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Sorption of phenol from waters on activated carbon impregnated with iron oxide, aluminum oxide and titanium oxide

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ABSTRACT

The efficiency of Fe₂O₃, Al₂O₃ and TiO₂ nanoparticles-loaded activated carbon (AC) for the adsorption of phenol from waters, was investigated. The raw and doped ACs were characterized by using Scanning Electron Microscopy, Energy Dispersive X-ray Spectroscopy, Thermogravimetric analysis and Brunauer–Emmett–Teller surface analysis. Batch adsorption experiments were performed to evaluate the effects of solution pH, agitation speed, contact time, adsorbent dosage and ionic strength on the phenol removal efficiency. Activated carbon impregnated with Fe₂O₃, Al₂O₃ and TiO₂ showed higher adsorption capacity compared to raw AC. The maximum removal of phenol was achieved by iron oxide, aluminum Oxide and titanium oxide doped AC under the optimum conditions of 200 mg dosage, at pH 7, 150 rpm agitation speed, 2 ppm initial phenol concentration and contact time of 2 h. While for raw AC, the maximum removal was achieved for an adsorbent dosage of 300 mg under the same treatment conditions. The Langmuir isotherm model best fitted the data of the adsorption of phenol using AC, AC–TiO₂, AC–Fe₂O₃ and AC–Al₂O₃, with correlation coefficient of 0.971, 0.96, 0.976 and 0.972. Surface characterization of both the impregnated AC showed an improvement in its surface area of the doped AC. The adsorption capacities, as determined by the Langmuir isotherm model were 1.5106, 3.1530, 3.2875 and 3.5461 mg/g for raw AC, AC–TiO₂, AC–Fe₂O₃ and AC–Al₂O₃.

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1. Introduction

Environmental laws are given general applicability and their enforcement has developed a growing interest in the treatment of the wastewater. Phenol is the priority organic pollutant due to its harmful nature and potential toxicity to human health. The toxic effect of phenol on human being includes diarrhea, impaired vision and excretion of dark urine [1]. Phenol sources are the wastewater from different chemical industries including pulp and paper, petroleum refinery, dye synthesis, coal gasification and pharmaceutical industries. Therefore, effective removal of phenol from the wastewater and reducing its concentrations to the permitted levels before discharging are challenging issues [1–3]. The US Environmental Protection Agency (EPA) regulations suggest phenol concentration below 1 ppm for wastewater [4–5]. Various techniques have been developed for the removal of phenol from

wastewater, such as electrochemical oxidation [6], adsorption by carbon fibers or activated carbon [2,7], wet air oxidation [8,9], chemical coagulation [10], solvent extraction [11], membrane separation [12,13], bioremediation [14,15] and photo catalytic degradation [16,17]. Activated sludge has been widely employed for the removal of phenol from wastewater because of relatively low cost and straightforward process [18]. However, this method is not efficient for the treatment of wastewater with high concentration of phenol due to low biodegradability. In addition, the regeneration process of the adsorbent is not only expensive, but also very complex [3,19].

Other process like bioremediation has also been extensively used for phenol and low molecular weight Polycyclic Aromatic Hydrocarbons (PAHs). The process of bioremediation is relatively simple, but has shown limited success for the degradation of high-molecular weight PAHs [13]. Ultraviolet (UV) radiation is another alternate process for mineralization/transformation of these contaminants, but it has the disadvantage of being expensive, particularly on a large scale [19]. Other physical and chemical remediation techniques applied to phenol

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contaminated water include venting, using solvents and surfactants. The limitation of these techniques is that high solvent concentrations are required for achieving good results [11].

Adsorption is favored by its potential to remove organic and inorganic constituents, even at low concentrations. Adsorption has the advantage of its relative ease of operation both in batch and continuous operation, the absence of sludge formation, potential of regenerative reuse of adsorbent and availability of low cost adsorbent materials [13]. Carbon-based adsorbent materials, which are hydrophobic and non-polar, have good potential for phenol removal in wastewater. Their large surface area, well-developed porosity and tunable surface-containing functional groups are features that enhanced their adsorption efficiency [13–15]. Many researchers [7,20–23] have reported the potential of carbon nanotubes and activated carbon for the removal of phenolic compounds. The large percentage of micropores and high pore volumes and surface area are typical characteristics of activated carbon that are responsible for the enhanced removal of phenols [20]. Although removal of phenol using raw activated carbon is reported by many researchers, however, the potential of modified activated carbon for the removal of phenol compounds is comparatively less reported in the literature [24–27]. Acid treatment of the activated carbon is one of the most effective ways for the enhanced removal of phenols. However, other modification techniques have also been employed to modify the surface of activated carbon [28]. Most of the modification techniques have reported to improve the removal efficiency of AC for the phenolic compounds.

Here, the work is to examine adsorption characteristic for phenol on modified AC by nanoparticles of iron oxide, aluminum oxide and titanium oxide. The physicochemical properties of the modified and unmodified AC were determined using SEM, EDS, TGA and BET surface area analysis. The effects of adsorbent dosage, contact time, pH, initial phenol concentration and shaking speed on the removal of phenol were investigated.

2. Material and methods

2.1. Materials

Activated carbons (ACs) used in this study were purchased from Calgon Ltd., supplied in 10–30 mesh (0.60–1.0 mm) size. The granular form was milled in a hammer-cutter mill to powder (<0.18 mm in particle diameter) for use in this study.

2.2. Impregnation of activated carbon

Aluminum nitrate was impregnated onto 5 g of the AC in ethanol (98% purity). Firstly, specific amount of AC were dispersed in ethanol, followed by sonication (110 V at 40% amplitude). Aluminum nitrate was also dispersed separately in ethanol and sonicated. Finally, both the solutions (AC and aluminum nitrate) were mixed and sonicated to ensure dispersion of the particles. Ethanol is then evaporated from the mixture and calcinated at 350 °C for 3 h to produce AC–Al₂O₃. Titanium oxide (TiO₂) and iron oxide (Fe₂O₃) from titanium isopropoxide and iron nitrate were imprinted on AC in a similar way to produce AC–TiO₂ and AC–Fe₂O₃ respectively.

2.3. Preparation of stock solution

The stock solution of phenol with initial concentration of 2 ppm was prepared by serial dilution of 1000 ppm solution. First 1000 mg of phenol was dissolved in 1 L deionized water. Solvents (1.0 M Nitric Acid and 1.0 M Sodium) were used to adjust the pH of the stock solution. The solution pH was maintained constant during the experiments, by means of buffer solutions.

2.4. Batch adsorption studies

Batch adsorption experiments were conducted at room temperature to study the effects of pH, contact time, adsorbent dosage and agitation speed on the phenol adsorption efficiency by the raw and AC impregnated aluminum oxide (Al₂O₃) and titanium oxide (TiO₂). Experiments were performed in volumetric flasks and the concentrations of phenol were measured using UV–VIS spectrophotometer. The adsorption capacity (q_e) was calculated using the following equations;

$$\text{Percentage Removal} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

$$\text{Adsorption Capacity, } q_e \text{ (mg/g)} = \frac{C_i - C_e}{M_s} \times V \quad (2)$$

where C_i and C_e are the initial and the equilibrium concentrations of phenol ions in the solution (mg/L), V is the total volume of solution (L), and M_s is the adsorbent dosage (g). The parameters considered for the study were adsorbent dosage, contact time, ionic strength of phenol, shaking speed and pH.

2.5. Adsorption isotherm models

Langmuir and Freundlich are the two commonly used isotherm models to represent the adsorption data. The Langmuir isotherm model is based on the assumption of monolayer adsorption from the homogeneous surface while Freundlich isotherm models assume adsorption from heterogeneous surface, with non-uniform heat distribution on the surface. The Langmuir and Freundlich models can be expressed mathematically by Eqs. (3) and (4) respectively:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{\max} K_L} + \frac{C_e}{Q_{\max}} \quad (3)$$

$$\text{Log } Q_e = \text{Log } K_f + \left(\frac{1}{n}\right) \text{Log}(C_e) \quad (4)$$

where: Q_e (mg/g) = amount adsorbed (mg/g). C_e (mg/L) = equilibrium concentration of phenol in the liquid phase. Q_m (mg/g) = maximum adsorption capacity. K_L = Langmuir constant.

K_f and n are the Freundlich constants.

3. Results and discussions

3.1. Characterization

3.1.1. SEM and EDS

The SEM images of ACs, AC–Al₂O₃ and AC–TiO₂ are depicted in Fig. 1a & b. The white spots in the SEM images of AC–Al₂O₃ and AC–TiO₂ show Al₂O₃ and TiO₂ particles, respectively, as confirmed by EDS analysis. The outcome of the analysis is shown as EDS spectrum in Fig. 2. Table 1 indicates the weight percentages of the elements. The peaks of iron, aluminum and titanium can be clearly observed in the spectrum [29].

3.1.2. Thermogravimetric analysis (TGA)

The thermal analysis results of thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTGA) curves obtained from the raw and modified AC with metals at a heating rate (10 °C/min) are shown below in Fig. 3 (a, b, c & d). The TGA thermograms were carried out in the air and it was noted that there was some residual remains of the samples, when it was heated up to 800 °C. The TGA curve for AC–TiO₂ (Fig. 3c) shows distinct behavior from the other three adsorbents. It can be seen that two degradation temperatures exist at 100 °C and 500 °C, respectively. The amount of the sample

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