Short Communication

Facile and simultaneous separation of nitrogen, phosphorus and bacteria from urine by using ash depth filters which harvest ammonium and phosphate as Struvite Enriched Ash

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ABSTRACT

Ash depth filters were developed which can simultaneously remove nitrogen, phosphorus and bacteria from human waste streams while simultaneously maintaining a flow rate which is acceptable for domestic use processing urine from one individual for 1 month if the filter bed has a volume of approximately 3 l. Nitrogen and phosphorus depletion is achieved by the formation of Struvite Enriched Ash, which can subsequently be used as a slow-release garden fertilizer. Depth filtration and sanitation by high pH removed all detectable bacteria from this home-based system.

Key words | ammonia, ash, depth filter, phosphate, struvite

INTRODUCTION

Nitrogen can form fixed compounds such as ammonium carbonate and ammonium bicarbonate by reacting with calcium carbonate, which separates easily. Phosphorus can be trapped as insoluble compounds, for example with magnesium, and can also be separated easily. Struvite (MgNH₄PO₄·6H₂O) is a desirable compound of both nitrogen and phosphorus, which is formed abundantly and easily from urine when magnesium is added and pH increased, for example using bittern and waste wood ash (Witty 2016a) followed by decanting.

Some large-scale processes are known for harvesting struvite from waste streams (Zamora et al. 2017), but these fluidized bed systems are not available in developing countries or rural districts. This paper shows that combining two waste products invents a new home-scale process which is not so limited. It uses minimal technology and equipment and achieves the depletion of both nitrogen and phosphorus in waste, a strongly sought process (Conley et al. 2009). The materials needed are globally available free of charge and consist of single use HDPE bottles, which are usually discarded, and wood ash. Sanitization of waste is also achieved because the flow and chemical properties of wood ash allow solutions to pass through the device described, but not bacteria.

MATERIALS AND METHODS

Urine was harvested and stored at 4 °C until used. This material was transparent and showed no obvious signs of bacterial growth. Kitchen oak ash was sieved to remove large particles (Witty 2016a), which improves urine and water flow characteristics through ash beds.
Thin-walled blow-molded 3.78 l HDPE bottles were recovered from domestic garbage and were trimmed to remove the bottom of the bottle but retain a curved lip which increases resistance to deformation. The screw cap lid was perforated (62 holes/cm²) using a 21 G hypodermic needle to create an end plate which allowed flow of water and dissolved material but not ash particles. This was filled with approximately 3 l ash and 24 h urine washed through in daily aliquots of 750 ml. Eluate was collected by placing the filter on the rim of the lower reservoir, transferring the entire apparatus to storage at 4 °C overnight, transferring back to room temperature, and then the filter bed was lifted to allow access to eluate in the lower reservoir. This handling of the flexible depth filter apparatus occasionally caused small visible and discouraging cracks in the ash bed. However, these cracks quickly ‘self-healed’ when the subsequent urine aliquot was added because suspended ash filled the cracks and operation was not significantly affected. Importantly, when used in domestic situations, the ash bed would not be picked up and put down to take measurements, so cracks would be less common than in the experimental apparatus. When bacterial counts of the eluate were >1 cfu/ml, the ash beds were washed with 1.5 l deionized water, transferred to a shallow glass dish, dried in a 50 °C oven, and stored at room temperature.

This Struvite Enriched Ash was stable for at least 2 1/2 years at room temperature but capable of releasing fixed ammonia when heated. Small amounts of ash were heated stepwise using a Benchmark heating block, and during heating, NH₃ gas was analyzed using the Eagle 2 gas monitor (RKI Instruments, Union City, CA, U.S.A.). Because ammonium carbonate, ammonium bicarbonate and struvite have such different temperatures of decomposition to release ammonia gas, it was possible to distinguish ammonia fixed as any of these three salts. Elements in ash were analyzed using a scanning electron microscope (JEOL JSM-IT100) and embedded energy-dispersive X-ray spectroscopy (EDS).

RESULTS

The apparatus which allows flow of large volumes of waste was invented, and the flow rate was excellent over long periods. The apparatus excludes bacteria, and when damaged, the ash bed is capable of self-healing to repair cracks which might otherwise form short circuits of urine directly to the end plate rather than through the ash bed.

Struvite Enriched Ash, which had been stored for 2 1/2 years, was decomposed using temperatures which were increased from 30 to 105 °C. Ammonia was usually only produced at 93 °C which shows the presence of struvite rather than other ammonium salts. Ammonia was only occasionally seen at approximately 58 °C (indicating ammonium carbonate) or 42 °C (indicating ammonium bicarbonate) in the various batches of Struvite Enriched Ash made (Figure 1).

The flow rate of urine through the system was excellent and liquid was passed through the ash quickly and consistently for long periods (Figure 2). In contrast, bacteria accumulated in a way which was noticeable, especially late in depth filter runs, as an organic matter, conspicuously indicating a physical mechanism for the removal of bacteria. No bacteria were detected in the eluate for most of the time the depth filter was used (Figure 2) despite the presence of nutrients and the absence of the aseptic technique, indicating the chemical suppression of bacterial growth. This
continued until pH declined to about pH 9 when numbers of bacteria in the eluate began to be seen. At this point, the ash bed was washed with water, dried and stored at room temperature in plastic bottles as described above. The process was surprisingly odor free when run at 4 °C. EDS detected 0.04% nitrogen and 1.57% phosphorus in Struvite Enriched Ash. Nitrogen was in the form of struvite, as shown by decomposition analysis. No heavy metals were detected by X-ray spectroscopy, which is expected from the analysis of kitchen wood ash.

CONCLUSIONS

Struvite has long been known as a granular precipitate in latrines (Witty 2016b), which is obtainable from urine in the laboratory when supplemented with reagent base and magnesium (Witty et al. 2017) and even in pilot plant experiments (Zamora et al. 2017), which achieve fertility depletion of city waste streams. Struvite may also have been obtained by fullers using ash in ancient times (Witty 2016a), but the intellectual property of this trade was protected in the usual way for the ancients, i.e. secrecy (Witty 2018) and then lost.

This natural process of crystallization has been improved to make Struvite Enriched Ash, where urine and ash are enriched to make a useful garden fertilizer. Struvite is the main store of ammonium in enriched ash. This is because of the solubility of ammonium carbonate and ammonium bicarbonate (10 g and 21.7 g/100 ml water, respectively, at 20 °C, Orentlicher & Simon 2016). Ash carbonates may fix ammonium temporarily but then be washed out of the system as it is presently organized. Using more ash or shorter run times might increase the efficiency of ash depth filters and result in the harvest of ammonium carbonates and bicarbonates in addition to struvite. Phosphorus is stored as a component of struvite and also as a component of the starting material, wood ash.

Ash depth filter flow characteristics were excellent, and tens of liters of wastewater can be treated before failure, after the consumption of excess calcium carbonate, as seen by pH decrease and the appearance of bacteria in the eluate. An excellent feature is self-healing of ash bed cracks because it allows use of flexible bottles to contain ash beds, increasing the simplicity and ease of the construction of ash depth filters tremendously. These devices are very easy to make, and no complex apparatus like fluidized beds (Zamora et al. 2017) are needed. After long-term use, these devices are not something to be thrown away. Instead, the ash is useful as a garden plant fertilizer, because of the fixed dried nitrogen and phosphate, and the plastic components are recycled.

The gas monitor was a convenient device to detect the form of nitrogen stored and released from ash, i.e. struvite, which can release ammonia (Figure 1), although quantitative measurements are difficult using this method. This is why improved accuracy and the proportion of elements in Struvite Enriched Ash was measured using EDS and

![Figure 2](http://iwaponline.com/washdev/article-pdf/10/2/374/713144/washdev0100374.pdf)

**Figure 2** Depth filtration is a component of sanitation by this apparatus, as shown by counting bacteria in eluate over 57 days of use. Symbols used are: pH, filled square symbol with gray color (except where flow was short circuited through a temporary crack in the ash bed, in which case the square is open, N.B. pH resumed the trend line immediately after the ash column cracks self-healed); bacteria, circle symbol.
0.04% w/w nitrogen was detected. The sanitation effect of this apparatus is achieved by high pH disinfection in addition to depth filtration.

REFERENCES

Conley, D. J., Paerl, H. W., Howarth, R. W., Boesch, D. F., Seitzinger, S. P., Havens, K. E., Lancelot, C. & Likens, G. E. 2009 Controlling eutrophication: nitrogen and phosphorus. Science 323, 1014–1015.

Orentlicher, M. & Simon, M. M. 2016 Process to recover ammonium bicarbonate from wastewater. U.S. Patent Application 14/852,836, filed July 14, 2016.

Witty, M. 2016a Ancient roman urine chemistry. Acta Archaeologica 87 (1), 179–191.

Witty, M. 2016b Hooke’s gravel was Struvite. Notes and Queries 65 (4), 569–570.

Witty, M. 2018 Athenaeus describes the most ancient intellectual property. Prometheus 35 (2), 137–143.

Witty, M., Dingra, N., Abboud, K., Felts, A. & Ayudhya, T. 2017 Nuclear magnetic resonance and X-ray crystallography to improve struvite determination. Analytical Letters 50 (16), 2549–2559.

Zamora, P., Georgieva, T., Salcedo, I., Elzinga, N., Kuntke, P. & Buisman, C. J. 2017 Long-term operation of a pilot-scale reactor for phosphorus recovery as struvite from source-separated urine. Journal of Chemical Technology and Biotechnology 92 (5), 1035–1045.

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