



Recovery of nutrients and volatile fatty acids from pig manure hydrolysate using two-stage bipolar membrane electro dialysis



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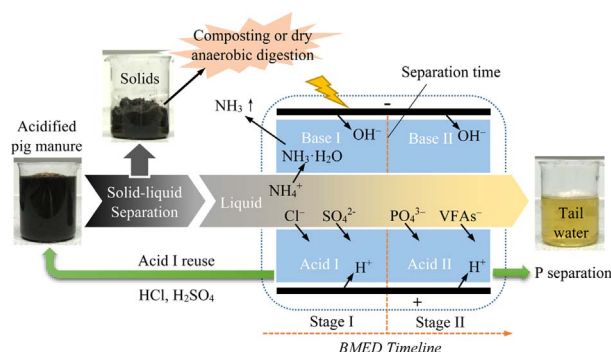
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GRAPHICAL ABSTRACT



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ABSTRACT

Animal manure should be regarded as a resource rather than a waste as it contains abundant nutrients (nitrogen and phosphorus) and organic matter. In this study, a laboratory-scale bipolar membrane electro dialysis (BMED) system was set up to assess the recovery of ammonium (NH_4^+), phosphate (PO_4^{3-}) and volatile fatty acids (VFAs) from both synthetic and real pig manure hydrolysate for the first time. Synthetic hydrolysate was used as feed first to investigate the ionic migrations in the BMED system. After 5.5 h of operation, 52% of NH_4^+ migrated to the base compartment, and 98% of PO_4^{3-} and 95% of VFAs migrated to the acid compartment. A BMED model was established to quantify the ion flux balance in the membrane stack. It substantiated that the low recovery efficiencies of NH_4^+ and the impurity of acid solution were primarily caused by the undesired diffusion of ions through bipolar membranes. Subsequently, a novel two-stage BMED operation based on the “inflection point” of voltage was developed to minimize the NH_4^+ loss and separate PO_4^{3-} and VFAs from the acid compartment. Through this operation, the recovery efficiency of NH_4^+ increased to 78%, and 75% of PO_4^{3-} and 87% of VFAs were separated from Cl^- and SO_4^{2-} in the acid compartment. Finally, real pig manure hydrolysate was tested and the variations of ions in the BMED were consistent as those using synthetic wastewater. This study demonstrates that it is feasible to recover valuable nutrients and VFAs from pig manure hydrolysate using two-stage BMED technology.

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1. Introduction

Global pork production has reached more than 116 million tonnes in 2016, resulting in not only the intensification of large-scale pig farms, but also a significant increase in manure production [1]. Pig manure is a mixture of flushed feces, urine, strewing material and spilled food [2]. As a natural fertilizer for crops, it has been extensively applied on farmlands [3]. Prior to land application, pig manure is commonly stored in animal houses for several weeks to several months (it is not rare that manure has been stored for one year before land application), leading to the release of ammonium (NH_4^+), phosphate (mainly PO_4^{3-}) and volatile fatty acids (VFAs) into the liquid fraction as hydrolysis occurs. Land application of pig manure at a rate exceeding the crops uptake threshold can pose significant environmental impacts via nutrients leaching and runoff, particularly in large-scale pig farms where manure generated exceeds the land carrying capacity [4]. The European Union Nitrates Directive Action plan has restricted the use of animal manure on grassland and cereals [5]. In Ireland, the maximum amount of nitrogen (N) that can be applied annually to land is 170 kg/ha [6]. Many farms are no longer suitable for land spreading of pig manure because organic N loading from grazing livestock is already at or approaching this limit. The land availability will be reduced further due to the imposed phosphorus (P) loading limit in 2017. Therefore, alternative options become essential to effectively recover nutrients, thus avoiding over-fertilization and thereby mitigating environmental impacts.

Pig manure contains abundant valuable resources including NH_4^+ -N, P and VFAs. Following anaerobic digestion, high concentrations of NH_4^+ -N and P (e.g. up to 4000 mg/L and 300 mg/L, respectively) remain in the digested pig manure [7,8]. While traditional approaches, such as struvite precipitation, can be adopted to recover NH_4^+ -N and P simultaneously from anaerobic effluent in the downstream processes, the low recovery yield has been reported due to the undesirable conditions, such as low pH, insufficient P concentration, and improper N/P mole ratio [9]. In addition, struvite precipitation cannot realize the recovery of VFAs, a carbon source widely used in the production of bioplastics and bioenergy, as well as for nitrogen removal from low C/N wastewater [10]. High concentration of VFAs from initial hydrolysis during the storage [11] renders its recovery imperative. Apart from being organic matter for biogas production through anaerobic digestion, VFAs can be alternatively harvested as by-products, which can be incorporated potentially into the recovery processes of NH_4^+ -N and P.

While traditional methods for pig manure management (e.g. anaerobic digestion and struvite precipitation) are used for resources recovery, membrane technologies, such as forward/reverse osmosis, nanofiltration, membrane distillation and electrodialysis, can also offer huge potentials. Nowadays, these membrane technologies are playing critical roles in energy production, chemical synthesis, clinical medicine, and water purification [12–18]. With respect to resource recovery, membrane technologies combined with air-stripping are effective and energy-efficient to recover NH_4^+ -N from animal manure [19]. In addition, membrane technologies can pre-concentrate P in the wastewater for the formation of struvite [20,21]. Amongst these membrane technologies, bipolar membrane electrodialysis (BMED) is a novel electrodialysis system equipped with bipolar membranes. It couples functionally the solvent dissociation of bipolar membranes and the salt dissociation of conventional electrodialysis. In other words, BMED takes advantage of the specific property of a bipolar membrane and effectively splits water into hydroxide ions (OH^-) and protons (H^+) under an applied electric field, thus separating cations and anions to different compartments. Ultimately, inorganic and organic salts can be converted to the corresponding acids and bases, thereby allowing the diversity of the products without chemical dosing. In this process, a high pH in the base compartment creates suitable conditions for NH_4^+ -N recovery via subsequent air-stripping, while P and VFAs migrating to the acid compartment in BMED can be recovered as weak acids. BMED

has been previously applied to produce acetic acid, lactic acid, citric acid, and other organic acids for industrial uses [14,15,22–24]. However, few studies have focused on the resource recovery from animal manure using BMED. Most importantly, simultaneous recovery of NH_4^+ -N, P and VFAs has never been reported in the literature.

Effective nutrients release and solid-liquid separation are essential prior to the nutrient recovery processes. Nutrients incorporated in the solids of pig manure, in particular P, need to be released to the liquid fraction prior to their recovery. The concentration of soluble P is associated thermodynamically with the pH value. Lower pH ($\text{pH} < 6.0$) of manure can facilitate 90% of the P release to the liquid fraction and reduce the evaporation loss of NH_4^+ during storage [25,26]. For this reason, pig manure is acidified (to $\text{pH} = 5.0$) in certain countries (e.g. Denmark) to increase its fertilizer value. In addition, the concentrations of VFAs in digestate are normally less than 1000 mg/L, while it can reach up to 10,000 mg/L in the hydrolysate [27–30]. The high concentration of VFAs leads to a lower pH in the hydrolysate, thus reducing chemical dosing during the acidification. In large scale pig farms, the solid-liquid separation of pig manure can minimize the transportation costs, reduce the nutrients load on adjacent lands, and produce energy-rich biomass [2]. From the perspective of resources recovery, the solid-liquid separation of pig manure can generate the liquid fraction for recovery and the solid fraction which can be used in dry anaerobic digestion or composting [31]. Therefore, recovery of nutrients and VFAs from pig manure hydrolysate would provide a management option alternative to conventional recovery of only nutrients from pig manure digestate.

Given these facts, a three-compartment BMED was set up in this study to assess the simultaneous recovery of NH_4^+ -N, P and VFAs from acidified pig manure hydrolysate. The primary objectives were to (1) simultaneously recover NH_4^+ -N, P and VFAs from pig manure hydrolysate using BMED; (2) establish a BMED model to simulate this process based on the ion flux balances and investigate the diffusion loss of ions; and (3) optimize the operation to separate the weak acids (phosphoric acid and VFAs) from Cl^- and SO_4^{2-} and improve the purity of products.

2. Materials and methods

2.1. Materials

Both synthetic and real pig manure hydrolysate were used as the feed wastewater to the BMED. The synthetic hydrolysate was prepared using distilled water to simulate the real pig manure hydrolysate. It consisted of (in g/L): ammonium chloride 8.02, ammonium sulfate 9.91, disodium hydrogen phosphate 3.15, sodium acetate 5.49, propionic acid 1.33, butyric acid 1.32, iso-butyric acid 1.32, valeric acid 1.02 and iso-valeric acid 1.02. The corresponding concentrations of NH_4^+ , PO_4^{3-} and VFAs were 5.40 g/L, 2.09 g/L and 10.03 g/L, respectively, with NH_4^+ -N and PO_4^{3-} -P being 4.20 g/L and 0.68 g/L, respectively. Pig manure hydrolysate, which had been stored in a storage tank for some time, was collected from a local pig farm in Co. Galway in Ireland (53.104° N, -8.306° W) with initial pH of 7.3. The characteristics of pig manure hydrolysate were detailed in Table S1. It was pretreated to facilitate the release of P to the liquid fraction and to remove the solid contents prior to the experiment. The pretreatment consisted of four steps as follows: (1) adjustment of pH to 5.0 using HCl (1 mol/L); (2) settlement for 24 h with foam removal; (3) centrifugation at 15,000g; and (4) filtration (10 μm) and microfiltration (0.45 μm).

Cation-exchange membrane (CM), anion-exchange membrane (AM) and bipolar membrane (BM) used in this study were heterogeneous membranes (MemBrain Company, Czech Republic). All membranes were soaked in deionized water for 48 h prior to the use. The properties of the membranes used in this study are presented in Table 1.

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