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Desorption of 4-chlorophenol from spent granular activated carbon in continuous flow ultrasonic reactor

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ABSTRACT

The purpose of this work was to explore the application of low frequency ultrasound for the desorption of 4-chlorophenol (4-CP) from saturated granular activated carbon (GAC) in a continuous flow ultrasonic reactor. The effects of operating parameters such as the amount of adsorbent, intensity of ultrasound irradiation, temperature, flow rate of desorbing solution, NaOH, ethyl alcohol, and mixture of NaOH and ethyl alcohol on the desorption process were investigated. The obtained results show that the desorption decreased with increasing the amount of adsorbent and intensity of ultrasound irradiation. The amount of 4-CP desorption gradually increased with the increase in temperature and flow rate of desorbing solution. Using NaOH or/and ethyl alcohol as desorbing solutions causes an enhancement of the desorption. Desorption was improved with increasing the concentration of NaOH and ethyl alcohol in the desorbing solution. Future studies should innovate the ultrasound-assisted desorption process in a pilot-scale test that applies this desorption procedure to a GAC adsorber used in organic compounds removal.

Keywords: Desorption; Activated carbon; Ultrasound; 4-Chloorphenol; Continuous flow reactor

1. Introduction

A wide range of methods has been developed for the removal of organic pollutants form water and wastewater. Among these methods, adsorption is by far the most versatile and widely used technique because of its initial cost, simplicity of design, ease of operation, and insensitivity to toxic substances. Adsorption processes using activated carbon represent a highly effective and versatile technique to remove low concentrations of organic pollutants from drinking water, wastewater, and groundwater. The US Environmental Protection Agency has designated adsorption onto granular activated carbon (GAC) as a "best available technology" for removing organic pollutants [1]. The progressive accumulation of pollutants adsorbed on the surface of the GAC leads to a gradual reduction in its adsorption capacity until adsorption is no longer possible. In the common practice, when an activated carbon reached to its saturation limit, the spent GAC was usually incinerated, discarded, or landfilled, which implies high economical and environmental costs. Thus, the study of GAC regeneration becomes a necessity as a solution to these problems.

The most common methods employed for the regeneration of spent GAC are thermal regeneration

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[2], solvent regeneration [3], direct oxidation and catalytic wet oxidation regeneration [4], and biological regeneration [5]. The main objective of regeneration is to remove adsorbed pollutants and recover the adsorption capacity of virgin adsorbent without loss of its active structure and to enable its reuse. The above cited regeneration methods suffer from severe drawbacks, such as requirement of high energy and time consumption, loss of carbon surface area, destruction of micropores, high cost of operation and low regeneration efficiency, and consequently are limited either technically or economically. To resolve the deficiencies of the common regeneration techniques, new alternative methods that use more advanced technologies are developed. Therefore, the regeneration of activated carbon has been recently investigated using microwaves [6], supercritical water oxidation [7], electric currents [8–11], and ultrasound [12–24].

When ultrasound passes through a liquid medium, the interaction between the ultrasonic waves, liquid, and dissolved gas leads to an exciting phenomenon known as acoustic cavitation. Acoustic cavitation is a phenomenon in which bubbles present in the liquid medium grow and collapse due to pressure fluctuations caused by ultrasound waves [25]. The most pertinent effects of ultrasound on liquid–solid systems are mechanical and are attributed to symmetric and asymmetric cavitation [26]. The motion of bubbles during oscillation and collapse causes significant fluid flow effects, such as micro-jetting and strong shear forces [27]. In addition, shock waves are produced which have the potential of creating microscopic turbulence within interfacial films surrounding nearby solid particles, also referred to as microstreaming [28]. Asymmetric collapse leads to the formation of microjets of solvent that impinge on the solid surface [26]. Acoustic streaming is the movement of the liquid induced by the sonic wave, which can be considered to be the conversion of sound to the kinetic energy, and is not a cavitation effect [29]. These phenomena increase the rate of mass transfer near the surface as well as possibly thinning the film [26]. Ultrasound may be very useful tool in intensifying the mass transfer process and regeneration of spent GAC by breaking the affinity between adsorbate and adsorbent [12–14,23,24,30–34].

In our previous works, ultrasound is employed to regenerate GAC saturated by 4-chlorophenol (4-CP) and good performance for GAC regeneration are obtained [12–14,23,24]. Nevertheless, ultrasound irradiation has been used in batch mode and GAC regeneration efficiency still requires improvements to be practically viable. In order to obtain a better performance of the process, studies must be carried out in continuous flow ultrasonic reactor. Therefore, this work aims to the desorption of 4-CP from spent GAC by low frequency ultrasound in continuous flow mode. The effects of operating parameters on the desorption process were examined.

2. Materials and methods

2.1. Materials

4-CP supplied by Sigma (99.5%) was used as an adsorbate. Aqueous solutions of 4-CP were prepared by dissolving the required amount in pure water.

The GAC used in this work was purchased from Sigma-Aldrich. The GAC has a mean granulometry of 1 mm. Prior to all experiments, the carbon was washed repeatedly with pure water to remove any fine particles and soluble materials. Finally, the washed activated carbon was dried in an oven at 110°C to constant weight and stored in a desiccator until use.

The scanning electron microscopy (SEM) technique was employed to observe the surface physical morphology of the activated carbon. Fig. 1(a) and (b) shows the SEM images of GAC surface at 400× and 4,000× magnification, respectively. These photographs clearly reveal the surface texture and different levels of porosity of the carbon. The carbon looks like a random pile of crystallized blocks of various sizes and shapes.

2.2. Experimental setup

The experimental setup used in this work corresponding to a continuous flow ultrasonic reactor is shown schematically in Fig. 2. A peristaltic pump (Kerlabo) whose volumetric flow rate can be changed enables to feed the ultrasonic reactor with the desorbing solution contained in the feed tank. The ultrasonic reactor consists of a 100 mL cell surrounded by a glass cylindrical jacket, allowing water-cooling of the reactor to control the temperature. A peristaltic pump ensured the flow out of the ultrasonic reactor in order to keep the volume of the desorbing solution constant. From the top of the reactor, the ultrasonic horn was immersed into the reactor. The ultrasonic irradiation was carried out with a programmable and microprocessor-based sonic processor Vibra-Cell (Sonics & Materials, Model: VCX 750). The ultrasonic probe of the processor was fabricated from high-grade titanium alloy and had a tip diameter of 25 mm. The ultrasound frequency generated by this probe was 20 kHz with maximum power output of 750 W. The processor had facility of power output control. The power control knob was set at 20% during sonication, except when the effect of power was examined. Acoustic power dissipated in the reactor was measured using standard calorimetric method [35,36].

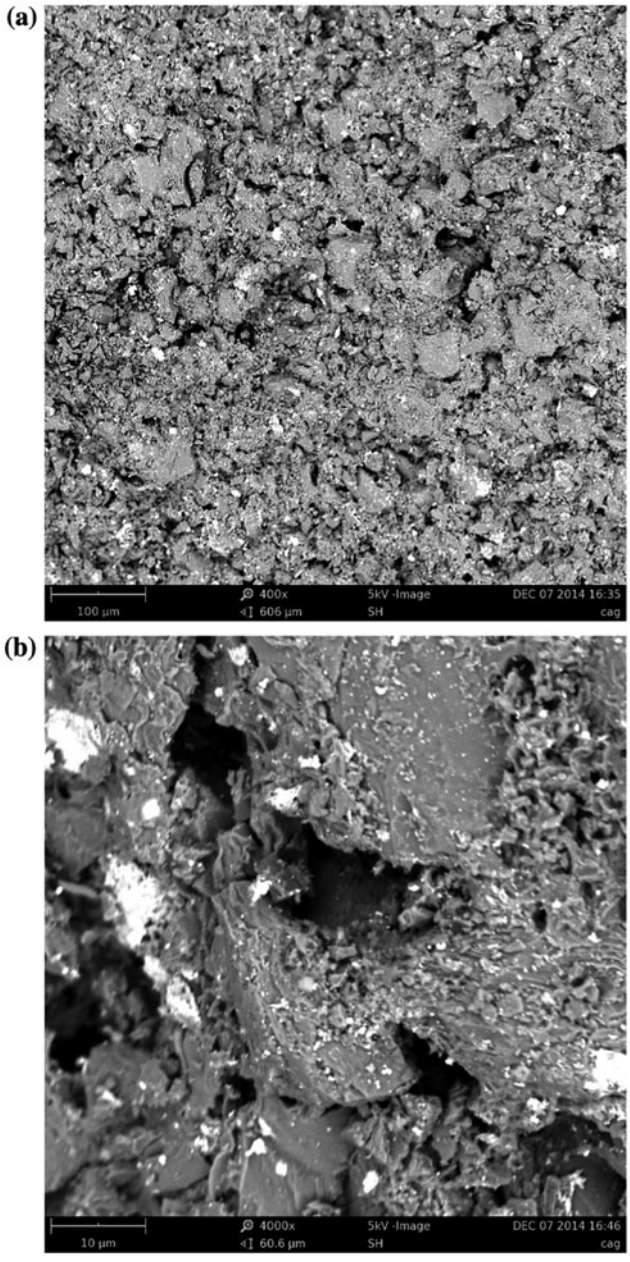

Fig. 1. SEM micrographs of GAC surface ((a) 400× and (b) 4,000×).

2.3. Adsorption experiments

For adsorption experiments, the GAC and 4-CP system were placed in a shaker and three days was allowed for equilibrium to be reached before the desorption experiments were performed. After three days, the carbon loaded with 4-CP was then separated from the solution. The concentrations of 4-CP in solution were determined by UV–vis spectroscopy (Lightwave II). The activated carbon loading was determined by mass balance.

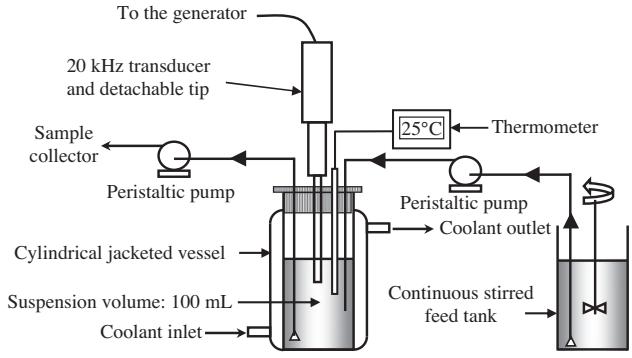

Fig. 2. Scheme of the continuous flow ultrasonic reactor.

2.4. Desorption of 4-CP from GAC

A definite mass of the prepared spent GAC was introduced into the continuous flow ultrasonic reactor containing the desorbing solution. After selected times of sonication, desorption kinetics was determined by following the 4-CP concentration change in the aqueous phase. The bulk concentration of adsorbate was measured by a UV–vis spectrophotometer. From the bulk concentration, the amount of desorption could be determined.

All experiments were conducted by triplicate and the mean values are reported. The maximum standard deviation was ±3%.

3. Results and discussion

3.1. Effect of amount of adsorbent

To determine the effect of amount of adsorbent on 4-CP ultrasonic desorption from GAC, experiments were conducted for GAC loading of 300 mg/g, ultrasound intensity of 20% (calorimetric power of 14 W) and temperature of 25°C, while the amount of adsorbent was varied from 0.05 to 0.4 g for various volumetric flow rates of desorbing water in the range of 3–10 mL/min. The time course of 4-CP concentration during ultrasonic desorption is shown in Fig. 3. It was observed that the concentration of 4-CP for a given volumetric flow rate of desorbing water increased with increasing the amount of adsorbent. This can be attributed to greater surface area and the availability of more adsorbed 4-CP molecules when higher amount of GAC was used. At higher GAC amount, there is a very fast desorption from the adsorbent surface that produces a higher 4-CP concentration in the desorbing water than when the GAC mass is lower. It was also seen from Fig. 3 that for a given amount of adsorbent the concentration of 4-CP in the

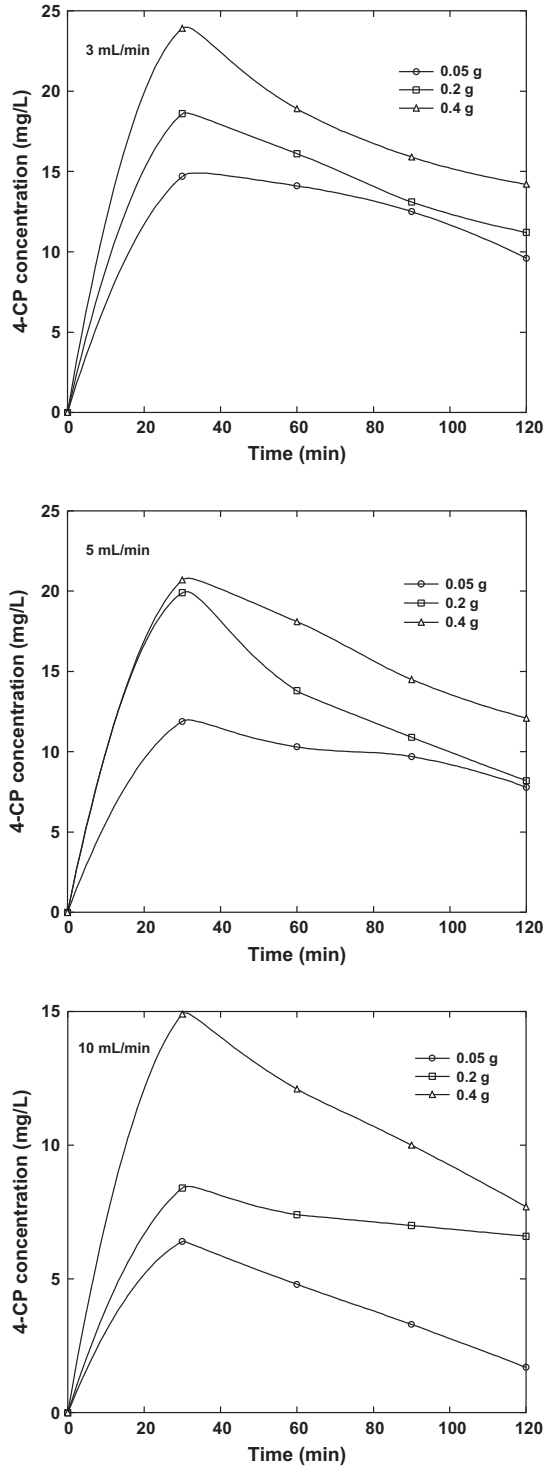

Fig. 3. Time course of 4-CP concentration during ultrasonic regeneration of spent GAC at various amounts of adsorbent and different volumetric flow rates (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25˚C, adsorbent mass: 0.05–0.4 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–10 mL/min).

desorbing water decreased with increasing the volumetric flow rate. This is due to the dilution of the desorbing medium at higher flow rate.

During this study, different runs were compared using the amount of 4-CP desorbed by ultrasound after 120 min of sonication time, rather than the time course of 4-CP concentration obtained during the desorption in continuous flow ultrasonic reactor.

The effect of the amount of adsorbent in the range of 0.05–0.4 g on the desorption of 4-CP from GAC for different volumetric flow rates of desorbing water is shown in Fig. 4. It was observed that, for a give flow rate, desorption was decreased by increasing the amount of adsorbent. This is due to the split in the flux or the concentration gradient between solute concentrations on the adsorbent surface and in the desorbing water. The desorbed amount was high when the adsorbent mass was low because a great volume of desorbing water was used for comparatively less GAC. It was also be noticed that the amount of 4-CP desorption increased with increasing the volumetric flow rate. This is because higher flow rate ensured high concentration gradient between the surface of GAC and the desorbing water, which promotes the desorption of 4-CP from the adsorbent surface.

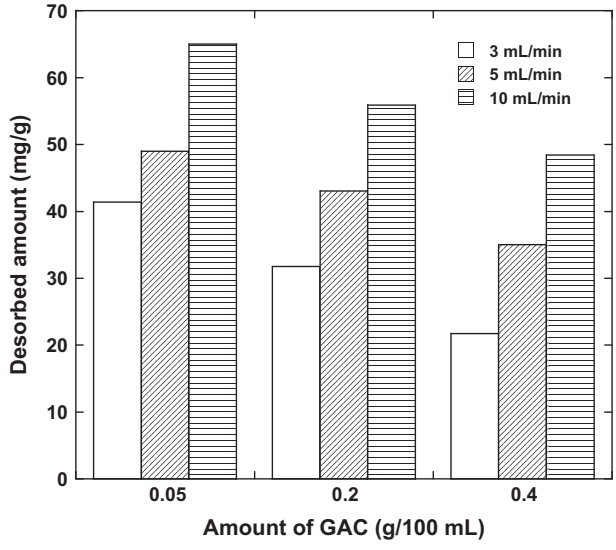

Fig. 4. Effect of amount of adsorbent on the ultrasonic desorption of 4-CP from GAC after 2 h at different volumetric flow rates (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25˚C, adsorbent mass: 0.05–0.4 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–10 mL/min).

Desorption by ultrasound could be related to the strong turbulence in the medium through different physical phenomena induced by ultrasound and cavitation. Desorption is improved by breaking bonds between 4-CP molecules and adsorbent surface and intensifying mass transfer phenomena through high-speed microjets, high-pressure shock waves, microturbulence, and acoustic vortex microstreaming [12–14,23,24,30–34]. When these physical phenomena hit a solid object, they tear off all molecules adsorbed onto it. Under the impacts of ultrasound and cavitation, the diffusion boundary layer gets thinner, the convective mass transfer increases and the diffusion significantly speeds up.

3.2. Effect of intensity of ultrasound irradiation

Before starting the ultrasound experiments, desorption tests without ultrasound were performed as a function of time. Experiments carried out in the absence of ultrasound (silent conditions) using a glass reactor (with the same geometry as the sonication reactor) and a magnetic stirrer with a stirring rate of 400 rpm. The reactor provides uniform mixing conditions due to continuous agitation. All other conditions were the same as those used with ultrasonic desorption. The obtained results showed that without ultrasound, no desorption was occurred even after 120 min of contact time.

In order to examine the effect of the intensity of ultrasound irradiation on the regeneration of GAC exhausted by 4-CP, experiments were carried out using an amount of adsorbent of 0.2 g at different volumetric flow rates of desorbing water in the range of 3–10 mL/min. The results of the desorption experiments in the presence of ultrasound are illustrated in Fig. 5. Desorption decreased with increasing the intensity of ultrasound from 20 to 35%. At three chosen intensities, the lower one was more effective. This behavior showed that the influence of ultrasound on desorption was dependent on the studied system as the heat of adsorption determines the quantum of energy required by a cavitation event to overcome the affinity between an adsorbate and an adsorbent. Additionally, it seems that further experiments should be conducted at ultrasound intensities lower than 20%, but this is the lower intensity provided by the used ultrasonic generator.

For a given intensity of ultrasound, the desorption increased with increasing the volumetric flow rate of desorbing water. This is because higher flow rate ensured high concentration gradient between the surface of GAC and the desorbing water, which promotes the desorption of 4-CP from the adsorbent surface.

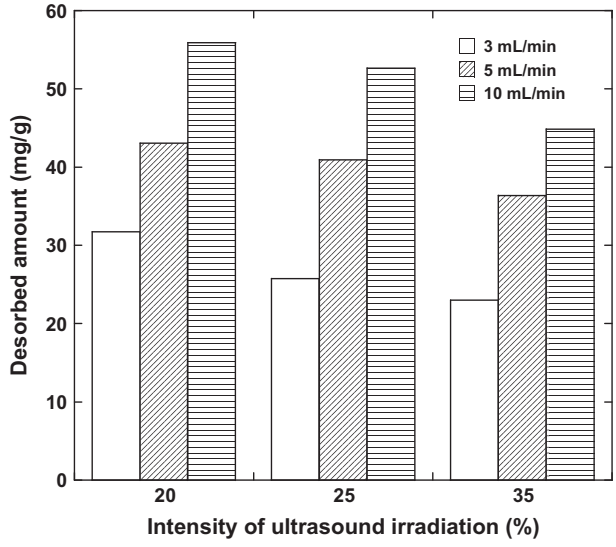

Fig. 5. Effect of intensity of ultrasound irradiation on the ultrasonic desorption of 4-CP from GAC after 2 h at different volumetric flow rates (frequency: 20 kHz, intensity of ultrasound: 20–35%, volume: 100 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–10 mL/min).

3.3. Effect of temperature

The effect of aqueous temperature on the desorption of 4-CP from GAC was investigated in the range of 15–45˚C at various volumetric flow rates of desorbing water (3–10 mL/min). Desorption results are shown in Fig. 6. Desorption increased with increasing temperature between 15 and 45˚C. This indicates that the desorption process is endothermic in nature. This improvement is felt to be due to the acceleration of the desorption process by the increased movement of adsorbate molecules from the adsorbent surface to the bulk solution at higher temperatures, owing to the decrease in the viscosity of the solution. Additionally, cavitating bubbles are more easily produced at high temperature because of the decrease of the liquid tensile stress and viscosity [28]. Desorption is promoted if such bubble collapse occurs near the adsorbent surface. This enhancement of desorption is attributed to the physical and thermal effects of ultrasound. The physical phenomena are mostly produced by the acoustic vortex microstreaming and by the high-speed microjets and high-pressure shockwaves induced by acoustic cavitation [12–14,23,24,30–34]. The thermal effect is mostly given by localized hot spots formed when bubbles cavitated as well as by piezoelectric transducer heating [24]. Thus, the improvement of desorption with temperature may be due to the increase in the breaking of bonds between

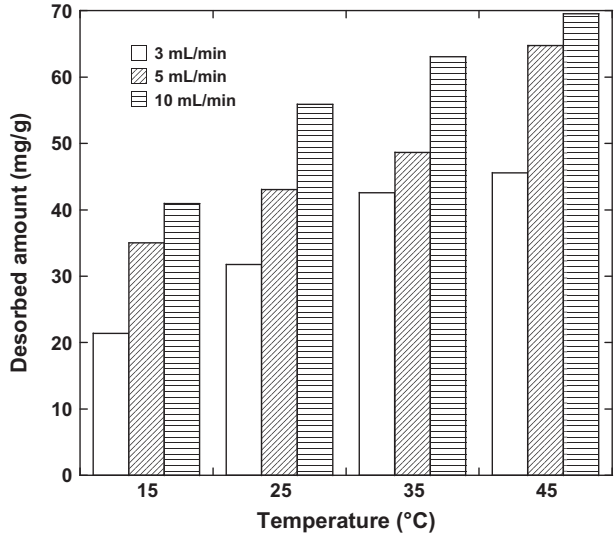

Fig. 6. Effect of temperature on the ultrasonic desorption of 4-CP from GAC after 2 h at different volumetric flow rates (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 15–45°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–10 mL/min).

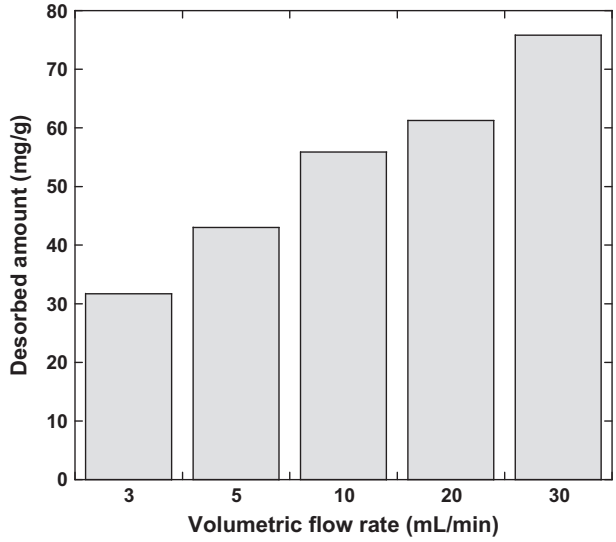

Fig. 7. Effect of volumetric flow rate on the ultrasonic desorption of 4-CP from GAC after 2 h (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–30 mL/min).

adsorbate molecules and the activated carbon surface and/or the increase in the diffusion rate of adsorbate molecules into the pore at higher temperature as diffusion is an endothermic process. From Fig. 6, it was noticed that the desorption of 4-CP from GAC increased with increasing the volumetric flow rate of the desorbing solution, whatever the temperature is.

3.4. Effect of volumetric flow rate

The influence of the volumetric flow rate on the regeneration of the spent GAC was investigated between 3 and 30 mL/min at different volumetric flow rates and 25°C. The obtained results are illustrated in Fig. 7. It was observed from this figure that the amount of 4-CP desorption increased with increasing the flow rate. This flow rate dependence can be accounted for by the fact that for higher value of flow rate, the contact time is shorter, and hence the concentration gradient between the desorbing water and the adsorbent surface is greater. This leads to higher desorption of 4-CP molecules from GAC. On the other hand, for lower flow rate, the contact time is higher, and the desorption of 4-CP is lower due to low concentration gradient between adsorbent surface and desorbing water. The higher flow rate caused higher desorbed amount, but higher flow rate decreases the concentration of 4-CP in the effluent due to the dilution of the desorbing water.

3.5. Effect of NaOH

In our previous study [37], we found that a higher solution pH (11) decreased the adsorption of 4-CP onto GAC. Based on the previous result, the effect of NaOH as a desorbing solution on the ultrasonic desorption of 4-CP from GAC was investigated using three different sodium hydroxide concentrations (0.01, 0.05, and 0.1 M) at various volumetric flow rates in the range of 3–10 mL/min. Fig. 8 show the results of desorption in sodium hydroxide solution after 120 min of sonication. From this figure, it was observed that the use of NaOH as a desorbing solution drastically enhanced the desorption. It was also noticed that the desorption increased with increasing NaOH concentration. This can be attributed to the dependency of 4-CP ionization on the pH value. The ionic fraction of 4-CP ion (φ_{ions}) can be calculated from:

$$\varphi_{ions} = \frac{1}{1 + 10^{(pKa-pH)}} \quad (1)$$

Obviously, φ_{ions} increases as the pH value increased. The dissociation constant of 4-CP (pKa) is 9.2. Hence, at pH ≥ 12, the compound exists mainly in ionic form ($\varphi_{ions} \geq 0.998$) due to deprotonation of the phenolic group. Ionic form of 4-CP is much more

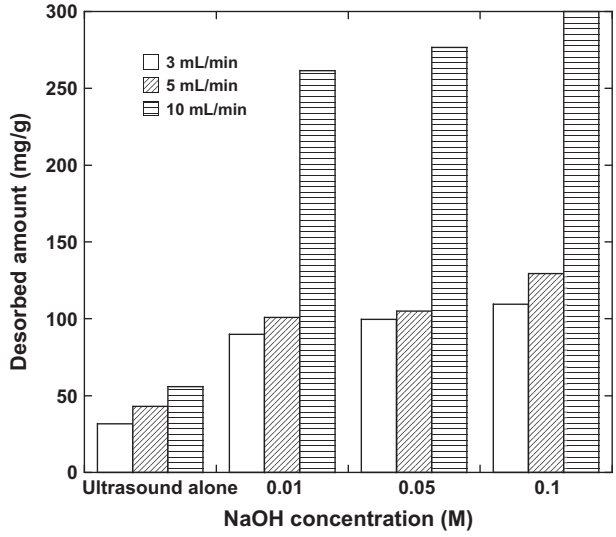

Fig. 8. Effect of NaOH concentration on the ultrasonic desorption of 4-CP from GAC after 2 h (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 3–10 mL/min).

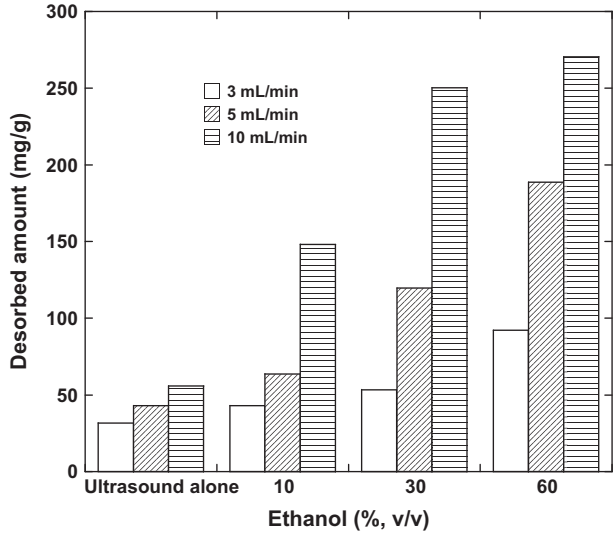

Fig. 9. Effect of ethyl alcohol concentration on the ultrasonic desorption of 4-CP from GAC after 2 h (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 3–10 mL/min).

hydrophilic and soluble than the neutral state. In NaOH solutions, both the surface of the GAC and the adsorbed 4-CP are negatively charged and because of electrostatic repulsions between chlorophenolate and adsorbent surface and also between chlorophenolate molecules, 4-CP is favorably desorbed as chlorophenolate species [12,13,23,24]. Moreover, NaOH solution can hydrolyze some chemical bonds between 4-CP hydroxyl groups and the surface oxygen groups of the GAC [7,38]. While spent GAC was treated with NaOH desorbing solution, 4-CP molecules were easily brought into aqueous phase resulting in an improvement of the desorption of 4-CP from GAC surface.

As can be observed in Fig. 8, it was noticed that the desorption of 4-CP from GAC increased with increasing the volumetric flow rate of the desorbing solution, especially at a volumetric flow rate of 10 mL/min. After being regenerated by 0.1 M NaOH at a volumetric flow rate of 10 mL/min, whole of the adsorption capacity of GAC was restored.

3.6. Effect of ethyl alcohol

Desorption of 4-CP from GAC was examined in continuous flow ultrasonic reactor at different ethanol concentrations in the range of 10–60% (v/v) and various flow rates (3–10 mL/min). As shown in Fig. 9, it can be observed that the desorption increased with increasing the ethanol percentage in the desorbing solution. This result is expected because ethyl alcohol decreases the tensile stress of the liquid and thus reduces the cavitation threshold and facilitates the generation of cavitating bubbles [12,13,23,24]. The production of more transient cavitation bubbles helps to produce easily physical and thermal effects. Furthermore, ethanol could not only reduce the threshold for cavitation, but also capture the primary radicals (HO• and H•) to form secondary radicals (C_2H_4OH•) beneficial for the desorption [39]. From Fig. 9, it was also noticed that the amount of 4-CP desorption increased gradually with increasing the volumetric flow rate.

3.7. Effect of NaOH and ethyl alcohol

Desoption of 4-CP from GAC was investigated using a mixture of 10% (v/v) ethanol and 0.05 M NaOH as desorbing solution at different flow rates in the rage of 3–10 mL/min. Fig. 10 reported the desorption results obtained in a mixture of ethanol and sodium hydroxide. Desorption was improved when a mixture of ethanol and NaOH was employed as desorbing solution. This might be due to the lowering of cavitation threshold and the creation of repulsion forces between activated carbon surface and 4-chlorophenolate anions. Additionally, it was remarked that the desorption of 4-CP from GAC increased with increasing the volumetric flow rate of the desorbing solution, especially at a volumetric flow rate of 10 mL/min.

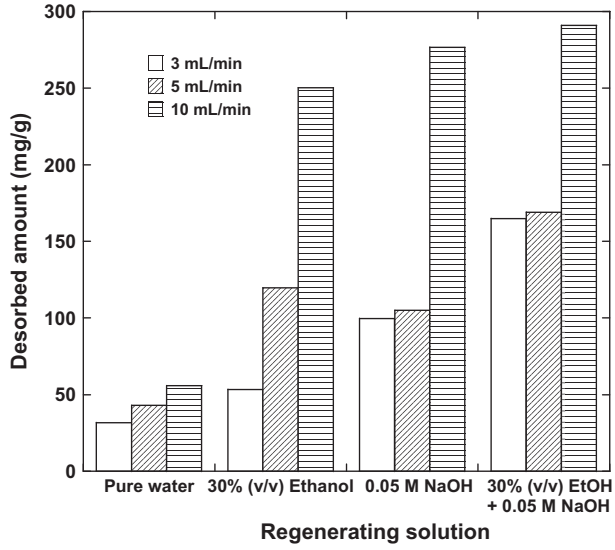

Fig. 10. Effect of a mixture of ethanol (30%, v/v) and NaOH (0.05 M) on the ultrasonic desorption of 4-CP from GAC after 2 h (frequency: 20 kHz, intensity of ultrasound: 20%, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 3–10 mL/min).

3.8. Adsorption on regenerated GAC

The adsorption kinetics of 4-CP onto virgin GAC and ultrasound regenerated activated carbon after three cycles of adsorption–regeneration are presented in Fig. 11. From this figure, it clearly appears that adsorption capacities of the activated carbon regenerated by ultrasound and that of the virgin carbon remained the same. This indicates that recovery of the initial adsorption capacity of activated carbon was attained and regeneration of GAC was complete.

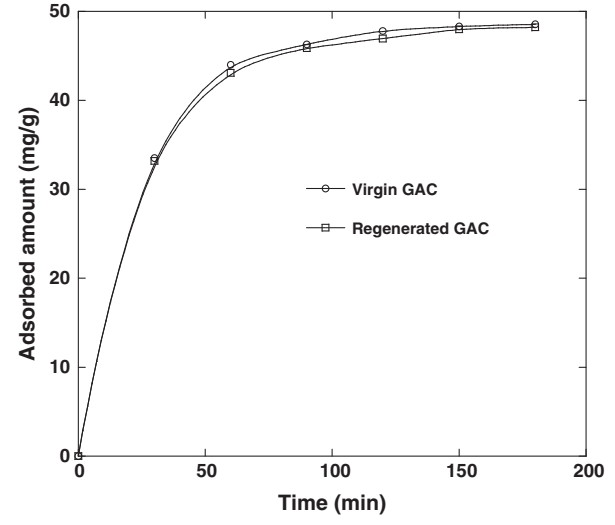

Fig. 11. Adsorption kinetics of 4-CP onto virgin GAC and regenerated GAC (volume: 100 mL, adsorbent mass: 0.2 g, 4-CP initial concentration: 100 mg/L, stirring speed: 300 rpm, temperature: 25°C).

4. Conclusion

A series of tests were performed in continuous flow ultrasonic reactor in order to desorb 4-CP from exhausted GAC. The obtained results show that the desorption was decreased by increasing the amount of adsorbent and the intensity of ultrasound from 20 to 35%. The amount of 4-CP desorbed from GAC surface increased with increasing the volumetric flow rate of desorbing solution and temperature between 15 and 45°C. This indicates that the desorption process is endothermic in nature. The use of NaOH as a desorbing solution drastically enhanced the desorption, and the amount of 4-CP desorbed increased with increasing the NaOH concentration. Desorption increased with increasing the ethanol percentage in the desorbing solution. Desorption was improved when a mixture of ethanol and NaOH was employed as desorbing solution. Ultrasound-assisted desorption is an innovative technique because the adsorbed molecules may be released into solution by breaking bonds and intensifying mass transfer phenomena through acoustic vortex microstreaming, shockwaves, microjets, and thermal effects from cavitational collapse. The next step is validation in a pilot-scale test that applies this desorption technique to a GAC adsorber employed in organic compounds removal.

Acknowledgments

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