

# Adsorption Properties of Chitosan Modified Diatomite for Anionic Dye Reactive Red 120

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## **Abstract:**

Water pollution has become a problem we need to be addressed. A large number of printing and dyeing wastewater was discharged into the environment, polluting water resources. In this study, sodium dodecyl sulfate and chitosan were used as modifiers to modify the surface of diatomite to prepare chitosan modified diatomite, which was used to treat printing and dyeing wastewater of containing reactive red 120. The physical and chemical properties of diatomite modified by sodium dodecyl sulfate and chitosan were studied and characterized by a specific surface area analyzer and FT-IR. The effects of the dosage of chitosan modified diatomite, adsorption time, pH and the concentration of printing and dyeing wastewater on the adsorption were studied. The results showed that sodium dodecyl sulfate and chitosan could modify diatomite effectively. Chitosan modified diatomite could effectively improve the treatment effect of printing and dyeing wastewater compared with diatomite. When the dosage of chitosan modified diatomite was 2g/L, the adsorption time was 110 min, and the pH value was between 5 and 6, the removal rate of reactive red 120 was more than 99%.

**Keywords:** Chitosan, Diatomite, Adsorption, Printing and dyeing wastewater.

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## I. INTRODUCTION

Industrial wastewater had become an urgent problem to be solved. In all kinds of industrial wastewater, printing and dyeing wastewater was an important part of China's industrial wastewater [1-4]. According to the relevant statistics show that China's annual discharge of printing and dyeing industrial wastewater more than 900 million tons, large emissions and great harm to the water body. However, the reuse rate of dye wastewater was very low, less than 7%, there are many dye wastewater had not been effectively purified [5-7]. Directly into the natural waters. It had caused great damage to the environment. Wastewater of printing and dyeing mainly comes from bleaching, mercerizing and printing processes. Due to the different tasks of each process, the types of dyes used are also different. Printing and dyeing wastewater had the characteristics of high chromaticity, high toxicity, difficult biodegradation, complex components and the like. This could reduce the photosynthesis of the water

environment when the wastewater was discharged into a water body [8-12]. This further increased the treatment difficulty of dye wastewater [13-17]. Due to the characteristics of dye wastewater, the treatment of dye wastewater usually needed to combine a variety of treatment methods according to the characteristics of different dye wastewater [18-23]. At present, the commonly used industrial methods to deal with wastewater were biological method, chemical oxidation and so on [24-26].

Adsorption was a common treatment method, which has many advantages, low cost, strong recovery ability and so on. The adsorption method was mainly based on electrostatic attraction and the formation of chemical bonds between functional groups on the surface of various modified adsorbents and dyes [27-29]. Various dye substances in the solution were adsorbed and the pollutants in the water were finally removed by a solid-liquid separation method [30-32]. At present, the adsorbents for treating printing and dyeing wastewater mainly include zeolite, chitosan, activated carbon, alumina and the like. Among these adsorbents, chitosan had a greater potential for development, which was mainly determined by the structural characteristics of chitosan itself [33-36]. The amino of chitosan could be protonated, so that chitosan had a positive charge. It was very suitable for the treatment of anionic dye wastewater [37-39]. In addition, chitosan also had the characteristics of biodegradable, non-toxic, low cost and wide source. However, the adsorption capacity and application range of chitosan were limited.

In recent years, diatomite in wastewater treatment, showing a unique advantage and better treatment effect. The main chemical composition of diatomite was amorphous  $\text{SiO}_2$ . Diatomite was a porous biological silicalite. Diatomite had many excellent properties, such as porosity, light weight and large specific surface area. These excellent characteristics were very conducive to the adsorption of adsorbate [40-43]. In this paper, sodium dodecyl sulfate and chitosan were used as modifiers to modify the surface of diatomite, and finally chitosan modified diatomite was prepared. The surface of diatomite was activated by anionic surfactant sodium dodecyl sulfate, which could increase the surface electronegativity of diatomite, and then increased the adsorption sites of chitosan. The main functional groups of chitosan were  $\text{C}_2\text{-NH}_2$ ,  $\text{C}_3\text{-OH}$  and  $\text{C}_6\text{-OH}$ , and the amino groups of chitosan could be protonated, which could make the surface of diatomite had positively charged. And provide a new idea for effectively developing and utilize diatomite.

## II. MATERIALS AND METHODS

### 2.1 Materials

Diatomite (Linjiang Co., Ltd., China). Chitosan (Shanghai Zhanyun Chemical Co., Ltd.). SDS (Guangfu Co.,Ltd.). Reactive red 120 (Tianjin Fuchen Chemical Reagent Co.,Ltd.). NaOH and HCl were supplied by Beijing Guangfu Chemical Reagent Co.,Ltd.

## 2.2 Preparation of Chitosan Modified Diatomite Adsorbent

Diatomite (10 g) was put into a 500mL beaker. And then, 300mL of deionized water was added to the beaker. After full stirring, a certain proportion of SDS was added to the beaker, and then, the pH of the previous mixture was adjusted to 1.0 with a 0.1 mol/L HCl solution. The previous mixture was stirred using a magnetic stirrer at room temperature for 5 h. Subsequently, the mixture was centrifuged for 0.5 h, and the supernatant was removed. The mixture was centrifuged for another 0.5 h, and the supernatant was removed. The precipitate was washed six times with deionized water. Subsequently, the precipitate was dried at 55°C and passed through a 200 mesh sieve; thus, the complex (SDS-M) of diatomite and SDS was obtained. 10 g of SDS-M was added to 150mL the chitosan solution with a concentration of 20 g/L. The mixture was stirred at room temperature for 5 h and soaked 1 h. Subsequently, the mixture was soaked in sodium hydroxide solution for 24h. Then, the mixture was washed with deionized water until neutral, dried at 80°C, and passed through a 200 mesh sieve; Thus, chitosan modified diatomite adsorbent was obtained.

## 2.3 Characterization of Chitosan Modified Diatomite

Diatomite and chitosan modified diatomite were characterized using UV-2602 UV-visible spectrophotometer, V-SORB2800 type specific surface area analyzer, IRAffinity-1 FTIR spectrophotometer and Zeta potentiometer.

## 2.4 Adsorption Experiments

A certain amount of diatomite and modified diatomite were respectively put into conical flask. 100mL of 50 mg·L<sup>-1</sup> printing and dyeing wastewater (prepared by using reactive red 120) were respectively added to the conical flask. The mixture was shaken in a shaker at 25°C and 200 R·min<sup>-1</sup>, and then the supernatant was taken by centrifugation. The absorbance of the dye was measured at the maximum absorption wavelength  $\lambda_{\max}$  (Reactive Red 120  $\lambda_{\max}$  was 534 nm), the adsorption capacity was calculated according to the standard curve equation, and the removal rate was calculated according to the formula (1).

$$R = (C_0 - C) \times 100\% \times 1/C_0 \quad (1)$$

R is the removal rate,  $C_0$  is the concentration of dye wastewater before treatment (mg/L), and C is the concentration of dye wastewater after treatment (mg/L).

## 2.5 The Determination Method of reactive red 120 concentration

In this experiment, the concentration of anionic dye reactive red 120 in dye wastewater was detected by UV-2602 spectrophotometer. A standard curve of the dye wastewater was made, and then the concentration of the anionic dye reactive red 120 in the solution to be detected is detected by using UV-2602 ultraviolet-visible spectrophotometer in combination with the standard curve.

## III. RESULTS AND DISCUSSION

### 3.1 Characterization of Chitosan Modified Diatomite

The modified diatomite was characterized. The results were shown in Table I and Figure 1 below.

**TABLE I. The physical properties of different diatomite**

ADSORBENT	SPECIFIC SURFACE AREA ( $M^2 \cdot G^{-1}$ )	ZETA POTENTIAL (MV)	THE AVERAGE PORE DIAMETER (NM)
DIATOMITE	30.08	-35	9.52
CHITOSAN MODIFIED DIATOMITE	42.26	27.6	7.25

The change of diatomite was characterized with BET. Specific surface area of chitosan modified diatomite had increased compared with diatomite, this was mainly because that the diatomite was washed many times in the process of pretreatment modified with SDS. So this could increase the specific surface area. On the other hand, chitosan molecular chain could adsorbed into the micropore and surface of the diatomite particles, it could form more micropore, which increased the specific surface area of the chitosan modified diatomite. Average pore size was slightly decreased. Zeta potential of chitosan modified diatomite was increased mainly due to the protonation of the amino group on chitosan. It was very beneficial to the absorption of the anionic dye.

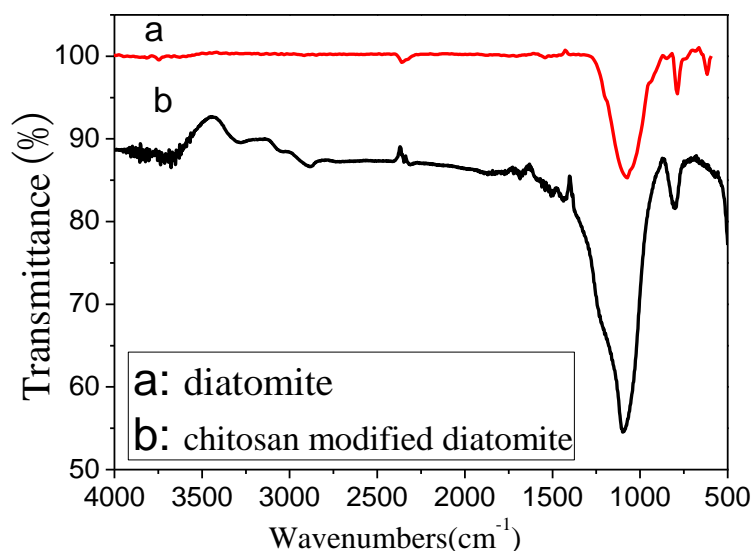


Fig 1: FT-IR spectra of diatomite (a) and chitosan modified diatomite (b)

As Figure 1 shows, the peak at  $1085\text{cm}^{-1}$  was Si-O stretching Vibrations, it was the peak of diatomite. The peak at  $750\text{cm}^{-1}$  was attributed to Si-O-Si stretching vibrations. The peak at  $510\text{cm}^{-1}$  was due to Si-O-Si antisymmetric stretching vibrations. Compared with the diatomite, DTA showed new characteristic peaks at  $2851\text{cm}^{-1}$  and belonged to the  $-\text{CH}_2$  in the SDS alkyl chain. It indicated that SDS had inserted into the pore and surface of diatomite. The peaks at  $1680\text{cm}^{-1}$  (amide bond I),  $1501\text{cm}^{-1}$  (amide bond II) and  $1380\text{cm}^{-1}$  (amide bond III) were the main characteristic peak of chitosan [44-46]. In addition, the peak at  $3395\text{cm}^{-1}$  belonged to the O-H stretching vibration in the chitosan. The peak at  $2875\text{cm}^{-1}$  belonged to the C-H stretching vibration in the chitosan. These characteristic peaks showed that chitosan had inserted into the pore or surface of diatomite. The main functional groups of chitosan are  $\text{C}_2\text{-NH}_2$ ,  $\text{C}_3\text{-OH}$  and  $\text{C}_6\text{-OH}$ . Nitrogen atoms of  $\text{C}_2\text{-NH}_2$  group have lone pair electrons, which can enter the vacant orbital of metal ions and form coordination bonds. So it can improve the removal rate of heavy metal ions

### 3.2 Effect of Concentration of Solution on Chitosan Modified Diatomite Adsorption Capacity of Reactive Red 120

100mL of the reactive red 120 dye solution with different concentrations were prepared respectively. 0.2g of the chitosan modified diatomite or diatomite were added to the reactive red 120 dye solution with different concentrations respectively. The mixture were oscillating at constant temperature and constant speed in a water bath oscillator. A sample was centrifuged after adsorption equilibrium, and the dye concentration of the supernatant was measured using an ultraviolet spectrophotometer. The result was shown in Figure 2.

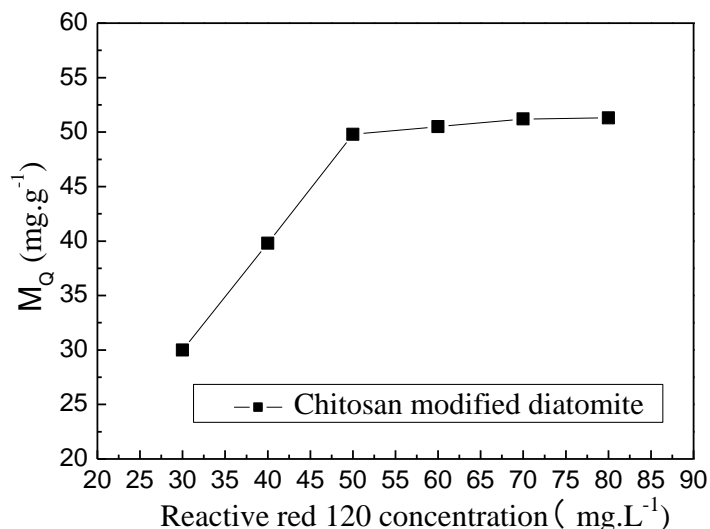


Fig 2: Effect of initial concentration of reactive red 120 on adsorption capacity

The adsorption capacity increased with the increase of adsorption time when the concentration of reactive red 120 solution was less than 50 mg/L in Figure 2. The maximum adsorption capacity of chitosan modified diatomite did not reach when the equilibrium adsorption concentration was reached. But the increasing trend of the adsorption amount became very gentle when the concentration of the reactive red 120 dye solution exceeded 50mg/L. The adsorption rate on the dye changed from fast to slow and had reached the equilibrium adsorption concentration. Reaching the maximum adsorption amount of the chitosan modified diatomite. If the concentration of reactive red 120 dye solution was further increased, not only the adsorption capacity would not be increased, but also the ions adsorbed to the chitosan modified diatomite may be affected due to electrostatic repulsion. The main reason was that the concentration of dye and the sites on the surface of chitosan modified diatomite changed. The outer surface of the chitosan modified diatomite was a chitosan molecule with positive charge. Nitrogen atoms in the structure of the chitosan modified diatomite could form hydrogen bonds with amino groups in anionic dyes. Strong electrostatic attraction was generated between the nitrogen atoms and the anionic dyes. After the adsorption of the outer layer was saturated, part of the adsorbed dye would diffuse into the interior of chitosan modified diatomite. Reactive red 120 would be adsorbed by in the chitosan modified diatomite interstice.

### 3.3 Effect of Dosage of Chitosan Modified Diatomite on Adsorption

Seven Portions of 100 mL 50 mg/L reactive red 120 solution were prepared and put into seven erlenmeyer flasks. 0.04 g,0.08 g,0.12g,0.16g,0.20g,0.24g and 0.28g of chitosan modified diatomite were

added into the seven erlenmeyer flasks respectively, and then adsorbed by shaking in a shaker at 25°C for 4 h. Samples were centrifuged. The concentration of the reactive red 120 solution in the supernatant was measured to determine the removal rate. Then 7 portions of 100 mL of reactive red 120 solution with the mass concentration of 50 mg/L were prepared. Diatomite was added. The addition amount and the treatment process were the same as those of the chitosan modified diatomite. The results were shown in Figure 3.

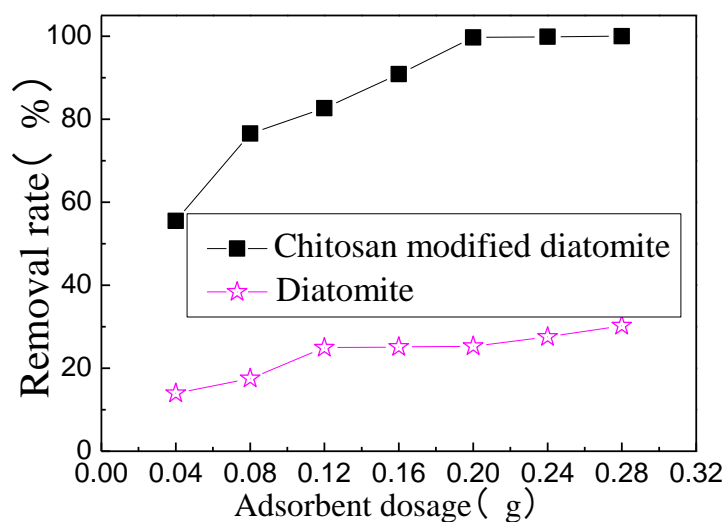


Fig 3: Effect of the amount of diatomite and chitosan modified diatomite on the removal rate

As shown in Figure 3. The removal rate of anionic dye reactive red 120 by chitosan modified diatomite was significantly higher than that by diatomite compared with diatomite. Because reactive red 120 itself was negative, while the surface of unmodified diatomite was negative in water due to its special structural characteristics, and the Zeta potential was negative. This leads to a certain degree of mutual repulsion between reactive red 120 and anionic dyes, which was not conducive to the adsorption of anionic dyes reactive red 120. The removal rate of anionic dye reactive red 120 was also improved with the increase of the dosage of chitosan modified diatomite. Because the adsorption sites of anionic dye reactive red 120 would increase with the increase of the dosage of chitosan modified diatomite, and the adsorption capacity would also increase. The detection results shown in Table 1. The surface electric property of the diatomite was changed, and the original electronegativity of the diatomite modified by the chitosan was changed into the electronegativity due to the amino group on the chitosan. The amino group could be protonated to be positively charged, thereby being easy to generate electrostatic attraction and combination with the anionic group in the dye. The adsorption effect of the diatomite on the anionic dye active red 120 was greatly improved. When the dosage of chitosan modified diatomite was less than 2g/L. The removal rate of anionic dye reactive red 120 increased rapidly with the increase

of the dosage of chitosan modified diatomite. When the dosage was more than 2g/L, the increasing trend of the removal rate became gentle and was close to 100%.

### 3.4 Effect of pH on Adsorption of Chitosan Modified Diatomite

Take 100mL of 50mg/L reactive red 120 solution. The pH of the solution were adjusted respectively, 0.20g of chitosan modified diatomite or 0.4g of diatomite was added into the corresponding conical flasks respectively. The solution was vibrated and adsorbed for 110min in a shaker at room temperature, and centrifuge for separation. The reactive red 120 removal rate was correspondingly determined. The results were shown in Figure 4.

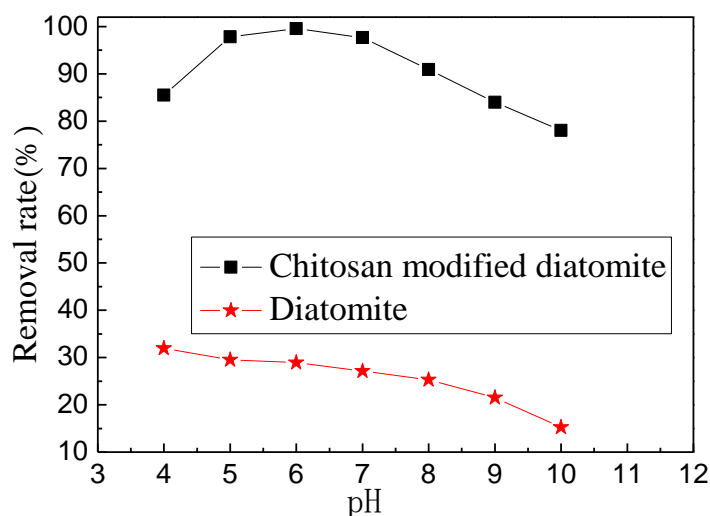


Fig 4: Effect of pH on removal efficiency

In Figure 4. The removal rate of reactive red 120 by chitosan modified diatomite was also increased with the increase of pH value when the pH was less than 6. When the pH value was greater than 6, the increase trend of the removal rate became slow. This was mainly due to the fact that free amino groups on chitosan molecular chains are combined with hydrogen ions to form  $\text{-NH}_3^+$  in an acidic solution. The excess hydrogen ions competed with the  $\text{-NH}_3^+$  on the molecular chain of chitosan when the pH value of reactive red 120 solution was low. This was not conducive to the adsorption process. When the pH value of the chitosan solution was more than 6, the free amino groups on the molecular chain of the chitosan could not be fully protonated, so that the Zeta potential of the surface of the modified diatomite was reduced. The positive charge was reduced, and the electrostatic attraction capacity was lowered, so that the adsorption process was not favorable. When the pH was less than 6, most of the free amino groups were protonated, which was beneficial to improve the adsorption of chitosan modified diatomite to the



reactive red 120. With the increase of pH, the adsorption effect of diatomite on reactive red 120 increased slightly in the early stage, and then gradually decreased. This was mainly because the mutual repulsion between diatomite and reactive red 120 was reduced at the initial stage due to the low pH of the wastewater liquid. However, the surface electronegativity of diatomite was further increased with the increase of pH. The mutual repulsion between diatomite and reactive red 120 was increased, which was not conducive to the adsorption process. However, the removal rate of reactive red 120 by chitosan modified diatomite was significantly improved compared with diatomite. The pH should be controlled between 5 and 6, which was more conducive to the adsorption of reactive red 120.

### 3.5 Effect of Adsorption Time on Adsorption Efficiency of Chitosan Modified Diatomite

100 mL of reactive red 120 solution with the concentration of 50 mg/l were prepared, 0.20g of chitosan modified diatomite or 0.4 g of diatomite was added into the corresponding conical flasks, and the solution was vibrated and adsorbed in a shaker at room temperature for different time. The solution were centrifuged, and the reactive red 120 concentration in the supernatant was measured to determine the removal rate. The results were shown in Figure 5.

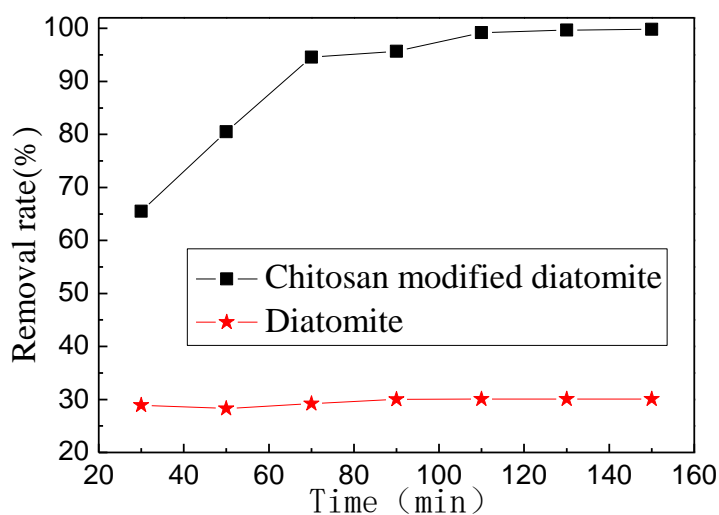


Fig. 5: Effect of Adsorption Time on Removal Rat

As can be seen from Figure 5. In the initial stage of adsorption, the adsorption speed of reactive red 120 by chitosan modified diatomite was faster when the adsorption time less than 70 min. The removal rate was obviously improved. In the middle period of adsorption, the adsorption rate slowed down. The removal rate of reactive red 120 increased slowly between 70 min and 110 min. When the adsorption time was 110 min, the removal rate of reactive red 120 was close to the highest value. When the

adsorption time was more than 110 min, the removal rate of reactive red 120 increased slowly. This was mainly because that reactive red 120 could be quickly adsorbed into the micropores of chitosan modified diatomite by electrostatic attraction in the initial stage of adsorption. In the middle stage of adsorption, with the adsorption process, the adsorption space became smaller when the micropore adsorption capacity of chitosan modified diatomite gradually increased. The electrostatic attraction also weakened, so the removal rate of reactive red 120 increased slowly. In the later period of adsorption, the adsorption was close to saturation. The removal rate of reactive red 120 was more than 99%. Therefore, increasing the adsorption time had little effect on the removal rate of reactive red 120.

#### IV. CONCLUSION

Diatomite was modified by chitosan and sodium dodecyl sulfate. FT-IR analysis showed that chitosan and sodium dodecyl sulfate modified diatomite effectively. Compared with diatomite, the specific surface area and Zeta potential of chitosan modified diatomite increased. Chitosan modified diatomite could effectively improve the removal rate of reactive red 120 in printing and dyeing wastewater. When the dosage of chitosan modified diatomite was 2g/L, the solution pH was between 5 and 6, and the adsorption time was 110 min, the removal rate of reactive red 120 was the best.

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