



Removal of nitrate and phosphate using chitosan/Al₂O₃/Fe₃O₄ composite nanofibrous adsorbent: Comparison with chitosan/Al₂O₃/Fe₃O₄ beads

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ABSTRACT

In the present study the chitosan/Al₂O₃/Fe₃O₄ composite nanofibrous adsorbent was prepared by electrospinning process and its application for the removal of nitrate and phosphate were compared with chitosan/Al₂O₃/Fe₃O₄ composite bead adsorbent. The influence of Al₂O₃/Fe₃O₄ composite content, pH, contact time, nitrate and phosphate initial concentrations and temperature on the nitrate and phosphate sorption using synthesized bead and nanofibrous adsorbents was investigated in a single system. The reusability of chitosan/Al₂O₃/Fe₃O₄ composite beads and nanofibers after five sorption-desorption cycles were carried out. The Box-Behnken design was used to investigate the interaction effects of adsorbent dosage, nitrate and phosphate initial concentrations on the nitrate and phosphate removal efficiency. The pseudo-second-order kinetic model and known Freundlich and Langmuir isotherm models were used to describe the kinetic and equilibrium data of nitrate and phosphate sorption using chitosan/Al₂O₃/Fe₃O₄ composite beads and nanofibers. The influence of other anions including chloride, fluoride and sulphate on the sorption efficiency of nitrate and phosphate was examined. The obtained results revealed the higher potential of chitosan/Al₂O₃/Fe₃O₄ composite nanofibers for nitrate and phosphate compared with chitosan/Al₂O₃/Fe₃O₄ composite beads.

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1. Introduction

The excess of nitrate and phosphate released in environment, is a very harmful to human and animal health [1–3]. Several methods of physicochemical and biochemical processes including electro coagulation [4], membrane process [5], catalytic reduction [6], ion exchange [7], and adsorption [8] have been applied to reduce the nitrate and phosphate amounts in aqueous systems. The adsorption method due to simplicity and economical is almost preferred [8–10]. The nanofibrous adsorbents have been highly used for the removal of nutrient anions such as nitrate and phosphate [11]. The high efficiency of nanofibrous adsorbents could be attributed to the high surface area and fine pores of nanofibers

[12–14]. Chitosan as a biodegradable, biocompatible and non-toxic polymer due to the presence of amino functional groups, has a high potential for nitrate and phosphate anions sorption [15–21]. However, the main problem of chitosan as an adsorbent is its poor chemical and mechanical stability [22]. Recently, organic/inorganic materials have been incorporated into the polymeric network to enhance the chemical and mechanical properties of polymer [17–19]. In the previous studies, PVA [23], PEO [24], hydroxyapatite [25], SiO₂ [26] and TiO₂ [13] particles were added into the chitosan nanofibers. Furthermore, the fabrication of chitosan composites through the incorporation of effective additives can increase the efficiency of chitosan for the removal of pollutants from aqueous systems. Hu et al. investigated the potential of chitosan-Fe³⁺ complex for nitrate sorption [21]. The potential of chitosan/zirconium beads for the phosphate ions removal have been investigated by Liu and Zhang [20]. In another study, silica dissolution method has been used to increase the efficiency of

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Table 1The experimental design and results of nitrate and phosphate sorption using chitosan/Al₂O₃/Fe₃O₄ composite bead and nanofibers.

Run	Nitrate (ppm)	Phosphate (ppm)	Adsorbent dose (%)	Nitrate r-Nanofiber	Fitted value	Phosphate-r-Nanofiber	Fitted value	Nitrate -r-Bead	Fitted value	Phosphate-r-Bead	Fitted value
1	20	20	0.6	52.30	50.92	41.00	40.65	22.00	23.88	16.80	17.78
2	100	20	0.6	28.50	29.71	17.30	18.05	16.50	16.75	9.50	8.86
3	20	100	0.6	29.60	28.73	22.10	23.13	16.00	15.75	13.40	12.91
4	100	100	0.6	18.30	20.02	11.00	13.13	8.80	6.91	5.40	3.98
5	20	60	0.2	14.85	16.98	12.70	13.12	10.45	8.80	8.05	7.90
6	100	60	0.2	8.25	7.79	5.10	4.42	7.70	7.69	3.25	4.37
7	20	60	1.0	42.90	45.98	38.20	38.42	33.00	33.00	26.10	25.75
8	100	60	1.0	24.75	25.24	15.40	14.52	16.50	18.14	10.50	11.42
9	60	20	0.2	17.25	16.66	11.05	11.86	11.35	11.10	7.20	6.92
10	60	100	0.2	9.20	8.10	6.25	5.68	6.00	7.89	5.75	6.60
11	60	20	1.0	48.80	47.26	33.60	34.61	36.10	34.20	24.00	23.92
12	60	100	1.0	26.00	23.95	18.70	18.33	19.20	19.44	13.45	14.50
13	60	60	0.6	33.00	32.35	25.50	23.74	19.25	19.23	14.90	14.46
14	60	60	0.6	32.50	32.35	25.00	23.74	19.10	19.23	15.00	14.46
15	60	60	0.6	32.25	32.35	24.30	23.74	19.35	19.23	15.05	14.46

Table 2Physical properties of synthesized chitosan/Al₂O₃/Fe₃O₄ composite bead and nanofibers.

Sample	S _{BET} (m ² g ⁻¹)	Pore volume (cm ³ g ⁻¹)	Average diameter (nm)
Nanofiber	251.7	0.421	3.36
Bead	72.3	0.258	2.52

Zr(IV) loaded cross-linked chitosan beads for the removal of nitrate and phosphate [18]. Chitosan–melamine–glutaraldehyde terpolymer was prepared by Sowmya and Meenakshi to remove nitrate and phosphate anions from aqueous systems [17]. In another study, Sowmya and Meenakshi have been evaluated the performance of protonated chitosan, aminated and carboxylated chitosan beads for the removal of nitrate and phosphate [16]. Jiang et al. were synthesized chitosan/Fe₃O₄/ZrO₂ nanocomposites for nitrate and phosphate sorption [15]. The nitrate removal by nano-alumina adsorbent has been employed by Bhatnagar et al. [3]. Katal et al. [27] reported the ability of Fe₃O₄/Polyaniline nanocomposite for nitrate removal. However, there is no study about the application of chitosan/Al₂O₃/Fe₃O₄ composites for the removal of nitrate and phosphate from aqueous solutions.

In the present study, chitosan/Al₂O₃/Fe₃O₄ composite nanofibrous adsorbent was prepared via electrospinning process and its application for the removal of nitrate and phosphate anions was compared with synthesized chitosan/Al₂O₃/Fe₃O₄ beads. The influence of pH, contact time, Al₂O₃/Fe₃O₄ content, initial concentration of nitrate and phosphate, and temperature was investigated on the removal efficiency of nitrate and phosphate using synthesized chitosan/Al₂O₃/Fe₃O₄ composites. Kinetic, isotherm and thermodynamic parameters were also evaluated to determine the mechanism of nitrate and phosphate sorption using synthesized composites. The efficiency of chitosan/Al₂O₃/Fe₃O₄ composites was carried out within six regeneration cycles. A response surface methodology based on Box-Behnken design was used to determine the simultaneous effect of pH, nitrate concentration and phosphate concentration on the removal efficiency of nitrate and phosphate. The effect of other anions such as chloride, and sulphate on the nitrate and phosphate removal was studied.

2. Experimental

2.1. Materials

Chitosan (92% degree of deacetylation (DD) with average molecular weight of 200 kDa), nano-alumina (Al₂O₃ nanopowder) and Glutaraldehyde solution (25 wt.% in H₂O) were purchased

from Sigma–Aldrich (USA). Acetic acid was provided from Merck. FeCl₃·6H₂O and FeCl₂·4H₂O were obtained from Fluka (Germany).

2.2. Synthesis of Al₂O₃/Fe₃O₄ composite

To synthesize Al₂O₃/Fe₃O₄ composite, 0.5 g of Al₂O₃ nanopowder was dissolved in water using an ultrasonic bath for 60 min. Then, 3.5 g FeCl₃·6H₂O and 0.65 g FeCl₂·4H₂O were added into the alumina solution under sonication for further 2 h. Finally, the Al₂O₃/Fe₃O₄ composite solution was dried under vacuum at 60 °C for 12 h.

2.3. Fabrication of chitosan/Al₂O₃/Fe₃O₄ composite beads

To synthesize chitosan/Al₂O₃/Fe₃O₄ composite beads, dried Al₂O₃/Fe₃O₄ composites with different concentrations (1, 2, 3 and 5 wt.% by weight of chitosan) were added to a solution of 4% chitosan in 0.5 M acetic acid and the prepared mixtures were continuously stirred for 2 h to obtain the colloidal solutions of chitosan/Al₂O₃/Fe₃O₄. Then, ethanol was added into the mixtures and the chitosan/Al₂O₃/Fe₃O₄ composites were separated by magnetic decantation, and washed for three times by deionized water and ethanol. Then, the wet chitosan/Al₂O₃/Fe₃O₄ beads were crosslinked with glutaraldehyde solution for 48 h. Finally, the chitosan/Al₂O₃/Fe₃O₄ beads were dried under vacuum at 60 °C for 12 h.

2.4. Fabrication of chitosan/Al₂O₃/Fe₃O₄ composite nanofibers

For preparation of chitosan/Al₂O₃/Fe₃O₄ composite nanofibers, the prepared chitosan/Al₂O₃/Fe₃O₄ solutions with different concentrations of Al₂O₃/Fe₃O₄ (1, 2, 3 and 5 wt.% by weight of chitosan) were poured into the 5 mL plastic syringes. The electrospinning conditions including high voltage of 20 kV, tip-collector distance of 12 cm, feeding rate of 0.5 mL/h and speed collector of 1000 rpm were carried out to form chitosan/Al₂O₃/Fe₃O₄ nanofibers. The set-up of electrospinning process was provided from Nanomeghyas Company (Iran).

2.5. Characterization tests

The functional groups of synthesized chitosan/Al₂O₃/Fe₃O₄ composites were determined using Fourier transform infrared (FTIR, Vector22-Bruker Company, Germany) spectroscopy. The morphology of composite beads and composite nanofibers were determined using scanning electron microscopy (SEM, JEOL JSM-6380) and tunneling electron microscopy (TEM, JEOL

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