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Development of new effective activated carbon supported alkaline adsorbent used for removal phenolic compounds

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Abstract

Phenolic (phenol) compounds are the major contaminates in wastewater, which can have a considerable negative influence on the environment and health of human. Adsorption is an efficient process that is widely applied in order to eliminate phenol in wastewater. In recent, Adsorption process has acquired a lot of attentiveness owing to its relative moderate operating conditions. However, adsorption process needs considerable ameliorations in terms of adsorbent modification, process type, productivity, and conversion rate. This work studies the development of a fast and effective adsorption process in a fixed bed adsorption column (FBAC) in order to reach safe and continuous elimination of phenolic compounds. Several adsorption parameters (reaction temperature, adsorbent bed height, feed flow rate and kind of adsorbent) were studied to achieve the highest removal of phenolic compounds. The adsorption process was conducted in the presence of two type of adsorbents (activated carbon (AC), and KOH/AC), 73% and 94% of phenol elimination were attained, respectively, at 10 cm bed height, 1 ml/s feed flow rate, and 75 °C reaction temperature. The adsorbents activity was investigated after six consecutive adsorption cycles at the best process conditions, and the adsorbents show high stability in terms of phenolic compounds adsorption. After that, the spent adsorbents were regenerated by utilizing various solvents (methanol, ethanol and iso-octane), and the results show that iso- octane achieved highest regeneration efficiency. The adsorption process was implemented in the adsorption column that the performance is possibly to be adjusted at an industrial scale since it can be scaled up predictably.

Keywords: Adsorption process; AC; KOH; FBAC; deactivation study

1. Introduction

The growing apprehension by government and environmentalist on the influence of the effluents of various industries on the public health gave rise to several of studies in the development of advance methods to eliminate pollutants from water and industrial wastewaters. Phenolic compounds found in the effluents of many industrial process such as petrochemicals, oil refining, coking operations, plastics, pharmaceuticals, resin manufacturing, paper, paint, pulp, and wood products are priority contaminants with high toxicity even at low concentrations. The inevitability of the elimination of phenolic compounds from polluted water owing to its harmful impacts on humans, animals, and aquatic systems [1-7]. A several methods are available for the elimination of phenolic compounds from industrial wastewater such as electrochemical, membrane filtration, adsorption and chemical oxidation. Adsorption is the most attractive method for the treatment of industrial wastewater because it is easy to control, does not require high operational costs and capital, and an adding of desorption step to the treatment process allows reuse of adsorbents and recovery of phenol compounds for recycling [8-12].

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Activated carbon (AC) can be defined as a carbonaceous material which having a porous texture and well-developed surface area. As a result, AC has been commonly utilized in separation processes or catalysis and as adsorbent [13-16]. MA et al. [17] studied the elimination of phenolic compounds by utilizing powder activated carbon as adsorbent in batch reactor and it was found that more than 80% of phenolic compounds was removed at 10 min. In some studies, using AC modified (prepared from agricultural wastes such as palm fronds, date stones and rice husk treatment via chemical and physical activation procedure and maximum capacity of removal (84.83%-94.65%)[13]. The study of the adsorption of phenol from an aqueous solution using tamarind seed powder (TSP) with adsorption process AD was investigated [25].

In this study, the adsorption process has been carried out in the fixed bed adsorption column (FBAC) by using loaded AC (10% KOH/AC) in order to improve the removal of phenol in comparison with related previous study MA et al. [17]. The loaded activated carbon has been prepared by loading alkaline (KOH) over AC in order to investigate the role of KOH in the enhancing the adsorption capacity of AC. Several adsorption parameters were studied so as to reach the most effective ADS system which involve feedstock temperature, feedstock flow rate, bed height and type of adsorbent. Finally, the deactivation and regeneration have been studies for the spent adsorbents.

The novelty of this study is performing the removal process of phenolic compounds in the continuous fixed bed adsorption column (FNAC) in the presence of new adsorbent in order to achieve highest removal of phenolic compounds as well as to simulate the industrial field.

2. Materials method and Experimental work

2.1. Chemicals and materials

2.1.1. Feedstock

The model wastewater feedstock has been prepared by mixing demineralized water and phenol (supplied from Alpha Chemika Company with purity of 99%) at concentration of 583 ppm.

2.1.2. Activated carbon (AC)

The activated carbon (AC) (obtained from Applichem GmbH, Germany) has been utilized as adsorbent and the AC specifications are listed in Table 1.

Table 1. Specifications of activated carbon

Properties	values	Unit
Surface area	804	m^2/g
Pore volume	0.491	cm^3/g
Pore size	2.423	nm
Particle size	2-3	mm

2.1.3. Potassium hydroxide

Potassium hydroxide (KOH) obtained from (Sigma Aldrich) has been employed to improve the adsorption capacity with purity of about 85%.

2.1.4. Regeneration solvents

Methanol, ethanol and iso-octane have been utilized as a solvents for the regeneration step of AC. The properties of these solvents are summarized in Figure 1.

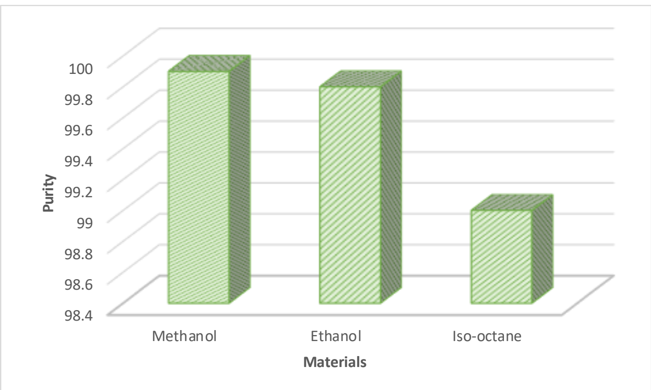


Fig. 1. Properties of solvents.

2.2. Adsorbent preparation

Incipient Wetness Impregnation method (IWI) was applied in order to prepare the adsorbent (10% KOH/AC). Initially, 10% KOH solution was formed by dissolve KOH in deionized water for one hour. Then, activated carbon was added to the prepared solution with mixing for 3 h. Finally, the impregnated solution was dried at temperature of 80 °C for 12 h to obtain (10% KOH/AC).

2.3. Adsorbent characterization

The adsorbents (AC and 10% KOH/AC) were characterized by utilizing the following equipment and tools: Brunauer, Emmett, and Teller (BET), X Ray Diffraction (XRD), Thermal Graphometry Analysis (TGA), Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM)

2.4. Experimental procedure

2.4.1. Fixed bed adsorption column (FBAC) unit

The removal of phenolic compounds via continuous adsorption process was implemented in a FBAC unit. The dimensions of the fixed bed adsorption column are listed in Table 2.

Table 2. Dimensions of the fixed bed adsorption column

Specification	values
Inner diameter (ID)	3 cm
length (L)	34 cm

The inlet of FBAC which is made of Pyrex (Corning, USA) is connected to the dosing pump (IML Company, Spain) which pumps the feedstock through a feed tank. The ranges of feedstock net flow rates are (0 – 1.433 ml/sec of 20 % pumping efficiency) and (0 – 5.15 ml/sec of 20 % pumping efficiency). The adsorption column is loaded ceramic balls in the top and bottom and the region between them contains adsorbent beds. The aim of this arrangement of ceramic balls is in order to enhance a complete wetting of adsorbent, ensure high contact and adsorption of the materials utilized and decrease bed porosity and radial dispersion. The fixed bed adsorption column (FBAC) unit is illustrated in Figure 2.

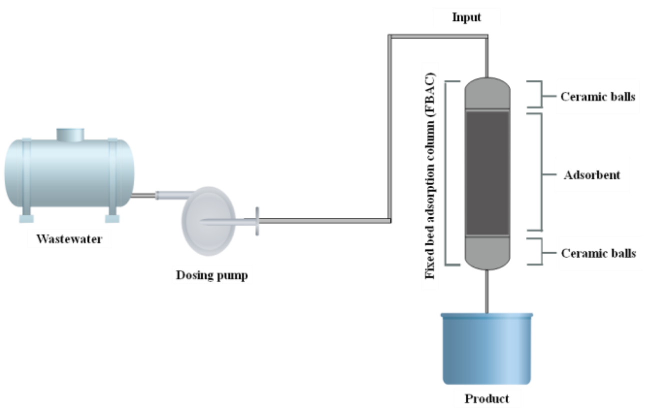


Fig. 2. Schematic diagram of the FBAC unit

2.4.2. Removal of phenol by adsorption process

The model wastewater is utilized as the feedstock in order to check the efficiency of normal and loaded activated carbon in the removal of phenolic compounds. The adsorption process was conducted in in a fixed bed adsorption column (FBAC). To perform the adsorption process, the model wastewater was pumped by employing a dosing pump from the feedstock tank to the top of the column at constant flow rate. The UV spectrophotometer can be utilized to estimate the phenol concentration of product.

2.4.3. Experimental conditions of adsorption process

Table 3 illustrates the variables of experimental (reaction temperature, feed flow rate, bed height, type of adsorbent) of this work:

Table 3: Experimental variables for FBAC process.

Variable	Value
Reaction temperature, °C	25, 50, 75
Bed height, cm	2.5, 5, 7.5, 10
Feed flow rate, mL/sec	1, 2, 3, 4
Type of adsorbent	AC, 10% KOH/AC

All experimental runs are carried out at atmospheric pressure.

2.4.4. Regeneration of the spent adsorbent

The activity of adsorbents was studied after six adsorption cycles at the best conditions that achieved higher removal of phenolic compounds. After that, the spent adsorbents were regenerated by utilizing batch regeneration system (BRS). Filtering the loaded activated carbon saturated with phenolic compounds from the model wastewater under suitable operating conditions is utilized to regenerate the spent adsorbents. In a (BRS), phenolic compounds are eliminated by treating the spent adsorbent with iso-octane, ethanol and methanol. About 10 mL of the solvent was employed to shake the suspension for (30 min) at 60 °C for each gram of spent adsorbent. After that, the washed adsorbent was filtered and putted in an oven in order to dry at 110 °C for 3 h.

3. Results and discussion

3.1. Adsorbent characterization

3.1.1. Surface area and pore volume analysis

The BET analysis was applied in order to estimate the surface area (SA) and pore volume (PV) of the normal and loaded activated carbon utilized as adsorbents in the removal of phenolic compounds. As listed in Table 4, it was observed that after the loading process of KOH over AC, the PV and SA of AC decrease. This demeanor is due to the occupation of KOH in some areas within the AC [17,18].

Table 4. BET results for adsorbents

Sample	pore volume (cm^3/gm)	specific surface area (m^2/gm)	pore size (nm)
AC	0 .487	804.014	2 .423
10% KOH/AC	0 .397	612.645	2 .562

3.1.2. X-ray diffraction (XRD)

The XRD patterns of the normal and loaded activated carbon are shown in Figure 3. As illustrate in Figure 3, It can be notice that the pattern of AC exhibited an amorphous halo at 2θ = 26.4°, which indicates to the reflection of the plane (002), a familiar characteristic of noncrystalline structures such as AC [19, 20]. After the loading of KOH, several peaks appeared at various positions (2θ =24°, 29.8°, 34°, 39.2°, 40.4°) refers to the potassium hydroxide phase [21,22].

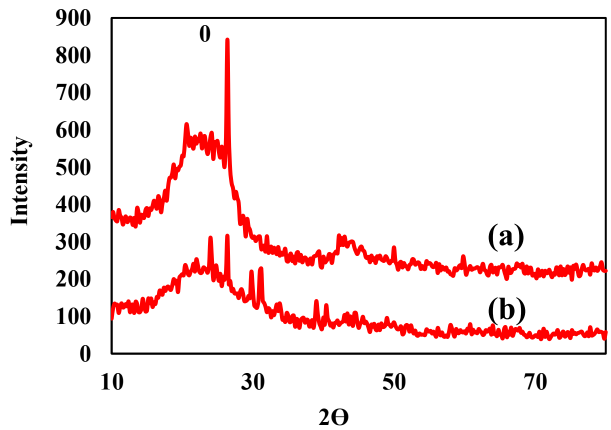


Fig. 3. XRD patterns for (a) activated carbon (b) 10% KOH/AC

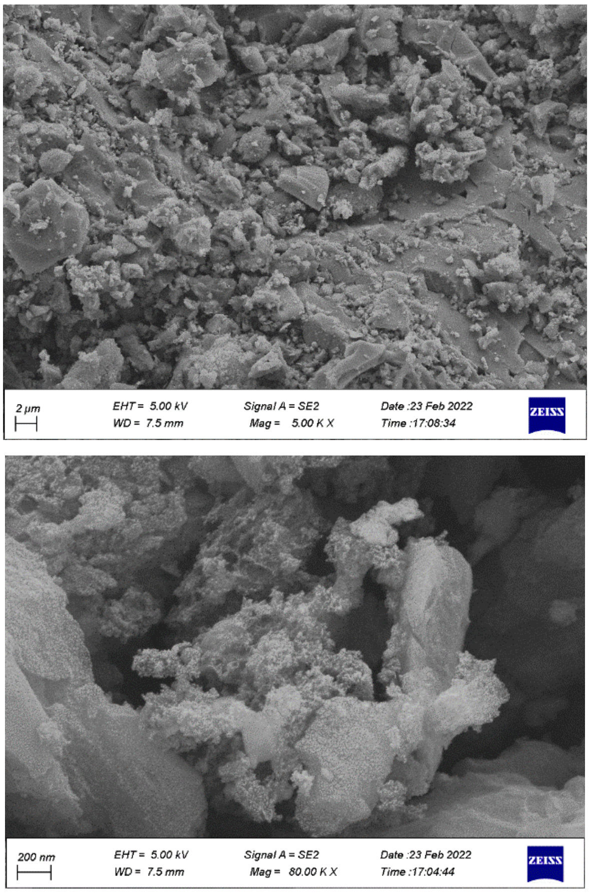
3.1.3. Scanning electron microscopy (SEM)

The surface nature of the normal and loaded AC is displayed by utilizing the SEM test. As shown in Figure 4(a), the SEM images denote several micropores located on the AC surface. The micropores presented on the surface of AC provide high surface area of the adsorbent. Figure 4(b) displays the SEM images of the loaded AC (10% KOH/AC). The SEM images of the loaded AC (10% KOH/AC) appear that the micropores on the surface of AC were blocked by the molecules of KOH, which emphasizes that molecules of KOH were substantially adsorbed onto the surface of AC.

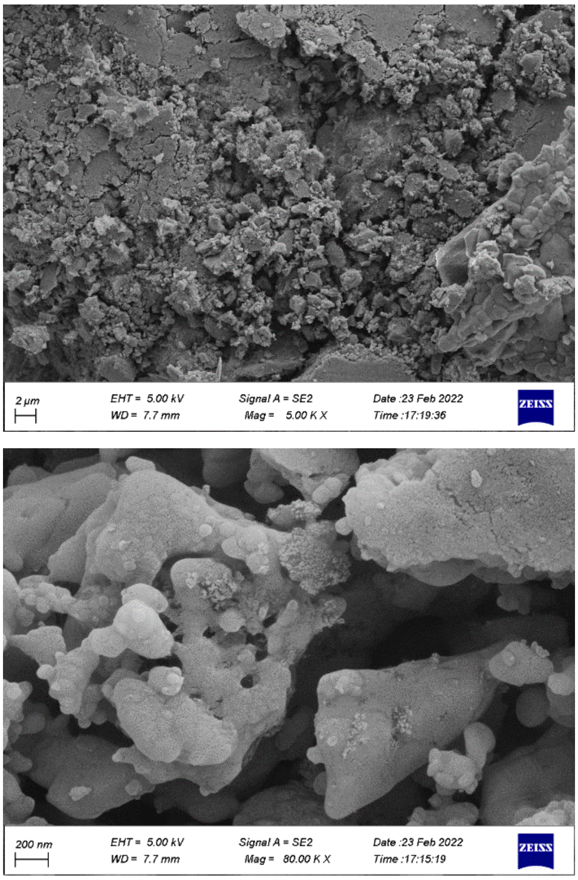
3.2. Results of Adsorption process

3.2.1. Influence of 10% KOH loaded over the AC on phenol removal efficiency

In Figure 5 the influence of the loading of KOH over activated carbon on the adsorption process of phenolic compound from wastewater in FBAC was studied under various operating condition. It was observed that the adsorption efficiency was enhanced by loading of KOH. This is due to the creation of functional groups (hydroxyl groups) on the surface of activated carbon, which are improved the adsorption characteristics of the AC [23, 24].



(a)



(b)

Fig. 4. (a). SEM images for AC, 4(b). SEM images for 10% KOH/AC

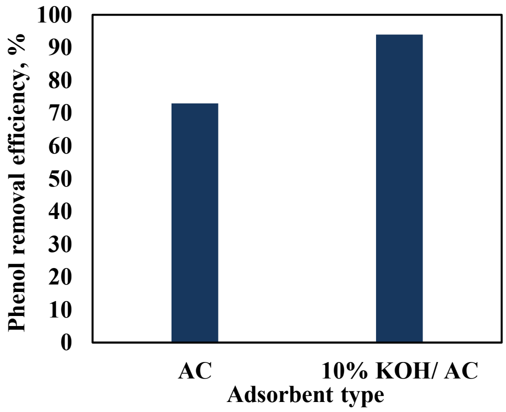


Fig. 5. influence of adsorbent type on phenol elimination efficiency at 10 cm bed height, 1 ml/s feed flow rate, and 75 °C reaction temperature

3.2.2. Influence of temperature on phenol removal efficiency

The influence of reaction temperature on phenol adsorption efficiency was studied by varying temperature from 25 to 75 °C. From Figure 6 and 7, it was noticed that the adsorption efficiency improved with increasing of reaction temperature above room temperature. This might be owing to an increase in the pores number on the surface of adsorbent. The high temperature increases the kinetic energy of phenol molecules and decreases the thickness of outer adsorbent surface. As a result phenol molecules are readily adsorbed on the surface of adsorbent [25-30].While the effect of the height of adsorbent bed on efficiency of adsorption process of phenolic compounds was detected at four various adsorbent bed heights 2.5 cm, 5 cm, 7.5 cm and 10 cm. as illustrated in Figures 8 and 9, the removal of phenolic compounds increased when the bed height was increased. A raise in the adsorbent bed height increases the presence of adsorption sites, which results an enhancing the contact between the adsorbent and feed, and leads to an increase in the surface area available for adsorption. According to the results of the adsorption process, high adsorption efficiency was achieved by using continuous process and in the presence of loaded AC (10% KOH/AC) and the removal of phenolic compounds are improved in comparison with related previous study [17].

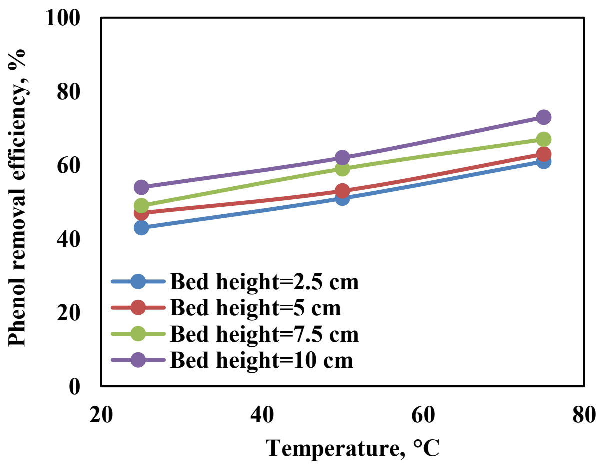


Fig. 6. influence of temperature on phenol elimination efficiency over AC (at feed flow rate = 1 mL/sec)

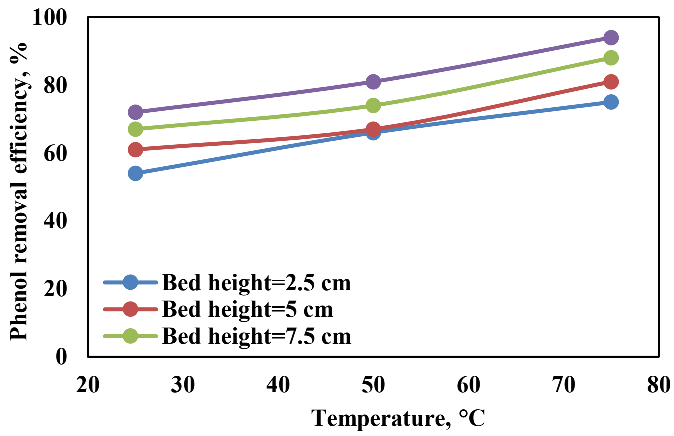


Fig. 7. influence of temperature on phenol elimination efficiency over 10%KOH/AC (at feed flow rate = 1 mL/sec)

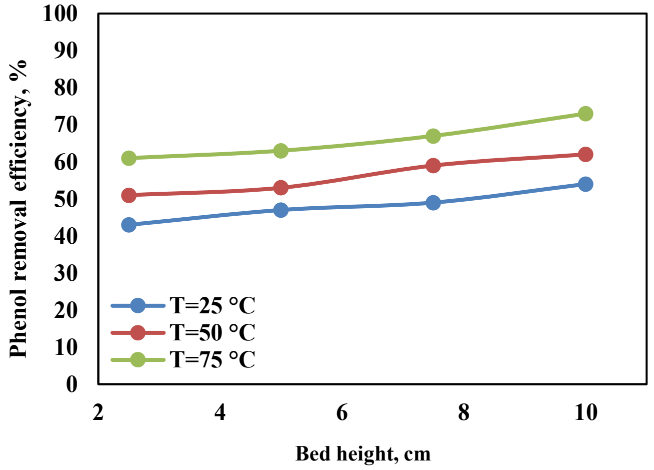


Fig. 8. influence of bed height on phenol elimination efficiency over AC (at feed flow rate = 1 mL/sec)

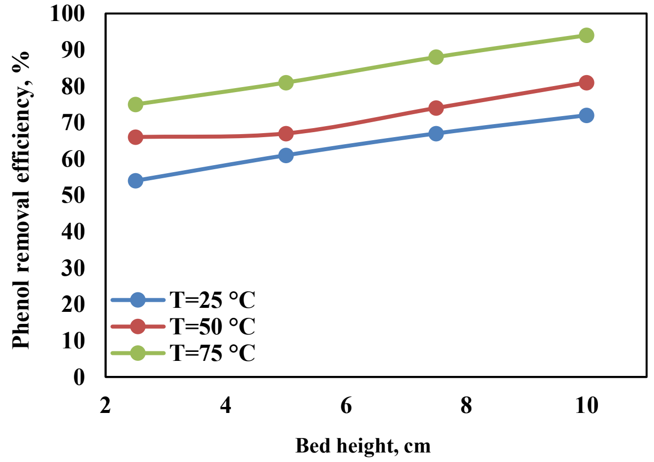


Fig. 9. influence of bed height on phenol elimination efficiency over 10% KOH/AC (at feed flow rate = 1 mL/sec)

3.2.3. Influence of feed flow rate on phenol removal efficiency

The influence of the feed flow rate on the efficiency of adsorption process was investigated with various feed flow rates (1, 2, 3 and 4 mL/sec). Figure 10 and 11 illustrate that the removal of phenolic compounds improved when the feed flow rate was reduced from 4 mL/s to 1 mL/s. This phenomenon is attributed to that with high feed flow rate, the residence time of the phenolic compounds in the FBAC is not long enough. Moreover, the contact time between the phenolic compounds and the adsorbent layer is so fast, leading to decrease in the efficiency of adsorption [31,32].

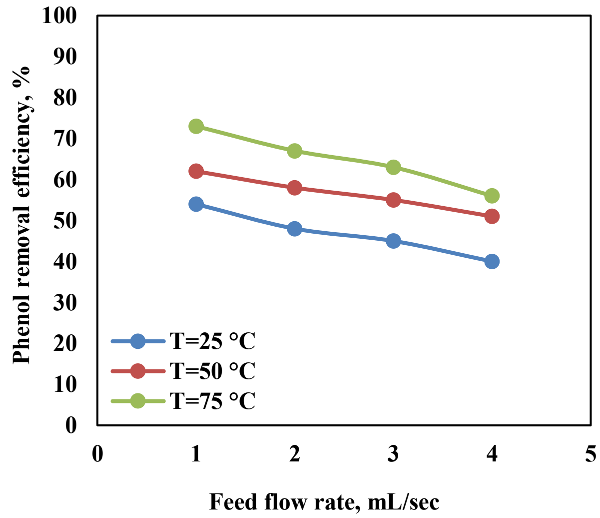


Fig. 10, influence of feed flow rate on phenol elimination efficiency over AC (at bed height = 10 cm)

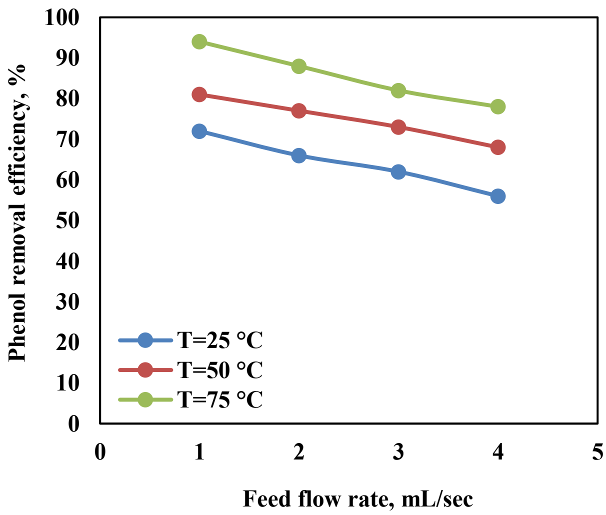


Fig. 11. influence of feed flow rate on phenol elimination efficiency over 10%KOH/AC (at bed height = 10 cm)

3.3. Deactivation and regeneration of the adsorbents

3.3.1. Deactivation study

In this work, the adsorbents efficiency was evaluated after six adsorption cycles at the best conditions (temperature = 75 °C, adsorbent bed height = 10 cm and feed flow rate = 1 mL/sec). The phenol removal efficiencies for the adsorbents after each cycle are illustrated in Figure 12. As shown in Figure, The adsorbents attained a peripheral reduction in overall phenol elimination after six cycles. This behavior indicates the high stability of the adsorbents under best operating conditions. The slight reduction in the efficiency of adsorbent may be owing to the loss of some active adsorption sites during the process of recovery [33,34].

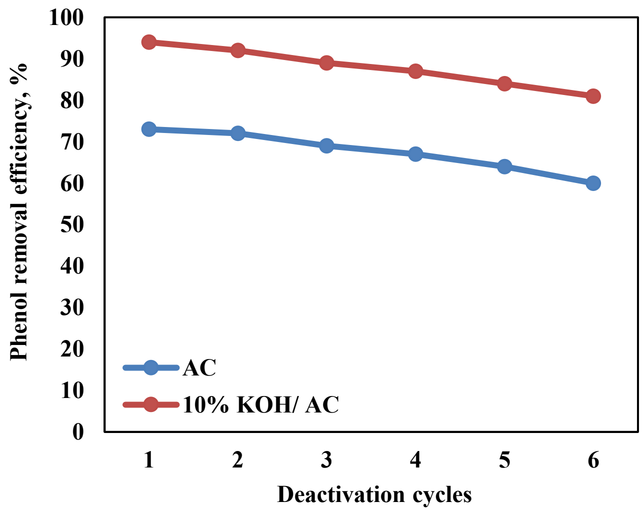


Fig.12. Recovery performance of the adsorbents in terms of phenol elimination capacity for six consecutive cycles

3.3.2. Regeneration study

Several solvents were utilized to evaluate the solvents performance in regeneration process of the spent adsorbents (after six cycles) in terms of phenolic compounds adsorption. In order to regenerate the used adsorbents, methanol, ethanol and iso-octane are employed as regeneration solvents. Figure 13 summarizes the adsorption efficiencies of the adsorbents after the regeneration process by using various solvents. As shown in Figure, the regeneration performance of the used solvents of adsorbents reduces as follows: iso-octane > ethanol > methanol. So, by utilizing iso-octane as a regeneration solvent, normal and loaded AC can be excellently regenerate.

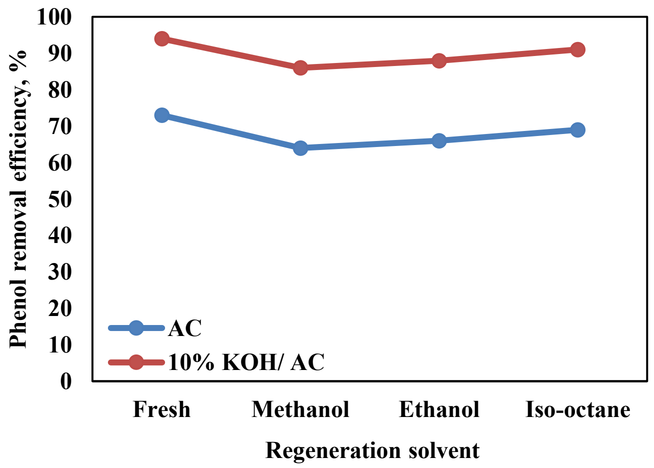


Fig.13. Regeneration performance of adsorbents in terms of phenolic compounds elimination efficiency utilizing several solvents.

4. Conclusions

The adsorption process of wastewater is performed in a laboratory-scale fixed bed adsorption column (FBAC), with bed height (2.5, 5, 7.5, 10) cm, reaction temperature (25, 50, 75) °C, feed flow rate (1, 2, 3, 4) mL/sec and various adsorbent types (AC, 10% KOH/AC). The adsorption efficiency with AC, and KOH/AC were 73% and 94%, respectively, at 10 cm bed height, 1 ml/s feed flow rate, and 75 °C reaction temperature. The activity of adsorbents was evaluated after six consecutive adsorption cycles at the best conditions, and the adsorbents show high stability in terms of phenolic compounds adsorption. Also, the spent adsorbents were regenerated by employing various regeneration solvents (methanol, ethanol and iso-octane), and the results show that iso- octane attained highest regeneration capacity.

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