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Mechanisms of phosphate removal from aqueous solution by blast furnace slag and steel furnace slag*

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Abstract: We report the adsorption of phosphate and discuss the mechanisms of phosphate removal from aqueous solution by burst furnace slag (BFS) and steel furnace slag (SFS). The results show that the adsorption of phosphate on the slag was rapid and the majority of adsorption was completed in 5~10 min. The adsorption capacity of phosphate by the slag was reduced dramatically by acid treatment. The relative contribution of adsorption to the total removal of phosphate was 26%~28%. Phosphate adsorption on BFS and SFS follows the Freundlich isotherm, with the related constants of k 6.372 and $1/n$ 1.739 for BFS, and of k 1.705 and $1/n$ 1.718 for SFS. The pH and Ca^{2+} concentration were decreased with the addition of phosphate, suggesting the formation of calcium phosphate precipitation. At pH 2.93 and 6.93, phosphate was desorbed by about 36%~43% and 9%~11%, respectively. These results indicate that the P adsorption on the slag is not completely reversible and that the bond between the slag particles and adsorbed phosphate is strong. The X-ray diffraction (XRD) patterns of BFS and SFS before and after phosphate adsorption verify the formation of phosphate salts ($CaHPO_4 \cdot 2H_2O$) after adsorption process. We conclude that the removal of phosphate by BFS and SFS is related to the formation of phosphate calcium precipitation and the adsorption on hydroxylated oxides. The results show that BFS and SFS removed phosphate nearly 100%, indicating they are promising adsorbents for the phosphate removal in wastewater treatment and pollution control.

Key words: Phosphate removal, Blast furnace slag (BFS), Steel furnace slag (SFS), Adsorption, Precipitation
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INTRODUCTION

In wastewater treatment and pollution control technology, inorganic sorbents, such as blast furnace slag (BFS), have been widely used for the removal of phosphate, heavy metals and organic pollutants (Dimitrova, 1996; 2002; Kostura *et al*., 2005; Ramakrishna and Viraraghvan, 1997; Dimitrova and Mehandjiev, 1998; 2000; Johansson, 1999a; Srivastava *et al*., 1997; Johansson and Gustafsson, 2000; Agyei *et al*., 2002; Khelifi *et al*., 2002; Jain *et al*., 2003; Oguz, 2004; 2005; Shilton *et al*., 2006; Drizo *et al*., 2006). The on-site wastewater treatment systems such as constructed wetlands (CSW) and soil filtration systems have been used for decades for the removal of phosphate in domestic, agricultural and industrial wastewaters (Johansson, 1999b; Mann and Bavor, 1993; Mann, 1997; Sakadevan and Bavor, 1998; Drizo *et al*., 1999; Ortiz *et al*., 2001). In CSW, industrial wastes slag is often used as the adsorbent or filter for a wetland substrate (Drizo *et al*., 1999; 2002; Johansson, 1999a). The major advantage of slag is cost effectiveness and abundance, which makes phosphate removal economical. BFS, a byproduct generated from iron and steel production, is a complicated mixture of alkali-earth silicates and alumino-silicates formed at high temperatures in the blast furnace. The ability of various types of slag to remove phosphate has been tested (Johansson and Gustafsson, 2000; Kostura *et al*., 2005; Drizo *et al*., 2006). High phosphate sorption capacity of BFS has been demonstrated by batch and column experiments as well as field research (Jo-

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hansson, 1999a; Johansson and Gustafsson, 2000; Kostura *et al*., 2005). Johansson and Gustafsson (2000) studied slag and opaka as a sorbing filter material for on-site wastewater treatment, and suggested the possible use of PO_4^{3-} saturated slag as fertilizer; when the P is retained in a hydroxyapatite form, it can be absorbed by plants. Khelifi *et al*.(2002) examined the adsorption of phosphate ions from seawater by acid-treated slag. Jha *et al*.(2004) used steel slag thermally activated at various temperatures to remove Ni^{2+}, NH_4^+ and phosphate.

It has been established that the removal/retention of phosphate by BFS is resulted from either adsorption or precipitation (Drizo *et al*., 2006; Kim *et al*., 2006). Although slag absorbs phosphate, mechanisms involved in the adsorption remain unclear. Understanding of P removal is necessary for both improving the design of wastewater treatment systems and predicting their long-term sustainability for phosphate removal under environmental conditions. This paper reports the removal of phosphate from aqueous solution by industrial slag, and discusses its mechanisms of phosphorus retention. The adsorption and precipitation of the BFS will be studied to evaluate their roles in removing phosphate from the aquatic environment.

MATERIALS AND METHODS

Materials

Two types of steel industry byproducts, the blast furnace slag (BFS) and steel furnace slag (SFS), were obtained from Hangzhou Iron and Steel Co. Ltd, China. BFS was formed when limestone, coke and iron ore were fused at high temperature in ironmaking furnace. A general chemical composition of BFS used in this study can be expressed on the basis of oxides as: SiO_2 36.11%, Fe_2O_3 0.63%, Al_2O_3 8.46%, CaO 36.23% and MgO 3.14%, although it varies with the composition of ores used. SFS was formed during the conversion of pig-iron to steel, a process that removes some impurities. The main composition of SFS includes SiO_2 16.11%, Fe_2O_3 20.63%, Al_2O_3 2.46%, CaO 35.33% and MgO 6.21%. The particle size of both BFS and SFS varies from 0.01 to 15 mm. Probationary tests showed that phosphate adsorption was greatly affected by the size of slag particles, and that the phosphate adsorption occurred mostly in fine particles (<0.1 mm). We, therefore, ground the slag into the grains of less than 0.1 mm in diameter in the batch experiment. Both BFS and SFS were treated with hydrochloric acid (1%, v/v) and washed with distilled water to remove residual acid and soluble compounds.

The pH of the slag was determined with a pH meter in 1:5 (w/v) mixture after equilibrium for 24 h. Electric conductivity (EC) was determined using a conductivity meter on a 1:5 slag/water mixture. The changes of pH of BFS and SFS were determined by recording the pH values of a suspension consisting of 20 g slag and 100 ml deionized (DI) water in every 1 h interval for a period of 24 h. The mineral composition of the slag was analyzed by X-ray diffraction (XRD) using a Rigaku D/Max 2550 PC (Rigaku Corption, Japan) diffractometer employing a Cu K_α radiation (40 kV, 40 mA) in the range of 2θ=2°~80° at a speed of 2°/min.

Batch adsorption experiments

Working solutions were prepared by diluting a standard phosphate solution (1000 mg/L) with DI water. The pH value of the phosphate working solution was adjusted to 6.7~6.8 using a diluted HCl or NaOH solution before adsorption experiments. 0.5 g of the treated or untreated slag was suspended into a 25 ml solution containing 500 mg/L phosphate in a 50 ml centrifuge tube. The tubes were mechanically agitated at room temperature (25 °C) and were taken for analysis after 5, 10, 15, 30, 45 min, 1, 2, 4, 8, 16 and 24 h. The solution was separated from the slag by filtration (0.45 μm filter paper) for determining the concentration of phosphate. The amount of phosphate sorbed was calculated by the changes of phosphate concentration. In the end of experiment, slag solids were collected and dried for XRD analysis.

Adsorption isotherm test: In a 50 ml centrifuge tube, 0.5 g sample was added to a 25 ml solution containing various concentrations of phosphate (50~8000 mg/L) and equilibrated by shaking at room temperature for 6 h. The suspension was then filtered through a 0.45 μm filter paper and the resulting filtrate was used for the analyses of phosphate, pH and Ca^{2+} concentration. The amount of phosphate sorbed was calculated by the concentration differences between the initial and measured solutions.

Desorption experiments

To determine the stability of the phosphate adsorbed by slag, desorption experiments were conducted. An extracting solution (pH 2.93) was prepared using acetic acid and the extraction fluids used in the EPA Toxicity Characteristics Leaching Procedure (USEPA, 1992). Another extracting solution (pH 6.93) was prepared by diluting HNO_3 and NaOH solutions. After adsorption tests, the remaining residual solids that had been treated with 100 and 500 mg/L phosphate were washed twice with DI water, and the supernatants were discarded immediately after centrifugation. The washed residues were then treated with 25 ml of extracting solutions (pH 2.93 or 6.93), and shaken at room temperature for 6 h. The suspension solution was filtered and analyzed for the concentration of the desorbed phosphate using the similar method described above. The amount of desorbed phosphate was determined by the amount of phosphate in solution after the desorption experiment.

Analytical methods

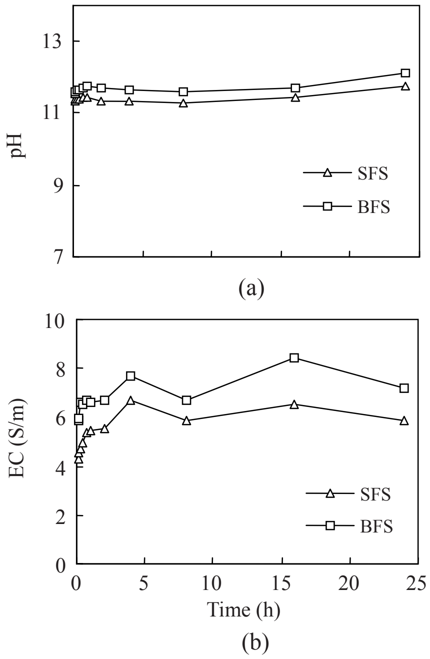
Concentration of phosphate was determined using the molybdenum blue spectrophotometric method according to the procedure stated in APHA (1995). The Ca^{2+} concentration was determined by EDTA titrimetric method (Klute, 1986). All measurements were performed in duplicate and the data was expressed as the average value and standard deviation.

RESULTS

Characterization of the sample

Fig.1 shows the changing kinetics of pH and EC in BFS and SFS. The final pH values of the untreated BFS after reaction in DI water were within a range of 12.0~12.2, while the acid treated samples had the final pH of 7.4~7.7. The pH of SFS varied in a range of 11.3~11.8 and decreased to 7.3 after acid treatment. The high pH value of the slag sample in DI water is due to the dissolution of Ca^{2+} into the solution. With an acid treatment, a large amount of calcium and salts were removed from the sample. The ECs of BFS and SFS were found to increase along with the increases of contact time with DI water, and finally reach 7.2 S/m for BFS and 5.8 S/m for SFS, respectively.

The pH of the solution plays a critical role in the phosphate removal rate. It was reported that chemical precipitation was dominant when pH was above 8, whereas adsorption became dominant when pH was below 6 (Lee *et al*., 1997). Thus, the adsorption phase of phosphate by the treated BFS and SFS could be the main process involved in the phosphate removal.



(a)

(b)

Fig.1 Changing kinetics of pH (a) and electric conductivity (EC) (b) as a function of contact time for BFS and SFS

Phosphate adsorption kinetics

The kinetics of phosphate removal by BFS and SFS are shown in Fig.2. The phosphate was rapidly removed in the first 5~10 min, and 83.0%, 86.8% and 89.8% of phosphate was eliminated after 15, 30 and 60 min, respectively. Two distinct stages of phosphate removal were identified. An initial large removal (almost 80%) lasting only a short time (5~10 min) was then followed by a further removal sustaining over a longer period of adsorption. The rapid initial removal is mainly attributed to the precipitation of phosphate with exchangeable and dissolved Ca^{2+} rather than the adsorption. The liberated Ca^{2+} from the exchange site or from the dissolution of $CaCO_3$, CaO and $Ca(OH)_2$ are preferably precipitated by phosphate in an alkaline solution, which gives a high initial rate of adsorption. While the precipitation stage has the greatest influence on the total removal of phosphate, the exact contribution of the adsorption and precipitation phases to the removal of phosphate remains unclear.

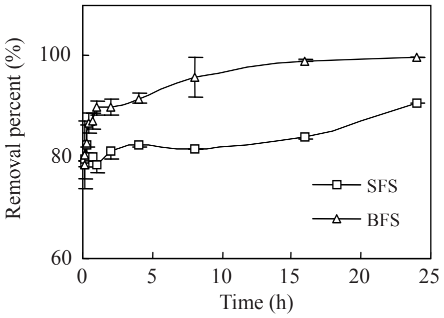


Fig.2 Kinetics of phosphate removal by BFS and SFS in the initial phosphate concentration of 500 mg/L. Bars represent one standard deviation of the mean of two replicates

The reductions of the Ca^{2+} and the pH of the solution by an acid treatment significantly influence the phosphate removal. The untreated BFS showed a maximum adsorption capacity, and the acid treatment reduced phosphate removal ability of BFS. The phosphate removal capacities of BFS and SFS were reduced dramatically by the removal of metals (Fig.3). It is expected that the slag containing $CaCO_3$ and $Ca(OH)_2$ adsorbs phosphate and promotes the precipitation of Ca phosphates. The comparison of the phosphate adsorption between the treated and untreated BFS and SFS clearly shows relative contributions of adsorption and precipitation to the total removal of phosphate. Fig.3 shows the changes of phosphate concentration after treatment. Phosphate concentration of the untreated BFS declined more rapidly than the treated BFS, due to its direct precipitation with calcium. The equilibrium relationship indicates that the capacity of the treated BFS is considerably reduced as compared with that of the untreated. In the current study, 26% (BFS) and 28% (SFS) of the total phosphate removals were estimated due to the slag adsorption. This observation is consistent with the findings of Johansson and Gustafsson (2000) and Agyei *et al.*(2002), who suggested that the predominant Ca-P precipitation via a surface adsorption of the slag contributes to the phosphate removal.

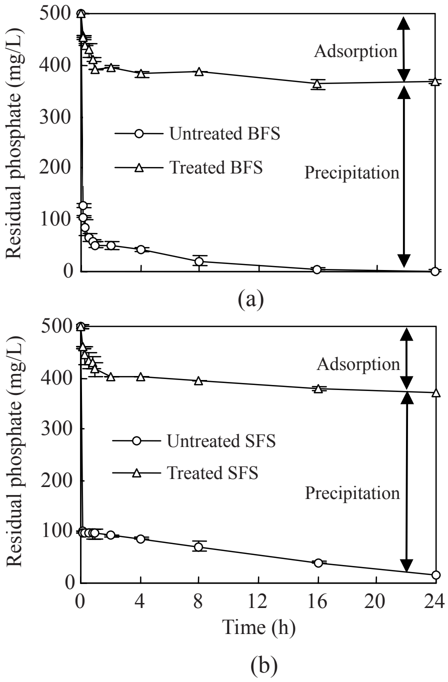


Fig.3 Change of phosphate concentration with untreated and treated BFS (a) and SFS (b). Initial phosphate concentration=500 mg/L. Bars represent one standard deviation of the mean of two replicates

Phosphate adsorption isotherm

The results of phosphate adsorption isotherm experiments are shown in Fig.4. The phosphate adsorption capacity drastically increased with the increases of phosphate concentration (from 0 to 8000 mg/L). The adsorption capacities of BFS and SFS were approximately 46.5 and 33.3 mg/g, respectively, at the initial phosphate concentration of 1000 mg/L. Three isotherms (Freundlich, Langmuir and Temkin models) were used for analyzing the experimental data. According to correlation coefficient, the data obtained in this work well fits in Freundlich adsorption isotherm, expressed by the following equation:

$$q=kC^{1/n}, \tag{1}$$

where C is the phosphate equilibrium concentration (mg/L), q is the amount adsorbed at equilibrium (mg/g), and k and n are Freundlich constants related to the capacity and energy of adsorption. In Fig.5, the related constants were k=6.372 and $1/n$=1.739 for BFS, and k=1.705 and $1/n$=1.718 for SFS. The parameter k, representing the amount of phosphate adsorption when the solution concentration is 1 mol/L, is taken as a relative indicator of adsorption capacity, while $1/n$ is an indicative of the intensity of the reaction. As indicated by Freundlich k values, BFS has higher phosphate adsorption capacity than SFS, in accordance with the fact that BFS contains higher level of Ca^{2+} concentration and pH value than SFS.

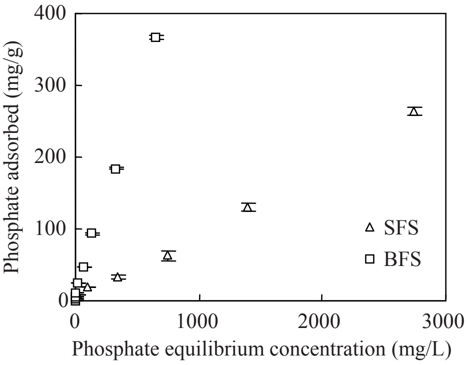


Fig.4 Phosphate adsorption isotherms of BFS and SFS. Bars represent one standard deviation of the mean of two replicates

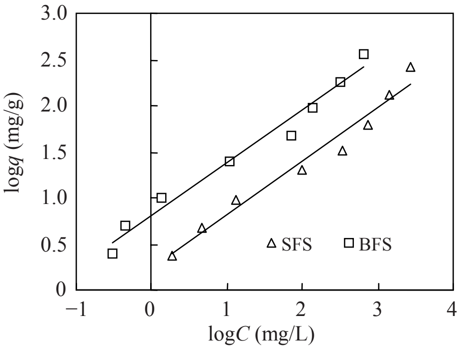


Fig.5 Freundlich isotherm plots for phosphate adsorption on BFS and SFS
BFS: $q=6.372C^{1.739}$, $R^2=0.976$; SFS: $q=1.705C^{1.718}$, $R^2=0.971$

Descriptions of phosphate adsorption models and capacities of slag vary among researchers. Khelifi *et al.*(2002), Oguz (2004), and Kostura *et al.*(2005) reported that phosphate adsorption of BFS followed Freundlich isotherm, which is consistent with our findings. However, others reported that phosphate adsorption followed Langmuir isotherm (Sakadevan and Bavor, 1998). Recently, Oguz (2005) indicated that all three models (Freundlich, Langmuir and Dubinin-Radushkevich) provide an adequate description of phosphate adsorption of slag. A few studies reported that the sorption capacity of BFS varied from 0.65 to 44.2 mg/g (Lee *et al.*, 1997; Kostura *et al.*, 2005). Apparently, the large discrepancies between the results of phosphate adsorption of BFS are related to the fact that BFS does not have well-defined chemical, mineralogical and phase compositions. Apart from the nature of the BFS, factors affecting the phosphate adsorption capacity of BFS in batch experiments also include the particle size, the period and temperature of contact between material and solution, the solution:material ratio, the type and concentration of the supporting electrolyte, the moisture content of the material prior to shaking, and the range of the initial phosphate concentrations used. The phosphate adsorption capacities of electric arc furnace (EAF) steel slag derived from batch experiments can vary by several orders of magnitude depending on the experimental condition applied (Drizo *et al.*, 2002). In our batch experiment, the use of small particle size of slag (less than 0.1 mm in diameter) could overestimate the phosphate adsorption capacity of slag. In designing the on-site wastewater treatment systems such as CSW and soil filtration systems, the phosphate adsorption capacity of a type of material is a key parameter for comparing and selecting candidate materials to be phosphate removal media. This is also the reason that Drizo *et al.*(2002; 2006) recommended that phosphate batch experiments should be coupled with a long-term investigation of material performance in columns for the estimation of phosphate removal efficiencies and retention capacities.

Phosphate desorption

Phosphate desorption was studied using two initial phosphate concentrations (200 and 500 mg/L) at the pH of 2.93 and 6.93, respectively. The P desorbability was defined as the ratio of the desorbed P to the total adsorbed P by the adsorbent. Fig.6 shows that the desorbability of phosphate depends on the pH of the extraction solutions. After the initial phosphate concentration of 500 mg/L was reacted, the desorbabilities of phosphate in the extraction solutions (pH 2.93 and 6.93) were about 39% and 9%, respectively. High acidity (at pH 2.93) likely induces the dissolution of calcium phosphate and weakly-bound phosphate and leads to the increase of phosphate desorption. Low desorbability (at pH 6.93) indicates that phosphate is not completely precipitated as calcium phosphate, and that a fraction of phosphate is weakly adsorbed or complexed on the surface of the slag. These results indicate that the P adsorption on the slag is not completely reversible and that the bond between the slag particles and adsorbed phosphate is strong. It is relatively difficult to desorb phosphate from the slag. These results imply that the slag can be a potential adsorbent of phosphate for wastewater treatment.

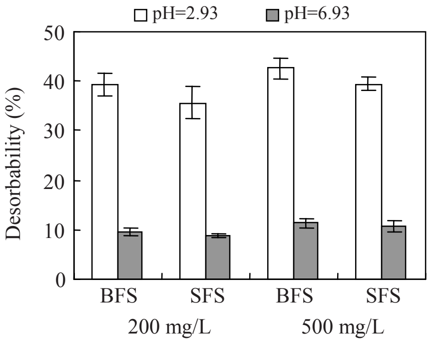


Fig.6 Effect of pH on phosphate desorption after 200 and 500 mg/L phosphate reaction with BFS and SFS. Bars represent one standard deviation of the mean of two replicates

Changes of pH and Ca^{2+} concentrations by the addition of phosphate

As Fig.7 shows, the Ca^{2+} concentration of the slag decreased with the addition of initial phosphate, suggesting a Ca-P precipitation. The pH also shows a decrease with the addition of P. This is true for all materials, but, in particular, the effect in BFS is strong.

DISCUSSION

The mechanism of phosphate removal by BFS and SFS is based on the adsorption (ion exchange and adsorption) and precipitation of Ca phosphates on the surface of the slag. The pH of the solution plays a critical role in the rate and mechanisms of phosphate removal. The chemical precipitation is dominant when pH is above 8, and the adsorption becomes dominant when pH is below 6. At the pH of 3~8.5, phosphate removal probably is due to ion exchange between phosphate hydrolysis products ($H_2PO_4^-$, HPO_4^{2-}) and the precipitation of the metallic salts of phosphate (Al^{3+}, Ca^{2+}, Fe^{3+}). Kostura *et al.*(2005) concluded that the adsorption of the dissolved phosphate is controlled by the presence of calcium on the slag surface.

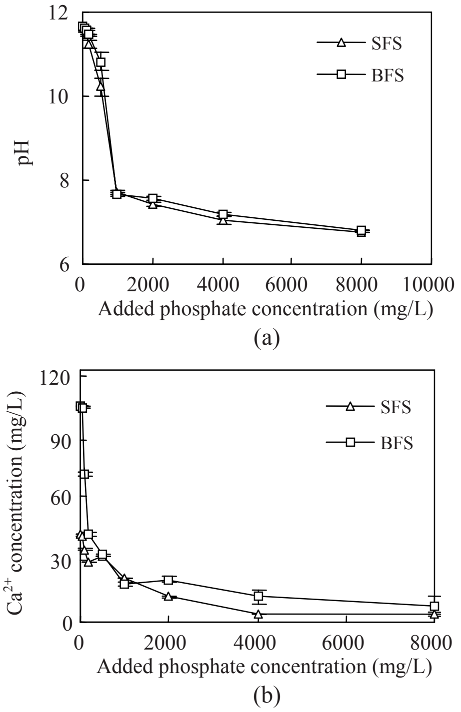
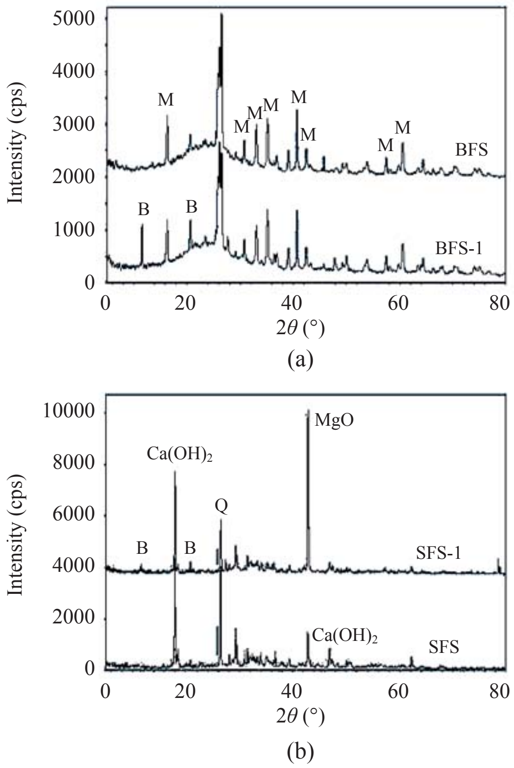


Fig.7 Change of pH value (a) and Ca^{2+} concentration (b) as a function of the amount of phosphate added. Bars represent one standard deviation of the mean of two replicates

Depending on the pH and concentrations of Ca and P ($H_2PO_4^-$ or HPO_4^{2-}) in the solution, different species of calcium phosphate form (amorphous calcium phosphates (ACPs), octacalcium phosphate (OCP), dicalcium phosphate (DCP), dicalcium phosphate dihydrate (DCPD), tricalcium phosphate (TCP) and hydroxyapatite (HAP)) can be precipitated (Lindsay, 1979; Kim *et al.*, 2006). The pH values of BSF suspension (12.2) and SFS suspension (11.8) were within the right range to support the formation of hydroxyapatite (HAP). The Ca-P precipitation depends on the degree of supersaturation and the formation of stable nuclei after which crystal growth takes place. The state of supersaturation develops due either to the increased concentrations of Ca^{2+} and phosphate or to the elevated pH of the solution. Generally, a high total Ca^{2+} concentration and a high pH are required for the precipitation of Ca^{2+}. Fig.8 shows XRD patterns of BFS and SFS before and after phosphate adsorption. The XRD patterns show a considerable change in the peaks of chemical composition intensities in BFS and SFS after phosphate adsorption, which suggests the presence of phosphate salts in the structures of the adsorbed slag. Comparison of the XRD peaks between the untreated and treated slag shows the presence of two sharp and intense peaks of brushite ($CaHPO_4 \cdot 2H_2O$). This result confirms that the precipitation of phosphate by Ca into a brushite is the prevailing mechanism of phosphate removal. The crystals of brushite ($CaHPO_4 \cdot 2H_2O$) are formed on the surfaces of seed particles in the following reaction:

$$Ca^{2+} + H^{+} + PO_4^{3-} + 2H_2O \rightarrow CaHPO_4 \cdot 2H_2O\downarrow.$$



(a)

(b)

Fig.8 X-ray diffraction diagrams of BFS (a) and SFS (b) before (BFS and SFS) and after (BFS-1 and SFS-1) adsorption (initial phosphate concentration of 500 mg/L). B: Brushite ($CaHPO_4 \cdot 2H_2O$), M: mullite [$Al(Al_{1.272}Si_{0.728}O_{4.864})$], Q: quartz ($SiO_2$)

In addition to brushite, the presence of calcium phosphate compounds such as hydroxyapatite (HAP), tricalcium phosphate [$Ca_3(PO_4)_2$] and calcium hydrogen phosphate ($CaHPO_4$) may be also formed.

Chemisorption involved in phosphate removal by slag is a specific adsorption on metal hydroxides. The extent of phosphate removal by these adsorbents appears to be related to the percentage of CaO and/or Ca^{2+} released to the solution via hydration and dissolution. Both Fe and Ca are known to play an important role in P retention by soils and sediments (Lee *et al*., 1997; Sakadevan and Bavor, 1998). As slag is rich in Fe and Ca oxides, phosphate ions can react with iron oxides via ligand exchange to form inner-sphere complexes. These results indicate that precipitation is the major mechanism of P removal, which accounts for more than 60% of the capacity of P removal of slag. In the pH range of 3~8.5, the removal of phosphate probably is through an ion exchange mechanism between the phosphate hydrolysis products and the precipitation of the metallic salts of phosphate (Al^{3+}, Ca^{2+}, Fe^{3+}).

CONCLUSION

We demonstrated that the BFS and SFS are effective adsorbents for the removal of phosphate. The sorption isotherms can be well described by the Freundlich equation. At pH 6.93, the phosphate desorbability was only approximately 10% and 11% for BFS and SFS, respectively. The low desorbability is attributed to a strong bonding between the adsorbed PO_4^{3-} and metal oxides in the slag, indicating that the slag firmly holds the adsorbed PO_4^{3-}. We conclude that the removal of phosphate by BFS and SFS is related to the formation of phosphate calcium precipitation and the adsorption on hydroxylated oxides. The results show that BFS and SFS removed phosphate nearly 100%, indicating they are promising adsorbents for the phosphate removal in wastewater treatment and pollution control.

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