

# From tremella-like MoS<sub>2</sub> to $\alpha$ -MoO<sub>3</sub> nanoplates: sintering synthesis and adsorption properties

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A simple calcination method was explored to synthesise the tremella-like molybdenum disulphide (MoS<sub>2</sub>), Mo trioxide (MoO<sub>3</sub>)/MoS<sub>2</sub> and MoO<sub>3</sub> nanoplates by using the pristine MoS<sub>2</sub> nanosheets as the precursor. The possible formation and growth mechanism from the pristine MoS<sub>2</sub> to the tremella-like MoS<sub>2</sub>, MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and MoO<sub>3</sub> nanoplates was proposed. It was found that the calcination temperature was a key factor for the formation, ingredient and phase structure of the three samples. The adsorption properties of the three samples for an aqueous solution of methylene blue (MB) at room temperature were investigated. The results revealed that the products exhibit the better adsorption capacities with increasing the ingredient of MoO<sub>3</sub> in the transformation process from MoS<sub>2</sub> to MoO<sub>3</sub>. Therefore, the pure MoO<sub>3</sub> nanoplates have the highest adsorption and removal rates, which reached 96.9% at MB concentration of 10 mg/l and 90.9% at MB concentration of 20 mg/l.

**1. Introduction:** Water contaminated by organic pollutant has become a serious environmental problem and has attracted global concern because of rapid industrialisation and urbanisation [1–4]. Thus, various techniques have been applied to remove organic pollutant from wastewater including chemical oxidation method, biodegradation [5], photocatalysis and adsorption [6–8]. Among them, adsorption has been considered as a promising and efficient method to remove organic pollutant from aqueous solutions due to low cost, convenient operation, flexibility, small amounts of harmful substances and simple design [8–11]. It is a universally acknowledged truth that adsorption efficiency relies on the development of high-performance adsorbent, and adsorption properties are significantly affected by the structures and chemical composition of adsorbents. Many of adsorption materials have been investigated extensively for this purpose and activated carbon is most widely used as adsorbents for the treatment of polluted water. However, activated carbon is relatively expensive, and alternative cost-effective adsorbents of different chemical composition are required for the treatment of wastewater [12, 13].

Recent years with the discovery of graphene, other two-dimensional (2D) materials such as metal chalcogenides and transition metal oxides have been attracted extensive attention. So far amongst 2D materials, molybdenum (Mo) compounds, Mo disulphide (MoS<sub>2</sub>) and Mo trioxide (MoO<sub>3</sub>) have played a significant role, and shown intriguing physical and chemical properties as well as exciting prospects for a variety of applications [14, 15] such as catalytic activity and adsorption property. For example, Xie *et al.* [16] demonstrated the successfully synergistic regulations of both structural and electronic benefits by controllable disorder engineering and simultaneous oxygen incorporation in MoS<sub>2</sub> nanomaterials, leading to the dramatically enhanced performance and activity. Song *et al.* [17] have synthesised fungus-like MoS<sub>2</sub> nanosheets with ultrafast adsorption capacities toward organic dyes of wastewater. Ma *et al.* [18] reported that  $\alpha$ -MoO<sub>3</sub> nanobelts with high adsorption capacity had been synthesised by a facile one-pot hydrothermal route using fluoroboric acid as the H<sup>+</sup> resource and the surfactant. Wang *et al.* [12] fabricated an MoO<sub>3</sub> hierarchical architecture with highly selective and efficient adsorption using a solvothermal method without adding any surfactants or templates. Recent researches have shown that the composition and

arrangement of atoms in materials play a crucial role in determining their fundamental properties [19–22]. Up to now, the synthetic conditions for these MoS<sub>2</sub> and MoO<sub>3</sub> nanomaterials are independent, i.e. the phase-controlled synthesis of MoS<sub>2</sub> nanosheet assembly, the composite MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and MoO<sub>3</sub> nanoplates through a simple synthetic route have rarely been reported. Consequently, it is necessary to develop a facile method to fabricate a series of samples with different composition and phase structures to answer their effects on adsorption properties.

In our work, we used as-prepared 2H-MoS<sub>2</sub> as precursor to calcine at different temperatures in air and finally obtained a series of products including 2H-MoS<sub>2</sub> assembly, the composite MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and  $\alpha$ -MoO<sub>3</sub>, respectively. X-ray powder diffractometer (XRD) and scanning electron microscope (SEM) were used to identify the composition and morphology of materials. Additionally, the surface adsorption properties were also investigated by adsorbing methylene blue (MB) aqueous solution. As a result, with the change of the composition and phase structure from 2H-MoS<sub>2</sub> to  $\alpha$ -MoO<sub>3</sub>, the enhanced adsorption activities of  $\alpha$ -MoO<sub>3</sub> were observed obviously.

**2. Experimental procedure:** Synthesis of tremella-like MoS<sub>2</sub> nano sheets: all the reagents used in this experiment process were analytical grade without further purification. Typically, 2.24 g thiourea (99 wt %, Sinopharm) and 1.24 g (99 wt %, Sinopharm) were mixed and dissolved in 40 ml deionised water under vigorous stirring for 30 min to form a homogeneous solution. Then, the solution was transferred into a 50 ml teflon-lined stainless-steel autoclave, which was carried out at 180°C for 12 h, and then the autoclave was cooled down to room temperature naturally. The black precipitate was collected and washed with deionised water for three times. Finally, the product of powder was dried at 60°C for 6 h under vacuum. The as-prepared MoS<sub>2</sub> was obtained, and named the sample T60.

Preparation of 2H-MoS<sub>2</sub>, MoO<sub>3</sub>/MoS<sub>2</sub>,  $\alpha$ -MoO<sub>3</sub>: To synthesise 2H-MoS<sub>2</sub>, MoO<sub>3</sub>/MoS<sub>2</sub>,  $\alpha$ -MoO<sub>3</sub>, the sample T60 (the as-prepared MoS<sub>2</sub>) was used as precursor. To fabricate 2H-MoS<sub>2</sub>, 0.4 g of the sample T60 was placed in a resistance furnace and annealed at 200°C for 2 h in air, and then the product was cooled down to room temperature naturally; the black powder of 2H-MoS<sub>2</sub> was obtained and named the sample T200. To obtain MoO<sub>3</sub>/MoS<sub>2</sub>,

the sample T60 was placed in a resistance furnace and treated at 300°C for 2 h and the obtained sample was named the sample T300. The sample  $\alpha$ -MoO<sub>3</sub> named T400 was synthesised by calcination of T60 in air at 400°C for 2 h.

The products were characterised with an X-ray powder diffractometer (XRD; Rigaku D/Max-2550PC) equipped with Cu-K $\alpha$  radiation, a scanning electron microscope (SEM; HITACHI S-4800) and a transmission electron microscope (TEM; JEOL JEM-2100F). Ultraviolet–visible (UV–vis) absorption spectra were detected by UV–vis 1901 spectrophotometer (AoXi).

In the adsorption experiments, MB was employed as the probe of organic pollutants in aqueous solution. Typically, a series of MB aqueous solutions were prepared with different concentrations (5–20 mg/l). Then, 20 mg absorbent was added into 50 ml MB aqueous solution and the solution was kept stirring at room temperature. At a constant time interval, 3 ml solution was sampled and centrifuged at 10,000 rpm for 3 min to separate the absorbent before analysis. The solution concentration was analysed by using a UV–vis spectrophotometer to determine the remaining concentration of MB at  $\lambda = 664$  nm. The MB concentration was calculated by the Lambert–Beer equation. Additionally, adsorbents were dealt with the sodium hydroxide (aq) to remove the adsorbed MB and then were rinsed mildly with distilled water and ethanol for several times for next test [22].

**3. Results and discussion:** The phase and the purity of as-synthesised samples were examined by the X-ray powder diffraction (XRD) patterns. Fig. 1 shows comparative XRD patterns of as-prepared samples at different temperatures, and reveals that a phase transformation occurs with different calcination temperatures. The XRD pattern of the sample T60 via hydrothermal method dried at 60°C was observed three broad peaks at 14.3°, 33.6° and 58.5° obviously, and the three broad and weak peaks can be well indexed to (002), (101) and (110) planes of 2H-MoS<sub>2</sub> (JCPDS Card No. 73-1508), respectively. It is noteworthy that a peak of the sample T60 appeared at 9°, and the special result reveals the formation of a lamellar structure which conforms to the previous reported by Xie *et al.* [16]. It confirms that the sample T60 is ordered 2H-MoS<sub>2</sub>. The XRD pattern of the sample T200 can be indexed to 2H-MoS<sub>2</sub>. It is noteworthy that the absence of other indexed diffraction peaks indicates the disordering lamellar stacking in the sample T200, and the sample T200 is disordered 2H-MoS<sub>2</sub>. Interestingly, compared with the sample T200, the XRD pattern of T300 shows the weaker peaks at 14.3°, 33.6° and 58.5° indicates the existence of MoS<sub>2</sub> in the sample T300; the other diffraction peaks in the XRD pattern can be indexed to the  $\alpha$ -MoO<sub>3</sub> (JCPDS Card No. 05-0508). It shows

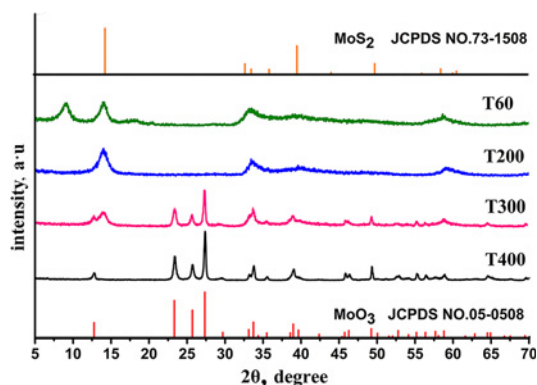


**Fig. 1** XRD patterns of the as-prepared MoS<sub>2</sub> via hydrothermal method dried at 60°C (T60) and other samples obtained by calcination of the sample T60 at 200°C (T200), 300°C (T300), at 400°C (T400). The standard patterns of MoS<sub>2</sub> (JCPDS Card No. 73-1508) and MoO<sub>3</sub> (JCPDS Card No. 05-0508) are also supplied

that the sample T300 is consisted of MoS<sub>2</sub> and MoO<sub>3</sub>. When the calcination temperature was raised to 400°C, the sample T400 displays a very standard XRD pattern of MoO<sub>3</sub> and no peaks of the impurity were found within experimental error. The sharp shape and narrow widths of the diffraction peaks indicate that the sample T400 has high crystallinity.

Interestingly, it can be clearly seen that the peaks indexed to (002), (101) and (110) planes of 2H-MoS<sub>2</sub> are becoming weaker and weaker, whereas the peaks corresponding to  $\alpha$ -MoO<sub>3</sub> especially (110), (040) and (021) planes have increasing intensity. That means, combined with Scherrer's equation  $D = K\gamma/B \cos \theta$  ( $D$  represents the size of crystalline domains,  $K = 0.89$ ,  $\gamma = 0.154056$  nm,  $B$  is full width at maximum intensity and  $\theta$  is the Bragg angle), the MoS<sub>2</sub> crystals fade away gradually while the crystalline grains of  $\alpha$ -MoO<sub>3</sub> begin to grow up. In particular, the distinct enhanced intensity of (021) plane indexed to  $\alpha$ -MoO<sub>3</sub> in T400 than T300 indicates the more orderly orientation growth. Thus, it can be concluded that the calcination of the sample T60 at different temperatures causes a phase transformation from MoS<sub>2</sub> to MoO<sub>3</sub>.

Fig. 2 shows the typical SEM images of these samples. From the low-magnification SEM image (Fig. 2a), the sample is composed of the hierarchical and tremella-like assemblies, with the diameters

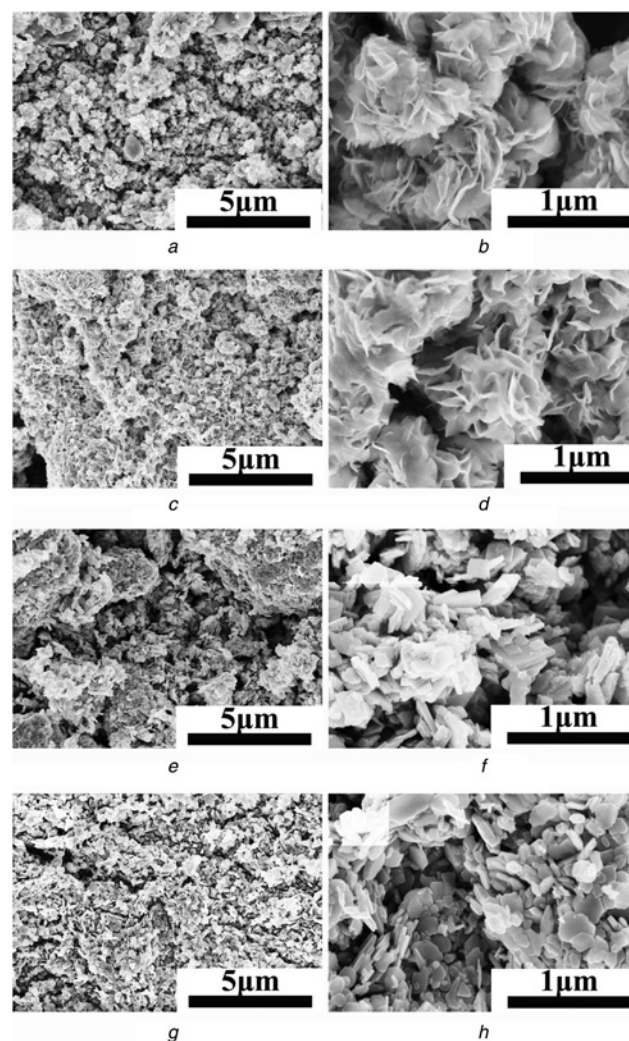


**Fig. 2** SEM images of as-prepared samples  
a, b Sample T60 (pristine MoS<sub>2</sub>)  
c, d Sample T200 (2H-MoS<sub>2</sub>)  
e, f Sample T300 (MoO<sub>3</sub>/MoS<sub>2</sub>)  
g, h Sample T400 ( $\alpha$ -MoO<sub>3</sub>)

of 1–3  $\mu\text{m}$ . The high-magnification SEM image of the typical assembly (Fig. 2b) illustrates that the tremella-like assembly consists of curved nanosheets which are 10–20 nm in thickness and  $\sim 150$ –800 nm in length. The tremella-like nanosheets provide a relatively large surface area for the pristine  $\text{MoS}_2$  assemblies, which is important for adsorption property. After calcination of the sample T60 at  $200^\circ\text{C}$ , the pristine  $\text{MoS}_2$  assemblies were transformed completely into 2H- $\text{MoS}_2$  assemblies, but the morphology was unchanged (Figs. 2c and d). After calcination of the sample T60 at  $300^\circ\text{C}$ , the nanoplates composed of  $\text{MoS}_2$  and  $\text{MoO}_3$ , with the width of 150–400 nm and the thickness of  $\sim 60$  nm can be synthesised (the sample T300, Figs. 2e and f). When the calcination temperature is  $400^\circ\text{C}$ ,  $\text{MoO}_3$  (the sample T400) is obtained showing a smaller sized plate-like shape with  $\sim 150$ –400 nm in width and  $\sim 40$  nm in thickness, as shown in Figs. 2g and h. Combined with the XRD and SEM results, the above results demonstrate that different calcination temperatures were found to be a key factor that can affect the composition, phase structure and morphology of as-prepared samples through the phase-controlled process.

Considering all results analysed above, the formation and transformation mechanism of the tremella-like  $\text{MoS}_2$  into  $\text{MoO}_3$  nanoplates is proposed as illustrated in Fig. 3. The seed nuclei of  $\text{MoS}_2$  are first formed using the hydrothermal route in process I. With the time extended in the process II, the seed nuclei of  $\text{MoS}_2$  are grown into the curved and ordered  $\text{MoS}_2$  nanosheets, and pristine  $\text{MoS}_2$  is synthesised. When the sample T60 is annealed at  $200^\circ\text{C}$  for 2 h in the process III, the XRD patterns reveal that the ordered structure of pristine  $\text{MoS}_2$  has transformed into the disordered structure of 2H- $\text{MoS}_2$  assemblies. When the sample T60 was treated at  $300^\circ\text{C}$  in the process IV, the  $\text{O}_2$  could be able to participate in the reaction and  $\text{MoO}_3$  formed on the surface of the  $\text{MoS}_2$  nanosheets; it indicates that the sample is consisted of  $\text{MoS}_2$  and  $\text{MoO}_3$  nanoplates. When the sample T60 was heated at  $400^\circ\text{C}$  in the process V,  $\text{MoS}_2$  will be completely oxidised to  $\text{MoO}_3$ , and  $\text{MoO}_3$  nanoplates are obtained.

To evaluate the potential application in water treatment of tremella-like 2H- $\text{MoS}_2$  assemblies,  $\text{MoO}_3/\text{MoS}_2$  nanoplates and  $\text{MoO}_3$  nanoplates, the adsorption capacities for MB are investigated. Fig. 4a shows the UV–vis absorption spectroscopy recording the absorption intensity of the MB solution after treatment with  $\text{MoO}_3$  nanoplates as adsorbent. It is distinct that the intensity of characteristic peak for MB remarkably decreases within 5 min and reaches almost zero after treatment for 35 min, suggesting the high adsorption property of the  $\text{MoO}_3$  nanoplates. Figs. 4b–d exhibit the adsorption curves measured with different MB concentrations of 5–20 mg/l at 664 nm and room temperature using the tremella-like 2H- $\text{MoS}_2$  assemblies,  $\text{MoO}_3/\text{MoS}_2$  nanoplates and  $\text{MoO}_3$  nanoplates as adsorbents. It could be seen that the adsorption rates of  $\text{MoO}_3/\text{MoS}_2$  nanoplates and  $\text{MoO}_3$  nanoplates are faster than that of the tremella-like 2H- $\text{MoS}_2$  assemblies at MB concentration of 5 mg/l; the adsorption rate of  $\text{MoO}_3/\text{MoS}_2$  nanoplates

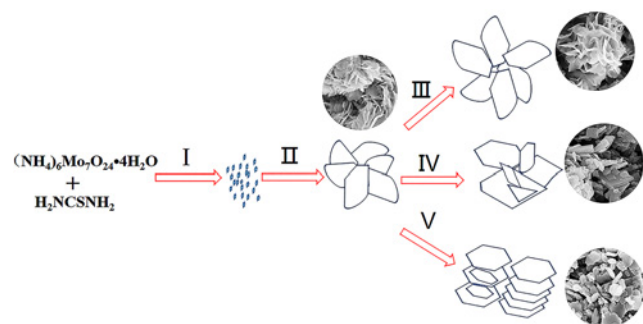


Fig. 3 Formation and growth mechanism of the samples

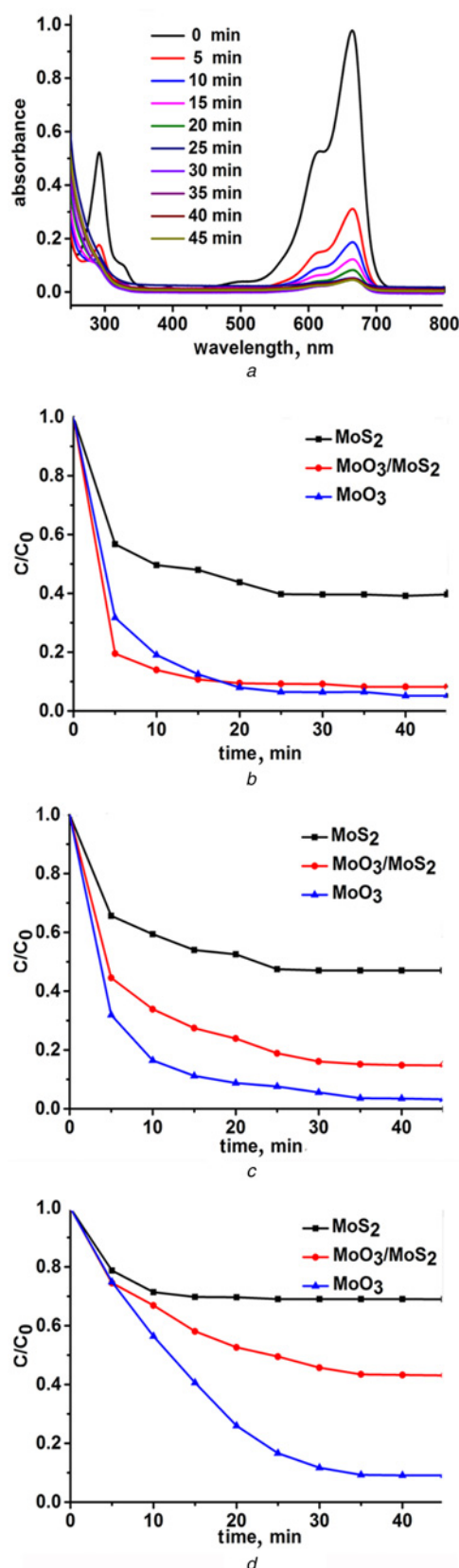


Fig. 4 Adsorption spectra and adsorption curves  
a Adsorption spectra of an aqueous solution of MB (20 mg/l, 50 ml) in the presence of  $\text{MoO}_3$   
b Adsorption curves of the aqueous MB in the presence of  $\text{MoS}_2$ ,  $\text{MoO}_3/\text{MoS}_2$  and  $\text{MoO}_3$  at the concentration of 5 mg/l  
c Adsorption curves of the aqueous MB in the presence of  $\text{MoS}_2$ ,  $\text{MoO}_3/\text{MoS}_2$  and  $\text{MoO}_3$  at the concentration of 10 mg/l  
d Adsorption curves of the aqueous MB in the presence of  $\text{MoS}_2$ ,  $\text{MoO}_3/\text{MoS}_2$  and  $\text{MoO}_3$  at the concentration of 20 mg/l

is faster than that of MoO<sub>3</sub> nanoplates at first, but the adsorption rate of MoO<sub>3</sub> nanoplates is faster than that of MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates after 18 min, while the removal rate of MoO<sub>3</sub> nanoplates was slightly higher than that of MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates. When the MB concentrations increase to 10 and 20 mg/l, the MoO<sub>3</sub> nanoplates exhibit both the highest adsorption rate and the largest removal capacity than those of the tremella-like 2H-MoS<sub>2</sub> assemblies and MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates; after adsorption for 35 min, the removal rate of MoO<sub>3</sub> nanoplates remains almost the same and the maximum removal rate reaches to 96.9% at MB concentration of 10 mg/l (as shown in Fig. 4c) and 90.9% at MB concentration of 20 mg/l (as shown in Fig. 4d). Additionally, the adsorbents were also recycled to test for three times again and excellent adsorption properties were remained. These results suggested that the products exhibit the enhanced adsorption capacities with the increasing ingredient of MoO<sub>3</sub> in the transformation process from MoS<sub>2</sub> to MoO<sub>3</sub>. The high adsorption property of MoO<sub>3</sub> may be contributed to its high surface area [17] and largely attributed to the electrostatic attraction between the surface and the MB molecules [18].

**4. Conclusion:** In summary, we have successfully synthesised the tremella-like 2H-MoS<sub>2</sub> assembly, MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and MoO<sub>3</sub> nanoplates by a simple calcination method using the pristine MoS<sub>2</sub> as precursor. A possible growth mechanism from the pristine MoS<sub>2</sub> to 2H-MoS<sub>2</sub> assemblies, MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and MoO<sub>3</sub> nanoplates was proposed. It was found that the calcination temperature was a key factor for the formation of different samples. Investigations of the adsorption property revealed that the tremella-like 2H-MoS<sub>2</sub> assemblies, MoO<sub>3</sub>/MoS<sub>2</sub> nanoplates and MoO<sub>3</sub> nanoplates with the increasing ingredient of MoO<sub>3</sub> in the samples have better adsorption activity for an aqueous solution of MB. In particular, the pure MoO<sub>3</sub> nanoplates have the highest adsorption and removal rates, which reached 96.9% at MB concentration of 10 mg/l and 90.9% at MB concentration of 20 mg/l. It is believed that MoO<sub>3</sub> nanoplates could be able to be a potential adsorbent for wastewater treatment.

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