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Original Article

Synthesis and Characterization of Nanosized Zirconium Titanate Dispersed Polyaniline Nanocomposite

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Abstract
The recent development in the synthesis of bi-metal oxide nanomaterials through a new synthetic technique upgrades the science and technology of nanoscale materials. Zirconium titanate ($ZrTiO_4$) is synthesized by solid-state combustion reaction using ZrO_2 and TiO_2 at high temperature. $ZrTiO_4$ nanomaterial dispersed polyaniline to form its nanocomposite ($ZrTiO_4$-PANI) is carried out by in-situ polymerization of aniline using ammonium peroxide as an oxidising agent. $ZrTiO_4$-PANI nanocompositeis well characterized by various characterization tools. Structural characterization is carried out by X-ray diffraction (XRD) and morphology by Scanning Electron Micrograph (SEM) tool. The bonding nature of the sample was studied by Fourier transform Infrared (FT-IR). Presence of metals like Zr and Ti in the nanocomposite was confirmed by Energy Dispersive X-ray microanalysis (EDX) study.

1. Introduction

Nanomaterials and nanocomposite materials have unique properties in comparison to the same materials in bulk form. Particle size is expected to influence three important groups of basic properties in any material. The first one comprises the structural characteristics, namely the lattice symmetry and cell parameters. Bulk materials are usually robust and stable system with well-defined crystallographic structures. In recent years, much attention has been paid to the synthesis and study of nanomaterials and nanomaterials dispersed polymeric materials because of wide range of potential applications [1-2].

Nanocomposites of directing polymer and metal nanoparticles have considered extensively because of the potential conceivable outcomes to make reasonable materials for electrolysis, substance sensors and microelectronics. The amalgamation of comparatively metals or solid chemical compound in key supreme polymers, for example, Polyaniline is the most interacted on account in the solid electronic connection in between the network of nanoparticles and the polymers [3]. $ZrTiO_4$ shows orthorhombic structure and is integrated by strong state response of oxide such as ZrO_2 and TiO_2 at raised temperature 600°C to 1200°C [4]. $ZrTiO_4$ scattered in PANI have been broadly concentrated on and brought out through oxidative polymerization strategy shaping a $ZrTiO_4$-PANI nanocomposite [5-6]. This shows fascinating properties and additionally numerous applications like quantum electronic gadgets attractive recording materials, sensors, capacitors. Polymer nanocomposite materials represent a new alternative to conventionally nanoscopic inorganic materials filled polymers [7-8]. Filler dispersion nanocomposites exhibit markedly improved properties when compared to the pure polymers or their traditional composites, because of their nanometersizes [9-10]. Properties include increased modulus and strength; outstanding barrier properties improved solvent, heat resistance and decreased flammability. Researcher have attempted to enhance the desired properties of Polymer nanocopmosites and, thus to extend their utility by reinforcing them with nanoscale materials to drive improved properties compared with to the more conventional particulate-filled microcomposites [11-12].

The present work reports the synthesis of zirconium titanate dispersed polyanilinenano composite materials. *In situ* oxidative polymerisation method is adopted for the synthesis of zirconium titanate dispersed poylanilinenano composite materials. Ammonium persulphate is used as oxidizing agent for the polymerisation process. Prepared nanocomposite was well characterised by various characterisation tools.

2. Experimental

2.1 Materials and methods

All chemicals used in the present work were of Analytical reagent grade (AR grade). Solid state combustion method was used for the preparation of Zirconium titanate sample using polyvinyl alcohol as a fuel. As prepared Zirconium titanate dispersed polyanilinenano composite was prepared by *in situ* polymerisation of aniline.

2.2 Preparation of $ZrTiO_4$-PANI nanocomposite

PANI was combined by means of an in-situ oxidative polymerization procedure, in which aniline and ammonium per sulphate (APS) were utilized as monomer and initiator, separately. At first, aniline (0.2M) was scattered in 1 M HCl by fiery mixing utilizing magnetic stirrer for 1hr and aniline hydrochloride is shaped. Known quantity of the zirconium titanate was added to the aniline hydrochloride for composite formation. APS (0.4M) was broken down in 1 M HCl and 0.2M aniline arrangement drop by drop utilizing burette tube into the measuring glass for 4 h. In the wake of dropping the initiator, the blend was kept consistent mixing and the arrangement is put inside an ice shower keeping up mixing for another 24 h. At the point when the response was done, the dull green accelerate coming about because of the polymerization response was separated and washed with refined water successively keeping in mind the end goal to expel the abundance initiator, monomer, and oligomer. Finally the product was dried under room temperature. The preparative apparatus and $ZrTiO_4$-PANI nanocomposite is given in the figure 1.

Figure 1(a): Experimental setup

Figure 1(b): PANI-$ZrTiO_4$nanocomposite

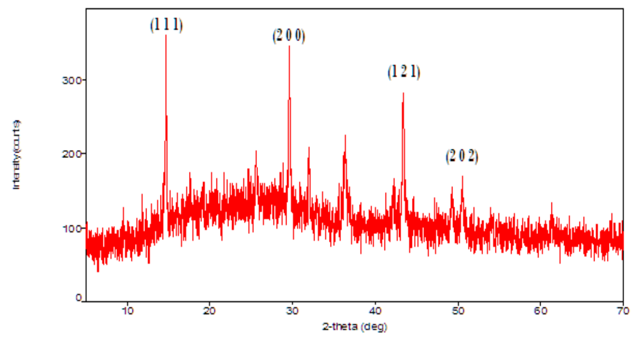


Figure 2: XRD pattern of as prepared ZrTiO4 dispersed in PANI

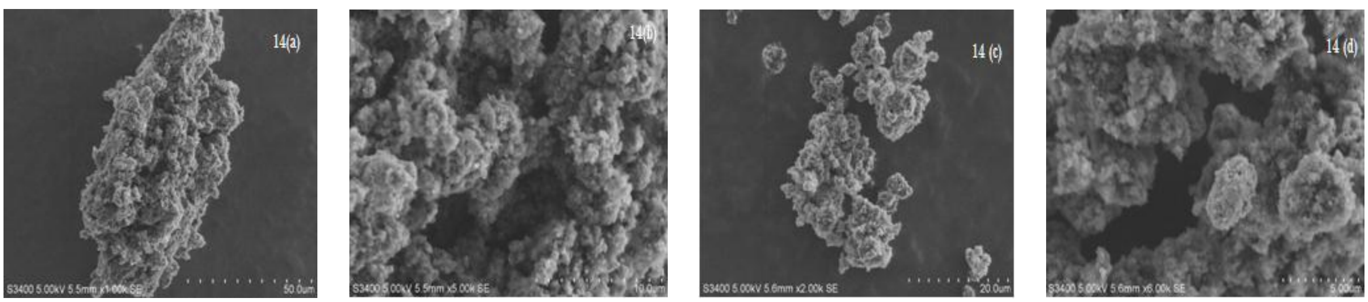
3. Results and discussion

3.1 X-ray Diffraction (XRD)

Figure 2 shows the indexed XRD pattern of as prepared $ZrTiO_4$-PANI nanocomposite material. The highly intense Bragg's reflections are observed in the composite pattern are due to crystalline nature of the composite material. Dispersed $ZrTiO_4$ peaks are observed and are indexed as per JCPDS file 00-034-0415 of $ZrTiO_4$nanomaterial in the composite pattern. The nearness of both polyaniline and $ZrTiO_4$ in the single XRD pattern confirms the development of $ZrTiO_4$ scattered Polyanilinenano composite. Observed 2θ values, literature 2θ values of and miller indices values are given in the table-1.

3.2 Scanning Electron Microscope Study

The surface morphology of the as prepared $ZrTiO_4$-PANInanocomposite is studied by scanning electron micrograph. Figure 3 (a-d) shows the SEM image of as prepared $ZrTiO_4$-PANI nanocomposite materials at various resolutions. The image shows irregular shaped particles of fine agglomeration with dense and compact structure. Dispersion of the oxide nanoparticles into PANI matrix masked with somewhat crystalline nature. However in higher resolution it is clearly observed that a sheet of polymer containing fine particles of oxide increased the compact nature confirms the formation of $ZrTiO_4$-PANI nanocomposite.



(a) (b) (c) (d)

Figure 3(a-d): SEM images of $ZrTiO_4$-PANI nanocomposite at a) 1K b) 2K c) 5K d) 6K

3.3 Energy Dispersive X-ray Study

The EDX tool is used to confirm the presence of Zr and Ti metals in $ZrTiO_4$-PANI Nanocomposite. Figure 4shows EDX pattern of as prepared $ZrTiO_4$-PANI nanocomposite material. This pattern shows presence of both Zr and Ti atom signals at particular KeV. These characteristic absorption peaks of Zr and Ti in a single pattern confirm the formation of $ZrTiO_4$-PANI nanocomposite material.

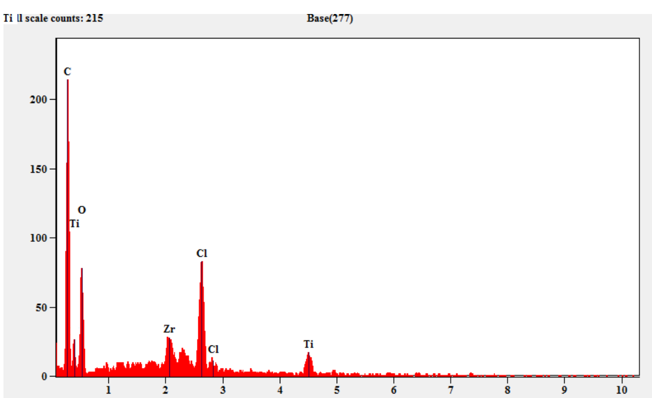


Figure 4: EDX of $ZrTiO_4$-PANI Nanocomposite material

3.4 Fourier Transform Infrared Spectroscopy study

Figure 5 shows FT-IR spectrum of $ZrTiO_4$-PANI nanocomposite. A broad peak from 3500-3000 cm^{-1} shows the presence of moisture moity. A straight peak at 1450 and 1150 cm^{-1} is due to presence of CH stretching and some overtunes. The peaks below 1000cm^{-1} correspond to metal oxygen modes. Peaks at 939, 617 cm^{-1} may be attributed due to Zr-O or Ti-O peaks confirm the formation of $ZrTiO_4$ nanomaterials.

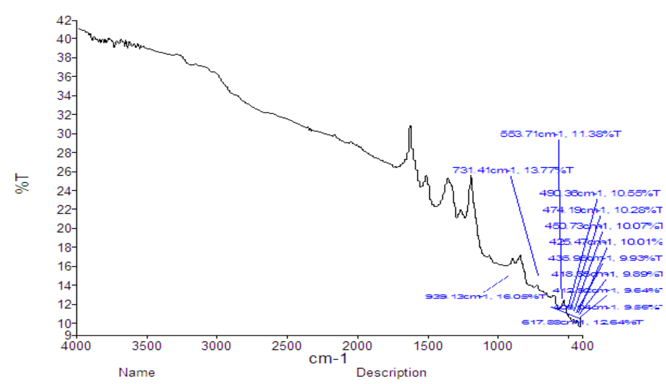


Figure 5: FTIR of $ZrTiO_4$-PANI nanocomposite material

4. Conclusions

Among leading polymers, Polyaniline family has pulled in much consideration of researchers overall on account of their simplicity of amalgamation, interesting conduction system, high natural solidness in nearness of oxygen and water.

They additionally show profoundly reversible redox conduct which is essential for some applications. A fruitful arrangement of $ZrTiO_4$ was done utilizing strong state technique. Polyaniline was orchestrated by oxidative polymerization method.

Consequently $ZrTiO_4$ scattered PANI was arranged and portrayed by various characterization tools. This method is known for its simplicity; hence this method may be used for the preparation of other nanocomposite materials.

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Table 1: Miller indices and 2θ values of Zirconium Titanate in the composite

Miller indices (hkl)	2θ values of Zirconium Titanate (Prepared sample)	2θ values of Zirconium Titanate (JCPDS No: 00-034-0415)
(1 1 1)	30.11	30.44
(2 0 0)	35.12	35.64
(1 2 1)	42.02	41.91
(2 0 2)	51.78	52.63

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