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Sorption of iron from phosphoric acid solution using polyacrylamide grafted activated carbon

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ABSTRACT

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Keywords: Sorption Iron Phosphoric solution Composite adsorbent Polyacrylamide grafted activated carbon (PAAm-Ac) was prepared from chemically activated carbon of rice husk and rice straw and polyacrylamide. The prepared composite adsorbent was used for removal of iron from wet-phosphoric acid solution. Different parameters that could affect the adsorption reaction were investigated such as pH, contact time, metal ion concentration, adsorbent dose and anion concentration. The adsorption isotherms were studied to evaluate the maximum sorption capacity of adsorbent. The experimental results were found to fit with Langmuir isotherm model with maximum sorption capacity 6.15 mg/g. Different kinetic models were applied upon the experimental data, and it was found to fit well with pseudo second order kinetic model with adsorption capacity of 5.29 mg/g, suggesting a chemisorptions mechanism. The prepared polymeric composite adsorbent was applied for removal of iron from wet process phosphoric acid solution through batch and column experiments.

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Introduction

Phosphoric acid was produced by two processes: the thermal process produces a pure acid with huge energy consumption and the wet-process which involve chemical reaction of phosphate rock with mineral acid. The wet-process is economically applied for the production of phosphoric acid in many countries [1–3]. The mineral acid used in phosphate rock dissolution also dissolves different impurities. Consequently the wet-process phosphoric acid (WPA) must be purified before using in several industries (food additives, fertilizers and detergents) [4]. Different techniques were studied for purification of WPA: precipitation as sulfides [5], adsorption on activated carbon [6], ion exchange and solvent extraction [7–10]. Solvent extraction is the widely used procedure for WPA purification either through separation of phosphate or separation of impurities. Column techniques were also studied for selective extraction of WPA [11]. Purification of WPA from heavy metals was studied through precipitation as sulfides [12]. Also other cationic impurities were removed from WPA by precipitation, as: Fe, Al, Mg, and Ca. Precipitation of metal cations in phosphoric acid was induced by increasing the pH. One of the most important impurities is iron. The content of iron as (Fe₂O₃) in fertilizer grade phosphoric acid $(50\% P_2O_5)$ must be not more than

1.5% Fe₂O₃ [13]. While higher iron content negatively affects the solubility of fertilizer P₂O₅ in water [14]. The Egyptian phosphate ores when leached with H₂SO₄ or recycled H₃PO₄, the produced phosphoric acid (50% P₂O₅) contains 4 or 2.7% Fe₂O₃, respectively. Decreasing iron content in wet phosphoric acid was studied using different precipitating reagents as sodium silicate, silicon dioxide, potassium sulfate, (silicon dioxide + sodium carbonate + potassium sulfate) [0.8885:1.5672:4.0000], calcium sulfate and potassium amyl xanthate $(0.01-6.00 \text{ g}/100 \text{ mL H}_3\text{PO}_4)$ [10]. Also iron content in WPA was studied through precipitation using different chelating compounds as amino tri-methylene phosphonic acid (IR3) [15]. The removal of iron from wet phosphoric acid produced by leaching of phosphate ore with nitric acid was studied using synthesized titanium silicate-poly(acrylic acid-acrylonitrile) (TS-P(AA-AN)) composite adsorbent [16].

In the present work composite adsorbent material was prepared by supporting activated carbon of rice husk and rice straw onto polyacrylamide polymer. The prepared adsorbent, firstly characterized and studied for adsorption iron from phosphoric acid solution with studying the concerned parameters.

Experimental

Materials

All chemicals used in this study were of analytical grade reagent and were used without further purification. Polyacrylamide (PAAm) with average molecular weight over 5,000,000 and

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N,N'-methylenebisacrylamide (NMBA) were purchased from Merck, hydrochloric acid and sodium hydroxide solutions were used to adjust the pH of the aqueous solution.

Sorbent preparation

The starting material, chemically activated carbon (CAC) was firstly prepared following the method: 20 g sample of rice straw and rice husk (1:1) was soaked in 70% H_3PO_4 and heated on a hot plate at 80 °C with stripping, then left in the furnace at 80 °C till complete dryness. After drying, the sample was left in the tube furnace at 500 °C for two hours. The sample was washed with bidistilled water till being neutral, and then dried at 110 °C for 24 h.

Polymeric composite sorbent containing chemically activated carbon was prepared according to the following procedure reported in previous work [17]: 1 g NMBA and 3 g PAAm (in 50.0 mL distilled water) were introduced into a 250 mL flask, equipped with stirrer, thermometer and nitrogen atmosphere. The mixture was vigorously stirred till complete dissolution of NMBA, and then appropriate amount (to achieve the needed percentage) of CAC was dispersed in the mixture. After being purged with nitrogen gas for 30 min (for removal of dissolved oxygen), the mixture was stirred for 2 h, the solution was stirred vigorously till obtaining complete homogeneity. Finally, the mixture was irradiated at ambient temperature in gamma radiation cell (MC-20 Russian) with dose of 30 kGy at fixed dose rate of 3.5 kGy h⁻¹. After irradiation, the composites were washed in acetone for removal of unreacted monomers, cut into small pieces, dried,

ground and stored for further use. The content ratio of CAC in prepared composite was 50% of total weight.

Sorption experiments

The removal of iron from phosphoric acid solution was studied using PAAm-Ac with respect to time, sorbent concentration, iron concentration, temperature and acid concentration. While a constant volume of the acid solution was mixed with the appropriate amount of solid composite adsorbent and left with shaking to appropriate time. After shaking the acid solution was separated from the solid adsorbent and the concentration of iron was measured using atomic absorption spectroscopy (AAS) (atomic absorption spectrometer GBC 932 AA (UK)). The removal percentage (R%) was calculated as:

$$R\% = \frac{(C_o - C_e) \times 100}{C_o}$$
(1)

The distribution coefficients K_d were calculated using the equations:

$$K_d = \frac{(C_o - C_e)V}{mC_e (mL/g)} \tag{2}$$

where C_o is the initial iron concentration in acid solution (mg/L), C_e is the equilibrium iron concentration in acid solution (mg/L), V is the volume of the solution (mL) and m is the weight of adsorbent (g). After optimizing the sorption condition batch sorption

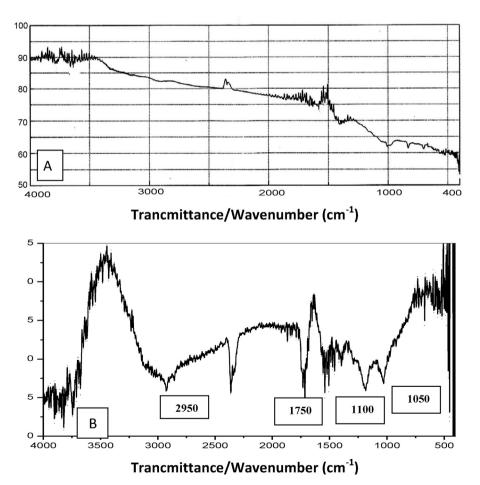


Fig. 1. FTIR spectrum of (a) carbon and (b) PAm-AC composite sorbent.

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