



Recovery of ammonia and phosphate minerals from swine wastewater using gas-permeable membranes



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ABSTRACT

Gas-permeable membrane technology is useful to recover ammonia (NH₃) from liquid manures. In this study, phosphorus (P) recovery via MgCl₂ precipitation was enhanced by combining it with NH₃ recovery through gas-permeable membranes. Anaerobically digested swine wastewater containing approximately 2300 mg NH₄⁺-N L⁻¹ and 450 mg P L⁻¹ was treated using submerged membranes plus low-rate aeration to recover the NH₃ from within the liquid and MgCl₂ to precipitate the P. The experiments included a first configuration where N and P were recovered sequentially and a second configuration with simultaneous recovery. The low-rate aeration reduced the natural carbonate, increased pH and accelerated NH₃ uptake by the gas-permeable membrane system, which in turn benefited P recovery. Phosphorus removal efficiency was >90% and P recovery efficiency was about 100%. With higher NH₃ capture, the recovered P contained higher P₂O₅ content (37–46%, >98% available), similar to the composition of the biomineral newberyite (MgHPO₄·3H₂O).

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1. Introduction

Conservation and recovery of nitrogen (N) and phosphorus (P) from livestock, industrial and municipal wastes is important because of economic and environmental reasons. More sustainable techniques using P recovery for both solid and liquid waste are important to close the P cycle in modern human society and address future scarcity (Desmidt et al., 2015; Keyzer, 2010). In the United States, the largest source of ammonia (NH₃) emissions is livestock farming, contributing 2.5 million tons/year (EPA, 2014). In addition, P build up in soils to excessively high levels due to animal manures often results in eutrophication and pollution of surface waters (Kleinman et al., 2015). Therefore, the removal and recovery of N and P is a desirable feature for new treatment technology for livestock effluents because the nutrients can be exported off the farm, which could solve the problems of N and P surpluses in concentrated livestock production, substitute for commercial fertilizers, help close the P cycle, and create new businesses (Keyzer, 2010; Szogi et al., 2015; Vanotti et al., 2009).

Technologies for recovery and reuse of P from livestock effluents

and also municipal effluents have focused mostly on struvite (MgNH₄PO₄·6H₂O) precipitation (Burns et al., 2001; Desmidt et al., 2015; Karunanithi et al., 2015; Nelson et al., 2003). Struvite forms from 1:1:1 ratios of magnesium (Mg²⁺), ammonium (NH₄⁺) and phosphate (PO₄³⁻). Addition of MgCl₂ and NaOH (Burns et al., 2001; Nelson et al., 2003; Westerman et al., 2008) have been used to balance Mg²⁺ to PO₄³⁻ ratio, increase pH, and improve process efficiency. Although the process also recovers N, in livestock wastewater only about <15% of the N contained in the influent could potentially be recovered through struvite because of the very high NH₄⁺ to PO₄³⁻ ratio in these effluents (i.e. 7.6:1 to 17.0:1, Burns et al., 2001), unless external phosphates are added to balance the NH₄⁺ (Liberti et al., 1986). Another way to recover P is through calcium phosphate precipitation with Ca(OH)₂ at pH > 9. Vanotti et al. (2005) used a biological nitrification step to eliminate the carbonate interference in swine wastewater before precipitating calcium phosphate with lime. Hao et al. (2013) indicated that future efforts should go to develop technologies based on other phosphate-based compound: any acceptable form of phosphate by the fertilizer industry as long as it contains appropriate P₂O₅ content (30–40% favored).

New technologies for NH₃ abatement in livestock operations are being focussed on N recovery. These technologies include: 1) reverse osmosis using high pressure and hydrophilic membranes (Masse et al., 2010; Thorneby et al., 1999); 2) nanofiltration (Kertész et al.,

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2010); 3) air-stripping using stripping towers and acid absorption (Bonmatí and Flotats, 2003; Liao et al., 1995); 4) zeolite adsorption through ion exchange (Milan et al., 1997); 5) co-precipitation with phosphate and magnesium to form struvites (Nelson et al., 2003; Uludag-Demirer et al., 2005); and 6) a new process using gas-permeable membranes at low pressure (Vanotti and Szogi, 2015; Garcia-Gonzalez and Vanotti, 2015). Zarebska et al. (2015) reviewed the pros and cons of all these N recovery methods and indicated the energy consumption for the gas-permeable membrane process was among the lowest (0.18 kW h kg NH₃⁻¹). However, its main drawback was the cost of alkali chemicals to increase manure pH (Zarebska et al., 2015).

The gas-permeable membrane process includes the passage of gaseous NH₃ through a microporous, hydrophobic membrane, and capture and concentration in a stripping solution on the other side of the membrane. The membrane manifolds are submerged in the liquid and the NH₃ is removed from the liquid before it escapes into the air (Vanotti and Szogi, 2015); the NH₃ permeates through the membrane pores reaching the acidic solution located on the other side of the membrane. Once in the acidic solution, NH₃ combines with free protons to form non-volatile ammonium (NH₄⁺) ions that are converted into a valuable NH₄⁺ salt fertilizer, which is desirable to export N off the farm to other regions where N is needed.

Gas-permeable membranes have been shown to effectively recover more than 97% of NH₄⁺ from raw and anaerobically digested swine wastewater (Garcia-Gonzalez and Vanotti, 2015; Dube et al., 2016; Garcia-Gonzalez et al., 2015). The process is responsive to increased pH through addition of alkali chemicals, which leads to an increased release of NH₃ from the manure and capture by the membrane. A strategy to reduce costs of this N recovery process and facilitate its adoption by farmers is to use simple and inexpensive alternatives for raising the pH of the manure in a farm setting. Vanotti and Szogi (2015) proposed the use of gas-permeable membranes with low-rate aeration and nitrification inhibitors to enhance the recovery of NH₃ without alkali and reduce costs. Such conditions applied to stored livestock effluents resulted in a pH increase of about 1 unit and increased NH₃ release. For the purpose of the enhancement of the recovery of NH₃ N recovery using gas-permeable membranes, the term “low-rate aeration” was defined as an aeration rate that is less than about 5% of the aeration rate used for biological ammonia removal/nitrification (Vanotti et al., 2016). Using the aeration approach, Dube et al. (2016) increased the pH of swine wastewater to 9.2 without alkali chemicals and obtained NH₄⁺ recovery efficiencies of 96–98% while reducing costs of treatment by 70%. The annualized of NH₄⁺ recovery from a 4000-head swine farm with anaerobic digester (12,547 kg N/year) using gas-permeable membranes with low-rate aeration and nitrification inhibitors was calculated at \$37,926/year: 55.5% for equipment, 37.1% for acid (120 kg H₂SO₄/day), 4.7% for nitrification inhibitor (0.5 kg/d), and 2.7% electrical cost (40.1 kW h/d for power use of blower and pumps) (Dube et al., 2016). Garcia-Gonzalez et al. (2015) compared the operational cost of using alkali (NaOH) vs. low-rate aeration (power and inhibitor) to increase manure pH and optimize N recovery using gas-permeable membranes. Relative to alkali addition, the aeration approach reduced the costs of NH₄⁺ recovery by 57%. Alkalinity is readily consumed in this system (72–73% reduction). The N recovery process produces wastewater with higher pH, lower NH₄⁺ concentration and lower carbonate alkalinity (Dube et al., 2016), which are modified conditions that could promote P recovery using precipitation processes (Desmidt et al., 2015; Liu et al., 2015; Nelson et al., 2003; Vanotti et al., 2003).

We hypothesized that, by implementing P precipitation in combination with the membrane N recovery system, the phosphorus recovery could also benefit. The objective of this study was to evaluate the potential advantages and technical feasibility of

simultaneous N and P recovery suitable for digester effluents. It combines a gas-permeable membrane technology (N recovery) with P recovery of solid products by precipitation. The P precipitating agent selected was MgCl₂ with or without alkali supplements.

2. Materials and methods

2.1. Basic process configuration

The basic configuration of the process evaluated in the experiments is shown in Fig. 1 (Vanotti et al., 2016). The overall goal was to synchronize the recovery of phosphorus (P) via chemical precipitation with the recovery of NH₃ through gas-permeable membranes and low-rate aeration by taking advantage of the increased pH and alkalinity destruction during the N recovery. Ammonia from anaerobically digested swine effluent was extracted in an ammonia-separation tank using a submerged gas-permeable membrane module and its was recovered in a stripping acid solution reservoir/nitrogen concentration tank. Low-rate aeration was provided in the ammonia separation tank to increase pH and the ammonium (NH₄⁺) recovery rate (Dube et al., 2016). A phosphorus recovery tank separated precipitated phosphorus. The two configurations evaluated in this work used the same NH₃ recovery system but varied in the location where the P precipitating agents were added. In the first configuration (experiment 1), the P precipitating agents (MgCl₂ with or without NaOH) were added to the liquid after NH₄⁺ was substantially removed. In the second configuration (experiment 2), the same P precipitating agents were added into the ammonia-separation tank at the start of N separation with the liquid containing high NH₄⁺ concentration. The two experiments were done under laboratory conditions.

2.2. Ammonia separation reactor

The NH₃ recovery portion of the study was done using the ammonia-separation reactor and protocol of Dube et al., 2016 (Fig. A.1). It consisted of a 2-L aerated ammonia-separation tank with an effective liquid manure volume of 1.5 L fitted with a submerged gas-permeable membrane connected with a stripping solution reservoir containing 0.25 L diluted 1N sulfuric acid (stripping solution). The acid solution was continuously recirculated at 4 mL min⁻¹ through the inside of the tubular membrane located in the ammonia-separation tank using a peristaltic pump (Cole-Parmer, Masterflex L/S Digital Drive, Illinois, USA). The tubular membrane was anchored to a glass rod inside the vessel to ensure submersion in the liquid manure. The tubular membrane was made of e-PTFE material (Phillips Scientific, Inc., Rock Hill, SC) with a length of 60 cm, outer diameter of 10.25 mm, and wall thickness of 0.75 mm. It had an average pore size of 2.5 μm and bubble point of 210 kPa. Bubble point was determined as the minimum pressure required to force air through the membrane which has been prewet with isopropylalcohol (ASTM, 2011). The ratio of the tubular membrane length per manure volume was 0.04 cm cm⁻³ and the ratio of e-PTFE membrane area per length was 0.0323 m² m⁻¹. Low-rate aeration was delivered to the bottom of the ammonia-separation tank at a rate of 0.12 L-air L-manure⁻¹ min⁻¹ using an aquarium air pump, a shielded air flow meter with a precision valve (GF-9260, Gilmont Instruments, Illinois, USA) and an aquarium diffuser stone that provided fine bubbles. The lid of the ammonia-separation tank was not sealed; it had one open port that allowed the air to escape. Nitrification inhibitor N-Serve (TCMP - 2-chloro-6 trichloromethyl pyridine, Hach, Loveland, CO, USA) was added to the manure at a rate of 22.5 mg L⁻¹ dosage to ensure nitrification inhibition (Dube et al., 2016). Small volume wastewater samples (2 mL) were drawn daily from the ammonia separation tank to test

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