



Acidified and ultrafiltered recovered coagulants from water treatment works sludge for removal of phosphorus from wastewater



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ABSTRACT

This study used a range of treated water treatment works sludge options for the removal of phosphorus (P) from primary wastewater. These options included the application of ultrafiltration for recovery of the coagulant from the sludge. The treatment performance and whole life cost (WLC) of the various recovered coagulant (RC) configurations have been considered in relation to fresh ferric sulphate (FFS). Pre-treatment of the sludge with acid followed by removal of organic and particulate contaminants using a 2kD ultrafiltration membrane resulted in a reusable coagulant that closely matched the performance FFS. Unacidified RC showed 53% of the phosphorus removal efficiency of FFS, at a dose of 20 mg/L as Fe and a contact time of 90 min. A longer contact time of 8 h improved performance to 85% of FFS. P removal at the shorter contact time improved to 88% relative to FFS by pre-acidifying the sludge to pH 2, using an acid molar ratio of 5.2:1 mol H⁺:Fe. Analysis of the removal of P showed that rapid phosphate precipitation accounted for >65% of removal with FFS. However, for the acidified RC a slower adsorption mechanism dominated; this was accelerated at a lower pH. A cost-benefit analysis showed that relative to dosing FFS and disposing waterworks sludge to land, the 20 year WLC was halved by transporting acidified or unacidified sludge up to 80 km for reuse in wastewater treatment. A maximum inter-site distance was determined to be 240 km above the current disposal route at current prices. Further savings could be made if longer contact times were available to allow greater P removal with unacidified RC.

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1. Introduction

Coagulation and flocculation is a key process at potable water treatment works (WTW). Whilst still considered a low-cost treatment method (accounting for ~5% of the total cost of water production and distribution, Niquette et al., 2004), it nonetheless consumes >325,000 tonnes of coagulant annually in the UK alone (Henderson et al., 2009). This generates >182,000 tonnes of waste sludge in the form of water treatment residuals (WTRs) (Pan et al., 2004), demanding significant costs for its disposal (UKWIR, 1999).

Wastewater treatment works (WWTW) also require coagulant to remove phosphorus. In China, industrial effluents are required to meet 0.5 mg/L P (Pan et al., 2009) and for protected waters in Europe and North America consents could become 50 µg/L and 10 µg/L (Remy et al., 2014; Sengupta and Pandit, 2011). Coagulants offer a simpler means of removing P compared to biological

nutrient removal (Blackall et al., 2002) but require 2–3-fold higher doses when P consents move from 2 mg/L to <1 mg/L (Ofwat, 2005) as they become less efficient at higher removals. Reuse of alternative chemical P removal agents could offer a more sustainable and cost effective treatment option for water and wastewater utility companies (Babatunde and Zhao, 2007). P removal from wastewater using WTRs is already widespread, as disposal of WTRs to sewer is convenient and frugal as it avoids sludge dewatering, haulage and disposal fees (Walsh, 2009). However, this approach is limited because fewer than 30% of the WTWs in the UK have a sewer connection. Furthermore, when WTRs are disposed to the sewer, it is usually carried out on an *ad hoc* basis with limited control on the process (UKWIR, 1999; Walsh, 2009).

Reuse of acid-recovered coagulants from WTRs has already been considered in potable treatment (Okuda et al., 2014). Recycling coagulant reduces coagulant demand and waste production. However, the acidification process required is non-selective and the carryover of organic compounds with the coagulant elevates formation of disinfection by-products if used in potable treatment (Keeley et al., 2014a). Numerous purification methods have been

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documented but at present none adequately combine selectivity with feasible implementation (Keeley et al., 2014b).

Reusing recovered coagulants (RCs) in wastewater treatment can provide similar advantages as reuse in potable treatment but is less sensitive to the presence of impurities. WTRs have proven to be effective and economically viable in a number of wastewater treatment configurations (King et al., 1975; Masides et al., 1988; Parsons and Daniels, 1999; Jiménez et al., 2007; Yang et al., 2014). However, the underlying removal mechanisms remain poorly understood (Thistleton et al., 2002; Szabo et al., 2008). This study aims to compare the removal mechanisms and the whole life cost (WLC) of several WTR reuse approaches with conventional chemical P removal using fresh coagulants.

Ferric coagulants typically remove 95–96% of P after 90 min and $M^{3+}:P$ molar ratios of 2–4:1 (Parsons and Daniels, 1999; Szabo et al., 2008) using two main mechanisms: precipitation and adsorption (Hsu, 1976). Firstly, metal sulphate or chloride salts rapidly hydrolyse, forming metal hydroxides and, when phosphorus is present, metal phosphates. Optimal mixing (average G values $> 100 \text{ s}^{-1}$; Szabo et al., 2008) and a $\text{pH} < 9$ (Galarneau and Gehr, 1997) can minimise wasted chemical and surplus sludge production (Thistleton et al., 2002) and allow rapid removal of up to 100 times more phosphate per mol of Fe than adsorption (Smith et al., 2008). Phosphate precipitation can be enhanced further by removing competing hydroxide species at pH s of 4.5–5.0 (Thistleton et al., 2002). As coprecipitation hydrolysis occurs, the precipitate particles grow in size (Takacs et al., 2006), before aggregating and settling (Jarvis et al., 2006).

Secondly, adsorption occurs through contact of phosphates with iron hydroxides (Yang et al., 2010). These have a high phosphate removal capacity ($\sim 340 \text{ mg P/g Fe}$ after 36 h) but at a much slower rate ($\sim 0.5 \text{ mg P/g Fe/minute}$; Parsons and Daniels, 1999) than for precipitation ($\sim 150 \text{ mg P/g Fe/minute}$, initially; Szabo et al., 2008). Phosphate adsorption onto the metal hydroxide surface is fast but limited by slow phosphate migration within the metal hydroxide micropores which has been estimated to be as slow as $< 4 \times 10^{-15} \text{ cm}^2 \text{ s}^{-1}$ (Makris et al., 2004; Wang et al., 2011).

Using a lower pH to neutralize hydroxides released by phosphate adsorption can increase adsorption efficacy by 2–3 fold (Razali et al., 2007; Babatunde et al., 2009). Unacidified WTRs and chemically similar ferric hydroxide media can match the performance of FFS (fresh ferric sulphate) in various configurations (Babatunde et al., 2009; Bai et al., 2014). However, the reliance of adsorption for P removal requires ten times higher molar doses of 50:1, $M^{3+}:P$ (Genz et al., 2004) than coagulants with the additional capability to remove P using the precipitation pathway. Solubilisation of WTRs by acidification to $\text{pH} 2$ can increase the chemical efficiency of P removal by facilitating precipitation pathways (Parsons and Daniels, 1999; Jiménez et al., 2007) and by favouring phosphate uptake by adsorption. The cost of acidification may be offset by the value of greater P removal efficiency than if WTRs were dosed at ambient pH . The contribution each mechanism makes is dependent on many factors but some suggest that adsorption dominates, accounting for 65% of total P removal (Yang et al., 2010). Other studies report that when sufficiently mixed to maximize precipitation, adsorption accounts for only 25% of total removal (Smith et al., 2008).

Understanding how the P removal mechanisms operate when using recovered RCs that have undergone varying degrees of purification is a very under explored area of research but is an essential consideration for the appropriate addition of WTRs into wastewater for P removal. These varying contributions are important considerations in the use of WTR-based P removal and were examined alongside other chemical and physical factors, in terms of their effect on performance and process economics, relative to FFS.

2. Methodology

2.1. Assessing RC treatment performance

Jar tests were used to replicate chemical treatment of primary wastewater and to examine the removal performance and treated effluent quality. Various forms of ferric based RCs were compared against the performance of commercial grade FFS (measured as 20% Fe). Screened municipal wastewater was collected daily from a 2000 population equivalent WWTW (Cranfield, UK). This wastewater was used for all jar test experiments (see Supporting Information (SI) A for details on the wastewater composition).

Dewatered sludge cake (14% dry solids; DS) was taken from a 120–180 MLD WTW treating upland water (Derbyshire, UK) that used ferric sulphate coagulant. Sludge cake (1 g, wet) was dissolved in 1 L of 0.1 M analytical grade nitric acid, before analysis for dissolved organic carbon (DOC) using a Shimadzu TOC-V analyser, and Fe using a PerkinElmer atomic absorption spectrometer (AAS). Acid demand and Fe solubilisation were measured with dilute sludge (1 g/L) titrated against dilute sulphuric acid.

A range of RC options were prepared from the sludge cake. These were: i) raw dewatered sludge RC; ii) unacidified RC (dewatered sludge diluted to 2.8% DS in deionised water); iii) Acidified RC (as previous but acidified using concentrated sulphuric acid, until the required pH was held); iv) Acidified and ultrafiltered RC (as previous followed by filtration through a 2 kD molecular weight cut off (MWCO) polyethersulfone membrane (Sterlitech Corporation, Kent, WA, USA), using apparatus previously described (Keeley et al., 2014b). See SI A for details on the RC chemical composition.

Jar tests were conducted at Fe doses of 0–50 mg/L for all RCs, using a Phipps & Bird PB-700 jar tester. The jar tester mixed cylindrical beakers containing 1 L of wastewater for 1 min at 200 rpm ($G = 128 \text{ s}^{-1}$), followed by 30 rpm ($G = 7.4 \text{ s}^{-1}$) for 15 min, and a 30 min unmixed settlement stage. Average velocity gradient conversions (G values) were taken from a previous study, using the same apparatus (Sharp et al., 2006). Samples were taken from the supernatant and immediately analysed for total P, total N and chemical oxygen demand (COD) using Hach cell test kits. Removal of contaminants was assessed by comparing its initial concentration with the concentration in the treated water. Residual Fe was analysed using atomic absorption spectroscopy (AAS). The sample pH and turbidity was also measured.

2.1.1. Examination of P removal mechanisms

Using an adaptation of a previous method (Szabo et al., 2008), jar tests were run using the different coagulants and were mixed with wastewater using a 90 s mix (200 rpm) and a 60 min mix (30 rpm). Samples were taken 2 min and 1 h after dosing with 20 mg/L Fe to determine P removal. These samples were immediately filtered (0.45 μm , nylon) and analysed for soluble P. This process was repeated with pre-hydrolysed and precipitated coagulants. To achieve pre-hydrolysis, the coagulants were adjusted to $\text{pH} 7$ prior to dosing. Acidified and unacidified RCs (2.8% DS) were fractionated using successive filtration through 840, 500, 210, 105, 60 and 10 μm polypropylene meshes (Spectrum Laboratories, Netherlands). Each fraction was analysed for Fe using AAS before being dosed into jar tests at normalised doses of 20 mg/L Fe.

2.1.2. Flocculation time and prolonged mixing

The optimum Fe dose was determined and repeated for all the coagulant types, with different flocculation durations of 5, 10, 30 and 120 min. Prolonged mixing for 2, 4, 8 and 24 h at 100 rpm ($G = 43 \text{ s}^{-1}$) was studied to simulate the effect of longer contact times that occur in settlement tanks or if Fe is dosed to the sewer, upstream of the WWTW ($\sim 1 \text{ h/km}$; Gutierrez et al., 2010). To

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