



Phosphorous adsorption on synthesized magnetite in wastewater



Jeongyun Choi^a, Jinwook Chung^a, Wonhee Lee^b, Jong-Oh Kim^{b,*}

^a R&D Center, Samsung Engineering Co. Ltd., 415-10 Woncheon-Dong, Youngtong-Gu, Suwon 443-823, Gyeonggi-Do, Republic of Korea

^b Department of Civil and Environmental Engineering, Hanyang University, 222 Wangsimni-Ro, Seongdong-Gu, Seoul 133-791, Republic of Korea

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ABSTRACT

The phosphorous adsorption on a synthesized magnetic iron oxide (MIO) was conducted using a batch type experiment. A dry-form MIO was used in adsorption experiment to obtain useful information for practical application. The Elovich model reasonably simulated the two-step adsorption process in the kinetic experiments. Freundlich isotherm model was better than the Langmuir model for describing the phosphate isotherm on MIO. The maximum adsorption capacity of MIO was 15.2 mg P/g MIO which was achieved at the pH less than 5. The adsorbed phosphorous was effectively detached from MIO within 30 min using 20 wt% NaOH solution.

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Introduction

Phosphorus is an essential nutrient for the growth of organisms in ecosystems and is the most important resource in many fields, such as the manufacture of fertilizers, detergents, and polishes. However, the increased discharge of phosphate into the water system contributes to eutrophication, leading to environmental problems in reservoirs and coastal areas [1,2]. Various techniques have been successfully applied for phosphate removal, such as chemical precipitation, adsorption, and biological processes. However, biological processes are unreliable because the quality of water has a significant impact on phosphate removal. Chemical precipitation often consumes a large amount of coagulant but requires sludge treatment and material disposal [3]. Adsorption is an alternative and promising phosphate removal method, especially for wastewater with a low phosphate concentration [4,5], due to the production of less sludge, easy operation, and high efficiency [6]. Therefore, the removal of phosphate using various adsorbents, such as aluminum oxides [7,8], iron oxides [9], mesoporous silicates [10], red mud [11], alum sludge [12], apatite [13], and clinoptilolite [14], has received increased attention.

On the other hand, despite its extensive use in various fields, phosphorus is a non-renewable and non-interchangeable finite resource. Therefore, the recovery and removal of phosphate should be considered. The material used for removing phosphorus must satisfy the following preconditions as an adsorbent: (1) high adsorption rate, (2) easy to recycle, and (3) high potential for practical application [15,16]. Magnetic iron oxide (MIO) was selected here as an effective alternative for the removal and recovery of phosphorus in solution because solid MIO can easily be separated from a solution using magnets.

Many researches have been carried out to adsorb phosphorous in solution using magnetic iron oxide [17]. The magnetic particles used in the previous researches were mostly nano-sized particles and were kept and used as suspended solid in the adsorption experiment. However, there was a little study using micro-size or dry-form magnetic solid. The relatively bigger size and dry-form magnetic solids is more appropriate to the practical application than the nano-sized suspended form because of cheap manufacturing price and easy handling.

In this study, we investigated the basic characteristic of dry-based MIO (20–100 μm) on adsorbing phosphorous from solution. Kinetic and isotherm experiments were conducted to evaluate the reaction kinetic behavior and the adsorption capacity of MIO. In addition, the effect of operational factors (pH and temperature) was examined to obtain the optimum condition of phosphorous removal by MIO. Finally, the desorption experiment was conducted to verify the reusability of MIO in practical application.

* Corresponding author. Tel.: +82 2 2220 0325; fax: +82 2 2220 1945.
E-mail address: jk120@hanyang.ac.kr (J.-O. Kim).

Materials and methods

Preparation of MIO

The Fe₃O₄ nanoparticles were synthesized using the co-precipitation method [17], mixing 2 L of 1 M Fe SO₄·7H₂O and 2 L of 2 M FeCl₃·6H₂O. The solution was then purged with nitrogen in order to prevent Fe²⁺ ion oxidation. After 30 min of purging, 1 L of 6 M NaOH was added drop-wise into the solutions noted above, and purging was continued for an additional 10 min. During the process, the solution was gradually changed to black and was washed 15 times with deionized water until the pH value of the solution became neutral. The resulting black solution was dried at 70 °C for 24 h in an electric furnace (SF-03, SciLab Co., Korea) and then cooled at room temperature. The dried magnetic iron oxide was crushed using a mortar and pestle.

Characterization of MIO

The crystal structure of MIO was characterized using X-ray diffractometry (XRD, D8-Advance, Bruker-AXS Co., Germany) after excitement with Cu K α radiation at 45 kV and 100 mA. The Fourier-transform infrared spectra (FTIR) were recorded on a Nicolet-380 Fourier-transform infrared spectrometer using the KBr method. The MIO particles were classified by size using the sieving method. The dried MIO was transferred to the nest of sieves arranged from top to bottom with decreasing size in the following order: 100-, 75-, 53-, 32-, 20- μ m, and pan. The sieves were then shaken on a sieve shaker for approximately three min. Each MIO particle fraction was weighed, and the average of five samples was calculated for use in further analysis.

Adsorption experiment for phosphate using MIO

The phosphate solution was obtained by dissolving potassium dihydrogen phosphate (KH₂PO₄) in deionized water. The adsorption process for the single-factor experiments was conducted as follows: 1 g of MIO was added to a 50 mL conical tube containing 20 mL of phosphate solution (40 mg/L). The sealed tubes were then placed in a rotator shaker (GTR-100, Green Tech Co., Korea) at room temperature for 2 h. After the reaction reached equilibrium (approximately 2 h), the supernatant was filtered through 0.45- μ m syringe filters. The phosphate concentration was analyzed using a HACH DR2800 spectrophotometer (method 8048 with a detection limit of 0.01 mg-P/L) with PhosVer 3 reagent powder pillows.

To determine the maximum adsorption capacities of the MIO, equilibrium batch tests were conducted using the aforementioned procedures with various initial concentrations (165, 325, 490, 650, 815, 980, 1140, 1305, 1,470, and 1510 mg-P/L) at an initial pH fixed at 4.0. In addition, the adsorption kinetic experiment was conducted as follows: 1 g MIO was added to a 50 mL phosphate solution with 50 mg-P/L at pH 4. At regular time intervals (1, 5, 10, 15, 30, 60, and 120 min), liquid samples were withdrawn, and the phosphate concentration was analyzed. The additional experiments were performed at 10 and 40 °C in order to examine the effect of temperature on phosphate removal and at an initial phosphate concentration of 50 mg-P/L and initial pH 4 with a MIO dose of 0.25–2.0 g in 30 mL of solution in order to evaluate the effect of adsorbent dosage. To investigate the effect of pH on adsorption, the change in initial pH was controlled at 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, and 9.0 using 1 M/L NaOH and HCl solutions for 120 min at an initial phosphate concentration of 50 mg-P/L.

Desorption of phosphate

A desorption experiment was carried out by 2 steps. In the first step, MIO was reacted with phosphorous at pH 4 and room temperature. The phosphorous concentration on MIO was 4.9 mg P/g MIO. In the second step, 1 g of MIO was added to a 50 mL conical tube containing 20 mL of NaOH solution (3, 5, 10, and 20 wt%). The MIO and alkaline solution was mixed using a multi-rotator at room temperature. Sampling was performed for 1, 5, 10, 15, 30, and 60 min.

Results and discussion

Physicochemical characteristics of MIO

The properties of the synthesized MIO were evaluated using X-ray diffraction (XRD), scanning electron microscopy (SEM), and particle size distribution. The XRD result in Fig. 1a showed that the *d*-space values obtained for the synthesized magnetite were 2.53, 1.48, and 1.61, which were analogous to the reference power diffraction file (JCPDS No. 86-1354). There was no noticeable peak for any of the other ferric or ferrous oxide-hydroxides except MIO. The *d*-space value of the MIO-adsorbing phosphate was almost the same as that of the synthesized MIO, which indicated that no structural disruption of MIO or

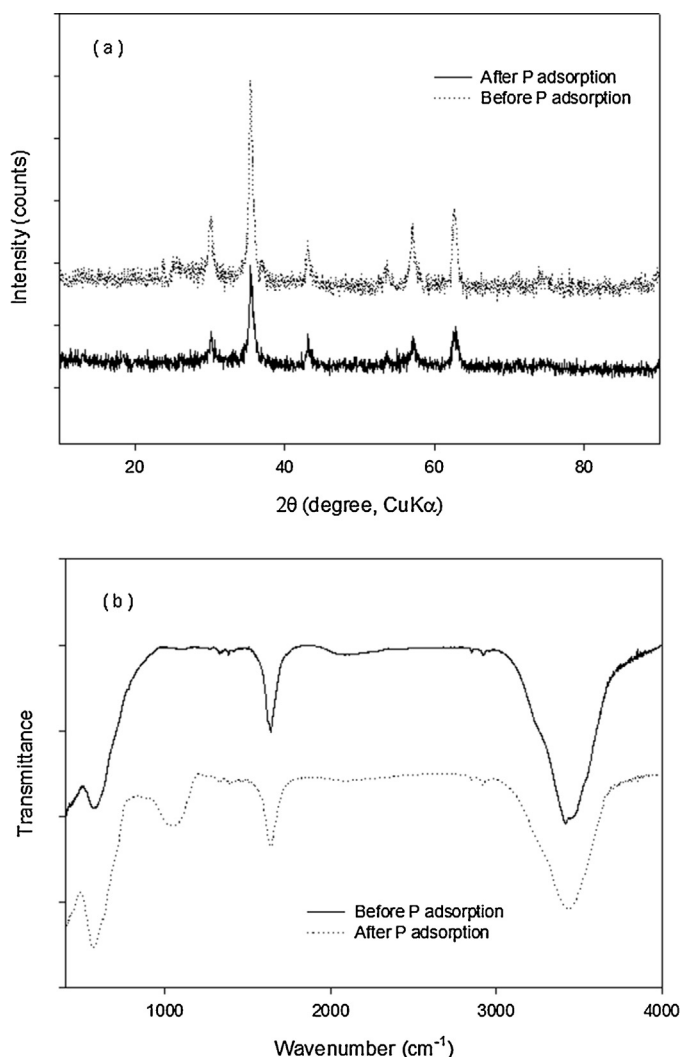


Fig. 1. Magnetic iron oxide used in this study: (a) XRD image and (b) FTIR pattern.

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