in *Experiment II* 30 mL fresh urine was added and dried 48 times. During prolonged drying cycles, the incubators were operated for an additional hour (total drying time being 4.5 h at 50 °C and 3.5 h at 60 °C).

parameters suggested by Warner (1942) (Supplementary Information, Fig. S1).

$$h_{\frac{1}{2}} = 1.352 \times 10^6 \times e^{-(0.1257T)} \quad (4)$$

Subsequently, the percentage recovery of urea after $n$ days of alkaline dehydration was calculated as:

$$100 \times \left( \frac{urea.rec_n}{\sum_i^n urea.add_i} \right). \quad (5)$$

### 2.4. Statistical analyses

The experimental data was tested for normality using the Shapiro–Wilk test and homogeneity of variances using the *F*-test. One-way analysis of variance (ANOVA) at 95% confidence level was used to check the mean differences between the dehydration media for $mass.red_{WB}$, $dry.rate_{WB}$, nutrient recovery, elemental composition, and physicochemical properties. Where a significant difference was observed, the mean values were compared using Tukey's Honest Significant Difference (HSD) test at 95% confidence level. For each dehydration media, independent two-sample Student's *t*-test was performed to compare the performance of the media at 50 and 60 °C. All statistical analyses were performed using RStudio (RStudio Team, 2016).

## 3. Results

### 3.1. Urine concentration and dehydration rate

Dehydrating urine considerably reduced its mass (Table 2). After dehydration, the end-product weighed 1/12th - 1/17th of the total mass of urine and alkaline media added in the experiments (Table 2). The concentration factor (Eq. (1)), which reflected the mass concentration of urine in the end-product in comparison to the total mass of urine and dehydration media used in the treatment, varied with both temperature and the type of alkaline media used in the experiment. Dehydrating urine in wood ash resulted in the highest mass concentration at both temperatures. Also, for the same media, at 60 °C the concentration factor ($p < 0.05$) and drying rate ($p < 0.001$) were significantly higher than at 50 °C.

There was a large variation in moisture removal (weight loss) throughout the experiment (Supplementary Information, Fig. S2), in part due to properties of the alkaline media but also because of the experimental setup (uneven drying at different positions on the grate and fixed operating times). Yet, high average dehydration rates were observed at both temperatures for all the media (Table 2).

**Table 2**
The concentration factor ($mass.cf_{WB}$) and dehydration rate of urine ($dry.rate_{WB}$) for all the media at the two investigated temperatures.

| | $Mass.cf_{WB}$ [−] | | | $Dry.rate_{WB}$ [kg day$^{-1}$ m$^{-2}$] | | |
|---|---|---|---|---|---|---|
| | 50 °C | | 60 °C | 50 °C | | 60 °C |
| Bran-Ash | 12.4 (0.3)$^{cd}$ | <<< | 15.9 (0.5)$^{b}$ | 19.3 (4.7)$^{a}$ | <<< | 27.1 (5.5)$^{a}$ |
| Char-Lime | 13.1 (0.4)$^{c}$ | << | 14.4 (0.2)$^{c}$ | 19.2 (4.8)$^{a}$ | <<< | 26.9 (5.1)$^{a}$ |
| Soil-Lime | 11.9 (0.4)$^{d}$ | < | 13.1 (0.7)$^{d}$ | 19.4 (4.0)$^{a}$ | <<< | 27.2 (4.6)$^{a}$ |
| Lime | 14.5 (0.7)$^{b}$ | << | 17.0 (0.4)$^{ab}$ | 19.4 (4.3)$^{a}$ | <<< | 27.1 (5.2)$^{a}$ |
| Ash | 16.4 (0.4)$^{a}$ | < | 17.2 (0.2)$^{a}$ | 19.4 (3.9)$^{a}$ | <<< | 27.1 (4.6)$^{a}$ |

Values are reported as mean (standard deviation). Within each column, values marked with the same letters show there is no significant difference at that temperature ($p < 0.05$). For each drying media, values that are significantly less at the two temperatures are indicated as: < ($\alpha = 0.05$), << ($\alpha = 0.01$), and <<< ($\alpha = 0.001$).

### 3.2. Nutrient recovery

A mass balance was carried out to estimate the efficiency of the dehydration treatment to recover macronutrients (NPK) from urine (Supplementary Information, Fig. S3). The primary focus for this study was on N recovery, as in theory, all the P and K in urine should be retained as they are non-volatile (Simha et al., 2018b). There was high N recovery (>90%) among all the media at 60 °C, and the recovery rates were overall higher than those for the media drying urine at 50 °C (Table 3). Drying urine in Char-Lime at 50 °C resulted in near complete recovery of P and K, yet the lowest N recovery, just 11 ± 1%.

The theoretical recovery of urea from urine, calculated using Eq. (3), was estimated as 98% at 50 °C and 95% at 60 °C. This calculation is based on the half-life of urea, which was approximated following Warner (1942), where urea-N content was estimated to be 88% of the total-N and considering the total experiment time to be 7.3 and 5.3 days at 50 and 60 °C, respectively (Supplementary Information, Fig. S1). However, we observed higher than expected N recovery at the higher dehydration temperature and *vice versa* at the lower temperature. There was also significant deviation between the expected and the actual N recovery from urine at 50 °C, particularly in Char-Lime, Bran-Ash and Lime. Overall, for recovering N when drying urine, all the tested media performed well (>90% recovery), but at a drying temperature of 60 °C.

### 3.3. Elemental composition

The end-products collected from the experiment were analysed for elemental composition (Table 4). For the same media, dehydrating urine at a higher temperature did not result in significantly different compositions, except for N, particularly in Char-Lime, Bran-Ash, and Lime. However, for the same temperature, there was a statistically significant difference in the composition of all the end-products ($p < 0.05$).

On dry matter basis, all the end-products had high N concentration, especially when drying urine at 60 °C. The N concentration was highest in Bran-Ash, yet similar to Char-Lime, Lime, or Ash at 60 °C ($P < 0.05$). Char-Lime at 50 °C was the outlier with the least N concentration (only 1.5%) as there was poor N recovery in this end-product (Table 3). Wood ash has high initial concentration of P and K (Table 1), and drying urine in this media results in end-products with the highest P and K concentrations.

The end-products also contained S, on average 0.5%, as it is excreted in urine (0.17–0.22 g L$^{-1}$ according to Kirchmann and Pettersson (1994)) and also present in the dehydration media. There was high C concentration in Char-Lime, and Ca in the Lime and Ash treatments, as the media initially had high concentrations of these elements (Table 1). Cu and Zn, heavy metals that are excreted in human urine, were present in very low concentrations (<0.005%) in most of the end-products. However, the end-products containing wood ash had high Zn content – approximately 49,000 mg kg P$^{-1}$ in Bran-Ash and 85,000 mg kg P$^{-1}$ in Ash, in proportion to the amount of ash present in these media, 50 and 100%, respectively.

**Table 3**
Recovery (%) of N from urine after dehydration in different media at 50 and 60 °C presented as mean ± standard deviation. The theoretical recovery of N at the two temperatures, calculated using Eq. (3) is also presented.

| | 50 °C | | 60 °C |
|---|---|---|---|
| Bran-Ash | 75 ± 3$^{b}$ | <<< | 95 ± 7$^{a}$ |
| Char-Lime | 11 ± 1$^{c}$ | <<< | 98 ± 2$^{a}$ |
| Soil-Lime | 88 ± 5$^{a}$ | = | 93 ± 9$^{a}$ |
| Lime | 78 ± 2$^{b}$ | <<< | 96 ± 2$^{a}$ |
| Ash | 95 ± 5$^{a}$ | = | 98 ± 1$^{a}$ |
| Theoretical | 98 | | 95 |

Values within each column marked with the same letters show no significant difference between the various media at that temperature ($p < 0.05$). For each media, values significantly different at the two temperatures are indicated as: <<< (significantly less than; $\alpha = 0.001$) and = (not significantly different; $\alpha = 0.05$).

**Table 4**
The elemental composition (%) of the end-products on dry matter basis is presented for all the dehydration media at both temperatures. Within each column, values marked with the same letters show there is no significant difference between the various media at that temperature ($p < 0.05$). For the same drying media, values that differ significantly at the two temperatures are indicated as: $p < 0.05$ (*), $p < 0.01$ (**), and $p < 0.001$ (***).

| | | N | P | K | C | S | Ca | Mg | Na | Fe | Mn | Al | Cu | Zn |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 50 °C | Bran-Ash | $8.5^{a,***}$ | $1.7^{a,*}$ | $6.9^{a}$ | $14.9^{b,*}$ | $0.5^{bc}$ | $5.2^{c}$ | $0.6^{b,*}$ | $2.7^{bc,*}$ | $<0.005^{b}$ | $0.070^{b}$ | $0.046^{c}$ | $<0.005^{b}$ | $0.06^{b,***}$ |
| | Char-Lime | $1.5^{c,***}$ | $1.3^{b,*}$ | $4.3^{b}$ | $26.9^{a,*}$ | $0.6^{ab}$ | $6.3^{bc}$ | $0.2^{d}$ | $3.8^{a}$ | $0.097^{b}$ | $0.009^{d}$ | $0.086^{bc}$ | $<0.001^{c}$ | $<0.005^{c}$ |
| | Soil-Lime | $7.2^{b}$ | $0.6^{c}$ | $2.8^{c}$ | $8.0^{d}$ | $0.4^{c}$ | $8.8^{bc}$ | $0.7^{b}$ | $2.2^{c}$ | $1.360^{a}$ | $0.031^{c}$ | $0.356^{a}$ | $<0.001^{c}$ | $<0.005^{c}$ |
| | Lime | $7.2^{b,*}$ | $0.8^{c}$ | $4.2^{b}$ | $12.0^{c}$ | $0.5^{abc}$ | $22.1^{a}$ | $0.3^{c}$ | $3.7^{a,*}$ | $0.076^{b}$ | $<0.005^{d}$ | $0.129^{b,*}$ | $<0.001^{c}$ | $<0.001^{c,***}$ |
| | Ash | $9.6^{a}$ | $1.7^{a}$ | $7.7^{a}$ | $11.7^{c}$ | $0.7^{a}$ | $15.6^{ab}$ | $1.1^{a}$ | $3.6^{ab}$ | $0.061^{b}$ | $0.148^{a}$ | $0.082^{bc}$ | $<0.001^{a}$ | $0.13^{a}$ |
| 60 °C | Bran-Ash | $9.9^{a,***}$ | $1.4^{a,*}$ | $6.1^{b}$ | $17.9^{b,*}$ | $0.6^{ab}$ | $5.8^{d}$ | $0.6^{b,*}$ | $3.1^{a,*}$ | $<0.005^{b}$ | $0.072^{b}$ | $0.046^{d}$ | $<0.005^{b}$ | $0.07^{b,***}$ |
| | Char-Lime | $9.7^{a,***}$ | $0.9^{b,*}$ | $3.6^{cd}$ | $26.5^{a,*}$ | $0.5^{bc}$ | $4.8^{e}$ | $0.2^{d}$ | $3.0^{a}$ | $0.062^{b}$ | $0.007^{d}$ | $0.072^{cd}$ | $<0.001^{c}$ | $<0.005^{c}$ |
| | Soil-Lime | $6.8^{b}$ | $0.5^{c}$ | $2.9^{d}$ | $7.6^{d}$ | $0.4^{c}$ | $7.6^{c}$ | $0.7^{b}$ | $2.4^{b}$ | $1.256^{a}$ | $0.029^{c}$ | $0.342^{a}$ | $<0.001^{c}$ | $<0.005^{c}$ |
| | Lime | $9.3^{a,*}$ | $0.7^{bc}$ | $3.9^{c}$ | $13.3^{c}$ | $0.6^{abc}$ | $16.4^{a}$ | $0.3^{c}$ | $3.1^{a,*}$ | $0.081^{b}$ | $<0.005^{d}$ | $0.140^{b,*}$ | $<0.001^{c}$ | $<0.001^{c,***}$ |
| | Ash | $9.7^{a}$ | $1.5^{a}$ | $8.4^{a}$ | $11.9^{c}$ | $0.7^{a}$ | $11.4^{b}$ | $1.0^{a}$ | $3.3^{a}$ | $0.053^{b}$ | $0.138^{a}$ | $0.088^{cd}$ | $<0.001^{a}$ | $0.13^{a}$ |

### 3.4. Changes in physicochemical properties

Alkalinity is a limiting factor when drying urine because the pH of the drying media must be kept above 10 to inhibit enzymatic ureolysis and recover urea (Simha et al., 2018b). At the start of the experiments, the drying media were strongly alkaline (pH > 11). The pH however decreased when fresh urine was added and dehydrated (Fig. 3a, b). The pH development of the media were very similar at the two temperatures. However, there was significant difference in the pH of different media at the same temperature across the treatment (Supplementary Information, Table S1). Lime and Soil-Lime had pH > 12 throughout the experiment (1440 mL urine added). In all the other drying media, the pH dropped below 10. The volume of urine dehydrated by 30 g Char-Lime, Ash, and Bran-Ash before the pH dropped below 10 was approximately 600, 340 and 120 mL at 50 °C and 680, 340, and 140 mL at 60 °C.

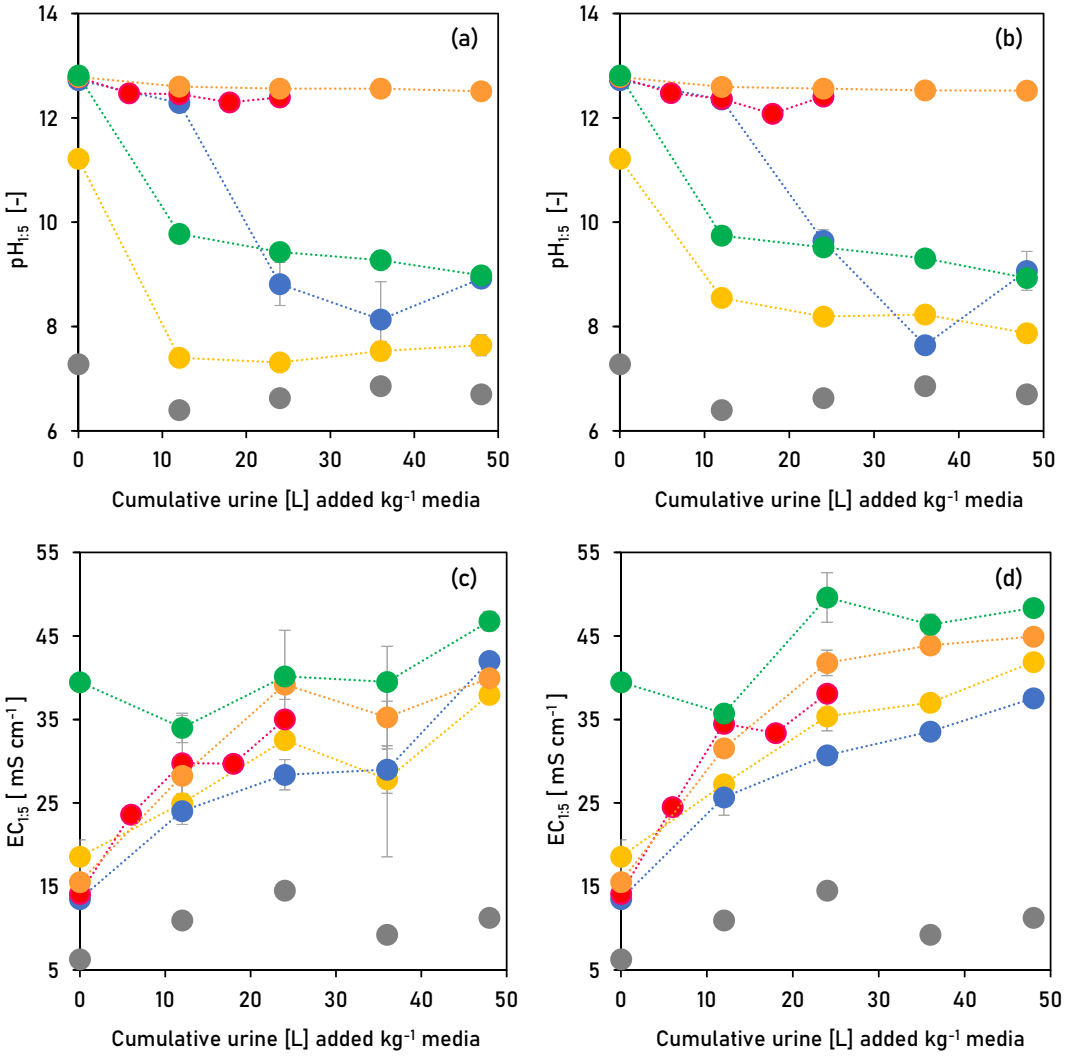


**Fig. 3.** The $pH_{1:5}$ (a, b) and $EC_{1:5}$ (c, d) of fresh urine (●), and the drying media (Bran-Ash (●), Char-Lime (●), Soil-Lime (●), Lime (●), and Ash (●)) at 50 °C (a, c) and 60 °C (b, d). The data was obtained by sampling all the media before the experiments (first point), at the end of *Experiment II* (last point), and during *Experiment I* (intermediary points). Mean values are plotted with error bars showing the standard deviation.

Electrical Conductivity (EC) reflects the total amount of salts in a solution. The EC of urine added to the media varied between 10.4 and 22.1 mS $cm^{-1}$ with a mean value of 14.4 ± 2.8 mS $cm^{-1}$ ($n = 163$). The EC of the drying media measured in 1:5 (media:urine) suspensions was <16 mS $cm^{-1}$, except for wood ash (39.5 mS $cm^{-1}$) (Fig. 3 c, d). The EC of the media gradually increased with cumulative addition and dehydration of urine, and the end-products had EC varying between 37.5 and 48.4 mS $cm^{-1}$.

## 4. Discussion

### 4.1. Dehydration temperature and N recovery

A prerequisite for dehydrating urine at elevated pH and recovering N as urea is inhibition of urease enzyme activity (Blakeley et al., 1982; Randall et al., 2016; Warner, 1942). Treating urine by alkaline dehydration (pH > 10) effectively eliminates the possibility of enzymatic hydrolysis of urea. Yet, urea can be lost by chemical hydrolysis, especially at high temperatures (Blakeley et al., 1982; Warner, 1942). The present study demonstrated that >90% of the N in urine can still be recovered at high temperatures (50 and 60 °C) by alkalising urine and dehydrating it efficiently in order to minimise the time urea is exposed to high temperature and high pH (thus minimising chemical urea hydrolysis).

While urine can be dehydrated at low temperatures (20–40 °C) to minimise the loss of urea (the half-life of urea at 25 °C is 40 years (Shaw and Bordeaux, 1955)), achieving high dehydration rates (per $m^2$) at such temperatures is practically challenging and can hinder implementation. A convective urine dehydrator operating at higher temperatures (>40 °C) is appealing as it can improve the dehydration rate but on the other hand, may require large energy input, also limiting its end use. This study suggested that, in the temperature range between 50 and 60 °C, the surface area required to dehydrate the urine produced by an average family of four, 6 kg $day^{-1}$ according to Vinnerås et al. (2006), is just 0.2–0.3 $m^2$. The area required is small enough that a urine dehydrator can be placed in most existing bathrooms by making use of unused or dead spaces, or even integrated with future toilet designs.

In this study we also found that drying urine at 60 °C as against 50 °C yielded higher N recovery in all the dehydration media. This is contrary to the expected recovery of N, calculated using Eqs. (3) and (4), where the half-life of urea was estimated using parameters suggested by Warner (1942). However, Warner's investigations on urea hydrolysis were performed in sealed tubes, presumably with little change in moisture content. During urine dehydration, the moisture content is initially high when urine is added to the media (1,1 v/w in this study), but is reduced considerably during dehydration, first on account of removal of unbound water during the constant rate drying period, and second due to the removal of bound water because of molecular diffusion when drying is continued in the falling rate period (Mujumdar, 1997). Moreover, at 60 °C, the rate of moisture removal is higher (27 L $d^{-1}$ $m^{-2}$) than that at 50 °C (19 L $d^{-1}$ $m^{-2}$) and is reflected in the total time required to dry urine at the two temperatures (5.3 days at 60 °C and 7.3 days at 50 °C). Hence, using Warner's approach to estimate the chemical hydrolysis of urea perhaps leads to an overestimation of the N loss during alkaline urine dehydration at high temperatures.

Nevertheless, Warner's approach is helpful to approximate the potential loss of urea. We have developed a design chart (Fig. 4) using this approach to estimate how much N can be recovered when drying urine at different temperatures and dehydration rates. For a given temperature, drying urine at a higher dehydration rate will result in higher N recovery, since chemical hydrolysis of urea is reduced by reducing the total dehydration time. For example, this study's setup dried urine at the rate of 27 L $d^{-1}$ $m^{-2}$ at 60 °C to result in >90% recovery of N. Yet, at the same temperature if urine is dried at the rate of 5 L $d^{-1}$ $m^{-2}$, the recovery of N will be <80%. The chart can thus help tentatively guide the design of urine dehydrators by identifying the dehydration rate that needs to be achieved at different temperatures. Further research is however needed to better quantify the loss of urea-N during alkaline urine dehydration.

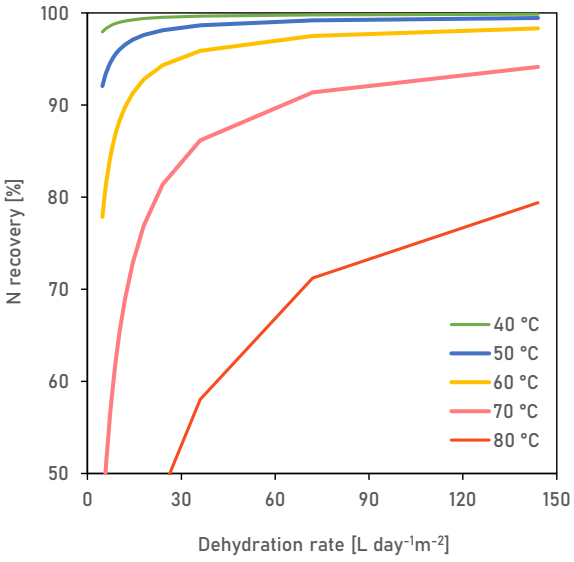


**Fig. 4.** The theoretical recovery of N (%) against the urine dehydration rate (L $day^{-1}$ $m^{-2}$) at different temperatures. The theoretical recovery was calculated using the approach suggested by Warner (1942) that estimates the half-life of urea (Eq. (4)) in the pH range 2–12 and by using the dehydration time at different dehydration rates. The simulation assumes fresh urine is added cumulatively and concentrated 48 times within the same alkaline media.

### 4.2. Dehydration media

This study explored the use of five different alkaline media for drying fresh human urine. The drying media differed in terms of N recovery, elemental composition, and physicochemical properties (Table 5). Not all the media used in this study could sustain pH > 10 throughout the treatment, which is necessary to ensure inhibition of urease enzyme activity. There is risk of reactivation of urease at pH < 10 and enzymatic hydrolysis of urea (Geinzer, 2017). As discussed in Simha et al., 2018b, the primary reason for the drop in pH is absorption of $CO_2$ and formation of carbonic acid. Two alkalising agents were used in this study, wood ash and calcium hydroxide. According to Randall et al. (2016), in the temperature range 50–60 °C, between 3 and 5 g of $Ca(OH)_2$ dissolves in 1 L of fresh urine. This suggested that for Lime, Soil-Lime, and Char-Lime there should be excess $Ca(OH)_2$ available to saturate urine and maintain pH > 10, since all the three media dehydrated 1.44 L urine (per 30 or 60 g media) and had ≥7.5 g $Ca(OH)_2$ at the start of the treatment. Yet, the pH dropped <10 in Char-Lime even though only between 4.3 and 7.2 g $Ca(OH)_2$ would have dissolved in urine. Urease enzymes may have reactivated as a response to this drop in pH (Fig. 3) and can perhaps explain the poor N recovery observed in Char-Lime at 50 °C (Table 3). The pH also dropped to <10 in Ash and Bran-Ash but no effect on urea degradation was detected. The system used in this study had a low bacterial load compared to that in a toilet system, since urine was collected in individual sterile flasks. With higher bacterial load such as that in a urine-diverting toilet setting, all the drying media would be affected by enzymatic urea hydrolysis upon the drop in pH. This suggests that, when using drying media of lower alkalinity (*e.g.* wood ash) to dry urine over long time periods, it is necessary to take precautions to keep the pH elevated. This can be done either by pre-treating the urine, *e.g.* by anion-exchange (Simha et al. (2018b)), by adding a stronger alkalising agent (*e.g.* $Ca(OH)_2$), or by decreasing the consumption of alkali by pre-treating air to remove $CO_2$.

**Table 5**
Summary of the study's findings are presented. For each media, the volume of urine treated (L $kg^{-1}$ media) before $pH_{1:5}$ drops to <10, the recovery of N (%), as well as N-P-K composition (as % of TS) is shown at both the drying temperatures (50 and 60 °C).

| Media | Urine treated [L $kg^{-1}$] | | N recovery [%] | | N [%] | | P [%] | | K [%] | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 50 °C | 60 °C | 50 °C | 60 °C | 50 °C | 60 °C | 50 °C | 60 °C | 50 °C | 60 °C |
| Bran-Ash | 4 | 4.7 | 75 | 95 | 8.5 | 9.9 | 1.7 | 1.4 | 6.9 | 6.1 |
| Char-Lime | 20 | 22.7 | 11 | 98 | 1.5 | 9.7 | 1.3 | 0.9 | 4.3 | 3.6 |
| Soil-Lime | 24 | 24 | 88 | 93 | 7.2 | 6.8 | 0.6 | 0.5 | 2.8 | 2.9 |
| Lime | 48 | 48 | 78 | 96 | 7.2 | 9.3 | 0.8 | 0.7 | 4.2 | 3.9 |
| Ash | 11.3 | 11.3 | 95 | 98 | 9.6 | 9.7 | 1.7 | 1.5 | 7.7 | 8.4 |

### 4.3. End-product composition and end-use implications

Treating urine by dehydration yielded end-products that can be used as fertiliser (Table 5). The end-products contained primary (N, P, and K), secondary (Ca, Mg, and S), and micro- plant nutrients (Table 4). Following conventional fertiliser labelling (*i.e.* N-P-K, given as percentage of weight), the fertiliser value of dehydrated urine was found to vary between 6.8 and 0.5-2.9 and 9.7-1.5-8.4. This value is much higher than that of fresh human urine (0.7–0.06-0.2 according to (Vinnerås et al., 2006)) and liquid dairy manure (0.4-0.1-0.25 according to (Brown, 2008)) but less than synthetic fertilisers like ammonium nitrate (33-0-0), diammonium phosphate (18-23.5-0), and potassium chloride (0-0-52) (Maguire et al., 2009) or blended fertilisers used in Sweden (21-4-7 for cereal crops or lawns and 11-4.6-17.6 for vegetables, fruits and berries) (Granngården, 2019). In any case, one tonne of fertiliser produced by drying urine in wood ash at 60 °C has >95 kg N, 15 kg P and >80 kg K (Table 4), which is enough NPK per hectare for cultivating cereal crops (Albertsson, 2008). Further research is needed to understand the plant availability of different nutrients present in dehydrated urine when applied as fertiliser.

The end-products also contained micronutrients (Table 4), of which B, Cu, Fe, Cl, Mn, Mo and Zn are plant-required micronutrients (Fraústo da Silva and Williams, 1997). Apart from Cl (2.3–2.5 g $L^{-1}$), urinary excretion of these nutrients is low (Kirchmann and Pettersson, 1994). Thus, any significant accumulation of these nutrients in the end-product were contributions from the substrates and not urine; *e.g.* Fe in Soil-Lime and Mn in Ash (Table 1). Applying dehydrated urine could minimise the risk of soil micronutrient deficiency (Jönsson et al., 2004). However, the gradual accumulation of urine salts in the media, reflected by the increase in EC of all the media (Fig. 3c, d), may be problematic, both from a process perspective as it results in boiling point elevation (Udert and Wachter (2012)) but also from an end-use perspective as regular fertiliser application may cause soil salinization (Mnkeni et al., 2008). On the other hand, standard fertilisers used in Sweden (*e.g.* Yara Mila 21-4-7) also contain 7% $Cl^-$ by weight.

Apart from the fertilisers produced using wood ash, heavy metals do not appear to be an issue limiting the end-use of the fertiliser. We found low Cu and Zn concentrations in the end-products, and since the concentrations of non-essential heavy metals (Cr, Ni, Pb, Cd, Hg) excreted in urine is normally 1/10th-1/100th of that of Cu and Zn (Vinnerås et al., 2006), it may be assumed that they would accumulate in negligible quantities. However, the Zn concentration of the fertiliser produced using wood ash (49,000–85,000 mg kg $P^{-1}$), despite dilution with wheat bran, will be much higher than that of sewage sludge (20,600 mg $kg^{-1}$ P; Eriksson (2001)) and farmyard manure (18,000 mg kg $P^{-1}$; Jönsson et al. (2004)) on organic cattle farms in Sweden. Hence, when drying urine, the concentration of non-essential heavy metals in the drying media can be of concern, as it can potentially contaminate fresh urine. This risk needs to be taken into consideration as it may affect the suitability of the end-product as crop fertiliser.

This study demonstrated that end-products with different nutrient concentrations as well as different physicochemical properties can be prepared, by drying urine at different temperatures and by using different drying substrates (Table 5). It may be possible to take advantage of this to prepare blended urine-based fertilisers suitable for specific end-use, *e.g.*, for cereals, grass or fruits.

## 5. Conclusions

In this study, we investigated the alkaline dehydration of fresh human urine in different drying media at two temperatures (50 and 60 °C). We demonstrated that it is possible to recover >90% N from urine even at these temperatures, by inhibiting enzymatic ureolysis at elevated pH and by minimising the chemical hydrolysis of urea with high urine dehydration rates. Our study also showed that a higher dehydration rate at 60 °C (27 L urine $d^{-1}$ $m^{-2}$) resulted in better N recovery than slower dehydration at 50 °C (19 L urine $d^{-1}$ $m^{-2}$).

The drying media used in this study concentrated urine 48 times, producing nutrient-rich end-products; *e.g.* the fertiliser value when drying urine in pure wood ash was 9.7-1.5-8.4. We found that the type of the media used to dry urine had significant influence on the fertiliser value of the end-products, especially regarding its concentration of heavy metals. While we found both wood ash and $Ca(OH)_2$ to be good alkalising agents for inhibiting urease enzyme activity, we also observed that the media produced using $Ca(OH)_2$ sustained alkalinity better throughout the treatment.

Treating human urine collected in new sanitation systems by alkaline dehydration to produce concentrated, nutrient-rich dry fertilisers is innovative and this study adds to the development of the technology by providing useful insights into the selection of the dehydration media and dehydration temperature, and how these aspects influence the fertiliser end-product.

## CRediT authorship contribution statement

**Prithvi Simha:** Conceptualization, Methodology, Investigation, Writing - original draft, Visualization. **Cecilia Lalander:** Conceptualization, Formal analysis, Writing - review & editing, Supervision. **Annika Nordin:** Conceptualization, Writing - review & editing, Supervision. **Björn Vinnerås:** Conceptualization, Methodology, Writing - review & editing, Resources, Funding acquisition, Supervision.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Supplementary information

A document containing Figs. S1–S3 and Table S1 is available as supplementary information. It provides further information on the theoretical half-life of urea, the study's experimental setup, the variation in urine dehydration rate, the mass balance for the dehydration treatment, and the changes in $pH_{1:5}$ and $EC_{1:5}$ of the media. Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2020.139313.

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