

Effect of microwave heating on the regeneration of modified activated carbons saturated with phenol

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Abstract The purpose of this work was to investigate the effect of microwave irradiation on the regeneration of modified activated carbons (GAC/MW, GAC/Ni, and GAC/Cu). The untreated activated carbon (GAC pure) was used for blank experiment. Microwave heating was used for preparation and regeneration of the modified activated carbons. The effect of loading Ni^{2+} and Cu^{2+} ions on the activated carbon adsorption capacity was investigated. The results showed that the activated carbon loaded with Ni^{2+} has no significant effect on phenol adsorption, while the adsorption capacity of activated carbon loaded with Cu^{2+} significantly decreased. Microwave irradiation showed a positive effect on activated carbon adsorption capacity. Under optimal conditions, the results showed that there was no effect when changing temperature and pH. The effect of ions Ni^{2+} and Cu^{2+} loaded into activated carbon were also investigated. During the regeneration process, the activated carbon loaded with Ni^{2+} showed a strong microwave energy adsorption than the activated carbon loaded with Cu^{2+} . The effect increasing Ni^{2+} quantity decreases the activated carbon regeneration efficiency. During the regeneration of activated carbons, the highest temperature was observed in the cases of GAC/Ni. During regeneration, the temperature increases when the quantity Ni^{2+} loaded increases. The regeneration efficiency of activated carbons reaches 98 % even after 10 times of regeneration cycles. After several regenerations, MW/GAC and GAC/Ni regeneration efficiency was high, while regeneration

efficiency of GAC/Cu decreased considerably. GAC regeneration efficiency also decreased several cycles. During regeneration process, phenol was simply desorbed from activated carbons under microwave irradiation.

Keywords Phenol · Microwave energy · Modified activated carbons · Microwave regeneration · Microwave irradiation

Introduction

The discharge of wastewater represents one of the major industrial pollutions (Nabarlatz et al. 2012). Among the hazardous organic wastes produced by the industries, phenolic compounds are the most common organic compound produced (Bo et al. 2008). Phenols are dangerous organic pollutants because they are toxic even at low concentrations (Ahmaruzzaman and Sharma 2005). These phenolic compounds usually have a low threshold in potable waters and also have a detrimental effect on human health at high level (Hui et al. 2011). One of the most important and more efficient techniques used to adsorb or retain the contamination (Manh et al. 2011) is adsorption. Among the materials commonly used as adsorbent, activated carbons is one of the most used materials (Lei et al. 2011) due to their high pore. Adsorption by activated carbons (AC) is a well-established technology widely used in wastewater purification (Bandosz 2006; Campbell et al. 2011).

Regeneration is time and energy consuming and very often there is a significant deterioration of the adsorbent's pore structure (Lu et al. 2011), thereby reducing the final adsorption capacity and the efficiency of the regeneration (Ania et al. 2004). In recent years, microwave (MW)

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irradiation has attracted attention as a tool for regeneration due to its capability of molecular-level heating. Interesting reports have appeared on the application of MW-heating technology for regenerating AC (Price and Schmidt 1997; Bradshaw et al. 1997; Coss and Cha 2000; Liu et al. 2004; Jones et al. 2002; Deming et al. 2011; Guido et al. 2008; Bermudez et al. 2012) with very promising results. Applications of microwave regeneration technologies are largely used in industries for economical purposes (Remya and Lin 2011). Microwave regeneration offers potential advantages over conventional treatment (Wang et al. 2012) and catalytic reactions (Huan et al. 2010). This technology can also be used for catalyst preparation (Xinliang et al. 2012). Researches on regeneration of the activated carbon loaded with metal are less reported in literature. To the best of our knowledge, loading metal on activated carbon decreases activated carbon adsorption capacity (Ania et al. 2007). In this work, microwave irradiation has a positive effect on modified activated carbon regeneration increasing their adsorption capacity after cycles of regenerations (Yuen and Hameed 2009). These investigations are done to evaluate the effect of microwave regeneration on the activated carbons loaded with metals. The effect of microwave irradiation may have many effects on the modified activated carbons. On the other hand, the effect of the nature and quantity of metal loaded upon activated carbons decreases activated carbon regeneration efficiency. So far interaction may occur between the metal and the activated carbon.

Experimental section

Reagents and instruments

A charcoal granulated activated carbon (GAC) with particle size of 0.1–1.1 mm, Cl (%) = 0.025 and Pb (%) = 0.005, Fe (%) = 0.02. pH (50 g/l, 25°C) = 5.0–7.0, Zn (%) = 0.05 was purchased from Tianjin Weichen chemical reagent. Phenol solid from Shenyang chemical industry 0.1 g was diluted in distilled water to make a 1,000 ppm solution and diluted to obtain the solution at different concentrations. Cu (NO₃), H₂O and Ni (NO₃), H₂O were purchased from Tianjin chemical industry China. The electronic scale balance (JJ 1,000, Max. 1,000 g. High-performance liquid Chromatography (HPLC) (LC-10UV, equipped with UV detector (UV-1575) and C18 reverse-phase column (250 × 4.6 mm, 5_μm ODS, China) was used for the analyses. Magnetic agitator was used to stir the phenol solution and also to set the temperature of the solutions. pH meter PHs-3C (Shang Hai, China) was used for pH measures.

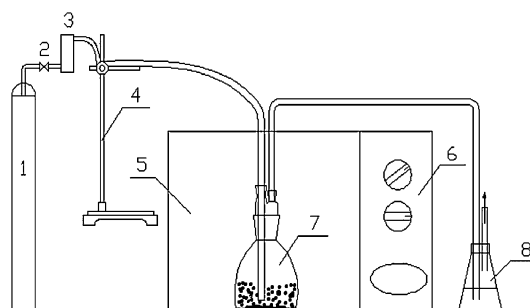


Fig. 1 Schematic of the apparatus used for the preparation and regeneration of the modified activated carbons 1 nitrogen gas container, 2 bottle opener, 3 flow pressure meter, 4 support, 5 microwave oven, 6 timer, 7 conical quartz glass, 8 water container glass

Experimental apparatus of the modified domestic microwave oven

The experimental device is a modified domestic microwave oven which operates at 2,540 MHz. The microwave oven was modified such as to introduce nitrogen into the conical quartz glass. Nitrogen gas bottle was connected with the flow pressure meter (Fig. 1). The conical quartz glass was used as reactor for each experiments. The modified activated carbons prepared were cooled at room temperature under the protection of nitrogen gas and were removed from the microwave oven to be used for the next experiments.

Preparation of the modified activated carbons

The impregnation was done by microwave calcination. The solutions were prepared as follow: 3.78 g of Cu (NO₃), H₂O, 5.01 g of Ni (NO₃), H₂O were introduced into two different conical flask, 50 ml of deionized water was added to make copper nitrate and nickel nitrate solutions, then 20 g of granular activated carbons (GAC) was introduced into the solutions. Then, the mixture was introduced in the machine for rotation at the speed of 232 rev/min during 2.5 h. The weight percentages of metals are, Cu:GAC = 1:20 and Ni:GAC = 1:20. The mixed solutions were introduced in the quartz conical flask reactor separately and placed in the microwave oven. The microwave irradiation power was 400 W and the solutions were treated under the same conditions: intermittently irradiated (for 1 min 46 s, 2 min, and 50 s) until nearly dryness. The Nitrogen gas was introduced at the pressure flow of 0.4 m³/h during 2 min before irradiation and was kept 5 min during the irradiation time at 400 W.

Saturation of the activated carbons

In each experiment, 2 g of activated carbons was used in 250 ml conical flash glass and 200 ml of phenol solution was introduced in the glass and stirred for 2 h until the activated carbons reached the adsorption equilibrium. 0.05 ml was taken each time to be analyzed. The concentration and removal efficiency of phenol were calculated following the Eqs. (1) and (2).

$$c = c_o \times \frac{A}{A_o} \quad (1)$$

$$q = 100 \% \times \left(1 - \frac{c}{c_o} \right) \quad (2)$$

C and C_o are, respectively, phenol solution concentration and phenol initial concentration (mg/l). A and A_o are, respectively, peak surface of phenol solution and peak surface of phenol initial solution ($\mu V \times s$). q is the percentage of phenol removal by the activated carbon (%).

Regeneration of modified activated carbons

Experiments were conducted in the conical quartz glass of 250 ml (Fig. 1). The experimental apparatus used to regenerate the activated carbons is shown in the figure (Fig. 1). After regeneration, the activated carbons were according to the optimized adsorption conditions. Under these conditions, the regeneration of activated carbons was done several times from 1 to 10 cycles of regeneration after several adsorptions. The regeneration of the activated carbons was done under a flow of nitrogen gas (Fig. 1) to avoid destruction of activated carbons. In our previous studies, we investigated the optimal condition for microwave regeneration of the modified activated carbon (Brim et al. 2013). The activated carbons were regenerated according to the optimized conditions.

Regeneration conditions

Microwave irradiation power and time have been optimized. After a series of regeneration experiments, MW power of 700 W and 3 min of regeneration time were selected as the optimized conditions. Under nitrogen atmosphere pressure, flow N_2 flow is $0.4 \text{ m}^3/\text{h}$. The gas was first introduced for 1 min before irradiation and during 3 min of irradiation time.

Analytical methods

Phenol was analyzed by a high-performance liquid, the detection wavelength was set at 270 nm, and the mobile

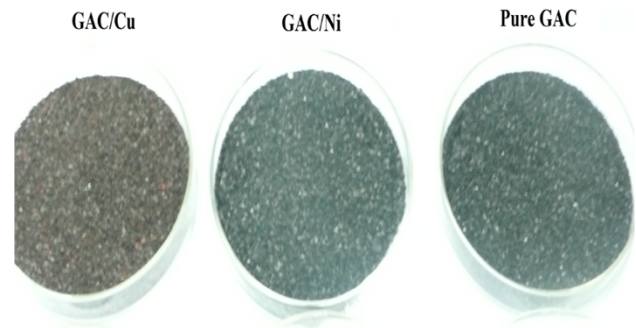


Fig. 2 Photograph of the microwave prepared modified activated carbons

phase was 40:60 (v/v) acetonitrile and water at a flow rate of 0.8 ml min^{-1} flow rate. 0.05 ml was taken each time to be analyzed. And the injection volume was 20 μl .

Results and discussion

Photograph-modified activated carbons

The photograph of the as-prepared modified activated carbon is represented. As we can see in the figure below (Fig. 2), the different activated carbons are presented with different color.

The activated carbon loaded with nickel as shown in the middle of this picture is colored in blue (Fig. 2). But the activated carbon loaded with copper is represented in a red brown color and as to make the comparison the activated carbon simply treated with microwave irradiation is colored in the black with the same color as that of the untreated activated carbon. These results shows that the color in the activated carbons loaded with copper and nickel are not the consequence of microwave irradiation but by the fact of loading metal in the activated carbon micro pores and each color, respectively, represents the nature of metal loaded inside of the activated carbon micro pores. The as-prepared colored activated carbons were directly used as they appear for next experiences.

Influence of regeneration parameters on activated carbon regeneration efficiency

Influence of regeneration time

Regeneration of activated carbons is a very crucial problem (Ania et al. 2004). Many investigations have been done to optimize the conditions for high regeneration efficiency of activated carbons. In this study, optimal condition for microwave regeneration of different types of modified activated carbons was investigated. GAC/Ni and GAC/Cu

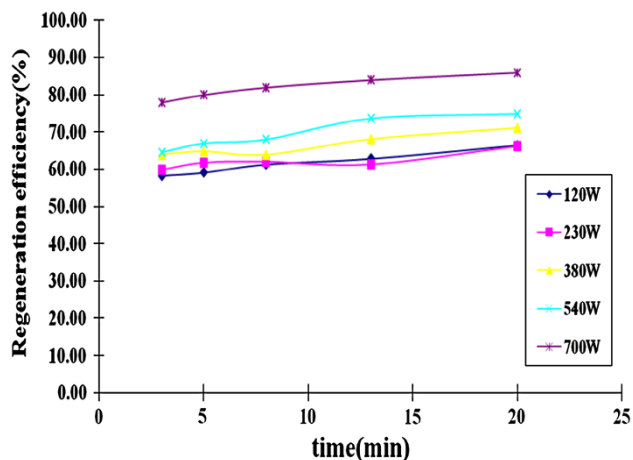


Fig. 3 Influence of time on the AC regeneration efficiency

were chosen as the modified activated carbon. (Fig. 2). The regeneration efficiency depends relatively on the microwave irradiation time (Fig. 3). The highest regeneration efficiency is obtained when obviously the irradiation power was set at 700 W. At lowest microwave irradiation power, there is not significant increase of regeneration efficiency (Fig. 3). The selected time was, respectively, 3, 5, 8, 13 and 20 min. The activated carbon regeneration efficiency increases with the irradiation time (Fig. 3).

Influence of microwave regeneration power

As it can be seen in the figure below, when the microwave irradiation power is high, activated carbon regeneration efficiency is also high (Fig. 4). Activated carbon removal efficiency reaches 80–90 % at the maximum microwave irradiation of 700 W. The more activated carbons is exposed

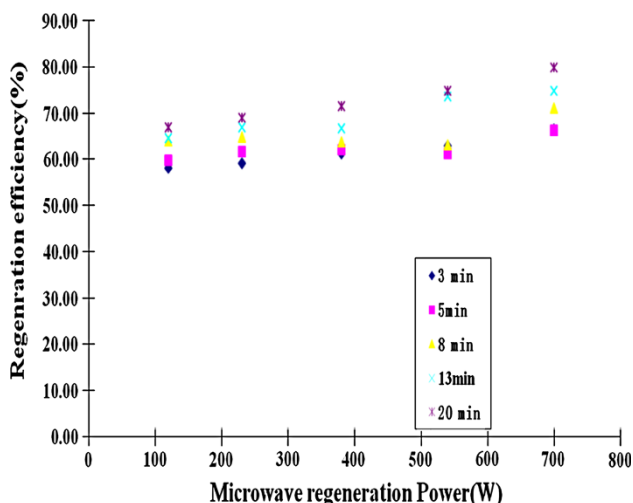


Fig. 4 Influence of microwave power on the AC regeneration efficiency

under MW irradiation, the high is the regeneration efficiency (Fig. 4). The highest regeneration efficiency was obtained at the microwave irradiation power of 700 W and 20 min of irradiation time. From 3 to 20 min, the difference of regeneration efficiency is not significant (Fig. 4). This slight difference may be explained by the fact that at 5 min, phenol compounds adsorbed by activated carbons were almost desorbed. For simple reasons of energy consumption, the time of regeneration was 3 min and the microwave irradiation power was set at 700 W for next experience unless otherwise mentioned. Three minutes and 700 W of microwave power were selected as the optimized regeneration conditions of the modified activated carbon regeneration. MW power was thus seated at 700 W and the regeneration time was 3 min.

Comparison of the effect of loading Ni²⁺ and Cu²⁺ on the Activated Carbons regeneration efficiency

The regeneration efficiency of GAC/Ni and GAC/Cu was investigated under similar conditions. The results were analyzed in comparison with the untreated activated carbon (pure GAC). The activated carbon impregnated with nickel presented the highest regeneration efficiency (Fig. 5). The result remains identical from 5 to 20 min. This result shows that the regeneration efficiency of GAC/Ni is higher than GAC/Cu. The effect of microwave heating on the regeneration efficiency of GAC/Ni is more efficient than GAC/Cu. This result can be explained by the fact that Ni adsorbs more microwave irradiation than Cu. The difference of regeneration efficiency of GAC/Ni and GAC/Cu is very significant. Microwave energy has a positive effect on the activated carbon impregnated with nickel than the activated carbon impregnated with copper (Fig. 5).

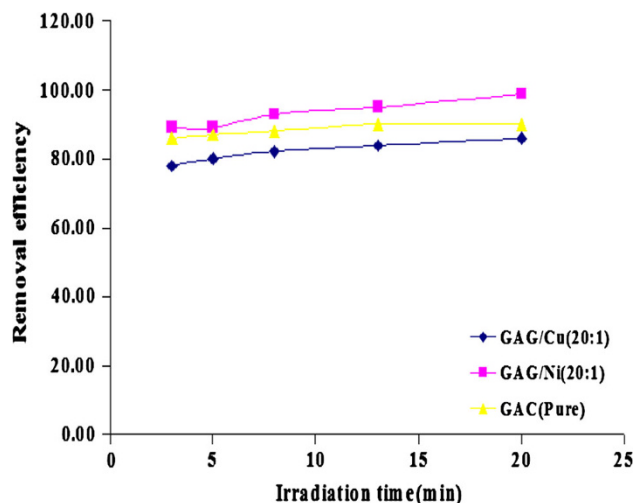


Fig. 5 Comparison of the effect loading Ni and Cu on the regeneration efficiency

Evolution of activated carbon temperature upon regeneration cycles

Temperature is a very important parameter in the regeneration of activated carbons (Ania et al. 2004). The evolution of temperature was controlled after each cycle of regeneration. Temperature plays an important role on activated carbon regeneration efficiency (J.M. et al. 2011). Carbons are used for dyes adsorption. The previous results showed that regeneration efficiency increases with the activated carbon temperature (Calis and Bermudez 2012). The activated carbons loading with nickel has the highest regeneration efficiency. The modified activated carbon (GAC/Ni) seems to absorb microwave energy better than GAC/Cu and this adsorption of microwave energy is the principal cause of elevation temperature in the GAC/Ni system. The temperature of activated carbons can reach 400° in the case of GAC/Ni, while it is below 400° in the case of GAC and GAC/Cu (Fig. 5). GAC/Cu adsorbs less microwave energy. These results can explain the low temperature observed in the activated carbon loaded with Cu^{2+} while activated carbons were regenerated (Fig. 6). The regeneration temperature of GAC/Cu is around 300°, while the regeneration temperature of GAC/Ni is around 450° (Fig. 6). These results show that nickel can absorb microwave energy more than copper while the activated carbons are being regenerated. On the other hand, GAC treated with microwave energy absorbs better microwave energy than the as-received GAC. The activated carbon pretreated with microwave energy (MW/GAC) seems to be able to absorb more microwave irradiation than the as-received activated carbon (GAC) (Fig. 6). The temperature of the modified activated carbons decreases with the number of regeneration cycle. When the modified activated carbons are regenerated, their temperature decreases

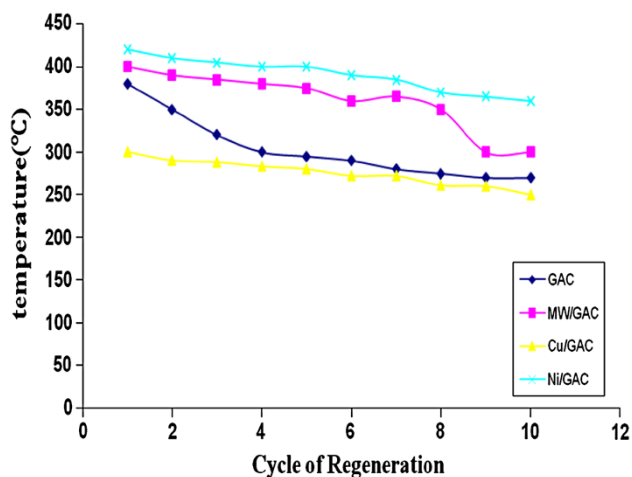


Fig. 6 Evolutions of activated carbons temperature function of number regeneration cycle

slightly (Fig. 6). More the activated carbons regeneration time is extended, less the activated carbons temperature is high.

Comparison of regeneration efficiency of activated carbons after several cycles

The different activated carbons could be regenerated with the percentage of regeneration reaching 94 % after 10 cycles of regeneration (Fig. 7). The microwave pretreated GAC and GAC/Ni have high adsorption capacity before the activated carbons are regenerated. After ten regeneration cycles the adsorption capacity of GAC/Cu is very low comparatively to GAC/Ni as (Fig. 7). Compared to GAC and GAC/Ni, the regeneration efficiency of GAC/Cu was very low (Fig. 7). The regeneration efficiency of activated carbon untreated GAC remained almost constant after several cycle of regeneration as shown in Fig. 6. This effect could be attributed to the action of microwave irradiation that acts to the GAC pore volume and pore size to increase the volume pore of the activated carbons (Bandosz 2006). Microwave irradiation has the effect on the activated carbon surface chemistry, thus increasing the activated carbon adsorption capacity. Obviously, when metal is uploaded into the activated carbon pore, it decreases their adsorption capacity. The results obtained in this study prove that the effect of MW irradiation on GAC seems to be more dominant (Bathen 2003) (Fig. 7).

After several regeneration cycles, the adsorptive capacity of GAC/Cu decreases considerably while GAC/Ni remained high during ten regeneration cycles as shown in Fig. 7. This fact could be attributed to the fact that Ni (II)

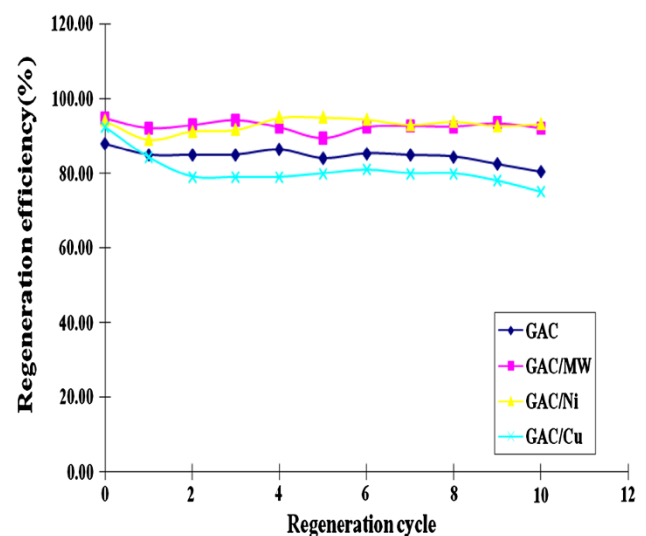


Fig. 7 Comparison of the adsorptive capacities of different activated carbon after 10 cycles of regenerations

adsorbs more MW irradiation, while Cu (II) has a negative effect on microwave irradiation or has more interaction with the activated carbon surface chemistry thus reducing the activated carbon adsorption capacity in aqueous solution. The adsorptive capacity of the as-received activated carbon (GAC) is high comparatively to GAC/Cu (Fig. 7). This result can be explained by the fact that the adsorption capacity of the activated carbon loaded with copper (GAC/Cu) decreases when the activated carbon is regenerated several times under microwave irradiation. These results confirm that the activated carbon loaded with Cu (II) does not adsorb sufficient microwave irradiations allowing the elevation of temperature meaning that Cu (II) adsorbs less or almost not the microwave irradiations allowing the decrease of GAC/Cu adsorptive capacity (Fig. 7).

Influence of nickel quantity on activated carbon regeneration cycles

The effect of the quantity of Ni loaded on activated carbon was also investigated when activated carbons were regenerated. To understand the effect of metal nickel loaded on activated carbon, the GAC/Ni (20:1) and GAC/Ni (10:1) were regenerated under similar conditions. The results show that the more nickel metal is loaded on activated carbon, the regeneration efficiency decreases significantly (Fig. 8). Obviously, because the activated carbon pores are saturated with Ni, the activated carbon adsorption capacity decreases (Fig. 8). It has been published that loading Ag^+ , Ni^{2+} , Cu^{2+} or Zn^{2+} ion on the activated carbon improves activated carbon adsorption capacity upon some

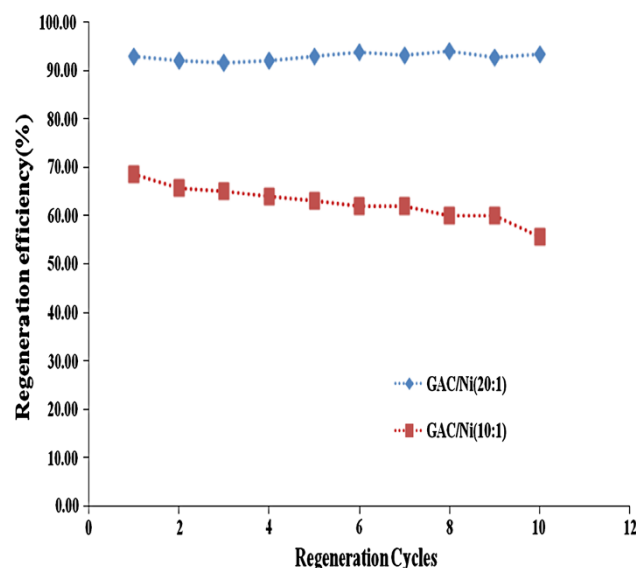


Fig. 8 Influence of nickel quantity upon regeneration efficiency after 10 cycles of regeneration

compounds like benzothiophene (Moxin et al. 2006; Loredana and Mihael 2012). During our experiments the activated carbon loaded with Ni^{2+} higher adsorption capacity upon phenol adsorption but when the Ni^{2+} increased, the activated carbon adsorption capacity considerably decreased. The activated carbon regeneration efficiency decreases with nickel quantity and upon the several regeneration cycles.

Conclusion

Under the optimum conditions, phenol was absorbed by the modified activated carbons. The modified activated carbons were saturated with phenol. After the maximum saturation, the modified activated carbons were regenerated by microwave irradiation under the protection of nitrogen gas. The activated carbon loaded with Cu^{2+} presented a considerable decrease upon phenol adsorption, while the activated carbon loaded with Ni^{2+} did not decrease upon phenol adsorption. The optimum conditions of the activated carbon regeneration were found. Under a microwave irradiation power of 700 W and 3 min of irradiation time, the regeneration efficiency of the modified activated carbons could reach around 98 %.

The activated carbons could regenerate with the regeneration efficiency of 98 % after 10 regenerations cycles. GAC/MW, GAC/Ni and GAC/Cu adsorptive capacity were higher than GAC before regeneration. The regeneration efficiency of GAC/Ni and GAC/MW remained high after 10 regenerations cycles, while GAC/Cu decreased significantly. This proves that Ni (II) plays an important role on the regeneration process compared with Cu (II). The regeneration efficiency of the activated carbon loaded with Ni^{2+} is higher than the activated carbon loaded with Cu^{2+} . Activated carbon loaded with Ni^{2+} can absorb more microwave energy than the one loaded with Cu^{2+} . During the regeneration process, the temperature of the modified activated carbons decreases with cycles of regenerations. The activated carbon temperature was measured. The regeneration temperature of GAC/Ni was higher than GAC/Cu, GAC/MW and untreated GAC over several cycles of regenerations. The quantity of metal Ni^{2+} loaded into activated carbon significantly decreases activated carbon regeneration efficiency. Even after several regeneration cycles, the regeneration efficiency remained decreased.

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