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## Use of solid phosphorus fractionation data to evaluate phosphorus release from waste activated sludge

S.P. Pokhrel <sup>a</sup>, M.W. Milke <sup>a,</sup>\*, R. Bello-Mendoza <sup>a</sup>, G. Buitrón <sup>b</sup>, J. Thiele <sup>c</sup>

a Department of Civil and Natural Resources Engineering, University of Canterbury, Private Bag 4800, Christchurch 8140, New Zealand <sup>b</sup> Laboratory for Research on Advanced Processes for Water Treatment, Instituto de Ingeniería, Unidad Académica Juriquilla, Universidad Nacional Autónoma de México, Blvd. Juriquilla 3001, 76230 Queretaro, Mexico

<sup>c</sup> Calibre Consulting, 323 Madras St., Christchurch 8013, New Zealand

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#### 1. Introduction

Waste activated sludge (WAS), derived from municipal wastewater treatment plants (WWTP), contains relatively high amounts of phosphorus (P), and is a potential source of recoverable P. [Cordell](#page--1-0) [et al. \(2011\)](#page--1-0) report activated sludge with 1.4% P by dry weight, while anaerobically digested sludge with 0.6% and urban compost with 0.4% P. Because of concern with eutrophication, WWTP can also be designed with chemical and biological processes to remove even more P in secondary sludge. WWTP sludge can become an expensive solid waste management problem. Dewatering followed by either incineration or landfill can be expensive, and can make P recovery of treated sludge challenging (for incinerator ash) or impossible (for landfill). Improving methods to recover P from WWTP sludge prior to landfill or incineration could improve the rate of reuse [\(Peccia and Westerhoff 2015; Garcia-Albacete et al.,](#page--1-0) [2012\)](#page--1-0), and would have environmental and resource benefits ([Sorensen et al., 2015\)](#page--1-0).

Phosphorus mass transfer from the solid phase into solution is a complex process and involves physical conditions (such as temperature), chemical factors (acid, alkaline, oxidation-reduction potential effects, and concentrations of other elements like iron or calcium), and biological processes (such as uptake, storage, growth,

⇑ Corresponding author. E-mail address: [mark.milke@canterbury.ac.nz](mailto:mark.milke@canterbury.ac.nz) (M.W. Milke).

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#### **ABSTRACT**

Waste activated sludge (WAS) can become an important source of phosphorus (P). P speciation was examined under anaerobic conditions, with different pH (4, 6 and 8) and temperatures (10, 20 and 35 C). Aqueous P was measured and an extraction protocol was used to find three solid phosphorus fractions. A pH of 4 and a temperature of 35 °C gave a maximum of 51% of total P solubilized in 22 days with 50% of total P solubilized in 7 days. Batch tests indicate that little pH depression is needed to release nonapatite inorganic P (including microbial polyphosphate), while a pH of 4 rather than 6 will release more apatite inorganic P, and that organic P is relatively more difficult to release from WAS. Fractionation analysis of P in WAS can aid in design of more efficient methods for P recovery from WAS.

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death and biological release) ([Boström et al., 1988, Jiang et al.,](#page--1-0) [2008; Ortuño et al., 2000](#page--1-0)). These are in turn affected by the form in which P arrives at the treatment facility and the unit processes used at the facility. An understanding of P release from WAS must rely on an understanding of the forms of P and their transformation processes.

In typical WAS, dissolved or aqueous P comprises a small fraction of total P (TP) [\(Ruban et al., 1999](#page--1-0)), and is mostly in the orthophosphate species. The inorganic solid forms of phosphorus can be separated into a fraction either loosely bound by electrostatic forces, or more tightly bound to exchange sites on minerals or cells [\(Ruttenberg, 1992\)](#page--1-0); this fraction is referred to herein as non-apatite inorganic phosphorus (NAIP). The second inorganic fraction of P found is in solid mineral compounds generally referred to as apatite P or Ca-bound P (herein termed AP) ([Ruban](#page--1-0) [et al., 1999](#page--1-0)), but which can also include Mg and Fe forms. Organic P (OP) in WAS solids is a complex fraction reflecting the many uses of P in bacteria. A final form of solid P is biologically stored inside cells as polyphosphates. This P form can be converted readily into soluble orthophosphate forms either within cells or after cell death, and is more similar to sorbed-P than to P incorporated into complex organic molecules. Phosphorus-accumulating microorganisms uptake P under aerobic and anoxic conditions and store it as polyphosphates, and then in anaerobic conditions release the P from storage as a source of energy to ensure survival when other aerobic bacteria lose activity ([van Starkenburg et al., 1993\)](#page--1-0).

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Stimulation and selection of these organisms in WAS is central to phosphorus removal and phosphorus recovery, and so the potential for recovery of P from WAS is dependent on the exact design of the biological wastewater treatment system.

Past research on waste activated sludge indicates that anaerobic conditions are more conducive to the release of P than aerobic conditions. In addition, pH is one of the dominant factors in P release as it affects sorption-desorption and precipitation-solubilization ([Chen et al., 2007; Xu et al., 2015; Latif et al., 2015](#page--1-0)). pH also changes biological reactions because it can stimulate or inhibit growth of specific micro-organisms that uptake or release P, while also affecting cell death and so P release from cell lysis.

[Chen et al. \(2007\)](#page--1-0) show that volatile fatty acid production and release of phosphorus from waste activated sludge varies over the range of pH 4.0–11.0. Anaerobic incubation of WAS resulted in significant increases of soluble phosphorus (PO $^{3-}_{4}$ P). The levels of phosphorus in the mixed liquor appeared to depend on sludge retention time and pH values. The concentrations of  $\mathrm{PO_4^{3-}P}$ reported were the highest at acidic pH (pH 4.0 and 5.0. The study did not report on the sources of the solid P released making it difficult to attempt to generalize the results to other WAS. [Xu et al.](#page--1-0) [\(2015\)](#page--1-0) report on the increase in soluble P and the changes in the solid P fractions; however, the study examined short reactions times of at most 150 min, with pH as low as 2 needed to release large fractions of P in the short time.

Although the release of P from WAS is highly dependent on the form of the P, there is a weak understanding of the relationship between P solubilization and P forms under conditions that might be applicable to WWTP when considering WAS treatment for P recovery. The aim of this study is to examine P release from WAS with a view to providing advice on P recovery based on solid P fractionation analysis.

#### 2. Material and methods

#### 2.1. Source of WAS

The WAS used in this study was obtained from a WWTP located in Christchurch, New Zealand, that receives municipal wastewater with a small industrial contribution. That plant uses primary sedimentation, a trickling filter as pre-treatment of a fraction of the wastewater, and then activated sludge aeration tanks with fine bubble diffusion. The sludge was directly collected from the main WAS pipe. Samples were placed in air-tight 20 L plastic containers, transported to the laboratory, and analyzed for initial parameters of WAS within one hour of collection. In the same day, the different treatments (e.g., pH adjustment) were applied and the P release tests started.

#### 2.2. Test procedures

P release batch experiments were performed in 1 L Schott Duran bottles. A similar amount of sludge (1 L) was put into each bottle. The bottles were capped tightly and incubated at three different temperatures (10, 20 and 35  $\degree$ C), and mixing was continuous with magnetic stirrers. The pH of the sludge was adjusted to 4, 6 and 8 by adding 1 M NaOH or 1 M HCl solution prior to testing. Every 12 h the pH was measured and readjusted to 4, 6 and 8 if any fluctuation was noticed. The readjustment process was continued until the desired pH had been maintained on two subsequent tests in 12 h intervals. Twenty ml samples from each bottle were withdrawn every 24 h to follow up P release kinetics. The sampled aliquots were immediately centrifuged in 50 ml centrifuge tubes at 4000 rpm (roughly, 1250g) for 10 min; temperature was monitored and changed less than  $2^{\circ}$ C during this step. The supernatant was used for the analysis of dissolved reactive phosphorus (DRP) and total dissolved phosphorus (TDP).

After centrifugation, the sludge residue was dried and ground to obtain a fine powder. The powdered samples were preserved in sealed plastic pots at room temperature. These preserved samples were used to analyze P fractionations.

Chemical analysis of the supernatant fractions for DRP and TDP were performed using a molybdovanadate method that forms a yellow complex measured by spectrophotometry at a wavelength of 430 nm. DRP as  $PO_4^{3-}$  mg/L was measured on the undigested sample, and TDP was measured after an acid persulfate digestion method. The instrument was calibrated from standard solutions with each use.

P fractions in the preserved solid residue sludge samples were determined using an extraction protocol previously applied by other researchers to fractionate the phosphorus in sewage sludge samples [\(González Medeiros et al., 2005](#page--1-0)) and in soil samples ([Zehetner et al., 2008\)](#page--1-0). This protocol determines a set of P fractions: total, inorganic P, organic P and apatite and non-apatite inorganic P. Non-apatite inorganic phosphorus was extracted by NaOH and HCl. Apatite phosphorus (Ca–P) was extracted by HCl. In a separate extraction, inorganic phosphorus (IP) was extracted by HCl, and the residual was treated at 450  $\degree$ C followed by HCl extraction as organic phosphorus (OP) in the solid phase. For all extractions, after treatment the sample was centrifuged and passed through a 0.45 um filter to separate the extract from the residue. Total phosphorus (TP) in the solid phase was determined by processing the samples at 450 $\degree$ C, and then extraction by HCl. The pictorial description of these methods is provided in [Appendix A.](#page--1-0) In all cases, phosphate concentrations of extracted solution were determined spectrophotometrically.

#### 2.3. Reproducibility testing

To test the variability of the P release results, five replicate reactors were run for 12 days. The coefficient of variation (standard deviation/mean) of the DRP and TDP of the five replicates over the eight sampling days averaged approximately 5%.

Four replicates of the solids fractionation were conducted on biosolids after anaerobic digestion collected from the Christchurch WWTP using the solid P fractionation protocol. The standard deviations were all 0.4 mg  $P/g$  or less, showing high replicability. The fractionation method also demonstrated internal consistency between the three sub-samples with values agreeing to within 10%.

The results presented in this paper are for one batch of WAS. Because of the need to confirm the accuracy of the methods and check on reproducibility, not all experiments could be conducted with the same sample of WAS. Over different batches of WAS, the phosphorus release pattern under control conditions (room temperature, without pH adjustment and anaerobic conditions) showed similar trends. Separate tests of the effects of pH and temperature were conducted using two batches under slightly different conditions. High reproducibility was found between separate batches of WAS. More details are provided in [Appendix B](#page--1-0).

#### 3. Results

#### 3.1. Characterization of P in WAS

The WAS was characterized within one hour of sampling for both dissolved and solid forms of P. Initial pH was 6.9. Total solids were 4900 mg/L. [Fig. 1](#page--1-0) shows the initial P forms in both the water and solid phases. The WAS had roughly 93 mg P/L with 92% in the sludge solids.

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