#### Journal of Environmental Management 157 (2015) 1-7

Contents lists available at ScienceDirect



Journal of Environmental Management

journal homepage: www.elsevier.com/locate/jenvman

## Phosphorus recovery from pig manure solids prior to land application

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#### ARTICLE INFO

Article history: Received 17 January 2015 Received in revised form 6 April 2015 Accepted 7 April 2015 Available online

Keywords: Phosphorus Nitrogen N:P ratio Pig manure Nutrient recovery Animal waste

### ABSTRACT

Land disposal of pig manure is an environmental concern due to an imbalance of the nitrogen to phosphorus (N:P) ratio for crop production, leading to excess phosphorus (P) in soils and potential risks of water pollution. A process called "quick wash" was investigated for its feasibility to extract and recover P from pig manure solids. This process consists of selective dissolution of P from solid manure into a liquid extract using mineral or organic acid solutions, and recovery of P from the liquid extract by adding lime and an organic polymer to form a P precipitate. Laboratory tests confirmed the quick wash process selectively removed and recovered up to 90% of the total (TP) from fresh pig manure solids while leaving significant amounts of nitrogen (N) in the washed manure residue. As a result of manure P extraction, the washed solid residue became environmentally safer for land application with a more balanced N:P ratio for crop production. The recovered P can be recycled and used as fertilizer for crop production while minimizing manure P losses into the environment.

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#### 1. Introduction

Land application of large amounts of manure generated by animal feeding operations is an environmental concern often associated with excess P in soils and potential risks of water pollution. Because of a disproportion of N and P contents in pig manure and harvested crops, repeated applications at optimal N rates for crop growth result in accumulation of P in soils. For instance, the average N:P ratio in plant biomass of most grain and hay crops is 8:1, whereas animal manure has a smaller N:P ratio (<4:1) (Zhang et al., 2003). Due to this nutrient imbalance, eutrophication of rivers and lakes can be accelerated as excess soil P is lost to aquatic environments through soil leaching and runoff (Vitousek et al., 2009; MacDonald et al., 2011). To reduce adverse effects of manure P losses into the environment, a substantial amount of P needs to be moved off the pig farm by transporting manure to P-deficit croplands (NRCS, 2003). However, transportation of pig manure in bulk becomes less cost and energy-effective with increasing distance from manure production sites (Keplinger and Hauck, 2006). Ideally, manure management in concentrated livestock regions could include new technologies for the recovery of manure P in a concentrated, usable form. This approach would make more economical the long distance transfers of manure P while reducing

\* Corresponding author. E-mail address: ariel.szogi@ars.usda.gov (A.A. Szögi). both agronomic P imbalances and adverse effects of soil P losses on water resources.

A number of physical, chemical, and biologic processes have been identified as potentially useful for the recovery P from animal manure and biosolids (Szögi and Vanotti, 2009; Vaccari, 2011). In particular, there is much interest on P recovery from solid animal manure by wet extraction of P from the byproducts of thermal treatment. In the thermal treatment approach, the organic matter is destroyed through incineration, pyrolysis, gasification, or liquefaction, and the phosphorus is extracted from the residue. For instance, P was recovered in a two-step precipitation process from chicken manure incineration ash extracted with a 1.0 M hydrochloric acid solution. Heavy metals impurities were first precipitated at pH 3.05 using sodium hydroxide and removed by filtration. Phosphorus was then recovered by raising the pH of the filtrate to 8.2 forming a precipitate containing 92% of its P as calcium phosphate (Kaikake et al., 2009). Another thermal method for conversion of biomass is pyrolysis, which produces a combination of gas, liquid fuel, and charcoal also known as biochar (Cantrell et al., 2008). Following pyrolysis of manure, P can be recovered from the biochar after wet extraction using mineral acids. For instance, about 92%-97% of the P present in fresh manure was recovered in the biochar fraction, much of which (60%-75%) was extracted as inorganic P using a 0.2 M sulfuric acid solution (Azuara et al., 2013). Thermal conversion processes such as incineration and pyrolysis require a relatively dry feedstock such as poultry litter or beef feed lot manure (Cantrell et al., 2008). Other thermal processes such as

wet pyrolysis and supercritical water gasification can use wet feed stocks such as liquid animal manure, and recover P from their char products by precipitation after acid extraction (Libra et al., 2011; Yanagida et al., 2009). These thermal conversion methods can provide byproducts rich in recoverable P but partial or total loss of N during thermal conversion make them less attractive for use as balanced N and P sources for crop production.

As an alternative to improve the N and P balance in manure byproducts, a new treatment process called "quick wash" was developed for rapid wet extraction of P from raw solid manure and recovery of manure P in solid concentrated form (Szögi et al., 2014). This process consists of selectively extracting P from solid manure using mineral or organic acid solutions, and recovery of P from the extract by adding lime and an organic polymer forming a calciumcontaining P precipitate. The quick wash process has three products: 1) a washed solid with a N:P ratio optimal for use in crop production; 2) a concentrated solid P material that can be transported long distance and used as an effective P fertilizer; 3) a liquid effluent that could be applied to nearby cropland as liquid fertilizer or recycled into the treatment system. Compared with thermal methods described above, the quick wash process avoids loss of oxidizable organic C and N from treated manure because it is conducted at ambient temperature. Therefore, the washed manure would be more environmentally safe for land application given that its N:P ratio is better balanced to match specific crop needs. In addition, the recovered P product could be transported to P-deficient croplands for use as a plant fertilizer. The objectives of this paper were to gain new insights into this new treatment process, and to evaluate its technical feasibility for improving pig manure N:P ratio prior to land application while reducing the potential P losses into the environment.

#### 2. Materials and methods

#### 2.1. Basic process configuration

The basic quick wash process includes three steps (Szögi et al., 2014): (1) selective P extraction, (2) P recovery, and (3) P recovery enhancement (Fig. 1). In the first step, manure solids are mixed with an acidic solution to form a washed solid residue (with a higher N:P ratio than the untreated manure solids) and a liquid P extract. In step 2, P is precipitated under alkaline conditions by adding lime to the liquid extract, and in the final step (step 3) the addition of an organic anionic polymer, polyacrylamide (PAM), enhances the formation and recovery of a calcium-containing P precipitate.

#### Table 1

Rates of acids applied to manure solids for extraction of P in step 1 of the quick wash process.

Treatment	Acid rate	Citric	HCl <sup>a</sup>
	(mmol $L^{-1}$ )	$(g L^{-1})$	
0	0	0	0
1	2.5	0.48	0.24
2	5	0.96	0.49
3	10	1.92	0.98
4	20	3.84	1.96
5	40	7.69	3.92
6	80	15.37	7.84

<sup>a</sup> 37.2% HCl with specific gravity 1.19 at 15 °C.

#### 2.2. Experimental

The feasibility of applying the quick wash process to pig manure was evaluated by performing the following two sets of experiments: 1) selective extraction of P from pig manure solids using either organic (citric) or mineral (hydrochloric) acids; and 2) P precipitation and recovery by precipitation under alkaline pH without and with precipitation enhancement by anionic PAM addition.

For the first experiments, pig manure was collected from a farrow to finish farm in Florence Co., SC that housed 280 heads in a barn that used an open gutter and flushing system to handle the manure. A composite fresh manure sample was collected from the inclined gutter between flushes using a shovel and placed in a 20-L plastic container, transported on ice to the laboratory, and stored at 4 °C until analysis. The manure sample had a fresh weight content of 8.1 g TN kg<sup>-1</sup>, and 3.9 g TP kg<sup>-1</sup> with 78.7% moisture, or 37.8 g TN kg<sup>-1</sup>, and 18.2 g TP kg<sup>-1</sup> on dry weight basis, and an equivalent dry weight P<sub>2</sub>O<sub>5</sub> content of 4.2%. For the second set of experiments, manure was obtained from a barn with a flush system under a slatted floor at the Pig Unit, North Carolina State University, Lake Wheeler Road Field Laboratory, Raleigh, NC. To obtain the pig manure solids, the flushes were held for 24 h to allow manure solids accumulate under the slatted floor. Again, manure solids were collected with a shovel, placed in a plastic container, transported on ice to the laboratory, and placed in cold storage until the start of the experiment. This second manure sample had fresh weight contents of 9.9 g TN kg<sup>-1</sup>, and 7.1 g TP kg<sup>-1</sup>, with 70% moisture, or 33.1 g TN kg<sup>-1</sup>, and 23.7 g TP kg<sup>-1</sup> on dry weight basis, and an equivalent dry weight P<sub>2</sub>O<sub>5</sub> content of 5.4%.

#### 2.2.1. First experiment: selective P extraction

Citric and hydrochloric (HCl) acids were tested for their ability to selectively remove P. Each acid was applied at six concentration levels (2.5, 5, 10, 20, 40, and 80 mmol  $L^{-1}$ ) plus one treatment

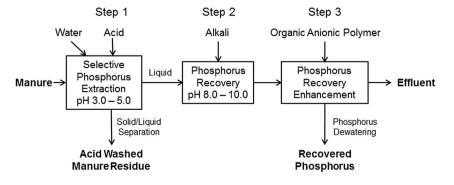


Fig. 1. Quick wash process schematic.

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