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Preparation and Adsorption Properties of Sulfur-Nitrogen Co-Doped Activated Carbon

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Abstract

Chromium is one of the heavy metal pollutants. Heavy metal chromium-contaminated water will seriously endanger human health after use. There are many ways to remove chromium-containing sewage, and the adsorption method is the most effective and convenient method. The adsorption amount of traditional activated carbon is limited, so it needs to be modified to improve the adsorption rate. This experiment determined a reasonable modification method, and the calcination method was selected for the modification. This paper mainly compares the surface morphological characteristics of activated carbon before and after modification. The modified X-ray diffraction peak is increased and the infrared ray absorption peak increased, and the results show that the surface of the modified activated carbon is more rough than that of the modified activated carbon, the functional groups are increased, and the sulfur and nitrogen are doped on the activated carbon. Therefore, the modified activated carbon has a high removal rate and the best performance under acidic conditions.

Keywords

Sulfur-Nitrogen Co-Doped Activated Carbon

1. Introduction

The discharge industry of heavy metal sewage is increasing, so heavy metal pollution is becoming increasingly serious. Industries involved in the discharge of heavy metal sewage include metal smelting, petrochemical industry, mining, etc. [1], even some illegal enterprises have made the discharge problem of heavy metal sewage more serious. Human factors also increase the pollution of heavy metals. The wastewater containing heavy metals mainly comes from factories, smelting, metal processing and other processes [2]. However, there are many uncertainties

in the production process, and it is difficult for the staff to control the uncertain factors in the chemical reaction, so that the production indicators fluctuate, resulting in the emission of heavy metals exceeding the standard [3]. The main human factor is that the students negligence in the experiment, and the waste water containing heavy metals was not poured into the waste liquid bucket, and the sewer also caused the pollution of heavy metals. The heavy metal wastewater discharged flows into the river, and the river evaporinto rainwater containing heavy metals. The rain washing the ground causes heavy metal pollution in the soil and heavy metal pollution in the fields, causing serious damage to the ecological environment. These heavy metal pollution causes great damage to the human body and can cause death when serious [4]. Therefore, a series of effective methods are needed to treat the problem of heavy metals in the environment.

There are many heavy metals in the wastewater, among which chromium is one of the most important heavy metal pollutants [5]. There are many forms of chromium and chromium compounds in natural water environments. There are four valence states in the environment, including zero valence, positive bivalent, positive trivalent and positive hexadrivalent [6]. The most common is mainly hexavalent chromium and trivalent chromium. The trace elements necessary for the human body contain trivalent chromium, a small amount of trivalent chromium is harmless, and the metabolic process of the human body is essential [7]. There are researchers [8]. The presence of chromium was detected from various substances in the environment, and the presence of chromium was detected from the atmosphere, water, land, plants, etc. [9]. Although the industrial wastewater containing heavy metal chromium has been centrally treated in the factory, sometimes the by-products and derivatives of the production process are poorly understood, and the improper treatment makes the chromium discharged into the environment, and eventually leads to the serious pollution and destruction of the ecological environment.

Adsorption method is also a physical method, which is mainly conducted with adsorbent, usually choose large surface area, loose porous substances as adsorbent. Physical adsorption usually refers to that the adsorbent is adsorbed to the surface or inside of the adsorbent [10]. Common adsorbent mainly have activated carbon, silicic acid [11]. The earliest adsorbent is activated carbon, activated carbon operation is simple, high efficiency, but there are also some disadvantages, such as high cost, limiting factors, it is difficult to recycle [12] in order to improve efficiency, activated carbon fiber adsorbent [13]. Although this adsorbent is higher than the traditional adsorbent, its price is more expensive [14]. Studies have shown [15], organic toresin is an effective adsorbent, treating 1 L 10 mg/L of chromium containing wastewater, with about 5 g adsorbent can make the chromium content of chromium containing wastewater lower than 0.5 mg/L. Meet the national emission standards [16].

The adsorption method can be divided into physical adsorption method and chemical adsorption method. Physical and chemical adsorption method mainly

uses intermolecular physical and chemical action to remove heavy metal ions in wastewater [17]. Adsorbent generally have a large specific surface area, such as activated carbon, zeolite, molecular sieve, etc. Chu xiao medium [18] [19] with coconut shell as raw material, the high specific surface area of coconut shell activated carbon, this activated carbon can effectively remove hexavalent chromium. Wang Chunfeng *et al.* [20] NaA zeolite was synthesized from fly ash as raw material, which was effective for Cu (II), Cr (VI) and Zn (II).

Activated carbon is generally made of wood, fruit shell and other herbs into the processing and production, is a kind of uncertain carbon, the output of Chinas activated carbon increased year by year, ranking the second in the world [21]. Activated carbon has many holes of different sizes. According to the different sizes of pores, it can be divided into three types of large pores and micropores, among which determines the adsorption rate and plays the main role is the micropores. Large pores and medium pores are mainly to allow the adsorption material into the activated carbon. The structure of the hole mainly includes void volume, surface area and so on [22].

The study shows that the larger the surface area of activated carbon, the more voids, the more functional groups, and the stronger the adsorption capacity [23]. Activated carbon can effectively remove metals from water, but the traditional live

The specific surface area of sexual carbon is small, less functional groups and less micropores, so it is necessary to modify the activated carbon to effectively improve the performance of activated carbon. However, activated carbon has low chemical stability, low surface activity and easy conversion. An effective modification method is the calcination method. Heteroatom doping is a way to effectively change the surface properties of activated carbon, such as nitrogen, sulfur, boron, halogen, etc. [24] [25]. The introduction of nitrogen and sulfur atoms can form local functional groups on the surface of carbon materials, and the isolated electrons of nitrogen atoms can emerge from carbon sp^2. The hybrid structure separates the system with negative charge, improves the conductivity and changes the reactivity [26].

2. Experimental Materials and Analytical Methods

2.1. Test Reagents, Instruments and Equipment

The raw materials and reagents used during the experiment are shown in **Table 1**.

Table 1. Raw materials and reagents.

reagent name	molecular formula	reagent grade	manufacturer
acticarbon	/	AR	Tianjin Fuchen Chemical Reagent Factory
sulfocarbamide	CH_4N_2S	AR	Chengdu Cologne chemical reagent factory
ortho-phosphoric acid	H_3PO_4	AR	Urumqi City Chemical Co., Ltd
hydrochloric acid	HCl	AR	Urumqi City Chemical Co., Ltd
caustic potash	KOH	AR	Tianjin Fuchen Chemical Reagent Factory

Continued

parylene carbonyl dihydrazine (chromogen)	$C_{13}H_{14}N_4O$	AR	Aladdin Chemical Reagents Ltd
acetone	CH_3COCH_3	AR	Tianjin Fuchen Chemical Reagent Factory
bichrome	$K_2Cr_2O_7$	AR	Tianjin Fuchen Chemical Reagent Factory
caustic soda	NaOH	AR	Chengdu Cologne chemical reagent factory
sulphuric acid	H_2SO_4	AR	Urumqi City Chemical Co., Ltd

2.2. Main Test Instruments and Equipment

The instruments and analytical equipment used during the experiment are shown in Table 2.

Table 2. Instruments and equipment.

instrument name	specifications	manufacturer
analytical balance	GL2241SCN	Guangzhou Xinghua Technology Co., Ltd
pipe furnace	OTF-1200X	Hefei Kejin Material Technology Co., LTD
Electric heating constant temperature blast drying box	DHG-9101	Jiangsu Jinyi Instrument Technology Co., LTD
The constant temperature shake bed	THZ-103B	Shanghai Yiheng Instrument Technology Co., LTD
pH count	pHS-4F	Shanghai Electric Instrument Co., LTD
ultraviolet spectrophotometer	7230G	Guangzhou Xinghua Technology Co., Ltd
Gold spray instrument	JEC-3000FC	Japan Electronics Corporation
Field-emission SEM	D8 ADVANCE A25	Japan Electronics Corporation
X-X	IR Prestige-21	Shimadzu Ltd., Japan
infrared spectrometer	Bruker Vertex 70	Bruke Co., Germany

2.3. Material Characterization

1) Scanning electron microscopy (SEM)

During the SEM test, the powder sample was coated on the conductive adhesive and blown out with the ear washing ball. Then, the morphology and microscopic surface structure of the sample were observed at the voltage of 20 V and 20 μ A.

2) X-ray diffraction (XRD)

XRD testing was performed using Shimadzu IR Prestige-21. Sample crystal structure was analyzed by X-ray diffractometer. The rotating cathode is the Cu target K α-ray source with tube voltage 40 kV and current 40 mA. XRD scan range according to sample selection.

3) Infrared spectroscopy (FT-IR)

FT-IR spectroscopy test Brooker Fourier transform infrared spectrometer to analyze the functional groups contained in the sample. The powder sample was mixed with potassium bromide, fully ground, pressed, and placed into the sample

chamber to start testing in a range of 4000 - 500 cm^{-1}.

The solution containing heavy metal chromium adds nitric acid and sulfuric acid and reacts with the chromoagent (diphenyl dihydrazide), and the solution becomes purple. The maximum absorption wavelength is 540 nm. The upper limit of this method is, the minimum concentration is 1 mg/L, and the minimum amount is 0.2 μg.

The absorbance of the adsorption solution was added into the standard curve to obtain the solution concentration *Ce*. The calculation formula of the adsorption capacity of the sample is as follows: $Qe = \frac{(Co - Ce)V}{m}$

where: *Qe* is the equilibrium adsorption amount of the sample, mg/g;
Co and *Ce* are Cr () concentration before and after adsorption, mg/L;
V is the solution volume, mL;
and, *m* is the sample mass, g;

Cr () removal rate is calculated as follows: removal rate $(\%) = \frac{Co - Ce}{Co} \times 100\%$.

Among them: *Co* and *Ce* are the same as above.

2.4. Methods of the Adsorption Experiment

The modified activated carbon was added to a tapered bottle filled with chromium solution and put into a constant temperature shaker for 10 hours. After the full reaction, the reacted solution took the filtrate from a syringe and determined the hexavalent chromium ion content. From the three variables of adsorbent dose and pH, the effect of different factors on the effect of hexavalent chromium ions of modified activated carbon in water was studied, and the adsorption capacity of modified activated carbon was explored. The comparison between the front and back of adsorption is shown in Figure 1.

Figure 1. Flow chart of comparison before and after adsorption.

2.5. Preparation of the Modified Activated Carbon

Mix 5 ± 0.01 g activated carbon (powder), thiourea 5 ± 0.01 g and potassium hydroxide 5 ± 0.01 g for one hour and place in tube furnace 500˚C for calcination for more than two hours. After the temperature is reduced, 10 percent hydrochloric acid is added. After the acid solution, wash it to neutral and place it in the

electric thermal constant temperature blast drying box. After the sample is dried, put it into the sample bag for backup.

2.6. Solution Configuration

2.6.1. Configuration of the Hexavalent Chromium Solution

Cigh potassium dichromate ($K_2Cr_2O_7$, Analytical pure) 0.2829 ± 0.0001 g, dissolved in a beaker with distilled water and moved into a 1000 mL volumetric flask, and diluted to 1000 mL with distilled water. This solution is a 100-mg/L hexavalent chromium solution.

2.6.2. Allocation of the Color Developer

Weigh 0.2 ± 0.0001 g of diphenyl dihydrazine in 50 mL of acetone into a brown volumetric flask, add distilled water and dilute to 100 mL, shake well and store in a 4˚C thermostatic refrigerator.

2.6.3. Allocation of the Phosphoric Acid

Transfer 50 mL of phosphoric acid solution into a 100 mL volumetric flask with distilled water to 100 mL.

2.6.4. Configuration of Sulfuric Acid

Transfer 50 mL of sulfuric acid solution to a 100 mL volumetric flask to 100 mL.

2.7. Experimental Methods

1) Effect of initial chromium concentration on adsorption

Transfer a 200 mg/L hexavalent chromium solution of 25 mL, 50 mL, 75 mL, 100 mL, and 125 mL into a 250 mL volumetric flask, and adjust it to 250 mL at 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L, and 50 mg/L, respectively. Transfer 30 mL of chromium solution at 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L and 50 mg/L each to the taical flask, and add 0.05 g of adsorbent in a thermostatic shaker for 25˚C for 6 hours. Take out the conical bottle, take the filtrate, according to the determination principle of hexavalent chromium to measure the absorbance, this experiment using diphenyl spectrophotometry of hexavalent chromium content, adsorption of hexavalent chromium solution diluted one hundred times, take 0.1 ml of adsorption solution to 10 mL of color tube, after volume to add 0.5 mL 1:1 phosphoric acid and sulfuric acid, then add 2 mL color agent. The absorbance was measured by a visible spectrophotometer at 540 nm, and then the adsorbed concentration was calculated.

2) The effect of the injection amount of the adsorbent on the adsorption performance

The amount of adsorbent was 10 mg, 20 mg and 30 mg, the chromium concentration of 20 mg/L was transferred to the conical flask, 10 mg, 20 mg and 30 mg adsorbent were added to the conical flask for three sets of parallel tests, and then the conical flask was placed in a constant temperature shaker for 25˚C for 10 hours. Take out the conical bottle, take the filtrate, measure the absorbance according to the determination principle of hexavalent chromium, and calculate the

chromium concentration.

3) Effect of pH on the adsorption properties

The chromium solution with a concentration of 20 mg/L was transferred into the tapered flask, 10 mg adsorbent was added, and pH was adjusted to 2, 4, 6, 8 with HCl and NaOH, and three sets of parallel tests were performed. The conical bottle was placed in a temperature shaker for 25˚C for 10 hours. Take the filtrate, measure the absorbance according to the determination principle of hexavalent chromium, and calculate the chromium concentration. The experimental results show that Cr () from activated carbon is mainly removed by electrostatic adsorption, and the increase of pH will reduce the adsorption effect.

3. Test Data and Analysis

3.1. X-Ray Diffractometer (XRD)

The principle of X-ray diffraction is used to analyze the composition of the substance, and the composition of the substance is used to analyze the properties of the adsorbent. According to Figure 2, four obvious diffraction peaks, respectively at 13˚, 21˚, 27˚ and 29˚, were found, indicating that the activated carbon contains impurities, and the modified X-ray diffraction spectrum is seriously broadened, indicating that the crystallization of the sample is poor, and most of them are amorphous.

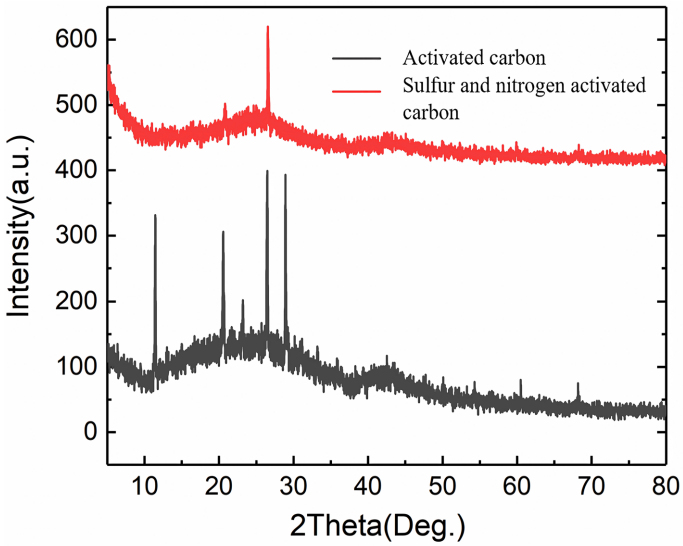

Figure 2. X-ray diffraction pattern of codoped activated carbon and sulfur and nitrogen.

3.2. Fourier Transform Infrared Spectrometer (FT-IR)

Infrared spectroscopy (IR) is an effective method for analyzing chemical functional groups. Black activated carbon is easy to absorb infrared light. The infrared spectrometer is mainly used to analyze the functional groups of the samples to be tested. According to the measurement, it can be seen from Figure 3 that the functional groups before the modification mainly include hydroxyl group, carbon-

hydrogen bonds, carbon and oxygen bonds, etc., and the modified adsorbent has an absorption peak at 3400 and a hydroxyl group, still at 2930, 1100, 960 and 650 cm^{-1}. There are infrared absorption peaks, in addition to the functional groups contained in the activated carbon before modification, there are carboxylate and other functional groups after modification [23].

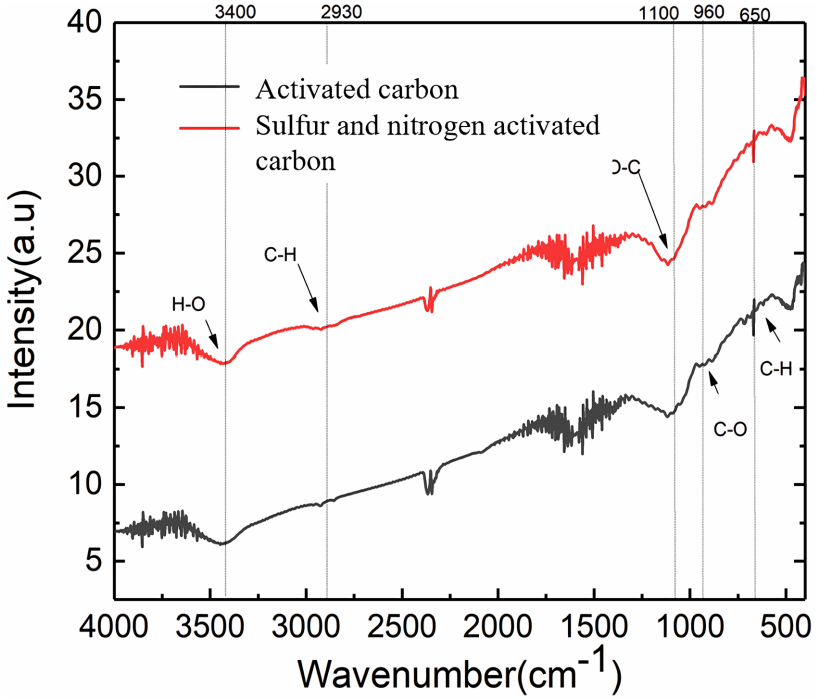

Figure 3. Infrared ray diffraction pattern of activated carbon and sulfur and nitrogen cod-oped activated carbon.

3.3. Scanning Electron Microscope (SEM)

From Figure 4, we can see the surface of activated carbon is smooth, enlarge ten thousand times can see activated carbon surface contains some functional groups and activated carbon pore, activated carbon contains a lot of carbon microspheres, gap small spherical degree is higher, relatively uniform distribution, carbon microsphere size mainly concentrated between 1 - 10 μm, the smallest ball diameter of about 1 μm, and the ball diameter of 5 μm ball distribution most.

This Figure 5 shows that the sulfur and nitrogen are attached to the surface of the activated carbon. By a can see the modified activated carbon surface is very rough, magnified by Figure 5(b) ten thousand times, the modified activated carbon surface attached to some small particles compared with Figure 4(b) is more rough, by c can clearly see the modified activated carbon surface attached with many functional groups, visible sulfur nitrogen has doping to the surface of activated carbon, and not modified the modified adsorbent more rough, surface more functional group, pore is more, so the modified activated carbon adsorption effect is better. From Figure 6, we can see that sulfur and nitrogen are doped on the surface of activated carbon.

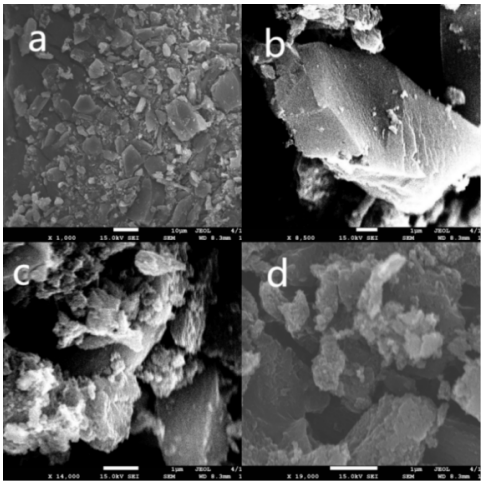

Figure 4. Chart of activated carbon of SEM.

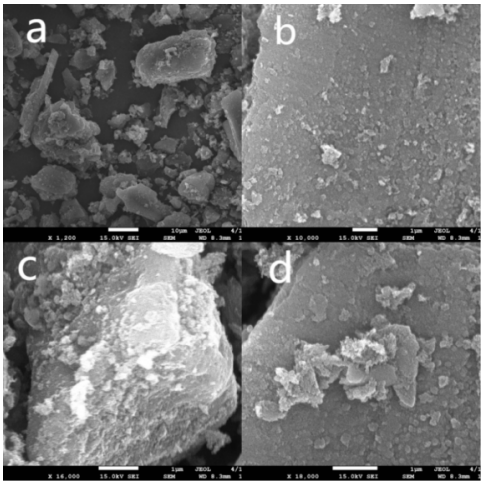

Figure 5. Chart of sulfur nitrogen activated carbon of SEM.

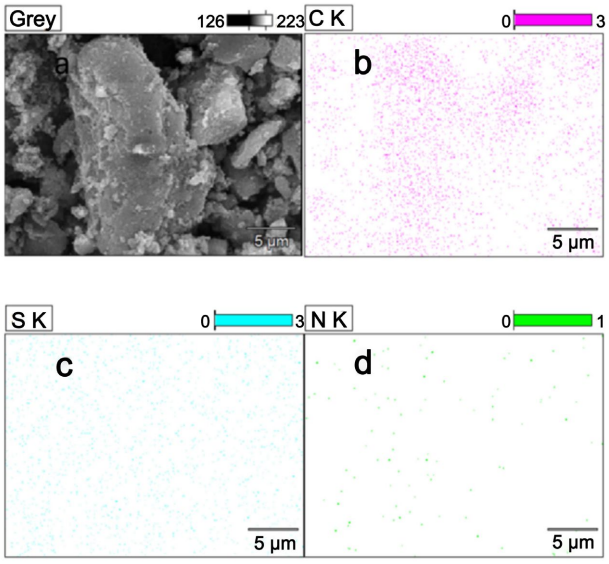

Figure 6. Chart of sulfur nitrogen activated carbon.

3.4. Analysis of the Results

According to the calculation, the removal rate reached 80% to 90% at the initial concentration of 10 mg/L, 60% at the initial concentration of 20 mg/L, 40% and 20% at 50 mg/L, respectively. Since the removal rate is the largest when the initial concentration of chromium solution is 10 mg/L, the adsorption capacity is the minimum when the initial chromium concentration is the lowest, the maximum adsorption capacity is about 19 mg/g and the minimum is about 5 mg/g, as shown in Figure 7.

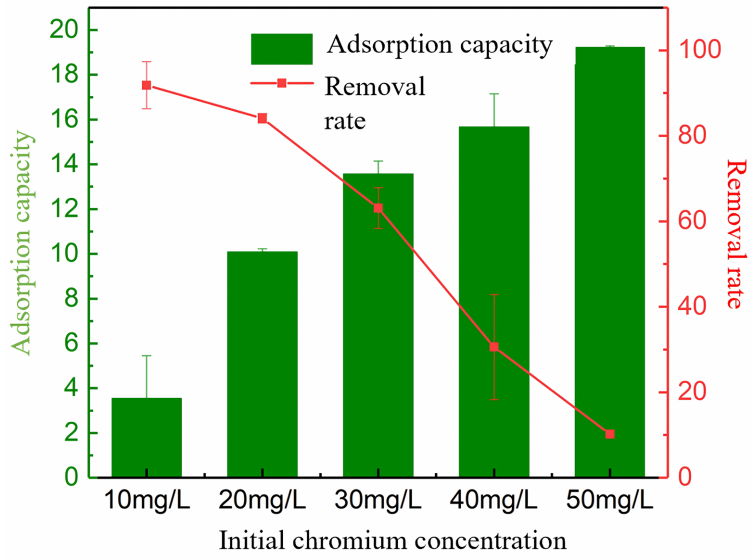

Figure 7. Chart of intial concentration of adsorption capacity and removal rate.

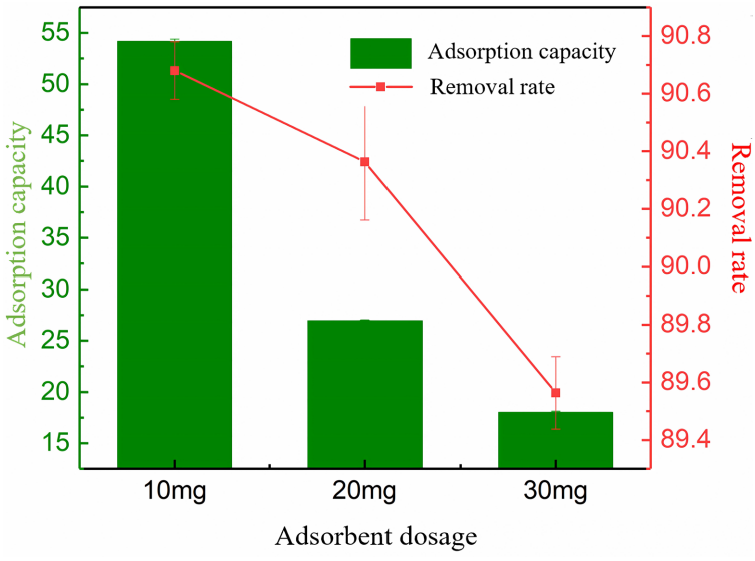

Figure 8. Chart of effect of dosage of adsorbent of adsorption capacity and removal rate.

It can be seen from Figure 8 that when the adsorbent is 10 mg, the removal rate and adsorption capacity are the largest, and the removal rate is 90% and the

adsorption capacity is about 54 mg/g. When the addition amount is 30 mg, the removal rate is about 89%, the adsorption capacity is about 16, the addition amount is 10 mg, 20 mg, and the removal rate of 30 mg is not fluctuating.

From Figure 9, we know the effect of pH on adsorption capacity and removal rate, and the size of pH is inversely proportional to the adsorption effect, and the adsorption effect is best when pH is 2.

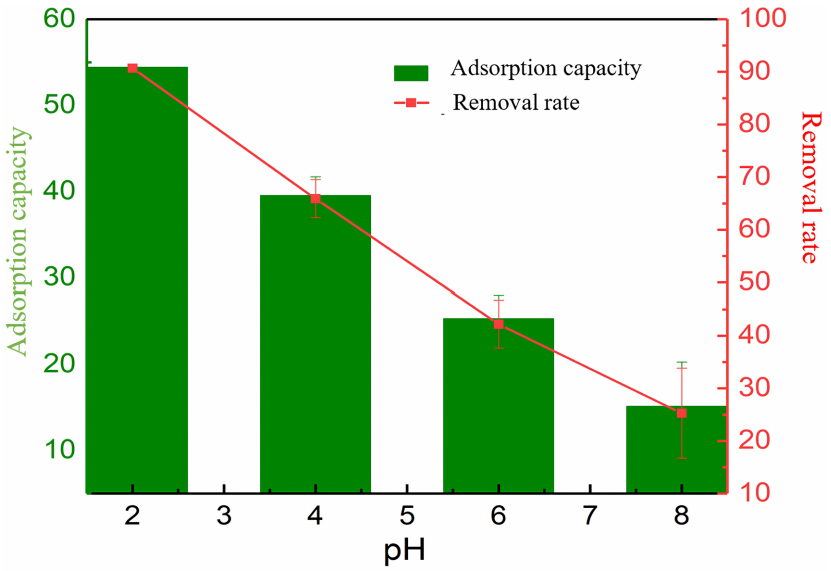

Figure 9. Chart of effect of pH on adsorption capacity and removal rate.

4. Conclusions

The modified activated carbon by X-ray derivative peak increases that sulfur and nitrogen are doped to the activated carbon, and the modification of adsorbent is successful. From the infrared ray absorption peak, we know the functional groups and the modified functional groups. The modified adsorbent is conducive to the adsorption of hexavalent chromium in water. It can be seen from the scanning electron microscope graph that the surface of activated carbon is smooth. By 10,000 times, the surface of activated carbon contains some functional groups and activated carbon pores, and activated carbon contains many carbon microspheres. The surface of the modified activated carbon is rougher, with more carbon microspheres, more micropores, and fewer large pores. Because the micropores play a major role in the adsorption process, the modified adsorbent is more conducive to removing the hexavalent chromium in the water.

According to the modified activated carbon adsorption experiment, when the amount of adsorbent is the same, the lower the initial concentration of chromium, the higher the removal rate, the higher the initial concentration of chromium, the greater the adsorption capacity. When the initial concentration of chromium is 10 mg/L, the less the adsorbent dose, the higher the removal rate, the greater the adsorption capacity, the highest removal rate is 89%, and the adsorption capacity is 59 mg/g. The best adsorption effect at pH 2.

Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

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