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Preparation And Characterization Of Psudobrookite (Fe_2TiO_5) Nano Composite For Fuel Cell Applications

S.Ponmani, R. Vishnupriya, T. Rajeshwari and M. Ramesh Prabhu*

Department of Physics, Alagappa University, Karaikudi – 630 003, India.

Abstract - *Nano crystalline psudobrookite (Fe_2TiO_5) has been prepared by using a simple, cost effective sol gel process. The prepared nanocomposites were studied by using XRD, SEM, Raman Spectroscopy, FTIR analyses. The structural and morphological studies were carried out to analyze its structural shape and size of the material. The XRD pattern reveals the formation of orthorhombic structure with Bbmm63 space group. The particle size of the materials was calculated by using the Debye Scherer formula. It is well coordinated with the standard JCPDS no (89-8065). The SEM analysis exhibit the formation of the nano sized particles .The FTIR and Raman analysis confirmed the orthorhombic structure and also its space group.*

Keywords *- Pseudobrookite, XRD, SEM, Raman spectroscopy, FTIR.*

I. INTRODUCTION

The nanoparticles and nanomaterial have been studied intensively due to their physical properties and potential applications [1-3]. The nanomaterial has some interesting special properties, the resulting materials at the nanoscale. Compared to the bulk materials the nanomaterial has improved surface activity and large surface to volume ratio. The perovskite oxide chemical composition can be written as $ABO_{3-\delta}$, where A and B denotes two different cations and the δ is the oxygen deficiency per perovskite unit cell.

The Fe-titanates exist principally as three minerals, namely ilmenite ($FeTiO_3$), Pseudobrookite (Fe_2TiO_5) and ulvospinel (Fe_2TiO_4) [4]. The iron and titanium are extensively studied for many technological applications, such as biotechnology, corrosion, electronics, microelectronics, spintronics technologies, heat resistant colorants for thermoplastics, industrial paints, magnetic materials for telecommunications, audio and video power transformers, ceramic pigments, gas sensing applications, and other applications involving low and high temperature treatments [5-7].

The pseudobrookite type Fe_2TiO_5 (FTO) with orthorhombic structure has been synthesized by numerous methods, such as solid state reactions, hydrothermal, co-precipitation and sol-gel methods. The bulk material properties depend on the size of the prime particles. In the synthesis process the control of particle size and morphology are very crucial role. The electrical, magnetic properties and crystallographic are strongly depends on the preparation methods, conditions, stoichiometry and particle size. The oxide was obtained by using sol - gel technique. The sol –gel technique has a lot of advantages over other fabrication techniques, such as homogeneity, stoichiometry control, purity, ease of processing and controlling the composition. It also enables in a relatively simple way at low temperature as bulk, powder or film, both in crystalline or amorphous form [8-10].

II. EXPERIMENTAL AND CHARACTERIZATION TECHNIQUES

The Ferric nitrate, titanium isopropoxide and oxalic acid were used as the starting materials, Fe_2TiO_5 nanoparticles were prepared by using a sol-gel technique. All the chemicals used in this method were of analytical grade and used as received without any purification. At first, a solution containing 3M of oxalic acid as chelating agent. 30 ml of ethanol was added drop wise into a solution involving 1M of titanium (IV) isopropoxide. Subsequently, 2M of $Fe(NO_3)_3.9H_2O$ and 2M of surfactant (CTAB) were dissolved in ethanol and added to the above solution under constant stirring. Afterwards, the final mixed solution was kept at stirring to form a gel at 60° C and then, was further heated in an oven at 90° C to remove the excess solvent. Finally, the obtained product was calcined for 3h at 900° C in the furnace. The obtained Fe_2TiO_5 (FTO) nanoparticles was characterized.

The morphological and crystal structure of the prepared FTO nanopowder was analyzed by XRD analysis with the help of X'pert PROPAN analytical X-ray diffractometer and morphology of the nanopowder was examined by gold sputter coated scanning electron microscopy (SEM) (FEI Quanta 250 Microscope, Netherlands).The complex formation between the Fe and Ti has been confirmed by FTIR spectra using SPECTRA RXI, Perkin Elmer spectrophotometer in the range of 400-4000 cm^{-1}. The space group of the nanopowder was analyzed by the Micro-Laser Raman (Seiki, Japan).

III. RESULT AND DISCUSSION

A. XRD Analysis

The crystalline structure and phase purity of as prepared Fe_2TiO_5 nanoparticles have been determined by using XRD. The XRD pattern of as-prepared Fe_2TiO_5 is shown in figure 1.The XRD patterns of the obtained powders shows the hematite (Fe_2O_3) and anatase phase (Tio_2) with corresponded JCPDS no of 89-8065. Based on the Fig.1.The observed diffraction peaks can be indexed to the pure orthorhombic phase of Fe_2TiO_5 (a=9.808 A°; b=10.07 A° ; c=3.750 A°) with space group Bbmm63 and their 2θ values are 24.256, 32.722, 35.775, 37.553, 41.083, 46.124, 49.644, 62.621 and 64.207˚ respectively. The obtained sample is a pure orthorhombic structure because there is no impurity phase was detected for other species by the XRD analysis. The average crystalline size of the nanopowder was calculated by using the following Scherer equation. No impurity phases were detected by XRD analysis.

$$D = k\lambda/\beta \cos \theta$$

where K is the shape factor, λ is the wavelength of the X-ray source, β is the breadth of the observed diffraction peak at its full width Half maximum (FWHM) [11]. By using the above formula, the average size of the Fe_2TiO_5 nanoparticles was calculated as 78 nm. The tolerence factor (V. M. Goldschmidt) was calculated by using the following equation

$$t = (r_A+r_O) / [2^{1/2}(r_B+r_O)]$$

where r_A is the radius of A-site cation, r_B is the radius of the B-site cation, r_O is the radius of the oxygen ion.The value of the tolerence factor is found to be t = 0.7222 (t<0.8). From this value the prepared nanocomposite exhibits the perovskite structure. The dislocation density (δ) and strain can also be calculated by using the following equations,

$$\delta = 1/D^2$$

The average dislocation density and strain is found to be 7.222×10^{-3} and 0.1869 respectively.

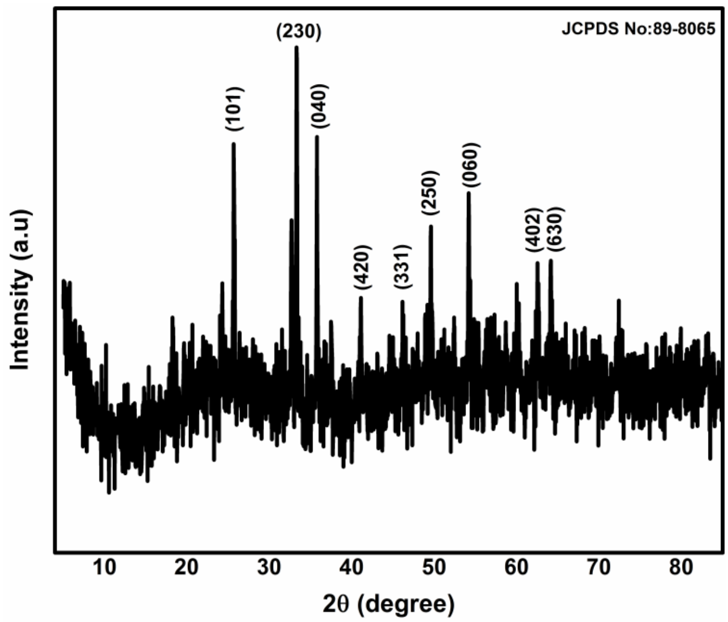

Figure 1. XRD analysis of the Fe_2TiO_5

B. Fourier Transform Infrared Spectroscopy

The FTIR spectra support the formation of Fe_2TiO_5, as shown in figure 2.The obtained spectrum for Fe_2TiO_5 powders calcined at 900°C. The broad absorption band is observed at 3418 cm^{-1} for Fe_2TiO_5 nano powder was assigned to the stretching vibrations of –OH group of absorbed water. This may be due to moisture content. Moreover the absorption band at 806, 557 and 498 cm^{-1} can be assigned to the absorption band of Fe-O, and the strong absorption peak at around 663 cm^{-1} can be assigned to Ti-O band can be assigned Fe_2TiO_5 corresponds to which confirms the formation of iron titanate [12].

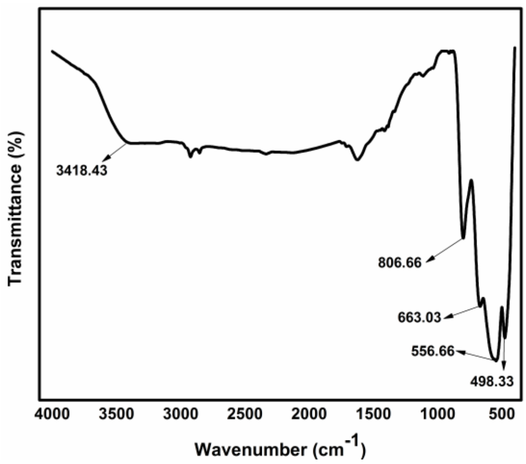

Figure 2. FTIR analysis of the Fe_2TiO_5

C. SEM Analysis

The surface of morphology of the synthesized Fe_2TiO_5 nano powder was investigated by SEM. The figure.3(a), (b) and(c) reveals the different magnification of the samples. Micrographs indicates that a cryofractured surface of the nanocomposite. The average particle size was estimated in the range of 75-90 nm. SEM reveal particles have agglomerated graining structure [13].

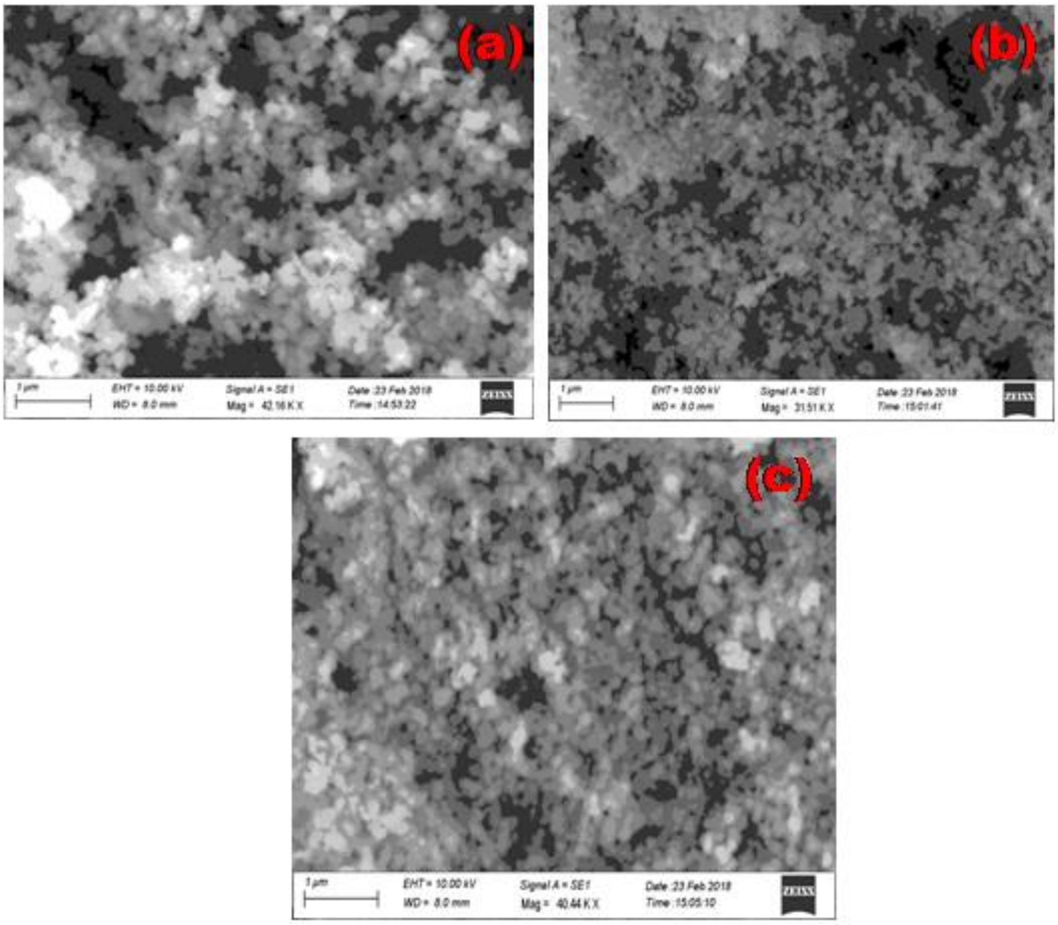

Figure 3. SEM analysis of the Fe_2TiO_5

D. Raman Analysis

The Raman spectra for the prepared pseudobrookite (Fe_2TiO_5) phase are obtained for the powder heterogeneous mixtures of iron (hematite) and titanium (anatase) oxides. The Raman spectrum of the (Fe_2TiO_5) nanocomposite was shown in the figure 4. The (Fe_2TiO_5) nanocomposite shows the crystalline phase. At 900°c the sample exhibit the anatase (TiO_2) phase and also hematite (Fe_2O_3) phase. The pseudobrookite phase is confirmed from the spectrum. The spectrum is a superposition of the broadened bands of the anatase and hematite bands for pseudobrookite features at 285,405,665 cm^{-1}. The present Raman peaks confirm the presence of anatase and hemitate in the prepared nanocomposite and also pseudobrookite. The (Fe_2TiO_5) had separated out due to the stoichiometry of Fe and Ti [14].

The (230) plane reflection shows the presence of Pseudobrookite and the FTIR shows the presence of Ti-O and Fe-O bands. The XRD and FTIR reveal the presence of pseudobrookite.

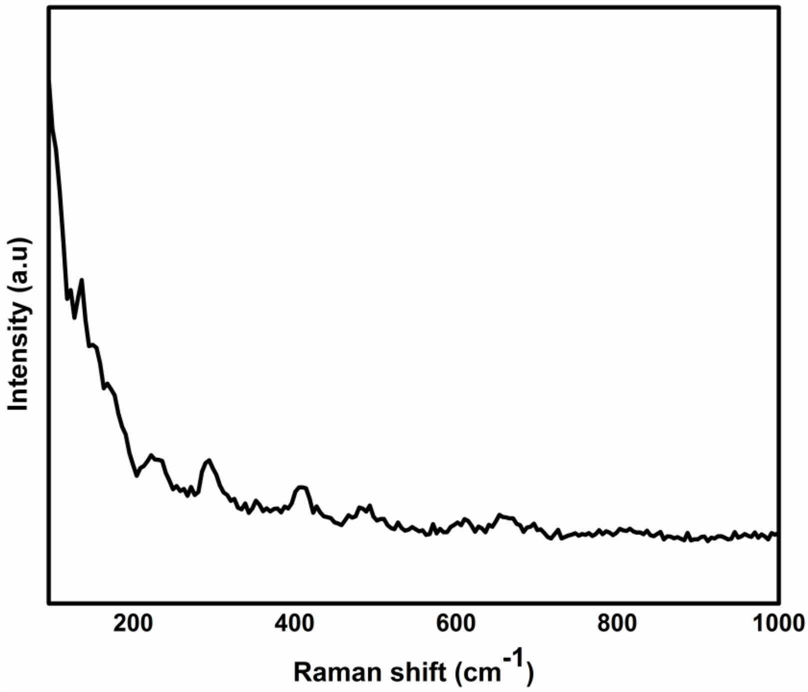

Figure 4. Raman spectroscopy analysis of the Fe_2TiO_5

IV. CONCLUSION

The pseudobrookite Fe_2TiO_5 nanoparticles were successfully synthesiszed by using the sol-gel method. The oxalic acid and the CTAB was used as chelating agent and surfactant. From the XRD the average grain size, space group and also crystalline nature was well matched to JCPDS no 89-8065 and it was confirmed. The FTIR reveals the formation of Fe-O, Ti-O and –OH groups in the prepared nanocomposites. The SEM analysis shows its morphology structure and its average particle size. The Raman spectroscopy reveals the space group of the prepared samples.

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