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Synthesis and characterization of novel manganese oxide nanocorals and their application for the removal of methylene blue from aqueous solution

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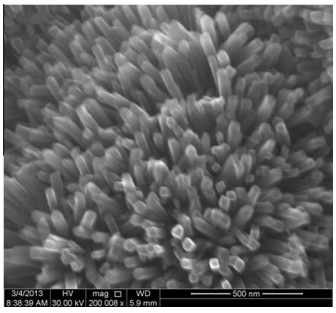
HIGHLIGHTS

- Novel manganese oxide nanocorals (MONCs) synthesized and characterized.
- MONCs were used for environmental remediation of model solution from methylene blue (MB) dye.
- The effect of different parameters that affected the adsorption process was explored.
- The adsorption was analyzed kinetically and thermodynamically.
- The MONCs exhibited a tremendous adsorption ability to remove MB from a model solution and a wastewater sample.

GRAPHICAL ABSTRACT



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ABSTRACT

Novel manganese oxide nanocorals (MONCs) were synthesized, characterized, and successfully used for the removal of toxic methylene blue (MB), as an example of organic dyes, from a model and the real solution. The MONCs were characterized by XRD, scanning and transmittance electron microscopes, and surface area analysis. The results confirm the formation of MONCs as nanocrystalline orthorhombic γ-MnO_2 phase with nanowires with diameters between 20.0 and 40.0 nm. The MONCs used for the removal of MB from aqueous solution and the different parameters that affected the adsorption process were studied and optimized. The adsorption process was studied kinetically and thermodynamically to elucidate the removal mechanism for better efficiency. The results showed that the removal process followed the pseudo-second-order kinetics model, and the spontaneity of the adsorption process at ambient temperatures. Finally, MONCs were used for the removal of MB ions from a wastewater sample, and the result indicated that they were highly efficient at removing the toxic MB.

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1. Introduction

One of the important methods used for the treatment of polluted environments is adsorption, because it is considered an efficient, cost-effective, versatile, and environmentally safe method; it does not generate secondary pollutants, and is characterized by the ability to regenerate both adsorbent and pollutants [1]. Nowadays, one of the great challenges in adsorption technologies is finding new potential adsorbents that might be used successfully for the removal of certain pollutants from different media. Recently, research scientists have explored and developed

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new materials that could be used as adsorbents for different pollutants. For example, zirconium-modified zeolites were used for the adsorption of phosphate from lake water [2], single-walled carbon nanotubes-doped walnut shell composite was used for the adsorption of lead from aqueous solutions [3], multi-walled carbon nanotubes decorated with ferrite nanoparticles were used for the adsorption of crystal-violet dye from aqueous solutions [4], surface-modified carbon materials such as activated carbon, carbon nanotubes and carbon xerogel, for the adsorption of the antibiotic ciprofloxacin from water [5,6], micro-mesoporous polymeric adsorbents were used for the adsorption of VOCs vapor [7], and modified kaolinites [8] and high surface-area mesoporous ceria [9] were used for the adsorption of CO_2 from air. However, there is still a great need to identify new types of adsorbent approaches characterized by a high adsorption capacity and rapid process kinetics. Manganese oxide nanoparticles are a group of emergent nanomaterials that have recently been synthesized, developed, and used in different applications [10–15]. Pristine and modified manganese oxide nanoparticles were used as solid adsorbents for the removal of heavy metal ions from aqueous solutions such as MB, As(V), Ag(I), Pb(II), Cu(II), Cd(II), Zn(II) [15–23], hydrogen sulfide [24], and anions such as phosphate [25] and fluoride [26]. However, the application of manganese oxide nanoparticles for the removal and remediation of organic pollutants from the aquatic environment is limited in the literature [27,28]; additional work is needed to explore the application of novel adsorbents such as manganese oxide nanoparticles as an efficient adsorbent for the removal of organic dye, as an example of organic pollutants—a major class of environmental pollutants that are persistent in the environment and cause adverse effects on both fauna and flora.

In the present work, the synthesis of manganese oxide nanocorals (MONCs) was studied for the first time. The prepared MONCs were characterized with different techniques; a scanning electron microscope (SEM), a surface area analyzer (SAA), and X-ray diffraction (XRD). The synthesized MONCs were used for the adsorption and removal of methylene blue dye (MB)—as an example or organic pollutants—from a model solution and a wastewater sample. The effects of the environmental and operational conditions, contact time, MNOCs mass, and solution pH on the adsorption/removal process were explored. The adsorption of the MB from the model solution was studied kinetically, using different kinetic models, and thermodynamically, to gain a better understanding of the adsorption process.

2. Materials and methods

2.1. Chemical

All chemicals were analytical grade and obtained from Sigma–Aldrich. The experiments were performed by using de-ionized water with resistivity that did not exceed 18.2 MΩ cm and that was obtained with a Millipore Milli-Q system (Billerica, USA). A stock solution; 100 mg L^{-1}, was prepared by dissolving a known quantity of MB dye in de-ionized water. The stock solution was further diluted to the desired concentration for other experiments.

2.2. Synthesis of manganese oxide nanocorals

Potassium permanganate solution (75.0 mL, 0.5 M) was heated to 343 K and stirred at 600 rpm by using a heater/magnetic stirrer, then NaOH solution (50.0 mL, 2.5 M) and manganese sulfate solution (75.0 mL, 2.0 M) were added drop-wise to the hot potassium permanganate solution using two separate burettes until a brown precipitate formed. The brown precipitate was then stirred over night at 343 K, filtered, washed with de-ionized water, and then dried in an oven at 383 K.

The MnO_2 was formed according to Eqs. (1) and (2):

$$MnSO_4 + 2NaOH \longrightarrow Mn(OH)_2 + NaSO_4 \quad (1)$$

$$3Mn(OH)_2 + 2KMnO_4 \longrightarrow 5MnO_2 + 2KOH + 2H_2O \quad (2)$$

2.3. Characterization techniques

A FEI-Field Emission Scanning Electron Microscope (FISEM) Quanta FEG 450 was used to characterize the morphological structure of the MONCs. TEM/STEM (JEOL 2100F) operating at 200 KV with a Field Emission Gun, obtaining a point resolution of 0.19 nm. X-ray diffraction (XRD) patterns were recorded for phase analysis and the measurement of crystallite size on a Philips X pert pro diffractometer, which was operated at 40 mA and 40 kV by using CuK_α radiation and a nickel filter in the 2θ range from 2° to 80° in steps of 0.02°, with a sampling time of one second per step. The specific surface areas of the MONCs were determined from nitrogen adsorption/desorption isotherms which were measured at 77 K by using a Nova 2000 series Chromatech. Prior to analysis, the samples were outgassed at 250 °C for 4 h.

2.4. Adsorption experiments

Kinetic experiments were carried out to establish the effect of time and temperature on the adsorption process and to identify the adsorption rate. The experimental procedures are described as follows: (1) A series of 10 ml solutions containing 40 mg/L of MB solution were prepared in a 250-ml glass bottle and kept at a certain temperature; (2) the pH of the solution was adjusted to 2.0 by using $NaOH/HNO_3$ solutions; (3) a certain amount of the MONCs was added to the solution; (4) the solution was shaken continuously for a designated period of time; (5) after the completion of preset time intervals, the solution was then immediately passed through filter paper to collect the supernatant; and (6) the residual MB concentration in the aqueous solution was then determined by a UV/Vis spectrophotometer at 662 nm. The percentage of removed MB in the solution was calculated using Eq. (3):

$$\%\ \text{removed} = \frac{(C_o - C_t)}{C_o} \times 100 \quad (3)$$

where C_0 is the initial MB concentration (mg L^{-1}); C_t is the final MB concentration (mg L^{-1}) after a certain period of time. The amount of MB adsorbed per unit mass of MONCs was calculated according to Eq. (4):

$$q_t = \frac{(C_o - C_t)V}{m}, \quad (4)$$

where q_t is the amount of MB ions adsorbed by the solid MONCs (mg g^{-1}) at any time (t); V is the initial solution volume (L); and m is the MONCs mass (g).

The adsorption experimental data was analyzed using the Lagergren pseudo-first-order kinetic model, and pseudo-second-order kinetics model [29].

The Lagergren pseudo-first-order rate equation can be written as follows:

$$\ln(q_e - q_t) = \ln q_e - k_1 t, \quad (5)$$

The pseudo-second-order kinetic model rate equation can be written as follows:

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