

Combined seawater toilet flushing and urine separation for economic phosphorus recovery and nitrogen removal: a laboratory-scale trial

H. R. Mackey, Y.-S. Zheng, W.-T. Tang, J. Dai and G.-H. Chen

ABSTRACT

Freshwater toilet flushing consumes 20–35% of typical household water demand. Seawater toilet flushing, as practised by Hong Kong since 1958, provides an alternative water source. To maximise the benefits of this unique dual water supply, urine separation could be combined to allow low-cost struvite production and subsequent urine nitrification – in-sewer denitrification. This paper reports on a laboratory-scale study of seawater urine phosphate recovery (SUPR) and seawater–urine nitrification. A laboratory-scale SUPR reactor was run under three phases with hydraulic retention time between 1.5 and 6 h, achieving 91–96% phosphorus recovery. A urine nitrification sequencing batch reactor (UNSBR) was also run for a period of over 650 days, averaging 90% ammonia removal and loading of up to 750 mg-N/L.d. Careful control of the SUPR phosphate removal was found necessary for operation of the downstream UNSBR, and system integration considerations are discussed.

Key words | magnesium ammonium phosphate (MAP), seawater toilet flushing, source separation, struvite precipitation, urine nitrification

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ABBREVIATIONS

ACP	amorphous calcium phosphate
HAP	hydroxyapatite
HRT	hydraulic retention time
MLVSS	mixed liquor volatile suspended solids
NLR	nitrogen loading rate
OCP	octacalcium phosphate
SI	saturation index
SP	soluble phosphorus
SRP	soluble reactive phosphorus
SUPR	seawater urine phosphate recovery
SWTF	seawater toilet flushing
TAN	total ammoniacal nitrogen
TCOD	total chemical oxygen demand
TN	total nitrogen
TP	total phosphorus
UNSBR	urine nitrifying sequencing batch reactor
XRD	X-ray diffraction

INTRODUCTION

Through rapid urbanisation and the age of megacities increased pollution, competition for water resources and energy minimisation have become leading issues. In many water-scarce regions seawater desalination has been chosen to overcome water issues. However, in most cities using this technology, toilets are flushed with high quality water which has been reported to account for 20 to 35% of typical household freshwater demand (Crook & Rimer 2009). As an alternative water resource, Hong Kong has practised seawater toilet flushing (SWTF) since 1958, serving 80% of the population. While implementation of dual pipelines is costly, power demands of SWTF are only 0.013–0.025 kWh/m³ (Leung *et al.* 2012), significantly better than desalination, 2.4–3.6 kWh/m³, or reclaimed water, 0.4–2.0 kWh/m³ (Desalination Committee 2011). In addition, only minimal treatment involving screening and electrochlorination is required before pumping to storage reservoirs. The overall result is that SWTF continues to be a more economical means of saving freshwater than use of

reclaimed water for new areas in Hong Kong based on life cycle cost analysis, despite the high cost of installing dual pipelines (Tang *et al.* 2006). Given around two-thirds of the world's population live within 150 km of the ocean (Hinrichsen 1996), typically in dense urban settlements, the use of SWTF is a widely applicable solution for freshwater scarcity, which should also be considered when looking at alternative water sources. For further information on advantages, disadvantages and engineering considerations of SWTF the reader is referred to Tang *et al.* (2007) and Leung *et al.* (2012).

Urine separation is another technology providing sustainable solutions for densely populated urban cities where urine collection is cost-effective. The motivation for urine separation is efficient removal and recovery of nutrients since urine contains around half of the phosphorus and the majority of nitrogen and potassium in municipal wastewater. Numerous studies have investigated ways of efficiently extracting these nutrients, which have been summarised by Maurer *et al.* (2006). Removal and recovery of phosphorus as struvite has proved popular, typically using magnesium (Mg) oxide or salts as precipitants. However, handling of such salt solutions is not optimal for decentralised treatment systems, and struvite precipitation alone does not remove nitrogen from urine. As eutrophication of coastal waterways is a prevalent and growing issue for densely populated coastal cities (Diaz & Rosenberg 2008), the strongest motivation for urine separation should be to provide efficient and cost-effective nutrient removal. In view of these issues and to maximise the benefits of SWTF and urine separation, a

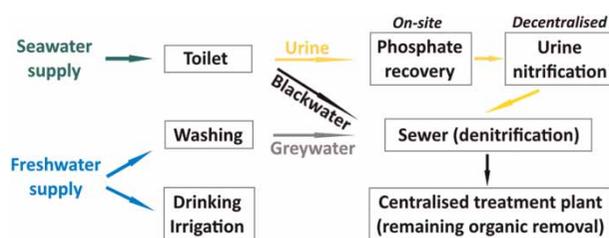


Figure 1 | Schematic of the proposed seawater–urine decentralised treatment scheme. Phosphate recovery would take place at building level, urine nitrification would serve a block of apartments (for high-rise urban area).

combined system with seawater urine phosphate recovery (SUPR), utilising the magnesium in seawater, combined with nitrified urine sewer discharge technology (Jiang *et al.* 2011) is proposed (Figure 1). This combined system allows the possibility of downsizing nutrient removal requirements at centralised plants (Wilsenach & van Loosdrecht 2003) while also providing sewer sulphide control.

Although phosphate precipitation using seawater and other alternative magnesium sources has received attention previously due to its economy (Kumashiro *et al.* 2001; Crutchik & Garrido 2011; Etter *et al.* 2011), each system is specific due to different saturation of various compounds, and as such the engineering feasibility study of the SUPR system is warranted. Furthermore, despite a number of studies on urine nitrification (Udert *et al.* 2003a; Oosterhuis & van Loosdrecht 2009; Sun *et al.* 2012) no study has investigated urine post struvite precipitation or high salinity nitrification of a urine–seawater mixture. This paper therefore investigates the performance and integration of urine struvite precipitation and nitrification, and the products formed through phosphate recovery, in a seawater-based sewerage system.

MATERIALS AND METHODS

Urine collection

Urine for the SUPR reactor was collected in sterilised bottles from a group of 20 healthy males, promptly cooled to 4 °C and used the same day by mixing with flushing seawater collected from a urinal flush nozzle. For a small period of the experiment SUPR effluent was sufficiently available to feed straight to the urine nitrification sequencing batch reactor (UNSBR). At other times a synthetic SUPR effluent was prepared from the supernatant of stored hydrolysed urine (stored at 4 °C for a number of weeks) and seawater after mixing and settling. The supernatant was used as the influent for the UNSBR with similar properties to the real SUPR effluent including phosphorus levels (Table 1).

Table 1 | Influent conditions for UNSBR – abbreviations given in 'Analytical methods' section

Influent	TAN mg-N/L	TN mg/L	TCOD mg/L	TP mg/L	SP ^a mg/L	Cl ⁻ mg/L	SO ₄ ²⁻ mg-S/L
Hydrolysed urine + seawater	1,268 (168)	1,468 (213)	972 (184)	2.58 (2.55)	0.88 (0.39)	13,647 (902)	682 (49)
SUPR effluent (day 399–524)	1,482 (197)	1,619 (145)	1,043 (163)	1.95 (0.65)	0.87 (0.39)	12,468 (1045)	648 (71)

^aSP – soluble phosphorus. Values in brackets show standard deviation.

SUPR reactor setup and operation

Two continuous-flow SUPR reactors were constructed from polymethylmethacrylate with an effective volume of 840 mL. An internal concentric cylinder divided the reactors into a hydrolysis–precipitation zone and a settling–separation zone (Figure 2). Influent entered the top of the middle cylinder and effluent was collected from a pipe into the outer cylinder acting as a weir. A 45- μm nylon mesh was used to empty and dewater the solids from the bottom settling cone every 10–14 days by gravity. Due to clogging issues in the annulus of the first reactor, the external diameter was increased on the second reactor from 55 to 66 mm with a corresponding reduction in wall height to maintain the effective volume. This resulted in a decreased surface area from 0.194 to 0.118 m². During phase I (Reactor 1) 50% urine at a hydraulic retention time (HRT) of 6 h was applied. For phase II and III (Reactor 2) the HRT was reduced to 3 and 1.5 h respectively with urine fractions of 50 and 25%. This gave phase 1 a urine loading rate of 1.68 L/d and phase II and III a rate of 3.36 L/d. In phase I and II a 50% urine mixture was recirculated within the reactors for 1 week during start-up. A table of various parameters over each phase is available in the online supporting information (available at <http://www.iwaponline.com/wst/070/335.pdf>).

Urine nitrifying SBR reactor setup and operation

The UNSBR had a diameter of 102 mm, decanted volume of 1.1 L and an exchange volume of 45 to 75% dependent

on influent loading conditions. Seed sludge was from an activated sludge plant treating brackish wastewater and cultivated on a 25% urine–seawater mixture. Aeration was through fine bubble diffusers at a flow rate of 0.5 L/min and was continuous except during settling. Initially, due to resource limitations, the reactor was manually operated by feeding influent containing sufficient alkalinity (and precipitated phosphorus) in batch mode until the pH had decreased to a stable value. From day 55 onwards this resulted in an average of three feedings of 425 mL/day. At day 160 a timer control system was installed to allow consistent monitoring, operated with a 24-h cycle, four influent additions of less than 5 min at 6 h intervals, and a settling period of 8–15 min. Only the supernatant (excluding precipitate) of the synthetic SUPR effluent was used from this point on and did not contain added alkalinity. Alkalinity was instead dosed in the form of 1 M NaHCO₃ when the pH decreased to 5.5 using a pH controller. This set point was increased to pH 6.5 and 7 at days 192 and 507. Influent conditions for the reactor are shown in Table 1. Changes to operation and major reactor parameters are summarised in the online supporting information.

Analytical methods

Unless noted, analytical methods were in accordance with the standard methods (APHA 2005). Phosphorus was measured by ascorbic acid method after persulfate digestion (APHA 2005). Total ammoniacal nitrogen (TAN)

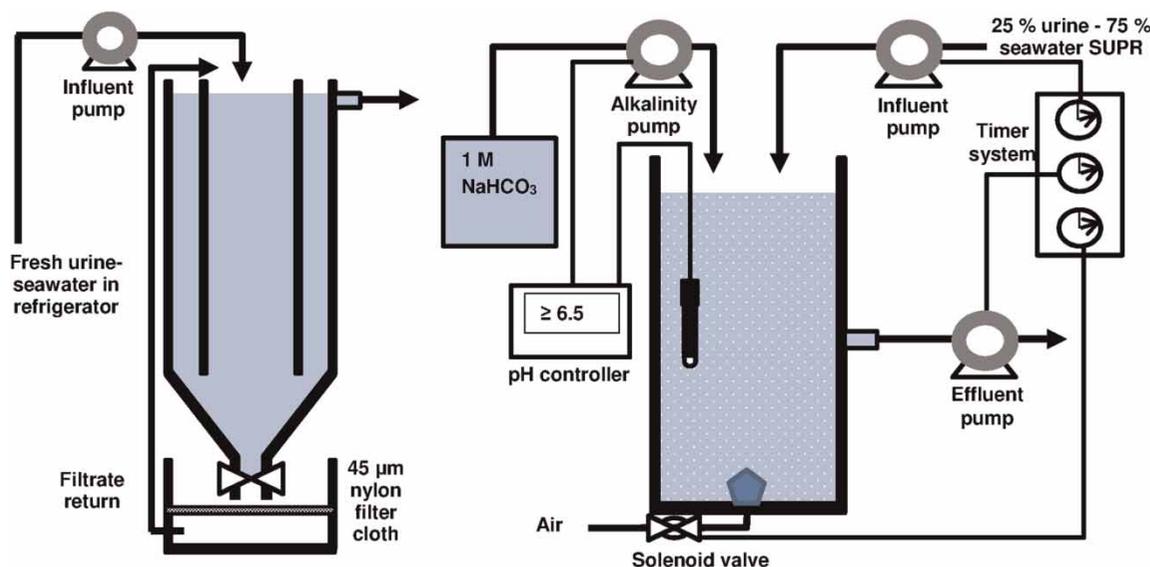


Figure 2 | Schematic of SUPR reactor (left) and UNSBR (right).

was measured by flow injection analysis (FIA; Quick-Chem 8500 Series II, Lachat); total organic carbon (TOC) and total soluble nitrogen (TN) were analysed using a total carbon analyser with total nitrogen module (TOC-VCPH plus TNM-1, Shimadzu). Anions were analysed using ion chromatography (LC-20A Super, Shimadzu), except nitrite which was measured by FIA when chloride interference was significant (low nitrite concentrations). Total chemical oxygen demand (TCOD) was determined with Hach test kits while magnesium and calcium were determined by atomic adsorption spectroscopy (SpectrAA-220FS, Varian). Mixed liquor volatile suspended solids (MLVSS) were determined in accordance with the standard methods (APHA 2005) and pH by a multimeter (Multi3420 with Sentix 940-3 probes, WTW). Samples were filtered (0.45 μm) except for total phosphorus (TP), total reactive phosphorus and TCOD. Biomass samples analysed for P content were first digested using a Hach Digesdahl[®] apparatus following the manufacturer's instructions.

SUPR product samples were vacuum filtered (GFC glass fibre) and dried at 45 °C for 48 h before subsequent analysis. Product for chemical analysis was dissolved in 100 mL 1 N HCl. X-ray diffraction (XRD; PW 1830, Philips) was conducted using monochromatic Cu K α 1 radiation through the 2θ range 5–70° in increments of 0.033°. Spectra mapping was done using Jade5.

UNSBR phosphorus batch test

On day 559 a batch test to confirm the influence of phosphorus deficiency on nitrifying activity was conducted. Sludge was taken from the UNSBR and washed with 75% seawater. Sludge samples were prepared at 0, 0.2 and 4.0 mg-P/L in quadruplet and controls at 0 and 4.0 mg-P/L in duplicate. Volatile suspended solids (VSS; 12.3 mg), 2.19 mL of urine and 1 mL of 1 M NaHCO₃ were added to 100-mL plastic containers before diluting to 35 mL with 75% seawater. All

samples were initially sealed to minimise oxygen transfer and placed on a shake table until 27 h to ensure carbon removal. At 6.5, 15.5 and 27.5 h the first set of sample duplicates were tested to confirm carbon removal and absence of nitrification. At 27 h the caps were taken off the remaining duplicate samples and the controls. These were sampled at 72 h to determine the degree of nitrification. All samples were analysed for TN, TAN, TOC, nitrite and nitrate.

RESULTS AND DISCUSSION

SUPR system

Reactor 1 was operated initially at a HRT of 6 h and a urine fraction of 50%. During this stage an average removal efficiency of 96% TP was achieved from the urine–seawater mixture (Figure 3) with an average effluent TP of 6.25 mg/L (Table 2). However, it was noted that a gradual clogging between the external and baffle walls was occurring. Therefore, for phase II and III a new reactor with the same effective volume but a slightly larger external diameter (but smaller height and surface area) was used. During phase II and phase III TP removal efficiencies of 94 and 91% were achieved at HRT and urine fractions of 3 h, 50% and 1.5 h, 25% respectively. Effluent TP concentrations were in the order of 10 mg/L for both phases. Rate-limiting urea hydrolysis (Udert *et al.* 2003b; Liu *et al.* 2008) appeared near complete at all HRTs tested, showing the rapid hydrolysis capability of the SUPR reactor.

Saturation indices (SIs; Udert *et al.* 2003b) over the operating pH range for possible phosphate solid phases were determined in Visual Minteq (supporting information, available online at <http://www.iwaponline.com/wst/070/335.pdf>). Amorphous calcium phosphate (ACP), octacalcium phosphate (OCP), hydroxyapatite (HAP) and struvite reached SIs greater than 1. Although OCP and HAP supersaturation was the highest, their transformation from ACP

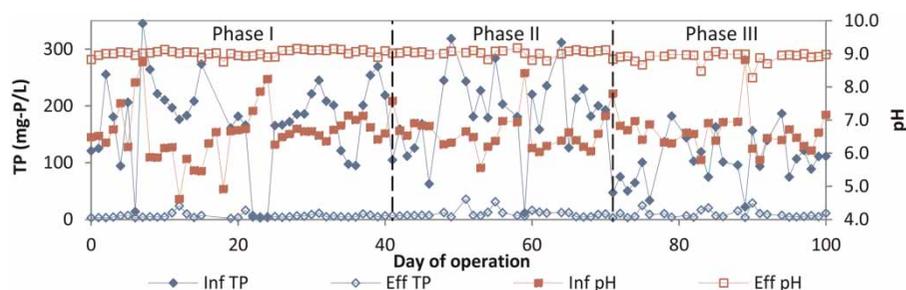


Figure 3 | Operation of SUPR reactor over three phases.

Table 2 | Summary of the SUPR reactor performance

Phase	Type	pH	TN mg/L	TAN mg-N/L	TP mg/L	Mg mg/L	Ca mg/L	Hydrolysis %
I	Influent	6.59	3,920 ^b	462	174	545	274	11.8
	Effluent	9.02	3,900	3,520	6.25	413	34.6	90.3
II	Influent	6.59	3,470	235	187	572	301	6.8
	Effluent	9.03	3,380	2,840	10.6	409	84.6	84.0
III	Influent	6.66	1,750	143	104	826	374	8.1
	Effluent	8.88	1,740	1,627	9.27	692	177	93.5

should be inhibited by the high magnesium to calcium ratio as well as by carbonate and urine organics (Boskey & Posner 1974). ACP showed similar saturation for all phases of operation while struvite was greatest for phase I and lowest for phase III. Differences in struvite saturation were strongly influenced by the available TAN in the system at lower pH. This is a possible factor for the reduced removal efficiencies in phase II and III, although washout of fine crystal particles at the reduced HRTs was also a contributing factor. Both magnesite and calcite were also supersaturated in all phases of operation. The SI at pH 9 for ACP was 2.8–3.1 and for struvite was 2.8–3.1 (using effluent TAN measurements).

Wet chemical analysis of the SUPR products showed similar molar ratios of TAN, Mg and Ca with Mg slightly greater than TAN (Figure 4). Mg showed an increase in concentration during phase III as a result of the higher proportion of seawater used. As the combined molar ratio of cations to P was in the range of 1.5–2.0 calcite, magnesite or a combined Mg-Ca carbonate should exist in the precipitate. This cation ratio was significantly higher during phase III indicating increasing carbonate precipitation with increased seawater fraction. The measured fractions give an N:P fertiliser rating of approximately 20:4. XRD analysis

of all products showed strong struvite-like spectra and minor monohydrate calcite and calcite spectra, similar to results of Malone & Towe (1970) investigating precipitation in seawater cultures from nutrient broth. Phase III showed the highest degree of noise (Figure 5). No strong amorphous spectra with broad peaks at 25° 2θ were observed, indicating ACP was only present in small quantities if at all (Zhao *et al.* 2011). This was consistent with faster formation rates of struvite to ACP (Ekama *et al.* 2006) and SIs, indicating struvite would first precipitate as hydrolysis proceeded and pH increased (online supporting information).

UNSBR performance

The UNSBR treating a 25% urine and 75% seawater mixture showed good nitrification performance with substantial periods of complete ammonia removal at nitrogen loading rates (NLR) between 0.5 and 0.75 g-N/L.d. Complete nitrification was observed for substantial periods including the operation on real, rather than synthetically produced, SUPR effluent despite chloride concentrations of around 13,000 mg/L (Figure 6). These periods of satisfactory performance were disrupted by a number of

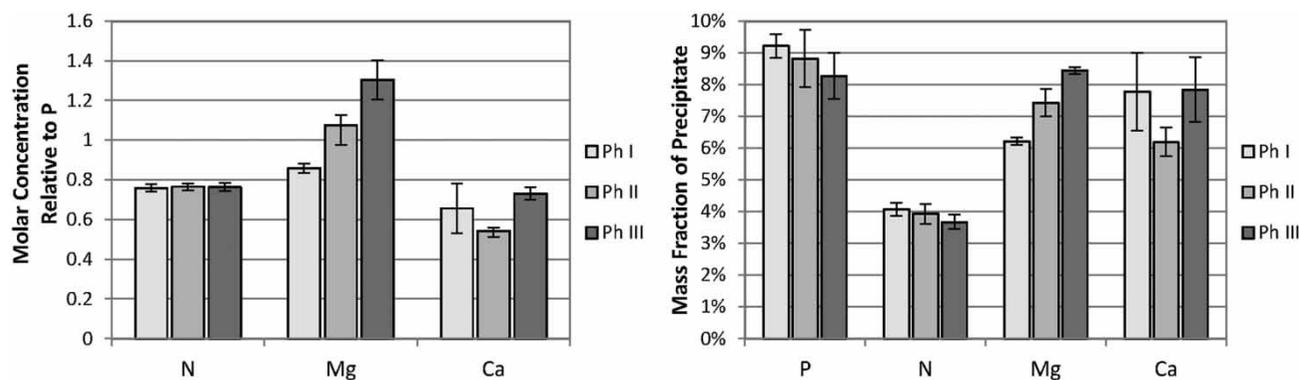


Figure 4 | Molar ratios of product relative to phosphorus from each SUPR operation phase (left) and mass fractions of total precipitate (right) determined from HCl dissolved precipitate. Error bars show standard deviation.

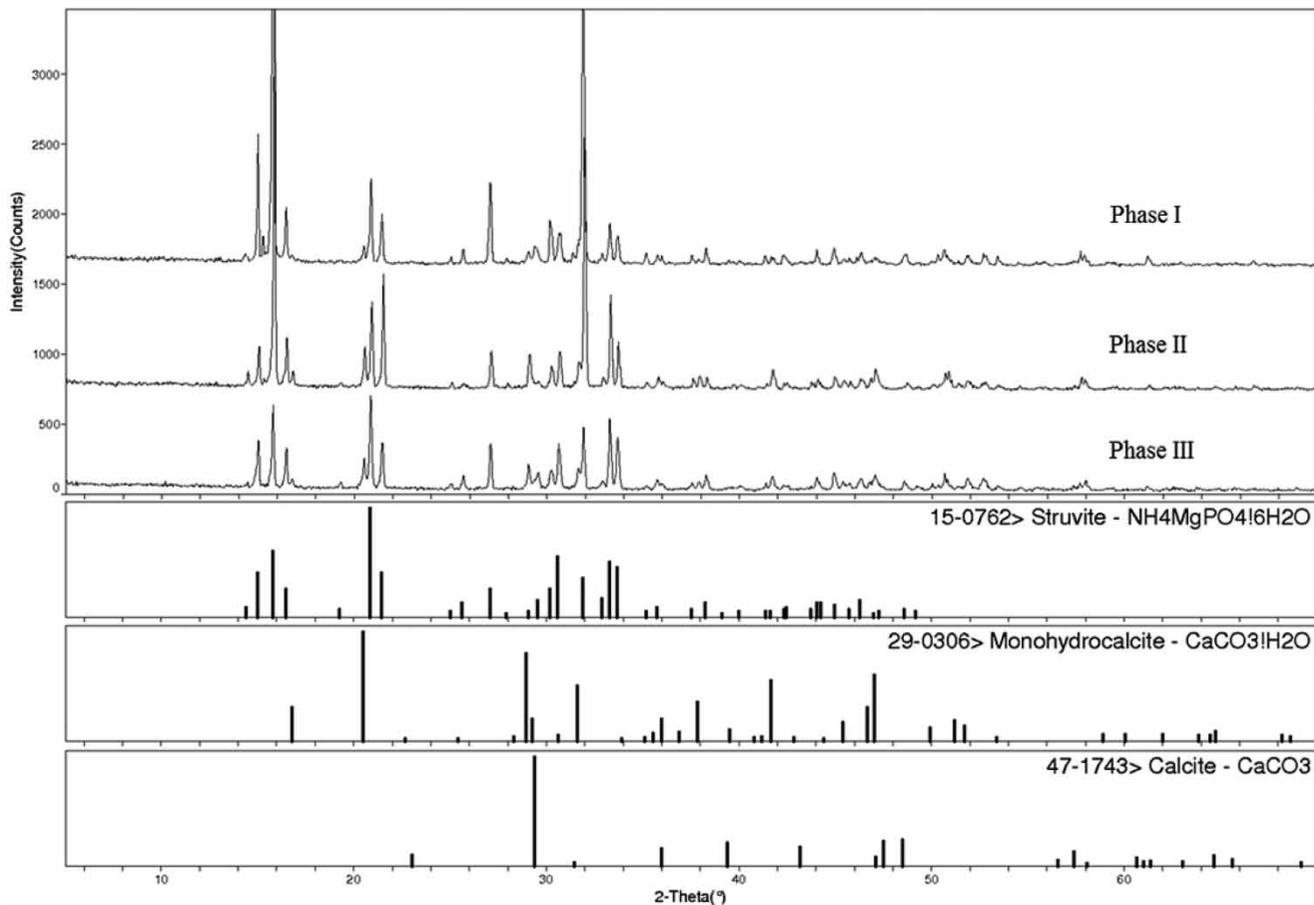


Figure 5 | XRD profiles for SUPR reactor products (top), with line profiles of major-phase struvite, and minor-phases monohydrocalcite and calcite below.

alkalinity dosing failures and two periods of clogged aeration diffusers due to the low air flow rate. However, in periods of sustained treatment performance it was observed that a gradual decrease in treatment performance would also occur. This was first observed from day 200 onwards where a gradual decrease in TAN removal occurred. A single addition of phosphorus (0.39 g, 1:10 ratio of N in reactor) at day 231 provided an immediate but short-lived recovery. Similar additions at day 298 (0.32 g) and 330 (0.21 g) also showed small but brief improvements. The high NLR over this same period between days 200 and 325 was believed to contribute to the decline in treatment performance through a gradual build-up of free ammonia in the reactor.

Real SUPR effluent was fed to the reactor for a period of 125 days during which TAN removal averaged 99.7% (excluding periods of mechanical failure) at an average NLR of 0.71 g-N/L.d. Despite this, a consistent decline in reactor biomass was observed, which had begun immediately after changing to a fixed controller system

and phosphorus-deficient influent at day 160 (Figure 6). Routine phosphorus measurements from day 470 onwards (online supporting information) showed that the effluent soluble reactive phosphorus (SRP) was on average 0.25 mg/L and soluble phosphorus (SP) was 0.39 mg/L. Such low levels of phosphorus were previously found to be limiting on nitrification (Manoharan *et al.* 1992).

From day 575 onwards phosphorus was added to the reactor as part of the alkalinity at varying concentrations. During this extended period nitrification showed good performance, with average TAN removal of 99.0% from day 580 onwards (excluding periods of mechanical failure) at an average NLR of 0.61 g-N/L.d. Moreover, a slow increase in biomass was observed.

Assessment of UNSBR phosphorus deficiency

A batch test was conducted on day 559 to determine the impact of P deficiency on the sludge nitrification. The first

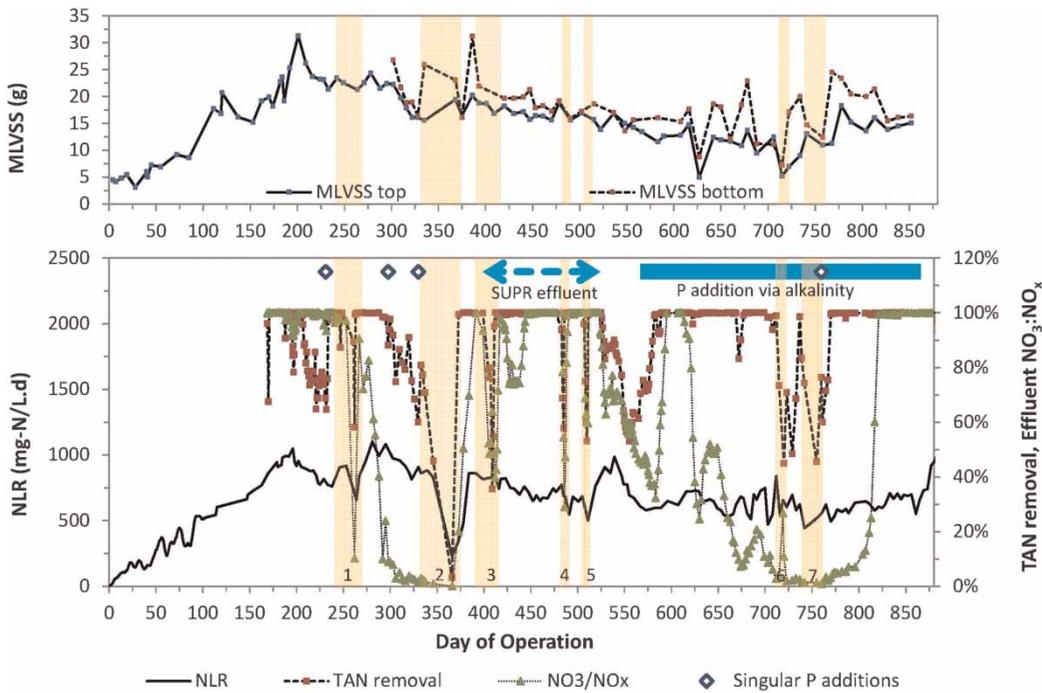


Figure 6 | Nitrification treatment performance and suspended solids of UNSBR through study period. Vertical shaded regions represent mechanical system failures. Failures number 2 and 7 were clogging of aeration diffusers, the remainder were alkalinity dosing failures. Performance measurements began after cultivation period.

set of samples at 6.5, 15.5 and 27.5 h confirmed that organics degradation was complete with negligible ammonia oxidation. At this point the second sample set including control samples was opened to the atmosphere on a mixing table. Results after 72 h indicated a pronounced impact of phosphorus on the nitrification. Due to the low aeration conditions and high pH of the batch test no nitrate was produced and significant ammonia volatilisation occurred as evidenced by the control samples. However, nitrite production and TAN removal clearly increased with each increasing addition of P (Figure 7). At a SRP value of

0.2 mg/L, similar to UNSBR effluent concentrations, nitrification was clearly inhibited.

In further analysis of the P deficiency of the system a P balance of the reactor was conducted from day 399 to 573. It was found that the total TP into the reactor was 347 mg and the SP out of the reactor was 103 mg. Based on a measured biomass P content of 1.27% the effluent solids accounted for an additional outflow of 357 mg P, resulting in a net loss of 113 mg P through normal operation. A further P balance of influent TP, dosed P and effluent SP during days 621 to 653 and 791 to 809 was conducted to determine the required P

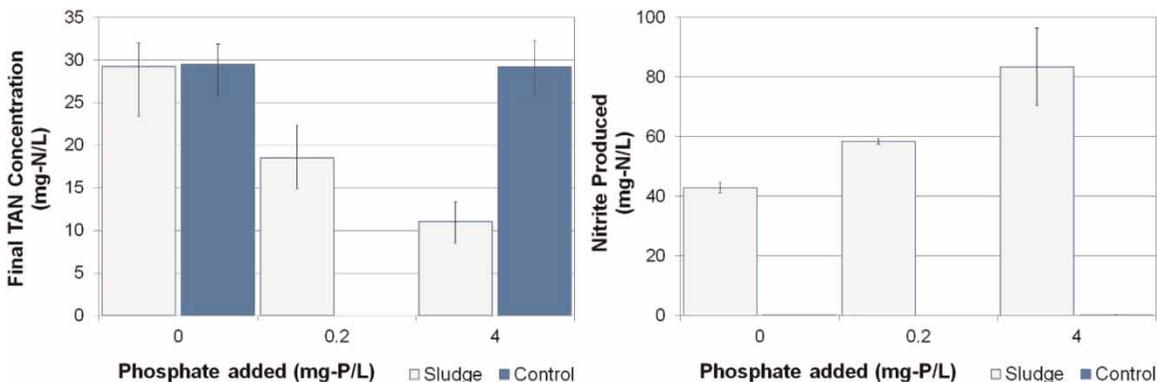


Figure 7 | Results of phosphorus addition batch test on UNSBR sludge.

for stable operation. In these periods consistent levels of P were dosed and in excess. It was found that 3.4 ± 1.1 mg-P/g-TAN oxidised (95% confidence interval) or 300 ± 80 μ g-P/g-MLVSS.d was required to meet biomass consumption. The required P was significantly greater than the influent P:N ratio of 1.6 mg-P/g-TN.

Integration of SUPR components

The results of the SUPR system have shown high levels of phosphate removal with downstream implications on biomass growth and treatment performance in the UNSBR system. The results indicate a minimum of 4–5 mg-P/g-TAN to be oxidised should be provided to the UNSBR to ensure stable operation. Based on effluent TP and TN concentrations from the SUPR system, direct coupling of the two reactors would only be possible under phase III conditions in this study. In cases where influent phosphorus is not sufficient, a small portion of urine, in the order of 1%, could be directly bypassed to the reactor to allow sufficient biomass growth. However, these results suggest that running the SUPR system at a lower HRT and slightly lower recovery is beneficial for the overall system, which also reduces the size of the SUPR reactor.

CONCLUSIONS

This study investigated a biological hydrolysis SUPR reactor and its integration with a subsequent UNSBR. The following can be concluded from this study.

- Full urea hydrolysis can be achieved within 1.5 h with 91–96% phosphorus removal achieved at HRTs of 1.5–6 h. P recovery increases with HRT and urine fraction.
- Product analysis indicated struvite as the main product component. Higher purity struvite was achieved with higher urine content. However, calcite was also present, especially at lower urine concentrations.
- UNSBR operation of a 25% urine and 75% seawater mixture was successful with greater than 99% TAN removal during mechanically stable operation, but was sensitive to incoming P levels.
- To maintain biomass within the reactor, and hence stable nitrification in the UNSBR, 3.4 ± 1.1 mg-P/g-N oxidised (95% confidence interval) or 300 ± 80 μ g-P/g-MLVSS.d is required for influent.
- Integration of these two systems for implementation of P recovery and nitrified urine sewer discharge is feasible

when lower HRTs and P recovery are used in the SUPR system to allow some P carry-over. If insufficient P is available, a small amount of urine can be bypassed around the SUPR reactor to maintain biomass growth.

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