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Regeneration of exhausted granular activated carbon by low frequency ultrasound in batch reactor

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ABSTRACT

The aim of the present work is to investigate the use of low frequency ultrasound (20 kHz) for the desorption of 4-chlorophenol (4-CP) from granular activated carbon (GAC) in batch reactor. The influence of experimental conditions such as amount of adsorbent, acoustic power, pulsed ultrasound, temperature, concentrations of NaOH, ethyl alcohol, and salt and saturating gases on the desorption was examined. The obtained results show that continuous wave ultrasound was more effective than pulsed modulation. The desorption was enhanced with the rise in temperature from 15 to 45˚C and was reduced in the presence of salt (NaCl) and saturating gases such as argon and nitrogen. The regeneration of GAC was increased with increasing the concentration of NaOH from 0.01 to 0.1 M and decreased afterward. The desorption increased with increasing the ethanol percentage. Using different mixtures of ethanol and NaOH at various concentrations, it has been shown that these regenerating mixtures improve the desorption of 4-CP. A mixture of 30% ethanol and 0.1 M NaOH produces a synergistic effect and a significant intensification of desorption. The findings revealed the potential to regenerate exhausted GAC by ultrasonic irradiation.

Keywords: Regeneration; Granular activated carbon; Ultrasound; Batch reactor

1. Introduction

Granular activated carbon (GAC) has been extensively used as an efficient and versatile adsorbent in the removal of organic pollutants from water and wastewater due to its excellent adsorbent properties, large surface area, tailored pore distribution, and high degree of surface reactivity [1–6]. The progressive accumulation of pollutants adsorbed on the GAC surface leads to exhaustion of the available adsorption sites and a gradual reduction in adsorptive capacity until adsorption is no longer possible. However, the use of GAC adsorption alone is limited in that the pollutants are not removed, but instead transferred to the solid phase after adsorption. Thus, in the common practice, the exhausted carbon was usually incinerated, discarded, or landfilled. In order to meet the environmental and economical requirements, the spent carbon should be regenerated. The main objective of regeneration is to remove adsorbed pollutants in order to recover the original adsorptive capacity of the GAC without loss of its active structure and to enable its reuse.

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A wide variety of GAC regeneration techniques have been proposed and applied. The most common methods are thermal regeneration [7,8], solvent regeneration [9], direct oxidation, catalytic wet oxidation regeneration [10], and biological regeneration [11]. Nevertheless, these techniques usually have disadvantages 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 solve these drawbacks, new alternative methods that use more advanced technologies are developed. Thus, the regeneration of activated carbon has been recently examined using microwaves [12], supercritical water oxidation [13], electric currents [14–17], and ultrasound [18–30].

Ultrasound irradiation in a liquid medium can induce acoustic cavitation under specific experimental conditions. The process of cavitation refers to the rapid growth and implosive collapse of bubbles in a liquid resulting in an unusual reaction environment within and in the vicinity of bubbles. Two unique cavitation effects have been intensively researched for decades, namely, sonochemical and sonophysical effects [31–36]. Sonochemical effects include pyrolysis of chemical species inside the cavitation bubbles and radical reactions inside and adjacent to the bubbles. The sonophysical effects are attributed to symmetric and asymmetric cavitation [35]. The motion of bubbles during oscillation and collapse causes significant fluid flow effects, such as micro-jetting and strong shear forces [37]. 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 [32]. Asymmetric collapse leads to the formation of microjets of solvent that impinge on the solid surface [35]. 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 [38]. These phenomena increase the rate of mass transfer near the surface as well as possibly thinning the film [35]. Ultrasound may be a very useful tool in intensifying the mass transfer process and regeneration of spent GAC by breaking the affinity between adsorbate and adsorbent [18–20,29,30,39–43].

Although in our previous papers, we have studied the regeneration of saturated activated carbon by ultrasound [18–20,29,30], comprehensive studies on the effects of process variables are yet to be carried out. Additionally, to the best of our knowledge, there is no well-documented publication on the influence of NaOH and ethyl alcohol concentrations, salt and saturating gases on the ultrasonic regeneration of GAC. Thus, the objective of the present work is to examine the desorption of 4-chlorophenol (4-CP) from exhausted GAC by low frequency ultrasound in batch reactor under various experimental conditions that include amount of GAC, acoustic power, pulsed ultrasound, temperature, concentrations of NaOH, ethyl alcohol, and salt and saturating gases. The regeneration of exhausted GAC in different mixtures of NaOH and ethyl alcohol at various concentrations was also carried out.

2. Materials and methods

2.1. Materials

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.

4-Chlorophenol (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.

2.2. Adsorption experiments

For adsorption experiments, the GAC and 4-CP system was placed in a shaker and 3 d was allowed for equilibrium to be reached before the desorption experiments were performed. After 3 d, 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.

2.3. Ultrasonic reactor

Desorption experiments were performed in a batch ultrasonic reactor, which consists of a 100 mL cell surrounded by a glass cylindrical jacket, allowing water cooling of the reactor to control the temperature. For sonication of the reaction mixture, a programmable and microprocessor-based sonic processor Vibra-Cell (Sonics & Materials, Model: VCX 750) was used. 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% (acoustic power of 14 W) during sonication, except when the effect of power was examined. Acoustic power dissipated in the reactor was measured using standard calorimetric method [44,45].

2.4. Regeneration of exhausted GAC

A definite mass of the prepared exhausted GAC was introduced into the batch ultrasonic reactor containing the regenerating solution. After selected times of sonication, the 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 exhausted GAC

To study the effect of amount of spent GAC on 4-CP desorption, experiments were conducted at GAC loading of 300 mg/g, calorimetric power of 14 W, and temperature of 25°C, while the amount of adsorbent was varied in the range of 0.025–0.6 g. Fig. 1 shows the effect of amount of adsorbent on the desorption of 4-CP. It was observed that the amount of 4-CP desorption decreased with the increase in amount of adsorbent. After 120 min of sonication, the desorbed amount decreased from 56.80 to 10.25 mg/g for an increase in GAC amount from 0.025 to 0.6 g. This is due to the split in the flux or the concentration gradient between solute concentrations on the adsorbent surface and in the regenerating 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. On the other hand, the concentration of 4-CP in desorbing water increased with increasing the amount of adsorbent. The increase in pollutant concentration was due to the increase of the available sorption surface and availability of more adsorption sites. At higher amount of adsorbent, there is a very fast desorption from the GAC surface that yields a higher concentration of 4-CP in the regenerating water than when the amount of adsorbent is lower.

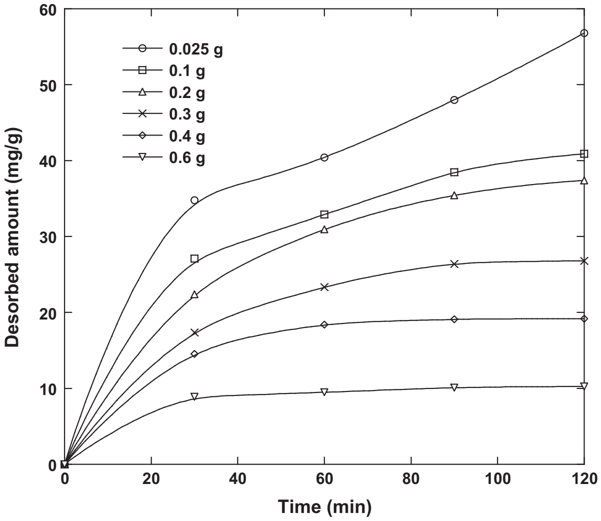

Fig. 1. Effect of amount of adsorbent on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.025–0.6 g, GAC loading: 300 mg/g).

The desorption by ultrasound could be related to the strong turbulence in the medium through different physical phenomena induced by ultrasound and cavitation. When the ultrasonic energy is high enough to overwhelm the bonding energy between the adsorbent and adsorbate, the adsorbed 4-CP molecules would be desorbed. The regeneration of GAC is improved by intensifying mass transfer phenomena through high-speed microjets, high-pressure shock waves, microturbulence, and acoustic vortex microstreaming [18–20,29,30,39–43]. 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 acoustic power

Acoustic power can influence the cavitation threshold, cavitation intensity, and active bubble population, which in turn can affect the cavitation activity [46] and hence the desorption of 4-CP from GAC surface. The effect of acoustic power on the desorption of 4-CP from GAC was examined using an amount of adsorbent of 0.2 g and the obtained results were shown in Fig. 2. The desorption decreased with increasing the acoustic power from 14 to 24 W. The results indicated that at three selected powers, the lower one was more effective. The adsorbed molecules may be released into solution by breaking bonds and intensifying mass transfer phenomena through acoustic vortex

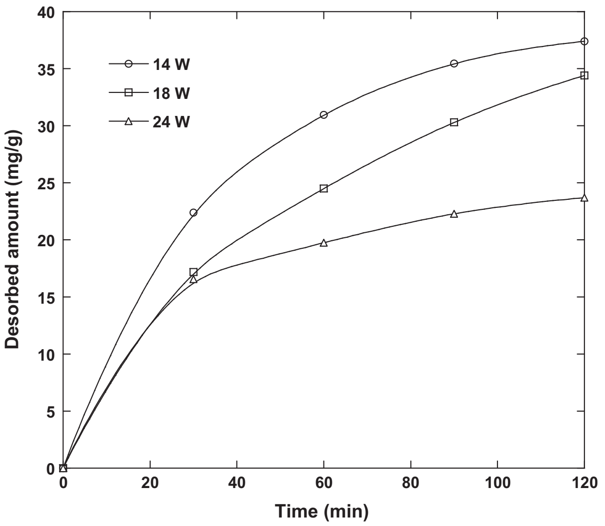

Fig. 2. Effect of acoustic power on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14–24 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

microstreaming, shockwaves, microjets, and thermal effects from cavitational collapse. The effects 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.

3.3. Effect of pulsed ultrasound

Fig. 3 shows ultrasound-mediated desorption of 4-CP under continuous wave and pulsed wave at two different times of exposure: 1 s on/1 s off and 2 s on/1 s off. The higher amount of desorption was obtained using continuous wave ultrasonic irradiation. This might be due to the action of acoustic cavitation, especially its transient cavitation bubbles. In continuous ultrasonic wave, the physical effects of cavitation and ultrasound impinge incessantly the surface of GAC, which weaken the forces between adsorbate and adsorbent, and result in the increase of desorption. Additionally, cavitation activity can be reduced by pulsed mode due to the decrease of the bubble density and the diminishing of the bubble clustering.

3.4. Effect of temperature

Generally, temperature plays a dominant role on nucleation as well as on collapse intensity of cavitation bubbles. The effect of temperature on desorption was studied by carrying out a series of experiments at 15, 25, 35, and 45°C using an acoustic power of 14 W and an amount of GAC of 0.2 g. The obtained results are shown in Fig. 4. It was indicated that the desorption increases with the increasing temperature, indicating that the desorption is an endothermic process. The increase in desorption with the rise of temperature may be due to the weakening of adsorptive forces

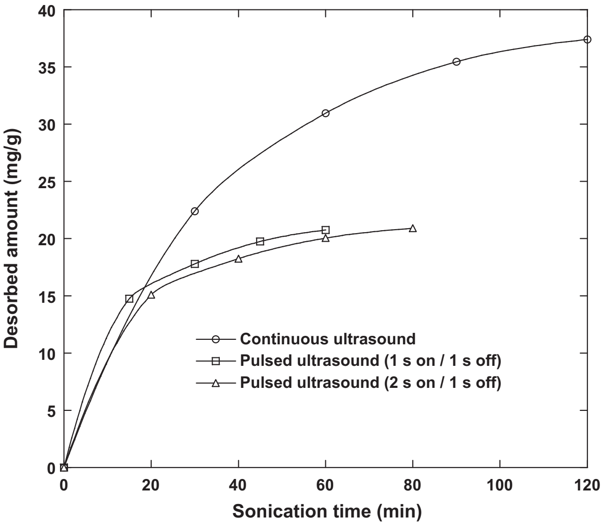

Fig. 3. Effect of pulsed wave on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

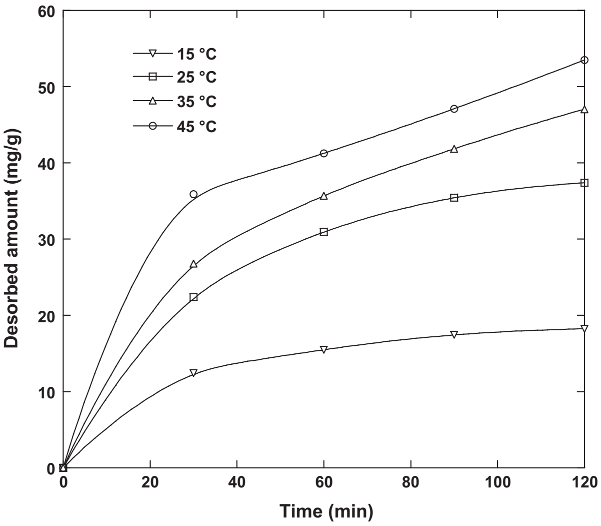

Fig. 4. Effect of temperature on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 15–45°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

between the active sites of the adsorbent and adsorbate species and also between the adjacent molecules of the adsorbed phase. Additionally, this may be a result of increase in the mobility of the 4-CP with increasing temperature. The amounts of 4-CP desorbed were determined as 18.25, 37.40, 47.05, and 53.50 mg/g at 15, 25, 35, and 45°C, respectively. Cavitating bubbles are more easily produced at high temperature because of the decrease of the liquid tensile stress and viscosity [32]. 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 [18–20,29,30,39–43]. The thermal effect is mostly given by localized hot spots formed when bubbles cavitated as well as by piezoelectric transducer heating [30]. Thus, the improvement of desorption with temperature may be due to the increase of the breaking of bonds between adsorbate molecules and the activated carbon surface and/or the increase of the diffusion rate of adsorbate molecules into the pore at higher temperature as diffusion is an endothermic process.

3.5. Multistage regeneration

In multistage regeneration of GAC, the sonicated adsorbent was separated from regenerating water after 120 min of sonolysis. The separated GAC was sonicated in new fresh pure water. Namely, regenerating water was changed five times with new pure water. The desorbed amount of 4-CP vs. sonication time for various stages was shown in Fig. 5. The desorbed amount per stage was initially relatively high, but decreased progressively as the ultrasonic treatment continued. The desorption amount was 37.15 mg/g after one stage of sonolytic treatment. After the sixth stage of ultrasonic treatment, approximately 3.00 mg/g was desorbed. It was likely that the samples after each cycle retain in their porous systems enough 4-CP desorbed as to hinder further desorption in the following cycle, while always in the first cycle, the pores fill with pure water and the driving for desorption of the adsorbate is thus higher. Additionally, the 4-CP molecules adsorbed onto low-energy sites and outer surface of GAC particles were desorbed first, while the more recalcitrant inner layers of adsorbate on the adsorbent surfaces had to be removed in the subsequent stages of treatment. In contrast, the total amount of adsorption increased with increasing the number of stage.

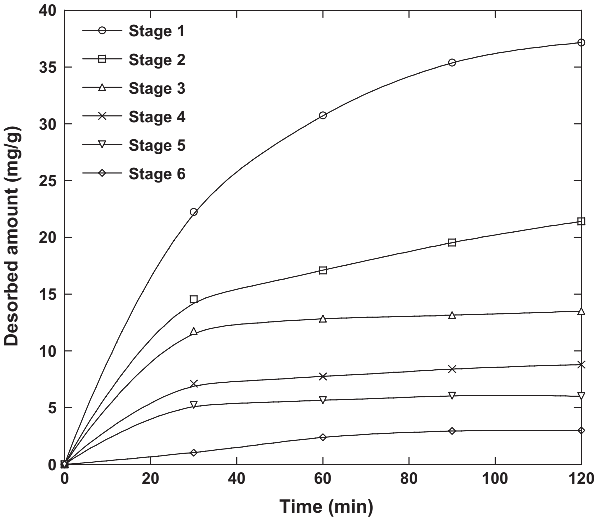

Fig. 5. Multistage ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

3.6. Effect of saturating gases

In order to investigate the influence of the saturating gas on the desorption of 4-CP from GAC, continuous injection of air, argon, or nitrogen at equivalent flow rates was performed during sonication, and the 4-CP concentration was monitored. The control experiment was carried out in the presence of air without any external injection. Fig. 6 shows the desorption kinetics under air, argon, and nitrogen. It can be seen from this figure that the order of desorption amount was as follows: air > control > argon > nitrogen. It seems that the best desorption was obtained in the presence of air. The external inclusion of air contribute to the reduction of cavitation threshold in water. The air dissolved in water ensures an excess of cavitation nuclei and improves the conditions of bubble collapsing [47]. Nevertheless, the desorption in the presence of argon and nitrogen is not consistent with this rule. It appears that the nature of gas used to saturate the water during ultrasonic irradiation plays an important role. Nitrogen-saturated solution had the worst effect on the desorption among the different saturating gases tested. Thus, the regeneration in air-saturated solution would be preferred.

3.7. Effect of salt

To study the effect of salt addition on the desorption of 4-CP from GAC, experiments were conducted in the presence of NaCl at two different concentrations

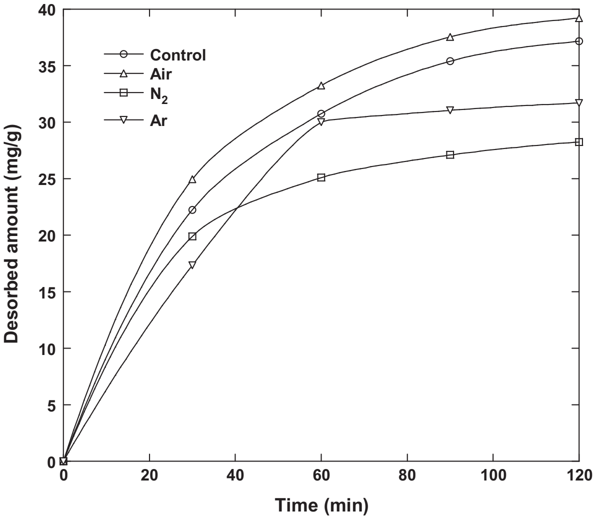

Fig. 6. Effect of saturating gases on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

of 0.1 and 1 M. Fig. 7 presents the effect of NaCl as regenerating solution on the sonolytic desorption. As it can be seen from Fig. 7, desorption in pure water was far higher than those assessed in NaCl solutions. It was also observed that the desorption decreased slightly with increasing NaCl concentration. It seems that excessive amount of NaCl may interfere with the introduction of ultrasound into the regenerating solution and thus decreased the physical effects of ultrasound and cavitation.

3.8. Effect of NaOH

The influence of NaOH as a regenerating solution on the sonolytic desorption of 4-CP from GAC was examined at different sodium hydroxide concentrations in the range of 0.01–1 M. Fig. 8 showed the results of desorption kinetics in sodium hydroxide solution. From this figure, it was observed that the use of NaOH as a regenerating solution drastically enhanced the desorption. It was also noticed that the desorption increased with increasing the NaOH concentration up to 0.1 M and decreased afterward. Hence, it can be concluded that the optimal concentration for NaOH solution was 0.1 M.

The enhancement of desorption in the presence of NaOH 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:

$$\varphi_{\text{ions}} = \frac{1}{1 + 10^{(\text{p}K_\text{a} - \text{pH})}} \tag{1}$$

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

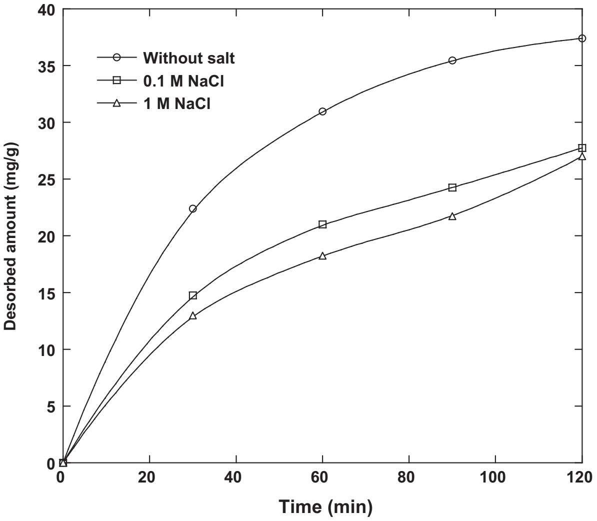

Fig. 7. Effect of salt (NaCl) concentration on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

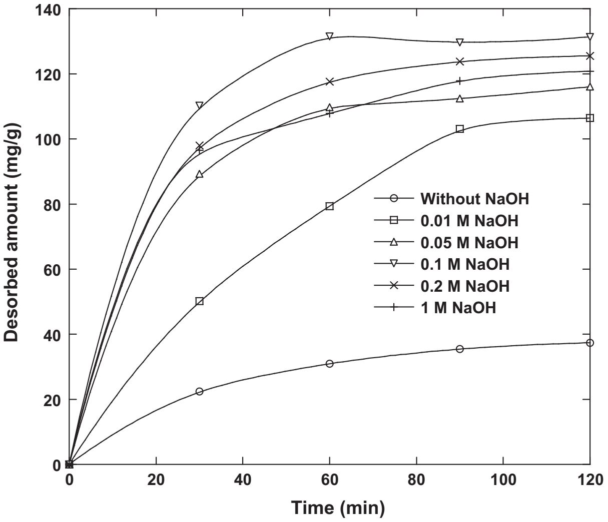

Fig. 8. Effect of NaOH concentration on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

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 [18,19,29,30]. Moreover, NaOH solution can hydrolyze some chemical bonds between 4-CP hydroxyl groups and the surface oxygen groups of the GAC [13,48]. While exhausted GAC was treated with NaOH regenerating solution, 4-CP molecules were easily brought into aqueous phase resulting in an improvement of the desorption of 4-CP from GAC surface.

3.9. Effect of ethyl alcohol concentration

The sonolytic desorption of exhausted GAC was investigated at various ethanol concentrations in the range of 20–70% (v/v). As shown in Fig. 9, it can be observed that the desorption increased with increasing the ethanol percentage in the regenerating 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 [18,19,29,30]. 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 ($HO^{\cdot}$ and $H^{\cdot}$) to form secondary radicals ($C_2H_4OH^{\cdot}$) beneficial for the regeneration of GAC [27].

3.10. Effect of mixtures of NaOH and ethylic alcohol at different concentrations

Mixed regenerants offer the possibility of synergistically combining multiple mechanisms to enhance desorption efficiencies. The ultrasonic regeneration of exhausted GAC was examined using different mixtures of ethanol and NaOH at various concentrations as regenerating solutions. Fig. 10 reported the desorption results obtained in different mixtures of ethanol and sodium hydroxide. The 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 establishment of repulsion forces between activated carbon surface and 4-chlorophenolate anions.

From Fig. 10, it was very important to notice that a mixture of 30% ethyl alcohol and 0.1 M NaOH produces a significant intensification of both the rate and the amount of desorption. It can be concluded that this mixture generates a synergistic effect because the desorption of 4-CP from the activated carbon is greater than the sum of the two separate processes.

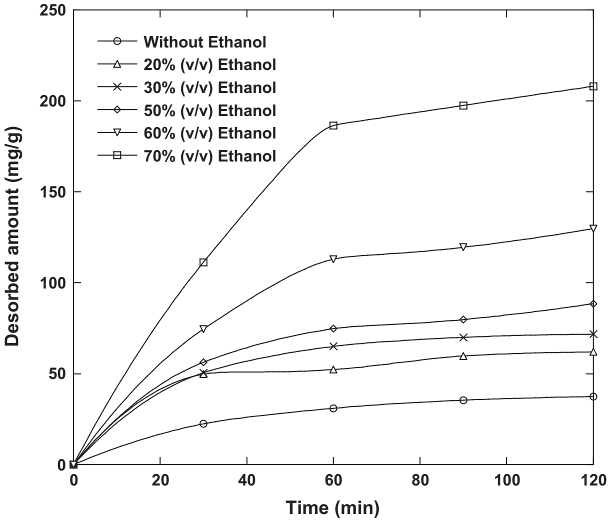

Fig. 9. Effect of ethyl alcohol concentration on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

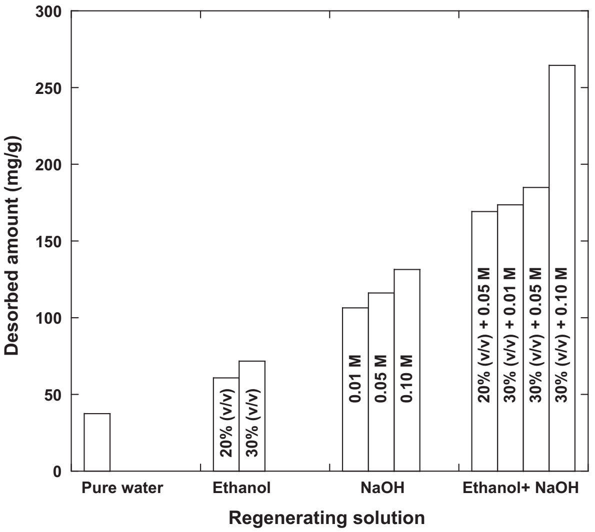

Fig. 10. Effect of mixtures of ethyl alcohol and NaOH at different concentrations on the ultrasonic desorption of 4-CP from GAC (frequency: 20 kHz, acoustic power: 14 W, volume: 100 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g).

4. Conclusion

This study shows that the amount of 4-CP desorption decreased with increasing the amount of adsorbent and acoustic power. Continuous wave ultrasonic irradiation was more effective than pulsed ultrasound. The desorption was enhanced with the rise in temperature from 15 to 45˚C and was reduced in the presence of salt (NaCl). The saturation of the regenerating water with air enhanced the desorption. In the presence of argon and nitrogen, the desorbed amount was reduced. Using NaOH or/and ethyl alcohol as regenerating solutions causes an improvement of the desorption of 4-CP from GAC. The desorption increased with increasing the ethyl alcohol percentage and the concentration of NaOH from 0.01 to 0.1 M and decreased afterward. Using different mixtures of ethanol and NaOH at various concentrations, it has been shown that these regenerating mixtures enhance the desorption of 4-CP. A mixture of 30% ethanol and 0.1 M NaOH produces a synergistic effect and a significant intensification of desorption. The regeneration of exhausted GAC by ultrasound is attributed to the thermal effect and physical forces including mechanical agitation, microjets, shear forces, microstreaming, hot spots, and shockwaves. This study highlighted the versatility of ultrasound-assisted technique for regeneration of GAC exhausted by 4-CP. In order to obtain a better performance of the process, further studies must be carried out in various reactor configurations.

Acknowledgment

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