

SYNTHESIS AND CHARACTERIZATION OF Eu_2WO_6 BY HYDROTHERMAL METHOD

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ABSTRACT : Metal oxide nanocomposites play an important role in nanoworld. Materials with photo catalytic properties are widely studied because of their numerous applications. These applications include solar cell application and other photodegradation properties too. This photodegradation property include the waste water management too. This paper reports the synthesis and characterization of europium tungstate nanocomposites and explain their photocatalytic applications. The synthesis is carried out with hydrothermal method. XRD, FTIR, SEM and PL studies have been used to study the structural and morphological and optical studies. From these characterizations the sample is studied and further studied for its future uses.

Keywords:

1.1 INTRODUCTION.

The cornerstones of nanoscience and nanotechnology are known as nanomaterials. In a few years back, the nanomaterials are grown for the development activities in an interdisciplinary area of research. It has a broad area for conducting the research. In structural and nonstructural applications, the nanomaterial has some useful properties which can be exploited. It is already having a significant commercial impact, which will assuredly increase in the future. In all around the world, the nanotechnology can be used to develop the technical aspects and for the improvement in life and social benefits. In nanomaterials there are different forms such as nanorods, nanotubes, nanocomposites, clusters etc.

The material which can be converting nanosized particle into a matrix form of standard materials is known as nanocomposites. This type of materials can be prepared by any combination of materials and the material such as metal, ceramics, and polymers. By the addition of particles, the result showed that the drastic improvement in properties can be changed, the changing properties of the materials such as mechanical strength, toughness, electrical and thermal conductivity. The nanoparticle is very effective when the amount of materials is added normally in a range between 0.5% & 5% by weight. Like nanomaterials, nowadays nanocomposites also used in common in this technological society[1-2]. Applications of nanocomposites include electro catalyst in batteries, in fuel consumption this material is used as a lightweight materials, used as a artificial joints, carbon nanotubes can be made with nanocomposites fibers , marine application, food packaging, fuel tanks, films, environmental protection, flame ability reaction , erosion and corrosion application, capacitors for computer chips, Automotive engine parts, Oxygen and gas barriers, non-linear optics, battery cathodes, and ionic, nanowires, sensors, etc.

1.2 MATERIAL AND METHOD

The materials were used for the synthesis of Eu_2WO_6 were Sodium tungstate (Na_2WO_4). (0.05M)(1.65 g), Sodium hydroxide (NaOH) (4g), Europium nitrate [$\text{Eu}(\text{NO}_3)_3$] (0.243g) purchased from Sigma-Aldrich and Deionized water(100 mL). The preparation was done by using hydrothermal method. For the preparation of a sample Eu_2WO_6 . First, take a 1.65g of sodium tungstate and dissolved it with 100 ml of deionized water and stirred it for 30 min. After stirring it to add 0.243g of Europium nitrate $\text{Eu}(\text{NO}_3)_3$ into a sodium tungstate solution at room temperature and stirred the solution. When the stirring is going on, the NaOH solution is poured into the solution drop by drop till the solution were adjusted into a pH level 10 to get the complete precipitation of Eu_2WO_6 . Then the resulted solution was filtered by using filter paper. After the filtration, the sample was collected and kept it in the hot air oven for 14 hr at 110°C [11]. When the sample is taken from the hot air oven, the sample is filled with distilled water and keeps the sample in a cold place till the white precipitate is present under the container. After the white precipitate is appear the sample is taken into an oven at 120°C and the sample is kept in a muffle furnace at 110°C . After this procedure was done, the sample was seen in the form of powder, so the powder sample is collected from the furnace and further carried out for different characterization.[12].

1.3 RESULT AND DISCUSSION
XRD RESULT

XRD analysis of the prepared sample was taken out using the instrument XPERT-PRO using CuK α radiation. The generation voltage and current was setup at 30mA and 40 kv respectively. The samples were scanned in the 2 θ ranges in 0°-90° ranges in continuous scan mode. By continuous scan mode, the XRD analysis of the Eu₂WO₆ nanocomposites exhibited Bragg's reflections, which was indexed on the basis of the hexagonal phase. The diffraction peaks can be found out using the value of 2 θ in a given sample[7-8]. From this sample, the wavelength values can be in the range of 1.5406 Å. The measurement temperature 25°C and the specimen length can be measure as 10 mm also can be used in this material for the measurement. The sample's XRD analysis graph are shown in a below figure 1

Fig 1 XRD pattern of the sample

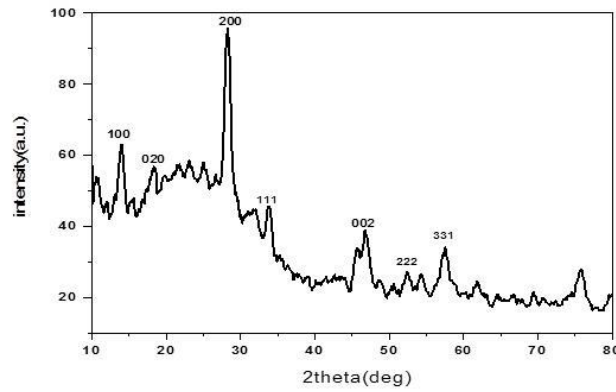


TABLE 1: XRD ANALYSIS OF Eu₂ WO₆

2 θ	d(Å) calculation	hkl
13.8458	2.8389	100
28.2658	4.8445	200
33.8385	2.4548	111
46.8449	2.5595	002

From the obtain spectra of the sample Eu₂ WO₆, the diffraction peaks that are located at 2 θ values are 13.8458, 28.2658, 33.8385 and 46.8449 which are corresponding to the peak 100, 200, 111 and 002 respectively for monoclinic and hexagonal phase in JCPDS file no 33-1387. The crystal parameter of the material can be denoted as K α_1 =1.54060, K α_2 =1.54443 and K β =1.39225. By using this value of 2 θ the particle size of the material can be find out using Debye-Scherer's equation ;

$$\text{The grain size of the sample } D = \frac{0.9\lambda}{\beta \cos \theta}$$

Where, D particle diameter

- K \rightarrow is a constant equal to 0.9
- λ \rightarrow wavelength of the X-ray source
- β \rightarrow Full width half maximum(FWHM)
- θ \rightarrow half diffraction angle

From this equation the strong sharp peaks indicates the good crystalline of Eu_2WO_6 . The size of Eu_2WO_6 by using Debye-Scherrer's equation. From this peak, the highest peak was obtained in 2θ value 28.2658 with a hkl value (200). The total mean size of the crystal grain is estimated about 27.4nm so it shows that the material is in average particle size.

FTIR RESULT

The FTIR technique is used to identify the functions groups that are present in the sample. The FTIR spectrum is recorded in alpha T, Bruker, Germany using ATR technique. The range of frequency in between 4500-500 cm^{-1} to identify functional groups in the synthesized particles. The FTIR peaks obtained in the present study are presented in figure 2. FTIR peaks are assigned to corresponding functional groups which is present in the sample Eu_2WO_6 .

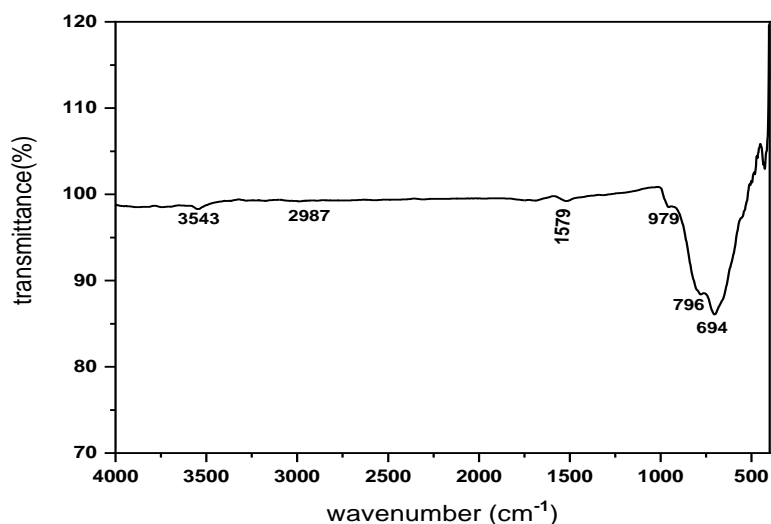


Fig 2 FTIR spectrum of the sample

TABLE 2 OBSERVED VIBRATIONAL WAVE NUMBER AND THEIR CORRESPONDING ASSIGNMENTS

Wave number(cm^{-1}) [Eu_2WO_6]	Assignments
3543	O-H stretching vibration
2987	hydration modes
1579	O-H stretching vibration
979	W=O bending band
796	O-W-O bending band
694	O-W peak

In table 2 shows that observed frequency and their corresponding assignments. The europium tungstate oxide sample shows absorption peaks at 3543, 2987, 1579, 979, 796, and 694 cm^{-1} . Metal oxide generally gives absorption band below 1000 cm^{-1} that arising from inter atomic vibration in the present study it is observed at 979, 796, and 694 cm^{-1} . The strongest absorption band of the sample can be assigned. The strongest absorption band is formed in the region 694 cm^{-1} . The wave number at 3543 and 1579 shows that O-H stretching band. The absorption band at 979, 796, and 694 represents to O-W bending vibration; [9-10] this confirms the presence of tungstate oxide in the europium doped tungstate oxide nanoparticles.

SEM RESULT

The morphological structures were studied by using scanning electron microscope, JOEL model: JSM-6390 LV. By using this instrument, the sample is taken into aluminum stubs inside the chamber and it can be analyzed for studying the morphological structure of the sample. The voltage which is used for analyzing was 20 kV for imaging process. The obtain images of sample by using SEM were shown below.

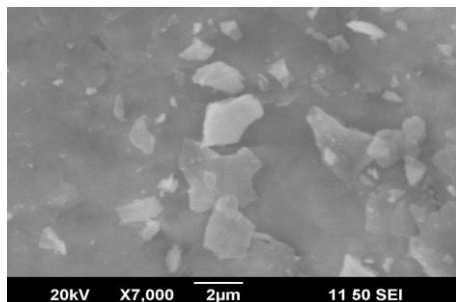


Fig 3 SEM image of the sample

By this image (fig 3), the result can be revealed that the images are produced with the high acceleration voltage which is focus to the surface change of europium tungstate oxide nanocomposites [6]. The lateral dimensions of the sample are taken approximately from $1\mu\text{m}$ to $50\mu\text{m}$ range for taking the image of the sample. The average particle size of the sample is calculated 28.2 from the SEM micrograph.

PHOTOLUMINESCENCE SPECTROSCOPY

Photoluminescence (PL) spectra of the europium doped tungstate oxide nanocomposites material were carried out by the spectra fluorometer with a 450W high pressure xenon lamp as an excitation source at room temperature. PL describes the phenomenon of the light emission from the matter after by absorption of photons [13]. It is one of many forms of luminescence and is initialized by photo excitation. Figure shows that the PL emission peak for the europium doped tungstate oxide. The prepared samples were excited at 200nm. For europium doped tungstate oxide the emission peak occurs at 359 nm. The result of the growth sample suggests that it may be used as violet light emitting material. This result is consistent with that observed. The size effect of the surface states have been discussed. By this result it shows that the sample have a presence of photocatalytic properties and the value of band gap energy was 3.4 eV. It shown in the figure 4.

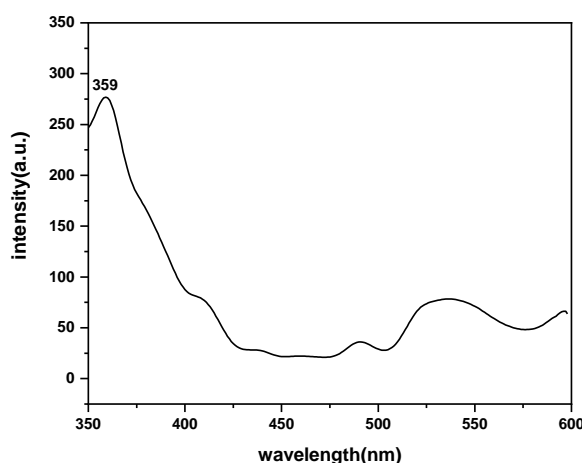


Fig 4 Photoluminescence pattern of the sample

CONCLUSIONS

In the present study, a Europium tungstate nanocomposite is synthesized successfully by hydrothermal method. Europium nitrate and Sodium tungstate were used as starting materials. The compounds thus prepared have been carried out for different characterization. Average particle size is calculated using XRD techniques. The average particle size was found to be 27.4nm. From FTIR spectra the vibration peaks were obtained at 694cm^{-1} which represents the WO band. The surface morphology of the sample was analyzed by SEM. SEM analysis indicated major part surface is clear and free from dislocation. However the hydrothermal methods helped the morphological studies. From photoluminescence, the sample showed photo catalytic properties and the emission peak was obtained at 359nm and the band gap energy was found to be 3.4eV. In future the properties can be enhanced by adding other dopants and the photocatalytic properties can be increased.

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