Sulfur-doped porous carbon derived from waste polyphenylene sulfide for efficient adsorption removal of Cd\(^{2+}\) from simulated wastewater

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Abstract: Sulfur-doped porous carbon materials were prepared from waste polyphenylene sulfide (PPS) by chemical activation using potassium hydroxide as activator. The elemental composition (sulfur content) and the number of acid oxygen-containing groups on the surface of the samples were determined by elemental analysis and Boehm method, respectively. The effects of preparation temperature and adsorption conditions on the removal of Cd\(^{2+}\) from simulated wastewater were studied according to the amount of cadmium removal. The results showed that the sulfur-doped porous carbon materials prepared under the optimal preparation conditions had better adsorption properties as compared to HNO\(_3\) modified bamboo-derived biochar. This result is mainly due to the Lewis acid-base interaction between sulfur and Cd\(^{2+}\) in addition to the coordination of Cd\(^{2+}\) and oxygen-containing groups on the surface. Under the optimal adsorption conditions, the removal of cadmium in simulated wastewater can reach 98.74%. In conclusion, this cheap porous carbon material may be a useful absorbent for cadmium.

1. Introduction
The development of economy and society is inseparable from the development of industry, but the process of industrial production often causes certain damage to the environment. For example, the wastewater from mining, industrial production, nonferrous metal smelting, electroplating and battery production often contains heavy metal wastewater [1]. Heavy metal ions in wastewater are more and more harmful to the ecological environment and human health. Such heavy metal wastewater will not only enter into rivers and lakes with surface runoff, pollute the source of human drinking water, threaten the survival of aquatic animals and plants, but also infiltrate into groundwater, pollute soil, enrich heavy metals in crops, and finally endanger human health. Wastewater containing heavy metal ions is very harmful. The most representative is cadmium (II), which is not abundant in nature, but can be enriched in the food chain [2, 3]. As the three major food crops, rice plants have a strong ability to enrich cadmium, so in the world's major rice production areas are often high incidence of cadmium poisoning[4-6]. At the same time, due to the high solubility of these substances in the aqueous
environment, they are easily absorbed by organisms. Once these heavy metal ions enter the food-chain, they may accumulate in the human body [7, 8]. When the concentration exceeds a certain limit, it will cause health damage to the human body. The damage of cadmium to the human body mainly lies in the kidney [9, 10], which will cause irreversible damage to the filtration structure of the kidney; in addition, cadmium will lead to a large loss of calcium in the body, and eventually lead to fracture deformation due to calcium [8, 11]. The well-known itai-itai disease[12] is caused by cadmium poisoning.

At present, the research of heavy metal wastewater treatment technology mainly includes adsorption, chemical treatment, and biological treatment and so on. The heavy metals in the water can be adsorbed onto the surface of the adsorbent, which can not only effectively reduce the content of heavy metals in water, but also improve the quality of water. There are many kinds of adsorbents; the most common one is activated carbon. Activated carbon is a kind of porous non-polar adsorbent [13-15]. Because of its special pore structure, which has a large specific surface area, more surface compounds and good mechanical strength, it is often used. Activated carbon can absorb many kinds of heavy metal ions at the same time, with large adsorption capacity, but with high cost, short service life, regeneration and high operating cost. Considering the development of environmental protection, it is also necessary to reduce production costs as much as possible. Therefore, the preparation of adsorbents with low value and large amount of solid materials is an increasingly popular method [16-18]. Ore, sludge, industrial by-products, waste, agricultural waste and domestic waste can be used as adsorbent materials after modification.

Polyphenylene sulfide (PPS) is a kind of special engineering plastics with excellent chemical resistance, excellent mechanical properties, good thermal stability and high cost performance [19, 20]. Due to the special properties of its materials [21], more and more industries have a stable demand for PPS. At present, there is still a big gap in the international polyphenylene sulfide Market. Most of the world's polyphenylene sulfide is produced by Japanese enterprises[22]. As more and more enterprises enter into the production of PPS, the production capacity of PPS will be improved [23]. Therefore, it is very reasonable and feasible to prepare activated carbon adsorbent for the treatment of cadmium in industrial wastewater with waste PPS and recycled PPS as raw materials. KOH [8, 24] is used as activator through chemical activation, and the mass ratio of raw material and activator is 2:1. The porous carbon adsorbent prepared from waste polyphenylene sulfide (PPS) was characterized. The sulfur content of the adsorbent was determined by elemental analysis, and the number of acid groups on the surface of the adsorbent was determined by Boehm titration.

At the same time, the effects of different adsorption conditions on the removal of cadmium ions from simulated cadmium wastewater by adsorbents prepared from waste polyphenylene sulfide were studied. The effects of adsorption temperature, adsorption time and adsorbent dosage on the removal rate of cadmium in simulated wastewater were studied. The best adsorption conditions were found, and the causes of the best adsorption conditions were discussed based on the characterization data of carbon materials. The results provide a theoretical and practical basis for the treatment of cadmium in industrial wastewater by waste PPS derived sulfur-doped porous carbon materials.

2. Experimental

2.1. Reagents and raw materials
Potassium hydroxide, potassium iodide, phenolphthalein, methyl orange, L (+) - ascorbic acid, sodium hydroxide, sulfuric acid, hydrochloric acid, ethanol, polyvinyl alcohol, Rhodamine B purchased from Sinopharm Group Chemical Reagent Co. Ltd. Waste polyphenylene sulfide was obtained from a local factory.

2.2. Preparation and modification of materials
Weigh 4.0 g waste polyphenylene sulfide (PPS) and 2.0 g potassium hydroxide. Measure 10ml ethanol and 10ml deionized water, mix and stir until potassium hydroxide is completely dissolved. At this time,
the solid phase is completely immersed in the liquid phase. The system was placed in an ultrasonic cleaner for 10 min (in order to make the system more uniform). After treatment, the system was immersed in room temperature for 12 h, and then dried in an oven at 60 °C for 12 h. After drying, the material was transferred to a tubular furnace, and nitrogen was introduced. The temperature control program was set. The activation temperature (maximum temperature) was controlled at 400 °C, 450 °C and 500 °C for 3 h. After cooling down, the carbonization step is completed. The material is crushed and placed in the water bath pot of magnetic stirrer with water of 700ml. The temperature is set at 100 °C. After water washing, dilute hydrochloric acid is used, and then water washing is carried out after pickling until the filtrate is neutral. Finally, porous carbon materials derived from polyphenylene sulfide (PPS) were dried in an oven at 100 °C for 12 h (denoted as P400, P450 and P500 respectively, where p represents polyphenylene sulfide, and 400, 450 and 500 represent activation temperature).

2.3. Static adsorption experiment

0.5078 g of CdCl₂ · 2.5H₂O was dissolved in distilled water and diluted to 1000ml with distilled water to prepare simulated wastewater containing Cd²⁺ with mass concentration of 250PPM. 25ml simulated Cd²⁺ wastewater and certain amounts of porous carbon material (0.03 g-0.15g) were placed in a 50ml round bottom flask. The pH of the system was adjusted with 0.1 mol / L NaOH solution (range 3-7). The system was placed in a constant temperature water bath (temperature range: 30 °C -90 °C) and magnetic stirring for a certain time (20-140min). The concentration of Cd²⁺ in the filtrate was determined by UV-Vis spectrophotometer and the removal rate of Cd²⁺ was calculated according to (1).

\[
\text{Removal rate (\%) = } \frac{(C_0 - C_E)}{C_0} \quad \text{(1)}
\]

Where, \( C_0 \) is the initial concentration of Cd²⁺ in the simulated wastewater, and \( C_E \) is the concentration of Cd²⁺ in the filtrate after saturated adsorption. Each experiment was carried out three times, and the deviation of observation data of three parallel experiments was less than 5%.

2.4. Characterization of porous carbon materials

0.05g of waste polyphenylene sulfide porous carbon material was accurately weighed by analytical balance, and put into a 250ml conical flask. 20ml sodium hydroxide solution (concentration: 11.00mmol / L) was added into the conical flask to seal the conical flask, and ultrasonic machine was used for one hour. Filter the solution and rinse with deionized water for several times to ensure complete collection of sodium hydroxide. Add phenolphthalein to the filtrate as indicator (red at this time), titrate with hydrochloric acid (concentration: 10.92 mmol / L) until colorless, which is the end point. The total number of acid groups[8, 25] on the surface of porous carbon was calculated from the titration results. Keeping other conditions unchanged, the titrant was changed from sodium hydroxide to potassium bicarbonate, and the indicator was changed from phenolphthalein to methyl orange. The total number of carboxyl groups[26, 27] on the surface of porous carbon materials can be calculated from the titration results.

The sulfur content in porous carbon materials was determined by elemental analysis.

3. Results and Discussion

3.1. Characterization of representative porous carbon material

3.1.1. The sulfur content of porous carbon

Fig.1 shows the sulfur content of carbon materials at different activation temperatures. It can be seen from Fig.1 that the sulfur content of porous carbon decreased with the increase of activation temperature. The sulfur content of porous carbon at 450 °C has little change compared with that at 400 °C, but the sulfur content of porous carbon greatly decreased at 500 °C and 550 °C. It can be attributed to the etching effect of potassium hydroxide (KOH) on waste polyphenylene sulfide (PPS)[28]. At a lower temperature (400 °C -450 °C), the etching effect of potassium hydroxide on polyphenylene
sulfide is not too strong, so the loss of sulfur content is little with the increase of temperature; when the temperature is further increased (>450 °C), the etching effect of potassium hydroxide on polyphenylene sulfide increased sharply. As a result, the sulfur content in the prepared carbon materials decreased significantly with the increase of temperature.

3.1.2. Acid group and carboxyl groups on the surface of porous carbon materials
Table 1 shows the number of acid and carboxyl groups on the surface of carbon materials prepared from waste polyphenylene sulfide at different carbonization temperatures. The results showed that the amount of total acid group and total carboxyl group increased with the increase of carbonization temperature in the temperature range of 400 °C -450 °C. However, when the temperature is higher than 450 °C, the number of total acid groups and total carboxyl groups decreased with the increase of activation temperature. This is due to the formation and decomposition of acid groups and carboxyl groups in the activation process. At low temperature (400 °C - 450 °C), with the increase of temperature, more acidic groups begin to form; at higher temperatures (> 450 °C), the decomposition of acid groups caused by high temperature cannot be ignored, and the decomposition of acid groups begins to dominate in the process. Therefore, the number of surface groups of porous carbon materials increased and then decreased with the increase of temperature[29, 30].

Table 1: The total number of acid and carboxyl groups at different carbonization temperature

| Sample | Carbonization temperatures (°C) | Total number of acid groups(mmol/g) | Total number of carboxyl groups (mmol/g) |
|--------|---------------------------------|-------------------------------------|-----------------------------------------|
| P-400  | 400                             | 1.323                               | 0.767                                   |
| P-450  | 450                             | 1.833                               | 0.902                                   |
| P-500  | 500                             | 1.576                               | 0.826                                   |

3.2. \( \text{Cd}^{2+} \) adsorption properties of different porous carbon materials
Table 2 compares the adsorption capacity and cadmium removal of the porous carbon materials obtained at different activation temperatures. The results showed that with the increase of activation temperature, the adsorption capacity and the amount of cadmium removal increased and then decreased, reaching the highest value at 450 °C. At this time, the prepared porous carbon material has the best performance (the highest cadmium removal). Compared with the characterization data of carbon materials, it is obvious that the carbon material prepared at 450 °C has the most acidic groups, and the porous carbon materials have the best adsorption performance. It can be seen that the number of acid groups on the surface plays a decisive role in the adsorption properties of carbon materials. This can be attributed to the coordination between the oxygen-containing acid groups on the surface of activated carbon and the heavy metal ions in the solution[31-33] i.e. the lone pair electrons on the oxygen-containing acid group transfer to the empty orbit on the cadmium ion, resulting in coordination[34]. Therefore, the number of acid groups on the surface of the activated carbon plays a leading role in the adsorption of cadmium ions on the porous carbon materials. In addition, the sulfur content of porous carbon materials also has a greater effect on promoting the cadmium removal of the adsorbent. Compared with the carbon materials modified [6, 35-37] by sulfuric acid or nitric acid, the surface of porous carbon materials also has acidic groups. However, the cadmium removal of porous carbon materials prepared by polyphenylene sulfide under the optimal conditions is obviously better than that of the carbon materials after acid modification, indicating that the performance is higher the sulfur content also has a certain influence on the cadmium removal of carbon materials [5, 38], which may be due to the weak Lewis acid-base interaction between sulfur and cadmium in solution.
| Carbon materials | Adsorption capacity (mg/g) | Cadmium removal (%) |
|------------------|---------------------------|---------------------|
| P-400            | 18.93                     | 75.73               |
| P-450            | 24.69                     | 98.74               |
| P-500            | 12.34                     | 49.36               |
| P-550            | 8.84                      | 35.43               |

3.3. Effect of adsorption conditions

3.3.1. Effect of temperature on Cd\(^{2+}\) removal percentage

The effect of temperature on the adsorption removal of Cd\(^{2+}\) is shown in Fig. 2. With the increase of temperature, the removal percentage of Cd\(^{2+}\) on porous carbon has little change. Besides, simple operation and energy saving should be considered in the actual production process[39]. Therefore, 35°C will be selected as the optimal adsorption temperature in the next step.

3.3.2. Effect of adsorbent amount on removal percentage of Cd\(^{2+}\)

The effect of the amount of adsorbent on the adsorption removal of Cd\(^{2+}\) is shown in Fig. 3. With the increase of adsorbent dosage, the removal percentage of Cd\(^{2+}\) on porous carbon increased. When the amount of adsorbent is greater than 0.100g, the removal percentage of Cd\(^{2+}\) on adsorbent basically unchanged.

3.3.3. Effect of contact Time

The effect of contact time on the adsorption removal of Cd\(^{2+}\) is shown in Fig. 4. Under other constant conditions (the dosage of porous carbon was 0.0500g and the temperature was 35 °C.). As shown in Fig. 4, with the increase of contact time, the removal rate of Cd\(^{2+}\) increased. When the time is 90 min, the removal reached the highest value, and with the extension of time, the removal rate of Cd\(^{2+}\) basically remained unchanged. In the initial stage, high concentration of Cd\(^{2+}\) and more adsorption sites are helpful for rapid adsorption. With the adsorption process going on, the concentration of Cd\(^{2+}\) and the surface sites of adsorbent decreased, which led to the slowdown of adsorption rate, and until the adsorption gradually reached equilibrium [40, 41]. Therefore, 90 min is the best adsorption time.
To sum up, when the concentration of Cd\(^{2+}\) is 250 ppm, the best adsorption conditions of Cd\(^{2+}\) are as follows: the adsorption temperature is 35 \(^\circ\)C, the amount of adsorbent is 0.1000g/25ml, and the adsorption time is 90 min. At this time, the maximum adsorption removal percentage of cadmium ion is 98.74%.

4. Conclusions

In general, in this work the sulfur-doped porous carbon materials prepared by using KOH to activate waste polyphenylene sulfide at 450\(^\circ\)C have the best adsorption performance of cadmium ion. When the concentration of cadmium ion was 250 ppm, the removal of cadmium was 98.74% under the optimal adsorption conditions (adsorption temperature 35 \(^\circ\)C, adsorbent dosage 0.1000g/25ml, adsorption time 90min). The total surface acid groups and the sulfur content of the materials were determined. It was found that the number of acid groups and the sulfur content had a close correlation with the removal of cadmium. The preparation of this material has the following advantages: cheap and a wide range of raw materials, simple process and high adsorption efficiency without special modification of material. Therefore, this material may be a promising adsorbent for the removal of cadmium ion from industrial wastewater.

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