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Fabrication and magnetic properties of Polyimide/Nickel Oxide nanocomposite

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ABSTRACT

Polyimide/Nickel oxide (polyimide/NiO) nanocomposite was successfully synthesized by sol gel technique, using Pyromellitic dianhydriede (PMDA), 4,4-oxydianiline (ODA) and N,N-dimethylacetamide (DMAC) and 10 wt% Nickel titanate nanopowders (NPs). Fourier transform infrared spectrometry (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and vibrating sample magnetometer (VSM) were used to characterize the structure and properties of the obtained nanocomposite. The results indicated that the average size of polyimide/NiO nanocomposite were estimated 65nm. Saturation magnetizations results indicate that by adding of NiO nanoparticles in polyimide matrix, magnetization decreases. The superparamagnetic behavior for NiO nanoparticles and polyimide/NiO nanocomposite of 10 wt% is reported whereas Polyimide has diamagnetic behavior.

Keywords: *Nanocomposite; Polyimide; Polymer; XRD; SEM; VSM.*

INTRODUCTION

Composite consisting of a polymer matrix and dispersed ceramic particles is a kind of materials with great potential properties and applications [1]. Aromatic polyimides exhibit many useful properties such as high transition temperatures, excellent dimensional stability, low dielectric constants, and outstanding thermal and thermo–oxidative stability. Therefore, some of these materials are used in such applications as high performance structural materials and packaging in printed electronic circuity. Polyimides are primarily used in the aerospace and microelectronics industries in the forms of films, moldings, and foams [2]. Adding inorganic materials to polymer matrix can develop the physical properties of polymers. Especially in recent years the inorganic compounds improve polymer material are used in nanosized. Nickel Oxide is a positive semiconductor that can function as an electron receiver.

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Therefore this material can be used in catalysts, cathode batteries, gas sensors, electrochromic films, magnetic materials, active optical fibers; recently, nickel oxide (NiO) nanoparticles have been extensively studied due to its mechanical, electronic, magnetic and optical properties [3-6]. So many methods for the synthesis of nickel oxide nanoparticles, such as thermal decomposition, de-electrode deposition, sol-gel are used. The wet-chemistry synthesis technique used in this study, including sol-gel, sol-precipitation, combustion synthesis, chemical coprecipitation, and hydrothermal synthesis, offers many distinctive advantages over solid-state method in the production of powders such as a controlled morphology, a narrow size distribution and high purity. The properties of nanocomposites are depended on the primary materials which are used to Synthesis of nanocomposites, the amount and percentage of these original materials, and particles sizes, [7-11] because of this, nanocomposites are used widely, such as packing materials, Electronic Industries, Electronic circuit, layer dielectric materials, Antennas and capacitors with high capacity. [12-13] In this study, polyimide/NiO NCs with 10 wt% content loading of NiO were prepared. The chemical structures of the polyimide/NiO NCs are characterized by Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD). Scanning electron microscope (SEM) is used to characterize the dispersion of NiO NPs and the morphology of the polyimide/NiO NCs. Magnetic properties of the nanocomposite was investigated by vibrating sample magnetometer (VSM).

EXPERIMENTAL

Materials and methods

Polyimide/NiO nanocomposite was prepared along a synthetic procedure as summarized in Figure 1. Stearic acid, Nickel acetate, ethanol, 3-amino-propyl-triethoxysilane (APTS), Pyromellitic dianhydride (PMDA), 4, 4-Oxydianiline (ODA) and N, N-dimethylacetamide (DMAc) used in experiments were all of Merck.

The whole procedure and structural characterization of Polyimide/NiO nanocomposite phases have been investigated by FTIR, XRD and SEM. The FTIR spectrum was recorded with a model Perkin Elmer spectrum RX1 of Fourier transform infrared spectrometry using a KBr pellet. The XRD patterns of the powders were recorded on a Model PTS 3003 of SEIFERT diffractometer using Cu Ka radiation (λ=1.5418 Å) in the range from $2\theta=20^{\circ}$ to 70° to examine the crystallization and structural development of Polyimide/NiO nanocomposite. The SEM pictures were recorded with KYKY Model EM 3200 instrument at the accelerating voltage of 25 kV. Magnetization measurements were performed on vibrating sample magnetometer (VSM) Model BHV-55 at room temperature.

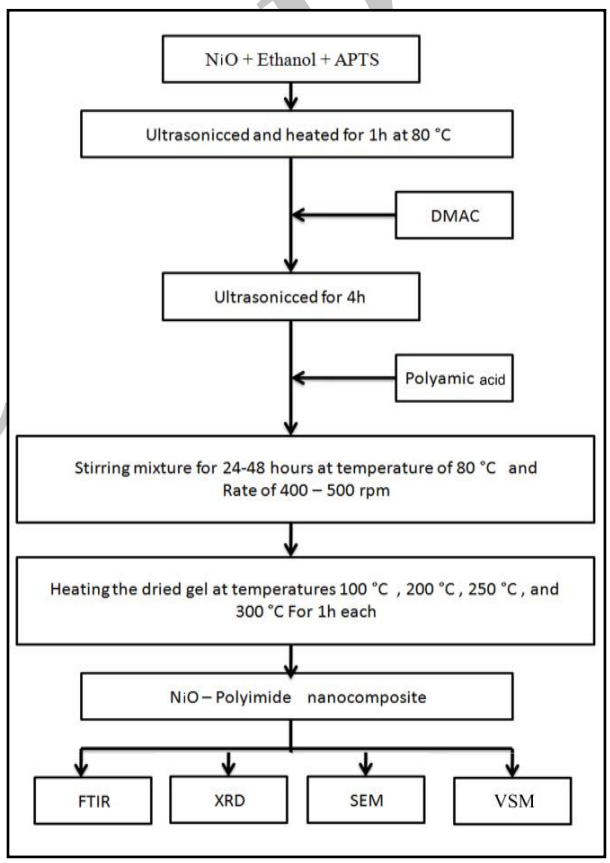

Fig. 1. Flowchart of the polyimide/NiO nanocomposite preparation

Synthesis of NiO nanoparticles

NiO nanoparticles were prepared through a modified wet-chemistry synthesis method. In this procedure, An appropriate amount of stearic acid was first melted in a beaker at 73℃, and then a fixed amount of nickel acetate was added to the melted stearic acid and stirring to form a

homogeneous light green sol, naturally cooling down to room temperature, and drying in an oven for 12 h to obtain dried gel. Finally, the gel was calcined at different temperatures in air to obtain nano-crystallites of NiO. The dried gel is calcinated in 4 stages. In the first stage the primary material is heated until 400℃ with 3℃/min speed. In the second stage heatings is continued at 400℃ for 40min without change. Then the temperature will be increased to 500℃, 600℃ and 700℃. In the last stage the temperature is remained in the same position for 2 hours.

Synthesis of Polyimide/NiO nanocomposite

In order to preparation of Polyimide/NiO nanocomposite (with a nanoparticle loading of 10 wt%), 1 gram of nickel oxide is poured in to the solvent (including: ethanol 95%-3 Amino-propil, Apts) and after 10min, for 1 hour with temperature of 80℃, the sufficient amount of N, N-dimethylacetamide (DMAc) was added and ultrasonicated for 4 hours. In this stage, Nickel oxide suspension is ready. 2 hours before this stage, the materials that are made the polyamic acid must had been combined with the same portions. In this manner the enough amount of 4,4-oxydianiline (ODA) and Pyromellitic dianhydriede (PMDA), In the presence of N,N-dimethylacetamide (DMAc) with the speed of 400-500 rpm and temperature of 80℃ for 2 hours are combined until the (polyamic acid) is made.

After 2 hours when the suspension and polyamic acid were obtained, the suspension was added to the polyamic acid and they were kept in the same temperature and speed for 24-48 hours, until it'll get thick enough that the magnet can't move the materials. After that the materials are poured in the melting pot, and they are kept in these conditions: 1hour at 100℃-1hour at 200℃ – 1hour at 250℃ and 1 hour at 300 ℃.

RESULTS AND DISCUSSION

FT-IR analysis

Figure 2 Shows the FT-IR spectra of the NiO nanoparticles after it was heated until 700°C and polyimide/ NiO nanocomposite with a nanoparticle loading of 10 wt%. In Figure 2a and Figure 2b, the absorption peak at 450 cm-1 corresponds to the Ni-O band. In the spectrum of pure polyimide, the characteristic absorption bands of the imide ring were observed near 1780 (asym. C=O str.), 1720 (sym. C=O str.) and 1380 (C–N str.), cm^{-1} [2]. The FT-IR spectra of the polyimide/NiO nanocomposite, Figure 2b are almost the same as that of pure polyimide. This result indicates that the polyimide/NiO nanocomposite have been successful synthesized.

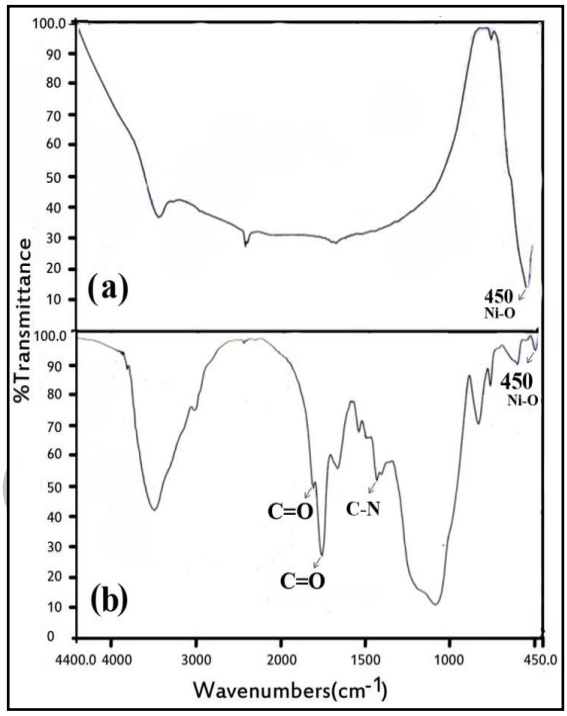

Fig. 2. The FT-IR patterns of (a) NiO nanoparticles, (b) the nanocomposite with a nanoparticle loading of 10 wt%.

XRD study

Figure 3 shows the XRD patterns of NiO NPs, pure polyimide and polyimide / NiO NCs with 10 wt% nanoparticles loading. The XRD pattern of as prepared NiO matches with JCPDS (file number 76-1158). The XRD of pure polyimide [14] exhibits a phase pure amorphous structure, which is associated with the broad peak of 2θ centered on 18°. As shown in Figure 3a, 3b and 3c the diffraction peak at 2θ=18° corresponds to polyimide. Also the prominent peaks at 2θ=37.25°, 43.30° and 62.90°, indicated the formation of phase pure, cubic nickel oxide at this temperature [15]. On comparison of XRD patterns of NiO, polyimide and polyimide /NiO composite, it is confirmed that NiO has retained its structure on dispersion in polyimide matrix during in situ polymerization

reaction. It was indicated that the crystal structure of NiO was still stable when it was doped into the polyimide matrix.

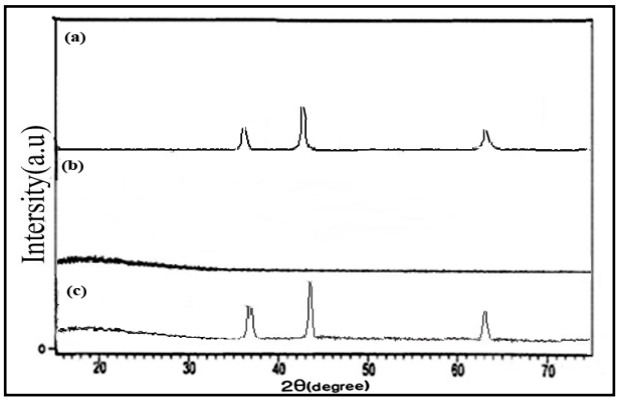

Fig. 3. The XRD patterns of (a) NiO nanoparticles, (b) pure polyimide and (c) the nanocomposite with a nanoparticle loading of 10 wt%.

Morphology of samples

Figure 4a and 4b shows the scanning electron micrograph of polyimide-NiO nanocomposite with 10 wt% of NiO NPs loading. The particles have agglomerated graining structure. In the scanning electron micrograph of the nanocomposite, with the increase of NiO content, the agglomeration become more appreciable and display some connections in some regions. The particle sizes were estimated 65 nm. SEM images reveal a homogeneous dispersion of NiO NPs in the polyimide matrix.

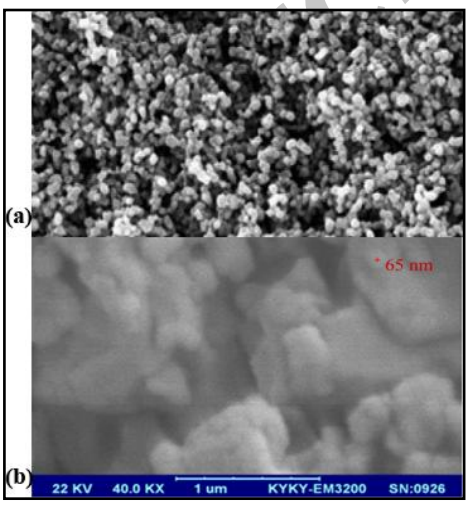

Fig. 4. SEM images of (a) NiO nanoparticles, (b) the nanocomposite of polyimide–NiO with a nanoparticle loading of 10 wt%.

Magnetic properties

Figure 5a and 5b show the field dependent magnetization curves of NiO nanoparticles and polyimide/NiO nanocomposite of 10 wt%. NiO are reported as superparamagnetic materials [16]. Under applied magnetic field NiO nanoparticles and polyimide/NiO nanocomposite show the positive magnetizations. It indicates the superparamagnetic behavior for NiO nanoparticles, and polyimide/NiO nanocomposite of 10 wt%, whereas diamagnetic behavior for Polyimide is reported [17], and the saturation magnetization values of NiO nanoparticles, and polyimide/NiO nanocomposite are 0.5, and 0.34 emu/g, respectively. The results indicate that by adding of NiO nanoparticles in polyimide matrix, magnetization decreases.

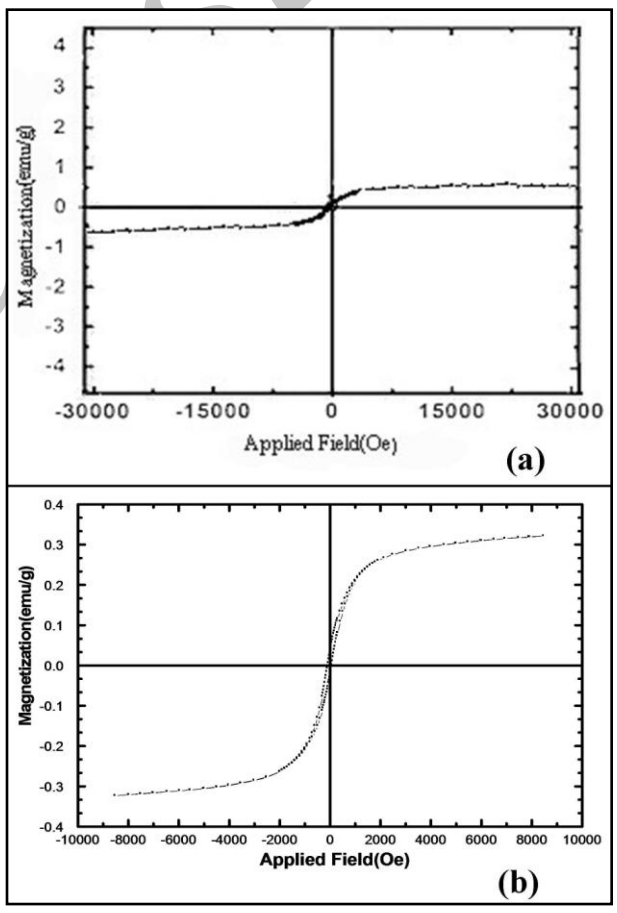

Fig. 5. VSM images of (a) NiO nanoparticles, (b) the nanocomposite of polyimide–NiO with a nanoparticle loading of 10 wt%.

CONCLUSIONS

In this study, polyimide /NiO nanocomposite with 10 wt% of NiO calcined at 700 °C temperature were successfully synthesized. The average size of polyimide-NiO nanocomposite was estimated 65nm. Saturation magnetizations results indicate that by adding of NiO nanoparticles in polyimide matrix, magnetization decreases. The superparamagnetic behavior for NiO nanoparticles and polyimide/NiO nanocomposite of 10 wt% is reported whereas Polyimide has diamagnetic behavior.

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