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Adsorptive removal of phosphate from wastewater using mesoporous titanium oxide

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Abstract. The adsorption of phosphate onto mesoporous TiO_2 was investigated in order to reduce phosphorus concentrations in wastewater and provide a potential mode of phosphorus recovery. Three equilibrium isotherms were used to optimize and properly describe phosphate adsorption ($R^2 > 0.95$). The maximum capacity of phosphate on the adsorbent was found to be 50.4 mg/g, which indicated that mesoporous TiO_2 could be an alternative to mesoporous ZrO_2 as an adsorbent. A pseudo-second order model was appropriately fitted with experimental data ($R^2 > 0.93$). Furthermore, the suitable pH for phosphate removal by TiO_2 was observed to be in the range of pH 3–7 in accordance with ion dissociation. In contrast, increasing the pH to produce more basic conditions noticeably disturbed the adsorption process. Moreover, the kinetics of the conducted temperature study revealed that phosphate adsorption onto the TiO_2 adsorbent is an exothermic process that could have spontaneously occurred and resulted in a higher randomness of the system. In this study, the maximum adsorption was observed at 30 °C.

Keywords: equilibrium isotherm; kinetics; mesoporous TiO_2 adsorbent; phosphate adsorption

1. Introduction

Phosphates are crucial for all living creatures and are extensively used in industry and agriculture (Ozacar, 2006). However, phosphorus is a resource that is nonrenewable and irreplaceable. Therefore, the recover and reuse of this limited resource is a priority for sustainable development (Cheng *et al.,* 2009). Phosphorus is found in natural waters and wastewaters owing to human activities, such as industrial, domestic, and agricultural activities, that continuously increase the amount of phosphorus in these bodies of water (Henry and Heinke, 1989). A phosphorus concentration increase to more than 0.1 mg/L may cause eutrophication (Henry and Heinke, 1989), in which excessive growth of organisms, such as algae, reduces the soluble oxygen concentration in waters and has a detrimental effect on aquatic life (Genz *et al.*, 2004). Therefore, it is important not only to address the recovery of phosphorus, but also its removal from wastewater (Ebie et al., 2008).

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Phosphorus removal by chemical means has been a considerable concern since the late 1960s (Onar *et al.*, 1996). However, it is difficult to recover phosphate, as opposed to removal, using these processes as some of them also produce excess amounts of sludge, which require subsequent disposal (Urano and Tachikawa, 1991). Adsorption is an alternative technology that appears to be more technically and economically feasible for such recovery operations as well as for ultra-high removal. Metals, such as iron, aluminum, calcium, manganese, lanthanum, and zirconium, have been used to study the adsorption of phosphate onto their oxides and/or hydroxides (Chitrakar et al, 2006).

For adsorption, the use of highly porous material is more effective because of ion exchange that occurs on the surface of the materials. Mesoporous structures have been synthesized using surfactant micelles as a template, thereby resulting in these structures having high surface areas and definite pore size distribution (Davis, 2002; Stein, 2003; Jutidamrongphan et al., 2012). The advantages of adsorption using mesoporous materials over those of other technologies, i.e., coagulation or chemical precipitation, are that additional sludge is not produced, additional reagents to overcome high alkalinity are not required, and the pH of the discharged wastewater is unaffected (Urano and Tachikawa, 1991; Choi et al., 2012). As phosphate adsorbents, zirconium-based mesostructured materials have been broadly studied (Iwamoto *et al.,* 2002; Lee *et al*., 2007). However, the selection of an appropriate adsorbent should be carefully considered; the economic feasibility of these absorbents has not been fully verified because of the high cost of zirconium. Therefore, various low-cost materials have also been investigated in several studies to reduce the production costs of zirconium-based mesostructured materials (Falcaro et al., 2004; Naidich, 2000). Titanium-based mesostructures have received attention in terms of their heterogeneous reactions, which are facilitated by the support of reagents on various solid inorganic surfaces (Esumi *et al.*, 1998). Mesoporous TiO_2 has been found to be effective in arsenic removal (Haron *et al.*, 2006) but has hardly been used for the removal of phosphate.

Hence, in the present study, mesoporous TiO_2 was reviewed and selected as an interesting alternative adsorbent for phosphate. This work focused on the equilibrium isotherm and adsorption kinetics of phosphate onto TiO_2 adsorbent. The influences of pH and temperature on phosphate removal from wastewater were evaluated with the aim of utilizing this technology in practical applications.

2. Materials and methods

The mesoporous TiO_2 adsorbent is a new material and was synthesized by the following procedure. Titanium (IV) isopropoxide (11.76 g) and 2,4-pentanedione (4.14) were used as the titanium precursor. Four grams of the triblock copolymer (as a surfactant) was dissolved in 100 mL of distilled water at 40 °C. After the surfactant had been dissolved sufficiently, 1.5 g of sulfuric acid was added. The dissolved surfactant was mixed dropwise with the prepared titanium precursor. For hydrothermal treatment, the reaction was carried out at 100 °C for 24 h without stirring. After the reaction was finished, the precipitate mixtures were washed three times with ethanol and distilled water remove excess surfactant. Calcination of mesoporous TiO_2 was carried out to remove the surfactant at 550 °C for 6 h.

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