Effect of Reaction Temperature on Adsorption Efficiency using Computer Mathematical Statistics Visible Spectorphotometer

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Abstract. In this paper, waste shrimp shells extracted from the head of the chitosan material; static adsorption experiments manner by wastewater containing Fe(III) added chitosan, vis spectrophotometer absorbance before and after the measurement experiment was obtained by reacting the size of the metal ion concentration, adsorption conditions whereby chitosan Fe(III) and the ability to explore. Experiments show that. In the case where the reaction temperature is less than 55℃, chitosan has adsorption rate Fe(III) smaller rise, the optimum temperature was 55℃, but the effects on the reaction temperature adsorption rate is not large; Adsorption when control time 30 min to 50 min, the absorption effect is increased with increase in the time, to reach the optimal reaction time 50 min.

Keywords: Chitosan, temperature, adsorption.

1. Introduction
Since China's reform and opening up, with the rapid development of chemical industry, chemical industry to contain heavy metal raw material usage is becoming more and more popular, the result is the generation of heavy metal wastewater is also more and more [1], the main sources of waste water containing iron ore refining, electroplating process and separation process, etc., while iron of biological toxicity is not large, However, when the content of iron compounds in wastewater reaches a certain value, it is easy to appear abnormal water color and emit special odor, which will affect the sensory properties of water environment. According to the relevant departments to statistics [2], our country is water accounted for more than four 5 of heavy metal pollution of heavy metals has the characteristics of difficult to biodegradation, enter human body with protein and enzyme reaction, make its active disappear or gathered in the human body, which can lead to such as headache, neurasthenia and diseases such as malignant tumor, as the hidden killer of human health, therefore, The disposal of heavy metal wastewater is urgent. Adsorption method is very common in the treatment of heavy metal wastewater. Compared with the traditional method of heavy metal wastewater, adsorption method is easy to operate, widely used and easy to recover pollutants and other advantages, and is becoming a more common method in the treatment of heavy metal wastewater [3].

In terms of the preparation of chitosan, the preparation methods of chitosan mainly include physical method, acid-base method, EDTA method and lye method. With the rise of the concept of
energy conservation and environmental protection, research shows that microwave treatment can improve the reaction efficiency, reduce energy consumption, improve the quality of finished products and enhance the reaction activity. Ju Hongfang [4] used several different crab shells to extract chitin, and then used microwave radiation technology to deacetyl the chitin, improved the extraction method of chitosan, and finally prepared high quality chitosan. The results show that under certain conditions, the degree of deacetylation can reach 86.1% by microwave initial treatment for 20 min, which is more effective than other methods. Compared with the traditional method, the production process of chitosan prepared by this method is more simplified, more energy saving and less consumption, and the reaction time is shortened to a great extent. However, this method has some limitations, such as low reaction time and temperature threshold, which makes it difficult to control the reaction conditions. Once the operation is slightly careless, the reaction will fail. Microbial treatment, Liu Zhenli [5] to explore the nutrition in fermentation matrix in different nitrogen source, carbon source, inorganic matter on refined radiation sporanox effects of chitosan, take only one factor to design experiment and response surface method, the control to the medium corn flour and corn slurry additive quantity proportion, to explore the best effect of dosing ratio, The process of preparing chitosan deacetylation from chitin was optimized and deleted to reduce the pollution to the environment during the preparation process. Compared with the optimized medium, the yield of chitosan increased by 64.30%, which had the advantages of high quality and simple operation. However, the processing technology of the raw materials of this method is relatively complex, and the cultivation conditions of the strains are also relatively harsh, so there is no condition for large-scale application at present.Huang Daqing [6] chitosan as adsorbent, water adsorption effect of chitosan copper printed circuit board abandoned removed to produce ions, lead ions and zinc ions, in single factor pH, temperature, chitosan The dosage and other variables are variables, and the optimal reaction conditions are obtained; research shows that the appearance of heavy metal ions and the external charge of chitosan are changed due to the influence of pH, which greatly affects the adsorption results of the adsorbent.

2. Test portion

2.1. Instruments and reagents

Instrument part:

Vis: 722s, Shanghai Electronics Co., Ltd. Analytical Instruments; electronic balance ALC-210.4, Guangzhou Branch Instrument Co., Ltd. granted; Drying oven: 101-0, Beijing Kewei Instrument Co., Ltd. Yongxing centrifuge ; low-speed large capacity centrifuge: TDL-40B, Shanghai Anting scientific instrument Factory; multi-purpose high-speed mill: HL-FS500, medical equipment, Ltd. Henan Hua Kanghong; collector constant temperature heating magnetic stirrer: DF-101S, Di equipment Co., Ltd. Shanghai Eagle.

Reagent part:

100μg/mL stock iron solution: Accurately weigh 0.2163g ammonium ferric sulfate, after dissolved in water to drain 250mL volumetric flask, add sulfuric acid 3.80mL, diluted with deionized water to the mark, the standby.

10μg/mL standard solution of iron: iron stock solution was accurately weighed 25.00mL, placed in 250mL flask, diluted with deionized water to volume, shake up.

0.1g/mL of hydroxylamine hydrochloride solution: Weigh accurately hydroxylamine hydrochloride 2.5002g, dissolved in water after draining to the 250mL volumetric flask and dilute to the mark with deionized water, shake up.

1.5mg/mL phenanthroline solution: Weigh accurately phenanthroline 0.1520g, with 1mL of absolute ethanol was placed in a 100mL volumetric flask and dissolved, adding deionized water to the scale, shake up.

Preparation of acetic acid-sodium acetate buffer solution with pH=4, and reserve.
2.2. Preparation of chitosan

The collected waste Xiatou eviscerated washed, dried in a thermostatic oven, milled shrimp pulverizer to form a powder after drying was placed. By demineralization to proteins, decoloring, four steps of deacetylation of chitosan and the like in the waste extraction head of the prawn powder, yield of different under different conditions.

2.3. Adsorption of Fe (III) by Chitosan

This experimental method Maxue Lian [7] (2009) for detecting iron ions, the method for improving determination of iron ion content GB320-2006 (phenanthroline spectrophotometry), having a simple and rapid having, color sensitive, buffer system stable.

2.3.1. Work curve drawing.

GB320-2006 reference standard curve, the specific steps are as follows. 7 Take 100mL volumetric flask with a pipette imbibe an iron ion concentration of 20μg/standard solution and 0, 8.00, 14.00, 20.00, 26.00, 32.00mL 38.00mL; were added 2.00mL suction mass concentration 0.1g/mL solution of hydroxylamine hydrochloride, was added 8.00mL of acetic acid - sodium acetate buffer solution and 2.00mL concentration of 2mg/mL solution of phenanthroline. Aqueous ammonia solution was adjusted to no color deepened, diluted with deionized water to volume, and mix for 15 minutes. Without addition of Fe$^{3+}$ reagent blank was sequentially measuring absorbance at 510nm, and take ρ(Fe$^{3+}$) as the abscissa and absorbance as the ordinate, draw the working curve, and perform linear regression on the experimental results. The adsorption effect of chitosan is expressed by the adsorption rate, which is calculated in Equation (1).

$$\text{Absorbance rate} = \frac{\rho_0 - \rho_t}{\rho_0} \times 100\%$$

In the formula: $\rho_0$ is the initial mass concentration of Fe(III), μg/mL; $\rho_t$ is the mass concentration of Fe(III) in the solution at the end of the experiment, μg/mL.

![Figure 1. Standard curve of initial mass concentration determination of Fe(III).](image)

Obtained from the regression curve in FIG. 1 is a linear equation $y = 0.2086x - 0.0039$, correlation coefficient $R^2 = 0.9999$, good linearity, indicating that this method of (III) reliable measurement result.

2.3.2. Effect of different chitosan dosage on adsorption effect.

The study of the adsorption properties of chitosan, so the priority adsorption experiments investigate the effect of chitosan in different amounts. Exact amount of 6 parts of 10μg/mL standard iron solution 50.00mL, transferred to a conical flask 6, an amount of chitosan were added 0.05, 0.10, 0.15, 0.20, 0.25, 0.30g, the solution is adjusted to pH 2.0, the reaction temperature was centrifuged after stirring at 25°C for 2h, 11.00mL supernatant
were transferred to a 50mL volumetric flask, a spectrophotometer to measure absorbance at 510nm under conditions and results calculated and recorded.

2.3.3. Effect of different reaction temperatures on adsorption effect. In general, temperature will also have an impact on the adsorption effect. Then, 0.20g chitosan was added to each conical flask. The pH of the solution was adjusted to 2.0, and the temperature of the solution was successively adjusted to 25, 35, 45, 55, 65, 75℃. After stirring for 2h, centrifugation was carried out. 11.00ml of the supernatants were transferred to a 50mL volumetric flask, and the absorbance was measured under the condition of 510nm spectrophotometer, and the experimental results were calculated and recorded.

2.3.4. Effect of different adsorption time on adsorption effect. To explore the influence of adsorption time on the adsorption effect, 10μ Transfer 50.00ml of iron standard solution (g/ml) to 8 conical flasks, add 0.20g of chitosan to each conical flasks, adjust the pH value of the solution to 2.0, and then stir at 60℃ for 30, 40, 50, 60, 70, 80, 90, 100, 110 min. after centrifugation, transfer 11.00ml of supernatant to 50ml volumetric flask, The absorbance was measured at 510nm, and the experimental results were calculated and recorded.

2.4. Analysis of the influence of reaction temperature on adsorption effect

Table 1 and Fig. 2 show the effect of Chitosan on the adsorption of Fe(III) under different reaction temperatures. The results showed that when the reaction temperature was lower than 55℃, the adsorption rate of chitosan for Fe(III) had a small increasing trend. At 25℃, the adsorption rate of 81.52% slowly increased to 86.21% at 55℃. The results showed that the percentage of active molecules increased with increasing temperature, which promoted the adsorption of Fe(III) on chitosan. In this experiment, the optimum temperature is 55℃, but the change of reaction temperature has little effect on the adsorption rate. In practical application, the reaction temperature can be adjusted according to the situation.

| Temperature (℃) | 25  | 35  | 45  | 55  | 65  | 75  |
|----------------|-----|-----|-----|-----|-----|-----|
| Absorbance     | 0.3821 | 0.3589 | 0.3287 | 0.2842 | 0.2787 | 0.2812 |
| Adsorption rate (%) | 81.52 | 82.62 | 84.05 | 86.21 | 86.42 | 86.34 |

Figure 2. Absorbance at different reaction temperatures.
2.5. Analysis of the influence of adsorption time on adsorption effect

Table 2 and Fig. 4 show the effect of Chitosan on the adsorption of Fe(Ⅲ) under different adsorption time. The results showed that when the adsorption time was controlled from 10 min to 50 min, the adsorption effect increased with the increase of time, and reached the maximum at 50 min. When the reaction time was 40-50 min, although the adsorption rate continued to increase, the rate slowed down. The reason for this is that the specific surface area of chitosan adsorbent and Fe(Ⅲ) decreases continuously with the reaction going on, and the adsorption sites on the adsorbent surface are gradually occupied. Therefore, the adsorption rate is faster in the initial stage and slower in the later stage, and the saturation time is about 50 min.

Table 2. Effect of different adsorption time on adsorption effect.

| Adsorption time (min) | 10  | 20  | 30  | 40  | 50  | 60  | 70  | 80  |
|-----------------------|-----|-----|-----|-----|-----|-----|-----|-----|
| Absorbance            | 1.4910 | 1.1372 | 0.8461 | 0.5102 | 0.3671 | 0.3752 | 0.3689 | 0.3721 |
| Adsorption rate (%)   | 28.32 | 45.32 | 59.25 | 75.36 | 82.24 | 81.82 | 82.12 | 81.98 |

Figure 3. Effect of different reaction temperature on adsorption effect.

Figure 4. Absorbance under different adsorption time.
3. Conclusion
The experimental results show that single chitosan has a good adsorption effect on Fe(Ⅲ), and the adsorption effect is affected to a certain extent by the reaction conditions, temperature, reaction time and the amount of chitosan. Under the same other conditions, the optimal reaction conditions were 55℃ and 50 min. The experimental results show that chitosan can solve the problem of wastewater containing Fe(Ⅲ), which can not only contribute to the cause of environmental protection, but also realize the transformation of waste into treasure, and reduce the environmental pollution pressure caused by the discharge of waste shrimp shells from industry and catering industry.

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