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Research Article

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Synthesis and Characterization of MgO/TiO_2 Nanocomposites

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Abstract

The nanostructured material properties were showing surprisingly different from the bulk materials. Among these nanostructured materials nanocomposites are of great technological importance because of their small size and high surface to volume ratio. Due to these reason the nanocomposites were used in various potential applications such as optoelectronics, microelectronics, water purification, paints, biomedical field and different sensor fabrication. Present work concentrates on synthesis and characterization of MgO/TiO_2 nanocomposites by Microwave-assisted method using ionic liquid as the initial precursor and different temperature effects were investigated on it. The obtained MgO/TiO_2 nanocomposites have been characterized by X-Ray Diffraction (XRD), particle size analyser (PSA), transmission electron microscopy (TEM), Fourier transform infrared spectrometer (FTIR), thermo gravimetric/differential thermal analyzer (TG/DTA) and Keithley electric meter for average crystallite size, average particle size, morphological studies, bond analysis, thermal stability and resistance respectively.

Keywords: MgO/TiO_2 Nanocomposite; Microwave-assisted method; XRD; TEM; FTIR

Introduction

Nanotechnology research work is growing very fast in recent days, because of its extraordinary properties in all the fields. The nanostructured metal oxides play an important role in optoelectronics applications such as global positioning systems, satellites and cellular phones [1]. Generally nano scale materials were available in various dimensions, such as zero-dimension, one-dimension, two-dimension and three-dimension. The nanocomposite rods category is in the one-dimensional nanostructured materials only. These nanostructured composite rods were used in many potential applications [2]. MgO and TiO_2 metal oxide nanomaterials are having extensive properties, due to this reason these materials were used in various applications. MgO nanostructured materials were shown high melting point, chemical stability, electric resistivity and high thermal expansion coefficient. Whereas TiO_2 nanostructured materials having high optical, electrical, dielectric and catalytic properties. MgO and TiO_2 nanostructured materials were used as high temperature insulator and optoelectronic devices respectively [3-6]. The MgO/TiO_2 metal oxide nanocomposite materials were observed broad importance in the electronics field. The composition of above materials produced different structures like nano tubes, nano rods, nano belts and nano dots [7,8]. This present paper was focusing on MgO/TiO_2 metal oxide nanocomposite rods synthesis, characterization and sensing applications. For the synthesis of nanostructured metal oxide materials many methods are available, such as chemical co-precipitation [9], solution combustion [10], chemical reduction [11], micro wave assisted method [12] and sol-gel synthesis [13]. Microwave assisted method is very easy, simple and fast synthesis method to prepare nanostructured materials compared with all above mentioned methods.

Materials and Methods

Magnesium Acetate, Titanium isopropoxide, Sodium hydroxide and 1-butyl-3-methyl imidazolium tetra fluoroborate [bmim]BF_4 were taken as initial precursor materials. MgO/TiO_2 nanostructured metal oxide composite rods were prepared by using microwave assisted synthesis method. The MgO and TiO_2 nanostructured metal oxides were prepared separately and add these two materials with the help of mechanical milling. For MgO preparation, 0.1M of magnesium acetate dissolved in 100 ml of distilled water, added 0.1M of NaOH standard solution and 0.8 ml of [bmim]BF_4 solution to the above solution under vigorous stirring. The light black colour solution was formed. This solution was kept in microwave oven. After 5 minutes the black colour precipitate was formed, this precipitated solution was filtered, washed with water and ethanol and dried at 60°C for 1 hour. Finally MgO nanostructured materials were obtained. Whereas in the preparation of nanostructured TiO_2 materials, 5 ml of titanium isopropoxide dissolved in 100 ml of distilled water, added 0.1M of NaOH standard solution and 0.8 ml of [bmim]BF_4 solution to the above solution. A white colour solution was formed. After 5 minutes of microwave irradiating, a milky white solution was formed. This solution was filtered; washed with water and ethanol and dried it mentioned conditions. The TiO_2 nanostructured materials were obtained. These obtained MgO and TiO_2 nanostructured materials were mixed by mechanical milling using mortar and pestle. The MgO/TiO_2 nanocomposite metal oxides were heat treated at 500°C and 600°C.

Characterization Techniques

The structural properties and average crystallite sizes of the materials were studied by Bruker D8 X-ray diffractometer. From the HORIBA SZ-100 Particle Size Analyser the average particle size and zeta potential were measured. The shape, morphology and d-spacing values were observed by JEM-100 CXII Transmission Electron Microscope. Broker FTIR used for bond analysis. From S-II EXSTAR-6000, TG/DTA-6300 thermal analyser used to measure the thermal properties

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of the materials. Keithley Electric meter is used for measuring of resistance [14].

Results and Discussions

X-ray diffractometer

XRD pattern of MgO/TiO_2 nanocomposite rods were prepared by microwave assisted method using room temperature ionic liquids. The XRD pattern of is shown MgO/TiO_2 nanocomposite rods heat treated at two different temperatures 500°C and 600°C is shown in Figure 1. The peaks were representing MgO, TiO_2, $MgTiO_3$ and Mg_2TiO_4 nanostructured materials. For MgO, peaks at 36°, 43°, 62°, 74° and 75° with (h k l) values (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) respectively. Whereas TiO_2, 25°, 37°, 48° and 68° peaks with (1 0 1), (0 0 4), (2 0 0) and (1 1 6) respectively (Figure 1).

In case of $MgTiO_3$ and Mg_2TiO_4 the peak positions are 27°, 38°, 41° and 54°, 56°, the corresponding (h k l) values are (2 1 4), (3 0 9), (2 3 2) and (2 2 3), (3 2 1) respectively. At 500°C heating temperature the MgO and TiO_2 peaks were matching with the JCPDS card numbers 65-0476 and 21-1272. Whereas 600°C, $MgTiO_3$ and Mg_2TiO_4 phases by JCPDS card numbers 02-0874 and 79-0830 were appeared along with MgO and TiO_2. The reason behind this is whenever the heating temperature is increases the Ti ions reacted with Mg ions and formed new phases like $MgTiO_3$ and Mg_2TiO_4. The average crystallite sizes were measured as 29 nm and 36 nm by Debye-scherrer's equation ($D=K\lambda/\beta\cos\theta$) for different heating temperatures 500°C and 600°C respectively [15].

Particle size analyzer

The particles distribution of MgO/TiO_2 nanocomposite rods at different heating temperature was as shown in figure. The MgO/TiO_2 nanocomposite rods were dispersed in ethanol solution using ultra sonicator. The particles size analyser is works on the principle of dynamic light scattering (Figure 2).

Here the YD-laser-532 nm light source is used for to measure the particle size distribution. The mean value of the distribution histograms is taken as average particle size. The average particle sizes were 38 nm and 43 nm for different heating temperatures such as 500°C and 600°C respectively. These results were nearly equal to the XRD average crystalline sizes [16].

Zeta Potential: The MgO/TiO_2 metal oxide nanocomposite rods stability was observed by particle size analyser using zeta potential measurement. Generally the positive and negative zeta potential values were obtained due to the strong coulomb attraction bonds of the nanostructured materials (Figure 3).

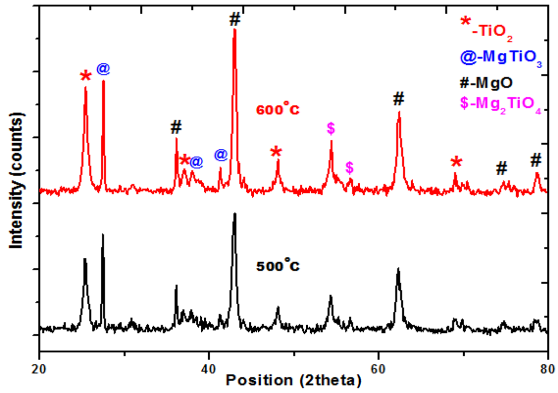

Figure 1: XRD pattern of MgO/TiO_2 nanocomposite rods.

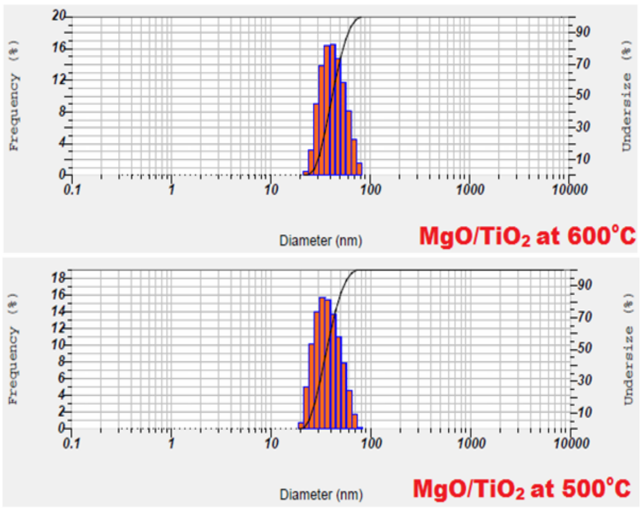

Figure 2: Particles distribution in particle size analyzer.

From the zeta potential verses intensity graphs the negative zeta value (below 50 mV) is shown in both 500°C and 600°C. These values infer that the MgO/TiO_2 metal oxide nanocomposite rods were having high stability [17].

Transition electron microscope

The morphology and d-spacing values were measured by Transmission Electron Microscope. The TEM images of MgO/TiO_2 metal oxide nanocomposites at different heating temperatures were shown in the Figures 4 and 5.

The images infer that the nanocomposite rods were formed. At 100 nm magnification the nanocomposites were formed as rod like structures, showing average length 250 nm and diameter 80 nm for both heating temperatures. The d-spacing values of nanocomposite rods were measured as 0.343 nm and 0.339 nm for 500°C and 600°C respectively. These d-spacing values are exactly coinciding with XRD d-spacing values [18].

Fourier transform infrared spectroscope

The bond analysis of chemical reactions was investigated by FTIR. FTIR spectrums of MgO/TiO_2 nanocomposite rods for both heating temperatures were shown in figure. FTIR analysis was done in the range of 500 cm^{-1} to 400 cm^{-1}. Almost equal bonds are shown in both the temperatures. The O-H stretching vibration bonds represented in the range of 4000 cm^{-1} to 3400 cm^{-1} are due to the water molecules. From 3400 cm^{-1} to around 1600 cm^{-1} endothermic peak observed due to very weak bonding vibration of water molecules (Figures 6 and 7).

The stretching vibrations were possible with tetra fluoroborate (BF_4) anion from 1400 cm^{-1} to 1200 cm^{-1}. Around 1100 cm^{-1} the peaks obtained with the effect of C=O bonds. In the case of 500°C heating temperature Ti-O-Ti vibration bonds were formed with in the range of 1000 cm^{-1} to 800 cm^{-1}, whereas at 600°C the more stretching vibrations occurred may be the phase transition of nanocomposite rods. The possible stretching bond is Ti-O-Mg. The strong Mg-O stretching bond is observed below 700 cm^{-1} in both heating temperatures [19].

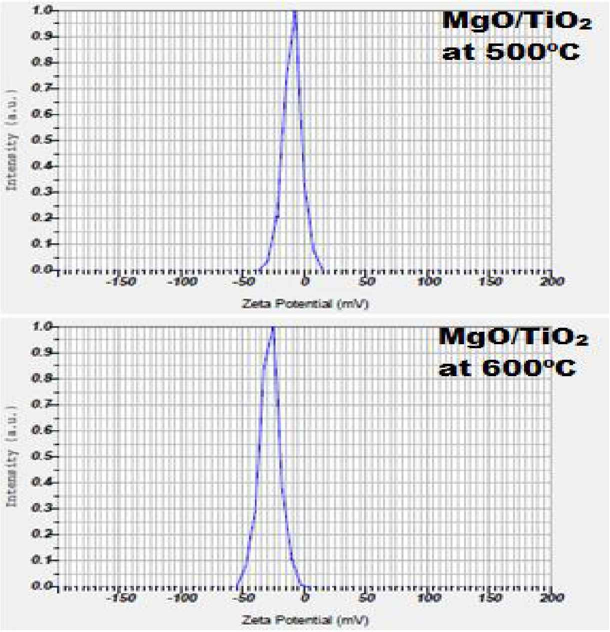

Figure 3: Zeta potential of MgO/TiO_2 nanocomposite rods.

Thermo gravimetric and differential thermal analyser

The thermal properties of MgO/TiO_2 nanocomposite rods were observed by thermo gravimetric and differential thermal analyser. The TG/DTA curves of MgO/TiO_2 nanocomposite rods at different temperatures were shown in figure. In the TG/DTA analysis the TG and DTA curves were taken in the function of temperature, the temperature range is room temperature to 800°C. The total weight loss of the 500°C heat treated nanocomposite rods were 7%. Below 100°C the weight loss occurred due to the water evaporation. After this temperature up to 350°C, the weight loss was observed by evaporation of unreacted inorganic materials in the sample. Beyond 350°C the weight loss was caused by decomposition of organic materials from the rods. These results were supported by DTA curve (Figures 8 and 9).

Whereas in case of 600°C heat treated nanocomposite rods the total weight loss was observed as 3.5%. Weight loss occurred below 100°C due to the evaporation of water molecules. From 100°C to 350°C, the weight loss occurred by evaporation of inorganic materials. At 350°C the endothermic peak of DTA curve represents evaporation of inorganic materials. Immediately after that temperature the exothermic peak was formed, due to the phase transition of MgO/TiO_2 nanocomposite rods which is explained in XRD. Again the weight loss was observed beyond 450°C due to the evaporation of organic materials in the rods [20].

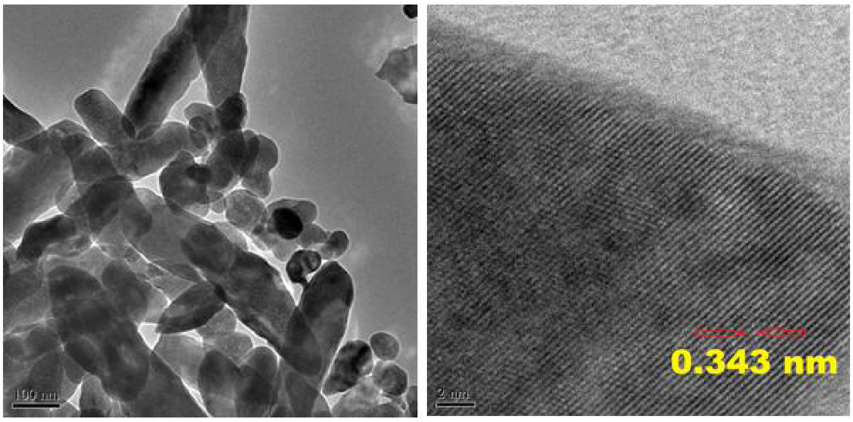

Figure 4: TEM images of MgO/TiO_2 nanocomposite rods at 500°C.

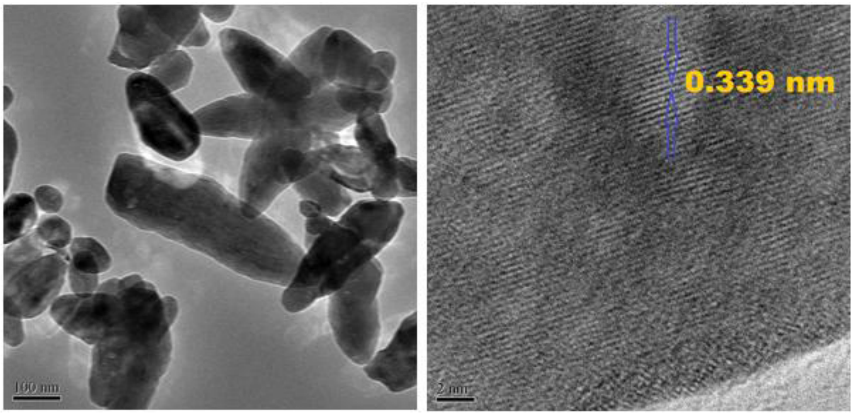

Figure 5: TEM images of MgO/TiO_2 nanocomposite rods at 600°C.

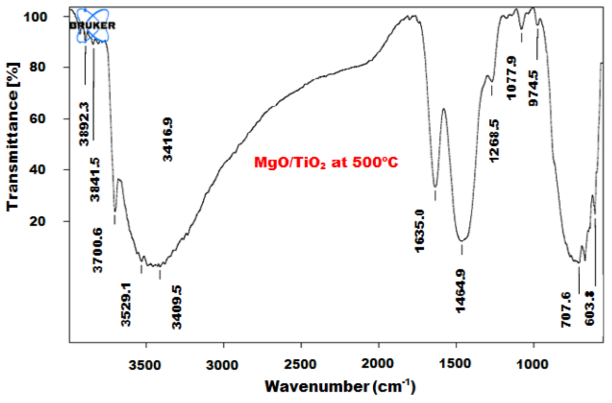

Figure 6: FTIR spectrum of MgO/TiO_2 nanocomposite rods at 500°C.

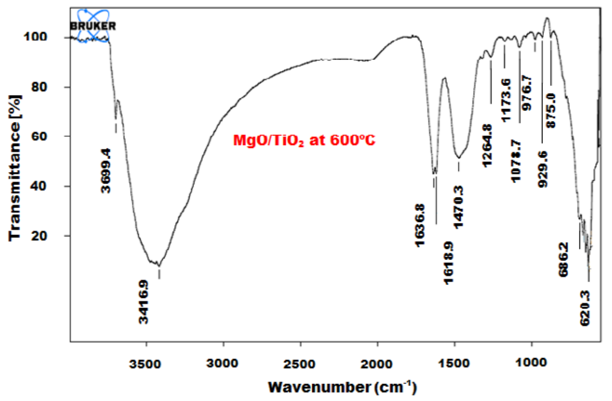

Figure 7: FTIR spectrum of MgO/TiO_2 nanocomposite rods at 600°C.

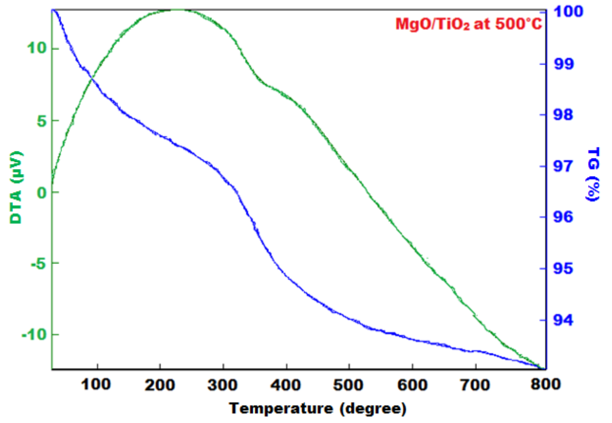

Figure 8: TG/DTA curves of MgO/TiO_2 nanocomposite rods at 500°C.

Resistance studies

The resistance studies were studied by using simple zig set up. The zig set consists of pellet holder along with two Cu electrodes. This Cu electrode probes were connected to electric meter and observed the resistance values of the material. The pellets were prepared using high applied pressures. In this present study, the MgO/TiO_2 nanocomposite rods at different heating temperatures were made as pellets, kept in pellet holder and resistance readings were taken with the help of electric meter (Figure 10).

At 500°C heating temperature the resistance is 114.35 MΩ, whereas in 600°C the resistance is 168.92 MΩ. This means that whenever the heating temperature increases the corresponding resistance value increases [21].

Conclusions

In this present paper, MgO/TiO_2 nanocomposite rods were successfully synthesized by microwave assisted method using room temperature ionic liquids. The structural properties of the nanocomposite rods were investigated with the help of XRD, PSA and TEM. In XRD observation, whenever the heating temperature increases from 500°C to 600°C the MgO/TiO_2 nanocomposite rods changed its phase. The newly obtained phases were $MgTiO_3$ and Mg_2TiO_4. This XRD patterns also matched with JCPDS card numbers. The average crystallite sizes were measured as 29 nm and 36 nm. From the particle size analyzer, the average particle sizes were obtained as 38 nm and 43 nm, the zeta potential values infers that the nanocomposite rods were having high stability. The nanocomposite rods structure was confirmed by TEM, and the measured d-spacing values were matched with XRD. The bond analysis was done by FTIR; it states that the phase transition

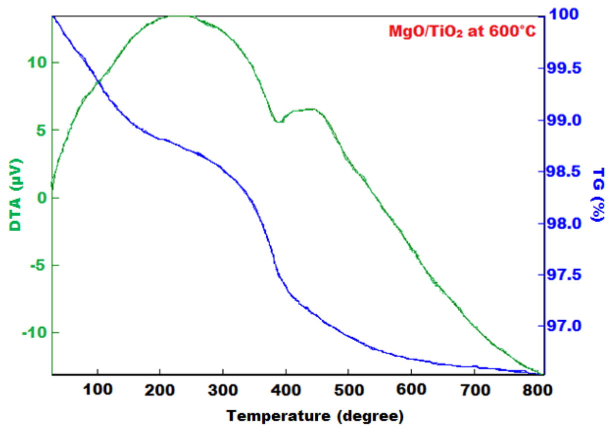

Figure 9: TG/DTA curves of MgO/TiO_2 nanocomposite rods at 600°C.

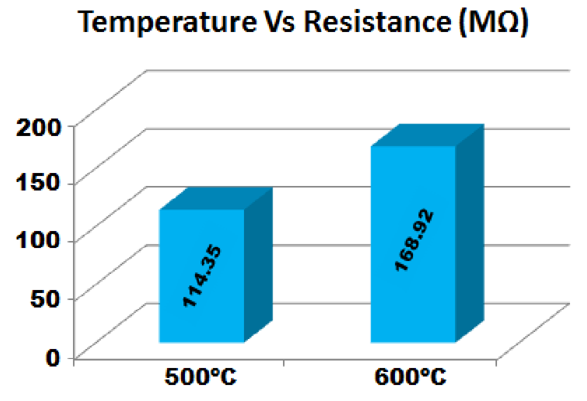

Figure 10: Resistance in the function of heating temperatures.

was formed in the nanocomposite rods. TG/DTA analysis supports the phase transition of the material as well as weight loss was decreased along with increasing of heating temperature. The heating temperature increases resistance values increases.

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