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Perspective

Investigation on mechanism of phosphate removal on carbonized sludge adsorbent

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ABSTRACT

For the removal of phosphate (PO_4^{3-}) from water, an adsorbent was prepared via carbonization of sewage sludge from a wastewater treatment plant: carbonized sludge adsorbent (CSA). The mechanism of phosphate removal was determined after studying the structure and chemical properties of the CSA and its influence on phosphate removal. The results demonstrate that phosphate adsorption by the CSA can be fitted with the pseudo second-order kinetics and Langmuir isotherm models, indicating that the adsorption is single molecular layer adsorption dominated by chemical reaction. The active sites binding phosphate on the surface are composed of mineral particles containing Si/Ca/Mg/Al/Fe. The mineral containing Ca, calcite, is the main factor responsible for phosphate removal. The phosphate removal mechanism is a complex process including crystallization via the interaction between Ca^{2+} and PO_4^{3-} ; formation of precipitates of Ca^{2+} , Al^{3+} , and PO_4^{3-} ; and adsorption of PO_4^{3-} on some recalcitrant oxides composed of Si/Al/Fe.

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Introduction

Excess phosphorus is a key factor contributing to the eutrophication of water bodies receiving wastewater treatment effluents (Z.H. Wang et al., 2016). On the other hand, phosphorus is a limited resource and gradually declining in other areas (Van Vuuren et al., 2010). Therefore, the recovery of phosphorus from sewage will not only reduce water pollution, but also phosphorus resource shortages. Phosphorus in sewage is generally in the form of phosphate (PO_4^{3-}). The amount of phosphate in municipal sewage is close to 5.5% of the amount of phosphorus fertilizer in China (Zhou et al., 2017). The common treatment methods of sewage, such as chemical and biological methods, generally remove phosphate as a pollutant by forming precipitates, but it is

very difficult to recover phosphate from the generated sludge (Chen et al., 2016). It may be possible to recover phosphate from wastewater using adsorption and crystallization methods (G.R. Cui et al., 2016; Hermassi et al., 2015). At present, the preparation of adsorbents for phosphate removal from water mainly focuses on natural minerals, industrial wastes, and novel materials prepared via synthesis or modification (Chen et al., 2016; X.Q. Cui et al., 2016; Seliem et al., 2016), whereas different types of phosphate adsorbents show remarkable discrepancies regarding efficiency and cost. The key hot spot for adsorption research is to select or prepare adsorbents with low cost and high capacity that are considered environmentally friendly (Bal Krishna et al., 2016), allowing for an economical and simple pathway to achieve phosphorous recovery.

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68 Many studies have shown that adsorbents containing Ca/Mg/
69 Al/Fe oxides exhibit good ability to remove phosphate from
70 sewage (F.H. Li et al., 2016; Nair and Ahammed, 2015). Addition-
71 ally, some researchers prepared adsorbents by introducing the
72 elements Zr and La to form oxides to enhance phosphate
73 adsorption (Z.H. Wang et al., 2016; Zhang et al., 2012). These
74 adsorbents mainly remove the phosphate in water that exists in
75 several other forms, including H_3PO_4 , H_2PO_4^- , or HPO_4^{2-} , under
76 different environmental conditions (Seliem et al., 2016). So far,
77 the generally accepted mechanisms of phosphate adsorption
78 mainly include (Chen et al., 2015; X.Q. Cui et al., 2016):
79 (I) electrostatic attraction between phosphate ions and the
80 positively charged hydroxylated surface of metal oxides derived
81 from protonation, (II) ligand exchange between H_2PO_4^- or HPO_4^{2-}
82 phosphate and the hydroxyl group ligand on the surface of
83 adsorbents, and (III) formation of water-insoluble precipitates
84 with the metal ions introduced. All of those might occur when
85 adsorbents containing Fe/Al/Mg/Zr/La metal elements are ap-
86 plied. For Ca-rich adsorbent materials, however, phosphate
87 removal is dependent on Ca-P crystal or precipitate formation
88 (Nair and Ahammed, 2015).

89 Preparing carbonaceous adsorbents through biomass pyro-
90 lysis or carbonization is a hot topic in pollution control (Ahmed
91 et al., 2016). There have been some researchers preparing
92 biochar capable of capturing phosphate, utilizing marine
93 macroalgae waste (*Undaria pinnatifida* roots) as feedstock (Jung
94 et al., 2016b). Further study found that higher values of Ca/P and
95 Mg/P ratios in biochars improved phosphate adsorption (Jung
96 et al., 2016a). In addition, nanosized MgO particles on the surface
97 are also considered to be the critical sites for phosphate removal
98 (G.R. Cui et al., 2016), because they would raise the isoelectric
99 point, resulting in improving adsorption dominated by electro-
100 static attraction. Moreover, the generation of hydroxyl group
101 ligands for ligand exchange would be increased (Yao et al., 2013).
102 The problem, at present, is that the effects and mechanisms of
103 phosphate removal of adsorbents derived from diverse bio-
104 masses will lead to differentiation in composition and structure
105 (Nguyen et al., 2014).

106 Sewage sludge, the by-product of sewage treatment, is also
107 recognized as a sort of biomass, possessing the potential of being
108 an adsorbent if processed by carbonization or pyrolysis (Jain
109 et al., 2016). There is some research on preparing carbonized
110 sludge adsorbents (CSAs) for pollutant removal from water (Hadi
111 et al., 2015). However, the mechanisms for removal of various
112 pollutants might rely on diverse characteristics of CSAs, and
113 these characteristics will be dominated by the carbonization
114 parameters and conditions. At present, reports on phosphate
115 adsorption using CSAs are rare, and the mechanisms of such
116 were not explicit. Thus, the objective of this study was to
117 investigate the mechanisms of phosphate removal by analyzing
118 the relationship between the chemical composition of the
119 adsorbent and phosphate removal. The composition of the
120 CSA adsorbent was complex since it comprises a carbon matrix
121 and minerals containing several metallic elements. In order to
122 determine the active sites binding phosphate, an acid washing
123 process including one-step and two-step acid washing was
124 introduced to affect the chemical and structural characteristics,
125 and the corresponding influence on phosphate removal was
126 also analyzed. It is expected that this work could provide more
127 support for further improving the applicability of this method.

1. Materials and methods 129

1.1. Experimental materials 130

1.1.1. Carbonized sludge adsorbent 131

132 The adsorbent used in the experiments was prepared from
133 sewage sludge collected from a municipal sewage treatment
134 plant in Beijing, where an A_2O process with a sewage capacity of
135 600,000 tons/day is employed. For pretreatment, the sludge was
136 first dried at 105°C to constant weight in a laboratory oven,
137 then the dry sludge was ground and sieved to a particle
138 size < 0.15 mm. The proximate analysis of dried sewage sludge
139 showed that the contents of volatiles, ash, and fixed carbon
140 were 54.35%, 45.08%, and 0.58%, respectively. Elemental analy-
141 sis was also carried out. To be specific, the weight percentages
142 of C and O were considerably higher than those of other
143 non-metal elements (N, S, Si and P). In addition, the presence of
144 several metallic elements (Ca, Mg, Al, Fe, Na and K) was also
145 revealed, in which Fe and Ca were determined to have the
146 highest contents of up to 7.66% and 4.83%, respectively. In the
147 preparation process, the dry sludge particles were placed in a
148 porcelain crucible with a lid and transferred into a muffle
149 furnace and heated at a heating rate of $23^\circ\text{C}/\text{min}$ from ambient
150 temperature to 690°C , and kept at that temperature for 1 hr.
151 After being cooled back down to ambient temperature, the
152 residual was ground to a particle size < 0.15 mm and labeled as
153 CSA. In the current study, a majority of carbon black material
154 was observed with a thin ash layer on its surface.

1.1.2. Carbonized sludge adsorbents with surface mineral components depleted 155

156 The adsorbent CSA was treated by acid washing to eliminate
157 mineral particles on the surface of the adsorbent. Two
158 methods for acid washing were employed. (I) Adsorbent
159 modification by one-step acid washing (CSA-D-1): The CSA
160 was mixed with 1 mol/L HCl at a ratio of 1 g:10 mL in a beaker,
161 and then placed in a 25°C rotary shaker for 30 min followed by
162 centrifugation and removal of the supernatant. This process
163 was repeated three times. After acid washing, the solid was
164 rinsed with deionized water until the effluent had a neutral
165 pH. Finally, the solid was placed in a 105°C oven for
166 dehydration, resulting in an adsorbent modified by one-step
167 acid washing, designated CSA-D-1. (II) Adsorbent modification
168 by two-step acid washing (CSA-D-2): The CSA-D-1 was mixed
169 with 1 mol/L HCl + HF solution at a ratio of 1 g:10 mL in a
170 beaker. The beaker was placed in a 25°C rotary shaker for
171 30 min followed by centrifugation and removal of the
172 supernatant. This process was repeated three times. After
173 acid washing, the solid was rinsed with deionized water until
174 the effluent was neutral. After dehydration in a 105°C oven,
175 the resultant solid was designated as an adsorbent modified
176 by two-step acid washing and labeled CSA-D-2. 177

178 The stock solution of phosphate, with a concentration
179 of 500 mg/L (calculated as P), was prepared with KH_2PO_4
180 and deionized water. Other solutions containing various
181 amounts of phosphate were obtained by diluting the stock
182 solution. The reagents HCl, HF, and NaOH purchased from
183 Sinopharm Chemical Reagent Co., China were all of analyt-
184 ical grade.

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