RESEARCH PAPER

Synthesis and Characterization of TiO_2-(MoO_3)/Al_2O_3 Nanocomposite Using Hydrothermal Method for Environmental Application

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ARTICLE INFO

Article History:
Received 10 September 2022
Accepted 20 December 2022
Published 01 January 2023

Keywords:
Aluminium oxide
Hydrothermal method
Molybdenum Trioxide
Nanocomposite
Oxidative degradation
Titanium dioxide

ABSTRACT

In the current research, a hydrothermal synthesis was used to create a nanocomposite of titanium dioxide (TiO_2), aluminum oxide (Al_2O_3), and molybdenum trioxide (MoO_3) for use in possible environmental applications. With the assistance of calcination at temperatures of 400 and 800 degrees Celsius, direct hydrothermal synthesis of TiO_2-Al_2O_3/MoO_3 powder was effectively accomplished in the presence of ethanol at low pH values at 70 degrees Celsius. The zeta potential and dynamic light scattering techniques, together with scanning electron microscopy (SEM), were utilized in order to evaluate the physicochemical features of the nanoparticles (DLS). Additionally, energy dispersive x-ray (EDX) was utilized in order to do an element distribution analysis on the nanocomposite that was manufactured. According to the findings, a TiO_2-Al_2O_3/MoO_3 nanocomposite with an average crystal size of 36.1 nm was successfully manufactured. According to the findings, the new features of this nanocomposite have the potential to be utilized in the development of future environmental applications.

How to cite this article
Radhi A., Al-Hilfi J A., Albukhaty S. Synthesis and Characterization of TiO_2-(MoO_3)/Al_2O_3 Nanocomposite Using Hydrothermal Method for Environmental Application. J Nanostruct, 2023; 13(1):104-109. DOI: 10.22052/JNS.2023.01.012

INTRODUCTION

Nanoparticles are used in many aspects of human existence, including in medical, industrial, and environmental applications because of their tunable physicochemical properties resulting from their ultrafine size and high surface area [1-5]. Numerous applications, including drug delivery, tissue engineering, biosensing, nanomedicine, photocatalysis, and electrochemical sensors, make extensive use of nanoparticles [5-10]. Nanoparticles are produced using a variety of techniques, including laser ablation, chemical coprecipitation, sonochemistry, sol-gel, and hydrothermal methods [10-15], the hydrothermal technique is the most effective way to produce nanoparticles of the methods mentioned. This process doesn't require calcination; it merely uses heat and water as a solvent. The appropriate alignment of crystals and the requirement for their growth at high temperatures and pressures are additional advantages of this technology. Typically, a pressure of less than 25 MPa and a temperature of less than 300 °C are needed for a hydrothermal reaction [16]. Nano-adsorbents for treating wastewater have recently received a lot of attention due to their large surface areas and flexibility in surface modification [17]. TiO_2 and other photocatalyst nano-composites have

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undergone extensive research as traditional photocatalysts in several sectors over the past few decades [18]. Wahyuono, Ruri, et al. also found that TiO_2 mixed (WO_3) nanocomposite photocatalyst was a powerful adsorbent for methylene blue in an aqueous media. [19]. Numerous investigations have demonstrated that connecting or doping other oxides can improve the performance activity of adsorption. Hanh, Nguyen Thi, et al. [20], for instance, were successful in synthesizing Co_3O_4/N, S-TiO_2 nanoparticles to improve the properties for using accelerated degradation of Direct Blue under visual irradiation.

Managing environmental quality will be of utmost importance in the near future. In fact, as a result of environmental pollution and/or shortage, regulatory systems would tighten in order to decrease the impact of waste pollutants on the environment and to allow for the recycling of the environment. The standards for tap water quality also need to be adjusted to account for the recently identified contaminants that are being found in rivers or soil. These commitments won't be met until new, efficient, and effective water treatment technologies are developed. Nanomaterials are advancing the study of more effective oxidation processes [21]. Alternative operating procedures like photo-active nanoparticles, which influence the oxidation of pollutants found in industrial and wastewater effluents and work through the hydroxyl radical to influence the degradation of organic species, have drawn a lot of interest as alternative treatment methods (OH) [22,23]. Strong oxidizing agents and highly reactive hydroxyl radicals with one free electron pair act as the main catalyst for the breakdown of a variety of organic pollutants, such as dyes, aromatic compounds, or chlorinated hydrocarbons, into carbon dioxide, organic acids, and inorganic ions as end products [24]. The amount of high-temperature and high-pressure regions was increased due to the presence of semiconductor (i.e., TiO_2, Al_2O_3) particles, which improved the process of breaking up the microbubbles produced by the ultrasonic irradiation into smaller bubbles [25] This causes an increase in the number of hydroxyl radicals produced, which attack the pollutant and cause it to degrade.

Additionally, nanocomposites are highly intriguing. To suggest environmentally responsible solutions to the world's environmental challenges, this study has concentrated on the evaluation of a novel TiO_2-(MoO_3)/Al_2O_3 nanocomposite for environmental applications that were hydrothermally synthesized and characterized.

MATERIALS AND METHODS

Alumina (nano material), Sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) from Merck, Germany Titanium isopropoxide (97%, Sigma-Aldrich), ethanol (99.9%), hydrochloric acid (37%), Ascorbic acid, Sodium decyl sulfate (SDS).4

Procedure

Prepare TiO_2 nano articles was as carried out according to previous published study by Mahata, S., et al. [26]. With some modifications. In brief, a combination of (2.5 ml) ethanol and (3.5 ml) diluted HCl was stirred while titanium isopropoxide (5 ml) was added dropwise to generate a clear solution. 10 ml of SDS (1.0 wt%) was added, and after being stirred for 15 seconds at room temperature, the mixture was placed to an autoclave lined with Teflon and heated to 110 C for 24 hours. The product was centrifuged, rinsed with water and ethanol, then dried at 60 for 24 hours after cooling to ambient temperature.

According to Michailovski, Alexej, and Greta R. Patzk [27] - MoO_3 nano-belts have been prepared with some modifications by using 1 mmol of sodium molybdate dihydrate and 7 ml of diluted HCl (added in the form of drops) while stirring for 15 mintues. Then, 10 ml of ascorbic acid solution was added while mixing with a magnetic stirrer. the mixture was transferred to steel Teflon tube autoclave and hydrothermal reaction was carried out at 180 for 6 hr , the result is also separated and washed with water and ethanol , and dried in an oven at 70 for 5 hr.

Using the ultrasonic technique, the TiO_2- Al_2O_3/MoO_3 nanocomposite was made with a weight/weight ratio of (0.25gm/0.5gm/0.25gm) accordingly.

The required quantities of (powder) nanomaterials were dissolved in ethanol and placed in each component's own baker before being shocked for two hours at 70 with an ultrasonic frequency of 60 HZ. The smaller solutions are then added to the bigger ones in the shape of drips while they are still in the ultrasonic at 70 . After that, the mixture is kept for an hour.

The product is then transferred from the solution to a magnetic stirrer while the ethanol is still liquid, washed with ethanol and water, and dried overnight at 60 .

The material was calcined at a temperature of 400, or 800 °C for 2 hours in ambient air (Germany's Nabertherm P320 Controller) as the last phase. These calcination temperatures were chosen to enhance the synthetic binary oxide systems' physicochemical characteristics, such as their crystalline structure.

Characterization

The size and stability of nanocomposites were determined using DLS and Zeta potential, respectively. SEM was used in conjunction with EDX to analyze the surface morphology and determine the elemental composition of the samples.

RESULTS AND DISCUSSION

The process of hydrothermal synthesis is the most widely used method for creating nanomaterials. It essentially employs a methodology based on solutions and reactions. From very low to very high temperatures, hydrothermal synthesis can be used to create nanomaterials. Low-pressure or high-pressure circumstances can be used to regulate the morphology of the materials to be synthesized, depending on the vapor pressure of the primary component in the process.

Both Al_2O_3 and TiO_2 are utilized as distinct catalysts for numerous chemical activities and have a number of benefits. Particularly, TiO_2 has high photocatalytic activity under UV irradiation but a very small specific surface area, whereas Al_2O_3 has better heat stability and a higher specific surface area but worse catalytic properties. Many researchers have tried to address these issues [28]. By combining the distinctive structural characteristics of the different oxides, materials with the benefits of the different oxides can be created, such as Mo/TiO_2 materials for high-performance oxidative desulfurization [29].

Figs. 1a and b show the synthesized nanocomposite's DLS and Zeta potentials analyses. The samples with 154 nm particle size, high (41.0 mV) Zeta potentials, and good values (0.700) of polydispersity index are suggestive of an efficient synthesis, according to the data (PDI),

Nano size is one of the essential properties of the synthesized material due to its effect on surface area.

SEM and EDX studies were used to identify the

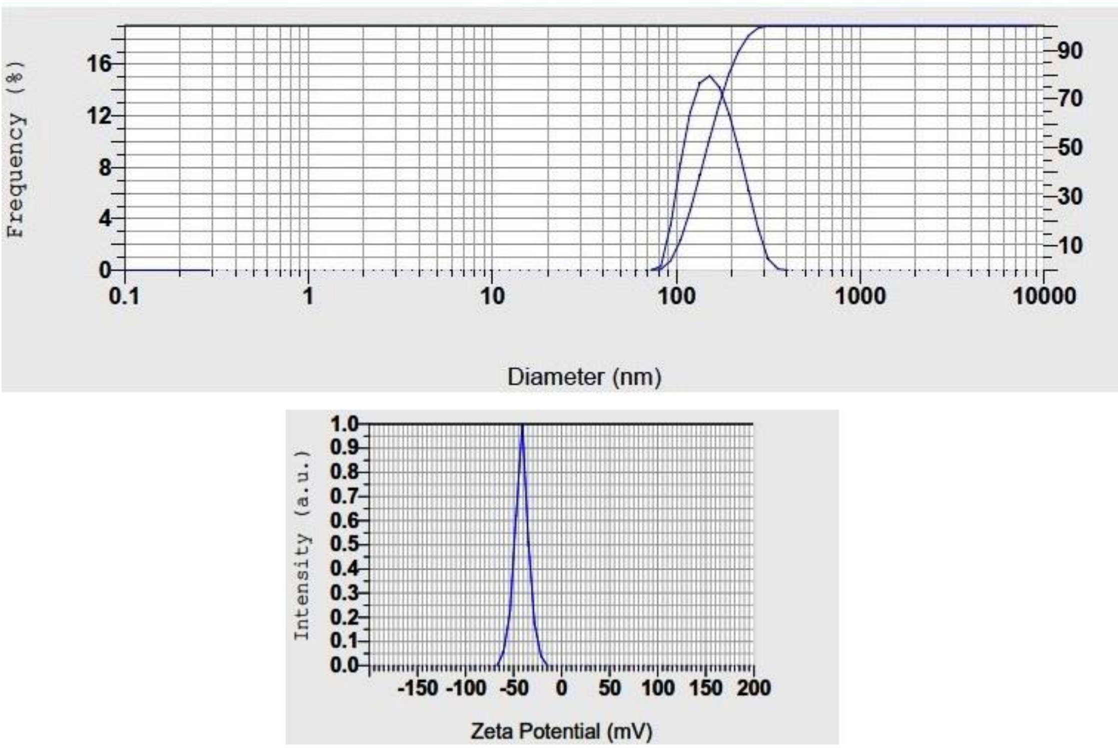

Fig. 1. The size distribution of prepared nanocomposite by dynamic light scattering DLS and Zeta potential value of prepared Nanocomposite

morphology and components of the composite. SEM images are displayed in Fig. 2A and the EDX Fig 2C.

The generated oxide NPs have a similar shape. The NPs materials revealed aggregated spherical particles with average size about 35 nm. The purity

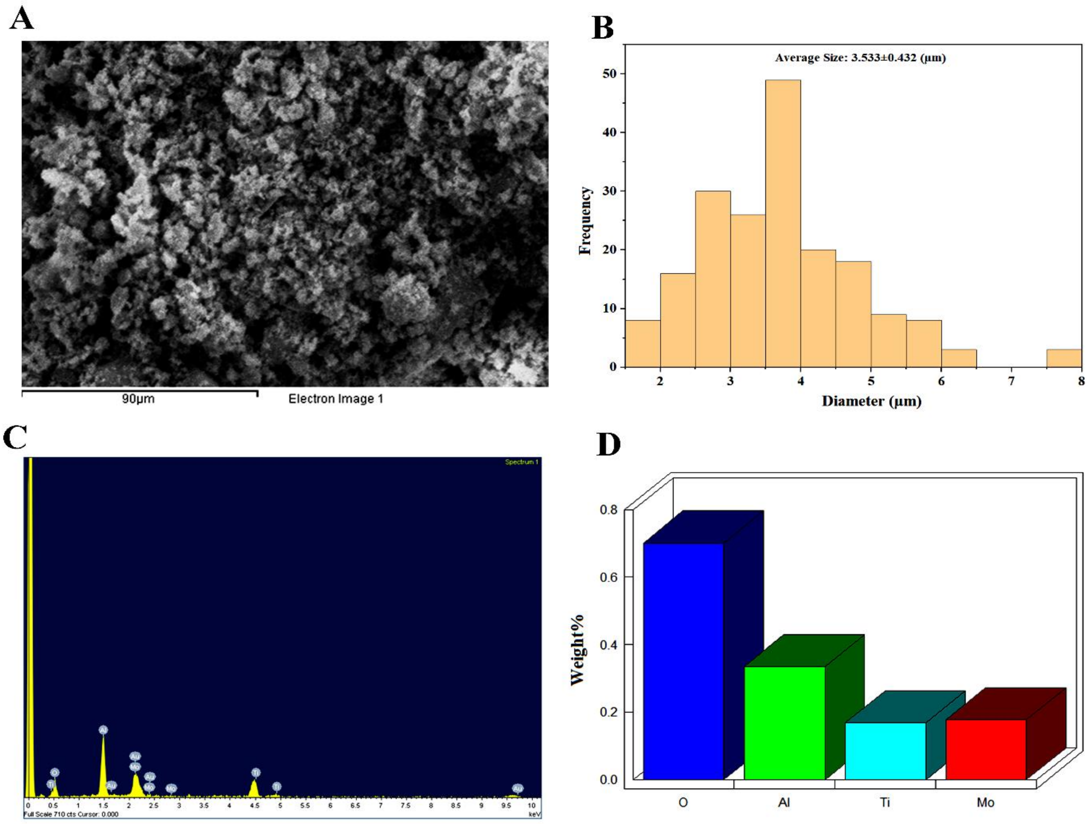

Fig. 2. A) SEM image, B) particle size distribution, C) EDX spectrum, and D) elemental analysis of prepared TiO_2- Al_2O_3/MoO_3 nanocomposite respectively.

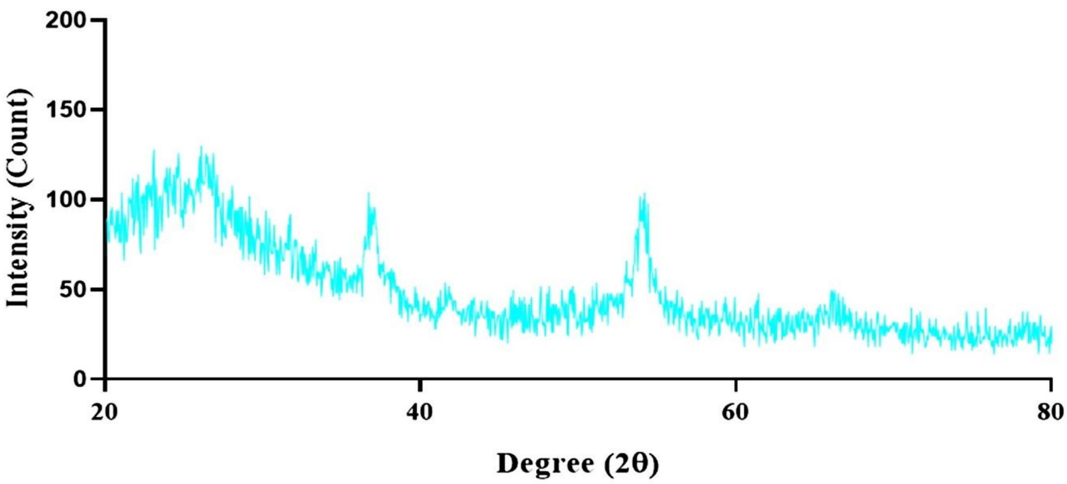

Fig. 3. The XRD patterns of prepared nanocomposite.

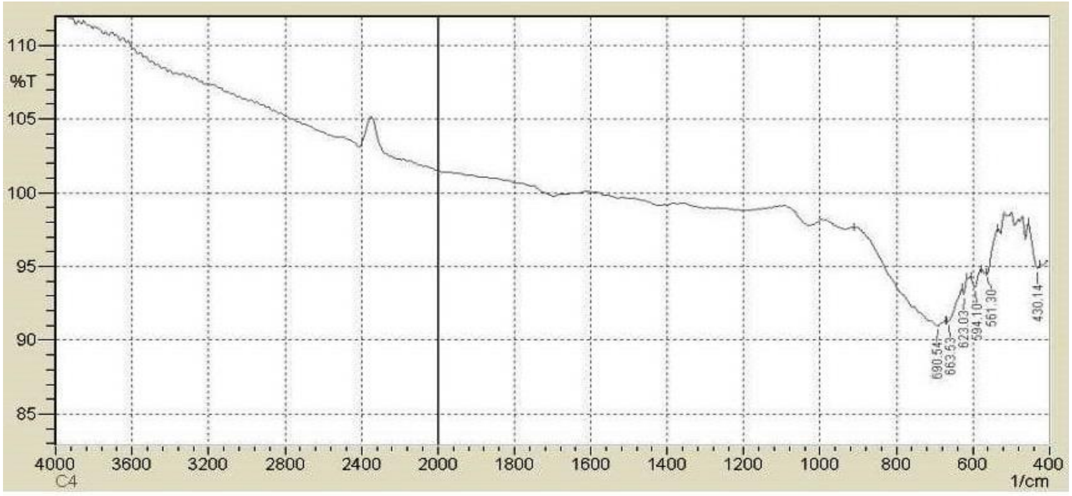

Fig. 4. The FTIR spectrum of prepared nanocomposite.

of the TiO_2- Al_2O_3/MoO_3 NPs was confirmed by an EDX test (Fig. 2C,D)

Therefore, while colloids with low zeta potentials coagulate or flocculate, those with high negative or positive zeta potentials are stabilized [23,24]. Intermolecular interaction, lattice mismatch, and the presence of residual oxides were thought to be the causes of the variance in surface shape [64]. EDX examination, which exclusively detects aluminum, titanium, molybdenum, and oxygen, proved the purity of TiO_2- Al_2O_3/MoO_3 NPs.

The crystalline phases of anatase TiO_2 and MoO_3 and Al_2O_3 observed in the XRD patterns of composite are shown in Fig. 3 and the patterns are well correlated with the JCPDS files (01-071-1167, 00-005-0506, and JCPDS 46-1212)).), respectively. It confirms the formation of crystalline MoO_3 and alumina phase on the titanium dioxide surface Fig. 3.

The FT-IR spectra for all of the materials contained four characteristic bands: for stretching vibrations of ≡Ti–O (623 cm^{-1}) and Al–O–Ti/Mo (690 cm^{-1}) and for hydroxyl group (–OH) bending vibrations (1600 cm^{-1}) and stretching vibrations (3500 cm^{-1}) (Fig. 4). The calcination temperature was found to influence the intensity of the characteristic bands for Ti–O and Al–O–Ti-Mo. The FT-IR analysis proved the effectiveness of the proposed synthesis methodology.

CONCLUSION

In conclusion, we have created a simple hydrothermal process that has produced a well-characterized TiO_2-Al_2O_3/MoO_3 nanocomposite. The shape of the TiO_2-Al_2O_3/MoO_3 nanocomposite, size distribution, stability, dispersity, and zeta potentials all supported the validity of the good formation findings. In each of the aforementioned scenarios, we have identified how temperature and pressure affect the physicochemical properties of the TiO_2-Al_2O_3/MoO_3 nanocomposite intended for environmental applications.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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