Synthesis and Characterization of Modified Sepiolite for the Adsorptive Removal of Rhodamine B from Wastewater

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Abstract

SDS/sepiolite was successfully synthesized as a low-cost adsorbent used in removal of Rhodamine B (RhB) from aqueous solution. The adsorbing capacity of RhB onto SDS/sepiolite shows a significant increase than the acid sepiolite. Analysis of the specific surface area, morphology, microstructure, chemical structure, chemical bonding substance and functional groups by BET, SEM and FT-IR proved the better adsorption capacity of SDS/sepiolite. The adsorption quantity was calculated to be 24.91mg/g under the optimum condition: SDS/sepiolite loadings of 0.02g, pH of 3 and 50°C, the concentration of RhB is 35mg/L obtained by analysis of influencing factors. It was found that a pseudo-second-order equation provided the best correlation to the process of adsorption, R²=0.999. Adsorption isotherm was fitted with Langmuir equation on different temperature, all R²>0.99. Re-adsorption showed SDS/sepiolite has reusable capacity in the adsorption of RhB in environmental protection.

Introduction

It is well known that Rhodamine B (RhB) was widely used in leather, textile and paper industries. “Blood River” event, the river becomes red dyed by Printing and dyeing waste water in Wenzhou, Zhejiang Province in July 2014. HeNan Ruyang “Red River” event is caused by the dyeing waste water which penetrates land from the dye-stuff plant nearby in September 2014. At the same time, a giant sewage pool discovered in Inner Mongolia, Ningxia Ming sheng dyeing co, LTD and other dye production companies involved among them. Rhodamine B (RhB) as a typical kind of alkaline dye has a structure of triphenylmethane molecules. RhB is a toxic kind of carcinogenicity and has great destructive to the environment [1-3]. Rhodamine B solution, migrated by diffusion and water removal, physical and chemical migrated through a series of biochemical reactions such as oxidation - reduction, hydrolysis and dissolve the precipitate, complexation-chelation, bio-analysis, photo-degradation and biological organisms migrated through the absorption, metabolism, growth, and death process, appeared in soil, groundwater, plants, animals and eventually caused human health problems through the food chain and other sectors.

Sepiolite is a kind of two-layer silicon-oxygen tetrahedron, the middle layer of magnesium oxide octahedral structure. And a layered structure of a chain. Each of the six top silicon-oxygen tetrahedron vertexes opposite, forming a layered structure on the key elements 2:1 parallel channels arranged in the lower phase [4-6]. Mg²⁺ and Si-OH groups are the main active centers for adsorption [7]. This particular structure determines its excellent adsorption performance, better stability also determines Sodium Dodecyl Sulfate (SDS) supported on sepiolite better stability [8-9]. However, due to natural sepiolite impurities, contaminants and partially blocked pores smaller channels which limits the role of sepiolite [10]. Therefore, sepiolite it has been studied using the modified method by scholars. Marjanović V [4], Le-taief S [11], Miura A [12], Franco F [13], et al., studied the adsorption capacity of sepiolite acid-modified. Miura A [12], Duan E [14], et al., studied the adsorption capacity of sepiolite heated-modified. Franco F [13] researched on the Microwave assisted acid treatment of sepiolite. Zhang G [15], Lazarević S [16], Pina-Zapardiel R [17], et al., studied the adsorption and catalysis capacity of sepiolite loaded metal or metallic oxide. Adsorption capacity of sepiolite loaded anionic surfactants has been studied by few people.

SDS as an anionic surfactant was used to modify sepiolite in this article. Adsorption capacity to RhB with SDS/sepiolite was compared with sepiolite, acid sepiolite and Polyvinylpyrrolidone (PVP)/sepiolite in the present study. Then the obtained SDS/sepiolite was characterized using Brunauer-Emmett-Teller (BET), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared spectroscopy (FT-IR). To evaluate the potential of SDS/sepiolite, studies of the kinetics, adsorption mechanisms and isotherms were conducted. Adsorption mechanisms were tested under the optimum conditions obtained by analyzing the influence factors. Finally, Re-adsorption experiments executed was detected to show whether there is recycling of SDS/sepiolite. All results indicated that SDS/Sepiolite is a good adsorbent using in the environmental field.

Experimental Section

Activation of sepiolite

All chemical reagents in this study were of analytical grade and used without further purification. A certain amount of sepiolite was
immersed into 10% dilute nitric acid for 6 h. Afterward, the solid was filtered, dried, and calcined at 120°C for 12 h. Finally, the 100-150 um particle was chosen as carrier.

**Synthesis of SDS/sepiolite and PVP/sepiolite composites**

In the synthesis of SDS/sepiolite composites, 5.0g of sepiolite powder and 0.5g of SDS were added into 85ml of water-ethanol solution (deionized water: absolute ethanol = 75:10, v/v), stirred for 30 min. Added some hydrochloric acid (0.1M) until pH=2, stirred for 4h on magnetic stirrer and then let stand for 1h, removed of the supernatant, repeat three times, then the mixed solution was centrifuged using a centrifuge. The sediment at 120°C for 12 h. Finally, the 100-150 um particle was chosen as adsorption carrier.

Synthesis of PVP/sepiolite accord to the way of synthesis of SDS/sepiolite. And then adsorption capacity to RhB with SDS/sepiolite was compared with sepiolite, acid sepiolite and Polyvinylpyrrolidone (PVP)/sepiolite.

**Characterization of adsorbents**

The specific Brunauer-Emmett-Teller (BET) Surface area (SBET) for each composite was derived from N₂ adsorption isotherms that were measured using a BELSORP. The morphologies and microstructure analyses were performed with a JSM-6700F field-emission scanning electron microscope. Fourier transform infrared (FT-IR) spectra (400-4000cm⁻¹) were recorded on a MAGNA-IR 750 FT-IR apparatus using KBr disks.

**Analysis of factors affecting the adsorbent**

Adsorption reactions were conducted in some 25ml Serum bottles, m(g) of SDS/sepiolite and 15ml RhB (C-mg/L) were added, adjusted pH, sampling 20(min), 40(min), 1h,1.5h,2h,3h and 5h after adsorption reactions. Adsorbance of samples were detected at 554nm using a visible spectrophotometer. Specific figures were seen in the table 1.

**Adsorption kinetics and isotherm of adsorption**

Adsorption kinetics: To determine the effect of SDS/sepiolite and the adsorption rate of RhB, experiment were performed under the optimum condition that mSDS/sep=0.02g, C₀=35mg/L, pH=3, sampling 0.5h, 1h, 1.5h, 2h, 3h, 4h, 6h, 8h and 10h after adsorption reactions. Adsorption capacity to RhB with SDS/sepiolite was compared with sepiolite, acid sepiolite and Polyvinylpyrrolidone (PVP)/sepiolite.

Adsorption reactions were conducted at 10, 25 and 35 degrees Celsius in the constant temperature water bath. Sampling 0.5h after adsorption reactions and adsorbance of samples were detected at 554nm using a visible spectrophotometer. The equilibrium adsorption data were analyzed using the Freundlich (Equation 5) and Langmuir (Equation 6) equilibrium isomerths.

\[ q_e = k_1 c_e^{1/n} \]  \hspace{1cm} (5)

\[ q_e = \frac{b c_e}{1 + b c_e} \]  \hspace{1cm} (6)

Where, \( q_e \) is the amount of RhB adsorbed (mg/g), \( c_e \) is RhB concentration in equilibrium solution (mg/L).

**Re-adsorption experiments**

Adsorption reactions were conducted in some 500ml beaker, 0.3g of SDS/sepiolite and 150ml RhB (25mg/L) were added. Adsorption reaction was conducted at 25 degrees Celsius in the constant temperature water bath. Sampling 0.5h, 1h, 1.5h, 2h, 2.5h, 3h, 3.5h and 4h after adsorption reactions. Then removing of the supernatant and filtrating the sediments with vacuum suction filter machine. And then conducting adsorption reaction (mSDS/sep:vRhB=1:500(g:ml) two times. Adsorption of samples were detected at 554nm using a visible spectrophotometer.

**Results and Discussion**

**Adsorption performance comparison**

The adsorption quantity at different adsorption time with several different absorbents was shown in figure 1. As can be seen from figure 1, SDS/sepiolite have a strong adsorption capacity, with physical adsorption and chemical adsorption.
Acid sepiolite, the adsorption quantity increased with time rising, rising from 11.88 at 0.5h to 12.15 at 4h. At the beginning of 4h, adsorption is mainly based on physical adsorption with a fast increased rate, since the sepiolite surface charge is determined by the hydrolyzed ruptures of the Si-O bond and Al-O bond on the surface, which can become both acid and alkali. The hydroxyl groups in R-OH (-OH) generated by the hydrolyzed bond breaking have both acid and alkali properties. The modified effect is mainly to make the structural and surface charges of sepiolite in aqueous solution change and thus change the charge and adsorbing activity of sepiolite colloid.

The modified PVP and SDS surface made plug some of the surface micro pores, however, the surface modified adsorption rate slope of PVP and SDS was the biggest, In the adsorption of 1 hours, the adsorption value is higher than that of acid modified sepiolite, PVP/sepiolite adsorption slope is more than SDS/sepiolite adsorption slope, may PVP of high surface activity and more load on the surface of sepiolite, In the adsorption of PVP in the time zone contact with the Rhodamine B large area, with more quantities. In 3 hours, the several sepiolites adsorption slope is close, subsequently adsorbed slope of PVP/Sepiolite is minimum, acid modified sepiolite is second, SDS/sepiolite is maximum, because PVP and Rhodamine B in the sepiolite surface reaction together, to some extent hindered the sepiolite adsorption of Rhodamine B, which is the reason, the absorbance of PVP/Sepiolite adsorption of Rhodamine B than acid change high absorbance of sepiolite adsorption of Rhodamine B at 10 hours.

**Structural characterization**

Figure 2 shows the nitrogen adsorption isotherm for the acid sepiolite and SDS/sepiolite composites, illustrating slow adsorption and desorption (type III isotherm). The date indicates that SDS/sepiolite has a better adsorption capacity to the N\textsubscript{2} than acid sepiolite (Table 2). The nitrogen adsorption capacity increases with the increase of the relative pressure, the growth rate is slower, mainly because at the start of the sepiolite surface contains a large number of microporous and mesoporosities, which is the reason, the adsorption quantity of acid sepiolite is smaller than that of the SDS/sepiolite (44.81 m\textsuperscript{2} g\textsuperscript{-1}), because the method of Adsorbent preparation and the concentration of SDS affect the specific surface area and the total pore volume of adsorbent.

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found at 2927 and 2839 for SDS/sepiolite. It indicated that SDS successfully supported on sepiolite. Acid sepiolite and SDS/sepiolite exhibit various bands at 624-677 (bending vibration of Mg-OH), at 753-795 (bending vibration of Mg-Fe-OH) [21], which shows the basic structure of sepiolite has not changed because magnesium ions as a supporting role has not changed in sepiolite [12]. SDS/sepiolite exhibits the band at 2839-2927 (asymmetric stretching vibrations of C-H groups) [21].

Analysis of factors affecting

The effects of the SDS/sepiolite dosage on the reaction rate and adsorption quantity are shown in figure 5. The best date of adsorption quantity was 16.91mg/g when the SDS/sepiolite dosage was 0.02g. The adsorption quantity was only 7.44mg/g corresponding mSDS/sepio-lite=0.05g. The maximum adsorption quantity was 7.5mg/g on 0.05g adsorbent in theory, it is very close to experimental data, thus it indicated a small number of adsorption quantity because of low RhB concentration. The reason of the low adsorption quantity of dosage (0.03g and 0.04g) is like this. The maximum adsorption quantity on 0.02g SDS/sepiolite was 18.75 in ideal model, which indicated there some mass RhB unadsorbed in solution. Thus, 0.02g of SDS/sepiolite was appropriate. Then the date of adsorption quantity to different RhB concentration verified the explanation in figure 6.

To compare the pH dependencies of the activities of SDS/sepiolite, its relative activities were determined in the pH range 3-9 and the results were shown in figure 7. The data showed that optimum pH is 3. Magnesium ions of octahedral layer were easily replaced by hydrogen ion in acid solution and the structure of sepiolite remains unchanged. Magnesium ions are eluted and Si-O-Mg-O-Si formed two Si-OH which become active centers of adsorption. Besides, internal channels are through, microporous empty and macroporous extended due to eluted magnesium ions in sepiolite [4,12,13,16]. In light of this theory, the maximum adsorption quantity was obtained in the solution pH=3.

Moreover, the surface charge of SDS/sepiolite becomes dependent on the pH of the solution due to protonation and deprotonation. Figure 8 is Zeta potential distribution of SDS/sepiolite at varied pH. From figure 8, we can know that the pH at zero point charge of SDS/sepiolite is around 5, indicating that the surface charge is positive when pH < 5 and negative at pH > 5. The results indicate that SDS/sepiolite has a better adsorption effect on RhB under acidic conditions.
In conclusion, the optimum condition was $m_{\text{SDS/sep}}=0.02\,\text{g}$, $c_{\text{Rh-B}}=35\,\text{mg/L}$, $pH=3$. The result is the same as the date of orthogonal analysis in tables 3 and 4.

### Adsorption kinetics

The kinetics of RhB adsorption under the optimum condition is presented in figure 9. The R2 of pseudo-first, pseudo-second order and Morries-Wede equations were 0.705, 0.999 and 0.707, respectively. Equilibrium adsorption capacity obtained from the pseudo-second order was closed to the experimental data. In summary, adsorption process can be good fitted pseudo-second order. The assuming rate-limiting step was chemical adsorption in pseudo-second adsorption kinetics (Table 5). With diffusion within the absorbent and adsorption in the pores, the concentration of adsorbate become lower and lower. The tangent of line fitted Morries-Wede equation didn’t pass the x-axis and y-axis intersection which indicated there are little inner adsorption in the adsorption process [22,23].

![Figure 7: Effect of the adsorption capacity on different solution pH.](image)

![Figure 8: Zeta potential of SDS/sepiolite.](image)

![Figure 9: Adsorption kinetics of SDS/sepiolite (a): the relation of adsorption and time, b): pseudo first-order kinetics fitting, c): pseudo first-order kinetics fitting, d): Morries-Wede fitting.](image)

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**Table 4: Orthogonal experiment results of adsorbing capacity.**

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**Table 3: Orthogonal design table.**

### Isotherm of adsorption

Adsorption isotherm and its fitting results are presented in figures 10-12 and tables 6 and 7. The correlation coefficients fitted ($R^2<0.99<R^2$) and $q_m$ equaled the saturation adsorption quantity of experiments both indicated adsorption of RhB with SDS/sepiolite can be well fitted by the Langmuir equation in the experiment examined the range of temperature and concentration. Single saturation adsorption amount and the equilibrium constant $b$ were positively correlated to temperature obtained from figure 10.

![Figure 10: Adsorption isotherm of SDS/sepiolite.](image)
It revealed the adsorption process is an endothermic process. Adsorption of RhB with SDS/sepiolite is preferentially adsorbed due to \( b > 0 \). \( 1/n < 1 \) also showed the adsorption process is chemical adsorption in table 6. The correlation coefficients \( R_1^2 \) was positively correlated to \( n \) (when the \( n < 1 \), \( R_1^2 \) nearly equal to 1) which also indicated adsorption is a monolayer adsorption [24-27].

### Table 5: Adsorption kinetics parameters.

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<th>Fitting Type</th>
<th>pseudo-first order</th>
<th>pseudo-second order</th>
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<td>( K_1 )</td>
<td>( R^2 )</td>
<td>( K_2 )</td>
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<tr>
<td>( 0.5545 )</td>
<td>( 0.705 )</td>
<td>( 2.5 )</td>
<td>( 0.041 )</td>
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Re-adsorption analysis

In the study of this experiment, the first adsorption quantity was much greater than the second adsorption and the third adsorption quantity (Figure 13). Each adsorption amount is not large, however, the sum of each saturation adsorption amount (30.39mg/L) was larger than the maximum adsorption quantity (24.91mg/L) under optimal conditions. Desorption generation was appeared in the process of adsorption and some mass RhB adsorbed desorbed in the filtration and drying experiments. It indicated SDS/sepiolite as a reusable sorbent was used in environmental protection.

### Table 6: Freundlich linear fitting parameters.

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<th>Temperature (°C)</th>
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<th>( n )</th>
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### Table 7: Langmuir linear fitting parameters.

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Conclusion

A novel adsorbent SDS/sepiolite composite was successfully prepared. Adsorption properties of SDS/sepiolite indicate that it has a good adsorption capacity in the comparison of adsorbing RhB and Analysis of the specific surface area, morphology, microstructure, chemical structure, chemical bonding substance and functional groups. Maximum adsorption quantity was 24.91 mg/L under the optimum conditions (mSDS/sep=0.02g, cRhB=35mg/L, pH=3.) verified by orthogonal experiment. Adsorbing kinetics and adsorption isotherm revealed main adsorption reaction was chemical adsorption and single adsorbing. Adsorption quantity increases with increasing temperature. Re-adsorption analysis indicated SDS/sepiolite as a reusable sorbent was used in removal of RhB. All results indicated anionic surfactants SDS modified sepiolite primarily to enhance the chemistry adsorption properties of surface. In summary, SDS/sepiolite can as an efficient and low-cost adsorbent used in environmental field.

References

Journal of Anesthesia & Clinical Care
Journal of Addiction & Addictive Disorders
Advances in Microbiology Research
Advances in Industrial Biotechnology
Journal of Agronomy & Agricultural Science
Journal of AIDS Clinical Research & STDs
Journal of Alcoholism, Drug Abuse & Substance Dependence
Journal of Allergy Disorders & Therapy
Journal of Alternative, Complementary & Integrative Medicine
Journal of Alzheimer’s & Neurodegenerative Diseases
Journal of Angiology & Vascular Surgery
Journal of Animal Research & Veterinary Science
Archives of Zoological Studies
Archives of Urology
Journal of Atmospheric & Earth-Sciences
Journal of Aquaculture & Fisheries
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Journal of Cancer Biology & Treatment
Journal of Cardiology: Study & Research
Journal of Cell Biology & Cell Metabolism
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Journal of Clinical Studies & Medical Case Reports
Journal of Community Medicine & Public Health Care
Current Trends: Medical & Biological Engineering
Journal of Cytology & Tissue Biology
Journal of Dentistry: Oral Health & Cosmesis
Journal of Diabetes & Metabolic Disorders
Journal of Dairy Research & Technology
Journal of Emergency Medicine Trauma & Surgical Care
Journal of Environmental Science: Current Research
Journal of Food Science & Nutrition
Journal of Forensic, Legal & Investigative Sciences
Journal of Gastroenterology & Hepatology Research
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