Recovery of Ammonia and Production of High-Grade Phosphates from Side-Stream Digester Effluents Using Gas-Permeable Membranes

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Abstract. Phosphorus recovery was combined with ammonia recovery using gas-permeable membranes. In a first step, the ammonia and alkalinity were removed from municipal side-stream wastewater using low-rate aeration and a gas-permeable membrane manifold. In a second step, the phosphorus was removed using magnesium chloride (MgCl₂) and reduced amounts of alkali. The side-stream wastewater contained 730 mg N/L, 140 mg P/L and 2900 mg/L alkalinity. The process recovered approximately 79–93% of the ammonia and 80–100% of the phosphorus. Surprisingly, the phosphates produced were very-high grade (42–44% P₂O₅) with a composition similar to the bio-mineral newberyite. However, lower grade phosphate products (27–29% P₂O₅) were produced whenever the N recovery step was bypassed or carbonate alkalinity was added. Therefore, removal of ammonia and alkalinity are important considerations for production of very-high grade phosphate product.

Keywords: Ammonia recovery · Phosphorus recovery · Newberyite

1 Introduction

Conservation and recovery of nitrogen (N) and phosphorus (P) from municipal, industrial and agricultural effluents using anaerobic digesters (AD) is important because of economic and environmental reasons.

A promising new method to recover ammonia (NH₃) from wastewater is the use of gas-permeable membranes (Vanotti and Szogi 2015). The gas-permeable membrane manifolds are submerged in the liquid manure, and the gaseous NH₃ is removed from the liquid matrix before it escapes into the atmosphere. The N removal is done with low-rate aeration in the reactors that naturally increases the pH of the liquid and accelerates the rate of passage of NH₃ (>96%) through the submerged gas-permeable membrane manifold and further concentration in an acid stripping solution reservoir (Garcia-Gonzalez et al. 2015; Dube et al. 2016). The effluent after ammonia treatment is low in ammonia and carbonates. In turn, these conditions improve precipitation of phosphate minerals of high-grade.
The objective of this work was to develop new technology for simultaneous N and P recovery suitable for municipal digester effluents (Vanotti et al. 2016). It combines a gas-permeable membrane technology (N recovery) with P recovery of solid products by precipitation of phosphates. Phosphorus precipitating compounds such as for example, magnesium chloride (MgCl₂), are added to the system after the N removal. The new system was first tested using livestock wastewater (Vanotti et al. 2017). In this work, municipal side-stream wastewater was used. Results of this study and others were used to file a US Patent on the new process.

2 Materials and Methods

In this case study, the wastewater was side stream collected from James River municipal plant, Hampton Roads Sanitation District, Virginia. The side stream wastewater was a centrate effluent from waste sludge that was subjected to anaerobic digestion and solids separation and contained about 140 mg/L P and 730 mg N/L. Ammonia was substantially removed in a first treatment step (Fig. 1). In a second step, MgCl₂ was added to the N treated effluent in the phosphorus recovery tank. The gas permeable membrane module was connected with a stripping solution reservoir containing diluted acid as described in Dube et al. (2016) and Garcia-Gonzalez et al. (2015). Low rate aeration was delivered to the bottom of tank. Gas-permeable membrane was tubular and made of e-PTFE material. Nitrification inhibitor (22 ppm) was

Fig. 1. Schematic diagram of nitrogen (N) and phosphorus (P) recovery system using ammonia separation tank and P recovery tank
added to ensure nitrification inhibition. Concentrated acid was added to the stripping solution to an end-point pH of 1 when the pH increased above about 2 as result of active ammonia capture. In a second step, the treated effluent from the N recovery tank was transferred to phosphorus separation tank where it was mixed with MgCl₂ and NaOH to obtain a phosphorus precipitate and an effluent without phosphorus or ammonia. MgCl₂ was applied to obtain a Mg:P ratio 1.2:1. Alkali NaOH was applied to pH 9.2. The chemicals were mixed for about one minute. After about a 0.5 h gravity sedimentation period, the phosphorus precipitate was dewatered using glass fiber filters, and characterized for total N, P, Mg, Ca, and K and plant available phosphorus.

3 Results and Discussions

Phosphorus recovery of anaerobically digested municipal wastewater via MgCl₂ precipitation was enhanced by combining it with the recovery of NH₃ through gas-permeable membranes and low-rate aeration. The low-rate aeration stripped the carbonates in the wastewater and increased pH, which accelerated NH₃ uptake by the gas-permeable membrane system (Fig. 2). The ammonia capture process substantially reduced carbonate alkalinity, from 2990 mg/L to 130 mg/L, and ammonia concentration, from 730 mg N/L to 50 mg/L. These conditions benefited subsequent P recovery. The combined process provided quantitative (ca 100%) P recovery efficiency (Table 1).

![Ammonia Removed and Recovered in N Concentration Tank](image)

**Fig. 2.** Mass removal and recovery of nitrogen (N) from municipal wastewater using gas-permeable membranes and aeration

With active NH₃ extraction, the magnesium phosphates that were produced contained high P₂O₅ grade (42%) and high plant availability (Table 2). The phosphorus product was similar to the composition of newberyite (MgHPO₄·3H₂O), a biomineral found in guano deposits, which has approximately 40.8% P₂O₅ and 13.9% Mg composition and 1:1 P:Mg molar ratio.
However, in other tests conducted with the same municipal wastewater, whenever the N recovery step was bypassed or carbonate alkalinity was added, the phosphate minerals obtained had lower grade (27–29% P$_{2}$O$_{5}$) (Vanotti et al. 2016). Therefore, removal of ammonia and carbonates are important considerations for production of very-high grade phosphate products.

### 4 Conclusions

These results showed that it is possible to produce Mg phosphates with high P$_{2}$O$_{5}$ content by removing the NH$_3$ from the liquid with the gas-permeable membrane process. In a first step, the ammonia and alkalinity were removed from municipal side-stream wastewater using low-rate aeration and a gas-permeable membrane manifold. In a second step, the phosphorus was removed using magnesium chloride (MgCl$_2$) and reduced amounts of alkali. The phosphates produced were very-high grade (42–44% P$_{2}$O$_{5}$) with a composition similar to the bio-mineral newberyite. This is an important finding because recovered phosphates with high P$_{2}$O$_{5}$ content are more in line with mineral commercial fertilizers and favored by the fertilizer industry.
References


Frontiers in Wastewater Treatment and Modelling
FICWTM 2017
Mannina, G. (Ed.)
2017, XXIV, 745 p. 355 illus., Hardcover
ISBN: 978-3-319-58420-1