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Alkaline dehydration of anion−exchanged human urine: Volume reduction, nutrient recovery and process optimisation

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ABSTRACT

In urine-separating sanitation systems, bacterial urease enzymes can hydrolyse urea to ammonia during the pipe transport and storage of urine. The present study investigated whether it was possible to reduce the urine volume without losing the nitrogen as ammonia. A method for stabilising the urine prior to dehydration was developed. Briefly, fresh human urine was stabilised by passage through an anion−exchanger, added to an alkaline media (wood ash or alkalised biochar), and dehydrated. Urine dehydration was investigated at three temperatures: 40, 45 and 50 °C. The influence of various factors affecting the dehydration process was modelled and the rate of urine dehydration was optimised. Results indicated that 75% (v/v) of the urine has to pass through the ion−exchanger for alkaline stabilisation of urine to occur. At all investigated temperatures, the dehydrator accomplished >90% volume reduction of ion−exchanged urine, > 70% N retention and 100% recovery of P and K. To realise high degree of nutrient valorisation, this study proposes combining source−separation of human urine with alkaline dehydration.

1. Introduction

New sanitation systems based on source separation of human urine offer the exciting prospect of creating and establishing a new sustainability paradigm in wastewater management (Guest et al., 2009). This emerging paradigm seeks to challenge the rationale of current approaches to wastewater collection, treatment, disposal, and reuse (if it exists) (Larsen et al., 2009). Conventional sanitation systems are designed and operated on the presumption as well as perception that human excreta are ‘wastes’; this consideration is subsequently used to justify its disposal (Esrey et al., 2001). However, sewer networks connecting household toilets with centralised treatment plants utilise (potable) water to mediate the transport of excreta thereby, magnifying the volume of waste at the end−of−the−pipe (Lettinga et al., 2001). This is paradoxical because, if human excreta are indeed waste streams, it is surprising that reducing its volume in the environment is not a treatment objective of conventional sanitation systems. In the EU however, municipal solid waste management, a utility that shares several similarities with sanitation does operate according to a hierarchy where, reducing waste generation is considered the ultimate management option (EC, 2016). Such a hierarchy of treatment options has not been applied to wastewater management the exception being, household demand−side measures that reduce water consumption (Inman and Jeffrey, 2006).

Another growing concern is the continuity of agricultural production to meet the demands of a resource−scarce, yet growing world population (FAO, 2017). In this regard, human excreta, especially urine, has been proposed as an alternative to chemical fertilisers as a source of plant nutrients (Winker et al., 2009). However, urine contains only 0.6% N (Vinnerås et al., 2006) and is an aqueous, dilute fertiliser in comparison to chemical fertilisers such as urea (46% N) or ammonium nitrate (35% N) (Kirchmann and Pettersson, 1994). Taking into account that per capita urine generation is 550 kg yr^{-1} (Vinnerås et al., 2006) and that the world population is approximately 7.5 billion, the annual urine load that needs to be safely handled, treated and assimilated in the environment is more than 4000 million tonnes.

Waterless urine diverting toilets that separately collect human urine and faeces, at source, are in use in several parts of the world (von Münch and Winker, 2011; Cheng et al., 2018). However, storing, transporting and applying large volumes of collected urine as a

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liquid fertiliser can pose financial and logistical burden on users of urine recycling systems (Johansson et al., 2001). While recycling urine, one problem is the hydrolysis of urea which occurs during the pipe transport and storage of urine, catalysed by the bacterial enzyme, urease (urea amidohydrolase) (Krajewska, 2009). The bacteria producing urease are ubiquitous, and can trigger mineral precipitation in urine collecting systems, causing pipe blockages and maintenance issues (Udert et al., 2003). The enzymatic hydrolysis of urea is however strongly dependent on the pH of the solution. Sissons et al. (1990) reported that around pH 9, urease activity falls to less than 10% of its maximum, while Kabdaşlı et al. (2006) observed no urea hydrolysis at pH 10. Recent studies have demonstrated that stabilising urine with wood ash (Dutta and Vinnerås, 2016; Senecal and Vinnerås, 2017) and lime (Randall et al., 2016) inhibits urea hydrolysis.

Ion–exchange resins have been used previously for the selective removal or recovery of compounds from polluted water and wastewater (Milmile et al., 2011). Ion-exchangers have also been used for recovering nutrients from source-separated urine (O'Neal and Boyer, 2013; Tarpeh et al., 2017, 2018). O'Neal and Boyer (2013) were able to selectively adsorb phosphate from synthetic urine solution using anion exchange resin impregnated with hydrous ferric oxide nanoparticles. Tarpeh et al. (2017) investigated the use of cation exchange resins for recovery of ammonium from hydrolysed urine. Tarpeh et al. (2018) demonstrated the feasibility of anion and cation exchangers for the recovery of N and K from hydrolysed urine. Despite these studies on ion-exchange of urine, some research gaps exist. Previous investigations focused on using ion-exchange resins for nutrient recovery from synthetic/hydrolysed urine. To the best knowledge of the authors, anion-exchange resins have not been applied for alkalising and stabilising fresh human urine. Human urine contains several anions, such as HCO_3^-, Cl^-, NO_3^-, PO_4^{3-} and SO_4^{2-} (Putnam, 1971; Ferslew et al., 2001) (Table S1). The concentration of Cl^- ions (as NaCl) is relatively large when compared to the rest of the ions present in urine. Thus, one approach to increase the pH of urine to levels required for urea stabilisation (>10) (Geinzer, 2017) could be to use an anion exchange resin that exchanges Cl^- ions in urine with hydroxide ions (OH^-).

In order to reduce the volume of urine, dehydration technologies have been the subject of recent investigations (Antonini et al., 2012; Dutta and Vinnerås, 2016; Senecal and Vinnerås, 2017). Convective air drying is a relatively simple technological operation used extensively in several industries, especially for food preservation (McMinn and Magee, 1999). Water constitutes more than 95% of the volume of human urine (Putnam, 1971). In principle, this water can be removed to a large extent by heating urine and forcing a current of air over its surface. In practice however, evaporation of human urine is inhibited by a thin oily film that forms over the liquid surface (Tettenborn et al., 2007). Ventilation would also result in the loss of N as NH_3 (Jönsson et al., 2000). Dehydrating human urine is therefore a process challenge, since the objectives of volume reduction and nutrient stabilisation need to be realised simultaneously.

As illustrated in Fig. 1, the use of an alkaline media over which stabilised urine is spread can circumvent the formation of oil films (Dutta and Vinnerås, 2016; Senecal and Vinnerås, 2017). Subsequently, dehydrating urine by convective heat and mass transfer can remove water while the media retains urine nutrients. Hence, the broader question this study seeks to answer is whether human urine can be dehydrated to produce a dry fertiliser. The specific objectives of the study were to: (i) evaluate the effectiveness of anion–exchange to alkalise human urine; (ii) investigate the possibility of reducing the volume of alkalised urine through alkaline dehydration by designing a dehydrator that removes $\geq 90\%$ of urine's water content and, assess the nutrient recovery potential of the combination of anion–exchange and alkaline dehydration; and, (iii) identify, understand, and optimise the functional relationship between parameters that affect dehydration and the rate of urine dehydration.

2. Materials and methods

2.1. Materials

Fresh human urine was collected in sterile 1 L polypropylene bottles. Urine was obtained anonymously from both men and women (10 persons, 24–65 years old) at different times of the day. Freshly collected urine was used immediately in experiments studying the ion-exchange of urine. In the urine drying experiments, fresh urine and ion-exchanged urine was stored at $3 \pm 1\,^{\circ}C$ for <1 week. Prior to use, the urine was brought to room temperature ($20 \pm 0.5\,^{\circ}C$).

Industrial grade Amberlite™ IRA410 type–2 resin (Merck Chemicals GmbH, Darmstadt, Germany) was used to exchange the anions present in urine with OH^- ions. The resin comprised of spherical beads (harmonic mean Ø 0.60–0.75 mm)[1] of styrene divinylbenzene copolymer with dimethyl ethanol ammonium functional group ($R - N^+(C_2H_5)_2(C_2H_5OH)$) and had an exchange capacity[1] of 1.25 eq L^{-1}.

For dehydrating human urine, wood ash and alkalised biochar were used as the dehydration media. Ash produced from the burning of birch wood used for domestic heating in Uppsala, Sweden was collected, sieved (<Ø 1 mm) and thermally treated at 500 °C for 5 h in a furnace (LH30/12, Nabertherm GmbH, Germany). Biochar was manufactured through pyrolysis (450 °C) of chopped willow trees (*Salix*) grown in Germany and sieved to < Ø 1 mm. To increase the pH of the biochar to ≥ 12.5, the procedure suggested by Tseng and Tseng (2005) was used: KOH pellets and biochar were mixed in a weight ratio of 1:4, dissolved using deionised water and kept aside for 1 h to ensure uniform dissolution. Subsequently, the mixture was oven–dried overnight at 110 °C and used in further experiments.

2.2. Experimental setup

The objective of the first set of experiments was to test the resin's efficiency in alkalising urine (Fig. 1). Experiments were performed to estimate the minimum volume of the sub–sample of urine that needed to be ion–exchanged in order for the mixture of ion–exchanged urine and fresh urine to attain a final pH > 10. Using 50 different fresh urine samples, composites comprising: 0, 20, 40, 60, 80 and 100% of ion–exchanged urine mixed with 100, 80, 60, 40, 20 and 0% fresh urine, respectively, were prepared and pH of each composite investigated.

In the second set of experiment, the possibility of reducing the volume of ion–exchanged urine through alkaline dehydration was investigated through three *treatments* performed as singlets: wood ash at 40 °C (ash_{40}) and 50 °C (ash_{50}) and alkalised biochar at 45 °C ($char_{45}$) (Table 1). These treatments were exploratory in nature and were performed so as to provide insights into the dehydration process and help guide the development of the optimisation model (*Experiment III*). The nutrient recovery potential of the treatments was also investigated.

In the third set of experiments, the objective was to identify, understand, and optimise the functional relationship between parameters that affect dehydration of ion–exchanged urine and the

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[1] Data available from manufacturer.