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Investigation of the Optimal Condition for Microwave Regeneration of Modified Activated Carbons Saturated with Phenol

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Abstract. Activated carbons (AC) are the most used materials for pollutant adsorption in aqueous solution. Regeneration allows activated carbons to be used several times after saturation. This process is industrially economic. Methods for AC regeneration are being developed and are a new field of investigation. Microwave heating is the most efficient method used to regenerate the activated carbons.AC modifications increase AC adsorption capacity. The purpose of this work was to investigate the optimal conditions for the regeneration of the modified activated carbons saturated with phenol. Microwave energy was used to prepare and regenerate the modified activated carbons (GAC, GAC/MW, GAC/Ni, and GAC/Cu). The effect of loading $Ni^{2+}$and Cu^{2+}ion on the optimal condition of regeneration of the modified AC is evaluated. Microwave heating parameters were also investigated. The microwave (MW) heating power and microwave irradiation time were optimized .The results showed that under the optimal condition of 3 minutes and 700 W irradiation power the modified AC could be regenerated ten times with high regeneration efficiency. The regeneration efficiency of the activated carbon impregnated with nickel is higher than the AC impregnated with cooper. The results showed that when the quantity of nickel is high, the AC regeneration efficiency is low. The results showed that the AC regeneration efficiency decreases with the quantity of metal loaded into AC. When MW irradiation power is high the AC regeneration efficiency increases considerably.

Keywords: Modified Activated Carbons, Microwave heating, Microwave Regeneration, MW irradiation.

1. Introduction

Our environment is considerably polluted with many activities of human being. The discharge of wastewater represents one of the major industrial pollutions [1]. Among the hazardous organic wastes produced by the industries, phenolic compounds are the most toxic. These compounds are known to be very dangerous because they are toxic even at low concentrations. Fundamental sources of this pollution are petroleum refining, the plastics and also ink industries. In this regard, adsorption represents one of the most important and more efficient techniques used to adsorb or retain the contamination [5].Among the materials commonly used as adsorbent, activated carbons are one of the most used materials [6] .Common regeneration techniques of AC beds in industrial applications are based on thermal (steam, carbon dioxide or inert atmosphere) and chemical methods (pH-swing or extraction with solvents). However, regeneration is time and energy consuming and very often there is a significant deterioration of the adsorbent's pore structure, thereby reducing the final adsorption capacity and the efficiency of the regeneration [8].In recent years, MW irradiation has attracted attention as a tool for regeneration due to its capability of molecular-level heating, which leads to homogeneous and quick thermal reactions. Interesting reports have appeared on the application of MW-heating technology for regenerating activated carbons with very promising results.

Regeneration of the activated carbon loaded with metal is not largely reported in literature .The effect regeneration of activated carbons loaded with metal is not yet well investigated .some interaction may occur between the metal and the activated carbon. The purpose of this study was to investigate the optimal condition for microwave regeneration of the modified activated carbons.

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2. Materials and Methodology

2.1. Materials

A charcoal activated carbon (GAC). Phenol solid , Cu $(NO_3).H_2O$ and Ni (NO_3) H_2O. The electronic scale balance: (JJ 1000, Max.1000g China). High-performance liquid Chromatography (HPLC) (LC-10UV, China) equipped with UV detector (UV-1575) and C18 reverse-phase column (250mm ×4.6 mm, 5_m ODS, China).Magnetic agitators HJ-6A (China).

2.2. Experimental Apparatus

The experimental device is a modified domestic microwave oven with operates at 2540 MHz's. The microwave was perced on top as shown in the figure1.Nitrogen gas is introduced from the upper part of the microwave oven .The conical quartz glass was used as the reactor in the experiments.

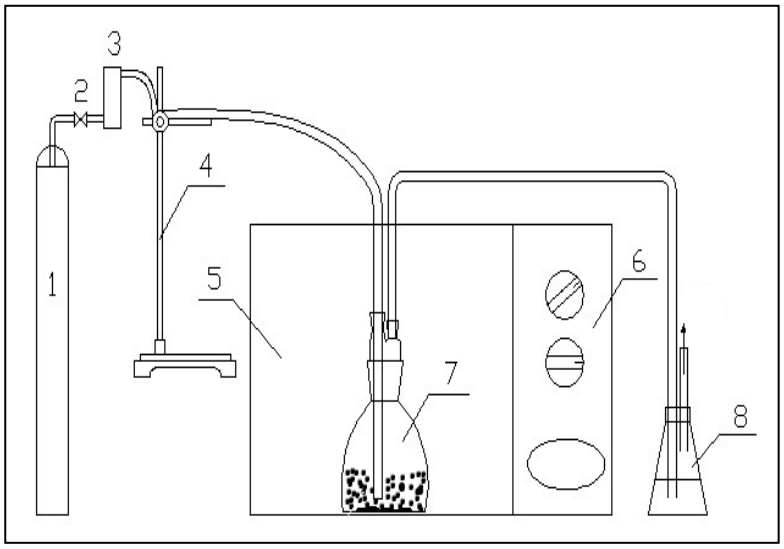

Fig. 1: Schematic of the apparatus used for regeneration of the modified activated carbons

1- Nitrogen gas container, 2-botle opener, 3-flow pressure meter, 4-support, 5-Microwave Oven, 6-Timer, 7-Conical quartz glass, 8-water container glass.

2.3. Preparation of the Modified Activated Carbons

The impregnation was done by microwave calcination. The solutions were prepared as follow: 3.78g of Cu $(NO_3).H_2O$, 5.01g of Ni (NO_3) H_2O were introduced into two different conical flask, 50ml of deionized .GAC were introduced into the solutions .Then the solutions were sharked at the speed of 232 rev / min during 2.5h. The weight percentages of metals are: Cu: GAC = 1:20. And Ni: GAC = 1:20. All was irradiated at 400W .intermittently irradiated (for 1min46s, 2min, and 50s) until nearly dryness. The Nitrogen gas was introduced at the pressure flow of $0.4m^3$ / h.

3. Results and Discussion

3.1. Influence of regeneration time

Many efforts have been done to optimize the conditions for high regeneration efficiency of activated carbons. In this study optimal condition for microwave regeneration of different type of modified activated carbons was investigated. GAC/Ni and GAC/Cu were chosen. The regeneration efficiency depends relatively on the microwave irradiation time. The highest regeneration efficiency is obtained when obviously the irradiation power was set at 700W. At lowest microwave irradiation power there is not significant increase of regeneration efficiency.

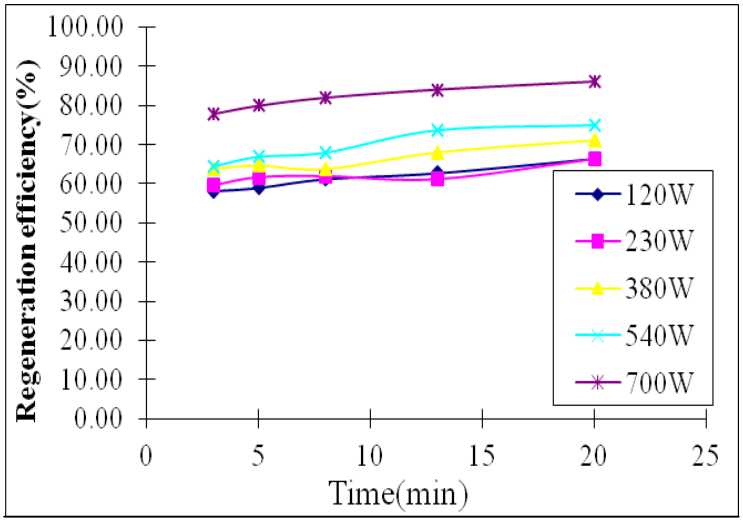

Fig. 2: Influence of microwave power on activated carbon regeneration efficiency

3.2. Influence of microwave regeneration power

When the microwave irradiation power is high, activated carbons regeneration efficiency is also high. Activated carbons removal efficiency reaches 80% to 90 % at the maximum microwave irradiation of 700W.

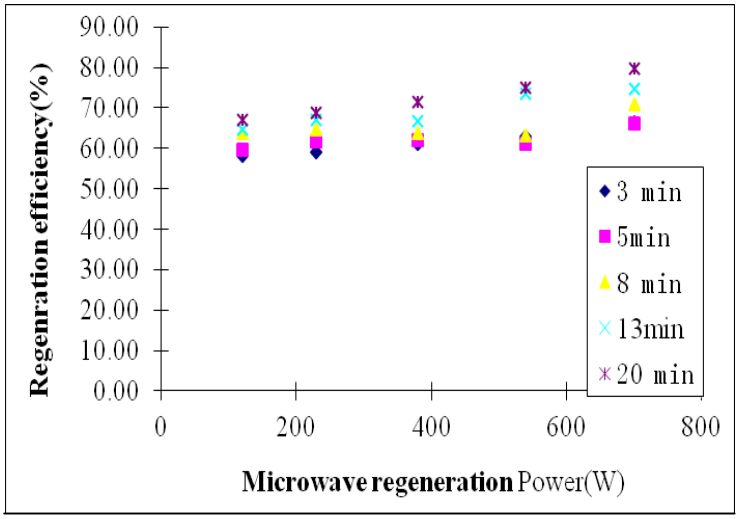

Fig. 3: Influence of microwave regeneration power GAC/Cu

The more activated carbons is exposed to MW irradiation, the regeneration efficiency is high. The highest regeneration efficiency was obtained at the microwave irradiation power of 700W and 20 minutes of irradiation time. From 3 minutes to 20 minutes, the regeneration efficiency isn't significant. This slightly difference may be explained by the fact that at five minutes phenol compounds adsorbed by activated carbons were almost desorbed. MW irradiation power was fixed at 700W and the regeneration time at 3minutes.

3.3. Influence of nickel quantity upon regeneration cycles

The effect of the quantity of Ni loaded on activated carbon was also investigated. 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 decrease significantly. Obviously because the activated carbons pores are saturated with Ni thus the activated carbon adsorption capacity decreases. It has been published that loading Ag^{+}, Ni^{2+}, Cu^{2+} or Zn^{2+} ion on the activated carbon improves adsorption of benzothiophene. In our work the activated carbon loaded with Ni^{2+} has a better adsorption but when the quantity of Ni^{2+} the opposite result is obtained. The activated carbons regeneration efficiency decreases with nickel quantity and upon the several regeneration cycles.

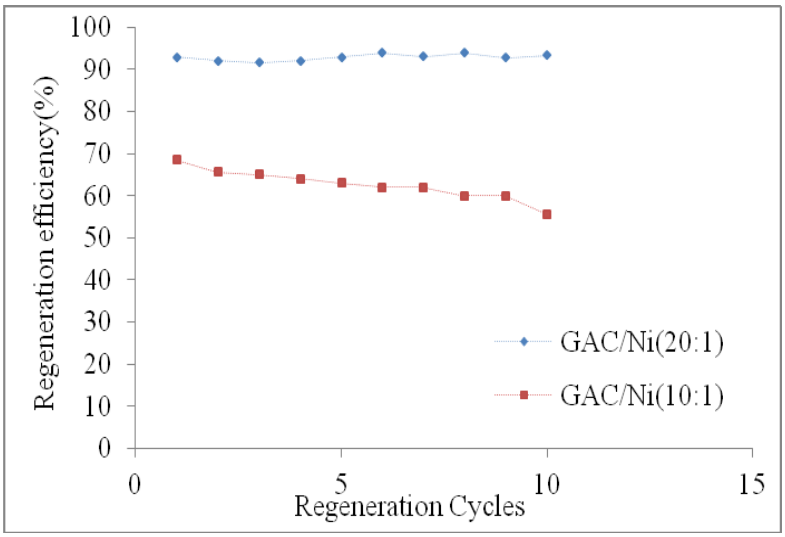

Fig. 4: Influence of nickel quantity on AC the regeneration efficiency (GAC/Ni)

4. Conclusion

Under the optimum conditions, the modified activated carbons were regenerated several times. After the maximum saturation the modified activated carbons were regenerated by microwave irradiation under the protection of nitrogen gas.

The modified activated carbons were saturated with phenol and regenerated ten time with the regeneration efficiency constant after cycles of regenerations.

The optimum conditions of the activated carbons regeneration were found. Under a microwave irradiation power of 700W and 3 minutes of irradiation time, the regeneration efficiency of the modified activated carbons could reach 98%.The activated carbons could be regenerated with the regeneration efficiency of 98% after 10 regenerations cycles. Time of irradiation of microwave power plays an important role on increasing activated carbons regenerations efficiency.

The experiments showed that the quantity of impregnated metal plays an important role on the activated carbons regeneration efficiency. The regeneration efficiency of the activated carbon decreased considerably when nickel quantity was high.

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