



Dissolution of particulate phosphorus in pig slurry through biological acidification: A critical step for maximum phosphorus recovery as struvite



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ABSTRACT

Recycling phosphorus as struvite from pig slurry requires an acidification step to dissolve the inorganic solids containing most of the phosphorus. This study focused on the biological acidification of several pig slurries using sucrose as a model organic co-substrate. Lactic acid fermentation occurred systematically, dissolving 60–90% of TP (total phosphorus) and T-Mg (total magnesium) at pH 6 or lower. Optimal pH range for maximum P dissolution aimed at struvite recovery was 5.5–6. A simple model was developed correlating pH, sucrose and buffer capacity to optimize P dissolution and future recovery using real organic waste.

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1. Introduction

The anthropogenic use of phosphorus presents a double problematic: on one hand it is contributing to water bodies pollution due to a combination of fertilization excess and leakage/runoff (MacDonald et al., 2011), and on the other hand it is a fossil resource critical for agriculture and worldwide food production (Cordell et al., 2009). As a result, recovering phosphorus from pig slurry as a mineral fertilizer could be a useful tool to mitigate water pollution from pig breeding and participate in a more sustainable food production (Ashley et al., 2011). Brittany (France) possesses only 6% of the national arable land but represents 60% of French the pig production (Comité régional porcin de Bretagne, 2013). Stringent regulations now prevent the unlimited spreading of the slurry into the fields and various handling strategies are currently used in the region (Landrain et al., 2013). Among them is anaerobic digestion

(Levasseur and Lemaire, 2006), in which various organic wastes are co-digested with the slurry in order to produce energy and heat. Exportation of manure out of Brittany is another strategy to reduce the environmental impact on soil (Martinez et al., 2009). Part of the digested and undigested manure is separated using screw press or centrifuge decanter to collect a liquid phase rich in nitrogen that is treated via biological processes, while most of the phosphorus remains in the solid phase under a mineral form (Christensen et al., 2009; Daumer et al., 2005). A large part of this solid phase has to be exported out of Brittany (usually as organic fertilizer) but this product has trouble finding its market. It cannot compete with compost because of its high P-content when organic matter is the customer's driving choice, and cannot compete either with mineral fertilizers because of the transport cost of organic matter when P is the component demanded.

Phosphorus recovery as struvite ($\text{NH}_4\text{MgPO}_4 \cdot 6 \text{H}_2\text{O}$), a slow-release fertilizer, would make the export of P more cost effective, meet the market demand for a concentrated, P-based mineral fertilizer and allow the organic matter in the digestate/compost to be maintained locally thanks to its low P content. Phosphorus recovery as struvite has been studied in a large variety of N and P concentrated waste streams: WWTP centrate liquor from anaerobic digestion (Battistoni et al., 2001; Jaffer et al., 2002), industrial

Abbreviations: ANOVA, Analysis of variance; Ca, Calcium; LAB, Lactic acid bacteria; Mg, Magnesium; MgO, Magnesium oxide; N, Nitrogen; P, Phosphorus; T, Total; TS, Total solids; TSS, Total Suspended solids; VS, Volatile solids; VSS, Volatile suspended solids; VFA, Volatile fatty acid.

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wastewater (Abma et al., 2010), municipal landfill leachate (Di Iaconi et al., 2010), dairy wastewater (Zhang et al., 2010) as well as swine lagoon effluent (Nelson et al., 2003), digested swine wastewater (Song et al., 2011; Vanotti et al., 2017; Ye et al., 2011) and raw swine manure (Burns et al., 2001; Çelen et al., 2007). Several full scale reactors have been built for struvite crystallization, such as the OSTARA, WASSTRIP (Cullen et al., 2013) and PHOSPAQ processes (Remy et al., 2013). However these technologies have been so far used only to treat municipal and industrial wastewater. In the case of animal manure, precipitation of phosphorus as struvite is still investigated at laboratory and pilot scales (Rahman et al., 2014), focusing mainly on reducing the dissolved phosphate concentration to meet the discharge standard for the liquid effluent. However, if the goal is to recover phosphorus, dissolving the inorganic solids that contain most of the phosphorus is necessary. It has been demonstrated that up to 70% of TP could be dissolved at pH 5.5 (Christensen et al., 2009). P recovery as struvite can therefore be achieved through acidic dissolution, solid-liquid separation and pH increase in the liquid phase using magnesium oxide (MgO) or magnesium hydroxide (Mg(OH)₂) leading to phosphorus precipitation (Capdevielle et al., 2016). The use of chemicals for acidic dissolution was demonstrated as too expensive and having a negative relative impact on the environment (Daumer et al., 2010). For the process to be efficient and sustainable, changing this acidification step is required. A biological process known as acidogenesis enables the natural acidification of a mixture under anaerobic conditions. During acidogenesis, organic matter is hydrolyzed and converted by fermenting bacteria into VFAs, lactic acid and alcohols. This acidifying reaction process is a crucial step in silage production (Dunière et al., 2013), a technic used to preserve the nutritive value of forage for cattle feeding (Buxton et al., 2003). Acidogenesis also occurs during anaerobic digestion of organic waste. The syntrophic cooperation of acidogenic, acetogenic and methanogenic organisms enables the conversion of organic waste into methane and carbon dioxide. Because acidogens have different optimal conditions compared to acetogens and methanogens (Liu et al., 2006), and especially a higher maximum growth rate, an overloading of a digester with easily fermentable substrates often leads to acidosis and inhibition of methanogens (McCarty and McKinney, 1961). As a result, the process can be physically separated into two consecutive reactors: one acidic and one methanogenic, each of them functioning under their respective optimum (Cohen et al., 1979). This two-stage process has been applied to crop residues (Kalia et al., 1992; Parawira et al., 2008), agro-industrial waste (Dareioti and Kornaros, 2014), and food waste (Uçkun Kiran et al., 2014).

Biological treatment of swine slurry in a two-stage anaerobic process has recently been studied (Ren et al., 2014; Schievano et al., 2012), often with the purpose of biohydrogen and methane production (Choi et al., 2015; Wu et al., 2010). It has been shown that a two-stage anaerobic digestion of fruit and vegetable waste mixed with swine manure at a ratio of 70/30 (w/w) lead to a pH of 4.2 in the acidogenic stage (Tenca et al., 2011), a pH at which phosphorus is known to be mostly dissolved in pig slurry (Sharpley and Moyer, 2000). Therefore, biological acidification of swine slurry with organic co-substrates could be proposed as the first stage in a bioprocess aimed at nutrient recovery (N & P) and green energy production (methane). Acidogenesis would dissolve P from pig slurry at lower cost than chemical acidification while providing VFAs and potentially increasing the biodegradability of the waste for the subsequent anaerobic digestion.

This batch test study focuses on the biological acidification of pig slurry using various concentrations of sucrose as a model organic co-substrate, adapting the methods developed by Braak

et al. (2016) and Guilayn et al. (2017) as a first step for phosphorus recycling. Biological acidification of pig slurry using glucose, cellulose or lactic acid has been tested by Hjorth et al. (2015) to minimize ammonia emission by lowering the pH. The results in terms of pH decrease and organic acids produced were very similar to this study. The objective here was to determine the relationship between on one hand initial amount of sucrose and slurry's characteristics (initial pH, buffer capacity) and on the other hand pH change, fermentation products and dissolution of phosphorus, magnesium, ammonia (the three molecules forming struvite) and calcium (which leads to the undesired precipitation of calcium phosphate). This endeavor could then translate into a targeted acidification step using real organic waste that would maximize P-dissolution, favor struvite formation over calcium phosphate and minimize the amount of reactant necessary to re-increase the pH.

2. Materials and methods

2.1. Pig slurry

The raw swine slurry ("slurry 1") was collected at a commercial pig fattening farm in Melesse (Brittany, France). Its composition can be found in Table 1. It was used to study the metabolism of acidogenesis in details. Three additional pig slurries (see Table 1) were also tested in order to correlate the level of sucrose added with the initial and lowest pH reached, as well as the buffer capacity of the slurry. The buffer capacity was measured in a beaker containing 100 mL of raw pig slurry with a magnetic stirrer, a pH probe and a graduated burette containing 2N sulfuric acid. The buffer capacity was calculated as the concentration of sulfuric acid necessary to reach pH 4, the lowest pH obtained during biological acidification of the slurries (pH 3.99 was reached in slurry 2 with 60 g/L of sucrose).

2.2. Biological acidification of pig slurry in batch tests using sucrose as co-substrate

One liter bottles were inoculated with 640 g of raw pig slurry 1 and six different sucrose concentrations (10, 20, 30, 40, 50 and 60 g/kg-slurry, in reactors called R10, R20, R30, R40, R50, R60 respectively) in triplicate, with an additional bottle inoculated with slurry only and used as a control. The bottles were sealed once inoculated. Temperature was set at 38 °C on heating plates and magnetic stirrers provided continuous mixing at 300 rpm. Samples were collected simply by removing the lid and pouring the liquid from the bottles. Contrary to previous batch tests with wastewater sludge, purging the gas in the head-space of the bottles at the beginning and after sample collection had no effect on the system when applied to pig slurry, therefore the bottles were never flushed with inert gas. Sample collection occurred after 12, 24, 36, 48, 72 and 96 h. The same protocol was applied to slurry 2, 3 and 4 using only 5 different sucrose concentrations for each, from 0 to 60 g/L, in order to investigate the variations between slurries.

2.3. Analysis

pH was measured using a WTW probe right after each sample collection. Total solids (TS), volatile solids (VS), total suspended solids (TSS), and volatile suspended solids (VSS) were measured in triplicates on the raw slurry with standard methods (APHA, 1998). An acidic dissolution of the ashes was realized to measure TP, T-Ca and T-Mg. 200 mg of ashes were added to 0.5 g of K₂SO₈ and 5 mL of a mix of H₂SO₄/HNO₃ (75:25) in triplicate. The samples were autoclaved at 110 °C during one hour at 1 bar.

The concentrations in TP, T-Ca and T-Mg (expressed in gram per

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