



The effect of solids residence time on phosphorus adsorption to hydrous ferric oxide floc



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ABSTRACT

The impact of solids residence time (SRT) on phosphate adsorption to hydrous ferric oxide (HFO) floc when striving for ultra-low P concentrations was characterized and an equilibrium model that describes the adsorption of P onto HFO floc of different ages was developed. The results showed that fresh HFO had a higher adsorption capacity in comparison to aged (2.8, 7.4, 10.8 and 22.8 days) HFO and contributed substantially to P removal at steady state. P adsorption onto HFO solids was determined to be best described by the Freundlich isotherm. P desorption from HFO solids was negligible supporting the hypothesis that chemisorption is the mechanism of P adsorption on HFO solids. A model that included the contribution of different classes of HFO solids (i.e. High, Low or Old, containing high concentration, low concentration or no active surface sites, respectively) to adsorption onto HFO from a sequencing batch reactor (SBR) system was found to adequately describe P adsorption onto HFO solids of different ages. From the model it was determined that the fractions of High and Low HFO decreased with SRT while the fraction of Old HFO increased with SRT. The transformation of High HFO to Low HFO did not limit the overall production of Old HFO and the fresh HFO solids contributed more to P removal at steady state than the aged solids.

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1. Introduction

Concerns over eutrophication in sensitive aquatic ecosystems have focused the wastewater community to produce effluents with ultra low P concentrations (i.e. <10 ug-P/L). In order to reliably meet these low effluent targets, chemical P removal with metal salts has been deemed to be necessary (Jenkins and Hermanowicz, 1991; Levesque et al., 2010; Benisch et al., 2013; Johnson et al., 2005; Johnson and Daigger, 2009). Removal of P with ferric chloride was the topic of this research.

A detailed understanding of the mechanisms of chemical P removal and models describing chemical interactions are required to effectively design P removal systems when extremely low P concentrations are targeted. Szabó et al. (2006) attributed the mechanisms of chemical P removal to co-precipitation and adsorption. Co-precipitation is defined as the simultaneous precipitation of a normally soluble component with a macro-component from the same solution by the formation of mixed

crystals, through adsorption, occlusion or mechanical entrapment (IUPAC, 1997). The addition of ferric salts to wastewater has been reported to result in the formation and rapid precipitation of HFO floc (Smith et al., 2008). PO_4^{3-} initially co-precipitates during the rapid HFO formation while subsequent uptake is slower and due to adsorption/complexation reactions on the surface of the HFO floc (Smith et al., 2008; Szabó et al., 2008). These complexation reactions involve iron and P sharing an oxygen atom (Smith et al., 2008). The availability of reactive oxygen atoms or “surface sites” has been found to depend on pH, mixing and aging.

Recycling solids through the biological treatment process provides additional solids retention time allowing for “slow” interactions between the liquid and solid phases. Recycling tertiary solids has been reported to enhance P removal and decrease metal dosing requirements (Takács et al., 2006; Newcombe et al., 2008; Takács et al., 2011; Falk et al., 2012; Johnson et al., 2012). However, recycling solids has also been found to lead to floc aging. Aging is expected to change the morphology of the precipitates. The transition from amorphous hydroxide floc to crystalline structures with floc aging is expected to impact on phosphate adsorption. Dzombak and Morel (1990) showed that aging decreased the adsorptive capacity of HFO. These findings were confirmed through experiments

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by Smith et al. (2008) where HFO aging lead to larger, denser structures which limited the surface area for binding and decreased the number of active surface sites. Reduced P removal as a result of aging has also been reported by Mao et al. (2012), Szabó et al. (2008) and Lijklema (1980). The aging described in these studies was over short time frames not typical of solid residence times (SRTs) observed in wastewater treatment processes. Therefore, a better understanding of the aging of chemical solids and the role of solids contact time and SRT on chemical use, achievable P limits, and adsorption mechanisms is required (Benisch et al., 2013).

Most studies have suggested that the mechanism of P adsorption on chemical solids is chemisorption where phosphate ions replace hydroxide ions (Briggs, 1996; Lijklema, 1980; Luedecke et al., 1989). Adsorption of P onto the surface of crystalline oxides such as goethite (α -FeOOH) has been well characterized (Weng et al., 2012; Kang et al., 2003; Rietra et al., 2001; Li and Stanforth, 2000; Geelhoed et al., 1997; Davis and Leckie, 1980; Golberg, 1985; Stumm et al., 1970; Dzombak and Morel, 1990; Hiemstra and Van Riemsdijk, 1996). However, this prior work focused on the use of crystalline oxides that had well-defined particle size and specific surface areas (Lijklema, 1980). Hence these studies can only be used as a starting point for understanding the mechanisms of removal via adsorption onto amorphous precipitates in wastewater. Experiments that have been conducted with fresh HFO were confounded by aging effects making quantitative interpretation difficult (Lijklema, 1980; Smith et al., 2008; Mao et al., 2012).

Further insight into the process of HFO aging was provided by Hauduc et al. (2013) and Takács et al. (2011) through the development of a model that combined chemical equilibrium, precipitation and co-precipitation reactions as well as adsorption and aging (surface consolidation) processes. Fig. 1 illustrates the model conceptually. From Fig. 1 it can be seen that iron addition results in the formation of two fractions of HFO (i.e. high HFO and low HFO) as a function of the mixing intensity (G) provided. High HFO is characterized by an open structure and a high specific surface area for adsorption while low HFO has a more compact structure and less accessible binding sites. P will co-precipitate with High HFO or co-precipitate/sorb onto low HFO. In addition, High HFO ages to form low HFO and low HFO ages to form Old HFO that contain no active surface sites but do have mechanically entrapped phosphates. The effect of SRT on the aging of HFO and thus the fractionation of these three types of HFO solids in wastewater treatment has yet to be quantified.

The objective of this work was to characterize the impact of SRT on phosphate adsorption to HFO floc. Batch adsorption experiments

were carried out with steady state HFO samples obtained from lab scale continuous flow sequencing batch reactors (SBRs) using synthetic natural water. A model to describe the overall adsorption behaviour of P onto HFO floc of different ages was applied to the data obtained.

2. Materials and methods

2.1. Materials

HFO flocs were collected from lab scale continuous flow sequencing batch reactors (SBR). The experimental set-up consisted of 4 SBRs (A–D) operating at one of four SRTs: 2.8 (A), 7.4 (B), 10.8 (C) or 22.8 (D) days. A schematic of the lab scale system is shown in Fig. 2. The SBRs were equipped with pH control, aeration, and mechanical mixing and allowed to react (floculate), waste, settle and decant. Table 1 summarizes the specific design parameters of the SBRs. SRT was controlled by adjusting the waste volumes weekly based on effluent and reactor solid concentrations, on the basis of the dynamic SRT model described by Takács et al. (2008). The SBRs were operated for 2 years under controlled conditions of pH, temperature and flow rates to minimize variability.

The SBRs were fed from a common flash mix tank (Table 1) that was fed with a synthetic natural water adapted from a recipe by Environment Canada (1990). The recipe (Table 2) was modified to provide alkalinity values typical of wastewater. A volume of stock phosphate solution was introduced into the flash mix tank to achieve the P concentrations in Table 2 via a fluid metering (FMI) pump. The stock phosphate solution (1 g PO_4^{3-} /L) was prepared by dissolving KH_2PO_4 in Milli-Q water. All chemicals used were reagent grade or better. All solutions were prepared with ultra pure water (Milli-Q, 18 M Ω).

The water in the flash tank was dosed with a stock iron solution to achieve a mean concentration of 16.1 mg Fe/L (95% CI of 13.3–19.0) under rapid mixing (350 rpm; 310 s^{-1}) and then transferred into each of the SBRs. The stock iron solution (1 g Fe/L) was prepared by dissolving $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in Milli-Q water. The resulting Fe:P molar ratio was 2.6. The iron dosing concentration was determined experimentally based on meeting effluent P concentrations in the range of 50 μg P/L under low influent P concentrations, while the mixing (rpm) corresponded to values reported by Szabó et al. (2008) and Gilmore (2009).

2.2. Sampling and sample preparation

Once steady state was achieved with respect to SRT the flash tank and SBRs were regularly sampled for characterization of P removal and to produce HFO solids that were employed in the off-line adsorption tests. Flash tank samples were collected after Fe dosing while the flash reactor contents were being transferred into the SBRs. SBR samples were collected from the waste and effluent streams. Waste samples were taken at the end of the react phase while the solids were still in suspension. Effluent samples were collected at the end of the decant phase. Grab samples were collected from the SBR during the react phase or from the flash mix tank while its contents were draining into the SBRs to obtain HFO solids for adsorption testing. Samples that were quantified for soluble P analysis were filtered immediately through a 0.45 μm nitrocellulose membrane filter (Whatman Millipore).

Flash tank and waste samples were also analyzed by microscopy. Microscopy samples were ‘washed’ to remove salts from the aqueous solution. The washing procedure involved centrifuging the samples to separate the floc from the aqueous solution, draining the aqueous solution, and resuspending the flocs in Milli-Q water. The procedure was repeated three times and the final aqueous

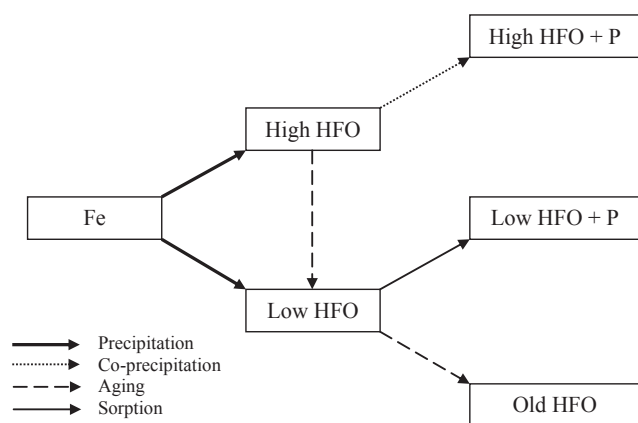


Fig. 1. Schematic of equilibrium-kinetic chemical P model (Hauduc et al., 2013; Takács et al., 2011) (adapted from Takács et al., 2011).

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