

Recovery of phosphorus from stored urine using continuous flow reactor in decentralised level operations

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Abstract

Past studies have shown that phosphate recovery in the form of struvite is relatively a simple process, which can be achieved by adding a magnesium source in stored urine. However, struvite recovery process at a decentralised level becomes uneconomical due to high input cost of magnesium salts and operational cost. While use of cheaper alternative magnesium sources such as bittern, low-grade MgO and wood ash could lead to partial cost reduction, it is also important to reduce the overall operational costs to make struvite recovery process economically viable and sustainable. In this study, a continuous flow reactor was developed for low-cost struvite recovery from stored urine at decentralised community scale operations. Our study revealed that over 81.2% of phosphate present in urine can be recovered in the form of struvite. Comparison of results from stirred and unstirred experiments shows that higher recovery efficiency is obtained due to minimal loss of fines. Operation and financial assessment of the process shows that struvite recovery can be profitable due to continuous operation of the reactor requiring minimal process control and manpower requirement.

Key words: bittern, decentralised, phosphorus, reactor, struvite, urine

Highlights

- Simple and low cost decentralised struvite reactors are not available.
- Use of low operating cost and continuous flow reactor can help in reducing the operational costs significantly.

INTRODUCTION

Source separation of urine can effectively reduce nutrient loads in wastewater streams. Around 80% of the total nitrogen (N), 70% of the potassium (K) and up to 50% of the total phosphate (P) present in the municipal wastewater originates from urine, which forms less than 1% of the volume total volume of wastewater (Larsen & Gujer 1996). Therefore, urine separation, while increasing efficiency of existing treatment plants, can reduce the overall resource consumption (Wilsenach & van Loosdrecht 2003). Struvite (magnesium ammonium phosphate hexahydrate (MAP), $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is one of the important compounds which can be recovered from human urine and used as fertiliser. It is also known for the devastating role it plays in the wastewater treatment plants causing blockages and scaling of pipelines and mechanical components. Struvite can be precipitated by simple addition

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of a magnesium source such as magnesium chloride (MgCl_2), magnesium sulfate (MgSO_4) or magnesium oxide (periclase, MgO) in source separated urine (Demirer *et al.* 2005). The chemical composition of source-separated hydrolysed urine with a pH value of 8–9 is optimal for recovery of struvite (Udert *et al.* 2003). Hydrolysed urine is the state in which the urea in human urine is converted into ammonia and carbonate, leading to volatilisation ammonia present in urine. However, as the cost of industrially produced magnesium salts are quite high, use of magnesium-containing waste products such as bittern (Etter *et al.* 2011), low-grade MgO (Chimenos *et al.* 2003) or wood ash (Sakthivel *et al.* 2012) are being explored for recovery of struvite from human urine and other waste water streams.

Although the process of struvite precipitation from source separated urine is relatively simple (Tilley *et al.* 2009), transportation cost of urine renders the process unviable at a central level (Etter *et al.* 2011). Therefore, decentralised struvite production at a community or institutional scale helps in offsetting the transportation cost of urine. The operations of fluidised bed reactors which are often used for struvite production at a centralised level from waste waters is relatively complicated (e.g. Abe 1995; Münch & Barr 2001; Ueno & Fujii 2001; Adnan *et al.* 2004; Udert & Wächter 2012), and it would hardly be viable on a decentralised scale (Wilsenach *et al.* 2007). Formation of other compounds such as newberyite ($\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$), bobietite ($\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$), cattite ($\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$), magnesium sodium phosphate hexahydrate ($\text{MgNaPO}_4 \cdot 6\text{H}_2\text{O}$), magnesium hydroxide ($\text{Mg}(\text{OH})_2$) and tricalcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) during struvite precipitation at the different reaction conditions are also reported (Kabdaşlı 2018). Use of stored urine for phosphate recovery is an emerging area of work, and the body of research is growing (Wilsenach *et al.* 2007; Ronteltap *et al.* 2010; Antonini *et al.* 2011; Etter *et al.* 2011; Barbosa *et al.* 2016).

A laboratory scale continuously stirred tank reactor (CSTR) with an improved solid–liquid separation arrangement for reducing scaling on the reactor surface was developed by Wilsenach *et al.* (2007) for struvite recovery. Synthetic urine having a pH of 9.4 and MgCl_2 (1 M) solution were added continuously to the reactor, having independent precipitation and settling chambers with 0.77 litre capacities. The solution was stirred using an axial flow impeller (propeller-type) submerged halfway into the precipitation zone. The study reported over 96% of phosphate removal in the form of struvite. The study revealed that particle size of precipitates could be controlled by limiting the supersaturation and degree of stirring. In the study, degree of stirring and hydraulic retention time (HRT) beyond certain minimum period (30 minutes) was found that it had no effect on the overall precipitation efficiency.

Ronteltap *et al.* (2010) utilised a laboratory scale CSTR of 1.1 litre capacity for recovery of struvite from stored urine using a simple flow through operation without recirculation of solids. The reactor was operated with a HRT of 11 hours and stirred with the help of magnetic stirrer. An overflow weir at the top for outlet of effluent and a sedimentation zone at the bottom for removal of crystals periodically were provided in the reactor for enabling continuous operation. Urine and magnesium chloride solutions were continuously pumped into the reactor at a rate of 88 ml h^{-1} and 12 ml h^{-1} to achieve an average molar Mg:P ratio of 1.33.

Antonini *et al.* (2011) reported the use of a 50 litre capacity batch reactor for struvite recovery from stored urine using MgO at an Mg/P molar ratio of 1.5:1. The authors presented a two-step chemical–physical process module called ‘Yellow Water Treatment’ with a precipitation reactor followed by a stripping and an absorption column for removal of both struvite and ammonium sulphate. In the study, struvite recovery reactor was stirred for 30 minutes and the contents were allowed to settle up to 3 hours for removal of precipitates using a nylon filter bag. The authors reported 98% phosphate removal from stored urine using this process.

Etter *et al.* (2011) developed a manually stirred batch reactor of 50 litres capacity for struvite recovery from stored urine at a decentralised level to minimise the operation and input costs. The authors used bittern as a magnesium source with a molar ratio of Mg:P 1.1. A filter bag attached to the outlet

below the reactor was used for filtering precipitates after each batch of stored urine is loaded and stirred for a period of 10 minutes. The retained filter cake in the filter bag was dried at ambient temperatures. An overall phosphate removal efficiency of 90% was reported in the study. Although this operation shows that struvite recovery from stored urine can be achieved using minimum process control in decentralised settings, the manual operations proposed rendered the process uneconomical.

Recovery of struvite from stored urine with minimum process control and operating parameters can substantially reduce the cost. According to the published literature, factors such as temperature, pH, HRT and degree of stirring should be taken into consideration for achieving these objectives. Zeng & Li (2006) proposed that temperatures in the range of 15 °C–35 °C are optimal for recovery of struvite from anaerobically digested cattle manure. However, based on a series of batch experiments conducted at 5 °C–30 °C using urine, Ronteltap *et al.* (2007) reported rapid rates of phosphate recovery of over 99% in all the experiments conducted. pH of hydrolysed stored urine is usually around 9.5 and therefore no pH modification is required (Stratful *et al.* 2001; Ronteltap *et al.* 2010). Rapid phosphate removal by bittern and water soluble magnesium sources ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) were reported from aqueous solutions due to their high dissolution property (Shin & Lee 1997; Burns *et al.* 2001). Etter *et al.* (2011) reported that HRT can be significantly reduced by employing filtration process as precipitation of phosphate from urine occurs within a short span of 10 minutes. In the experiments conducted using CSTR at mixing speeds from 0 to 600 rpm, Wilsenach *et al.* (2007) found that degree of stirring had very little effect on the overall phosphate levels in the effluent. Ronteltap *et al.* (2010) reported rapid nucleation of struvite crystals and formation particles in the range of 36–136 μm in the experiments conducted using urine. Although factors such as limiting supersaturation and stirring are reported to favour crystal growth of struvite particles over nucleation, in this study formation of large crystals were found limited due to conditions typically found in hydrolysed urine. The negative zeta-potential of struvite crystals at higher pH levels occurring in urine is the factor reported to limit growth of crystals (Le Corre *et al.* 2006).

Based on the review of past studies conducted using stored urine, it would be ideal to develop a continuous flow reactor with minimal process control and low-input costs to design astruvite recovery system at decentralised community scale operations. The following primary process parameters favourable for struvite precipitation from stored urine were taken into consideration in the present study to achieve the above objectives:

1. Stored urine does not require pH modification because of the high buffering capacity of ammonium present in stored urine (Stratful *et al.* 2001).
2. Phosphate removal from stored urine is a very rapid process especially when highly water soluble magnesium sources are used (Shin & Lee 1997).
3. Degree of stirring does not affect overall phosphate precipitation rate and beyond a certain minimum level it only aids in improving the property of precipitates (Wilsenach *et al.* 2007).

MATERIALS AND METHODS

Reactor design and set-up

A continuous flow reactor of 15 litres capacity was designed for phosphate removal from stored urine in this study (Figure 1). The vertical cylindrical shaped reactor partitioned into a reaction zone, a solid–liquid separation and a sedimentation zone which interconnects them at the bottom to facilitate the process of phosphate precipitation and sedimentation. The influents (stored urine and bittern) were dosed in the reaction zone and the effluent is allowed to overflow through an outlet weir

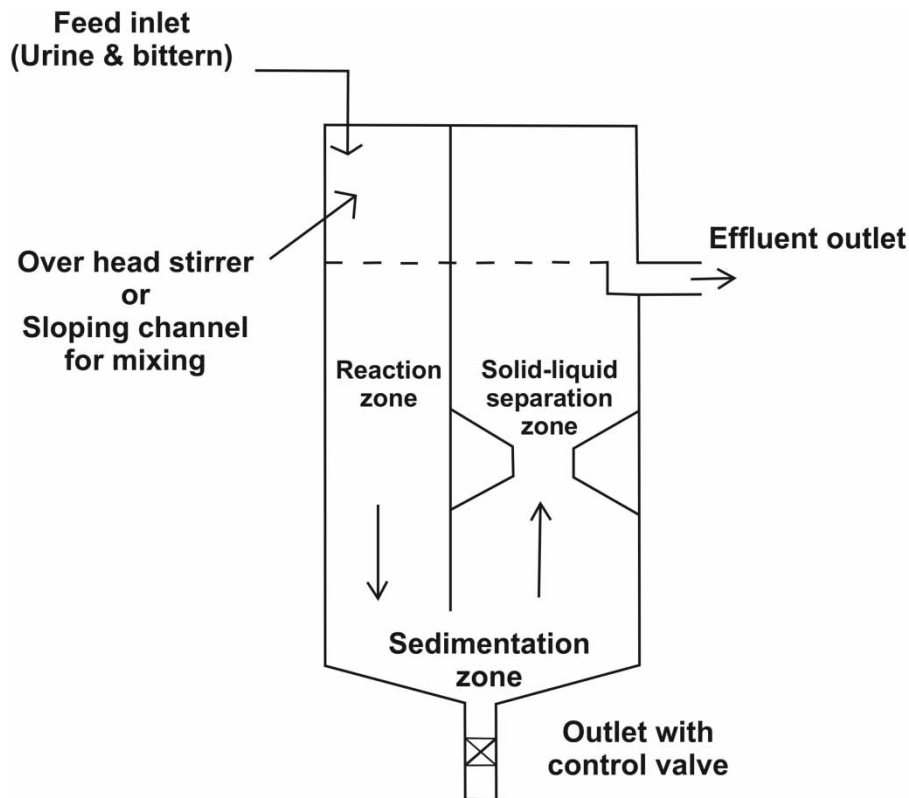


Figure 1 | Schematic of the pilot scale reactor.

provided at top of the solid–liquid separation zone which enables the reactor to work as a continuous up-flow reactor. The reactor was placed over a steel stand to facilitate periodic removal of struvite sediments from the bottom of the reactor through the filtration process. A cylindrical shaped nylon filter bag with pore sizes of 50 μm was used for filtering the precipitates at the end of each experiment. The design specifications of the pilot scale reactor used in this study are given in [Table 1](#).

Stored urine and bittern were allowed to flow into the reaction zone of the reactor by gravity from two separate storage tanks of 500 and 50 litres tank capacities respectively which were kept at an elevation using flow regulators. To improve the mixing of urine and bittern in the reactor, two sets of arrangements were made: (1) for the experiments conducted without stirring, a curved sloping

Table 1 | Specifications of the pilot scale reactor

| Description | Specification |
|-----------------------------------|--|
| Process | Continuous up-flow reactor |
| Treatment capacity | 180 litres (2 h HRT) to 720 litres (0.5 h HRT) |
| Reactor capacity | 15 litres |
| Total height | 0.45 m |
| Overall diameter | 0.25 m \varnothing |
| Reaction zone | 4.5 litres |
| Sedimentation zone | 1 litres |
| Solid–liquid separation zone | 9.5 litres |
| Solid–liquid separation mechanism | Sedimentation + filtration |

channel with a rectangular cross section having 18 mm width and 15 mm depth with an overall drop of 10 cm up from the top to the fluid level in the reactor was fixed to improve mixing of urine and bittern entering the reactor and (2) for the stirred experiments, an overhead stirrer with an axial flow impeller (Remi, India) was used to stir the solution at 100 rpm.

Urine and bittern

Urine required for the study was collected from waterless urinals installed in three schools of Musiri town of Trichy District, Tamilnadu, India. The concentration of phosphate and pH of urine used in the experiments are given in Table 2. Bittern was used as the magnesium source in the study. Bittern collected from a salt pan near Chennai had magnesium concentration of 65.4 g·L⁻¹. Dilution of bittern using deionised water was done as required to maintain a magnesium to phosphate ratio of 1.50 ± 0.1 mol Mg·mol P⁻¹ for all the experiments. Based on the initial assessment of phosphate concentration in the stored urine, the ratio of magnesium to phosphate ratio was adjusted using flow regulator installed in the tanks to obtain the desired ratio.

Table 2 | Operating parameters and effluent characteristics measured during the experiments

| Exp No. | HRT (hours) | Type of Mixing | Initial pH of Urine | Influent PO ₄ -P ^a (mg L ⁻¹) (± Standard Dev.) | Effluent ^b | |
|---------|-------------|---------------------|---------------------|---|---|--|
| | | | | | PO ₄ -P (mg L ⁻¹) (± Standard Dev.) | PO ₄ -P _{tot} (mg L ⁻¹) (± Standard Dev.) |
| 1 | 0.5 | No mixing (channel) | 10.2 | 181.9 (± 1.90) | 32.8 (± 0.93) | 43.5 (± 3.46) |
| 2 | 1 | No mixing (channel) | 10.1 | 180.1 (± 3.95) | 31.4 (± 4.73) | 37.7 (± 6.93) |
| 3 | 2 | No mixing (channel) | 10.2 | 184.1 (± 2.66) | 28.0 (± 1.94) | 34.6 (± 3.21) |
| 4 | 0.5 | 100 rpm | 10.2 | 182.2 (± 2.28) | 31.3 (± 3.65) | 47.9 (± 6.40) |
| 5 | 1 | 100 rpm | 10.1 | 178.0 (± 1.86) | 30.1 (± 2.34) | 48.1 (± 2.59) |
| 6 | 2 | 100 rpm | 10.2 | 179.5 (± 2.21) | 25.3 (± 2.99) | 44.2 (± 9.80) |

^aConcentrations based on the initial measurements taken in duplicate.

^bConcentrations based on the measurements taken at 2, 4, and 8 hour intervals of the experiments.

Experimental studies

The experiments were conducted at the Training and Research Centre located in Musiri, Trichy District in the State of Tamilnadu, India being maintained by the SCOPE, Trichy. The operational parameters of the six experiments conducted in this study are given in Table 2. Prior to each of the experiments, the reactor was initially filled with 15 litres of urine and dosed with bittern at an Mg:P ratio of 1.50 mol Mg·mol P⁻¹ and left for 30 minutes. Experiments were conducted for HRTs of 0.5, 1 and 2 hours, both with the aid of stirring and without stirring. Each experiment lasted for 8 hours duration. Bittern was dosed at a magnesium to phosphate ratio of ≈1.50 mol Mg·mol P⁻¹. At the end of each experiment, the reactor was emptied from the outlet at the bottom to collect precipitates for analysis, and later it was thoroughly cleaned using water dosed with acid solution for complete removal of precipitates from the reactor.

Sampling and analysis

To analyse phosphate removal from urine in the reactor, samples in triplicate were collected at 0, 2, 4 and 8 hour intervals and filtered using 0.45 µm filter cartridges (Durapore – Millipore, Germany). In order to determine concentration of fines escaping along with effluent, samples collected at the outlet

were analysed without filtering. Samples were acidified with 2 M HCl solution and were stored at 4 °C prior to analysis. Samples of struvite precipitated during the experiments were collected from the reactor and air dried prior to the X-ray diffraction and particle size analysis.

Concentrations of magnesium in bittern and phosphate in urine were analysed using EDTA Titrimetric method (IS 3025 – Part 46) and colorimetrically (SpectroquantPharo, Merck, Germany) respectively. Confirmation of crystals and the particle size were conducted using X'Pert PRO diffractometer (PANalytical B.V., Almelo, The Netherlands) using (Cu K α radiation 45 kV and 40 mA) and Scanning Electron Microscope (Zeiss EVO 50) respectively.

RESULTS AND DISCUSSION

Precipitation efficiency

The precipitation efficiency of phosphate removal was determined by comparing phosphate concentrations in the influent and effluent of the reactor (Table 2). The estimates of precipitation efficiencies for the six experiments conducted in the study from samples obtained in triplicate are 82, 82.6, 84.8, 82.8, 83.2 and 85.63 respectively (Figure 2). The first three experiments were conducted without stirring and the next three experiments were conducted with stirring at 100 rpm having HRTs of 0.5, 1 and 2 hour respectively. Short durations of HRT were employed in the experiments due to the rapid precipitation potential of struvite from stored urine. In the experiments conducted, only a marginal increase in the phosphate removal efficiencies were recorded with increased HRTs and stirring. The precipitation efficiencies found in the study corresponds well with the values reported by Pastor *et al.* (2008). The authors reported phosphate removal efficiencies up to 80% for experiments conducted with a 21 litres capacity CSTR fed by artificial phosphate reagent solutions resembling wastewater. Ronteltap *et al.* (2010) observed phosphate removal efficiencies up to 98.7% in experiments conducted using stored urine in a laboratory scale CSTR (stirred at 300 rpm, 11 HRT and

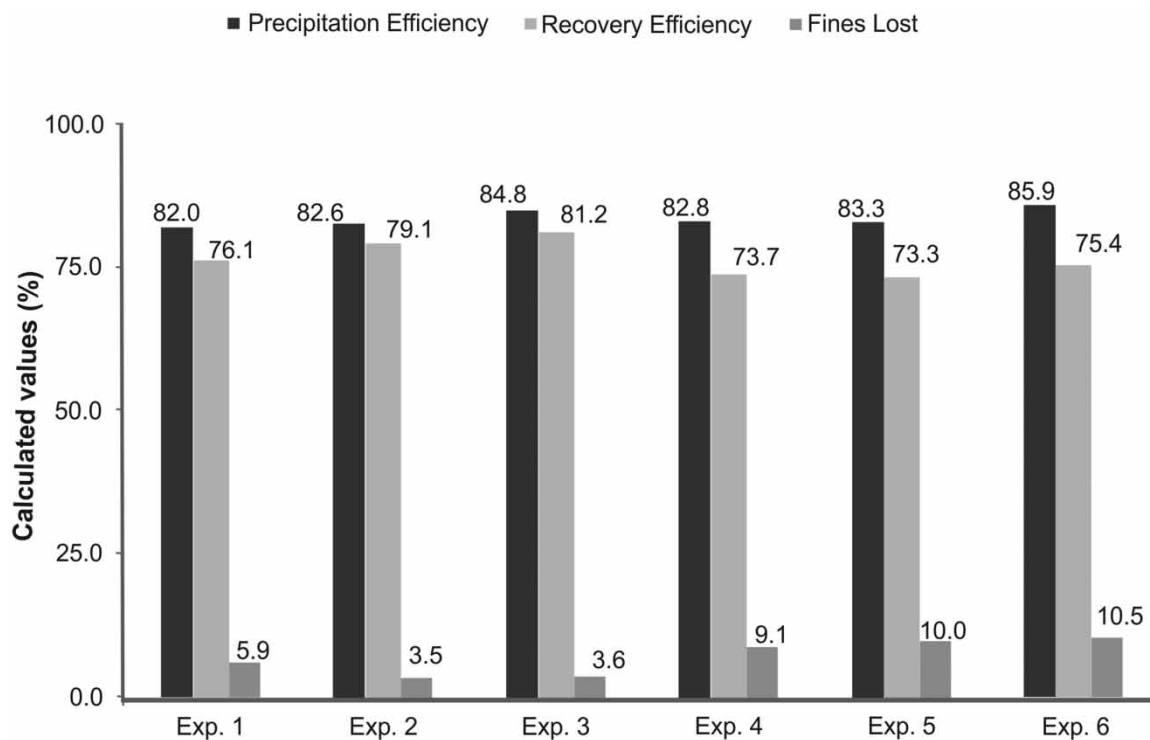


Figure 2 | Phosphate removal efficiencies and fines lost observed in the experiments.

1.1 litres capacity). Reduction in precipitation efficiency in the present study could be due to factors such as dilution of urine collected (Ronteltap *et al.* 2010). In addition, development of localised supersaturation in the reactor due to poor mixing and flow dynamics caused by continuous flow of urine resulting in premature flow of urine from the reactor without complete removal of phosphate could be the reason for the reduction of precipitation efficiency (Wilsenach *et al.* 2007). Etter *et al.* (2011) observed an overall efficiency of 90% in the experiments conducted using real urine in a manually stirred batch reactor.

Recovery efficiency

Recovery efficiencies were determined based on the difference in phosphate concentration in the influent and the total soluble phosphate concentration in the effluent. The percentages of recovery efficiencies from the six experiments are 76.1, 79.1, 81.2, 73.7, 73.3 and 75.4 respectively (Figure 2). The percentage of fines lost in these experiments, which is calculated based on the difference in precipitation and recovery efficiency, are in the order of 5.9, 3.5, 3.6, 9.1, 10 and 10.8 respectively. The increase in HRT for the experiments conducted without stirring resulted in a marginal increase in the recovery efficiency. This increase is due to the lesser amount of fines escaping the reactor with effluent. It is reported that an increase in the crystal retention duration was found to support better growth of struvite crystals (Battistoni *et al.* 2002). However, lower recovery efficiencies observed in the stirred experiments could be due to the transfer of power of stirring to the aqueous solution which causes fines to escape along with the effluent (Wilsenach *et al.* 2007). The low specific density of struvite of about 1.7 (Wilsenach *et al.* 2007) and typical formation of smaller struvite particles in the range of 36–136 μm in urine (Ronteltap *et al.* 2010) can affect recovery efficiencies.

Solids and particle size

X-ray diffraction (XRD) examination of the air dried precipitates collected from one of the experiments confirms the presence of struvite (Figure 3). The trapezoidal morphology of struvite observed in the scanning electron microscopy (SEM) picture (Figure 4) resembles the crystal shapes reported by Wilsenach *et al.* (2007) and Münch & Barr (2001), while x-shaped (Ronteltap *et al.* 2010), coffin-like (Wierzbicki *et al.* 1997) and needle-like (Abbona & Boistelle 1985) are also some typical morphologies of struvite. Particles in the range of 10.7–72.2 μm were observed for the experiments conducted with a crystal retention period of 8 hours in the present study. A maximum particle size of 72.2 μm was observed in the stirred experiment with 100 rpm with 2 hour HRT. The higher crystal size observed in the stirred experiment with longer HRT could be due to the secondary crystal growth effected as a result of good stirring (Wilsenach *et al.* 2007).

The particle sizes measured in the experiments are lower than the values reported by Ronteltap *et al.* (2010) for the experiments conducted with CSTR stirred at 300 rpm and a crystal retention period of 4–5 days in the reactor. The authors observed struvite particles with a median diameter of 91 μm . This difference in the particle size can be attributed to the lower crystal retention period with minimal stirrer speed employed in the present study. Le Corre *et al.* (2006) reported that struvite crystals do not agglomerate at higher pH levels due to the negative zeta-potential observed. Stored urine usually has higher pH and has very high buffering capacities due to the higher ammonium concentrations (Stratful *et al.* 2001).

Scale formation

In all the present experiments conducted, scaling on the reactor walls observed was minimal as the experiments were conducted with very mild stirring or without stirring and relatively with shorter

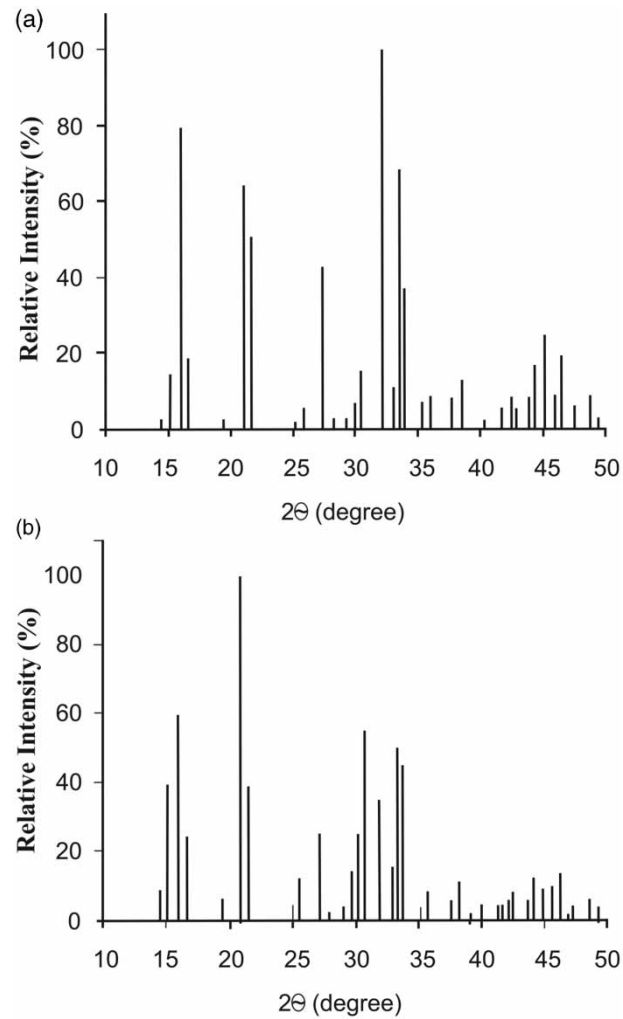


Figure 3 | X-ray diffractogram of the (a) precipitate obtained in the experiment and (b) struvite standard.

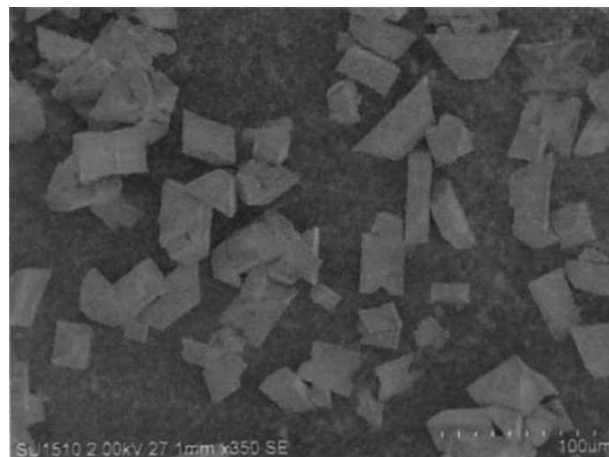


Figure 4 | SEM image of struvite precipitate obtained in one of the experiments.

HRTs. However, the long-term effects of scaling should be investigated, if the operations are to be continuously carried out. Also, scaling on the impeller surface of about 1–3 mm thickness during the stirred experiments and 1–2 mm thickness on the surface of sloping channel used for experiments conducted without stirring observed requires attention. As reported in the earlier studies (Wilsonach

et al. 2007; Abegglen 2008; Ronteltap *et al.* 2010; Etter *et al.* 2011), formation of scales on various components of the reactor can pose operational problems during struvite recovery process. Ronteltap *et al.* (2010) observed scaling on the reactor walls and a layer of 2 cm thick struvite deposit formed on the bottom surface of the reactor after a runtime of 227 days. Higher mixing speeds were found to cause increased scaling on the reactor surfaces (Wilsenach *et al.* 2007). Abegglen (2008) reported that scaling could be prevented if the sedimentation time was shorter than four hours. Etter *et al.* (2011) observed that shorter HRT reduced the scaling problems. The reduced effect of scaling reported by the authors, while operating the reactor in a batch processing mode, could also be due to the periodic recovery of precipitates carried out by emptying the reactor. Other recommended suggestions for preventing scaling in the past include: reducing supersaturation during struvite precipitation and avoiding rough surfaces in the reactor (Ohlinger *et al.* 1998); use of PVC and acrylic materials for the reactors components (Mohajit *et al.* 1989); and masking of reactor surfaces with removable plastic film (Wilsenach *et al.* 2007); seeding of reactor with struvite particles to promote growth of particles (Agarwal *et al.* 2019).

Economics of struvite production

In terms of operation and maintenance, the reactor designed in this study was found to be easy to operate. The continuous flow operation results in minimising the overall operation cost of struvite production. In order to compare the performance of the reactor, the results reported by Etter *et al.* (2011) for a manually stirred reactor of 500 litres capacity in Nepal was utilised. For this purpose, the selling price of struvite worked out using linear regression model (Equation (1)) is Rs. 33.01 kg⁻¹ (Tilley *et al.* 2009). Table 3 shows the calculations worked to arrive at the selling prices of struvite based on the current fertiliser prices in India (Indiastat 2020). The maximum recovery efficiency of 81.2% observed in experiment no. 3 conducted with 2 hour HRT and without stirring is taken into consideration for the calculations.

$$\text{Struvite Retail Price} = FV_{N:iN} + FV_{P:iP} + FV_{K:iK} + FV_{Mg:iMg} + FV_{S:iS}[\text{NPR} \cdot \text{kg}^{-1}] \quad (1)$$

where *ix* is the content of the nutrient in the fertiliser in (kgx·kg⁻¹) and FV_x is the fitted financial value of the respective nutrient. To calculate the financial value of struvite as a fertiliser, the constants FV_x were multiplied with the nutrient concentrations of struvite, which are 57 g N·kg struvite⁻¹, 12.6 g P·kg struvite⁻¹ and 99 g Mg·kg struvite⁻¹.

For the purpose of direct comparison, calculation for operations using a 500 litre capacity reactor was worked out based on the operational parameters of the 15 litres capacity reactor used in this study (Table 4). The analysis shows that struvite production can be made profitable using the reactor developed. The profit worked out for sites with minimum transportation of bittern for a minimum distance of 50 km and a maximum distance of 2,000 km is about Rs.21,048/- and Rs.7,048/- per annum respectively. However as a trade-off, if experiments with shorter HRT are chosen for analysis, instead of choosing higher recovery efficiencies, the profit margins can be further increased. For example, considering recovery efficiencies 76.1 or 79.1% observed in experiments conducted without stirring with 0.5 or 1 hour HRT respectively would increase the profit by three or two folds respectively due to higher volume of urine processed and struvite recovered for a given time. However, the manually stirred reactor of similar capacity developed by Etter *et al.* (2011) used for struvite recovery in Nepal failed to meet the operations costs due intensive labour operations required. The cost of collection and transportation of urine to the site has not been included as it can make struvite production from source-separated urine uneconomical (Etter *et al.* 2011). Struvite production from urine should not be taken into consideration purely from the point of economics of its recovery, but it should be viewed from the impact such projects will have on the environment and sustainability of

Table 3 | Prices of fertiliser and calculation of selling price of struvite

| Type of Fertiliser, Nutrients and Price | N | P ₂ O ₅ | K ₂ O | Mg | S | Price [INR/kg] |
|---|------|-------------------------------|------------------|---|-------------------------------|-----------------------|
| Complex Fertiliser | 20 | 20 | 0 | 0 | 0 | 19.00 |
| Complex Fertiliser | 24 | 24 | 0 | 0 | 0 | 24.40 |
| Complex Fertiliser | 28 | 28 | 0 | 0 | 0 | 25.51 |
| Complex Fertiliser | 10 | 26 | 26 | 0 | 0 | 24.34 |
| Complex Fertiliser | 12 | 32 | 16 | 0 | 0 | 24.54 |
| Complex Fertiliser | 14 | 35 | 14 | 0 | 0 | 25.73 |
| Complex Fertiliser | 15 | 15 | 15 | 0 | 0 | 21.20 |
| Complex Fertiliser | 17 | 17 | 17 | 0 | 0 | 25.00 |
| Diammonium Phosphate | 18 | 46 | 0 | 0 | 0 | 42.00 |
| Diammonium Phosphate (Imported) | 18 | 46 | 0 | 0 | 0 | 25.10 |
| Ammonium Sulphate | 20.6 | 0 | 0 | 0 | 23 | 13.17 |
| Ammonium Phospho Sulphate | 16 | 20 | 0 | 0 | 13 | 19.05 |
| Ammonium Phospho Sulphate | 20 | 20 | 0 | 0 | 13 | 20.09 |
| Muriate of Potash | 0 | 0 | 60 | 0 | 0 | 18.98 |
| Magnesium Sulphate | 0 | 0 | 0 | 9.9 | 13 | 12.00 |
| Calculation of Selling Price of Struvite Using Linear Regression Model | | | | Price/kg^a | Subsidy/kg^b | Total Price/kg |
| Nitrogen N | | | | 51.64 | 18.9 | 70.54 |
| Phosphorus P | | | | 47.92 | 15.21 | 63.13 |
| Potassium K | | | | 30.85 | 11.12 | 41.97 |
| Magnesium Mg | | | | 109.66 | 0 | 109.66 |
| Sulfur S | | | | 8.79 | 3.56 | 12.35 |
| Struvite (N:5.7%, P:28.9%,Mg:9.8%) | | | | Total price including subsidy based on N, P & Mg price calculated above | | 33.01 |

^aCalculated based on the current fertiliser prices in the market (Indiastat 2020).

^bFertiliser subsidy provided by the Government of India.

resources. Decentralised operations with low technological inputs can potentially reduce the operation costs and can provide opportunities to small communities and institutions to recover struvite using urine (Wilsenach *et al.* 2007).

SUMMARY

The following conclusions can be drawn from experiments conducted using the continuous up-flow struvite reactor developed in this study:

- Around 85% precipitation and 81% recovery efficiencies of phosphate by the continuous up-flow struvite reactor designed in study was recorded.
- The experiments conducted reveal that precipitation of struvite from urine using bittern can be conducted without the need for external mixing of the aqueous solutions in the reactor. Experiments conducted with 100 rpm mixing have shown higher loss of the fines precipitated to the effluent.
- The XRD analyses confirm that the precipitates obtained in the experiments are struvite. Morphology and the particle size studied are in agreement with previous studies.
- Scale formation on the components of the reactor was minimal due to shorter HRTs and relatively mild stirring employed, however this could pose problems during continuous operations. Periodic cleaning of the reactor components after recovery of struvite is necessary to reduce the scaling problem.

Table 4 | Economics of struvite production

| Description | Low-cost reactor developed in Nepal (Etter et al. 2011) ^a | Continuous up-flow reactor developed in this study ^a |
|--|--|--|
| 1. Operation parameters | | |
| Reactor operation | Filtration & Manual Stirring | Sedimentation + Filtration & No stirring |
| Reactor capacity | 500 L | 500 L |
| Cycles per day | 8 D ⁻¹ | Continuous (2hrs HRT-24 hrs) |
| Struvite recovery efficiency | 90% | 81% |
| Yearly struvite production (250 workdays) | 1,400 kg | 1,893 kg |
| Molar Mg:P ratio | 1.1 molMg-mol P ⁻¹ | 1.5 molMg-mol P ⁻¹ |
| 2. Installation costs | (based on inflation rates ^b) | (based on current prices in India) |
| Steel tank for reactor | Rs.17,023 | Rs. 30,000 |
| Additional tanks, fittings, pipes etc. | Rs.31,537 | Rs. 50,000 |
| Building | Rs.35,867 | Rs. 40,000 |
| Total | Rs.82,427 | Rs.1,20,000 |
| Operation duration | 10 years | 10 Years |
| Amortisation (per annum) | Rs.8,243 | Rs.12,000 |
| Capital costs (12% per annum) | Rs.990 | Rs.1,440 |
| 3. Expenses | | |
| Amortisation and capital costs (per annum) | Rs.5,150 | Rs.13,440 |
| Labour charges (per annum) | Rs.1,12,890 | Rs.27,000 ^c |
| Bittern price (per annum) | Rs.34,763 | Rs.1,000–15,000 ^d |
| Total (per annum) | Rs.1,52,803 | Rs.41,440–55,440 |
| 4. Revenue | | |
| Sale of struvite (per annum) | Rs.46,124 ^e | Rs.62,488 ^e |
| 5. Profit (per annum) | A loss of Rs.1,06,679 is incurred | Operation can be performed without making a loss. Profit for sites with minimum transportation of bittern for a distance of 50 km and a maximum distance of 2,000 km is about Rs.21,048/- and Rs.7,048/- per annum respectively. |

^aAll financial costs are given in Indian Rupees. Nepali rupee is converted using 1NRs = 0.63 INRs.

^bBased on total inflation rate of 79.19% from 2011 to 2020 in India has been considered for calculations.

^cLabour charges for two days per week @Rs.300 D⁻¹ for 45 weeks.

^dBittern transportation price @ Rs.3 per ton/km for transporting 1.5 ton of bittern (distance taken for calculation minimum: 50 km @ Rs.1,000/- and maximum 2,000 km @ Rs.15,000/- per annum).

^eStruvite price worked out for India based on current fertiliser rate using linear regression Rs.33.01 kg⁻¹.

- Struvite can be produced economically in decentralised operations using the continuous flow reactor developed in this study due to lower operational costs.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

REFERENCES

- Abbona, F. & Boistelle, R. 1985 Nucleation of struvite: single crystals and agglomerates. *Crystal Research and Technology* **20** (2), 133–140.
- Abe, S. 1995 Phosphate removal from dewatering filtrate by MAP process at Seibu treatment plant in Fukuoka City. *Sewage Works in Japan* 59–64.
- Abegglen, C. K. 2008 *Membrane Bioreactor Technology for Decentralized Wastewater Treatment and Reuse*. PhD thesis, Nr. 17998, Swiss Federal Institute of Technology Zurich, Switzerland.
- Adnan, A., Dastur, M., Mavinic, D. S. & Koch, F. A. 2004 Preliminary investigation into factors affecting controlled struvite crystallization at the bench scale. *Journal of Environment Engineering and Science* **3** (3), 195–202.
- Agarwal, S., Guest, J. S. & Cusick, R. D. 2019 Elucidating the impacts of initial supersaturation and seed crystalloading on struvite precipitation kinetics, fines production, and crystal growth. *Water Research* **132**, 252–259.
- Antonini, S., Paris, S., Eichert, T. & Clemens, J. 2011 Nitrogen and phosphorus recovery from human urine by struvite precipitation and Air stripping in Vietnam. *CLEAN – Soil, Air, Water* **39** (12), 1099–1104.
- Barbosa, S. G., Peixoto, L., Meulman, B., Alves, M. M. & Pereira, M. A. 2016 A design of experiments to assess phosphorous removal and crystal properties in struvite precipitation of source separated urine using different Mg sources. *Chemical Engineering Journal* **298**, 146–153.
- Battistoni, P., de Angelis, A., Prisciandaro, M., Boccadoro, R. & Bolzonella, D. 2002 P removal from anaerobic supernatants by struvite crystallization: long term validation and process modelling. *Water Research* **36** (8), 1927–1938.
- Burns, R. T., Moody, L. B., Walker, F. R. & Raman, D. R. 2001 Laboratory and in-situ reductions of soluble phosphorus in liquid swine waste slurries. *Environmental Technology* **22** (11), 1273–1278.
- Chimenos, J. M., Fernandez, A. I., Villalba, G., Segarra, M., Urruticoechea, A., Artaza, B. & Espiell, F. 2003 Removal of ammonium and phosphates from wastewater resulting from the process of cochineal extraction using MgO containing by-product. *Water Research* **37** (7), 1601–1607.
- Demirer, S. U., Demirer, G. N. & Chen, S. 2005 Ammonia removal from anaerobically digested dairy manure by struvite precipitation. *Process Biochemistry* **40**, 3667–3674.
- Etter, B., Tilley, E., Khadka, R. & Udert, K. M. 2011 Low-cost struvite production using source-separated urine in Nepal. *Water Research* **45** (2), 852–862.
- Indiastat 2020 *Grade-wise Maximum Retail Price of Fertilizers in India*. Available from: <https://www.indiastat.com/table/agriculture-data/2/fertiliser-prices/17825/1330716/data.aspx> (accessed 5 May 2020).
- Kabdaşlı, T. 2018 Nutrient recovery by struvite precipitation, ion exchange and adsorption from source-separated human urine – a review. *Environmental Technology Reviews* **7** (1), 106–138.
- Larsen, T. A. & Gujer, W. 1996 Separate management of anthropogenic nutrient solutions (human urine). *Water Science and Technology* **34** (3–4), 87–94.
- Le Corre, K. S., Valsami-Jones, E., Hobbs, P., Jefferson, B. & Parsons, S. A. 2006 Agglomeration of struvite crystals. *Water Research* **41** (2), 419–425.
- Mohajit, K., Bhattarai, E., Taiganides, P. & Yap, B. C. 1989 Struvite deposits in pipes and aerators. *Biological Wastes* **30**, 133–147.
- Münch, E. v. & Barr, K. 2001 Controlled struvite crystallization for removing phosphorus from anaerobic digester side streams. *Water Research* **35** (1), 151–159.
- Ohlinger, K. N., Young, T. M. & Schroeder, E. D. 1998 Predicting struvite formation in digestion. *Water Research* **32** (12), 3607–3614.
- Pastor, L., Mangin, D., Barat, R. & Seco, A. 2008 A pilot-scale study of struvite precipitation in a stirred tank reactor: conditions influencing the process. *Bioresource Technology* **99** (14), 6285–6291.
- Ronteltap, M., Maurer, M. & Gujer, W. 2007 Struvite precipitation thermodynamics in source-separated urine. *Water Research* **41** (5), 977–984.
- Ronteltap, M., Maurer, M., Hausherr, R. & Gujer, W. 2010 Struvite precipitation from urine – influencing factors on particle size. *Water Research* **44** (6), 2038–2046.
- Sakthivel, S. R., Tilley, E. & Udert, K. M. 2012 Wood ash as a magnesium source for phosphorus recovery from source-separated urine. *Science of the Total Environment* **419**, 68–75.
- Shin, H. S. & Lee, S. M. 1997 Removal of nutrients in wastewater by using magnesium salts. *Environmental Technology* **19** (3), 283–290.
- Stratful, I., Scrimshaw, M. D. & Lester, J. N. 2001 Conditions influencing the precipitation of magnesium ammonium phosphate. *Water Research* **35** (17), 4191–4199.
- Tilley, E., Gantenbein, B., Khadka, R., Zurbrugg, C. & Udert, K. M. 2009 Social and economic feasibility of struvite recovery from Urine at the community level in Nepal. In: *International Conference on Nutrient Recovery From Wastewater Streams* (Mavinic, D., Ashley, K. & Koch, F., eds). Vancouver, IWA Publishing, London, UK, pp. 169–178.

- Udert, K. M. & Wächter, M. 2012 Complete nutrient recovery from source-separated urine by nitrification and distillation. *Water Research* **46** (2), 453–464.
- Udert, K. M., Larsen, T. A. & Gujer, W. 2003 Estimating the precipitation potential in urine-collecting systems. *Water Research* **37** (11), 2667–2677.
- Ueno, Y. & Fujii, M. 2001 Three years experience of operating and selling recovered struvite from full-scale plant. *Environment Technology* **22** (11), 1373–1381.
- Wierzbicki, A., Sallis, J. D., Stevens, E. D., Smith, M. & Sikes, C. S. 1997 Crystal growth and molecular modeling studies of inhibition of struvite by phosphocitrate. *Calcified Tissue International* **61**, 216–222.
- Wilsenach, J. & van Loosdrecht, M. 2003 Impact of separate urine collection on wastewater treatment systems. *Water Science and Technology* **48** (1), 105–110.
- Wilsenach, J. A., Schuurbiers, C. A. H. & van Loosdrecht, M. C. M. 2007 Phosphate and potassium recovery from source separated urine through struvite precipitation. *Water Research* **41** (2), 458–466.
- Zeng, L. & Li, X. 2006 Nutrient removal from anaerobically digested cattle manure by struvite precipitation. *Journal of Environmental Engineering and Science* **5** (4), 285–294.

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