

# FIELD OPERATION OF A SIMPLE STRUVITE REACTOR TO PRODUCE PHOSPHORUS FERTILISER FROM SOURCE-SEPARATED URINE IN ETHEKWINI

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## ABSTRACT

Struvite was produced in a field setting from source separated urine in a simple reactor. Research was conducted as part of VUNA (Valorization of Urine Nutrients in Africa) project in which one aim is to recover nutrients from source separated urine collected in a field setting. Struvite (magnesium ammonium phosphate hexahydrate; MgNH4PO4•6H2O) precipitates are produced by adding soluble magnesium to urine. A simple struvite reactor was built and batch-operated. Dissolved magnesium (Mg) was dosed at ten percent percent excess to stoichiometric amount of P using the historical average measurement of 280 mg P·*l*-1. Struvite was filtered through cotton filter bags (by gravity or with external pressure added via a nylon rope), then subsequently air dried. A submersible pump was used to fill the reactor with stored urine; otherwise, all operations were human-powered. Over 1 000 L per day of urine was processed daily within 4 to 6 hours. Struvite yield was affected by insufficient addition of magnesum and some loss based on filtering method used. Emphasis was placed on simple methods and execution of tasks, such that the procedures tested could be replicated in decentralised locations in a rural or semi-rural setting.

## **1 INTRODUCTION**

Struvite, a phosphorus-rich solid precipitate, was produced in a field setting from source separated urine in a simple reactor, requiring little electricity or process control. Struvite (magnesium ammonium phosphate hexahydrate; MgNH4PO4•6H2O) was produced by adding dissolved magnesium sulphate (MgSO4•7H2O) to urine. Urine is an ideal source for fertiliser production, as the majority of nutrients found in wastewater originate from urine (Larsen and Gujer, 1996). Phosphorus (P) is essential for the growth of food for which there is no substitute (Cordell et al., 2009), thus is directly related to food security, yet at certain loadings is a pollutant to bodies of water and groundwater. As part of the VUNA (Valorization of Urine Nutrients in Africa, www.vuna.ch) project, research was conducted into the field operation of a simple struvite reactor for nutrient recovery and fertiliser production from source separated urine. For an overview on the different activities of the VUNA project, refer to Etter et al. (2014). One aim of VUNA is to test nutrient recovery processes (Udert et al., 2014) in a field setting that may lead to the development of technologies and techniques that could be used by entrepreneurs. One purpose for this approach is to incentivize improved toilet use by providing addition value in the sanitation system through the valuation of nutrients in urine. The concept for application is local recovery and recycling of nutrients to improve the overall health of people through improved sanitation, environmental health, soil health, and improved local food production where safely applied. As the cost of fertilisers is market influenced and therefore fluctuating, their purchase still requires capital expenditure. If labour is available, but capital is not, then access to these nutrients through collection, processing, and utilisation, could be purchased in the form of human power and effort. In this way, the methods evaluated here were not intended to be supported entirely through the market value of the nutrients recovered as fertilisers, but as value-added to the overall health of the community and sanitation system. When considering nutrient recycling from waste streams, questions naturally arise regarding their safety. Others are addressing aspects of health, hygiene and environmental fate related to the handing of urine and struvite (Decrey et al., 2011, Ronteltap et al., 2007, Schertenleib, 2014). Similarly, the efficacy of struvite in agriculatural applications is being evaluated by research partners (Bonvin, 2013).

The aim of this study was to evaluate if uncomplicated techniques were suitable for the recovery of phosphorus from source-separated human urine in a field setting to produce a solid fertiliser using a simple reactor vessel requiring little to no electrical power.

## 2 METHODS

Techniques were developed to process urine in a field setting to produce struvite targeting the recovery of phosphorus adapted from techniques developed by Grau et al. (2012). Observations made during this period focused on the use of implementing simple process techniques and resources, and their effect on process capacity and product yield.

## **Urine Source**

Sanitation services to approximately 80 000 households in the rural and semi-rural areas of eThekwini are provided through the use of urine diverting dehydrating toilets (UDDT), as described in Tilley et al. (2008). To support the VUNA project, approximately 700 household UDDTs were modified for urine collection (Joseph et al. 2014). The normal configuration would be to dispose of the urine in soak-away pits. Collected urine was consolidated and stored at a field site.

## Magnesium Source

Industrial grade magnesium sulphate heptahydrate (MgSO4•7H2O) was selected as the magnesium source, as it is available locally in South Africa (Strathmore Mine, Malelane,

Mpumalanga).

#### Urine Storage and Handling

The age of urine ranged from 2 weeks to 2.5 years depending upon available storage tanks. Storage tanks were either well-sealed with rubber gasket covers equipped with spring-loaded vacuum release vents (JoJo, Groblersdal, South Africa), or modified prefabricated septic tanks, which were open to atmosphere. Tank volumes ranged from 1 000  $\ell$  to 10 000  $\ell$ .

Phosphorus salts spontaneously precipitate during the storage of urine due to the natural presence of magnesium and calcium in urine (Udert et al., 2003). In addition, the stored urine contained foreign objects and general debris (for example, cigarette butts, and unspecified dirt). A submersible pump was used to transfer urine from storage tanks into the struvite reactor. Due to the debris in the tank sediments, first-pumped urine was wasted until the debris cleared. The submersible pump was the only electrical equipment used in the entire production process.

### **Chemical Analysis**

Chemical analyses were undertaken using a Merck NOVA 60 spectrophotometer and Merck test kits for dissolved phosphate (114848), total phosphate (114543), and ammonium (114752) (Darmstadt, Germany). A handheld device was used to measure pH, conductivity, and temperature (WTW pH/Cond 340i, Germany). Dissolved phosphate and ammonium ions were determined in filtered urine. Samples were filtered using a glass fibre filter with an average retention capacity of 0.4  $\mu$ m (Machery-Nagel, Düren, Germany) and diluted with distilled water to analyse dissolved species (PO4-P – 1:100; NH4-N – 1:10 000). Total phosphate was determined in unfiltered urine.

### **Field Production Facilities**

Facilities available at the field site where urine is stored included a sloped concrete pad leading to a drain connected to sanitary sewer, potable water, and single phase electrical power. An analytical laboratory for wet chemistry analyses was not available at that time.

#### Simple Struvite Reactor Design

A simple, transportable, manually-operated struvite reactor (40  $\ell$ ) was built and modelled after a design developed by Etter et al. (2011). The dimensions of the reactor were: diameter 300 mm; height 840 mm; with 160 mm of freeboard. The reactor was fitted with four vertical baffles and two mixing impellers for improved mixing (Figure 1). The mixing shaft was equipped with a bearing ring assembly making the mixing crank free moving and easy-to-use. A manual valve was used to contain or drain the reactor contents between reaction periods. The reactor was placed on a sloped concrete slab with a drain. The effluent was drained to a sanitary sewer. The reactor was rinsed with tap water after each use.

#### **Reactor Operators**

Two manual labourers from the municipal urine collection crew (extended public works programme) who demonstrated above average interest and initiative were trained in initial methods of struvite production. They were also requested to contribute to further developing and enhancing field production methods. Production days were generally supervised, but once trained, operators conducted processing tasks on their own.

### **Magnesium Dosing Strategy**

Dissolved magnesium was added at a 10 percent excess to stoichiometric amount of P using the historical average measurement of 280 mg P·{-1. To provide a magnesium (Mg) dose specific to each batch of stored urine, the influent phosphate concentration would have to be measured to prepare the dosing solution. This approach would have been counter to the objective of developing simple techniques. Consequently, the historic average phosphorus concentration observed during this project was used to prepare the dosing solution.



Figure 1: Simple, manually-operated struvite reactor, clockwise from left: overall assembly and placement; vertical baffles in interior; dual impellers; bearing assembly and dosing inlet port viewed from underside of cover.

The following simple dosing procedure was developed in conjunction with the reactor operators. A magnesium dosing solution for a phosphate concentration of 280 mg P· $\ell$ -1 delivered in 0.5  $\ell$  of tap water. The procedure to prepare the dosing solution was to add four (4) full measuring cups (1 000 m $\ell$  or 98 g) of magnesium sulphate to 20  $\ell$  of tap water in a mixing tank with a valve. The dosing solution was prepared in advance of processing. This was sufficient for 4 x 40  $\ell$  batches of urine.

#### Liquid Solids Separation

Cotton fabric was obtained from a local fabric store and sewn into cylindrical bags providing surface area of 3 100 cm2 (450 mm tall, 200 mm in diameter) and fitted with cotton pull string. Pore space of the clean, unused cloth was analysed with a scanning electron microscope (SEM) and shown to have a pore size of approximately 100  $\mu$ m (Figure 2). Multiple 40  $\ell$  batches were filtered through one filter bag with eight batches (320  $\ell$ ) being typical for daily processing.



Figure 2: SEM image of common cotton fabric used as filter material.

#### Drying

Filter bags air dried for 7 to 14 days and kept beneath a rain cover.

#### **Process Sequence**

The reactor was filled with urine to the 40  $\ell$  mark using the submersible pump. Magnesium dosing solution (0.5  $\ell$ ) was poured into the top of the vessel. Mixing was initiated immediately thereafter. Following mixing (counting one second per crank arm revolution for 60 counts), the valve would be opened and contents drained into the filter bag. Once the contents of the reactor had drained into the filter, the valve was closed and fresh stored urine pumped into the reactor to repeat the sequence. Effluent typically was drained to sewer, or captured to evaluate phosphorus recovery and amount of struvite not captured by the filter bag. Filtration by gravity required 30 minutes. The operators developed a technique whereby a nylon rope was wrapped around the exterior of the filter to apply external pressure.

### Process Technique Development and Training

In addition to processing training and improvements applied to a field setting, training and procedures included equipment maintenance, and data collection (time for processing, volumes processed, presence of debris in the urine, gross observations of product yield, drying times, notation of sampling for chemical analysis, process time, and change in preparation of magnesium dosing solution).

## **3 RESULTS**

### **Process Capacity**

Volumes of over 1 000  $\ell$  per day of urine were processed daily (Figure 3). This production capacity was successfully repeated for five days (Production Days 2 through 7). Overall, 4 to 6 hours was sufficient to process over 1000  $\ell$  in a day, including set-up and cleaning tasks. Process times were typically 5 minutes for fill, mix, and react, followed by 30 minutes filtration by gravity or 5 minutes with pressure enhanced filtration. Air drying required 7 to 14 days. Production Days 1, 2, 8, and 9 were devoted to other investigative objectives other than daily production capacity. Production occurred over six weeks during the months of March and April 2013, except for Production Day 9, which was in October of 2013.



Figure 3: Daily urine process capacity

### **Urine Characteristics**

Historic data available for dissolved phosphate were used to guide the Mg dose. A concentration of 280 mg P·ℓ-1 was selected to attempt to recover the most of urine processed as most samples analyzed fell within this concentration range (Figure 4, left). Similarly, historic data for the concentration of ammonium ions was reviewed to consider ammonium-nitrogen remaining in the struvite-depleted urine (Figure 4, right).

It could be observed that pH, temperature and conductivity of urine in different storage tanks were similar (Table 1).

 Table 1: Values for urine pH, conductivity, and temperature of urine processed.

|           | рН  | Conductivity<br>(mS/cm) | Temperature<br>(°C) |
|-----------|-----|-------------------------|---------------------|
| ave       | 8.8 | 25.3                    | 27.2                |
| Std. Dev. | 0.2 | 4.0                     | 3.3                 |
| max       | 9.2 | 28.2                    | 30.9                |
| min       | 8.6 | 17.4                    | 22.6                |
| n         | 7   | 7                       | 7                   |



Figure 4: Distribution of dissolved phosphate (left) and ammonia nitrogen (right) measurements in urine.

#### **Recovery and Yield**

Influent and effluent samples from the struvite reactor were collected and analysed (filter Bags 10, 15, 17, and 30). Phosphorus recovery from Bag 10, 15, and 17 were: 48, 59, 62 percent, respectively. This was lower than anticipated. During these days the reactor operators worked autonomously and it was suspected that magnesium was dosed insufficiently. Thus, a subsequent test for phosphorus recovery was conducted on Production Day 9. In all cases on Production Day 9, P recovery was greater than 95 percent. The relationship of yield, magnesium dosage, supervision, and application of external pressure was evaluated (Figure 5).

Using an assumed influent concentration of dissolved phosphate of 280 mg P· $\ell$ -1, 2.45 kg of magnesium sulphate were used per 1 000  $\ell$  urine processed. Overall, nearly 7 000  $\ell$  of urine were processed in six weeks generating a recoverable mass of struvite totalling 5 kg.

## **4 DISCUSSION**

Practical experience was gained in a field setting with the operation of a simple, manually-operated struvite reactor. Process capacities of up and greater than 1 000 l of urine per day were regularly achieved. Resources for time and materials required were manageable. Struvite yield from the process was less than expected likely affected by inadequate addition of Mg and increased loss from increased external pressure to filters which was made in in exchange for reduced time of filtration. Addition of magnesium salts must be adequate to get maximum stochiometric production and yield. The method to prepare the magnesium dosing solution was to add 4 scoops of magnesium sulphate to 20 l of water. A recommended improved practice would be to obtain a single vessel to contain the total magnesium sulphate that must be mixed with the 20 l of water. In this way, the possible error of counting errors in the addition of the magnesium sulphate to the stock tank could be avoided.



Figure 5: Yield of struvite harvested relative to dose, filtration, and supervision

Emphasis was placed on simple equipment, methods, and execution of tasks, so that the procedures tested could be replicated in decentralised field settings. A critical step was filtration. The liquid-solid separation process step was identified as a rate-limiting step in the overall process sequence with a similar, struvite reactor also under investigation as part of the VUNA project (Grau et al., 2013). A technique was developed and tested in which external pressure was applied to the hanging filter bag with a nylon rope. The desire by the operators to complete the process cycle quickly resulted in the incomplete capture of the struvite in the filter bag. Filter bags were utilised repeatedly, though showed deterioration over time. Dried struvite would break apart threads in the seams when emptying the filter bags, leading to reduced filter performance.

In this study, the struvite-depleted urine was wasted to sewer. The nitrogen content may be beneficial in for agricultural applications. Samples of the struvite-depleted urine were collected and are being evaluated in agricultural trials.

Process improvements to increase the recovery of struvite include: 1) improved processes to ensure sufficient magnesium is dosed; and 2) modification to the delivery of dosing solution to pass through a funnel and tube through a ring distributor near the mixing impellers possibly delivered in stepped doses.

## **5 CONCLUSION**

The reactor was successfully field tested for ease of-use, ability to process volumes of urine, and ability to transfer technology and training to municipal support staff. Production processes were quickly learned by operators, improved upon, and eagerly applied. Interest in utilising the product, struvite, for their home gardens was expressed by reactor operators. This is an indication that the semi-rural urine diversion toilet users may be willing to participate in collection, processing, and application of products generated in local nutrient recycling.

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