



Research Paper

A facile synthesis of magnetic particles sprayed gelatin embedded hydrotalcite composite for effective phosphate sorption

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ABSTRACT

The magnetic composite possess added advantages like high sorption capacity (SC), selectivity and quick separation. The eco-friendly, gelatin (Gel) encapsulated hydrotalcite (HT) GelHT composite was synthesized by dispersing HT in Gel polymeric matrix for phosphate removal. The magnetic biocomposite was synthesized by impregnating iron oxide (Fe_3O_4) on GelHT composite namely Fe_3O_4 @GelHT composite for phosphate remediation. The developed Fe_3O_4 @GelHT composite hold enhanced phosphate SC of 32.73 mg/g whereas phosphate SCs of GelHT composite, Fe_3O_4 @HT composite, HT and Fe_3O_4 were found to be 25.91, 18.35, 15.37 and 5.15 mg/g respectively. The adsorption influencing parameters include contact time, adsorbent dosage, initial phosphate concentration, solution pH, co-existing anions and temperature were optimized in batch mode. The synthesized adsorbents were characterized using FTIR, PXRD, BET, SEM and EDAX with mapping analysis. The feasible phosphate uptake mechanism of magnetic biocomposite was reported. The adsorption equilibrium data were adapted to various suitable isotherm models. Thermodynamic parameters indicate the spontaneous and endothermic of phosphate adsorption. The aptness of the biocomposites at field conditions was examined with collected field water. The reuse of synthesized Fe_3O_4 @GelHT composite was carried out using NaOH solution and it can be reused upto 4 cycles.

1. Introduction

Phosphorus is an essential macronutrient for biomass growth and development. However, the critical water contamination was expanded by surplus phosphate ejection from natural sources, human, industrial extension, detergent and municipal activities which escort malicious environmental troubles such as eutrophication and algae blooms [1,2]. The excessive nutrients growth in river, lake or other water bodies tends to stimulate the depletion of aquatic oxygen level which leads to the deterioration of natural aquatic ecosystem [3]. The tolerance limit of phosphate content in drinking water is < 0.5 mg/L [4]. Consequently, it is necessary to remove excess nutrients present in the aquatic system to protect the ecosystem.

The various remarkable attempts have been made towards phosphate removal which includes membrane processes [5], ion-exchange [6], electrocoagulation [7], biological methods [8], chemical precipitation [9] and adsorption process [10,11]. Among them, adsorption is considered as the best technique for water treatment due to its benefits like inexpensive, easy operation and efficient method [12,13]. The compilation of various inorganic clays play a massive role in the treatment of both synthetic and field water. The versatile inorganic

anion-exchanger namely hydrotalcite (HT) clay has brucite like layered structure with partial substituted Mg^{2+} and Al^{3+} cations whereas the interlayer could be filled by exchangeable anions together with water molecules [14,15]. HT has moderately weak interlayer bonding which improve its remarkable ability towards the capture of various toxic ions. The adsorption property of HT increases mainly due to its large surface area, superior thermal stability and elevated anion exchangeability of about 2–3 meq/g [16]. However, HT holds some drawbacks like pressure drops during column filtration which restricts its field applications.

To outweigh this problem, currently the biopolymer encapsulated composite materials have been developed [17,18]. The biodegradable and eco-friendly gelatin (Gel) biopolymer having both acidic and basic functional groups such as carboxyl and amine groups which could make it as very good adsorbent for toxic ions removal [19,20]. The limitations of gelatin such as poor mechanical strength, low specific gravity and easy gel formation diminish its valuable adsorption nature, hence it has been modified in an usable composite form by loading of HT onto the gelatin (Gel) polymeric matrix to give GelHT composite which would improve the adsorption nature towards phosphate.

Iron oxide (Fe_3O_4) is often called as magnetite which has a cubic

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inverse spinel structure with the interstitial octahedral and tetrahedral sites could be occupied by Fe cation whereas oxygen anion was located in cubic close-packed site. The importance of magnetic supported adsorbent for environmental remediation rapidly increases especially for water treatment includes adsorption of fluoride, cadmium, arsenate, arsenite, chromium and nickel [21–24]. The cross-linking ability and Lewis acidic nature of Fe^{3+} in Fe_3O_4 could be easily attracted by negatively charged species like phosphate. In addition to this, magnetic materials can be easily separated using the external magnet.

The desire of the present investigation is to develop the magnetic iron oxide (Fe_3O_4) coated GelHT (Fe_3O_4 @GelHT) composite for phosphate adsorption. The batch mode was used to determine the adsorption studies of Fe_3O_4 , HT, Fe_3O_4 @HT composite, GelHT composite and Fe_3O_4 @GelHT composite. The synthesized adsorbents were characterized using FTIR, PXRD, BET, SEM and EDAX with mapping analysis. The numerous adsorption influencing parameters such as contact time, initial phosphate concentration, adsorbent dosage, solution pH, co-existing anions and temperature were optimized for maximum phosphate adsorption. The possible phosphate removal mechanism was interpreted for synthesized biocomposites. The adsorption equilibrium data were adapted to various suitable isotherm models. Thermodynamic parameters indicate the spontaneity and endothermic nature of phosphate adsorption. The aptness of the biocomposites at field conditions was examined with field water sample collected from nearby area. The reusability of synthesized Fe_3O_4 @GelHT composite was carried out using alkaline solution and a maximum removal efficiency of 93.81% was observed.

2. Experimental section

2.1. Chemicals

Gelatin, ferric chloride hexahydrate, ferrous chloride tetrahydrate, magnesium nitrate hexahydrate, aluminum nitrate nonahydrate, anhydrous sodium carbonate, sodium hydroxide, ammonium heptamolybdate tetrahydrate and ammonium metavanadate were purchased from Merck, India. The anhydrous potassium dihydrogen orthophosphate of about 1.4329 g was dissolved in 1000 mL of double distilled water to prepare a typical phosphate stock solution.

2.2. Synthesis of GelHT composite

About 2 g of gelatin was dissolved in 50 mL of double distilled water at 40 °C with incessant magnetic stirring upto 2 h. Subsequently, about 700 mL mixed metal aqueous solution of both $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (1 mol/L) and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.5 mol/L) was prepared and dropped into the freshly prepared gelatin solution over 15 min by stirring for 1 h with the same temperature. Meanwhile, 1 L of both NaOH (3.5 mol/L) and Na_2CO_3 (0.94 mol/L) solution were added slowly onto the above polymeric mixture over 20 min at 50 °C and stirred until the mixture becomes homogeneous and leads to the formation of GelHT composite [25,26]. The prepared GelHT composite must be kept in the mother liquid for 24 h for ageing. Then the composite was washed with double distilled water, dried in hot air oven at 65 °C for 12 h and crushed to fine powdered using ball mill (IKA, Germany). Finally, the powdered GelHT composite was applied for phosphate removal.

2.3. Synthesis of Fe_3O_4 @GelHT composite

At first the magnetic iron solution was prepared using the mixture of 10 mL of both $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ (1.85 mmol) and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (3.7 mmol). Then the magnetic iron solution was added into GelHT composite medium over 15 min. During the addition of magnetic contents, the composite medium was maintained at basic pH in the range of 9 to 11 [27]. Once, the formation of Fe_3O_4 @GelHT composite was obtained and it was kept in mother liquid upto 24 h for aging. Then the magnetic

composite was filtered, washed with plenty amount of double distilled water to attain the neutral pH and dried at 68 °C for 18 h in hot air oven. The dried Fe_3O_4 @GelHT composite was crushed to fine powder by the use of ball mill (IKA, Germany) and then utilized for phosphate adsorption studies.

2.4. Batch adsorption studies

In batch series, the influence of various adsorption parameters include contact time, adsorbent dosage, initial phosphate concentration, co-existing anions, solution pH, and temperature were optimized for maximum phosphate adsorption. The effect of contact time was carried out by adding 0.1 g of the synthesized adsorbent into 50 mL of 100 mg/L initial phosphate solution taken into the stopper iodine flask. Then, the reaction mixture was placed onto the thermostat shaker by varying time interval in the range of 10–60 min at room temperature. To optimize the adsorbent dosage for high phosphate adsorption, the varied dosage level of 0.025–0.15 g was added with 50 mL of 100 mg/L phosphate solution at neutral pH. The different initial phosphate concentration range of 20–140 mg/L was utilized in batch study in order to know the optimum concentration range for the maximum phosphate adsorption. The effect of solution pH for phosphate removal was examined by varying the solution pH in the range of 3–11 using 0.1 M of HCl/NaOH solution. The effect of temperature onto the synthesized adsorbents was carried out by taking various initial phosphate concentrations viz., 80, 100, 120 and 140 mg/L with different reaction temperature in the range of 303, 313 and 323 K. The amount of phosphate removal was resolved by mass balance equation which is given as

$$\text{Sorption capacity (SC)} = \frac{C_i - C_e}{m} V \text{ mg/g} \quad (1)$$

where C_i is initial phosphate concentration (mg/L), C_e is the final phosphate concentration at equilibrium time (mg/L), V is volume of the solution (L) and m is the adsorbent dosage (g).

2.5. Analysis

The phosphate concentration was measured in UV–Visible spectrophotometer kit (Model: Spectroquant Pharo 300, Merck). In order to measure solution pH and to prepare various pH medium during pH studies, Thermo Orion Benchtop Multiparameter Kit (Model: VERSA STAR92) was used. The pH drift method was assist to measure the pH at zero point charge (pH_{zpc}) of the adsorbents [28]. All other water quality parameters like Cl^- , SO_4^{2-} , NO_3^- , HCO_3^- , total hardness and total dissolved solids were examined using standard methods [29].

2.6. Characterization of adsorbents

FTIR with JASCO-460 plus model was used to explain the informative chemical functional groups of the synthesized adsorbents. The exterior morphology of GelHT and Fe_3O_4 @GelHT composites were observed from SEM with Vega3 Tescan model. The elemental explorations of the synthesized composites were achieved by EDAX with Bruker Nano GMBH model. PXRD analysis of the adsorbents was given using X'pert PRO model instrument (PAN-alytical). The surface area of the synthesized adsorbents was measured using BET surface analyzer with NOVA 1000 model under nitrogen atmosphere.

2.7. Statistical tools

The computations of all the experimental data were worked out by the use of Microcal Origin (Version 8.0) software. The regression correlation coefficient (r), standard deviation (sd) and chi-square analysis (χ^2) were used for significance of data trends and well selected isotherm model.

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