SYNTHESIS AND CHARACTERIZATION OF ZIRCONIUM OXIDE NANOPARTICLES BY MICROWAVE IRRADIATION METHOD

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ABSTRACT

The morphology, structure, and optical properties of ZrO2 nanostructures produced by microwave irradiation have been thoroughly investigated. The ZrO2 nanoparticles' X-ray diffraction patterns confirm their monoclinic structure. The present method produces ZrO2 nanoparticles with a relatively lower band gap energy (4.88 eV), which could be used as a photocatalyst. The PL spectra for the sample zirconium oxide, synthesized for a wavelength range of 350-750 nm was recorded.

Keywords: XRD; UV and Band gap.

1. INTRODUCTION

Zirconium oxide (ZrO2) has bandgap in the range of 5-7 eV. It has received a great interest in technologically anchored current society due to its promising application in optical and electrode materials. ZrO_2 also has major consideration as a component to prepare composite materials for catalytic applications. Zirconium oxide has wide application in more than a few manufacturing fields, such as high performance ceramics, catalysts, high- temp fuel cells, oxygen sensor, damage resistant optical coatings, and bioceramics such as orthopedic and dental implants. Zirconia exists in three crystalline forms of monoclinic, tetragonal and cubic structures at atmospheric pressure. The martensitic conversion from the tetragonal to the monoclinic structure has grand importance in ceramic and catalytic applications of zirconia. The ZrO_2 nanoparticles have been prepared by various routes: sol- gel method, plasma sputtering, ultraviolet (UV) irradiatio, and ultrasound assisted technique. The uses of microwave irradiation for the preparation of nanoparticles have been developed in current years.

2. EXPERIMENTAL PROCEDURE

2.1. MATERIALS

Zirconium acetate (Sigma Aldrich) and ammonia solution (Merk, 98%) were used for the synthesis of ZrO2 nanoparticles. Zirconium acetate were of analytical grade and used as received without further purification was used for the synthesis. Double distilled water was used for all the experiments.

2.2. SYNTHESIS

The initial solution was prepared by dissolving 0.1 mol of zirconium acetate in 20 ml of ethanol, which was later mixed with 175 ml ofdeionized water. The pH of the solution was maintained at 8 by adding a liquid ammonia solution dropwise. The resulting product was filtered and washed with double distilled water and ethanol until it became free from impurities. The precipitate was irradiated for 5 minutes in a household microwave oven (radiation frequency - 2.45 GHZ, Power up to 1 KW) with convection mode, giving a white product. Finally the resulting powders were thermally treated at 300 °C sample for 12 hours.

2.3. CHARACTERIZATION

The resulting powders were analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument diffractometer with monochromatic CuKα1 wavelength of 1.5406 Å. The Fourier transform infrared

spectra (FT-IR) of the samples were recorded by using a Nicolet 5DX FTIR spectrometer. The ultraviolet (UV) spectrum of the ZrO2 samples was recorded on a Perkin Elmer UV-visible DRS spectrophotometer.

3. RESULTS AND DISCUSSION

3.1. X-RAY DIFFRACTION (XRD)

Figure 1 depicts the XRD patterns of the different samples prepared by the MWI method. the monoclinic ZrO2 crystalline structure by comparing the results with JCPDS card No. 89-9066.

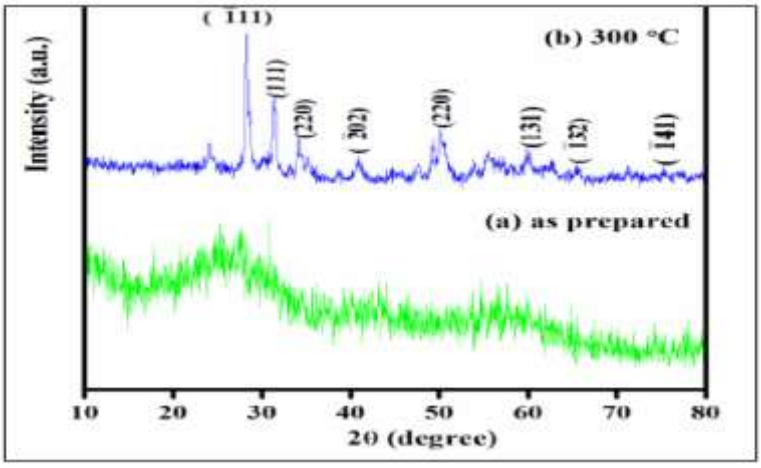

Figure 1. XRD patterns of ZrO_2 samples

The reflections correspond to the characteristic planes (111), (220), (202), (131), (132)and (141). The average crystalline size of the crystallites was evaluated using the Scherrer's formula. The average particle size of ZrO2 is estimated to be around 14 nm for sample B.

3.2. FOURIER TRANSFORM INFRARED (FTIR) ANALYSIS

The formation of ZrO_2 functional group from the zirconium hydroxyl group was also confirmed from FT-IR analysis. The FT-IR spectrum of ZrO_2 nanostructures of sample Fig. 3.3 .

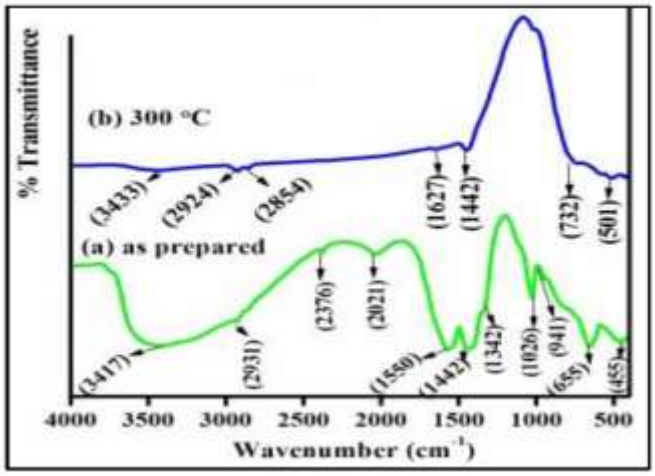

Figure 2. FT-IR spectra of ZrO_2 samples: (a) Sample A, (b) Sample B

3.3. UV-VISIBLE – DIFFUSED REFLECTANCE SPECTROSCOPY (DRS)

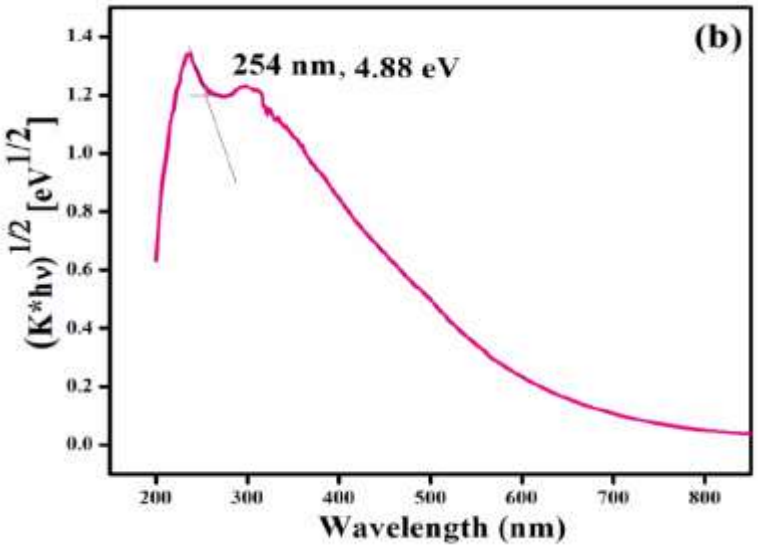

Figure 3. UV-Vis diffused reflectance spectra of ZrO_2 samples

The bandgap value is obtained from the Kubelka- Munk formula which can beexpressed by the following relation [4,5] .

$$K = \frac{(1-R)^2}{2R}$$

Where K is the reflectance transformed according to Kubelka Munk and R wasthe reflectance (%). The reflectance accepts onset peaks were located at 254 nm, corresponding to the band gap of 4.88 eV respectively [6].

3.4. PHOTOLUMINESCENCE SPECTRAL STUDIES

The PL spectra for the sample zirconium oxide, synthesized for a wavelength range of 350-750 nm was recorded and shown in figure 3.4. The spectrum (figure 3.4) shows the wide emission bands at 512 nm. The PL emission bands in current ZrO_2 could be caused by transitions from surface trap states in the conduction band to lower energy levels near the valance band. This is consistent with the findings in the literature [7,8-10]. Due to an extremely narrow particle size distribution, the small particle size was the primary cause of the broad fluorescence band. The presence of oxygen vacancies between 478 and 497 nm caused the bands to be noticed [9-11]. The produced ZrO_2 nanoparticles have a great deal of promise for future optoelectronic Nano device applications [12,13].

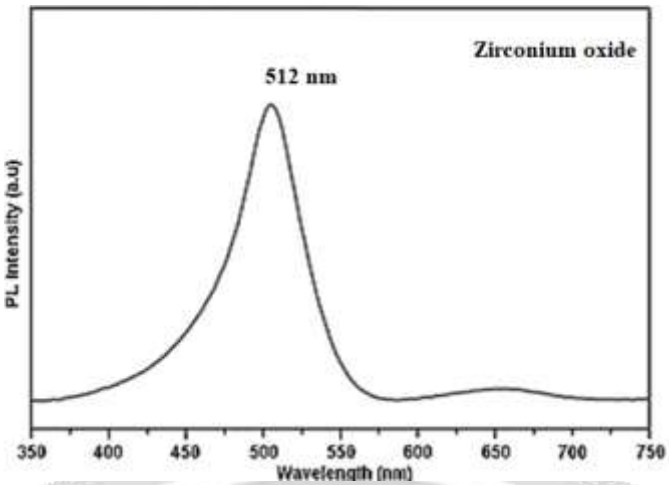

Figure 4. PL Spectrum of Zirconium Oxide

4. CONCLUSION

The morphology, structure and optical properties of ZrO2 nanostructures synthesized by microwave irradiation have been investigated in detail. The X- ray diffraction patterns of the ZrO2 nanoparticles confirm monoclinic structure. The ZrO2 nanoparticles obtained by the present method are found to have relatively smaller band gap energy (4.88 eV), which is potentially to be used as a photocatalyst. The PL spectrum shows the wide emission bands at 512 nm. In summary, results obtained show that the microwave-assisted method is a promising low temperature, cheap, and fast method for the manufacture of ZrO2 nanoparticles for optical and other related applications.

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