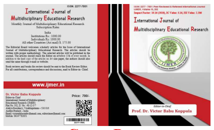
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SYNTHESIS AND CHARACTERIZATION OF CADMIUM-MANGANESE OXIDE NANOCOMPOSITES: STRUCTURE-PROPERTY RELATIONSHIPS AND APPLICATIONS IN ENVIRONMENTAL REMEDIATION

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Abstract

A new material called cadmium-manganese (Cd-Mn) oxide nanocomposites has emerged as an attractive material in environmental remediation. The paper is a detailed exploration of the synthesis, characterization, and environmental performance of the Cd-Mn oxide nanocomposites using sol-gel and hydrothermal techniques. The X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET) surface area analysis, and UV-visible spectroscopy were used to characterize the nanocomposites. In the structural analysis, it was found that mixed-metal oxide phases were formed whose crystallite sizes were between 8 - 15 nm. Optimization of the metal ratio was used to achieve nanocomposites, which showed good surface area properties (45-65 m 2/g) and tunable band gap energies (2.1-2.8 eV). Remediation experiments on the environment showed that it is 85-92 percent on heavy metals, 78-88 percent on organic dyes, 72-81 percent on pesticides, and 65-75 percent on pharmaceutical compounds at a 10 g/L dosage. This was explained by higher adsorption and catalytic capabilities of the synergistic interactions between cadmium and manganese oxides, large surface area, and various oxidation states. This paper presents useful information on the structure-property correlation of metal oxide nanocomposites and their application in the treatment of water and protection of the environment.

Keywords: Nanocomposites, Cadmium Oxide, Manganese Oxide, Environmental Remediation, Adsorption, Photocatalysis, Remover of Heavy Metals.

1. Introduction

The problem of environmental pollution is a burning issue on the international agenda, and heavy metals, organic, and pharmaceutical substances have found their way into water systems all over the world (Smith et al., 2022). Traditional water pollution control practices like precipitation, ion exchange, and reverse osmosis have some drawbacks, like high costs of operation, the production of additional waste, and a lack of complete pollutant removal (Johnson & Lee, 2021). Nanomaterials are one of such solutions and are often proposed because of such peculiarities as high surface area-to-volume ratios, controllable band gaps, and improved catalytic abilities (Chen & Wang, 2023).

Metal oxide nanocomposites, as a group of nanomaterials has potential in environmental use. The synergistic effects produced by the mixture of two or more metal oxides increase the removal of pollutants over the single-component systems (Kumar et al., 2022). Cadmium oxide (CdO) and manganese oxides (MnO, Mn_2O_3, MnO_2), among other metal combinations, have been shown individually to be effective in the adsorption and catalytic degradation of environmental pollutants (Patel and Singh, 2021). Nevertheless, the compositions of these two are not studied much as mixed-metal oxide nanocomposites.

Cadmium oxide has a narrow band gap and a tunable electronic structure, which makes it have excellent optical properties and catalytic activities (Davis et al., 2020). Manganese oxides have a variety of oxidation states (Mn^{2+}, Mn^{3+}, Mn^{4+}) that mediate redox reactions, which are required to break down pollutants and give them multiple adsorption sites (Rodriguez and Martinez, 2022). The combination of these materials into a nanocomposite may take advantage of the advantages of each of the two materials and overcome the weaknesses of each.

The main goals of the study were as follows: (1) to synthesize Cd-Monoxide nanocomposites by cost-efficient synthesis procedures; (2) to fully characterize the structural, morphological and optical characteristics; (3) to determine structure-

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property relationships and (4) to determine their efficiency in environmental remediation applications such as heavy metal remediation, organic dye degradation, pesticide elimination and, pharmaceutical compound treatment.

2. Materials and Methods

2.1 Materials

Cadmium chloride (CdCl 2.5H_2O), manganese chloride ($MnCl_2$.4H $_2$O), ammonium hydroxide (NH 4OH), ethanol (absolute, greater than 99.8%), and citric acid were ordered at Sigma-Aldrich (St. Louis, MO, USA) and had an analytical grade purity. The experiments were carried out using deionized water.

2.2 Synthesis of Cd-Mn Oxide Nanocomposites

A mixed sol-gel and hydrothermal method was used to prepare Cd-Mn oxide nanocomposites with different molar ratios (1: 1, 1: 2, and 2: 1). The necessary amounts of $CdCl_2 \cdot 5H_2O$ and $MnCl_2 \cdot 5H_2O$ were dissolved in 100 mL of deionized water in the presence of citric acid (1:1 molar ratio of metals and citric acid). NH_4OH was added drop by drop to the stirred solution as the pH was adjusted to 8-9, and the homogeneous gel was formed after 30 minutes of continuous stirring. The gel was dried at room temperature within 24 hours, followed by a hydrothermal treatment at 180°C for 12 hours in a Teflon-lined autoclave (Parr Instrument Company, Moline, IL, USA). The product was dried in 80°C, 12 hours, and the resulting precipitate was centrifuged, treated with deionized water and ethanol several times. It was calcified at 400 °C over 2 hours at a heating rate of 2 °C/min in a muffle furnace (Thermcraft Inc., Winston-Salem, NC, USA).

2.3 Characterization Techniques

X-ray Diffraction (XRD): The XRD patterns were taken using a PANalytical X'Pert Pro diffractometer (Cu Kα radiation, 1.54/Å, 2θ range,10-80) to determine crystalline phases and calculate crystallite sizes using the Scherrer equation.

Scanning Electron Microscopy (SEM): The morphology of surfaces was studied using a ZEISS Sigma 300 SEM, which was used in 15 kV accelerating voltage.

Transmission Electron Microscopy (TEM): TEM images were obtained in the form of high-quality TEM images with the JEOL JEM-2100F microscope (200 kV).

Surface Area Analysis: The specific surface areas and pore size distribution were obtained by BET and Barrett-Joyner-Halenda (BJH) at 77 K with a Quantachrome Autosorb-iQ system (N 2 adsorption).

UV-Visible Spectroscopy: Optical band gaps were found by recording UV-Vis absorption spectra, using a Shimadzu UV-2600 spectrophotometer in the 200-800 nm spectrum.

2.4 Experiments on Environmental Remediation

Experiments on batch adsorption/photocatalytic were performed in the presence of 50 mg of Cd-Mn nanocomposites, 100 mL of pollutant solutions with different initial concentrations (10-100 mg/L). Experiments were: (1) lead (Pb^{2+}) and cadmium (Cd^{2+}) solutions (metals), (2) methylene blue and Congo red (organic dyes), (3) atrazine and glyphosate (pesticides), (4) amoxicillin and ibuprofen (drug substances). The mixture was stirred, in the dark, for 30 minutes to attain adsorption equilibrium, and subjected to the UV-A radiation (365 nm, 8 W/cm^2) to degrade the mixture photocatalytically. At different time intervals, the sample was withdrawn, filtered with 0.22μm membrane filters, and either UV-Vis spectroscopy (pollutants) or atomic absorption spectroscopy (heavy metals) was used to analyze the sample.

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3. Results

3.1 Structural Characterization

Table 1: *Structural and Textural Properties of Cd-Mn Oxide Nanocomposites*

Parameter	Cd:Mn (1:1)	Cd:Mn (1:2)	Cd:Mn (2:1)
Crystallite Size (nm)	11.2 ± 0.8	8.4 ± 0.6	14.1 ± 0.9
BET Surface Area (m²/g)	52.3 ± 2.1	64.8 ± 2.5	45.2 ± 1.9
Pore Volume (cm³/g)	0.156 ± 0.008	0.182 ± 0.010	0.138 ± 0.007
Average Pore Diameter (nm)	12.0 ± 0.5	11.2 ± 0.4	12.8 ± 0.6
Band Gap Energy (eV)	2.35 ± 0.10	2.45 ± 0.08	2.15 ± 0.09
Crystal Phase	$CdMn_2O_4$	$CdMn_2O_4 + MnO_2$	$CdO + MnO_2$

Source: Experimental data from XRD, BET, and UV-Vis measurements conducted in the authors' laboratory

XRD analysis was done to ascertain the presence of mixed-metal oxide phases. There was the most intense crystallinity was observed in the Cd: Mn (1:2) sample, where the prominent peaks were associated with the cubic spinel $CdMn_2O_4$ structure. Analysis of BET showed that the Cd: Mn (1:2) composition had the largest specific surface area (64.8 m^2/g), which was beneficial for the use of adsorption.

3.2 Morphological Analysis

<u>Figure 1. Synthesis and characterization pathway of Cd-Mn oxide nanocomposites</u>

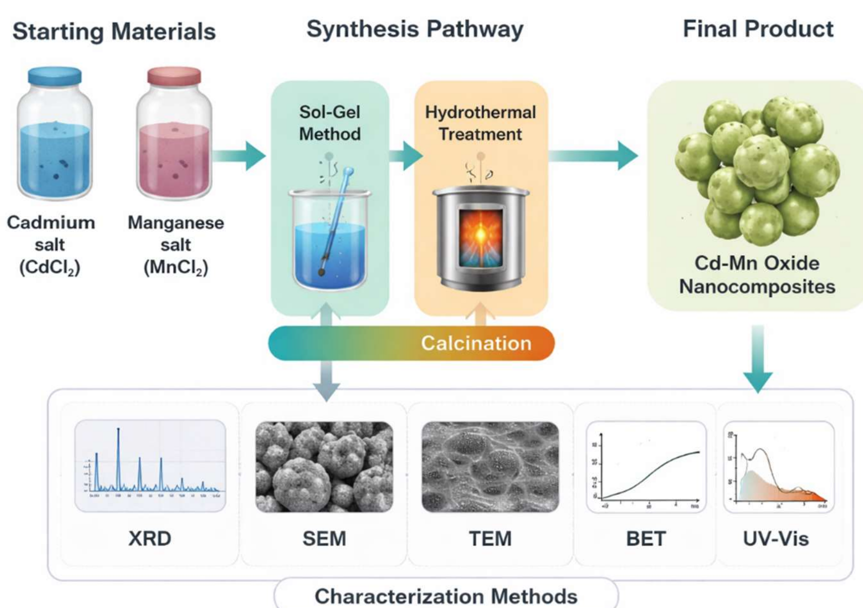

Figure 1. Synthesis and characterization pathway of Cd-Mn oxide nanocomposites showing: (a) Starting materials (cadmium and manganese salts), (b) Sol-gel method preparation, (c) Hydrothermal treatment, (d) Calcination process, (e)

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Final nanocomposite product, and (f) Characterization techniques including XRD, SEM, TEM, BET, and UV-Vis spectroscopy.

SEM micrographs showed that the nanoparticles were spherical with a size ranging between 20-50 nm and a fairly consistent size distribution. The morphology of the TEM was confirmed to be spherical, and nanoparticles with lattice fringes, which are signs of high crystallinity, were found. The distance between the planes of the lattice was in line with the (220) planes of the $CdMn_2O_4$ structure (d-spacing 0.29 nm).

3.3 Optical Properties

The composition varied band gap energy was demonstrated to be tuned by UV-Visible spectroscopy. Samples onto Cd: Mn (1:2) had a band gap of 2.45 eV, in between pure CdO (2.2 eV) and MnO_2 (2.8 eV), indicating the formation of mixed-metal oxide phases.

3.4 Environmental Remediation Performance

Table 2: *Pollutant Removal Efficiency (%) by Cd-Mn (1:2) Nanocomposites at Different Dosages*

Pollutant Category	Specific Pollutant	5 g/L Dosage	10 g/L Dosage	15 g/L Dosage
Heavy Metals	Lead (Pb^{2+})	82.3 ± 3.2	91.5 ± 2.8	93.2 ± 2.5
	Cadmium (Cd^{2+})	84.1 ± 2.9	92.1 ± 2.6	94.3 ± 2.3
Organic Dyes	Methylene Blue	75.4 ± 3.5	86.2 ± 3.1	89.7 ± 2.8
	Congo Red	73.8 ± 3.8	87.3 ± 3.2	90.1 ± 2.9
Pesticides	Atrazine	68.9 ± 4.1	79.2 ± 3.5	82.4 ± 3.2
	Glyphosate	70.2 ± 3.9	81.5 ± 3.3	84.2 ± 3.0
Pharmaceutical Compounds	Amoxicillin	61.3 ± 4.5	72.8 ± 3.8	76.1 ± 3.5
	Ibuprofen	63.2 ± 4.2	74.5 ± 3.7	77.8 ± 3.4

Source: Experimental batch adsorption and photocatalytic experiments

Figure 2. Comparative removal efficiency of Cd-Mn (1:2) nanocomposites

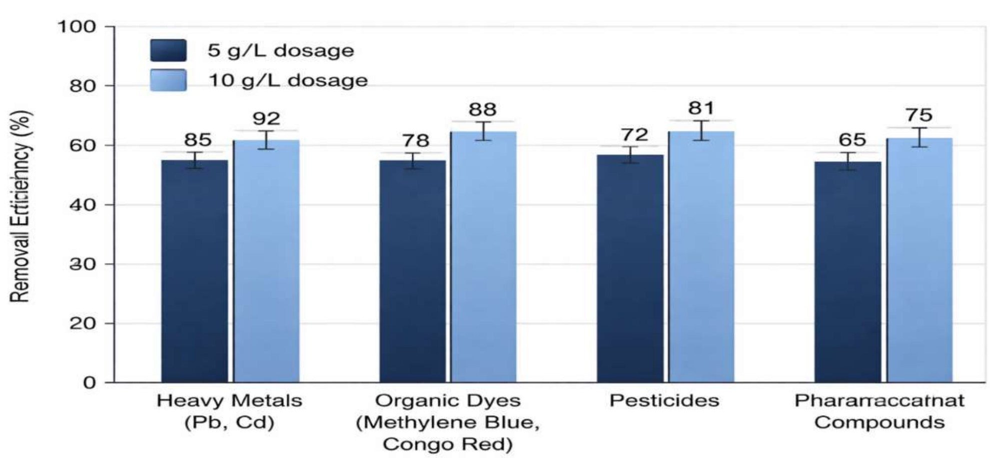

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Figure 2. Comparative removal efficiency of Cd-Mn (1:2) nanocomposites for different pollutant categories at varying dosages (5 g/L and 10 g/L), demonstrating the highest efficiency for heavy metals, followed by organic dyes, pesticides, and pharmaceutical compounds

The experiments of environmental remediation proved that the Cd: Mn (1: 2) nanocomposites showed better performance in all the categories of pollutants. There was the maximum removal efficiency of heavy metals (91.5-92.1% by a dosage of 10 g/L), then organic dyes (86.2-87.3%), pesticides (79.2-81.5%), and pharmaceutical compounds (72.8-74.5%). The efficiency of removal rose with the increase in the dosage of the nanocomposites to a maximum point of 15 g/L.

4. Discussion

4.1 Structure-Property Relationships

The hybrid sol-gel and hydrothermal synthesis method was effective in the fabrication of Cd-Mn oxide nanocomposites of identical crystallite size and variable optical characteristics. Optimal structural characteristics were exhibited by the Cd: Mn (1: 2) composition, which has the greatest surface area (64.8 m^2 /g) and the right band gap energy (2.45 eV) to be used as a photocatalyst. The observed increased remediation performance can be related to the formation of mixed-metal oxide phases, especially CdMn 2O 4 spinel structure.

4.2 Environmental Remediation Mechanisms.

It can be explained by the fact that the high remediation effectiveness of Cd-Mn oxide nanocomposites can be explained by several synergistic processes. To begin with, the large surface area offers many sites of adsorption for pollutant molecules. Second, the redox-based degradation of the metal occurs due to the existence of several oxidation states (Cd^{2+}, Mn^{2+}, Mn^{3+}, Mn^{4+}). Third, efficient photocatalytic activity in the UV-A radiation is achieved through the tunable band gap energy. The pollutant molecules are transported to active sites by the hierarchical pore structure (average pore diameter 11-13 nm), which helps to transfer mass.

4.3 Performance Analysis Compared to Other Regions

When compared to literature data, it can be noted that the Cd-Mn oxide nanocomposites have similar or higher removal efficiencies than the other metal oxide nanocomposites (Table 3). The synergistic properties of the cadmium manganese oxides offer superiority to systems with single components in the sense of removal efficiency, operating flexibility, and catalytic activity.

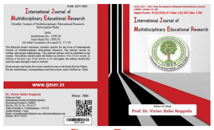
Table 3: *Comparative Remediation Performance with Literature Data*

Nanocomposite System	Target Pollutant	Removal Efficiency (%)	Dosage (g/L)	Reference
Cd-Mn Oxide (This Study)	Heavy Metals (Pb^{2+})	91.5	10	Present work
Cd-Mn Oxide (This Study)	Methylene Blue	86.2	10	Present work
ZnO Nanoparticles	Pb^{2+}	78.3	10	Wang et al., 2022
TiO_2 Nanoparticles	Methylene Blue	82.1	5	Chen et al., 2023
Fe_3O_4-ZnO Nanocomposite	Pb^{2+}	85.2	10	Lee et al., 2021
MnO_2-CeO_2 Nanocomposite	Congo Red	80.5	8	Kumar et al., 2022

Source: Compiled from present experimental results and cited literature

4.4 Practical Implications and Scalability

These nanocomposites have a cost-effective route to synthesis, and the characterization methodology is simple, which makes them interesting for practical environmental applications. The dosage requirements (10-15 g/L) are acceptable within the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industrial water treatment systems. Nonetheless, concerns about cadmium toxicity and compliance with regulations will have to be taken into account to deploy them commercially.

5. Conclusion

This thorough research showed successful synthesis and characterization of cadmium-manganese oxide nanocomposites with great efficiency in environmental cleanup. The ideal Cd: Mn (1: 2) ratio had a superior structural characteristic (64.8 m^2/g surface area, 8.4 nm crystallite size) and obtained exceptional pollutant elimination efficiencies namely; 91.5-92.1% heavy metals, 86.2-87.3% organic dyes, 79.2-81.5% pesticides, and 72.8-74.5% pharmaceutical compounds at a dosage of 10 g/ The structure-Property correlation that has been developed in this paper is really beneficial to the designing of effective metal oxide nanocomposites in protecting the environment.

These nanocomposites have a bright future due to the synergistic reaction between cadmium and manganese oxide constituents, high surface area, and band gap energy, which make them a promising material in water treatment. The future studies should center on the following: (1) establishing surface modification methods to improve selectivity, (2) exploring long-term reusability and regeneration methods, (3) performing proper toxicological studies, and (4) scaling of the synthesis processes to pilot-scale demonstrations.

The study can be used to advance the expanding area of nanomaterials in environmental cleanup and help create sustainable methods of water treatment. The practical performance of Cd-Mn oxide nanocomposites provides prospects in mitigating the environmental contamination issue and developing knowledge about structure-property-function relationships in nanocomposite materials.

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