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Ultrasound-assisted regeneration of granular activated carbon saturated by 4-chlorophenol in batch-loop reactor

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ABSTRACT

The present work reports an investigation for examining the use of low-frequency ultrasound (20 kHz) as an inventive technique for the regeneration of granular activated carbon (GAC) saturated by 4-chlorophenol (4-CP) in batch-loop reactor. The effects of experimental conditions such as the amount of adsorbent (0.05–0.4 g), intensity of ultrasound irradiation (20–35%), temperature (25–55˚C), flow rate (3–30 mL/min) and total volume of regenerating solution (200–300 mL), NaOH (0.01–0.1 N), ethyl alcohol (10–60%) and mixture of NaOH, and ethanol on the regeneration process were examined. The obtained results show that the amount of 4-CP desorption decreased with increasing the amount of adsorbent and intensity of ultrasonic irradiation. The desorption gradually increased with the increase in temperature and flow rate and total volume of regenerating solution. Using NaOH or/and ethyl alcohol as regenerating solutions causes an improvement in the desorption of 4-CP from GAC. The regeneration of spent GAC by ultrasonic irradiation is due to the generation of physical and thermal effects of ultrasound. It can be concluded that desorption by ultrasound can be an alternative technique to classical methods used for the regeneration of exhausted GAC.

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

1. Introduction

Adsorption onto granular activated carbon (GAC) is a well-established technique extensively used in water and wastewater treatment processes due to its high surface area, different pore networks and a wide spectrum of surface functional groups [1–3]. However, depending on the adsorption capacity, GAC will become saturated after some time and the spent activated carbon is considered as a hazardous waste because the contaminants are not removed, but transferred to the solid phase after adsorption. From environmental and economical viewpoints, it is required to investigate the regeneration of activated carbon in order to repeatedly use the adsorbent.

Currently, varieties of regeneration techniques for exhausted spent GAC, especially carbon saturated with phenols, are suggested. The most widely used regeneration techniques include thermal regeneration [4], solvent regeneration [5], direct oxidation and catalytic wet oxidation regeneration [6], and biological regeneration [7]. These regeneration methods suffer

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from severe drawbacks, such as requirement of high energy and time consumption, loss of carbon surface area, destruction of micropores, high cost of operation, requirement of further treatment to destroy or to recuperate the extracted pollutants and low regeneration efficiency, and consequently are limited either technically or economically. To resolve the deficiencies of the above discussed regeneration techniques, new alternative methods that use more advanced technologies are developed. Therefore, the regeneration of activated carbon has been recently investigated using microwaves [8], supercritical water oxidation [9], electric currents [3,10–12], and ultrasound [13–25].

The passage of ultrasound through a liquid induces tiny gas bubbles to oscillate and, under appropriate conditions, undergo a large change in volume and collapse in a near adiabatic process. This is commonly referred to as acoustic cavitation, and the concentration of energy upon collapse results in extreme localized conditions including high temperatures and pressures inside the cavity [26,27]. Ultrasound can create convection in the medium through various physical phenomena mainly attributed to symmetric and asymmetric cavitation such as microstreaming, microturbulence, shock waves, and microjets. During radial motion of the bubble, the fluid near the bubble is set into oscillatory motion, which is called microturbulence [28]. The velocity of the microturbulence varies directly with the amplitude of the oscillations of the bubble. 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. These phenomena increase the rate of mass transfer near the surface and within the pores as well as possibly thinning the film surrounding nearby adsorbent particles [28–32].

The process of regenerating of GAC exhausted by phenolic compounds poses a major challenge because of the high affinity of these compounds to the adsorbent surface. In our previous studies, ultrasound is employed to regenerate GAC saturated by 4-chlorophenol (4-CP) and good performance for GAC regeneration was obtained [13,14,24,25]. However, ultrasonic waves have been utilized in batch mode and GAC regeneration efficiency still require enhancements to be practically viable. The use of batch-loop ultrasonic reactor may be a feasible way to promote the utilization yield of ultrasound. To the best of our knowledge, there is no publication involving batch-loop reactor for the ultrasonic regeneration of GAC. Thus, the purpose of the present work is to investigate the desorption of 4-CP from GAC by low-frequency ultrasound in batch-loop reactor. 4-CP is widely known as an important contaminant of soil and groundwater and designated as priority pollutants by the US Environmental Protection Agency and the European Union. The permissible limit of 4-CP in drinking water supply is 0.5 mg/L [33]. The factors that influence the regeneration process were investigated as well.

2. Experimental

2.1. Batch-loop ultrasonic reactor

A schematic representation of the ultrasonic reactor under a batch-loop mode configuration is shown in Fig. 1. Desorption experiments were performed in a 100 mL cell surrounded by a glass cylindrical jacket, allowing water cooling of the reactor to control the temperature. The regenerating solution was continuously circulated in a closed circuit between the reactor and the alimentation tank. A peristaltic pump (Kerlabo) ensured the circulation. 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% during sonication, except when the effect of power was examined. Acoustic power dissipated in the reactor was measured using standard calorimetric method [34,35].

2.2. Materials

The GAC used in this work was purchased from Sigma–Aldrich. The GAC has a mean granulometry of 1 mm. The GAC had 17.74 Å average pore width.

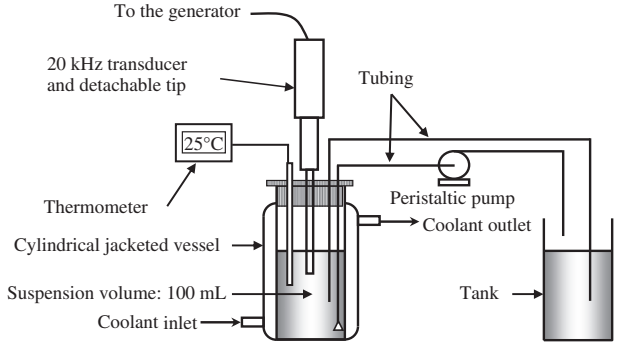

Fig. 1. Scheme of the batch-loop reactor.

According to the classification of IUPAC-pore dimensions, the pores of adsorbents are grouped into micropore ($d < 2$ nm), mesopore ($d = 2$–50 nm), and macropore ($d > 50$ nm). Given that the average pore width is lower than 20 Å, the GAC is microporous. The GAC showed a BET surface area of 1,090 m^2/g. 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.

2.3. Adsorption experiments

For adsorption experiments, the GAC and 4-CP system was placed in a mechanical shaker at 300 rpm and 3 d was allowed to ensure equilibrium was reached before the desorption experiments were performed. After 3 d, the carbon loaded with 4-CP was then separated from the solution using a sieve. Finally, the GAC was dried in an oven at 50˚C for 24 h and stored in a desiccator until use. The concentrations of 4-CP in solution were determined by UV–vis spectroscopy (Lightwave II, UK). The activated carbon loading was determined by mass balance.

2.4. Regeneration of GAC

A definite mass of the prepared spent GAC was introduced into the batch-loop 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 adsorbent

The effect of the amount of adsorbent in the range of 0.05–0.4 g on the desorption of 4-CP from GAC as a function of time is shown in Fig. 2. These experiments were carried out for ultrasound intensity of 20% (acoustic power of 14 W), temperature of 25˚C, GAC loading of 300 mg/g, regenerating water total volume of 200 mL, and flow rate of 30 mL/min. From Fig. 2, it was observed that 4-CP desorption was decreased by increasing the amount of adsorbent. The decrease in the desorption of 4-CP with increasing the amount of adsorbent 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. In contrast, 4-CP concentration in regenerating water (aqueous solution) increased with increasing the amount of GAC. The increase in 4-CP concentration in desorbing water was due to the increase in the available adsorbent surface and availability of more adsorbed molecules. 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. Control experiments were investigated in order to quantify the desorption as a function of time in the absence of ultrasound by simple mechanical stirring. The obtained results showed that without ultrasound, no desorption was noticed after 120 min of contact time.

The regeneration of GAC by sonication could be related to the strong convection in the medium through different physical phenomena induced by ultrasound and cavitation. The regeneration of GAC is enhanced by breaking bonds between 4-CP and adsorbent surface and intensifying mass transfer phenomena through high-speed microjets, high-pressure shock waves, microturbulence, and acoustic vortex microstreaming [13–15,24–32]. When these physical phenomena hit a solid object, they tear off all molecules adsorbed onto it. Additionally, they enhance the

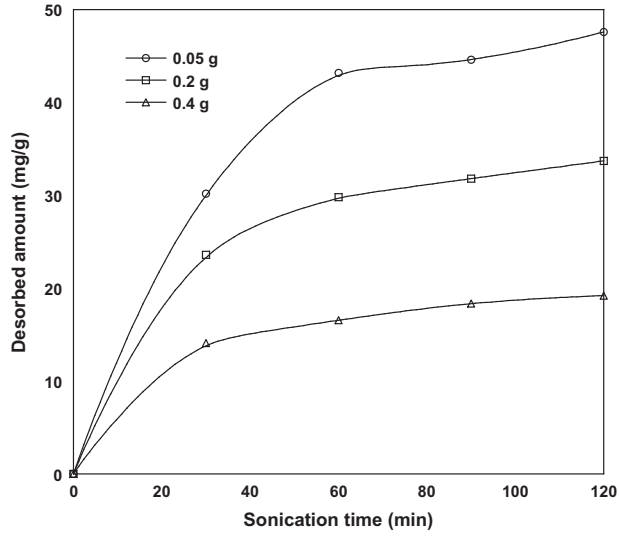

Fig. 2. Effect of adsorbent mass on the desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 25˚C, adsorbent mass: 0.05–0.4 g, GAC loading: 300 mg/g, regenerating water flow rate: 30 mL/min).

mass and heat transfer at interfacial films surrounding nearby adsorbent particles and within the pores.

Physical phenomena produced by the ultrasound and cavitation can influence the morphology and size distribution of adsorbent particles through the cavitation process. Ultrasound is only useful if it does not destroy the adsorbent in order to propose a reasonable process alternative to existing regeneration techniques. Hence, the size distribution of the GAC was carried out before and after 120 min of sonication. The mean granulometry of GAC particles has only shifted to 3% and thus the intensity threshold for GAC particle breakup had not been attained. A scanning electron microscope was used to look at the effect of ultrasound on the morphology of the activated carbon surface. It can be seen from the micrographs (Fig. 3) that the activated carbon showed similar surface morphologies before and after sonication. Additionally, sonication had no significant effect on BET surface area and pore diameter of the GAC.

3.2. Effect of intensity of ultrasound irradiation

In this study, the effect of intensity of ultrasound irradiation on desorption kinetics of 4-CP from GAC was studied at 300 mg/g adsorbent loading and GAC mass of 0.2 g. The used total volume of regenerating water and temperature was 200 mL and 25˚C, respectively. The effect of intensity on the amount of 4-CP desorption was shown in Fig. 4. The desorbed amount decreased with increasing the intensity from 20 to 35%. The results indicated that at three selected intensities, the lower one was more effective. The influence of intensity on the regeneration process is highly dependent on the examined substance as the heat of adsorption determines how much energy is required by a cavitation event to overcome the affinity between adsorbed molecules and adsorbent. Consequently, there is a great potential for process optimization in changing the frequency and intensity in order to preserve GAC properties and maximize the phenomena induced by ultrasound. Therefore, intensity of ultrasound is an important factor for the rate of 4-CP desorption from GAC surface.

3.3. Effect of temperature

When an ultrasonic wave traverses a medium, part of the energy that it transports is dissipated in the form of heat. The movements due to the passage of ultrasound engender frictions that induce a heating of the liquid because of the solution viscosity. We will make profitable this property to study the influence of temperature on desorption in the presence of ultrasonic irradiation. From the thermodynamics point of view, since adsorption is an exothermic process, higher temperature is unfavorable for adsorption and thus helps desorption. Fig. 5 presents the desorption of 4-CP as a function of time at different temperatures in the range of 15–45˚C. It was observed that the amount of 4-CP desorbed increased with rising temperature, indicating endothermic desorption process. 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. In addition, cavitating bubbles are more easily produced at high temperature because of the decrease in the liquid tensile stress and viscosity [27]. Desorption is promoted if such bubble collapse occurs in the vicinity of the adsorbent surface. This improvement of desorption is attributed to the non-thermal and thermal effects of

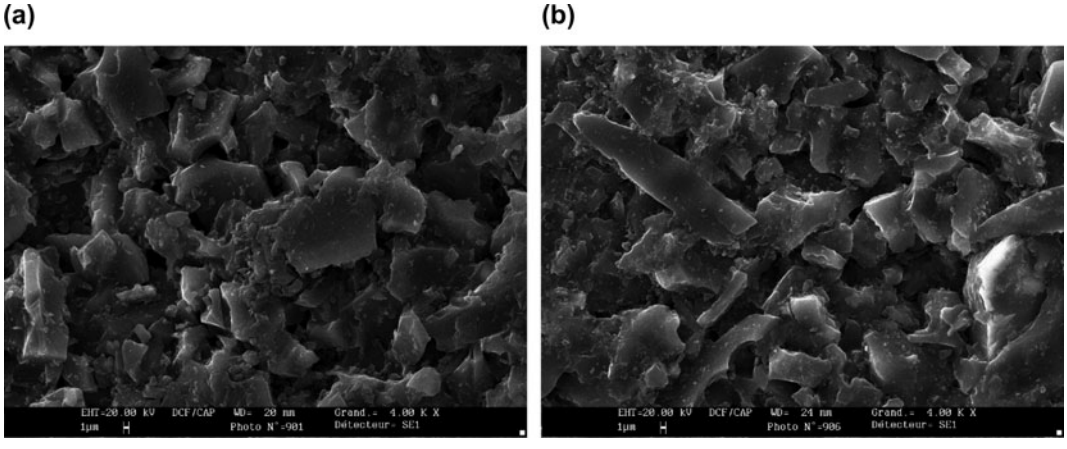

Fig. 3. SEM micrographs of GAC surface (4,000×): (a) before sonication and (b) after 120 min of sonication.

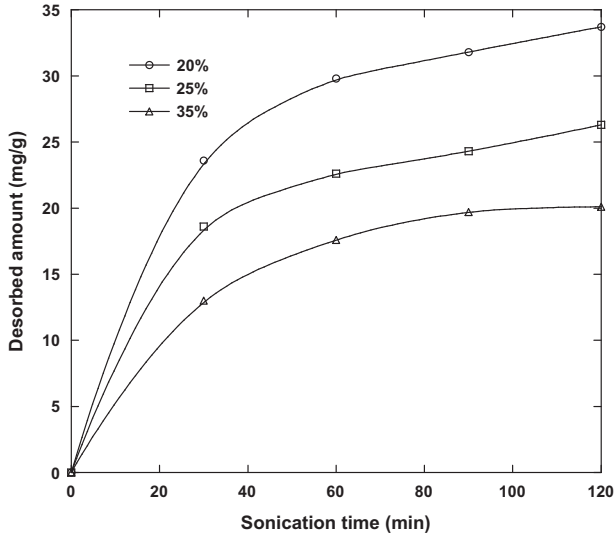

Fig. 4. Effect of ultrasonic power on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, Intensity of ultrasound: 20–35%, total volume: 200 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 30 mL/min).

ultrasound. The non-thermal effect is mostly produced by the acoustic vortex microstreaming and by the high-speed microjets and high-pressure shockwaves induced by acoustic cavitation [24,36]. The thermal effect is mostly given by localized hot spots formed when bubbles cavitated as well as by piezoelectric transducer heating [25]. Thus, the observed temperature-dependent desorption may be due to the increase in the breaking of bonds between 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.

3.4. Effect of flow rate

The effect of the flow rate of regenerating water was examined by subjecting the exhausted GAC to five different flow rates between 3 and 30 mL/min at 25˚C. The obtained results are shown in Fig. 6. It was observed that the amount of 4-CP desorbed increased with increasing the flow rate. The increase in flow rate in batch-loop reactor promotes desorption because the concentration gradient between the regenerating water and the surface of the adsorbent increased with increasing flow rate due to the dilution of the medium. Therefore, the increase in flow rate provides high concentration gradient and thus promotes the desorption. Ultrasound can work in a hydrodynamical way in addition to its simultaneous thermal functions. Sonication generates high-pressure shockwaves, high-speed microjets and microstream vortexes using the solvent media in the pores of GAC, which washes out additional adsorbed compounds and frees the pores [13–15,24–32]. Accordingly, all the regeneration

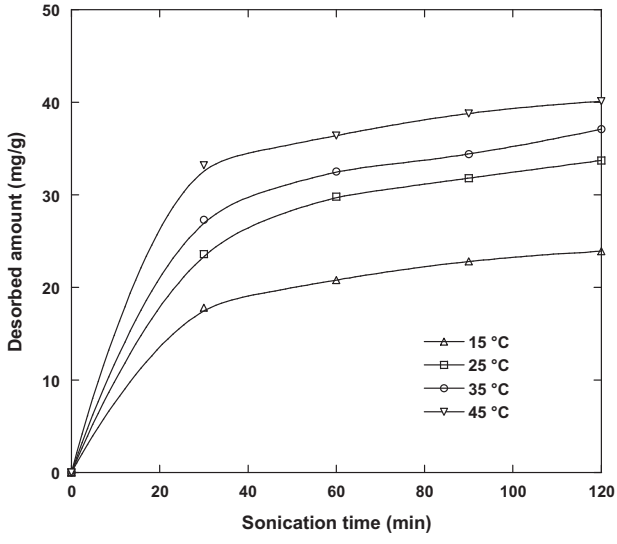

Fig. 5. Effect of temperature on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 15–45˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 30 mL/min).

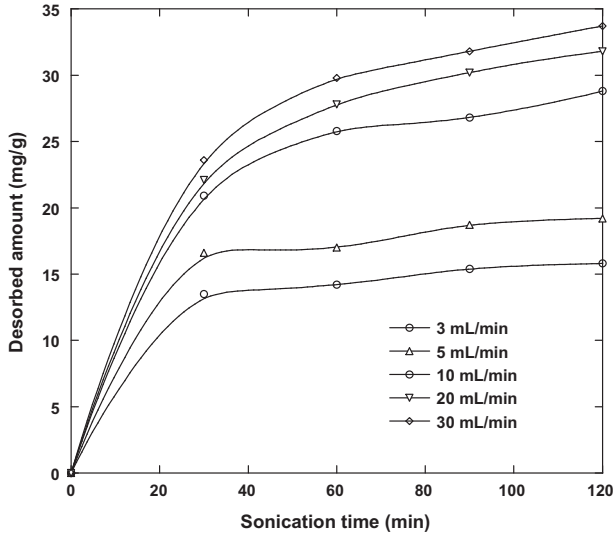

Fig. 6. Effect of regenerating water flow rate on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 3–30 mL/min).

experiments reported in this work were performed with a flow rate of 30 mL/min, thus guaranteeing best results.

3.5. Effect of volume

In order to investigate the effect of the total volume of regenerating water used in batch-loop reactor on the regeneration of GAC exhausted by 4-CP, experiments were conducted using two volumes: 200 and 300 mL. The obtained results are shown in Fig. 7. The increase in the total volume of the regenerating water induces an improvement of the amount of desorption and decreased the concentration of 4-CP desorbed in the reactor. The decrease in the concentration of 4-CP is related to the dilution effect due to the increased volume of the regenerating water. The increase in desorption with increasing the total volume of regenerating water is due to the gradient between solute concentrations on the GAC surface and in the desorbing water.

3.6. Effect of NaOH

To examine the effect of NaOH as a desorbing solution on the regeneration of GAC, experiments were carried out at three different NaOH concentrations (0.01, 0.05, and 0.1 M), using a total volume of 200 mL, intensity of ultrasound of 20%, temperature of 25˚C, adsorbent mass of 0.2 g, GAC loading of 300 mg/g, and flow rate of regenerating solution of 30 mL/min. The effect of NaOH concentration on the desorption of 4-CP from GAC is shown in Fig. 8. From this figure, it was observed that the rate and amount of desorption increased with increasing sodium hydroxide concentration from 0.01 to 0.05 M and decreased afterward. Therefore, it can be concluded that the optimal concentration for NaOH solution was 0.05 M. At basic conditions, 4-CP (pKa = 9.20) is predominantly in the form of chlorophenolate ions and its solubility in water is higher than that of the neutral 4-CP molecule. 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 [13,14,24]. Additionally, NaOH solution can hydrolyze some chemical bonds between 4-CP hydroxyl groups and the surface oxygen groups of the GAC [9,37]. Once spent GAC was treated with NaOH regenerating solution, 4-CP molecules were easily brought into aqueous phase resulting in an improvement of both the rate and amount of desorption of 4-CP from GAC surface.

3.7. Effect of ethyl alcohol

In order to investigate the effect of ethyl alcohol as a regenerating solution on the desorption of 4-CP from

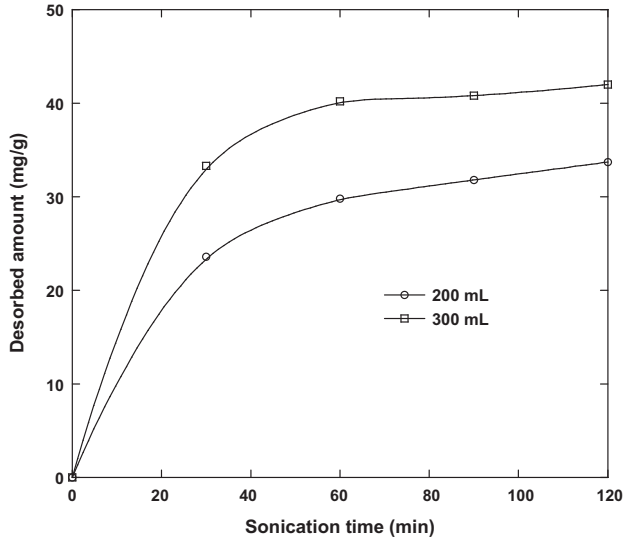

Fig. 7. Effect of regenerating water total volume on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200–300 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating water flow rate: 30 mL/min).

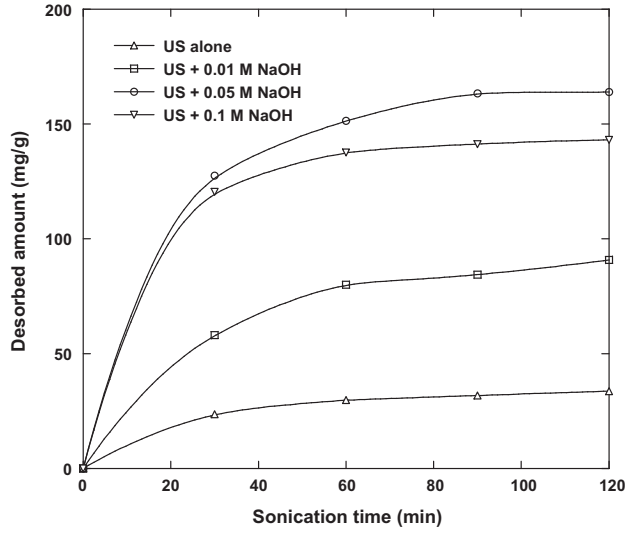

Fig. 8. Effect of NaOH concentration on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 25˚C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 30 mL/min).

GAC, experiments were realized using alcohol concentrations in the range of 10–60% (v/v). The obtained results are presented in Fig. 9. It was observed that the desorption efficiency increased with increasing the ethanol percentage in the regenerating solution. Ethanol decreases the tensile stress of the liquid and thus reduces the cavitation threshold and facilitates the generation of cavitating bubbles [13,14,24]. The production of more transient cavitation bubbles helps to easily produce physical and thermal effects. Additionally, 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 [38].

3.8. Effect of NaOH and ethylic alcohol

Using a regenerating solution containing 30% (v/v) ethanol and 0.05 M NaOH, the concentration of 4-CP in the solution desorbing the activated carbon was monitored with time. The results of the desorption experiments are shown in Fig. 10. Experimental results show that a mixture of ethanol and NaOH enhances the kinetics of desorption. The improvement of desorption rate and amount using a mixture of ethanol and NaOH in the presence of ultrasonic irradiation can be explained by the lowering of cavitation threshold and the creation of repulsion forces between activated carbon surface and chlorophenolate anions.

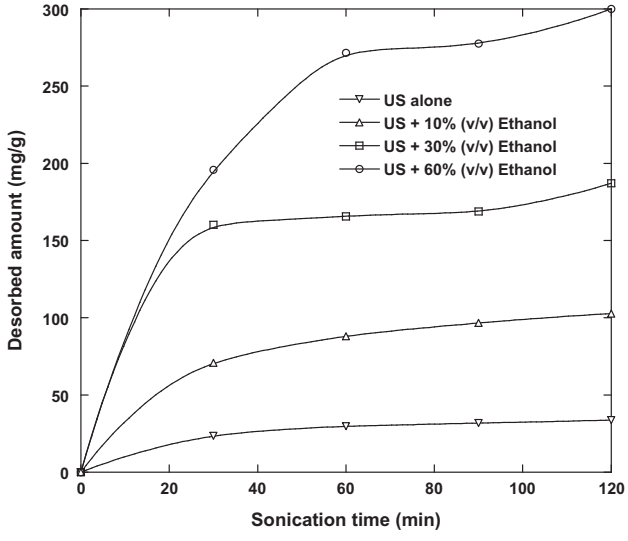

Fig. 9. Effect of ethanol concentration on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 30 mL/min).

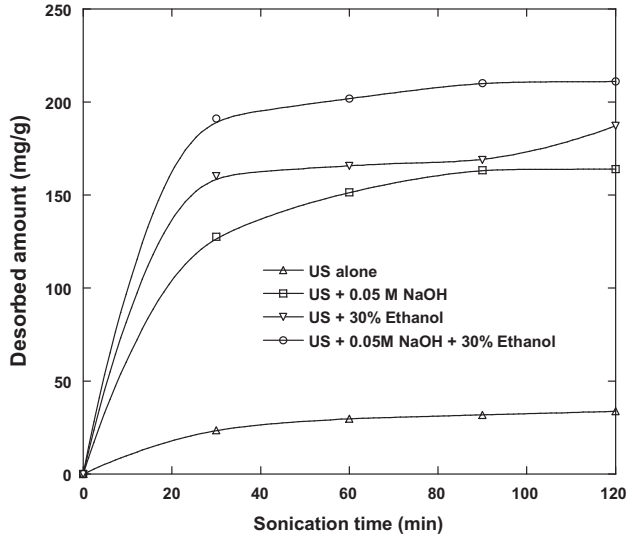

Fig. 10. Effect of a mixture of ethanol (30%, v/v) and NaOH (0.05 M) on desorption kinetics of 4-CP from GAC (frequency: 20 kHz, intensity of ultrasound: 20%, total volume: 200 mL, temperature: 25°C, adsorbent mass: 0.2 g, GAC loading: 300 mg/g, regenerating solution flow rate: 30 mL/min).

3.9. Variation of adsorption uptake with the regeneration cycles

The variation of adsorption uptake as a function of regeneration cycles in the five adsorption-regeneration experiments, together with the error bars representing

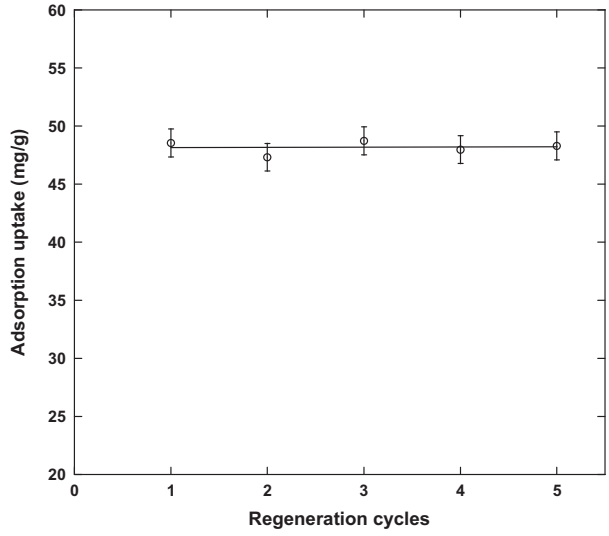

Fig. 11. The variation of adsorption uptake as a function of regeneration cycles (volume: 100 mL, adsorbent mass: 0.2 g, 4-CP initial concentration: 100 mg/L, stirring speed: 300 rpm, temperature: 25°C).

the deviation errors for the replicates data was shown in Fig. 11. From this figure, it appears that the adsorptive uptake of the regenerated activated carbon do not differ significantly with the virgin carbon, even after several adsorption-regeneration cycles.

4. Conclusion

This work shows that the ultrasonic desorption of 4-CP from GAC decreased with increasing the amount of adsorbent and intensity of ultrasound. The amount of 4-CP desorbed progressively increased with the increase in temperature and flow rate and total volume of desorbing solution. The desorption of 4-CP from GAC was drastically enhanced when NaOH or/and ethyl alcohol were used as regenerating solutions. The regeneration of exhausted spent GAC by ultrasound is attributed to both the thermal and non-thermal effects induced by ultrasonic irradiation. This work is fundamental to designing an alternative method to improve GAC regeneration. From a practical viewpoint, regeneration of GAC by ultrasound reported herein is a promising technique. In order to obtain a better performance of the process, further studies must be carried out in continuous flow reactor.

Acknowledgment

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