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A facile synthesis of nanostructured magnesium oxide particles for enhanced adsorption performance in reactive blue 19 removal

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ABSTRACT

Magnesium oxide (MgO) has been known as an excellent adsorbent for a variety of the environmentally polluted compounds. This work describes a synthesis of nanostructured MgO particles via a facile procedure by using cetyltrimethylammonium bromide (CTAB). Powder X-ray diffraction, thermal gravimetric, and differential thermal gravimetry (TGA/DTG) analyses were performed to characterize the physical properties of synthesized MgO particles and field emission-scanning electron microscopy (FE-SEM) was used to observe their morphology, whereas nitrogen adsorption–desorption isotherms and Brunauer–Emmett–Teller (BET) method were used to calculate the total surface areas of the samples. The adsorptive performance was studied by batch experiments for reactive blue (RB) 19 dye removal. The results showed that as-prepared MgO particles revealed hexagonal-like shaped platelets with an average diameter in the range of 49–91 nm and a mean thickness of 19–25 nm; meanwhile, MgO CTAB-free particles are aggregated, tiny nanoparticles with an average width of 22 nm and an average length of 77 nm. The maximum adsorption capacity of as-prepared nanostructured MgO for reactive blue (RB) 19 dye was 250 mg g^{-1}. Furthermore, the correlation between structural characterization (mean size, pore, surface) of the samples and the adsorption performance was also discussed in details.

1. Introduction

One of the most important industries using synthetic dyes is the textile and garment, which produces a strongly colored wastewater, typically with a concentration in the range of 10–200 mg L^{-1} [1,2]. Since high stability and toxicity are characteristic of synthetic dyes, their release into the environment causes serious environmental and health problems. Thus, industrial dye-effluents are a major concern and need to be treated before being discharged into the environment. Adsorption is widely used for dyes removal due to its simplicity and cost effectiveness. A number of synthetic adsorbents have been investigated for the dyes removed from colored wastewater [3–5]. Among those materials, activated carbon is one of the most frequently studied adsorbents for dye treatment, but its main disadvantage is its high production and treatment cost [4,6]. For this reason, it is necessary to develop alternative adsorbents with high adsorptive capacity and low cost.

Magnesium oxide (MgO) belongs to the group of basic oxides, but it has found many applications in the fields of catalysis, adsorption, additives in refractory, optically transparent ceramic windows, and others [7–10]. $MgO/Mg(OH)_2$ is a well-known adsorbent for removing dyes and soluble toxic ions from wastewater [11–14] as it is a non-toxic and environmentally friendly material with high surface reactivity and adsorption capacity. Furthermore, as the pH of zero point of charge of MgO is 12.4, it is a suitable adsorbent for anions due to its favorable electrostatic attractive mechanism [15]. Synthesis of MgO with high surface area is of great interest in catalytic and adsorptive materials [16–18]. Recent advances in nanotechnology have made much interest in preparing $MgO/Mg(OH)_2$ nanostructures and studying their properties. To date, several methods have been developed to synthesize $Mg(OH)_2$ and MgO nanostructures, such as hydrothermal route [19], solvothermal reaction [20–22], co-precipitation [23,24], and ultrasonic method [25]. Among them, hydrothermal method revealed some advantages in synthesis of nanostructured MgO particles such as high crystalline and homogeneous particles, but could lead to agglomeration of particles [26]. Besides cetyltrimethylammonium bromide (CTAB) has been widely used as a soft template in the morphology-controlled synthesis of nanoparticles [27], it could be a potential chemical to overcome the disadvantage of this method. Up to date, to the best of our knowledge, there has no report regarding use of CTAB as a template for controlling nanostructured MgO morphology.

In this study, our objective is to develop a facile route to synthesize nanostructured MgO particles with the use of cetyltrimethyl-

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ammonium bromide (CTAB). This chemical serves as a morphological control to obtain a high specific surface area and enhance the adsorptive performance of as-synthesized MgO particles. The resulted materials were tested to remove reactive blue dye RB 19 from aqueous solution. The effect of some key factors, such as MgO dosage, pH of the solution, contact time and initial concentration of dye on RB 19 removal, was also evaluated. A possible mechanism for the formation of nanostructured MgO was proposed to identify the role of CTAB in the formation of the nanostructures. Adsorptive capacity was assessed by both Langmuir and Freundlich models.

2. Experimental

2.1. Chemicals

All reagents were of analytical grade and used as received without further purification. Sodium hydroxide (NaOH), magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$), cetyltrimethylammonium bromide (CTAB), and reactive blue 19 ($C_{22}H_{16}N_2Na_2O_{11}S_3$, M = 626.5 g mol^{-1}, denoted as RB 19) were obtained from Sigma–Aldrich. Double distilled water was used for preparing all solutions and reagents.

2.2. Synthesis of MgO nanostructures

In the absence of CTAB, for a typical experiment, 1.6 g $MgCl_2{\cdot}6H_2O$ was dissolved in 40 mL distilled water. Subsequently, 80 mL of a 0.2 M NaOH solution was added dropwise into $MgCl_2$ solution and the resulting mixture was vigorously stirred at 40 °C for 4 h to form a white suspension. As-obtaining suspension was then transferred into a 200 mL Teflon-lined stainless steel autoclave where it was maintained at 180 °C for 24 h. Afterward, a white precipitate was collected and washed for several times with double distilled water, and then dried at 60 °C overnight, and finally calcined at 450 °C for 3 h. In the presence of CTAB, in a typical synthesis, a mixture of a specific amount of CTAB and 1.6 g $MgCl_2{\cdot}6H_2O$ was dissolved in 40 mL distilled water under stirring for 1 h to make a clear gel. CTAB amounts of 1.5, 2.2, and 2.9 g were used. 80 mL of a 0.2 M NaOH solution was then added dropwise into as-obtained gel, and the following steps were the same as for synthesis in the absence of CTAB. Finally, a series of samples was produced and denoted as 0CTAB, 1.5CTAB, 2.2CTAB, and 2.9CTAB, respectively, according to different CTAB amount used.

2.3. Characterizations

Powder X-ray diffraction patterns of the as-synthesized samples were recorded on a Siemens D5005 diffractometer using Cu Kα radiation (λ = 0.15406 nm). Thermal gravimetric and differential thermal gravimetry (TGA/DTG) analyses were performed under Argon flow at a heating rate of 10 °C/min, using SETARAM instrument (Caluire, France).

Powder morphology was examined by Field Emission Scanning Microscopy (S4800, Hitachi, Japan). The average diameter, thickness, and lengths of nanostructured MgO particles were measured from scanning electron microscopic (SEM) images.

Nitrogen adsorption and desorption isotherms were measured at −196 °C with a Micromeritics ASAP 2020 apparatus. The total surface areas were calculated by Brunauer–Emmett–Teller (BET) method, the micropore areas were calculated by *t*-plot method, and the mesopore areas were determined by the total surface area minus the micropore area. Pore size distributions were determined by Barrett–Joyner–Halenda (BJH) method using desorption curves, while the micropore size distributions were determined by Horvath–Kawazoe model.

2.4. Adsorption studies

2.4.1. Analytical technique

The calibration curve for RB 19 (its chemical structure was shown in Fig. S1, Supporting materials) obtained by recording absorbance values of dyes solutions in a range of known concentrations at the wavelength of maximum absorbance (λ = 592 nm). Absorbance measurements were taken by a UV–VIS spectrophotometer (Agilent 8453, USA).

2.4.2. Dye removal experiments

Key parameters such as MgO dosage, pH of solution, contact time, and initial dye concentration were investigated in RB 19 removal batch experiments under the following experimental conditions: the adsorbent dose varies from 0.005 to 0.12 g; pH from 3 to 12; contact time from 1 to 25 min; and initial RB 19 dye concentration in the range of 50–300 mg L^{-1}. The adsorption capacity of as-prepared samples for RB 19 dye was calculated using the following equation: $q_e = \frac{V(C_o - C_e)}{m}$, where C_o and C_e are initial and equilibrium dye concentrations (mg L^{-1}), V is the volume of the dye solution (L), and m is the amount of dry adsorbent (g). All the adsorption experiments were performed at room temperature (18 °C). The experimental data were averaged from triplicate determinations.

3. Results and discussion

3.1. Characterizations of MgO nanoparticles

First, in order to analyze the thermal stability and decomposition behavior of $Mg(OH)_2$ into MgO, the obtained precursors were

Table 1
Some characteristics of as-prepared MgO samples.

Sample codes	Mean diameter (nm)	Mean length (nm)	Thickness (nm)	Morphology
0CTAB	22	77	–	Needle-like particles
1.5CTAB	91	–	25	Hexagonal-like platelet
2.2CTAB	75	–	27	Hexagonal-like platelet
2.9CTAB	49	–	19	Hexagonal-like platelet

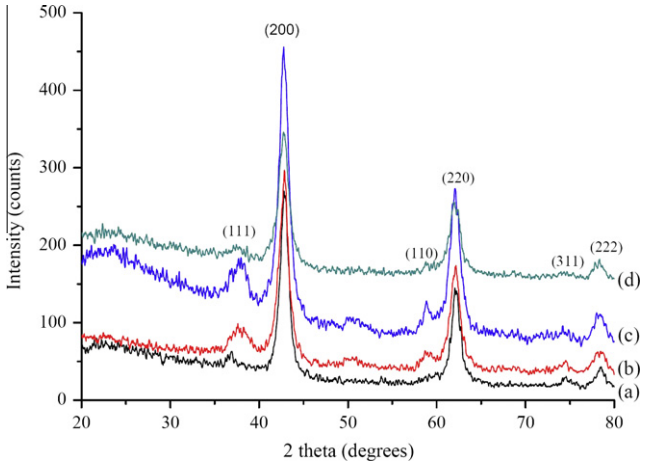

Fig. 1. XRD patterns of as-prepared samples with different amounts of CTAB (a) 0CTAB sample; (b) 1.5CTAB sample; (c) 2.2CTAB sample; (d) 2.9CTAB sample, calcined at 450 °C (remaining time 3 h).