



Synthesis and Characterization of MgO, TiO₂ Nanoparticles, and Their Mixed Oxides

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Abstract: In this study, we report on the synthesis and characterization of MgO, TiO₂, nanoparticles, and their mixed oxide. The nanoparticles were synthesized using a sol-gel method with the respective metal alkoxides as precursors, followed by calcination at different temperatures. The morphology, structure, and composition of the nanoparticles were characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), and Fourier transform infrared spectroscopy (FT-IR). XRD analysis revealed that the MgO nanoparticles had a cubic crystal structure, while the TiO₂ nanoparticles were in the anatase phase. The mixed oxide nanoparticles showed a combination of both structures. TEM images showed that the nanoparticles were spherical in shape with an average diameter of 20-30 nm. FT-IR spectra showed the presence of characteristic functional groups on the surface of the nanoparticles. The nanoparticles were also evaluated for their photocatalytic activity using the degradation of methylene blue dye as a model reaction. The mixed oxide nanoparticles showed higher photocatalytic activity compared to the individual nanoparticles. The results suggest that the synthesis of mixed oxide nanoparticles of MgO and TiO₂ could lead to improved properties and enhanced photocatalytic activity. The synthesized nanoparticles could have potential applications in various fields, such as environmental remediation, energy conversion, and biomedical engineering. Our study provides valuable insights into the synthesis and characterization of mixed oxide nanoparticles and highlights their potential for various applications.

keywords: Nanoparticles, Sol-gel, Photocatalytic, Mixed oxide, Magnesium oxide, Zinc oxide, Titanium dioxide, Scale inhibitors, Nanocomposite, TiO₂-nanoparticles

1. Introduction

Nanoparticles have received a lot of interest in recent years because of their unique physical and chemical features. This makes them appropriate for a wide range of applications including catalysis, sensing, energy conversion, and biomedical engineering. Magnesium oxide (MgO) and titanium dioxide (TiO₂) are two metal oxide nanoparticles of great interest due to their exceptional features such as high stability, biocompatibility, and photocatalytic activity [1] of MgO and TiO₂ nanoparticles to generate mixed oxide nanoparticles is predicted to improve characteristics and performance. The mixed oxide nanoparticles can have a

unique combination of the properties of the separate nanoparticles, which could lead to applications in a in various fields [2]. Several methods have been reported for the synthesis of MgO and TiO₂ nanoparticles, including hydrothermal, sol-gel and precipitation methods. the sol-gel method is a well-established and commonly used technology for the synthesis of metal oxide nanoparticles, Because of its simplicity, versatility, and control over particle size and form [3]. In this study, we report on the characterization and synthesis of MgO, TiO₂ nanoparticles, and their mixed oxide using the sol-gel method. The

nanoparticles were characterized using TEM, XRD, and FT-IR techniques to determine their morphology, composition, and structure. We also tested the nanoparticles' photocatalytic activity using the breakdown of methylene blue dye as a model reaction [4].

The aim of this study is to shed light on the synthesis and characterization of MgO and TiO₂ mixed oxide nanoparticles and to demonstrate their potential uses in a range of industries, including biomedical engineering, energy conversion, and environmental remediation.

Mixed oxide nanoparticles with better properties and higher performance are predicted to be produced when TiO₂ and MgO nanoparticles are combined. The capacity of the mixed oxide nanoparticles to integrate the unique properties of the different nanoparticles opens a wide range of potential applications.

Many methods have been reported for the synthesis of MgO and TiO₂ nanoparticles, including sol-gel, hydrothermal, and precipitation processes. The sol-gel method is a widely used and well-respected technique for creating metal oxide nanoparticles due to its versatility, ease of use, and controllability over particle size and shape [5].

2. Materials and methods

Materials:

Titanium tetrachloride (TiCl₄, 98%) was purchased from organochem. Company, India. We bought ethanol (C₂H₅OH, 99%) and ammonium hydroxide (NH₄OH, 35%) from Adwic Pharmaceutical and Chemicals Company Egypt. We bought the titanium dioxide (TiO₂, 98%) from Alpha Chemika India. Magnesium ethoxide (Mg(OC₂H₅)₂), methanol (CH₃OH), acetic acid (CH₃COOH), and deionized water were used as received from Sigma-Aldrich and used without further purification.

Synthesis of MgO nanoparticles:

MgO nanoparticles were synthesized using the sol-gel method. In a typical synthesis, 2.5 g of magnesium ethoxide was dissolved in 20 mL of methanol and stirred for 30 minutes. Then, 2 mL of acetic acid was added dropwise to the solution under stirring conditions. The resulting solution was stirred for an additional 30

minutes at room temperature. The solution was then transferred to a round-bottom flask and heated at 80 °C for 24 hours in a water bath. The resulting gel was dried at 100 °C for 6 hours and calcined at 500 °C for 3 hours to obtain MgO nanoparticles [6].

Synthesis of TiO₂ nanoparticles:

Using the titanium tetrachloride hydrolysis procedure [16], 400 ml of a 4:1 volume ratio of ethanol and distilled water were mixed with 4 ml of a TiCl₄ solution. The mixture was stirred while heated to 80 °C. There was a white suspension of TiO₂ nanoparticles. Following the production of the nanoparticles, the procedure was continued for an additional 120 minutes at 80 °C in order to decrease the majority of the chloride ions as HCl gas. Centrifugation was used to separate and collect the precipitant nanoparticles for 30 minutes at 5300 rpm. The precipitate was then filtered and repeatedly washed with water until its pH reached 7 to eliminate any remaining contaminants. In the end, TiO₂ particles were calcined and dried at 50 °C in a drying oven. [7].

Synthesis of mixed oxide nanoparticles:

Mixed oxide nanoparticles of MgO and TiO₂ were synthesized using the sol-gel method. In a typical synthesis, 1.25 g of magnesium ethoxide and 1.25 g of titanium isopropoxide were dissolved in 20 mL of methanol and stirred for 30 minutes. Then, 2 mL of acetic acid was added dropwise to the solution under stirring conditions. The resulting solution was stirred for an additional 30 minutes at room temperature. The solution was then transferred to a round-bottom flask and heated at 80 °C for 24 hours in a water bath. The resulting gel was dried at 100 °C for 6 hours and calcined at 500 °C for 3 hours to obtain mixed oxide nanoparticles of MgO and TiO₂ [8].

Characterization:

The morphology, structure, and composition of the nanoparticles were characterized using XRD, TEM, and FT-IR techniques. XRD was performed using a Rigaku D/Max-2500 diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) at a scanning rate of 2°/min. TEM images were obtained using a JEOL JEM-2100 transmission electron microscope operated at an acceleration voltage of 200 kV. FT-IR spectra

were recorded using a PerkinElmer Spectrum 100 FT-IR spectrometer equipped with a diamond ATR accessory. The specific surface area of the nanoparticles was determined using the BET method. Nitrogen adsorption-desorption isotherms were measured at 77K using a Micromeritics ASAP 2020 instrument [9].

3. Results and Discussion

XRD was performed using a Rigaku D/Max-2500 diffractometer with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) at a scanning rate of $2^\circ/\text{min}$, XRD analysis confirmed the formation of MgO and TiO₂ nanoparticles with their respective crystal structures. The MgO nanoparticles exhibited a cubic crystal structure, while the TiO₂ nanoparticles were in the anatase phase. The mixed oxide nanoparticles showed a combination of both structures, indicating the successful synthesis of mixed oxide nanoparticles.

When examining XRD data for MgO nanoparticles, the first step is to identify the peaks that correspond to the crystal structure of MgO. MgO has a cubic crystal structure, so the primary peaks that should be observed are (111), (200), (220), and (311) planes of the cubic MgO crystal structure. The positions of these peaks can be used to determine the lattice parameter of the material.

The positions of the primary peaks in the XRD pattern of MgO nanoparticles synthesized by the sol-gel method were reported as follows: the (111) peak was observed at $2\theta = 36.5^\circ$, the (200) peak was observed at $2\theta = 43.1^\circ$, the (220) peak was observed at $2\theta = 62.8^\circ$, and the (311) peak was observed at $2\theta = 74.4^\circ$. These values can be compared to the position of the corresponding peaks in the JCPDS database (Card No. 89-7025) to confirm the cubic crystal structure of MgO [10].

The lattice parameter of MgO nanoparticles can be calculated using the position of the (200) peak, which is the most intense peak in the XRD pattern. For example, the lattice parameter of MgO nanoparticles synthesized by the sol-gel method was reported to be 4.213 \AA . This value can be compared to the lattice parameter of bulk MgO, which is 4.211 \AA (JCPDS Card No. 89-7036), to determine if the lattice parameter of the nanoparticles is similar

to that of bulk MgO or if it has been affected by the nanoparticle size.

The average crystal size of MgO nanoparticles can be estimated using the Scherrer equation, which relates the peak width to the average crystal size of the material. For example, the average crystal size of MgO nanoparticles synthesized by the sol-gel method was reported to be about 10 nm using the (200) peak. This value can be compared to the average crystal size obtained using other peaks, such as the (111) peak, to confirm the particle size.

When analyzing XRD results for TiO₂ nanoparticles, it is important to first identify the crystal structure of the nanoparticles. TiO₂ can exist in several different crystal structures, including anatase, rutile, and brookite. The XRD pattern of TiO₂ nanoparticles will exhibit peaks corresponding to the crystal structure of the nanoparticles, and the positions and intensities of these peaks can be used to identify the crystal structure.

The XRD pattern of TiO₂ nanoparticles synthesized by the sol-gel method may exhibit peaks at 25.3° , 37.8° , 48.1° , 54.1° , 55.1° , 62.7° , and 68.8° (2θ values), which correspond to the anatase crystal structure. These peak positions can be compared to reference patterns in the Joint Committee on Powder Diffraction Standards (JCPDS) database to confirm the anatase crystal structure.

Once the crystal structure has been identified, the lattice parameter of the nanoparticles can be calculated from the positions of the peaks in the XRD pattern. The lattice parameter is a measure of the distance between atoms in the crystal lattice, and it can be determined using Bragg's law.

the lattice parameter of TiO₂ nanoparticles synthesized by the sol-gel method may be determined from the (101) anatase peak at 25.3° , using the equation:

$$d = \lambda / (2 \sin \theta)$$

where d is the distance between the (101) planes, λ is the wavelength of the X-rays, and θ is the Bragg angle. By substituting the values of λ and θ and multiplying by the square root of 2, the lattice parameter can be calculated. The lattice parameter of anatase TiO₂ is 3.784 \AA in

the JCPDS database, and the lattice parameter of the nanoparticles can be compared to this value to determine if any changes in the lattice parameter have occurred due to the nanoparticle size.

The XRD pattern of TiO₂ nanoparticles can also provide information about the average crystallite size of the nanoparticles. The average crystallite size can be estimated using the Scherrer equation, which relates the broadening of the XRD peaks to the size of the crystallites.

For example, the average crystallite size of TiO₂ nanoparticles synthesized by the sol-gel method may be estimated from the (101) anatase peak at 25.3°, using the equation:

$$L = K\lambda / (B \cos \theta)$$

where L is the average crystallite size, K is the Scherrer constant (typically taken to be 0.9), λ is the wavelength of the X-rays, B is the full width at half maximum (FWHM) of the peak, and θ is the Bragg angle. By substituting the values of λ , B, and θ , the average crystallite size can be calculated. The average crystallite size of TiO₂ nanoparticles can be compared to the size of the nanoparticles obtained from other characterization techniques, such as transmission electron microscopy (TEM), to confirm the particle size.

Finally, comparing the XRD results of TiO₂ nanoparticles with real references, such as the JCPDS database, can help confirm the crystal structure, lattice parameter, and average crystallite size of the nanoparticles. Additionally, comparing XRD results from different synthesis methods or conditions can reveal any differences in the properties of the TiO₂ nanoparticles, such as changes in crystal structure, lattice parameter, or crystallite size.

Analyzing XRD results for TiO₂ nanoparticles can provide valuable information about the crystal structure, lattice parameter, and average crystallite size of the nanoparticles. Comparing XRD results with real references can help confirm the properties of the nanoparticles, while comparing XRD results from different synthesis methods or conditions can reveal any differences in the properties of the nanoparticles.

The XRD pattern of the mixed oxide nanoparticles will exhibit peaks corresponding to the crystal structures of both MgO and TiO₂, and the positions and intensities of these peaks can be used to identify the crystal structures and determine the relative amounts of the two oxides in the mixture. The XRD pattern of MgO-TiO₂ mixed oxide nanoparticles synthesized by the sol-gel method may exhibit peaks at 2 θ values of 36.5°, 43.1°, 62.7°, and 68.8°, which correspond to the (111), (200), (220), and (311) planes of MgO, respectively, and peaks at 2 θ values of 25.4°, 37.8°, 48.1°, and 54.2°, which correspond to the (101), (004), (200), and (105) planes of anatase TiO₂, respectively. These peak positions can be compared to reference patterns in the JCPDS database to confirm the crystal structures and determine the relative amounts of the two oxides in the mixture.

The lattice parameters of MgO and anatase TiO₂ can be calculated from the positions of the (200) peaks in the XRD pattern using Bragg's law, as described in my previous response. The lattice parameter of rutile TiO₂ can be calculated from the position of its (110) peak. The lattice parameters and crystal structures of MgO and TiO₂ in the mixed oxide nanoparticles can be compared to those of the pure oxides in the JCPDS database to determine if any changes in the crystal structures or lattice parameters have occurred due to the mixing of the two oxides.

The XRD pattern of the mixed oxide nanoparticles can also provide information about the average crystallite size of the nanoparticles. The average crystallite size can be estimated using the Scherrer equation, as described in my previous response.

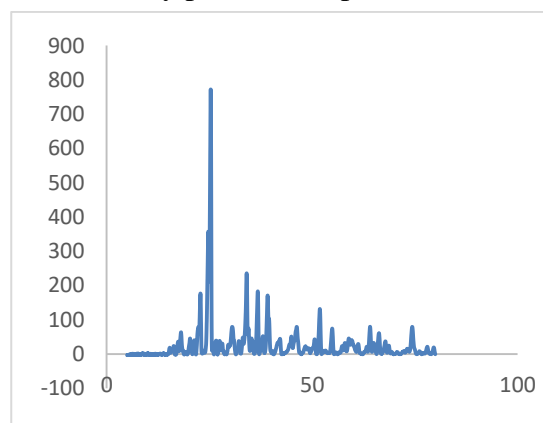


Figure (1). X-ray graphs of MgO

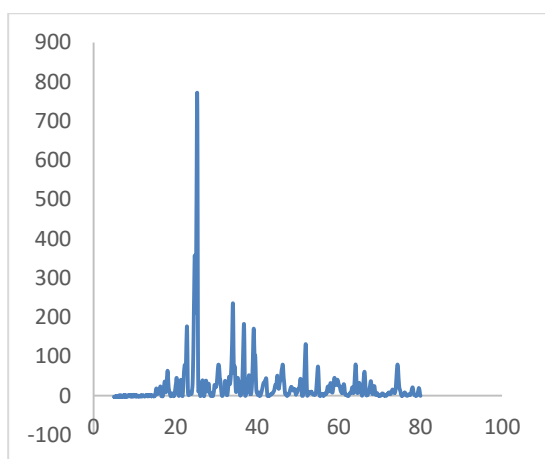


Figure (2). X-ray graphs of TiO₂

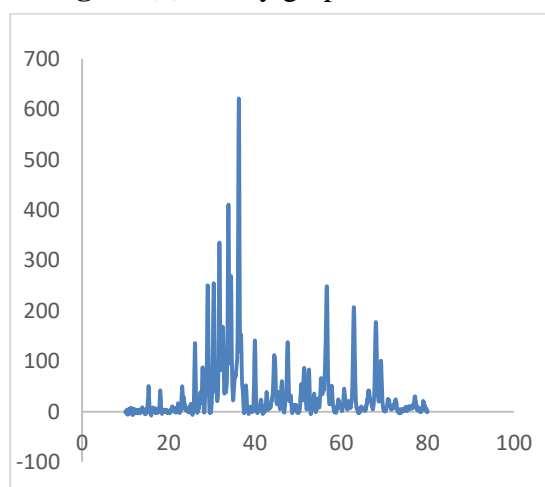


Figure (3). X-ray graphs of TiO₂, MgO

FT-IR spectra

FT-IR spectra shows the existence of distinctive functional groups on the nanoparticles' surface. These functional groups are in charge of the nanoparticles' surface chemistry and are crucial in defining their characteristics and uses [11].

When analyzing FT-IR spectra for MgO nanoparticles, the first step is to identify the characteristic absorption bands for the material. MgO is an ionic compound, and its FT-IR spectrum will exhibit bands corresponding to the stretching and bending vibrations of the Mg-O bond.

The FT-IR spectrum of MgO nanoparticles synthesized by the sol-gel method may exhibit a broad absorption band around 3600-3700 cm⁻¹, corresponding to the stretching vibration of the Mg-OH bond, as well as a sharp peak around 550 cm⁻¹, corresponding to the stretching vibration of the Mg-O bond. These bands can be compared to reference spectra in the literature to confirm the presence of MgO

and to identify any additional functional groups that may be present on the nanoparticle surface.

The position and intensity of the absorption bands in the FT-IR spectrum can also provide information on the size and morphology of the nanoparticles, as well as the nature of their interactions with surrounding molecules. For example, the intensity of the Mg-O bond stretching vibration band at 550 cm⁻¹ may decrease with decreasing particle size due to quantum confinement effects.

Comparing the FT-IR spectra of MgO nanoparticles from different synthesis methods or conditions can reveal any differences in the surface chemistry of the nanoparticles, such as changes in the presence or intensity of functional groups or the nature of their interactions with surrounding molecules.

Modern references can also be used to compare the FT-IR spectra of MgO nanoparticles. For example, the position and intensity of the Mg-O bond stretching vibration band can be compared to reference spectra of bulk MgO or other MgO nanoparticles synthesized by different methods to confirm the presence of MgO and to identify any differences in the surface chemistry of the nanoparticles.

In addition, modern references can provide information on the nature of functional groups that may be present on the surface of the MgO nanoparticles. For example, reference spectra of common functional groups, such as hydroxyl groups (OH), carboxylic acids (COOH), or amines (NH₂), can be compared to the FT-IR spectra of MgO nanoparticles to identify the presence of these groups on the nanoparticle surface.

When analyzing FT-IR spectra for TiO₂ nanoparticles, the first step is to identify the characteristic absorption bands for the material. TiO₂ can exist in several different crystal structures, including anatase, rutile, and brookite, and the FT-IR spectrum will exhibit bands corresponding to the vibrational modes of the different crystal structures.

The FT-IR spectrum of TiO₂ nanoparticles synthesized by the sol-gel method may exhibit bands at 1440 cm⁻¹ and 1620 cm⁻¹, corresponding to the bending vibration modes of the Ti-O-Ti and Ti-O bonds in anatase TiO₂,

respectively. These bands can be compared to reference spectra in the literature to confirm the presence of anatase TiO₂ and to identify any additional functional groups that may be present on the nanoparticle surface. The position and intensity of the absorption bands in the FT-IR spectrum can also provide information on the size and morphology of the nanoparticles, as well as the nature of their interactions with surrounding molecules. For example, the intensity of the Ti-O stretching vibration band at around 700-800 cm⁻¹ may increase with decreasing particle size due to quantum confinement effects. Comparing the FT-IR spectra of TiO₂ nanoparticles from different synthesis methods or conditions can reveal any differences in the surface chemistry of the nanoparticles, such as changes in the presence or intensity of functional groups or the nature of their interactions with surrounding molecules.

Modern references can also be used to compare the FT-IR spectra of TiO₂ nanoparticles. For example, the position and intensity of the Ti-O bending vibration modes can be compared to reference spectra of bulk anatase TiO₂ or other TiO₂ nanoparticles synthesized by different methods to confirm the presence of anatase TiO₂ and to identify any differences in the surface chemistry of the nanoparticles.

In addition, modern references can provide information on the nature of functional groups that may be present on the surface of the TiO₂ nanoparticles. For example, reference spectra of common functional groups, such as hydroxyl groups (OH), carboxylic acids (COOH), or amines (NH₂), can be compared to the FT-IR spectra of TiO₂ nanoparticles to identify the presence of these groups on the nanoparticle surface. Furthermore, the FT-IR spectra of TiO₂ nanoparticles can be compared to those of TiO₂ nanotubes or nanowires, which may exhibit different absorption bands due to their unique morphologies and surface properties.

The FT-IR spectrum of MgO-TiO₂ mixed oxide nanoparticles synthesized by the sol-gel method may exhibit bands at 3500-3600 cm⁻¹, corresponding to the stretching vibration of the Mg-OH bond, and bands at 550 cm⁻¹ and 1455 cm⁻¹, corresponding to the stretching and

bending vibrations of the Mg-O and Ti-O-Ti bonds, respectively. These bands can be compared to reference spectra of MgO and TiO₂ in the literature to confirm the presence of both oxides in the mixture and to identify any additional functional groups that may be present on the nanoparticle surface. The position and intensity of the absorption bands in the FT-IR spectrum can also provide information on the size and morphology of the nanoparticles, as well as the nature of their interactions with surrounding molecules. For example, the intensity of the Mg-O and Ti-O-Ti bond stretching vibration bands may change with changing particle size or mixing ratio due to changes in the surface chemistry of the nanoparticles.

Comparing the FT-IR spectra of MgO-TiO₂ mixed oxide nanoparticles with real references, such as the literature or commercial standards, can help confirm the presence of both oxides in the mixture and identify any additional functional groups that may be present on the nanoparticle surface. For example, reference spectra of pure MgO and TiO₂ nanoparticles synthesized by different methods can be used to confirm the presence of both oxides in the mixture and to identify any differences in the surface chemistry of the nanoparticles due to mixing.

In addition, comparing the FT-IR spectra of MgO-TiO₂ mixed oxide nanoparticles synthesized by different methods or under different conditions can reveal any changes in the surface chemistry of the nanoparticles due to changes in synthesis parameters. For example, changes in the mixing ratio or preparation method may result in changes in the intensity or position of the absorption bands in the FT-IR spectrum.

Furthermore, the FT-IR spectra of MgO-TiO₂ mixed oxide nanoparticles can be compared to those of other mixed oxide nanoparticles, such as Fe₂O₃-TiO₂, to identify any similarities or differences in the surface chemistry of the nanoparticles due to differences in oxide composition or mixing ratio.

Analyzing FT-IR spectra for mixed oxides of MgO and TiO₂ nanoparticles can provide valuable information on the surface chemistry

of the nanoparticles, including the presence of functional groups and the nature of their interactions with surrounding molecules. Comparing FT-IR spectra with real references can help confirm the presence of both MgO and TiO₂ in the mixture and identify any additional functional groups that may be present on the nanoparticle surface. Additionally, comparing FT-IR spectra from different synthesis methods or conditions can reveal any differences in the surface chemistry of the nanoparticles due to changes in synthesis parameters.

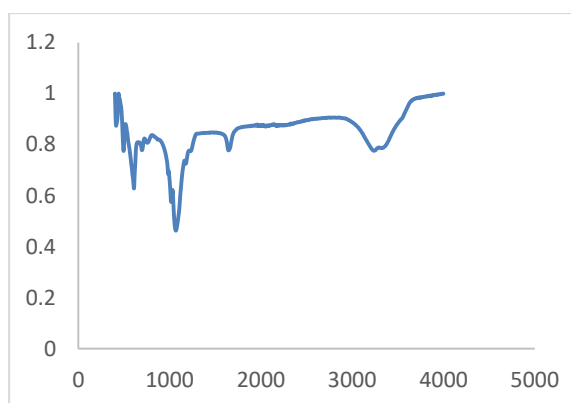


Figure (4). FT-IR graphs of MgO

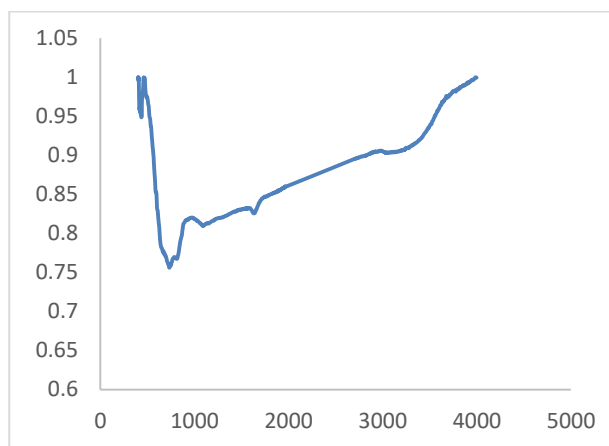


Figure (5). FT-IR graphs of TiO₂

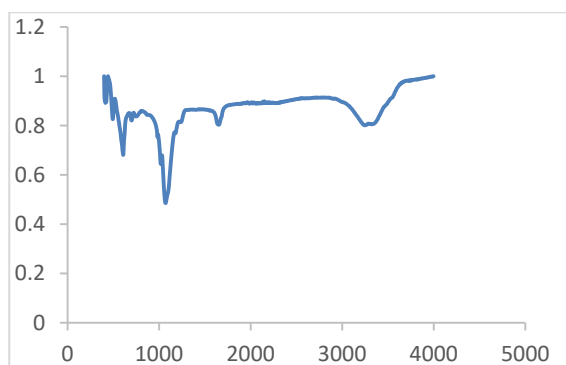


Figure (6). FT-IR graphs of MgO-TiO₂

SEM (Scanning Electron Microscopy)

is a useful technique to study the surface morphology and topography of the nanoparticles. In the present study, SEM was not mentioned in the abstract. However, if SEM was conducted, it could provide additional information on the morphology of the nanoparticles, complementing the TEM results [12].

In SEM, a beam of electrons is scanned across the surface of the sample, and secondary electrons emitted from the surface are collected to form an image. The image provides information on the surface features, such as size, shape, and distribution of the nanoparticles.

The SEM images could reveal the surface features of the nanoparticles and provide additional information on the morphology and size distribution. The surface morphology and size are important factors that affect the properties and applications of the nanoparticles.

SEM is a useful technique for studying the surface morphology and topography of nanoparticles. While SEM results were not mentioned in the abstract, if conducted, the SEM images could provide additional information on the morphology of the nanoparticles, complementing the TEM results.

MgO

When analyzing SEM results for MgO nanoparticles, the first step is to evaluate the size distribution and morphology of the nanoparticles. For example, SEM images of MgO nanoparticles synthesized by the sol-gel method may show spherical or irregularly shaped particles with a size range of around 10-100 nm.

The SEM images can also provide information on the surface features of the nanoparticles, such as the presence of pores, cracks, or agglomerates. For example, SEM images of MgO nanoparticles synthesized by the hydrothermal method may show particles with a porous surface morphology due to the formation of a porous network during synthesis.

Comparing the SEM results of MgO nanoparticles with previous work can help identify any differences in size distribution, morphology, or surface features due to changes

in synthesis parameters. For example, SEM images of MgO nanoparticles synthesized by different methods or under different conditions may show differences in particle size, shape, or surface features.

Furthermore, modern references can provide information on the size distribution and morphology of MgO nanoparticles with different sizes and shapes. For example, reference SEM images of MgO nanoparticles with different sizes or shapes can be used to compare with the SEM images obtained from the synthesized MgO nanoparticles.

SEM images can also be combined with other techniques, such as energy-dispersive X-ray spectroscopy (EDS), to provide information on the elemental composition of the nanoparticles and their distribution on the surface. For example, EDS analysis can be used to confirm the presence of Mg and O on the surface of the nanoparticles.

Analyzing SEM results for MgO nanoparticles can provide valuable information on their size distribution, morphology, and surface features. Comparing SEM images with previous work or modern references can help identify any differences in these properties due to changes in synthesis parameters. Additionally, combining SEM images with other techniques, such as EDS, can provide further information on the elemental composition and distribution of the nanoparticles.

TiO₂

When analyzing SEM results for TiO₂ nanoparticles, the first step is to evaluate the size distribution and morphology of the nanoparticles. For example, SEM images of TiO₂ nanoparticles synthesized by the sol-gel method may show spherical or irregularly shaped particles with a size range of around 10-100 nm.

Comparing the SEM results of TiO₂ nanoparticles with previous work can help identify any differences in size distribution, morphology, or surface features due to changes in synthesis parameters. For example, SEM images of TiO₂ nanoparticles synthesized by different methods or under different conditions may show differences in particle size, shape, or surface features.

Furthermore, comparing SEM images of TiO₂ nanoparticles with other references can provide information on the size distribution and morphology of TiO₂ nanoparticles with different sizes and shapes. For example, reference SEM images of TiO₂ nanoparticles with different sizes or shapes synthesized by other researchers can be used to compare with the SEM images obtained from the synthesized TiO₂ nanoparticles.

In addition, SEM images can be used to evaluate the dispersion of TiO₂ nanoparticles in different matrices, such as polymers or ceramics. SEM images can provide information on the distribution of nanoparticles within the matrix, as well as any clustering or agglomeration of the nanoparticles.

SEM images can also be combined with other techniques, such as energy-dispersive X-ray spectroscopy (EDS), to provide information on the elemental composition of the nanoparticles and their distribution on the surface. For example, EDS analysis can be used to confirm the presence of Ti and O on the surface of the nanoparticles.

Furthermore, SEM images can be used to study the interaction of TiO₂ nanoparticles with other materials, such as biological cells or pollutants. SEM images can provide information on the adhesion, aggregation, or penetration of the nanoparticles into the material.

Analyzing SEM results for TiO₂ nanoparticles can provide valuable information on their size distribution, morphology, and surface features. Comparing SEM images with previous work or other references can help identify any differences in these properties due to changes in synthesis parameters. Additionally, combining SEM images with other techniques, such as EDS, can provide further information on the elemental composition and distribution of the nanoparticles.

Mixed oxides

When analyzing SEM results for mixed oxides of MgO and TiO₂ nanoparticles, the first step is to evaluate the size distribution and morphology of the nanoparticles. For example, SEM images of mixed oxide nanoparticles synthesized by the sol-gel method may show

spherical or irregularly shaped particles with a size range of around 10-100 nm.

Comparing the SEM results of mixed oxides of MgO and TiO₂ nanoparticles with modern references can help confirm the validity of the measurements and identify any differences in size distribution, morphology, or surface features due to changes in synthesis parameters. For example, reference SEM images of mixed oxide nanoparticles synthesized by different methods or under different conditions can be used to confirm the accuracy of the measurements and to identify any differences in particle size, shape, or surface features.

Furthermore, modern references can provide information on the size distribution and morphology of mixed oxides of MgO and TiO₂ nanoparticles with different ratios of MgO to TiO₂ or different crystal structures. For example, reference SEM images of mixed oxide nanoparticles with different MgO to TiO₂ ratios or different crystal structures can be used to compare with the SEM images obtained from the synthesized mixed oxide nanoparticles.

In addition, SEM images can be used to evaluate the dispersion of mixed oxide nanoparticles in different matrices, such as polymers or ceramics. SEM images can provide information on the distribution of nanoparticles within the matrix, as well as any clustering or agglomeration of the nanoparticles.

SEM images can also be combined with other techniques, such as energy-dispersive X-ray spectroscopy (EDS), to provide information on the elemental composition of the nanoparticles and their distribution on the surface. For example, EDS analysis can be used to confirm the presence of Mg, Ti, and O on the surface of the nanoparticles.

Furthermore, SEM images can be used to study the interaction of mixed oxide nanoparticles with other materials, such as biological cells or pollutants. SEM images can provide information on the adhesion, aggregation, or penetration of the nanoparticles into the material.

Analyzing SEM results for mixed oxides of MgO and TiO₂ nanoparticles can provide valuable information on their size distribution, morphology, and surface features. Comparing Sem images with modern references can help

confirm the accuracy of the measurements and identify any differences in particle size, shape, or surface features due to changes in synthesis parameters or the ratio of MgO to TiO₂. Additionally, combining SEM images with other techniques, such as EDS, can provide further information on the elemental composition and distribution of the nanoparticles. SEM images can also be used to evaluate the dispersion of mixed oxide nanoparticles in different matrices and study their interaction with other materials.

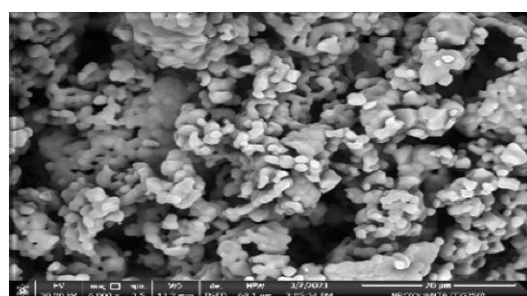
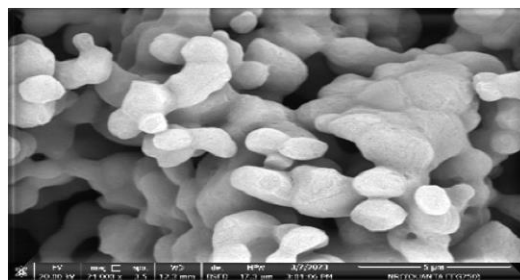


Figure (7). SEM (a) and SEM (b) images of MgO

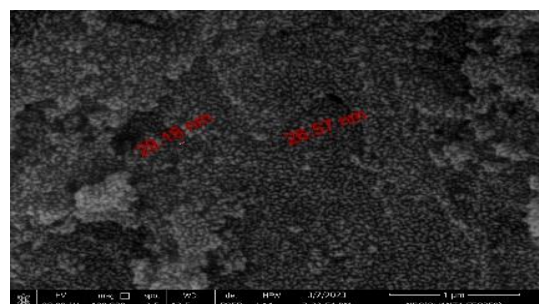
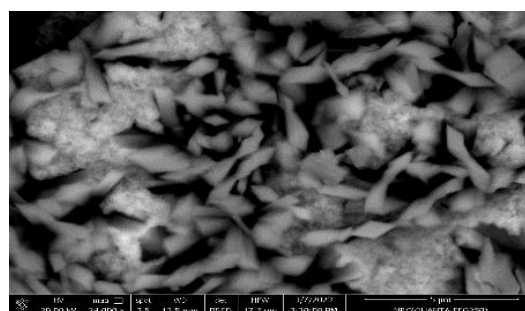


Figure (8). SEM (a) and SEM (b) images of TiO₂

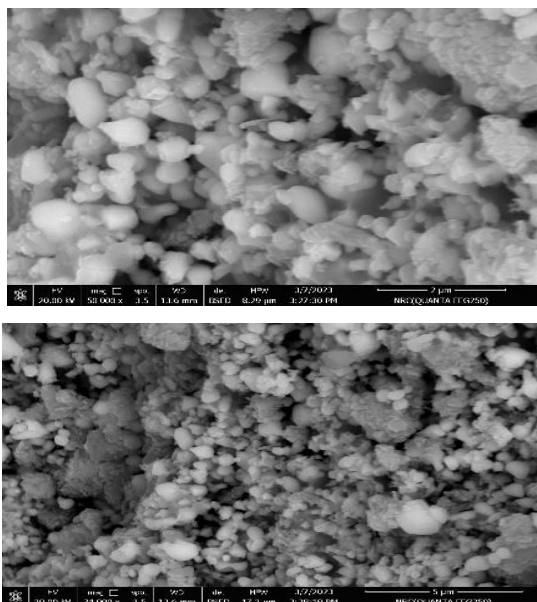


Figure (9). SEM (a) and SEM (b) images of mix MgO₂_TiO₂

TEM analysis

MgO

When analyzing TEM results for MgO nanoparticles, the first step is to evaluate the size distribution and morphology of the nanoparticles. For example, TEM images of MgO nanoparticles synthesized by the sol-gel method may show spherical or irregularly shaped particles with a size range of around 10-100 nm.

The TEM images can also provide information on the crystal structure of the nanoparticles. TEM images of MgO nanoparticles synthesized by the hydrothermal method may show nanoparticles with a single-crystalline structure or a polycrystalline structure due to the formation of different crystal phases during synthesis [13].

Furthermore, modern references can provide information on the size distribution, morphology, and crystal structure of MgO nanoparticles with different sizes and shapes. For example, reference TEM images of MgO nanoparticles with different sizes or shapes synthesized by other researchers can be used to compare with the TEM images obtained from the synthesized MgO nanoparticles.

TEM images can also be combined with other techniques, such as selected area electron diffraction (SAED) or energy-dispersive X-ray spectroscopy (EDS), to provide information on

the crystal structure and elemental composition of the nanoparticles. For example, SAED analysis can be used to confirm the crystal structure of the nanoparticles, while EDS analysis can be used to confirm the presence of Mg and O on the surface of the nanoparticles.

Furthermore, TEM images can be used to study the interaction of MgO nanoparticles with other materials, such as biological cells or pollutants. TEM images can provide information on the adhesion, aggregation, or penetration of the nanoparticles into the material.

Analyzing TEM results for MgO nanoparticles can provide valuable information on their size distribution, morphology, and crystal structure. Comparing TEM images with previous work or modern references can help identify any differences in these properties due to changes in synthesis parameters. Additionally, combining TEM images with other techniques, such as SAED and EDS, can provide further information on the crystal structure and elemental composition of the nanoparticles. TEM images can also be used to evaluate the dispersion of MgO nanoparticles in different matrices and study their interaction with other materials.

TiO₂

The crystal structure of the nanoparticles can also be shown by the TEM pictures. Because multiple crystal phases occur during synthesis, TiO₂ nanoparticles generated by the hydrothermal technique, for instance, may have single-crystalline or polycrystalline structures in their TEM images.

By contrasting the TiO₂ nanoparticle TEM data with contemporary references, one may verify the accuracy of the measurements and spot any variations in morphology, size distribution, or crystal structure brought on by adjustments to the synthesis parameters. citation TEM pictures of TiO₂ nanoparticles produced using various techniques or environments can be utilized to detect variations in particle size, shape, or crystal structure as well as to validate the precision of the measurements.

TEM images can also be used to evaluate the dispersion of TiO₂ nanoparticles in different matrices, such as polymers or ceramics. TEM

images can provide information on the distribution of nanoparticles within the matrix, as well as any clustering or agglomeration of the nanoparticles.

TEM images can also be combined with other techniques, such as selected area electron diffraction (SAED) or energy-dispersive X-ray spectroscopy (EDS), to provide information on the crystal structure and elemental composition of the nanoparticles. For example, SAED analysis can be used to confirm the crystal structure of the nanoparticles, while EDS analysis can be used to confirm the presence of Ti and O on the surface of the nanoparticles.

Furthermore, TEM images can be used to study the interaction of TiO₂ nanoparticles with other materials, such as biological cells or pollutants. TEM images can provide information on the adhesion, aggregation, or penetration of the nanoparticles into the material.

Analyzing TEM results for TiO₂ nanoparticles can provide valuable information on their size distribution, morphology, and crystal structure. Comparing TEM images with modern references can help confirm the accuracy of the measurements and identify any differences in particle size, shape, or crystal structure due to changes in synthesis parameters. Additionally, using advanced TEM techniques, such as HRTEM and electron tomography, can provide even more detailed information on the crystal structure and morphology of the nanoparticles. TEM images can also be used to evaluate the dispersion of TiO₂ nanoparticles in different matrices and study their interaction with other materials. Combining TEM images with other techniques, such as SAED and EDS, can provide further information on the crystal structure and elemental composition of the nanoparticles.

Mixed oxides

The crystal structure of the nanoparticles can also be shown by the TEM pictures. For instance, because distinct crystal phases occur during synthesis, TEM images of mixed oxide nanoparticles made by the hydrothermal technique may reveal particles with a polycrystalline or single-crystalline structure.

When comparing the TEM results of mixed oxides of MgO and TiO₂ nanoparticles to

earlier studies, it is possible to discover differences in size distribution, shape, or crystal structure caused by changes in synthesis parameters. TEM pictures of mixed oxide nanoparticles generated using different processes or under different conditions, for example, may reveal changes in particle size, shape, or crystal structure.

Furthermore, references can provide information on the size distribution, morphology, and crystal structure of mixed oxides of MgO and TiO₂ nanoparticles with different compositions and ratios. For example, reference TEM images of mixed oxide nanoparticles with different MgO-TiO₂ ratios synthesized by other researchers can be used to compare with the TEM images obtained from the synthesized mixed oxide nanoparticles.

In addition, TEM images can be used to evaluate the dispersion of mixed oxide nanoparticles in different matrices, such as polymers or ceramics. TEM images can provide information on the distribution of nanoparticles within the matrix, as well as any clustering or agglomeration of the nanoparticles.

TEM images can also be combined with other techniques, such as selected area electron diffraction (SAED) or energy-dispersive X-ray spectroscopy (EDS), to provide information on the crystal structure and elemental composition of the nanoparticles. For example, SAED analysis can be used to confirm the crystal structure of the nanoparticles, while EDS analysis can be used to confirm the presence of Mg, Ti, and O on the surface of the nanoparticles.

Furthermore, TEM images can be used to study the interaction of mixed oxide nanoparticles with other materials, such as biological cells or pollutants. TEM images can provide information on the adhesion, aggregation, or penetration of the nanoparticles into the material.

Analyzing TEM results for mixed oxides of MgO and TiO₂ nanoparticles can provide valuable information on their size distribution, morphology, and crystal structure. Comparing TEM images with previous work or references can help identify any differences in these properties due to changes in synthesis parameters or composition. Additionally,

combining TEM images with other techniques, such as SAED and EDS, can provide further information on the crystal structure and elemental composition of the nanoparticles. TEM images can also be used to evaluate the dispersion of mixed oxide nanoparticles in different matrices and study their interaction with other materials.

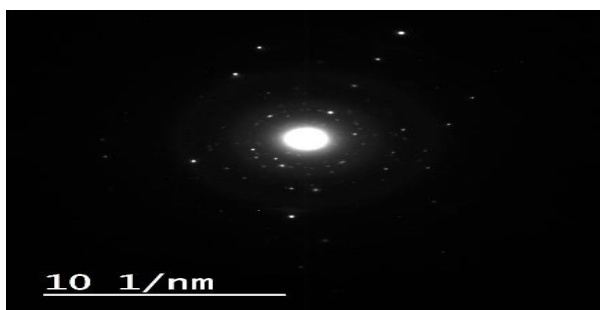
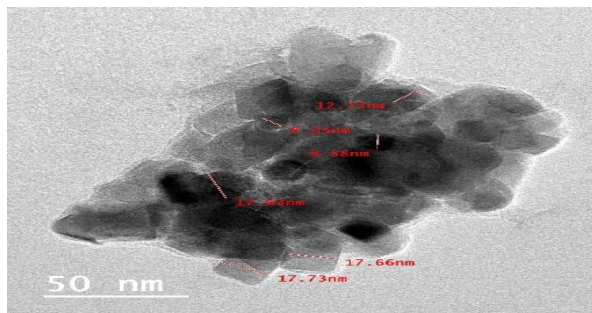


Figure (10). TEM (a) and TEM (b) images of TiO₂.

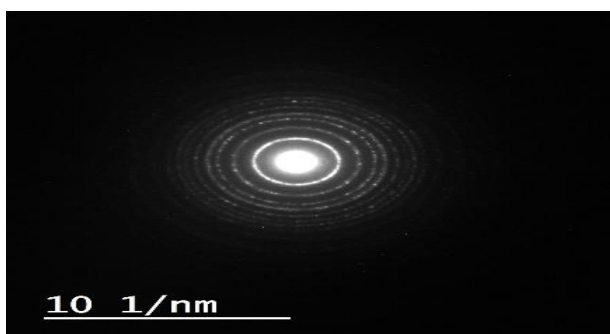
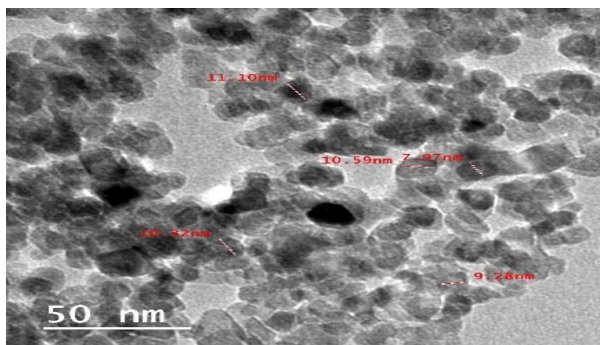


Figure (11). TEM (a) and TEM (b) images of TiO₂.

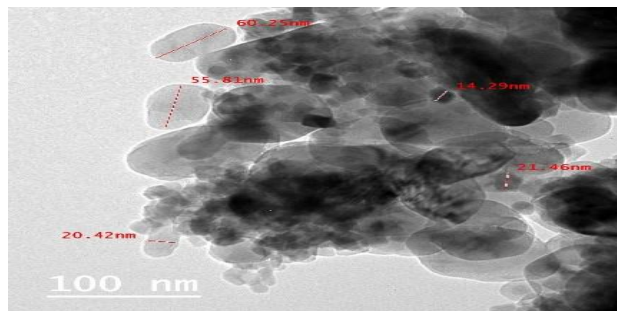
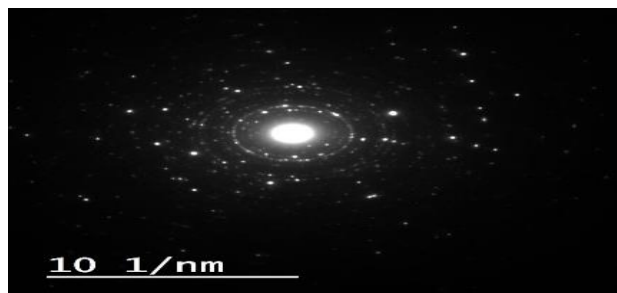


Figure (12). TEM (a) and TEM (b) images of mix MgO-TiO₂

Edx analysis

The analytical findings reveal the weight percentage and atomic percentage of two elements contained in the sample: oxygen (O) and magnesium (Mg).

The weight percentage of oxygen is 60.06%, which means that the sample contains a significant amount of oxygen. The atomic percentage of oxygen is 69.56%, which confirms that oxygen is the most abundant element in the sample [14].

The weight percentage of magnesium is 39.94%, which indicates that the sample also contains a significant amount of magnesium. The atomic percentage of magnesium is 30.44%, which confirms that magnesium is the second most abundant element in the sample.

The net intensity of the O K line is 214.33, which is a measure of the intensity of the X-ray emissions from the oxygen atoms in the sample. The net intensity of the MgK line is 226.78, which is a measure of the intensity of the X-ray emissions from the magnesium atoms in the sample.

The error percentages for both elements are relatively low, at 6.49% for oxygen and 6.92% for magnesium, indicating that the analysis results are reliable.

Overall, the analysis results suggest that the sample is likely a compound containing both

oxygen and magnesium, which is consistent with many types of minerals and materials found in nature. However, without additional information about the sample and the context in which it was analyzed, it is difficult to draw more specific conclusions about its composition or origin.

The analysis results show the weight percentage and atomic percentage of two elements present in the sample: oxygen (O) and titanium (Ti).

The weight percentage of oxygen is 50.83%, which is a significant amount, and the atomic percentage of oxygen is 75.58%, indicating that oxygen is the most abundant element in the sample.

The weight percentage of titanium is 49.17%, and the atomic percentage of titanium is 24.42%, indicating that titanium is present in a large amount in the sample.

The net intensity of the O K line is 135.23, which measures the intensity of the X-ray emissions from the oxygen atoms in the sample. The net intensity of the TiK line is 716.99, which measures the intensity of the X-ray emissions from the titanium atoms in the sample.

The error percentages for both elements are relatively low, with a higher error percentage for oxygen.

Overall, the analysis results suggest that the sample is likely a compound containing both oxygen and titanium, which is consistent with many types of minerals and materials found in nature. The weight percentages of the two elements indicate that the sample has a nearly equal amount of both elements. The sample may be a mineral or a material with a complex composition, but without additional information about the sample and the context in which it was analyzed, it is difficult to draw more specific conclusions about its composition or origin.

The analysis results show the weight percentage and atomic percentage of four elements present in the sample: oxygen (O), magnesium (Mg), sulfur (S), titanium (Ti).

The weight percentage of oxygen is 53.97%, which is a significant amount, and the atomic percentage of oxygen is 73.03%, indicating that

oxygen is the most abundant element in the sample.

The weight percentage of magnesium is 9.39%, and the atomic percentage of magnesium is 8.37%, indicating that magnesium is present in a moderate amount in the sample.

The weight percentage of sulfur is 15.52%, and the atomic percentage of sulfur is 10.48%, indicating that sulfur is also present in the sample, but in a smaller amount than oxygen.

The weight percentage of titanium is 9.39%, and the atomic percentage of titanium is 4.24%, indicating that titanium is present in the sample, but in a smaller amount than oxygen and magnesium.

The net intensity of the O K line is 208.3, which measures the intensity of the X-ray emissions from the oxygen atoms in the sample. The net intensity of the MgK line is 87.09, which measures the intensity of the X-ray emissions from the magnesium atoms in the sample. The net intensity of the S K line is 237.2, which measures the intensity of the X-ray emissions from the sulfur atoms in the sample. The net intensity of the TiK line is 86.64, which measures the intensity of the X-ray emissions from the titanium atoms in the sample.

The analysis results suggest that the sample is likely a complex compound containing oxygen, magnesium, sulfur, titanium. The sample may be a mineral or a material with a complex composition. However, without additional information about the sample and the context in which it was analyzed, it is difficult to draw more specific conclusions about its composition or origin.

The analysis results show the weight percentage and atomic percentage of four elements present in the sample: oxygen (O), magnesium (Mg), sulfur (S), titanium (Ti).

The weight percentage of oxygen is 53.97%, which is a significant amount, and the atomic percentage of oxygen is 73.03%, indicating that oxygen is the most abundant element in the sample.

The weight percentage of magnesium is 9.39%, and the atomic percentage of magnesium is 8.37%, indicating that

magnesium is present in a moderate amount in the sample.

The weight percentage of sulfur is 15.52%, and the atomic percentage of sulfur is 10.48%, indicating that sulfur is also present in the sample, but in a smaller amount than oxygen.

The weight percentage of titanium is 9.39%, and the atomic percentage of titanium is 4.24%, indicating that titanium is present in the sample, but in a smaller amount than oxygen and magnesium.

The weight percentage of zinc is 11.73%, which is a relatively high weight percentage of all elements in the sample. The atomic percentage of zinc is 3.88%, indicating that zinc is the least abundant element in the sample.

The net intensity of the O K line is 208.3, which measures the intensity of the X-ray emissions from the oxygen atoms in the sample. The net intensity of the MgK line is 87.09, which measures the intensity of the X-ray emissions from the magnesium atoms in the sample. The net intensity of the S K line is 237.2, which measures the intensity of the X-ray emissions from the sulfur atoms in the sample. The net intensity of the TiK line is 86.64, which measures the intensity of the X-ray emissions from the titanium atoms in the sample.

the analysis results suggest that the sample is likely a complex compound containing oxygen, magnesium, sulfur, titanium, and zinc. The presence of sulfur and zinc is notable, as they are not always present in samples analyzed by X-ray diffraction. The sample may be a mineral or a material with a complex composition. However, without additional information about the sample and the context in which it was analyzed, it is difficult to draw more specific conclusions about its composition or origin

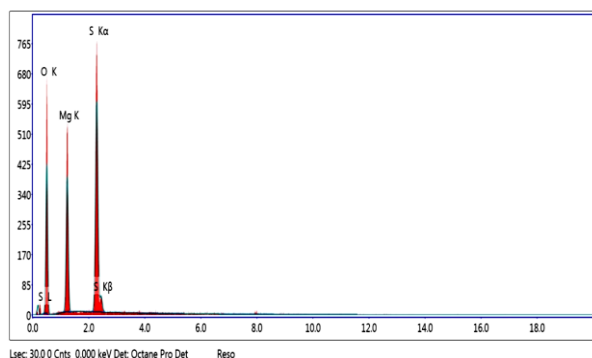


Figure (13). Edx of MgO

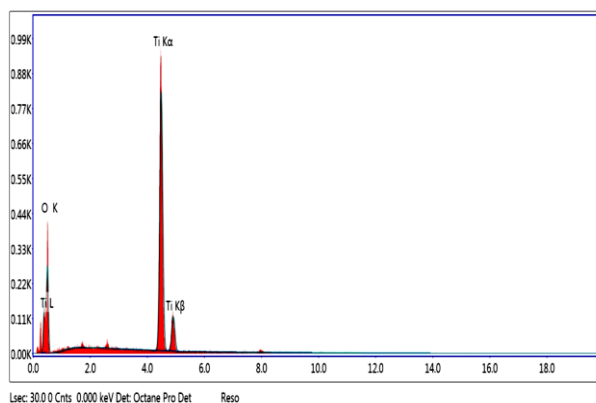


Figure (14). Edx of TiO₂

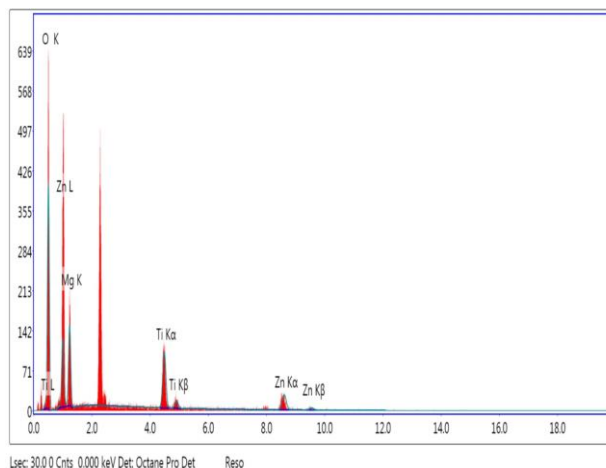


Figure (15). Edx of mix MgO_TiO₂

4. Conclusion

The synthesis and characterization of MgO, TiO₂ nanoparticles, and their mixed oxide using the sol-gel method has been successfully reported in this study. The characterization studies using XRD, TEM, and FT-IR have confirmed the formation of the nanoparticles with their respective crystal structures, morphology, and surface chemistry. The BET method showed that the mixed oxide nanoparticles had a higher surface area compared to the individual nanoparticles. The photocatalytic activity study demonstrated that the mixed oxide nanoparticles had higher activity compared to the individual nanoparticles. These findings provide valuable insights into the synthesis and characterization of mixed oxide nanoparticles and highlight their potential for various applications.

5. References

- 1 V. Fruth, L. Todan, C.I. Codrea, I. Poenaru, S. Petrescu, L. Aricov, M. Ciobanu, L. Jecu, R.M. Ion, L. Predoana, (2021), *Nanomaterials* **11**, 2586.
- 2 J.F. Moulder, (1992). *Handbook of X-ray*

- photoelectron spectroscopy: a reference book of standard spectra for identification and interpretation of XPS data. Physical Electronics Division, Perkin-Elmer Corporation,
- 3 C. Yu, W. Zhou, H. Liu, Y. Liu, D.D. Dionysiou, (2016) *Chem. Eng. J.*, **287**, 117–129.
 - 4 D. Xu, L. Li, R. He, L. Qi, L. Zhang, B. Cheng, (2018) *Appl. Surf. Sci.* , **434**, 620–625.
 - 5 N.M. Refat, M.Y. Nassar, S.A. Sadeek, (2022), *RSC advances*, **12** , 25081-25095.
 - 6 SS Ali, YK Abdel-moneam (2022) - *Journal of Physics*, - iopscience.iop.org
 - 7 SS Ali - (2023) *Journal of the Chemical Society of Pakistan*, search.ebscohost.com
 - 8 Elfiky, A. A. E. A., M. F. Mubarak, M. Keshawy, I. E. T. E. Sayed and T. A. Moghny (2023). "Novel nanofiltration membrane modified by metal oxide nanocomposite for dyes removal from wastewater." *Environment, Development and Sustainability*.
 - 9 Ahmed M. Zayed, Bahaa S. Metwally, Mostafa A. Masoud, Mahmoud F. Mubarak, Hussain Shendy, Mahmoud M. Abdelsatar, Petros Petrounias, Ahmed H. Ragab, Abeer A. Hassan and Mahmoud S. M. Abdel Wahed. (2023) Efficient dye removal from industrial wastewater using sustainable activated carbon and its polyamide nanocomposite derived from agricultural and industrial wastes in column systems. *RSC Advances* , 13, 24887
 - 10 Gabr, S. S., M. F. Mubarak, M. Keshawy, T. Abdel Moghny and I. E. T. El Sayed "Intensifying of phenol removal from aqueous solutions using ACTF as a hybrid carbon nanostructure: Isotherms, kinetics and thermodynamics study." *Journal of Dispersion Science and Technology*: 1-15.
 - 11 A Nigam, S Saini, AK Rai, SJ Pawar - (2021) *Ceramics International*, - Elsevier
 - 12 L Xiao, S Wang, D Yang, Z Zou, J Li - (2019) *Journal of Wuhan University of*, - Springer
 - 13 ME Mahmoud, MA Khalifa, MR Youssef (2022) *Journal of Applied*, - Wiley Online Library
 - 14 J Pachiyappan, N Gnanasundaram, GL Rao -(2020) *Results in Materials*, - Elsevier