Research Article

Synthesis of Magnesium Oxide Nanoplates and Their Application in Nitrogen Dioxide and Sulfur Dioxide Adsorption

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In this research, nanostructured magnesium oxide was synthesized through the sol-gel calcination or hydrothermal calcination method using various surfactants. The X-ray diffraction pattern of the materials confirmed that all the prepared magnesium oxide samples were single phase without any impurity. The scanning electron microscopy images and specific surface area values showed that sodium dodecyl sulfate was the most suitable surfactant for the synthesis of magnesium oxide nanoplates with the diameter of 40–60 nm, the average thickness of 5 nm, and a specific surface area of 126 m²/g. This material was utilized for nitrogen dioxide and sulfur dioxide adsorption under ambient condition. The saturated adsorption capacities of magnesium oxide were 174 mg/g for nitrogen dioxide and 160 mg/g for sulfur dioxide, making the magnesium oxide nanoplates a promising candidate for toxic gas treatment.

1. Introduction

Nowadays, air pollution caused by the emission of toxic gases from human and industrial activities poses not only tremendous threats to human health but also the destructive effects to the ecosystem. Nitrogen dioxide (NO₂) and sulfur dioxide (SO₂) are criteria pollutants that have adverse impacts on the environment. Both of NO₂ and SO₂ are released from natural and anthropological sources including forest fires, volcanic eruptions, transportation systems, and the combustion process. While NO₂ gas could be harmful to human due to cardiovascular damage and respiratory pathway [1, 2], SO₂ gas can affect the human health with breathing problems, cardiovascular complications, bronchitis, and eyes irritation [3, 4]. Some studies show that for every 10 ppb increase in SO₂ concentration, the risk of death increased by 0.2–2% [5]. This increasing risk requires feasible solutions to deal with. Various methods have been developed by scientists and experts

including molecular sieves, membrane separation, adsorption, and catalyst [6–9]. Among them, adsorption is one of the most effective methods for toxic gas treatment owing to simple processing and regeneration at low cost. Several materials such as activated carbon, hydroxide, metal oxide, sepiolite, and metal organic frameworks (MOFs) have been widely used as effective adsorbents [10–13].

During the development of nanoscience and nanotechnology, the application of inorganic nanomaterial as toxic gas adsorbents has attracted much attention because of their large specific surface area and high reactivity. Due to the economical and eco-friendly properties, nano-magnesium oxide (MgO) is a potential adsorbent of toxic gas [14–18]. However, to the best of our knowledge, there have been very few reports using MgO for the adsorption of NO₂ and SO₂.

Nanostructured MgO has been synthesized by various physicochemical techniques, such as sol-gel calcination,

chemical precipitation, and hydrothermal and microwave fabrication [19–28]. In this research, we successfully synthesized MgO nanoplates by sol-gel calcination and hydrothermal calcination methods. The influence of surfactant on the morphology of nanostructured MgO was investigated. The MgO nanoplates were used for NO₂ and SO₂ adsorption.

2. Materials and Methods

All chemicals were of analytical grade and directly used without further purification. Magnesium nitrate hexahydrate (Mg(NO₃)₂·6H₂O, 99.9%), sodium hydroxide (NaOH, 99.8%), polyethylene glycol (PEG 6000), cetyltrimethylammonium bromide (CTAB, 98%), sodium dodecyl sulfate (SDS, 98.5%), sulfur dioxide (99.9%), and nitrogen dioxide (99.5%) were purchased from Sigma-Aldrich and used without any further purification.

2.1. Synthesis of Nanostructured MgO. Nanostructured MgO was prepared by the sol-gel calcination or hydrothermal calcination method [29, 30] described as follows.

2.2. Sol-Gel Processing. Typically, 10 mmol Mg(NO₃)₂·6H₂O (2.56 g) was dissolved in 250 ml distilled water, and 0.5 mmol surfactant (3 g PEG 6000, 0.182 g CTAB or 0.144 g SDS) was added into the magnesium nitrate solution. Then, 100 ml NaOH 0.2 M solution was added dropwise into this mixture and stirred vigorously until a relatively high viscosity gel was formed. The gel was stirred gently overnight and then filtered, washed several times with distilled water, and dried at 80°C for 4 hours before calcinating at 500°C for 4 hours to obtain a white solid powder.

2.3. Hydrothermal Processing. The hydrothermal process was performed in a Teflon-lined stainless steel autoclave. 10 mmol Mg(NO₃)₂·6H₂O (2.56 g) was dissolved in 250 ml distilled water, and 0.5 mmol surfactant (3 g PEG 6000, 0.182 g CTAB or 0.144 g SDS) was added into the magnesium nitrate solution. Then, 100 ml NaOH 0.2 M solution was added dropwise into this mixture. Afterward, the resulting mixture was transferred into the autoclave and heated at 100°C for 2 hours. After cooling down to room temperature, the precipitate was collected by vacuum filtration and calcinated at 500°C for 4 hours to obtain a white solid powder.

2.4. Characterization of Materials. All the obtained MgO materials were characterized by different physicochemical techniques. The functional groups on the surface of materials were determined by Fourier-transform infrared spectroscopy (FT-IR, Thermo Fisher, USA). Phase identification was performed by the X-ray diffraction (XRD) technique, using Cu K α ($\lambda = 1.54056 \text{ \AA}$) radiation (D/max Ultima III, Rigaku, Japan). The morphology and particle size were given by scanning electron microscope (SEM, Hitachi, S-4800). The specific surface area was measured from the nitrogen adsorption/desorption at 77 K using the Brunauer–

Ennett–Teller (BET) method (TriStar II Plus, Micromeritics Instrument Corp, USA). The chemical composition of MgO material after adsorption was analyzed with energy-dispersive X-ray spectroscopy (SEM-EDX) (HORIBA-EMAX80; Hitachi High-Technology).

2.5. Adsorption Experiments. The experiments of evaluating the NO₂ and SO₂ adsorption on MgO nanoplates were carried out under ambient temperature. SO₂ or NO₂ gas was mixed with nitrogen carrier gas (5% SO₂ or NO₂ in N₂ atmosphere). A glass tube reactor was loaded with 100 mg of MgO powder with the pretreatment at 200°C for 1 hour. Then, a stream of NO₂ gas or SO₂ gas was supplied with a flow rate of 50 ml/min. The uptake quantities were recorded as a function of time. The adsorption of NO₂ and SO₂ on MgO nanoplates were monitored by FT-IR and EDX. The MgO nanoplates were collected and regenerated by calcination at 500°C for 4 hours. The schematic diagram for NO₂ and SO₂ adsorption experiment is shown in Figure 1.

3. Results and Discussion

3.1. Characterization of Magnesium Oxide Nanoplates. In this study, three different surfactants (PEG, CTAB, and SDS) were utilized for the synthesis of MgO nanocrystals by sol-gel and hydrothermal methods. Their crystalline structures are presented in Figure 2. The most intense peaks in XRD patterns of the synthesized MgO samples were observed at 2θ of 37.16°, 43.12°, and 62.40° which were well-matched with those of JCPDS data file of standard MgO no. 78-0430 [31]. The XRD patterns of MgO synthesized by using different surfactants and synthesis methods were basically similar to the surfactant-free MgO, indicating that the crystal structure of MgO was not affected by surfactants or synthesis methods. Furthermore, both sol-gel and hydrothermal methods produced MgO crystal with single phase.

The morphology of MgO samples is illustrated in Figures 3 and 4. BET specific surface area values of nano-MgO are shown in Table 1. Evidently, surfactant-free MgO samples exhibited undefined morphology and high aggregation.

Among the three surfactants, PEG showed less influence in controlling the size and morphology of MgO nanoplates. In addition, MgO materials synthesized using this surfactant have relatively low surface areas (98 m²/g for the sol-gel method and 110 m²/g for the hydrothermal method). In contrast, SDS was a suitable surfactant for the formation of MgO nanoplates with well-defined morphology and high surface area (115 m²/g for sol-gel method and 126 m²/g for hydrothermal method) (Figure 5). This phenomenon might be explained by the electrostatic interaction between surfactants and MgO surface. The nonionic nature of PEG does not allow effective interaction with positively charged MgO surface while the negatively charged SDS interacts effectively with the surface of MgO. Interestingly, MgO samples synthesized by the hydrothermal method had smaller average size and higher surface area compared with the samples prepared by the sol-gel method. There are two possible

FIGURE 1: Schematic diagram of the experimental setup for NO_2 and SO_2 adsorptions.

FIGURE 2: XRD patterns of nano-MgO synthesized by (a) the sol-gel method and (b) the hydrothermal method.

reasons for these higher surface areas. Firstly, during the hydrothermal process, the generating and loss of water molecules between adjacent layers of hydroxyl ions create defects, intercrystallite channels, and cracks inside the structure of MgO, which increase the surface area and porosity of the material [30]. Secondly, when increasing temperature suddenly, the formation of nucleations is facilitated which results in the decline of precursor concentration and hence, crystals are formed with smaller size. Due to the highest surface area and good morphology, the MgO nanoplate material synthesized by hydrothermal method using SDS was chosen for the adsorption of NO_2 and SO_2 gases.

3.2. Investigation of Adsorption Ability of the Material. Fourier-transform infrared spectroscopy was employed to determine the presence of NO_2 on the surface of MgO (Figure 6(a)). It was shown that after 10 min of adsorption, a new peak appeared at 1384 cm^{-1} which is assigned for NO_3^- vibration [32]. The appearance of NO_3^- is explained by the interaction between NO_2 and the metal sites of MgO which facilitates the dissociation of NO_2 into NO_3^- and NO [32, 33]:

The intensity of this peak increased with the increase of the adsorption time, indicating higher loading of NO_2 on MgO nanoplates. Figure 6(b) presents the results of the adsorption experiment for NO_2 . The uptake rate increased rapidly during the first 20 min with the adsorbed amount of $111\text{ mg}_{NO_2}/\text{g}_{MgO}$. After 60 min of adsorption, the adsorbed NO_2 reached 89% of the maximum capacity (the adsorption equilibrium was achieved after 180 min of adsorption with the uptake value of $174\text{ mg}_{NO_2}/\text{g}_{MgO}$). The EDX results (Figure 6(c)) verified the existence of N elements on MgO nanoplates with 4.55 wt.%, corresponding to the NO_2 adsorption of $176\text{ mg}/\text{g}_{MgO}$, which was in agreement with the adsorption result. The SEM image (inset in Figure 6(c)) illustrated the surface of MgO sample after the adsorption process without any change in comparison with the original sample. In addition, XRD patterns of MgO sample after 180 min of exposure to NO_2 showed no difference with the initial sample (Figure 6(d)).

The results of the SO_2 adsorption on MgO nanoplates are presented in Figure 7. Because of the number of basic sites over MgO, a $SO_{2,gas} + MgO \longrightarrow MgSO_3$ reaction could be expected [33, 34]. The presence of SO_2 on the MgO surface was affirmed by the broadband at 984 cm^{-1} [35] in the IR spectrum (Figure 7(a)). This band enlarged along the

FIGURE 3: SEM images of nano-MgO prepared by the sol-gel method using different surfactants: (a) surfactant-free, (b) PEG, (c) CTAB, and (d) SDS.

FIGURE 4: SEM images of nano-MgO prepared by the hydrothermal method using different surfactants: (a) surfactant-free, (b) PEG, (c) CTAB, and (d) SDS.

TABLE 1: BET specific surface area values of nano-MgO synthesized by using different surfactants.

	Method	Surfactant-free	PEG	CTAB	SDS
S_{BET} (m ² /g)	Sol-gel	66	98	100	115
	Hydrothermal	88	110	119	126

FIGURE 5: N₂ adsorption/desorption isotherms of MgO nanoplates synthesized using SDS by (a) the sol-gel calcination method and (b) the hydrothermal calcination method.

FIGURE 6: Continued.

(c)

(d)

FIGURE 6: (a) FT-IR spectra of MgO samples after different adsorption times; (b) quantity of NO₂ adsorbed on MgO; (c) EDX spectrum and SEM image of MgO after 180 min of adsorption; (d) XRD patterns of MgO samples after different adsorption times.

(a)

(b)

(c)

(d)

FIGURE 7: (a) FT-IR spectra of MgO samples after different adsorption times; (b) quantity of SO₂ adsorbed on MgO; (c) EDX spectrum and SEM image of MgO after 180 min of adsorption; (d) XRD patterns of MgO samples after different adsorption times.

FIGURE 8: Langmuir adsorption isotherm of NO₂ and SO₂ over MgO nanoplates.TABLE 2: NO₂ and SO₂ adsorption capacity of MgO nanoplates compares with well-known adsorbents.

Adsorbent	Experimental condition	Adsorption time (min)	Saturated adsorption capacity (mg/g)		Reference
			NO ₂	SO ₂	
Porous media prepared from sewage sludge ($S_{\text{BET}} = 21.2 \text{ m}^2/\text{g}$)	(i) Concentration: NO ₂ : 70 ppm (0.007%) in nitrogen SO ₂ : 100 ppm (0.01%) in nitrogen (ii) Moisture: 0%	70 (NO ₂); 100 (SO ₂)	1.12 (0.0244 mmol/g)	0.98 (0.0214 mmol/g)	[36]
Zeolite-coated porous media ($S_{\text{BET}} = 13.1 \text{ m}^2/\text{g}$)	(iii) Adsorbent weight: 0.55 g (iv) Gas flow rate: 17000 ml/min (v) Temperature: 25°C	40 (NO ₂); 65 (SO ₂)	2.08 (0.0451 mmol/g)	1.72 (0.0374 mmol/g)	
Activated carbon ($S_{\text{BET}} = 1077 \text{ m}^2/\text{g}$)	(i) Concentration: NO ₂ : 4856 ppm (0.4856%) in nitrogen SO ₂ : 1170 ppm (0.1170%) in nitrogen (relative pressure: 2.47) (ii) Moisture: 0% (iii) Adsorbent weight: 1.5 g (iv) Gas flow rate: 583 ml/min (v) Temperature: 30°C	60	135.70 (2.95 mmol/g)	42.10 (2.73 wt.%)	[37]
MgO nanoplates ($S_{\text{BET}} = 126 \text{ m}^2/\text{g}$)	(i) Concentration: 5% in nitrogen (ii) Moisture: 0% (iii) Adsorbent weight: 0.1 g (iv) Gas flow rate: 50 ml/min (v) Temperature: 25°C	180	174.00	160.00	This research

adsorption time. As shown in Figure 7(b), the amount of SO₂ measured after 10 min reached 95 mg/g_{MgO}. After 60 min, the adsorbed SO₂ reached 93% of the maximum capacity (the adsorption equilibrium was achieved after 180 min of adsorption with the uptake value of 160 mg/g_{MgO}). The EDX spectrum (Figure 7(c)) confirmed the presence of S on the MgO surface. The content of S element was 6.96 wt.%, corresponding to 13.92 wt.% SO₂ in the sample after 180 min of exposure. The calculated amount of SO₂ was about 162 mg/g_{MgO}, similar to the obtained SO₂ adsorption of MgO shown in Figure 7(b). The SEM image (inset in Figure 7(c)) and the XRD patterns (Figure 7(d)) show no

change in morphology as well as the crystalline structure after 180 min of SO₂ exposure compared with the original MgO sample, indicating the high stability of MgO nanoplates under the experimental condition.

The Langmuir model was used to evaluate the adsorption of NO₂ and SO₂ over MgO nanoplates. The Langmuir adsorption isotherm plots of NO₂ and SO₂ adsorption over MgO nanoplates are shown in Figure 8. The R^2 values of 0.9892 (in the case of SO₂) and 0.9962 (in the case of NO₂) illustrate the fitness of the Langmuir model for the adsorption. The equilibrium adsorption capacity value (q_e) was calculated with the relative pressure (p/p_0) value of 1. The q_e

FIGURE 9: Diagram of adsorption capacity of NO₂ and SO₂ over MgO nanoplates after three cycles.

calculated values for the adsorption of NO₂ and SO₂ are 178.52 mg/g and 161.29 mg/g, respectively. These values were similar to the obtained experimental values of 174 mg/g (in the case of NO₂) and 160 mg/g (in the case of SO₂). This result shows the compatibility between experimental and theoretical calculations.

Furthermore, the adsorption capacity of NO₂ and SO₂ over MgO nanoplates was compared with that of reported porous adsorbents. The adsorption capacity and experimental conditions of these materials are summarized in Table 2. Interestingly, the MgO nanoplates exhibited relatively higher adsorption capacity than microporous activated carbon and zeolite [36, 37].

In order to evaluate the recycle of MgO nanoplates, this adsorber was regenerated and reused for NO₂ and SO₂ adsorption. The NO₂ and SO₂ adsorption capacity at the third cycle only decreased 3.45 and 1.87%, respectively (Figure 9). The result further confirmed the potential applicability of MgO nanoplates for the adsorption of NO₂ and SO₂.

4. Conclusions

To summarize, MgO nanoplates were well synthesized by sol-gel and hydrothermal methods using PEG, CTAB, and SDS as the morphology-controlling agents. The MgO nanoplates prepared by the hydrothermal method using SDS surfactant exhibited high specific surface area (126 m²/g) and well-defined morphology of nanoplates with the diameter of 40–60 nm and the average thickness of 5 nm. The adsorption experiments showed high NO₂ and SO₂ uptake values on MgO nanoplates (174 mg/g and 160 mg/g, respectively). Moreover, the MgO samples were highly stable under the exposure of NO₂ and SO₂. These results indicate that MgO is a potential candidate for the adsorption of toxic gases. Further work is ongoing to investigate the adsorption behavior of the material in severe condition. In addition, the kinetic and mechanism of adsorption will be studied for the

future application of MgO nanoplates in toxic gas removal and enhancement of the performance of gas treatment on MgO.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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