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Hydrothermal synthesis of SnS2/MoS2 Nanospheres for enhanced adsorption capacity of organic dyes

Bowen Cui1, Yihui Wang1, Fan Zhang1,2, Xin Xiao1,2, Ziqin Su1, Xincheng Dai1, Hao Zhang1 and Shan Huang1

1 School of Chemical Engineering, Jiangsu Ocean University, Lianyungang 222005, People’s Republic of China
2 School of Chemical Engineering, Nanjing University of Science and Technology, Nanjing 210094, People’s Republic of China

E-mail: njuzzfan@163.com and xiaoxin@njjust.edu.cn

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Abstract
The SnS2/MoS2 nanospheres (NPs) were prepared by a facile hydrothermal route, and microstructure and morphology were investigated by SEM, TEM, XRD and BET. The SnS2/MoS2 NPs exhibited the excellent adsorption properties for organic dyes, and the maximum adsorption capacity of SnS2/MoS2 to Rhodamine B (Rhb) is 125 mg g−1 at room temperature. The adsorption process is well fit by the pseudo-second-order adsorption model and Langmuir isotherm model. Moreover, SnS2/MoS2 NPs has outstanding adsorption capacity for both cations and anions organic dyes, and the maximum adsorption capacity for methylene blue (MB), crystal violet (CV) and malachite green (MG) were 202, 165 and 175 mg g−1 respectively. It is attribute to the high specific surface area (101.06 m2 g−1) and small mesopores (3.23 nm) provide numerous adsorption active sites for adsorption of organic dyes. The reusability experiment demonstrated the SnS2/MnS2 NPs could be reused for 5 times. The result show that the SnS2/MoS2 NPs is a potential adsorbent for removal of organic dyes from wastewater.

1. Introduction

The unreasonable discharge of industrial wastewater contains high concentrations of various organic dyes and their derivatives, which are harmful to human health and eco-environment [1]. Rhodamine B (Rhb) as a representative of organic dyes, widely used in the field of textile and leatherwear [2]. In recent years, the removal of RhB has been extensively explored because it is difficult to decompose and harmful to human [3, 4]. Previously developed methods for removing organic dyes including chemical precipitation [5], photocatalytic degradation [6], membrane separation [7] and adsorption [8]. Compare with other methods, adsorption is a great potential method due to easy treatment, low cost and high efficiency.

Mesoporous transition metal sulﬁdes have attracted widespread attention the field of wastewater treatment, which attribute to high specific surface area, special microstructure and higher surface activity [9]. MoS2 as a representative of mesoporous transition metal sulﬁdes that widely used for adsorption of organic dyes [10, 11]. Wang et al [12] demonstrated the flower-like MoS2 used for an effective adsorbent for removal of RhB, and the adsorption capacity reached 49.2 mg g−1. Song et al [13] synthetized fungus-like MoS2 nanosheets with ultrafast, and investigated adsorption capacities for organic dyes. The application of traditional MoS2 is limited because of its low adsorption capacity. The composite of MoS2 and other similar materials is an effective method to improve the adsorption capacity of materials.

As a potential photocatalyst, SnS2 has attracted great interest from researchers in recent years [14]. In addition, SnS2 has potential to be an excellent adsorbent for wastewater treatment due to excellent adsorption properties, chemically stable and simple synthesis [15]. Wang using Sn4+ and Sn2+ as raw materials to synthesize the SnS2/SnO2 composites by a green hydrothermal method, and the hierarchical SnS2 were applied for removal of RhB [16]. Because both MoS2 and SnS2 have outstanding adsorption capacity for dye adsorption, the coupling...
structures of MoS2 and SnS2 could form a heterostructure to increase the adsorption capacity for organic dyes. In addition, there is no report of SnS2/MoS2 composites used for removal of organic dyes.

In this work, the SnS2/MoS2 NPs were synthesized by a one-pot hydrothermal method, and the adsorption properties of SnS2/MoS2 for organic dyes were discussed for the first time. The adsorption kinetics, adsorption equilibrium, adsorption thermodynamics and adsorption mechanism of RhB adsorption onto SnS2/MoS2 NPs were investigated in detail. In addition, the SnS2/MoS2 NPs exhibited the excellent adsorption capacity and significantly enhanced adsorption performance as compared with pure MoS2 and SnS2 composites. The results demonstrated the novel SnS2/MoS2 NPs is a potential adsorbent for removal of organic dyes in wastewater.

2. Experimental

2.1. Synthesis of SnS2/MoS2 NPs

All chemicals were obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China) and were not further purified. The SnS2/MoS2 NPs were synthesized by a hydrothermal method [17]. In a typical procedure, 6 mm thiourea (CN2H4S), 0.25 mm Ammonium molybdate tetrahydrate ((NH4)6Mo7O24·4H2O) and 2 mm stannous chloride dihydrate (SnCl2·2H2O) were added in 30 ml of distilled water. After stirring for 15 min, the mixture was transferred into a 40 ml autoclave and then reacted at 200 °C for 12 h. After cooling naturally, the products were obtained and washed with deionized water and ethanol for several times, and dried at 70 °C for 12 h in a vacuum oven. Finally, the SnS2/MoS2 was obtained. As a comparison, pure MoS2 and pure SnS2 were synthesized.

2.2. Material Characterization

The phase structure was tested by powder x-ray diffraction (XRD) on a RINT 2000 diffractometer (Rigaku) with Cu Kα radiation. The shape of the product was tested by a Hitachi S-4800 field emission scanning electron microscope (SEM) and a JEOL JEM 2100 F transmission electron microscope (TEM). The BET surface areas were performed using a micromeritics ASAP 2460. The Raman spectra was performed using a HORIBA HR-800. XPS of the samples were performed by a Thermo ESCALAB 250XI. The ultraviolet-visible (UV–vis) spectrophotometer was performed using a TU-1901.

2.3. Adsorption experiments

The adsorption studies was performed in the dark to prevent degradation of RhB. 20 mg of SnS2/MoS2 NPs were disperse to 50 mL RhB aqueous solutions with different concetrate (10–150 mg L−1). The solution was magnetically stirred at 1000 rpm for 2 h. 3 mL of RhB solution was collected at different time intervals. The supernant was collected and centrifuged at 10 000 rpm for 3 min. Then the supernatant was transferred into microcell for the analysis of RhB at 554 nm with a UV–vis Spectrometry. Blank experiments was carried out under the same conditions. The adsorption rate (a) and adsorption capacity (b) are calculated by the following equations:

\[
\text{Adsorption rate} = \frac{C_0 - C_e}{C_e} \times 100\% \quad (1)
\]

\[
\text{Adsorption capacity} = \frac{C_0-C_e}{W} \times V \quad (2)
\]

where \(C_e\) (mg L−1) is equilibrium concentration of RhB solution; \(C_0\) (mg L−1) is the initial concentration of RhB solutions; \(V\) (L) is the volume of sample solution; \(m\) (g) is the mass of the adsorbents.

3. Results and discussion

3.1. Characterization of SnS2/MoS2 NPs

Figure 1 is the XRD pattern of the products of SnS2/MoS2, MoS2 and SnS2. The diffraction peaks (figure S1 is available online at stacks.iop.org/MRX/7/015016/mmedia) at 16°, 29°, 33°, 50°, and 53° were originated from the (001), (100), (101), (110), and (111) planes of SnS2, respectively. The phase structure was indexed to the 2T-type hexagonal berndtite SnS2 (JCPDS, No. 23-0677). The purity was good because of no impurity peak was obtained. The XRD pattern of pure MoS2 can be indexed well to hexagonal phase (JCPDS, no. 37-1492). The XRD pattern of the SnS2/MoS2 has two kinds of diffraction peaks, Besides the diffraction peaks at 9°(002), and 33.6°(100) reflections assigned to the MoS2 (JCPDS card no.37-1492), the others are consistent with the SnS2, suggesting the presence of MoS2 grown on SnS2.
Figure 2 is shown the SEM images of SnS2, MoS2 and SnS2/MoS2. Figure 2(a) shows that the SnS2 was composed by irregular and non-uniform flake. It was found that the MoS2 nanospheres in figure 2(b). As a comparison, the morphology of SnS2/MoS2 is similar with the pure MoS2 (figure 2(c)).

TEM was used to further study the structure of the SnS2/MoS2 NPs. The image shows that the sample consists of nanosheets, which is consistent with SEM (figure 3(a)). The HRTEM image (figure 3(b)) indicates two interplanar distance of approximately 0.65 nm and 0.32 nm, that is consistent with the (002) and (100) lattice plane of MoS2 and SnS2. According to the corresponding energy dispersive x-ray spectrum (EDX) results (figure 3(c)), the mass ratio of Mo, S and Sn was to be 0.77: 0.39: 0.66, and the atomic ratio was 32.71%: 32.33%: 34.97%. In addition, energy-dispersive x-ray spectrometry mapping images reveal the elements of Mo, S and Sn were uniformly distributed throughout the surface of SnS2/MoS2 (figures 3(d)–(g)).

The BET surface of the SnS2/MoS2 was performed by N2 adsorption. The N2 adsorption-desorption isotherm of SnS2/MoS2 is exhibited in figure 4. The BET surface and average pore size are list in table 1. The N2 adsorption-desorption curve was assigned as type IV isotherm having H3 hysteresis loop, suggesting a mesoporous structure. The SnS2/MoS2 has the large surface area of 101.06 m² g⁻¹, which is much larger than SnS2 and MoS2. The large BET surface should add more active sites to the adsorption of Rhodamine B, which facilitated the rapid adsorption and transfer of the adsorbate.
### 3.2. Adsorption properties

The sample pH is a significant parameter for the adsorption properties of RhB by affecting both the existing forms of the RhB molecule and the charge species and density on the surface of SnS$_2$/MoS$_2$ NPs. As the result of zeta potential, the surface of SnS$_2$/MoS$_2$ NPs had negative charge with the zeta potential of $-40.6$ mV. The sample pH was adjusted by B-R buffer solution, and the effect of pH on the adsorption capacity was investigated at pH 3.0 to 11.0. As shown in Figure 5, the adsorption capacity of SnS$_2$/MoS$_2$ for RhB was the highest (180 mg g$^{-1}$) at pH 3.0. And the adsorption capacity of SnS$_2$/MoS$_2$ NPs decreases gradually from 125 to 55 mg g$^{-1}$ in the pH range of 3–11. It could be a reasonable explanation of the finding that, at pH $< 7$, RhB molecule exhibit a cationic form to promoted the electrostatic interactions between RhB and SnS$_2$/MoS$_2$ NPs.

To demonstrate the rapid adsorption properties of MoS$_2$/SnS$_2$ NPs, 20 mg SnS$_2$/MoS$_2$ NPs were added into 10 mg L$^{-1}$ RhB solutions. Figure 6(a) shown in the adsorption capacity and adsorption rate of RhB at different time intervals (0–15 min), the adsorption efficiency were 70%, 83% and 92% within 5, 10 and 15 min. As shown in the insert image of figure 6(a), the color of the dye solution disappeared after 15 min. The result show that the SnS$_2$/MoS$_2$ NPs quickly adsorbed RhB from dye solution. The adsorption kinetics model is a significant
parameter to illustrate the adsorption mechanism of RhB onto adsorbents. The experimental data were fitted using the pseudo-first-order model (3) and the pseudo-second-order kinetic model (4), which expressed by the following equation:

\[
\ln(Q_e - Q_t) = \ln Q_e - k_1t
\]  
(3)

\[
t/Q_t = 1/(k_2Q_e^2) + t/Q_e
\]  
(4)

Where \(k_1\) (min \(^{-1}\)) and \(k_2\) (g mg \(^{-1}\) min \(^{-1}\)) are the rate constant of pseudo-first-order adsorption and pseudo-second-order adsorption, respectively. \(Q_e\) is the adsorption capacity at different times and \(Q_t\) is the adsorption capacity at adsorption equilibrium. The fitting curve of adsorption kinetics models and relevant parameters are shown in figure 7(b) and table 2. It was found that the linearly fitting \(R^2\) of pseudo-second-order adsorption model is 0.9992, and the calculated adsorption capacity (24.2 mg g \(^{-1}\)) is similar to the actual adsorption capacity (23.6 mg g \(^{-1}\)). The pseudo-second-order kinetics fitting curve indicates a better linear relationship for the experimental data. The result show that the adsorption process is controlled by chemisorption, and adsorption process be attribute to the electrostatic attraction between the negative charge on the surface of SnS\(_2\)/MoS\(_2\) NPs and RhB molecule.

The adsorption isotherm could explain the adsorbate molecules distribution in solid-liquid adsorption system at the equilibrium. The Langmuir adsorption isotherm model illustrate the adsorbents is monolayer and the active site on the adsorbent surface [18]. The Freundlich isotherm model demonstrates the presence of heterogeneous adsorption surfaces and variable adsorption sites with different adsorption energies [19].
Langmuir isotherm model (5) and Freundlich isotherm model (6), which represented by the following equation:

\[
\frac{C_e}{Q_e} = \frac{C_e}{Q_m} + \frac{1}{K_o Q_m}
\]

\[
\ln Q_e = \ln K_f + \frac{1}{n_f} \ln C_e
\]

Where \(K_l\) and \(K_f\) are the Langmuir and Freundlich isotherm constant, respectively. Figure 8 shows the fitting curve of the adsorption experimental data using Langmuir and Freundlich isotherm models. As shown in table 3, the \(R^2\) of the Langmuir isotherm model is higher than Freundlich isotherm models. And the calculated adsorption capacity of Langmuir isotherm model is 125 mg g\(^{-1}\), is closer to the actual adsorption capacity (127 mg g\(^{-1}\)), which illustrated the adsorption process is well fit by the Langmuir isotherm model. The adsorption of RhB onto SnS\(_2\)/MoS\(_2\) NPs conforms to the single-layer adsorption model. The result show that the active sites are located on the surface of SnS\(_2\)/MoS\(_2\) NPs.

The thermodynamic studies is an important parameter for provide information on intrinsic energy changes associated with adsorption process. Three thermodynamic parameters were considered to describe the adsorption process; the enthalpy change (\(\Delta H\)), Gibb’s free energy (\(\Delta G\)) and entropy change (\(\Delta S\)) were calculated from the following equation:

\[
\Delta G = -RT \ln kd
\]

\[
\ln kd = \frac{\Delta S}{R} - \frac{\Delta H}{RT}
\]

\[
K_d = \frac{Q_e}{C_e}
\]

Where \(R\) is the universal gas constant (8.314 J mol\(^{-1}\) K\(^{-1}\)); \(T\) is the temperature (K); \(kd\) is the equilibrium constant. Plots of \(\ln kd\) versus \(1/T\) are linear as indicated in figure 7. The values of \(\Delta H\) and \(\Delta S\) can be calculated by the slope and intercept of \(\ln kd\) relative to \(1/T\). The thermodynamic parameters for the adsorption of RhB on SnS\(_2\)/MoS\(_2\) NPs are shown in table 4. The negative values of \(\Delta G\) except for RhB adsorption onto SnS\(_2\)/MoS\(_2\) NPs in the range of 293 to 333 K, which demonstrate the adsorption process is spontaneous. The positive value

| Tem. (K) | \(Q_m\) | \(K_l\) | \(R^2\) | \(n_f\) | \(K_f\) | \(R^2\) |
|---------|---------|---------|---------|--------|--------|---------|
| 293     | 127.47  | 0.2085  | 0.9913  | 5.330  | 53.68  | 0.9425  |
| 303     | 142.62  | 0.1584  | 0.9956  | 4.494  | 50.85  | 0.9420  |
| 313     | 162.71  | 0.1306  | 0.9868  | 4.197  | 52.73  | 0.9527  |
of $\Delta H$ (9.43 kJ mol$^{-1}$) illustrated the adsorption is endothermic process. The positive values of $\Delta S$ indicating the randomness of the solid-liquid interface increases during the adsorption processes.

To illustrate the excellent adsorption properties of SnS$_2$/MoS$_2$ NPs, the adsorption capacity of pure MoS$_2$ and SnS$_2$ were discussed respectively. Figure 9(a) show that the adsorption capacity of SnS$_2$/MoS$_2$ to RhB is significantly higher than MoS$_2$ and SnS$_2$. The adsorption for RhB is mainly attributed to the Van der Waals force and electrostatic attraction between adsorbents and RhB. Moreover, the introduction of SnS$_2$ increases the active center and specific surface area of MoS$_2$ and further increases the adsorption capacity of MoS$_2$ for RhB. To prove SnS$_2$/MoS$_2$ has excellent adsorption capacity for different organic dyes, Rhodamine B (RhB), methylene blue (MB), malachite green (MG) and crystal violet (CV) were carried out at room temperature. Figure 9(b) indicate the maximum adsorption capacity of SnS$_2$/MoS$_2$ to RhB, MB, MG, CV were 125 mg g$^{-1}$, 202 mg g$^{-1}$, 165 mg g$^{-1}$ and 175 mg g$^{-1}$ respectively. The results show that SnS$_2$/MoS$_2$ NPs has great potential for wastewater treatment because of its excellent adsorption capacity for both cationic and anionic organic dyes.

Table 4. Thermodynamic parameters for RhB by SnS$_2$/MnS$_2$ NPs at different temperature.

| Tem. (K) | $\ln K_d$ | $\Delta H$ (kJ mol$^{-1}$) | $\Delta S$ (J K$^{-1}$ mol$^{-1}$) | $\Delta G$ (kJ mol$^{-1}$) |
|---------|-----------|-----------------------------|----------------------------------|--------------------------|
| 293     | 0.7452    | 9.43                        | 38.24                            | −1.81                    |
| 313     | 0.9382    | −2.44                       | 3.35                             |                          |
| 333     | 1.2131    | −3.35                       |                                  |                          |

The reusability of an adsorbent is an important factor for its practical application. According to reported that the adsorption/desorption procedure of RhB could changed by the pH value and the polarity of the solven [20], the sodium hydroxide of ethanol solution (0.5 mol L$^{-1}$) is used to desorb RhB. The adsorbed SnS$_2$/MoS$_2$ NPs were washed three times with 0.5 mol L$^{-1}$ NaOH solution and distilled water respectively, then SnS$_2$/MoS$_2$ NPs were used for the adsorption of RhB again. The adsorption efficiency of SnS$_2$/MoS$_2$ NPs gradually decreased with increase of repeated times, because the RhB molecule cannot be completely desorbed from SnS$_2$/MoS$_2$ NPs and a little RhB molecule still occupied the active site onto the adsorbent. As shown in figure 10, the adsorption efficiency of SnS$_2$/MoS$_2$ NPs was maintain at 85.8% after repeated use for 5 times. The result illustrated the SnS$_2$/MoS$_2$ NPs has well recycling performance for adsorption of RhB.
3.2.1. Adsorption mechanism

To demonstrate the adsorption mechanism of RhB onto the SnS$_2$/MoS$_2$ NPs, the FT-IR spectrum of the SnS$_2$/MoS$_2$ NPs before adsorption (figure 11(a)) and after adsorption (figure 11(b)) to RhB was prepared and discussed. Figure 11(a) exhibited the strong characteristic diffraction peaks at 580 cm$^{-1}$, which is attribute to the

![Figure 10. Reusability tests of the SnS$_2$/MoS$_2$ NPs for the adsorption of RhB.](image)

![Figure 11. FT-IR spectrum of SnS$_2$/MoS$_2$ NPs before adsorption and after adsorption of RhB.](image)

| Organic dyes         | Adsorbent                  | Specific surface area (m$^2$ g$^{-1}$) | Adsorption capacity (mg g$^{-1}$) | References  |
|----------------------|----------------------------|----------------------------------------|-----------------------------------|-------------|
| Rhodamine B          | Fe$_3$O$_4$/MoS$_2$ composites | 72.07                                  | 24.0                              | [13]        |
|                      | MoS$_2$ nanosheets          | 106.98                                 | 76.0                              | [20]        |
|                      | MoS$_2$-glue sponge         | 123.7                                  | 125                               | [8]         |
|                      | SnS$_2$/MoS$_2$ NPs        | 101                                    | 125                               | This work   |
| Methylene blue       | MoS$_2$@MWCNT              |                                        | 96.7                              | [21]        |
|                      | Few-layered MoS$_2$        | 74.4                                   | 146.43                            | [22]        |
|                      | 2D-MoS$_2$ nanosheets      | 15.09                                  |                                   | [23]        |
|                      | MoS$_2$ nanosheets         | 297.2                                  |                                   | [11]        |
|                      | SnS$_2$/MoS$_2$ NPs        | 101                                    | 203                               | This work   |

Table 5. Comparison of SnS$_2$/MoS$_2$ NPs and other adsorbents for adsorption capacity of organic dyes.
bending vibrations of Sn-S. After adsorption of RhB, the spectrum of SnS2/MoS2 NPs exhibited the diffraction peaks at 1335 cm⁻¹ and 1585 cm⁻¹, which are attributable to the C-C stretching vibrations in the aromatic ring of RhB molecule [24]. The FT-IR spectra of SnS2/MoS2 NPs before and after adsorption show that the RhB is adsorbed on the surface of the SnS2/MoS2 NPs.

4. Conclusion

In this study, SnS2/MoS2 NPs were synthesized by a facile hydrothermal method, and the adsorption properties for organic dyes was investigated for the first time. SnS2/MoS2 NPs has excellent adsorption capacity (125 mg g⁻¹) for RhB, which much higher than SnS2 and MoS2. The SnS2/MoS2 NPs could adsorb RhB from aqueous solution within 15 min, and adsorption efficiency reached 92%. The adsorption process was fitted to pseudo-second-order kinetic model and Langmuir model. Moreover, the SnS2/MoS2 NPs has excellent adsorption properties for both cationic and anionic organic dyes. The result demonstrated the SnS2/MoS2 NPs as adsorbent to rapidly and effectively removal of different organic dyes from wastewater.

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ORCID iDs

Bowen Cui @ https://orcid.org/0000-0001-9916-3595
Fan Zhang @ https://orcid.org/0000-0002-1596-5695
Xin Xiao @ https://orcid.org/0000-0002-0852-7398

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