



Phosphorus removal performance and speciation in virgin and modified argon oxygen decarburisation slag designed for wastewater treatment



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ABSTRACT

Argon oxygen decarburisation (AOD) slag may be used for phosphorus (P) removal, as its high pH and weatherable calcium (Ca) minerals provide sufficient Ca^{2+} and OH^- for calcium phosphate (Ca-PO_4) precipitation. This study examined the P removal performance of AOD slag for use as wastewater treatment material. Batch experiments were carried out using both synthetic P solution and real wastewater, followed by chemical modelling and X-ray absorption near edge structure (XANES) spectroscopy. The influences of initial P concentration, slag dose and modification by polyethylene glycol (PEG), an effective agent for generation of porous materials, were investigated to determine the optimal conditions for P removal by AOD slag. It was found that virgin AOD slag removed 94.8% of P from a synthetic P solution in 4 h and 97.8% in 10 h. This high P removal was accompanied by a rapid increase in pH from 7.0 to 10.74. The maximum P removal capacity (PRC) from synthetic P solution ranged from 1.3 to 27.5 mg P g^{-1} . The optimal AOD dose for P removal from wastewater, determined in 8-h batch experiments, was 25 g L^{-1} . PEG modification increased the reaction rate and resulted in higher final pH, increasing PRC by 47.9%. Combined Visual MINTEQ and XANES analysis for detailed examination of P removal mechanisms revealed that the main P removal mechanism was precipitation of calcium phosphate. According to the XANES analysis, the main Ca-PO_4 precipitate formed on virgin AOD slag under low initial P concentration and high pH was apatite, while brushite was the dominant product at high initial P concentration and low pH.

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1. Introduction

Phosphorus (P) removal and recovery from wastewater is an important issue because of the rapid depletion of existing irreplaceable and indispensable P resources (Reijnders, 2014). A variety of physical, chemical and biological techniques have been developed and introduced to remove and recover P from wastewater. It is reported that a P recovery of >90% can be achieved by technologies such as acidic-alkaline sequential treatment (Petzet et al., 2012) and combined low pressure wet oxidation and nanofiltration (Blöcher et al., 2012). Although a high P recovery

can be achieved with these technologies, it is also desirable if alternative systems that are more energy- and cost-efficient are developed.

Phosphorus precipitation using slag for P removal and recovery from wastewater involves the capture of phosphate by reaction with metal cations dissolved from slag composed of calcium-rich oxides and silicates. It is considered an economical and effective technology for P removal from wastewater (Drizo et al., 2006; Kim et al., 2006; Gustafsson et al., 2008; Claveau-Mallet et al., 2013). So far, most research on P removal using slag has focused on: blast furnace (BF) slag, which is produced from the separation of iron from ore; electric arc furnace (EAF) slag, which is from the first step of stainless steel production; and blast oxygen furnace (BOF) slag, from the second processing step of carbon steel production (Santos et al., 2013a). The principal advantages of these slags are their wide availability and low cost due to their huge annual production

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volumes. Although they are by-products of different processes and their chemical composition varies, they share features such as high calcium content and alkalinity, which makes them favourable for P removal as they can provide sufficient Ca^{2+} and OH^- for the formation of calcium phosphate (Ca-PO_4) precipitates.

The most widely studied slag, BF slag, has been found to have a P removal efficiency (PRE) higher than 95% in laboratory experiments with both synthetic P solution and real wastewater (Oguz, 2004; Kostura et al., 2005; Rastas and Hedström, 2006; Johansson, 2010; Johansson Westholm, 2010). However its performance in pilot and field trials is less satisfactory, with P removal reported to range from 40% to 77% (Shilton et al., 2006; Asuman et al., 2007). Drizo et al. (2006) conducted column experiments to investigate the P removal performance of EAF slag and obtained consistently high PRE for 114 days. Bowden et al. (2009) investigated BOF slag as a reactive medium for P removal in both batch and continuous flow experiments for 406 days and observed high maximum P removal. Barca et al. (2012) evaluated EAF and BOF slags from four countries in Europe in terms of their P removal capacity using both synthetic P solution and real wastewater and found that BOF slag has a much higher maximum P removal capacity (PRC) than EAF slag. To our knowledge, research using argon oxygen decarburisation (AOD) stainless steel slag for P removal has not been reported previously.

As a by-product from the argon oxygen decarburisation refining process, 270 kg AOD slag are produced for every ton of stainless steel produced. The slag is mainly applied in cement production, road construction, civil engineering work, fertiliser production and landfill daily cover (Huawei and Xin, 2011). These do not completely consume the huge production volume and hence increasing amounts of AOD slag are accumulating. Alternative uses of AOD slag are therefore being investigated. AOD slag is reported to have a high alkalinity and to contain more than 35% Ca in the form of easily soluble calcium silicate (Kriskova et al., 2012; Santos et al., 2013a, 2013b). Hence it may be a promising wastewater treatment material for P removal and recovery via Ca-PO_4 precipitation.

It has been pointed out that the formation of calcium phosphates depends strongly upon the dissolution of calcium silicate from the slag. If BF slag is modified in an effective way the PRE is higher, since the modified slag surface with its higher porosity tends to release more Ca^{2+} and thus facilitate the precipitation of calcium phosphate (Gong et al., 2009). Inspired by previous research (Guan et al., 2014), a modification process using polyethylene glycol (PEG) was developed in this study, aimed at improving the P removal performance of AOD slag. PEG is a hydrophilic, non-toxic and water-miscible polymer used in controlled-release systems and in nanoparticle preparation (Ali and Lamprecht, 2013; García-Jimeno and Estelrich, 2013). It is produced in an industrial scale and low grade forms are available to a realistic price. In this study, it was mixed with AOD slag under hydrothermal conditions in order to facilitate the dissolution of Ca^{2+} by converting the original sparingly soluble calcium silicate species on the slag surface into more easily soluble forms.

This study explored P removal by AOD slag in an effort to devise a technically and economically feasible treatment solution that makes it possible to use this metallurgical by-product in wastewater treatment. To obtain reliable results when assessing AOD as a P removal material, batch experiments with both synthetic P solution and real wastewater were conducted to investigate the PRE and PRC of AOD. The influence of initial P concentration, slag dose and modification by PEG was investigated to determine the optimal conditions that maximise the PRC and P removal efficiency (PRE) of AOD slag. Visual MINTEQ and X-ray absorption near edge structure (XANES) spectroscopic analysis were combined for an in-depth study of the P removal mechanisms.

2. Materials and methods

2.1. Material

The AOD slag used in this study was obtained from Outokumpu Stainless AB, Sweden. This slag is porous and has a brown colour. The pH, determined by immersing the AOD slag in distilled water with a volume ratio of 1:2.5 for 24 h, was found to be 12.04. Chemical analysis revealed that the AOD contained 380 mg Ca g^{-1} and 150 mg Si g^{-1} (Table 1). The slag was crushed by hand in a mortar and sieved to get particle size fractions of 1–2 mm, 0.5–1 mm (labelled cAOD) and 0–0.5 mm (labelled fAOD). The last two fractions were used only for PEG modification. The particle size fraction of 1–2 mm was used in all other experiments because it is expected to be useful in practical applications. A wax-like PEG manufactured by Merck, with a molecular weight ranging from 950 to 1050 g mol^{-1} and a melting point at 33–40 °C, was used as the PEG treatment.

2.2. PEG1000 modification

Polyethylene glycol is a water-miscible polyether with varying molecular weight. In this experiment, PEG with a molecular weight of around 1000 was used (PEG1000). Silicates and calcium-rich solids on the slag surface can be transformed to calcium silicate hydrate (CSH) under hydrothermal conditions (Jing et al., 2007; Hosokawa et al., 2014). Guan et al. (2014) reported that the presence of PEG can effectively prevent the aggregation of small CSH particles during hydrothermal treatment, leading to the formation of CSH with larger surface area. The aim of the modification applied in the present study was to improve the reaction kinetics and the maximum PRC of AOD slag by modifying calcium silicate on the slag surface through hydrothermal treatment with PEG.

The modification process was as follows: a fixed amount of PEG was dissolved in deionised water at a solid:liquid ratio of 1:225 to form a PEG solution, which was stirred for 30 min to ensure complete dissolution. Then AOD slag with particle size of 0–0.5 mm (fine fraction) and 0.5–1 mm (coarse fraction) was added to the solution at a PEG:AOD slag mass ratio of 2:5. A control group was prepared by adding the same amount of slag to deionised water. All suspensions were then placed in a water bath for 2 h at 80 °C with a stirring rate of 60 rpm and later removed to an autoclave for hydrothermal treatment at 120 °C for 12 h (Guan et al., 2013, 2014). The suspension was cooled to room temperature. Centrifugation at 8000 rpm for 3 min was carried out to separate the AOD slag from the PEG solution. The modified AOD slag was dried at 105 °C and then collected in zip-lock plastic bags and referred to as fAOD-PEG (0–0.5 mm), cAOD-PEG (0.5–1 mm).

2.3. Experiment methods

The PRE of AOD was initially investigated in a series of batch experiments with synthetic P solution. A stock synthetic solution of 1000 mg P L^{-1} was prepared by dissolving KH_2PO_4 in distilled water. The solution was then stored in a refrigerator at 4 °C. The working P solution was prepared fresh daily by diluting a defined volume of stock solution with distilled water to an experimental concentration of 6.5 mg P L^{-1} , which is similar to the P

Table 1
Elemental composition of AOD slag (mg g^{-1}).

	Si	Mn	P	Al	Ca	Fe	Mg
AOD	150	4	0.04	9	380	3	33

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