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Adsorption of phenol on aluminum oxide impregnated fly ash

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ABSTRACT

In this work, the potential of fly ash impregnated with aluminum oxide (FA-Al_2O_3) for adsorption of phenol from aqueous solution was studied. Batch adsorption experiments were carried out to evaluate the effects of the experimental parameters pH, agitation speed, contact time, adsorbent dosage, and initial concentration on the phenol removal efficiency. The adsorption of phenol by FA-Al_2O_3 was found to be pH dependent with the best removal achieved at pH 7. The optimum set of parameters for the removal were, 200 rpm agitation speed, 200 mg adsorbent dosage, 2 h contact time, and 2 ppm initial phenol concentration. Both the Langmuir and Freundlich isotherm models represented the adsorption experimental data. However, the Langmuir isotherm model best fitted the data on the adsorption of phenol using FA and FA-Al_2O_3, with correlation coefficient of 97.7 and 97.9, respectively. The improvement in the adsorption efficiency of FA-Al_2O_3 over FA could be attributed to the increase in the surface area, which was found to be 11.889 m^2/g and 7.1 m^2/g for FA-Al_2O_3 and FA, respectively.

Keywords: Fly ash; Aluminum oxide impregnation; Phenol; Adsorption

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1. Introduction

Phenolic compounds are generated in petrochemical and petroleum, coal conversion, and phenol-producing industries, and have been identified as common contaminants in wastewaters. Phenols have found application in the production of wide varieties of phenolic resins, used in construction of automobiles and appliances, adhesives and epoxy resins, as well as other various applications [1]. The harmful nature of phenols to organisms, even at low concentrations have attracted their classification as a priority pollutants and given their potential toxicity to human health, several forms of phenols have been classified as hazardous pollutants. Hence, it is required by US Environmental Protection Agency regulations to lower phenol content of wastewater below 1 ppm [2].

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Several different techniques, such as electrochemical oxidation, chemical coagulation, solvent extraction, membrane separation, bioremediation, photo catalytic degradation, and adsorption have been studied for the removal of phenolic compounds from wastewater, with varying levels of success achieved and each technique also characterized by its inherent limitations [3–8]. The application of fly ash for phenol removal from wastewater is favored by its high adsorption capacity. The low-cost fly ash adsorbent has the advantages of ease of operation both at batch and continuous operation, the absence of sludge formation, potential of reuse and overall economic nature [9]. 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 enhancing their adsorption efficiency [10–12].

In this study, fly ash (FA) which has shown material success in the adsorption of heavy metals, other inorganic contaminants, and some organics was studied for phenol removal from water under batch condition. Prior to its application for phenol adsorption, surface modification by metal oxide impregnation, specifically aluminum oxide (Al_2O_3) was performed.

2. Material and methods

2.1. Preparation of adsorbent

FA used in this study is a particulate waste generated during the combustion of heavy fuel oils at a local power generating plant in Saudi Arabia. The FA is collected locally with the aid of electrostatic precipitator. It usually contains high percentage of unburned carbon and other metal impurities. The FA had size varying from 500 to 1,500 microns. Al_2O_3 from aluminum nitrate was impregnated onto 5 grams of the FA in ethanol (98% purity), followed by sonication (110 volts at 40% amplitude) and calcination at 350°C for 3 h.

2.2. Preparation of stock solution

Stock solution of phenol with initial concentration of 2 ppm was prepared by serial dilution of 1,000 ppm solution made from dissolved 1,000 mg of phenol in 1 L deionized water. The pH of the stock solution was adjusted using 1.0 M nitric acid (HNO_3) and 1.0 M sodium hydroxide (NaOH) and buffer solutions were added to maintain constant pH during the experiments.

2.3. Batch adsorption experiment

Mode adsorption experiments were conducted at room temperature to study the effects of pH, adsorbent dosage, contact time, and agitation speed on the phenol adsorption efficiency of the FA impregnated with Al_2O_3. Each experiment was conducted in a volumetric flask and the initial and final concentrations of phenol were obtained using UV–vis spectrophotometer. The adsorption capacity (q) was calculated using the following equations;

$$\%\text{removal} = \frac{C_i - C_e}{C_i} \times 100 \tag{1}$$

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

where C_i is the initial concentration of adsorbate ion in the solution (mg/L), C_e is the concentration of adsorbate ion in solution (mg/L), V is the total volume of solution (L), and M_S is the carbon dosage (g).

2.4. Adsorption isotherm models

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate between the liquid and adsorbent based on assumptions mainly related to homogeneity/heterogeneity of adsorbents, type of coverage, and possibility of interaction. The absence of interaction between adsorbate molecules is the main assumption of the Langmuir model, implying that adsorption is localized in a monolayer. The Freundlich isotherm model assumes that different sites with several adsorption energies are involved in the adsorption of solutes from a liquid onto the adsorbent's surface.

The Langmuir and Freundlich models are represented by the following linearization equations, respectively:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{\max} K_L} + \frac{C_e}{Q_{\max}} \tag{3}$$

$$\log Q_e = \log K_f + \left(\frac{1}{n}\right) \log(C_e) \tag{4}$$

where Q_e (mg/g) = Equilibrium concentration of phenol in the adsorbed phase. C_e (mg/L) = Equilibrium concentration of phenol in the liquid phase. Q_m (mg/g) = Maximum adsorption capacity corresponding to complete monolayer converge. K_L is the Langmuir constant related to energy of adsorption, it can be calculated from the slope of the linear plot of (C_e/Q_e) vs C_e. K_f and n are the empirical constants. These constants can be calculated from the slope and intercept of the linear plot of log C_e vs. log Q_e.

$$Q_e = Q_m K C_e / (1 + K C_e) \tag{5}$$

where Q_e is the adsorption density at the equilibrium solute concentration C_e (mg of adsorbate per g of adsorbent), C_e is the equilibrium adsorbate concentration in solution (mg/L), Q_m is the the maximum adsorption capacity corresponding to complete monolayer coverage (mg of solute adsorbed per g of adsorbent), and K is the the Langmuir constant related to energy of adsorption. We can describe adsorption with the Langmuir isotherm if there is a good linear fit. If the adsorption mechanism is not monolayer that can be described by the Langmuir isotherm, then the Freundlich isotherm will be attempted.

3. Results and discussion

3.1. Characterization of adsorbents

3.1.1. Scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM–EDS)

The Al_2O_3 impregnated fly ash (FA-Al_2O_3) were characterized using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Fig. 1 shows the SEM of FA in its raw state and following Al_2O_3 impregnation. The diameters of the produced FA vary from 20 to 40 nm, with an average diameter of 24 nm. The elemental composition of the carbon-based adsorbent following Al_2O_3 impregnation was obtained using the EDX, and it showed the metal oxide to constitute 5% of the total mass of the FA-Al (the FA-Al abbreviation will be used in this manuscript to represent FA-Al_2O_3 with 5% Al_2O_3.

3.1.2. Brunauer Emmett Teller (BET) surface area analysis

Aimed at determining the improvement in surface morphology following Al_2O_3 impregnation of the FA, BET surface area analysis was conducted. The analysis was performed using Micrometrics ASAP 2020 and results are interpreted based on the adsorption–desorption of N_2 at 77 K for the Al_2O_3 impregnated FA. The BET surface area values obtained for the raw FA and FA-Al were 7.1 and 11.889 m^2/g, respectively.

3.2. Adsorption of phenol from aqueous solution

3.2.1. Effect of pH

As a change in pH affects the surface charge of the adsorbent materials, the pH of aqueous solution is considered as an important factor in the adsorption process; therefore, influencing the removal of phenol as in this study. The extent of phenol removal in this study was evaluated at pH from 2 to 9, using FA-Al dosage of 50 mg, agitation speed of 100 rpm, and for a

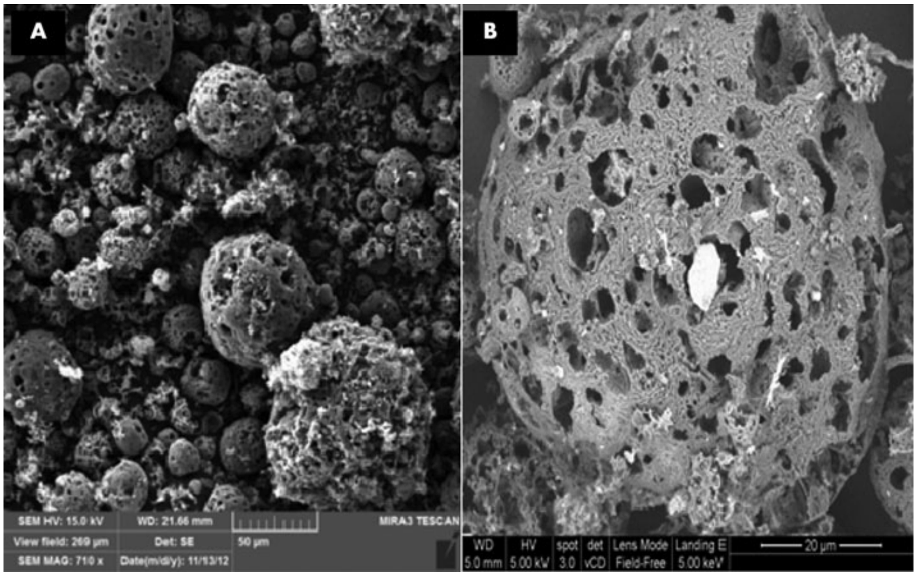

Fig. 1. SEM micrographs of raw FA (A) and FA-Al (B).

contact time of 2 h. Fig. 2 shows increase in phenol removal efficiency with rise in pH of the solution, with the peak removal attained at pH 7. This observation was attributed to the types and ionic state of the functional groups on the surface of the adsorbent, as well as ionic chemistry of the solution [13]. Given that phenol is a weak acid (pKa = 10) is expected to be less adsorbed at higher pH due to repulsive force prevailing at higher pH value. The peak adsorption of phenol at pH 7 suggests that negatively charged phenolate ions bind through electrostatic attraction to positively charged functional groups on the surface of the FA, due to their exposure at this pH level. However, beyond pH 7, it is assumed that the carbon possesses more functional groups carrying a net negative charge which tends to repulse the anions. Banat et al. [13] and Halouli et al. [14] reported similar pH behaviors in phenol adsorption onto activated charcoal and bentonite clay, respectively.

3.2.2. Effect of agitation speed

The role of agitation speed on the adsorption of phenol from the aqueous solution was also studied, aimed at understanding its effect on interaction between the active sites on the FA-Al and phenol. The study was conducted varying the speed from 50 to 250 rpm and from Fig. 3, it was observed that phenol removal efficiency increased with the increase in agitation speed. The peak removal was achieved at 200 rpm and no significant change was achieved beyond this point. This observation was attributed to improved contact between the ions in the solution and the active adsorption sites on the adsorbent with increase in agitation speed. Similar studies have reported more effective adsorption between carbon-based adsorbents and phenol at 200 rpm [15,16].

3.2.3. Effect of contact time

The effect of contact time in the removal of phenol from the aqueous solution was studied to determine the optimum adsorption time. From Fig. 4, increase in phenol removal efficiency was observed with increase in time until 2 h at which the optimum adsorption was attained. This observation was attributed to the adsorption equilibrium phenomenon whereby the rate of adsorption was higher than the rate of desorption up to 2 h of contact, which is the equilibrium adsorption point at which the rate of adsorption and desorption were same and no further removal of phenol from the solution was achieved. In addition, a clear desorption of phenol from all adsorbents was observed after two hours due to the saturation of the active sites on surfaces adsorbents. The enhanced adsorption of metal with an increase in agitation time may also be due to the decrease in boundary layer resistance in the kinetics energy of the hydrate layer [17].

3.2.4. Effect of carbon dosage

The effectiveness of adsorption technique for treatment is usually a subject of the number and availability of active adsorption sites on the surface of the adsorbent. Generally, increasing the dosage of the

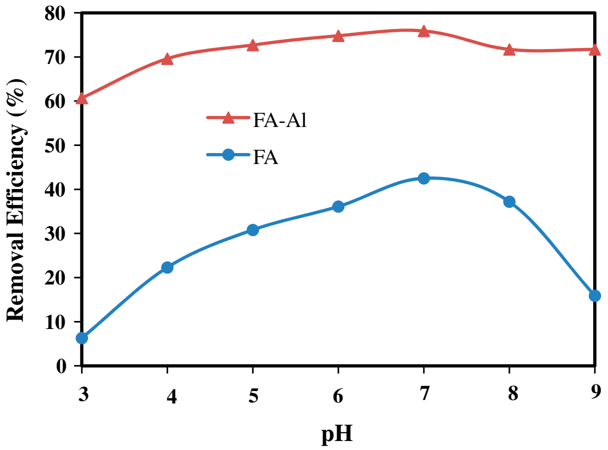

Fig. 2. Effect of pH on phenol adsorption using FA-Al at room temperature, 100 rpm, 2 h contact time, 50 mg adsorbent dosage, and 2 ppm initial concentration.

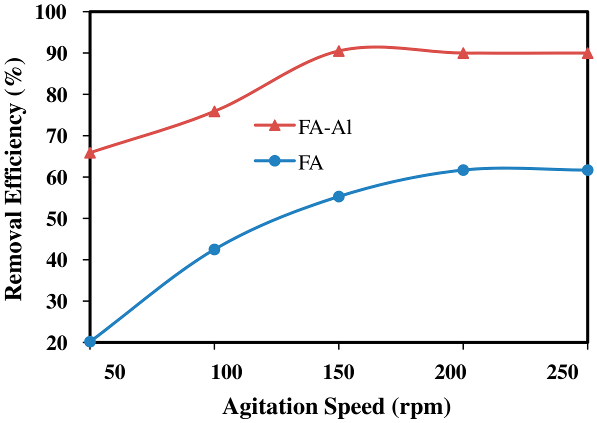

Fig. 3. Effect of agitation speed on phenol adsorption using FA-Al at room temperature, 2 h contact time, pH 7, 50 mg adsorbent dosage, and 2 ppm initial concentration.

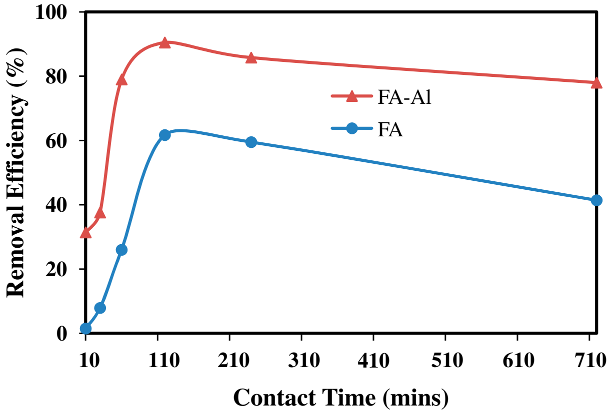

Fig. 4. Effect of contact time on phenol adsorption using FA-Al at room temperature, pH 7, 50 mg adsorbent dosage, and 2 ppm initial concentration.

adsorbent brings about an increase in the number of active adsorption sites available for interaction. In this study, the FA-Al dosage was varied from 10 to 500 mg and its effect on the phenol removal efficiency was evaluated. From Fig. 5, it was observed that there was an increase in the phenol removal efficiency with increase in dosage to 200 mg, beyond which there was no notable increase in removal with further increase in dosage. This observation was attributed to increase in the number of active adsorption sites until the optimum dosage was attained, beyond no further increase in removal efficiency was achieved with increase in dosage due to conglomeration or overlapping of the active adsorption sites at dosages beyond the optimum [18].

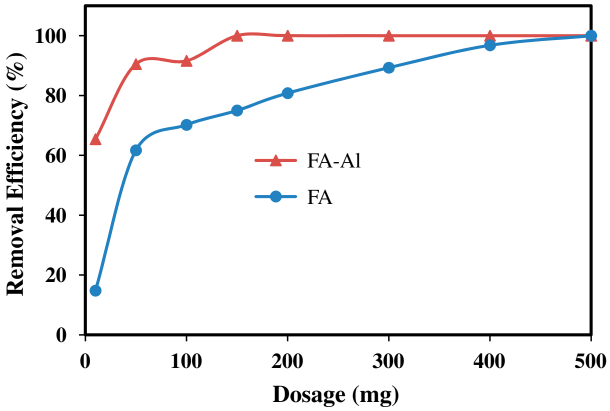

Fig. 5. Effect of adsorbent dosage on phenol adsorption using FA-Al at room temperature, pH 7, 2 h contact time, and 2 ppm initial concentration.

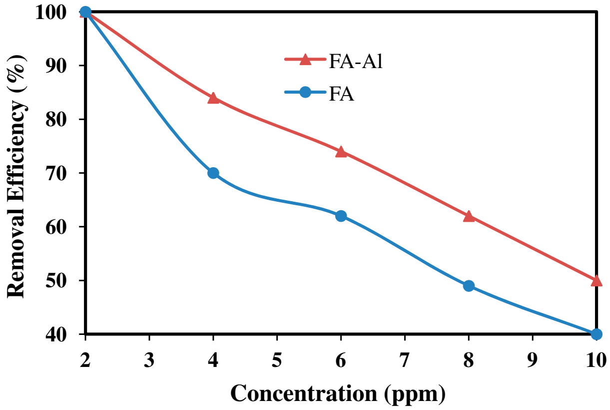

Fig. 6. Effect of initial concentration on phenol adsorption using FA-Al at room temperature, pH 7, 2 h contact time, and 450 mg adsorbent dosage.

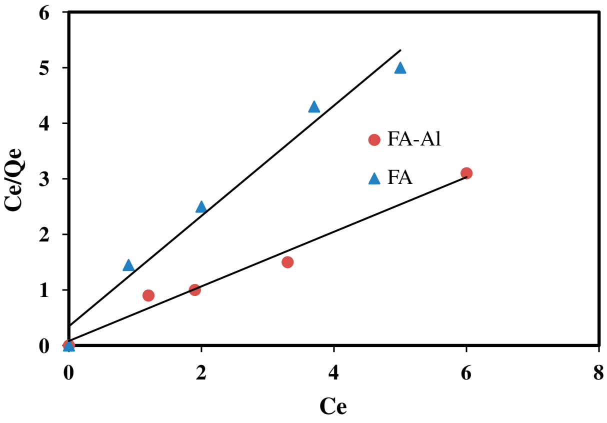

Fig. 7. Langmuir adsorption model for phenol at pH 7.

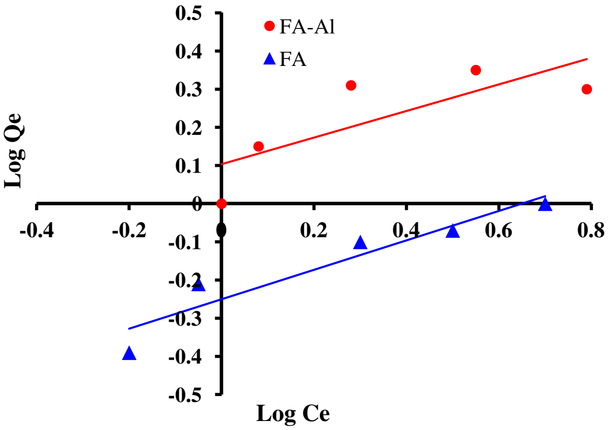

Fig. 8. Freundlich adsorption model for phenol at pH 7.

Table 1
Parameters of Langmuir and Freundlich adsorption isotherm models for phenol removal

	Langmuir			Freundlich		
Adsorbent	Q_m(mg/g)	K_L(L/mg)	R^2	n	K_F	R^2
FA	1.0070	2.8950	0.977	8.264	0.7345	0.925
FA-Al	2.105	7.196	0.979	2.786	1.265	0.625

3.2.5. Effect of initial concentration

The effect of phenol concentration in the aqueous solution on its adsorption by the carbon-based adsorbent was studied. For this purpose, the initial concentration of phenol in the solution was varied from 2 to 10 ppm, and its effect on the removal efficiency was ascertained. From Fig. 6, it was observed that increasing the initial phenol concentration in the solution resulted in decrease in the removal efficiency of the carbon-based adsorbent. This observation was attributed to high amount of phenol ion with limited active adsorbed sites on the adsorbent surfaces, which leading to increase the concentration of phenol ion in the bulk solution and thus decreasing on the removal of phenol [19].

3.2.6. Adsorption isotherm model

An evaluation of the maximum adsorption capacity of the adsorbents was taken from the conditions with optimum adsorption parameters. The equilibrium curves were modeled in Figs. 7 and 8. The Langmuir and Freundlich in Eqs. (3) and (4) were used to fit the data derived from the adsorption of phenol by the carbon-based adsorbents over the optimum set of parameters. Based on the Figs. 7 and 8, the maximum adsorption capacity (Q_m) and adsorption intensities were determined from the slope and the intercept of each adsorbent line, respectively [20–26]. The Langmuir adsorption model as compared to the Freundlich model best fits the adsorption behavior of adsorbent. The correlation coefficients values are given in Table 1. The above analysis also indicates that phenol ions strongly adsorbed to the surfaces of the adsorbent suggesting that these carbon-based adsorbents have great potential for adsorption and can be applied for the removal of phenol ions in water treatment, with the advantage of being of low cost comparing with others [27–42].

4. Conclusions

The potential of Al_2O_3 impregnated FA to adsorb phenol from aqueous solution was successfully demonstrated in this study. Al_2O_3 impregnation brought about a positive change in the surface properties of the FA, notably increase from 7.1 to 11.889 m^2/g in its BET surface area, consequently improved its phenol removal efficiency from aqueous solution. Solution parameters such as pH, agitation speed, adsorbent dosage, contact time, and initial phenol concentration were found to significantly affect the phenol removal efficiency. The optimum removal from the aqueous solution was achieved at pH 7, 200 rpm agitation speed, 200 mg dosage, 2 h contact time, and 2 ppm initial phenol concentration. The data from the phenol adsorption behavior of the carbon-based adsorbents were best fitted by the Langmuir adsorption isotherm model, with correlation coefficients of 97.7 and 97.9 for FA and FA-Al, respectively.

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