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Catalytic Ozonation with Activated Carbon for Degradation of Polyacrylamide in Wastewater

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Abstract: *With the development of tertiary oil, the technology of polymer oil displacement has been widely used. The treatment of the oilfield wastewater become harder, owing to the extensive use of the polyacrylamide (PAM) in oil displacement. This experiment explored the modified activated carbon surface properties on the removal rate of PAM, under the conditions of the temperature 50 ℃, the ozone concentration 5 L/min, the reaction time 1 h . The activated carbon(AC) was modified by copper, iron, nitrogen-containing compound. And the turbidity method was used to analyze the PAM concentration. The experimental results showed that the best modifier was ethylenediamine, which had less nitrogen; the optimum modification conditions of cupric nitrate were the calcination temperature 500 ℃, maceration 15 mL; the optimum modification conditions of iron nitrate were the calcination temperature 550 ℃, maceration 20 mL.*

Keywords: *Modified Activated Carbon, Pam, Ozonation, Degradation*

1. Introduction

At present, the demand for oil in every country is increasing day by day. Petroleum is a non-renewable energy source, and after extensive exploitation, there are less and less petroleum reserves in the formation. With the continuous shortage of oil resources, most oil fields have entered the middle and later stages of oil development. In order to improve oil recovery, most oil fields have already entered the third stage of oil extraction (Zhou 2013). The PAM as an oil displacement agent has obvious effects both in terms of crude oil recovery rate and economic benefits, and has been widely used in major oil fields (Fang 2016). However, the massive use of PAM flooding generally increases the viscosity and emulsification of the recovered water, thus it is more difficult to make the follow-up treatment of the produced water in the oilfield. After the wastewater treatment, there are still a large number of PAM. A more serious problem is that PAM will cause great harm to the surrounding environment (Stahl 1988), if it accumulates for a long time. It has become an urgent problem in the wastewater treatment process to solve the damage caused by PAM in wastewater. At present, physical and biological methods for treating PAM wastewater have not been widely applied, while chemical methods for treating PAM wastewater have obtained a good application in various oil fields. By adding aluminum sulfate (Li 2003), XG213 coagulant (Guan 2003), and flocculating agents LN-A (Lu 2004), which is composed of inorganic polymer and organic low molecular polymerization, into the wastewater containing PAM, the removal effect of suspended matter and residual oil in the produced water of oil field is good.

Activated carbon, with high specific surface area, good thermal stability and chemical stability, has been widely used as catalyst, catalyst carrier, adsorbent, battery electrode and fiber carbon structure hardening agent. In order to improve the adsorption performance of activated carbon, it is necessary to modify the activated carbon. Iron ion, copper ion and silver ion are commonly used as activated carbon agents (Smith 1997; Gong 2011). There have been a lot of studies on using modified activated carbon as catalyst to treat wastewater, many of which have achieved good results (Chen 2004). At the same time, the use of ozone oxidation can also make the degradation rate of organic matter in wastewater reach a high level (Ye 1989; Yang 2012; Pan 2007; Wang 2014; Ma 2010). Therefore, in this work, modified activated carbon was used as catalyst and ozone was added as oxidant to treat PAM in wastewater.

2. Experimental

2.1 Experimental Instruments

The instruments used in the experiment contain a 101-1AB electric blast drying oven (Beijing Zhongxing Weiye Century Instrument Co., Ltd.), a DF-101Z type heat collection, a constant temperature magnetic stirrer (Shanghai Lichen Bangxi Instrument Technology Co., Ltd.), an HY-010 type ozone generator (Chengdu Yifeng Hongyuan Environmental Protection Technology Co., Ltd.), an I3 type UV-visible spectrophotometer (Jinan Haineng Instrument Co., Ltd.) and a OTL1200 type tube furnace. Figure. 1 is the experimental devices as follows:

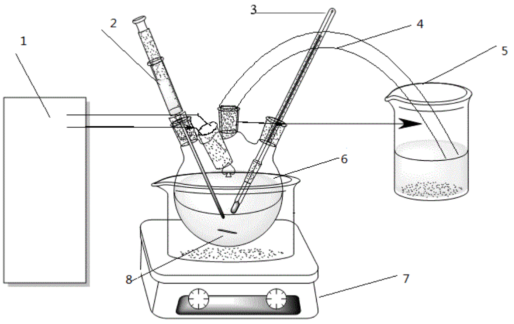


Figure. 1 Diagram of experimental devices

1-Ozone generator, 2-colloidal sampler, 3-thermometer, 4-exhaust pipe, 5-tail gas recovery cup, 6-four-necked flask, 7-magnetic stirring heater, 8-stirring rotor

2.2 Preparation of Catalyst

(1) Metal loaded activated carbon

54 mL hydrochloric acid solution with a mass fraction of 36%-38% was prepared with 146 mL deionized water as the cleaning solution. Three groups of activated carbon were weighed for 5 g for each group. An appropriate amount of hydrochloric acid impregnated activated carbon with 10% mass fraction was taken for 6 h to remove ash, and the activated carbon was washed to neutral with deionized water, followed by filtration and drying for 3 h. Then 0.5 mol/L modifier solution(copper nitrate or ferric nitrate) 10 mL, 15 mL, 20 mL impregnated with activated carbon for 12 h, extraction, drying for 8 h. Then it is calcined in a tube furnace for 3 h and the activation temperature is 300 ℃, 400 ℃ and 500 ℃.

(2) Nitrogen loaded activated carbon

Purification of AC: weigh 250 g activated carbon and immerse it in 1 mol/L hydrochloric acid solution for 6 h to remove ash. Then, rinse with ionized water until pH=5-7, place it in an oven at 105 ℃ for 4 h, and then put it in a dryer for standby use. The purified activated carbon was placed in 1 mol/L nitric acid, soaked at 40 ℃ for 6 h, washed with deionized water until neutral, and then dried in a drying oven at 105 ℃ for 8 h.

Preparation of ACN: The dried activated carbon was divided into five parts with 3 mol/L ethylenediamine solution, 3 mol/L diethylenetriamine solution, 3 mol/L triethylenetetramine solution and 3 mol/L tetrethylenepentamine solution as modifiers. The activated carbon was put into the modified agent, soaked for 12 hours, and then dried for 10 hours at 105 ℃. The activated carbon was respectively put into the tube furnace and injected with nitrogen, which was calcined at 450 ℃ for 2 hours. The activated carbon was cooled to below 30 ℃ under the protection of nitrogen, and then taken out and cooled in the dryer. Then, it was cleaned with deionized water for 3-5 times, drained, and then dried for 10 h at 105 ℃ in a dryer, cooled, and then put into a bag for standby use. The modified activated carbon AC, ACN_1, ACN_2, ACN_3 and ACN_4 were marked.

2.3 Catalytic Oxidation of PAM

An analytical balance was used to weight 300 mg of modified AC for later use. 250 mL PAM stock solution was removed from the 1000 mg/L PAM mother liquor to prepare a PAM reaction solution of the desired concentration. The prepared PAM solution was poured into a dry four-necked flask, and then a magnetic stirrer was put into the flask; the four-necked flask containing the solution was placed in a water bath to fix the height of the PAM solution below the height of the water layer. After turning on all the switches, the speed and temperature of the magnetic stirrer were set; the bottom of the meter and the bottom of the trachea were at the same level as the stirrer; when the reaction temperature of the PAM solution reached the specified temperature, it would pass through the four ports. After the modified AC catalyst was added into the flask, the ozone generator switch was turned on and the timer was started. The sampler was used to take 5 mL of sample liquid through the sampling port every time. The residue was filtered through the disposable filter head and then put into the sample tube for testing. The temperature was controlled during the reaction so that the temperature difference was lower than ±1 ℃.

2.4 Analysis Method

According to the turbidimetric analysis standard (Wu 2009), the volume ratio of 1:2:2 (PAM: acetic acid: sodium hypochlorite) was used to draw the standard curve. A certain concentration gradient of PAM solution was prepared, which were 50 mg/L, 100 mg/L, 150 mg/L, 200 mg/L, 250 mg/L, 300 mg/L, 400 mg/L and 500 mg/L, respectively. Move separately with the pipette take different concentrations of PAM solution 2 mL to 250 mL of iodine in the volumetric flask, according to the proportion to join 5 mol/L acetic acid solution, gently shake, static 1-2 min, and then according to the proportion to 0.461% NaClO solution shake, about 20 min after precipitation reaction is completed, measure the absorbance value, get the following standard curve shown in Figure 2 as following:

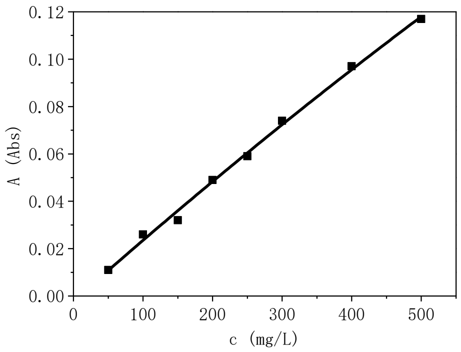


Figure. 2 Standard cruve

As shown in the Figure, the points in the Figure are basically on a straight line and show a linear relationship. The equation is as follows:

$$y=0.00024x+0.000003 \tag{1}$$

In the equation curve fitting, variance $R^2=0.99617$, indicating that the measured value is very close to the fitting formula, so this formula can be taken as the standard curve equation of the experiment.

The degradation rate is calculated according to the following formula:

$$D=\left(1-\frac{A}{A_0}\right)\times 100\% \tag{2}$$

Where D is the degradation rate (%), and A and A_0 are the absorbance before and after degradation, respectively.

3. Results and Discussion

3.1 Experimental Repeatability

The experimental temperature was 50 ℃, the ozone concentration was 5 L/min, and the reaction time was 30 min. The absorbance was measured by sampling.

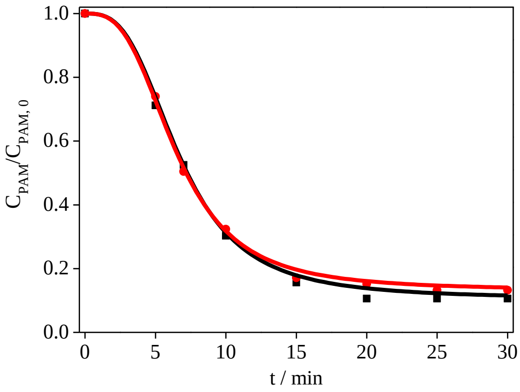


Figure. 3 Experimental repeatability

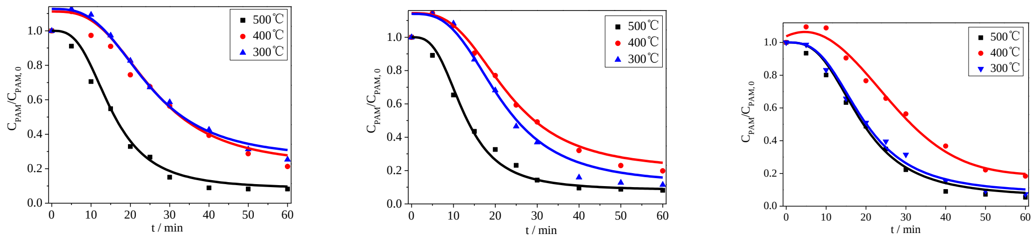
According to the analysis in Figure. 3, in the repeated experiment, the reaction was slow 2 min at the beginning of the reaction, then began to drop sharply 2 min later. After 15 min, the reaction tended to slow down and basically reached a balance. In addition, the linear basic repetition of two experiments has been achieved in several pre-experiments, meeting the requirements of pre-experiments.

3.2 Copper Loaded Activated Carbon

When the modifier is copper nitrate, the calcination temperature of activated carbon in this experiment is 300 ℃, 400 ℃ and 500 ℃, and the immersion amount of the modifier is 10 mL, 15 mL and 20 mL. Firstly, the effect of different calcination temperature on the catalytic degradation of PAM by activated carbon modification was analyzed, and then the effect of different impregnation amount on the catalytic degradation effect of PAM by activated carbon modification was analyzed.

(1) Effect of calcination temperature

In the experiment, the impregnation amounts of the modifier copper nitrate solution were 10 mL, 15 mL, and 20 mL. The influence of different impregnation amounts at the calcination temperature of 300 ℃, 400 ℃, and 500 ℃ on the catalytic ozonation of PAM by activated carbon modification was discussed and analyzed as shown in Figure 4:



(a) (b) (c)

Figure. 4 Effect of calcination temperature: (a) impregnation amounts is 10 mL; (b) impregnation amounts is 15mL; (c) impregnation amounts is 20 mL;

As shown in Figure 4, when the amount of the modifier was 10 mL, 15 mL, and 20 mL, and the calcination time was 3 h, the modified activated carbon catalyzed ozonation of PAM wastewater at the calcination temperature of 300 ℃, 400 ℃, and 500 ℃. The degradation rate with 500 ℃ had the best effect. The reasons may be as follows:(1) because the calcining temperature is too low, copper nitrate is not easy to decompose into metal oxide, which fails to make it better loaded, resulting in poor catalytic activity of modified activated carbon; (2) Due to insufficient activation of activated carbon by temperature, the specific surface area and porosity of activated carbon are not changed enough to improve the adsorption performance of activated carbon; (3) At the beginning of the reaction, the curve showed an increasing point, which may be because after adding modified activated carbon, the adsorption of activated carbon reached equilibrium. As the reaction proceeded, the concentration of PAM in the solution decreased, and PAM adsorbed in the activated carbon channel was released, so the concentration of PAM in the solution increased and the point of increase appeared. (4) There is a point in the Figure where the curve does not rise after the experimental reaction. The reason may be that under the calcination at this temperature, the activation temperature is sufficient to widen the activated carbon channel, increase the specific surface area, and shorten the time period for activated carbon to adsorb and release PAM at the beginning of the reaction.

(2) Effect of the amount of modifier

When the calcination temperature is 500 ℃, the influence of different impregnation amount of modified activated carbon on the catalytic ozonation of PAM wastewater is discussed and analyzed, as shown in Figure 5:

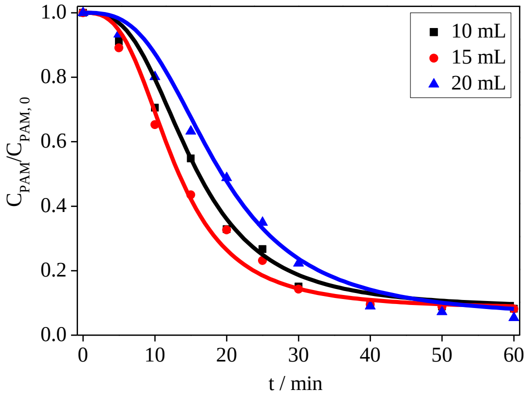


Figure. 5 Effect of the amount of modifier

As can be seen from Figure 5, the reaction appeared a sudden drop 5 min after the experiment began, and stabilized after 40 min. When the calcination temperature of modified activated carbon is 500 ℃, the influence of changing the experimental impregnation amount on the final removal rate of PAM wastewater has little difference, and the overall linear change trend is similar. But at the same time, the degradation rate of modified activated carbon was the fastest when the impregnation amount was 15 mL. Conclusion: When the calcination temperature is 500 ℃, the modified activated carbon has the best effect on the degradation of PAM wastewater by catalytic ozonization under the condition of 15 mL of the modifier.

Under the same calcination temperature, immersion fluid volume have great influence, too little or too much is bad for modification of activated carbon, the reason may be that: (1) when the amount of modifier dipping is 20 mL, the PAM degradation rate is the worst. May be too much metal oxide load activated carbon pore blockage, appear reunion phenomenon, thus reducing the activated carbon adsorption, hindered the PAM contact with the catalyst active component, the degradation effect of variation; (2) In the modification process of 20 mL immersion amount, too many active components enter into the micropores of activated carbon, and the micropore structure is usually not conducive to the diffusion and reaction of ozone and organic matter, so that the active components cannot give full play to the catalytic role and the degradation effect becomes worse; (3) When the impregnation amount is 10 mL, it may be due to the insufficient impregnation amount of the modifier, resulting in insufficient load, so that its catalytic activity fails to meet the modification requirements and its degradation effect is poor.

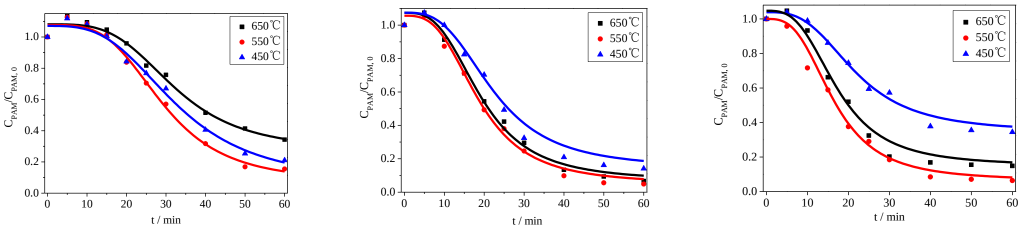
3.3 Iron Loaded Activated Carbon

When the modifier is iron nitrate, the calcination temperature of activated carbon in this experiment is 450 ℃, 550 ℃ and 650 ℃, and the immersion amount of the modifier is 10 mL, 15 mL and 20 mL.

Firstly, the effects of different calcination temperatures on the catalytic degradation of PAM by modified activated carbon were analyzed, and then the effects of different impregnation amounts on the catalytic degradation of PAM by modified activated carbon were analyzed.

(1) Effect of calcination temperature

In the experiment, the impregnation amounts of the modifier iron nitrate solution were 10 mL, 15 mL, and 20 mL. The influence of different impregnation amounts at the calcination temperature of 450 ℃, 550 ℃, and 650 ℃ on the catalytic ozonation of PAM by activated carbon modification was discussed and analyzed as shown in Figure 6:



(a) (b) (c)

Figure. 6 Effect of calcination temperature: (a) impregnation amounts is 10 mL; (b) impregnation amounts is 15mL; (c) impregnation amounts is 20 mL;

As shown in Figure 6, when the amount of the modifier was 10 mL, 15 mL, and 20 mL, and the calcination time was 3 h, the modified activated carbon catalyzed ozonation of PAM wastewater at the calcination temperature of 450 ℃, 550 ℃, and 650 ℃. The degradation rate with 550 ℃ had the best effect. The reasons may be as follows: (1) According to the Figure 6, when the modified calcination temperature is 650 ℃, the degradation effect is not good, indicating that too high calcination temperature makes activated carbon and metal oxide sintered, causing the hole collapse, the specific surface area is destroyed, reducing the catalytic activity of activated carbon, leading to its catalytic degradation effect of PAM. (2) When the modified calcination temperature is 450 ℃, the degradation effect is not good, indicating that the calcination temperature is too low, iron nitrate solution is not easy to decompose into metal oxides, and the load is low, resulting in poor degradation effect of PAM.

(2) Effect of the amount of modifier

When the calcination temperature is 550 ℃, the influence of different impregnation amount of modified activated carbon on the catalytic ozonation of PAM wastewater is discussed and analyzed, as shown in Figure 7:

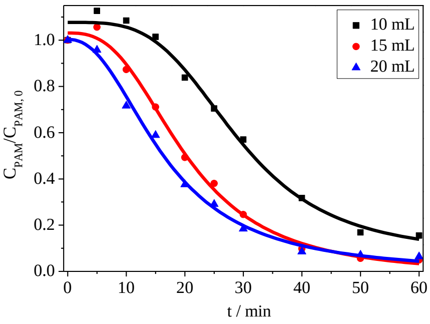


Figure. 7 Effect of the amount of modifier

As can be seen from Figure 7, at the calcination temperature of 550 ℃, the effect of the catalytic ozonation of PAM with modified activated carbon was the best. Within a certain range, with the increase of the amount of the modifier, the modified activated carbon can be used as a catalyst to catalyze the degradation of PAM. When the impregnation amount was 15 mL and 20 mL, the final degradation rate was the same, which was significantly better than the impregnation amount of 10 mL. It was concluded that the catalytic degradation of PAM was the best when the calcination temperature was 550 ℃ and the amount of the modifier was 20 mL. The reasons may be due to the insufficient impregnation amount of the modifier, resulting in a low iron load, which makes its catalytic activity fail to meet the modification requirements and its degradation effect is poor.

3.4 Nitrogen Loaded Activated Carbon

When the modifiers were ethylenediamine, diethylenetriamine, triethylenetetramine and tetrethylenepentamine, the effects of different nitrogen content compounds on the catalytic ozonation of PAM were discussed and analyzed.

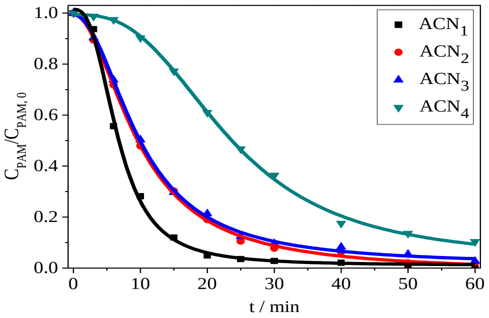


Figure. 8 Effect of nitrogen content of modifier

As shown in Figure 8, Within a certain range, as the nitrogen content of the modifier increases, the catalytic ozonation of PAM on modified activated carbons gradually deteriorates. The catalytic degradation effect of PAM after modified by ethylenediamine was the best, the effect difference between diethylenediamine and triethylenetetramine was not obvious, and the effect of tetrethylenetetramine was the worst. Thus the catalytic degradation of PAM with ethylenediamine modified activated carbon with less nitrogen content was the best. The reason may be as follows: (1) in the process of activation, load content of nitrogen and carbon atoms in the activated carbon to form a ring structure, is not conducive to activated carbon adsorption, with the increase of load in nitrogen content, the formation of ring type structure, the adsorption performance, reduces the activated charcoal catalytic activation, the catalytic degradation of PAM effect becomes poor. (2) With the increase of nitrogen content, the degradation effect becomes worse, which may be due to the high nitrogen content load that causes the micropores of activated carbon to be blocked, resulting in the reduction of specific surface area of activated carbon, which reduces the adsorption performance of activated carbon, hindering the contact between PAM and active components of catalyst, and making its degradation effect worse.

3.5 Nitrogen and Iron Loaded Activated Carbon

When the modifiers were diethylenetriamine +5%Fe, triethylenetetramine +5%Fe and tetrethylenepentamine +5%Fe, the effect of the catalytic degradation of PAM by modified activated carbon was discussed and analyzed.

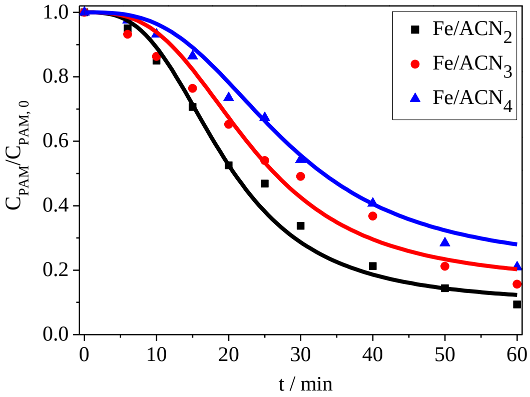
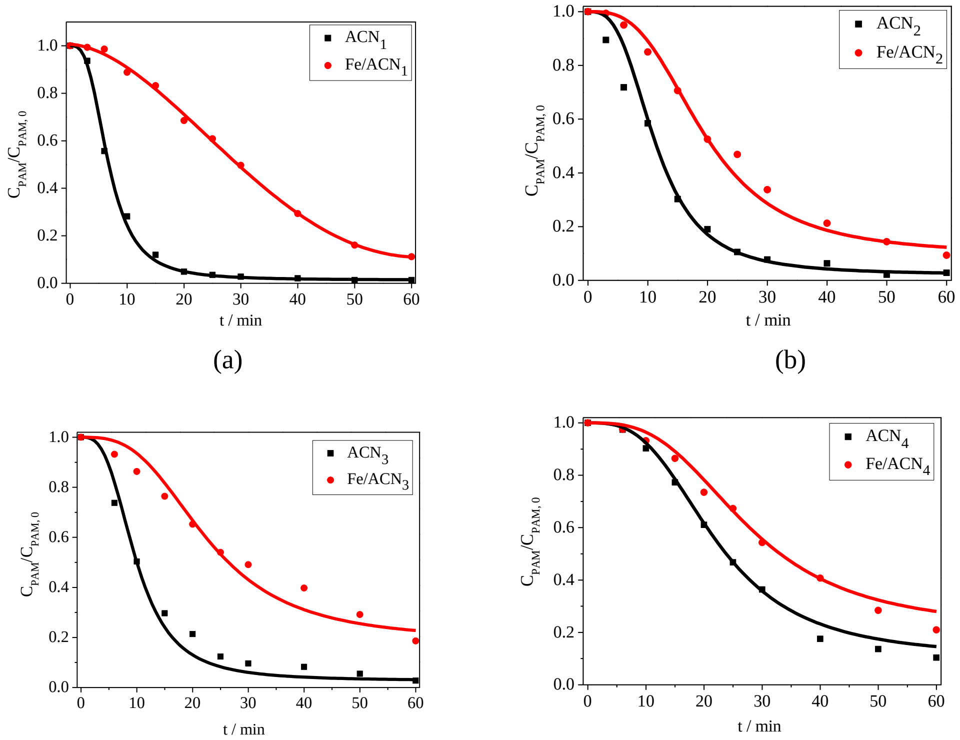


Figure. 9 Effect of Fe/ACN content of modifier

It can be seen from Figure 9, on the basis of modification with different nitrogen content and the same amount of 5%Fe, the activated carbon is secondary modified, and the modification result is that the catalytic degradation effect of PAM on activated carbon with diethylenetriamine +5%Fe, triethylenetetramine +5%Fe and tetrethylenepentamine +5%Fe becomes worse in turn, and the final degradation rate gradually decreases. Thus within a certain range, after the secondary modification of 5%Fe, the result of the modification is that with the increase of nitrogen content, the catalytic degradation effect of PAM wastewater by modified activated carbon is worse.

The reasons may be as follows :(1) the load containing high nitrogen makes part of the pore structure of activated carbon blocked, resulting in the reduction of specific surface area of activated carbon, which reduces the adsorption performance of activated carbon, hindering the contact between PAM and active components of activated carbon, reducing its catalytic activity and leading to poor degradation efficiency; (2) In the activation process, the nitrogen in activated carbon load and carbon atoms in activated carbon formed a ring structure, not conducive to the adsorption of activated carbon, with the increase of nitrogen content of the load, the formation of the ring structure more, adsorption performance decreased, reduced activated carbon catalytic activation, so that its catalytic degradation of PAM effect becomes worse.

Activated carbon was first modified with ethylene diamine, diethylenetriamine, triethylenetetramine and tetrethylenepentamine containing nitrogen compounds, and then modified in a solution containing 5% iron. The effects of primary modified and secondary modified activated carbon on the removal of PAM wastewater were compared.



(a) (b)

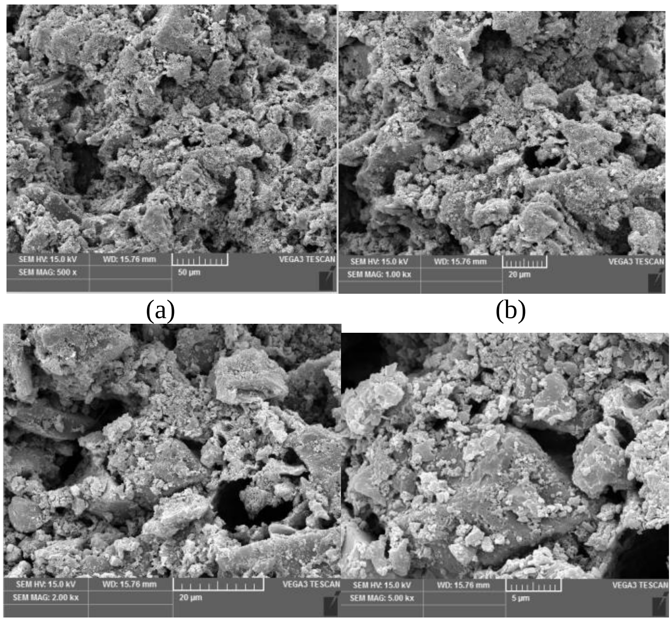
(c) (d)

Figure. 10. Comparison of CAN and Fe/ACN

It can be seen from Figure 10, the removal of PAM wastewater by ACN is better than by Fe/ACN in the four groups. When the activated carbon carries iron through secondary modification, the activated carbon is activated by secondary calcining, and the high temperature makes the microcrystal on the surface of the activated carbon appear sintering phenomenon, resulting in the destruction of the microporous structure of the activated carbon, which reduces its catalytic activity. It is also possible that the excess load causes the partial pore structure of activated carbon to be blocked, resulting in the decrease of specific surface area of activated carbon, the decrease of adsorption performance, and the degradation effect of PAM.

3.6 Characterization

The surface morphology of composite catalysts was analyzed by scanning electron microscopy (SEM). Different magnification multiples of ordinary activated carbon were used for SEM analysis, and the magnification multiples were 500, 1000, 2000 and 5000, respectively. The surface structure scanning diagrams were obtained, as shown in Figure 11.



(a) (b)

(c) (d)

Figure. 11 SEM of activated carbon

From the Figure11, it can be seen the various pore structures on the activated carbon cross section can be seen from different magnification Figures, indicating that the activated carbon has a very rich specific surface area.

The modified activated carbon with calcination temperature of 500 ℃ and the immersion(copper) amount of 15 mL of the modifier was subjected to SEM analysis at different magnification multiples, which were 500, 1000, 2000 and 5000 times respectively. The surface structure scanning diagrams were obtained, as shown in Figure 12.

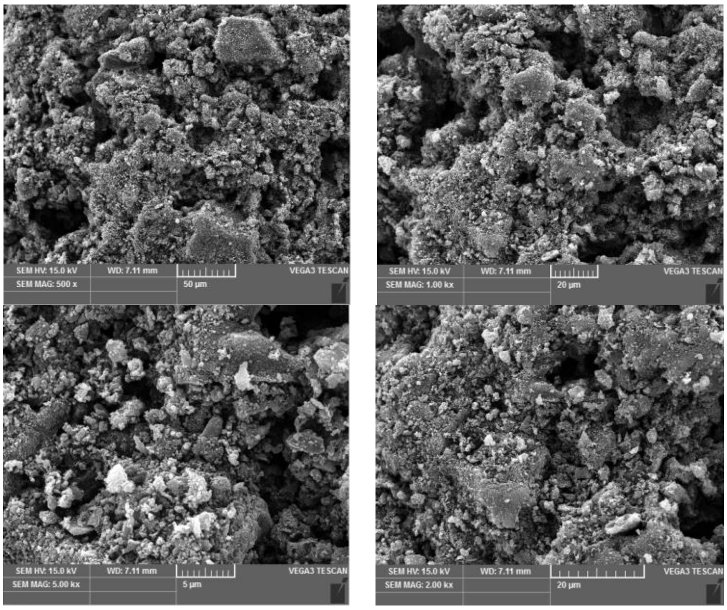


Figure. 12 SEM of AC/Cu

Figure12 is the SEM image of the copper-loaded activated carbon at different magnification times.

Compared with Figure11, the comparison at the same magnification times shows that the number of slightly larger pores in the activated carbon modified by copper nitrate decreases, and the number of micropores increases significantly, which greatly improves the specific surface area and the number of pores of the activated carbon. It can also be seen from Figure12 that some substances were added to the surface of activated carbon modified by copper nitrate, and it can be speculated that the oxide load of copper was successful.

The modified activated carbon with calcination temperature of 550 ℃ and the immersion(iron) amount of 20 mL of the modifier was subjected to SEM analysis at different magnification multiples, which were 500, 1000, 2000 and 5000 times respectively. The surface structure scanning diagrams were obtained, as shown in Figure 13.

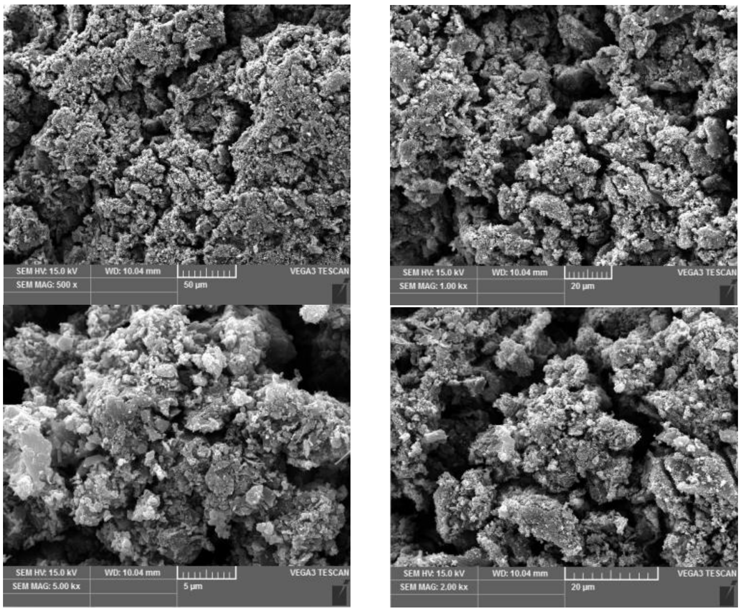


Figure. 13 SEM of AC/Fe

Figure13 is the SEM Figure of ferribearing activated carbon at different magnification times. Compared with Figure 3-15, it can be found that the number of slightly larger pores in the activated carbon modified by iron nitrate decreases, but the number of some small micropores increases. It was also observed that the surface structure of activated carbon modified by ferric nitrate appeared a large number of layers, which greatly increased the specific surface area of activated carbon. It can also be seen from Figure 13 that the surface of activated carbon modified by iron nitrate has increased substances, which can be speculated to be the success of iron oxide load.

4. Conclusion

In this paper, the surface properties of activated carbon modified by different agents were studied to investigate the effect of catalytic ozonation of modified activated carbon on the removal of PAM wastewater. When the modifier is copper nitrate, the calcination temperature is 500 ℃, and the impregnation amount is 15 mL, the removal effect of PAM wastewater is the best. When the modifier is iron nitrate, the calcination temperature is 550 ℃, and the impregnation amount is 20 mL, the removal effect of PAM wastewater is the best. When the modifiers are nitrogen compounds, they are ethylenediamine, diethylenetriamine, triethylenetetramine and tetrethylenepentamine respectively. Under the same conditions, the experimental results show that the removal effect of the PAM wastewater by modified activated carbon with ethylenediamine is the best, and the modification effect becomes worse with the increase of nitrogen content. When the modifier is nitrogen compounds and 5%Fe, the activated carbon modified with diethylenetriamine, triethylenetetramine and tetrethylenepentamine is secondary modified, and the same amount of 5%Fe is loaded. The results show that the removal effect of PAM wastewater by diethylenetriamine +5%Fe modified activated carbon is the best. After primary modification by ethylenediamine, diethylenetriamine, triethylenetetramine and tetrethylenepentamine, and then secondary modification in 5% iron solution, the effect of primary modification and secondary modification of activated carbon on the removal efficiency of PAM wastewater was compared. The results show that the removal of PAM wastewater by activated carbon modified by nitrogenous compounds is the best in the four groups of comparative experiments.

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