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NiO-Nanofillers Embedded in Graphite/ PVA-Polymer Matrix for efficient Electromagnetic Radiation Shielding

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Abstract. In this study, we report on the preparation of NiO/graphite sheets nanofillers in PVA-polymer matrix using a simple cost-effective hydrothermal process for EM shielding effectiveness applications. The careful optimization of the growth conditions and NiO/G/PVA relative ratios have resulted in NiO nanoparticles formation with homogeneous density. In this nanocomposite, the NiO nanoparticles and graphite sheets were incorporated into a polymer to enhance the electromagnetic shielding effectiveness. The morphological, structural, and chemical analysis have been conducted by SEM, EDX and XRD techniques. EDX and XRD analysis confirmed the exact chemical composition with high purity. SEM images showed the best morphology with homogenous NiO-nanoparticles distribution on graphite sheets for 15 wt% NiO relative ratio NiO/G/PVA nanocomposite. The nanocomposite was tested in different environments and shielding chambers that contained relatively high-level exposure to electromagnetic radiation. The shielding effectiveness (SE) measurements of NiO/G/PVA showed a significant increase of shielding effectiveness of about 17 dB compared to the commercial shielding paint. This can be ascribed to the homogenous distribution of NiO-NPs over the entire graphite sheets and the strong interaction of the incident electromagnetic radiation with the magnetic and electric dipoles in the nanocomposite. These finding is promising for enhanced flexible and cost-effective EMI shielding applications.

INTRODUCTION

Electromagnetic (EM) pollution is recently considered as one of the most significant challenges in modern electronics and optoelectronics technologies [1,2]. EM waves generated from various electronic devices and communication systems impact life and operational device performances [3,4]. The conventional solution is utilizing metal based materials as shielding materials [5,6]. However, these kinds of materials possess many issues, including heavy weight, less environmental stability and flexibility in structure, difficult in process ability and complexity in manufacturing technologies [7,8]. On the other hand, the polymer nanocomposites possess carbonaceous materials act as alternative solution to fabricate the shielding materials because of massive advantages such as low cost, weight saving excellent process ability, and strong corrosion resistant and dielectric tenability [9-11].

Polymer nanocomposites are a unique set of materials, which enable reduction of electromagnetic disturbances and present as a host matrix for new materials in order to enhance their magnetic, electrical, dielectric, mechanical, and thermal properties [12]. For example, there are many valuable features of metal-oxides nanomaterials which could improve EM wave absorption capability of polymeric nanocomposites because of its greater surface atoms, greater surface area, multi reflection, and thereby increased dielectric and/or magnetic loss [13,14]. Various nanofiller materials were utilized to accomplish polymer/carbon nanocomposites in combination with a wide range

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of electrical conductivity (σ), magnetic permeability (μ), and electric permittivity (ε) [15,16]. Even though polymer based nanocomposites possess benefits over metals, a percolation network could be formed through use of conducting metal-oxides nanofillers within the host polymer matrix, which could be the likely key solution for induction of electrical conductivity in the nanocomposites. However, improving their electrical conductivity and magnetic/dielectric properties is still challenging. In this work, nanocomposites were developed through a unique process [17,18]. The technique involved a specific combination of sonication, hydrothermal, annealing, and grinding processes. Through this process, different experimental parameters like temperature, sonication power, molarity, and pH etc. were strongly regulated to achieve reproducible stable nanocomposites to be used as a prototype for EMI shielding applications.

SYNTHSIS OF NICLE OXIDE/GRAPHITE/PVA-POLYMER NANOCOMPOSITE

Materials: Graphite (>99.0%) from Sigma Aldrich, Poly (vinyl alcohol) (PVA) from Wako Pure Chemical, Ethanol, Nickel Chloride Hexahydrate (NiCl2.6H2O) and absolute ethanol (C_2H_6O, 99.99%) were purchased from Aldrich, water was purified using Milli-Q system, sodium hydroxide (NaOH, 98.0%) from Aldrich, hydrazine hydride from Wako Pure Chemicals, and the commercial shielding paint from Germany.

Hydrothermal Synthesis of NiO-NPs: NiO-NPs were prepared by simple low-temperature hydrothermal process. Firstly, Nickel Chloride Hexahydrate (NiCl2.6H2O) was dissolved in a solution composed of deionized water and ethanol. After that, hydrazine hydride with concentration of 85% was injected in the solution. Then, the pH value was adjusted to 13.7 by using 5 M sodium hydroxide (NaOH) solution. The resultant solution was a blue transparent solution. This solution was transferred to a flask and kept at 80°C for 75 minutes. The result of this hydrothermal process was a colorless solution with black color NiO-NPs at the bottom. Finally, the NiO-NPs was collected from the solution, washed several times using distilled water and ethanol under applied magnetic field then dried at 60°C for 12 hours.

Synthesis of NiO-NPs/Graphite/PVA Polymer Nanocomposite: The nanocomposites were prepared by an optimized hydrothermal method as illustrated in the process flow chart below. The polymer matrix Poly (vinyl alcohol) (PVA) was dissolved in ethanol and mixed with graphite rigorous magnetic stirring. After that, NiO-Nanofillers were added to the PVA/graphite composite with different concentrations, as shown in Table1. Finally, the solution was sonicated for 40 minutes followed by a water bath at 65 °C for 1 hour with continuous rigorous magnetic stirring.

Sample Structure	PVA wt%	Graphite wt%	NiO-NPs wt%
Commercial	-	-	-
Graphite/PVA	50	50	0
NiO-NPs/PVA	50	0	50
NiO-NPs/Graphite/PVA	50	35	15
NiO-NPs/Graphite/PVA	50	25	25

TABLE 1. The prepared samples series with different nanocomposites and relative ratios of NiO-NPs/G/PVA polymer.

RESULTS AND DISCUSSION

NiO-Nanofillers/Graphite/PVA nanocomposites were evaluated by employing various structural and morphological characterization techniques. These investigations have been performed with the help of electron dispersive x-ray (EDX), scanning electron microscope (SEM), x-ray diffraction (XRD), while shielding effectiveness measurements were conducted using shielding box coated with the commercial shielding paint, single generator and spectrum analyzer.

Morphological study of the NiO-NPs/Graphite/PVA-Polymer Structure

SEM has been utilized for the purpose of morphological study of the prepared NiO-NPs/G/PVA nanocomposites. Fig.1(a) represents the surface morphology of the pure PVA polymer directly on the substrate. The PVA polymer observed to covers the entire surface of the substrate with a dense and smooth surface. However, some voids and cavities were observed in the microstructure on the top of polymer surface. This can be ascribed to the mechanical and thermal preparation of the samples prior to SEM analysis. On the other hand, the graphite sheets were observed to form on the top of the polymer with different sizes and thicknesses Fig.1(b). However, the PVA polymer is filling the gaps between the graphite sheets forming a new surface morphology of the G/PVA composite.

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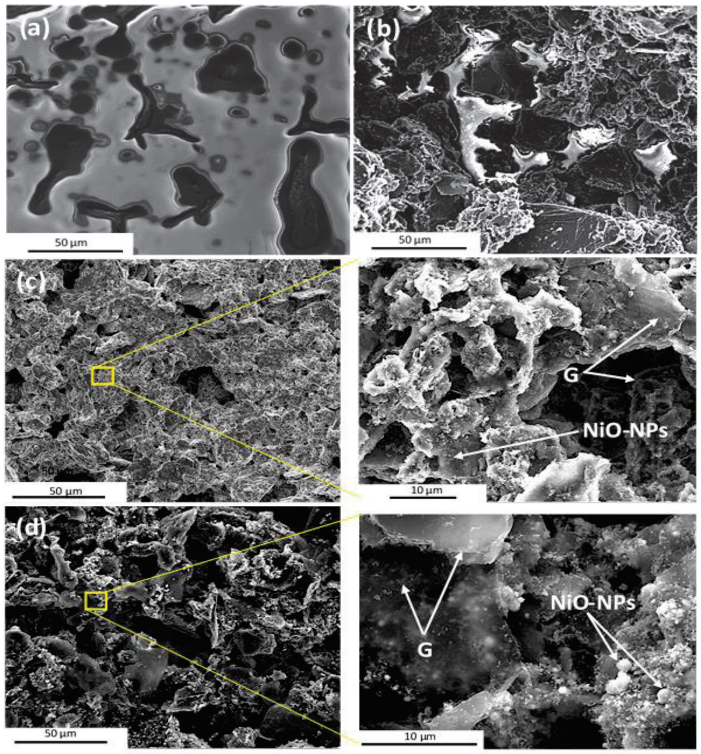

FIGURE 1. SEM images of the surface morphology for different NiO/G/PVA nanocomposites combinations and relative ratios: (a) PVA-polymer directly on the substrate. (b) Graphite/PVA-Polymer. (c) 15 wt% NiO-NPs/G/PVA nanocomposite. (d) 25 wt% NiO-NPs/G/PVA nanocomposite. (The insets in (c) and (d) depict magnified images of the selected areas).

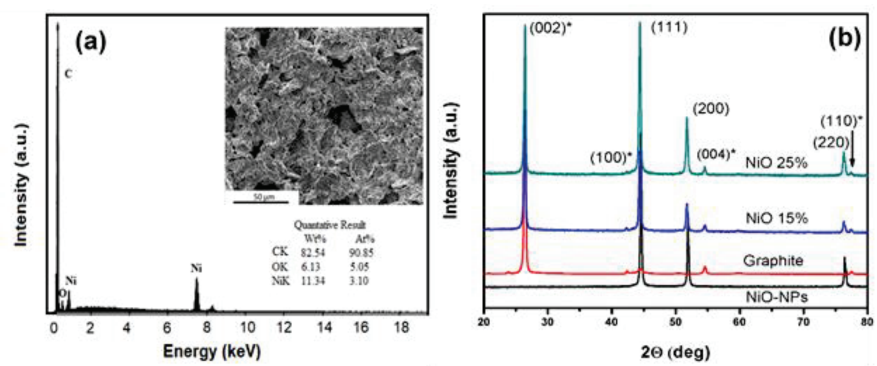

FIGURE 2. (a) EDX measurement of 15 wt% NiO-NPs/G/PVA nanocomposite as grown. The inset shows SEM image of the same NiO-NPs/G/PVA nanocomposite on graphene sheets. (b) XRD spectrums of NiO-NPs, Graphite, 15% NiO –NPs and 25% NiO-NPs, respectively. Graphite peaks are marked with (*).

Fig.(c) shows the surface morphology of nanocomposite with relative ratio 15 wt% NiO-NPs/G/PVA. NiO-NPs are observed to spread out over the entire surface with homogenous size and distribution. However, less aggregation has been observed at 15 % concentration compared to other concentrations. Fig.1(d) depicts the formation of bigger NiO-NPs size due the high aggregation that start to be more significant at higher NiO –NPs concentrations above 25 wt%. The nanoparticles were filling some of the spaces between the polymer and were quite dispersed throughout the sample.

The chemical composition of as-synthesized nanocomposite (NiO/PVA/G) was examined by energy dispersive spectra (EDS) technique. EDX spectrum in fig. 2(a) indicates the presence of only C, O and Ni in the prepared samples. The XRD patterns of the nanocomposites (NiO/PVA/G) observed at different concentrations of (G/NiO) as shown in Fig. 2b. The obtained diffraction peaks can be indexed to the (111), (200) and (220) plans of NiO [19], and (002)*, (100)*, (004)*and (110)* plans of graphite [20]. The obtained peaks became more intense with the increase of NiO concentrations. As a result, graphite peaks intensity decreases with the decrease of the amount of graphite and the increase of NiO concentration. These results and analysis confirm the exact chemical composition of the intended structure with high purity and without the formation of new phases.

Fig.3 depicts the shielding effectiveness (SE) measurements in the frequency range from 0 to 6 GHz. A clear shielding effectiveness improvement has been observed for NiO/G/PVA nanocomposites (c and d curves) compared with the commercial shielding paint (b-curve). The maximum shielding effectiveness of about 32 dB has been detected for the NiO/G/PVA nanocomposites (d-curve) with 15 wt% NiO concentration. This can be ascribed to the homogenous distribution of NiO-NPs over the entire large surfaces of graphite sheets. The strong interactions of the electromagnetic radiation NiO/G/PVA nanocomposite have resulted in this attenuation.

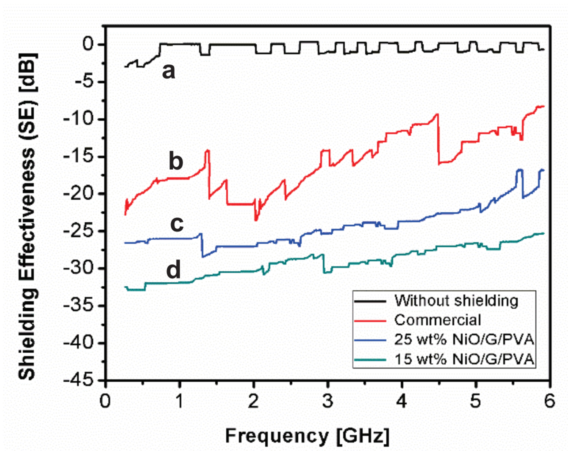

FIGURE 3. Shielding effectiveness (SE) measurements versus frequency: (a) reference sample without shielding paint. (b) commercial shielding paint. (c) 25 wt% NiO/G/PVA nanocomposite. (d) 15 wt% NiO/G/PVA nanocomposite.

The enhancement of shielding effectiveness is mainly due to the combined excellent conductivity of graphite sheets and the superior magnetic properties of NiO-NPs. The improved mechanism of shielding effectiveness based on multi-reflection, absorption can be attributed to the presence of more electrical and magnetic dipoles. These dipoles could create magnetic and electrical fields opposite to the fields of incident radiation. This results in weaker net EM-fields and enhanced overall SE after the total interactions with nanocomposite.

CONCLUSIONS

NiO/G/PVA nanocomposites with high purity have been successfully synthesized using simple and cost-effective hydrothermal process. NiO/G nanofiles in PVA- polymer matrix has proven to be an alternative key solution for EMI shielding applications. NiO with 15 wt% in NiO/G/PVA nanocomposite showed the best morphology and shielding effectiveness (SE) with about 32 dB. This SE enhancement has been ascribed to the percolation network formed through use of conducting metal-oxides nanofillers/Graphene within the host polymer matrix that contributes in presence of more electrical and magnetic dipoles inside NiO/G/PVA nanocomposite, which in turn improves the mechanism of SE. These findings are very promising for low frequency EMI shielding applications.

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