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Comparative study of physicochemical properties of $CoFe_2O_4$/MWCNT nanocomposites

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$CoFe_2O_4$/MWCNT (CFO/MWCNT) nanocomposites have been synthesized using Co-precipitation and solid state method. X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and impedance spectroscopy have been used to determine structural, morphological and dielectric properties of synthesized nanocomposites. The particle size was found to be 3 nm, 3.3 nm and 4 nm for CFO, C-CFO/MWCNT and G-CFO/MWCNT, respectively, using Scherrer formula. Aggregation of nanoparticles has been acquired by SEM analysis. From impedance spectroscopy, it has been observed that the dielectric constant decreases with increase in frequency and dielectric constant and loss both are more for C-CFO/MWCNT nanocomposites than that are for G-CFO/MWCNT nanocomposites.

Keyword: Synergistic effect, Co-precipitation, Nanocomposite, Dielectric constant

1 Introduction

Recently, magnetic nanoparticles loaded with CNTs exhibit great interest in fascinating applications such as adsorption of dye from solution, magnetic resonance imaging, drug delivery system[1,] etc. Ferrites possess attractive magnetic properties due to their spinel structures. The spinel structure can be defined by tetrahedral (A) and octahedral (B) lattice sites in a spinel cube[2]. $CoFe_2O_4$ possesses an inverse spinel type structure which might be responsible for its high cubic magneto-crystalline anisotropy, considerable saturation magnetization and coercivity[3]. Therefore, $CoFe_2O_4$ has been selected as magnetic material for covering CNT. It has been observed from properties of CNTs that its side walls have covalent bonds due to which magnetic nanoparticles can be attached appropriately[4]. Hence, authors have decided to synthesize CFO/MWCNT nanocomposites with two different methods for illustrating comparing dielectric properties as an electrical material.

2 Experimental Details

For synthesizing pure $CoFe_2O_4$, the solution of $CoCl_2.6H_2O$ (Merck, 99.9 % pure) and $FeCl_3.6H_2O$ (S.D. Fine Chem, 99.9 % pure) was prepared separately using distilled water and mixed with the ratio of Co to Fe as 1:2. Another solution of NaOH was prepared as an alkaline precipitant agent. The metal chloride solution was mixed drop by drop in the alkaline solution. Precipitation occurred after 2 h. Further, washed and dried it with 3 h sintering process. Consequently, two routes were followed to synthesize CFO/MWCNT nanocomposites. In first method, MWCNT was introduced during formation of chloride complex in the synthesis of CFO nanoparticles. The precipitation of C-CFO/MWCNT nanocomposites was washed and dried. In second method, MWCNT is grinded with CFO nanoparticles in presence of ethanol. Both particles mixture was grinded until the ethanol evaporated and at last fine G-CFO/MWCNT nanocomposites formed. Further, to design a pellet CFO/MWCNT mixed with epoxy properly.

3 Results and Discussion

3.1 Structural properties

The XRD spectra were shown in Fig. 1. It was observed from XRD spectra that there is no other phase/impurity in CFO nanoparticles (JCPDS No. 00-022-1086). The average sizes calculated using Scherrer formula[5] were found to be 3 nm for pure CFO nanoparticles, 3.3 nm for chemically synthesized C-CFO/MWCNT nanocomposites, and 4 nm for G-CFO/MWCNT nanocomposites, respectively. The extra peaks of CNT were illustrated at 25.4° and 30.2°

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in Fig.1 (b) and (c), which confirms the formation of CFO/MWCNT nanocomposites.

3.2 Morphological properties

Surface morphology of pure CFO, C-CFO/MWCNT and G-CFO/MWCNT nanocomposites has been investigated in Fig. 2 using JEOL-JSM 7500 SEM. The aggregation of particles was obtained from Fig. 2 in all samples.

3.3 EDX analysis

EDX spectrum of $CoFe_2O_4$ nanoparticles is shown in Fig. 3. It is evident from the Fig. 3 that Co:Fe ratio is 1:2 indicating that the stoichiometric proportion is maintained. Spectrum 1 shows the composition of cobalt ferrite absorbed by EDX spectrum. This shows the cobalt ferrite nanoparticles produces by Co-precipitation method is free from impurities.

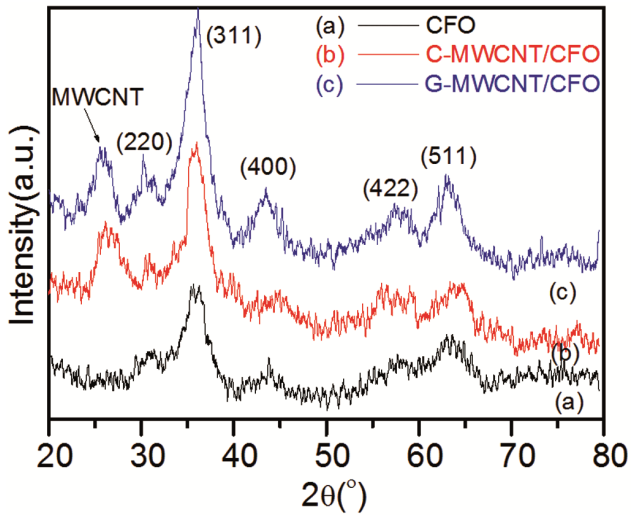

Fig. 1 — XRD patterns of (a) pure CFO nanoparticles (b) C-CFO/MWCNT nanocomposites synthesized by in-situ and (c) G-CFO/MWCNT nanocomposites synthesized by solid state method.

3.4 Dielectric properties

Figure 4 shows the variation of the dielectric constant of CFO/MWCNT nanocomposites as a function of frequency at room temperature. At low frequency region in addition to polarization due to epoxy and CFO/MWCNT, the space charge polarization plays a major role in increasing dielectric constant of composite. The space charge polarization arises from the epoxy-$CoFe_2O_4$/MWCNT interfaces. It is also found that in C-CFO/MWCNT nanocomposites, dielectric constant is higher than G-CFO/MWCNT. This may be attributed to the better interaction among the constituents in C-CFO/MWCNT nanocomposites.

Figure 5 shows the variation of the loss tangent of CFO/MWCNT nanocomposites which is a function of frequency at room temperature. The reactive shell contained epoxy group that plays a dual role in keeping the low dielectric loss of the composites and high dispersivity of the CFO/MWCNT. The values of losses continuously decrease with increase in frequency and after this gradual change at certain point it becomes constant with increasing frequency. This is due to the overlapping of relaxation process

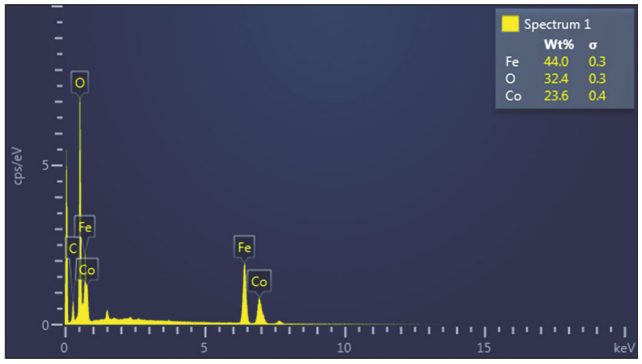

Fig. 3 — Energy dispersive X-ray spectra of $CoFe_2O_4$ nanoparticles.

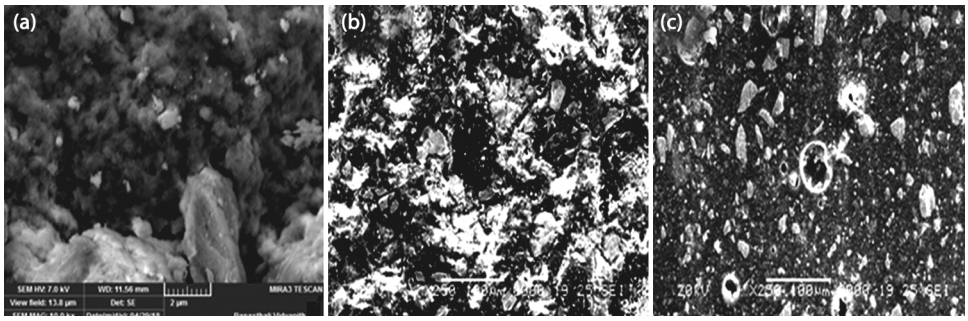

Fig. 2 — SEM images for (a) $CoFe_2O_4$ nanoparticles, (b) C-CFO/MWCNT and (c) G-CFO/MWCNT nanocomposites.

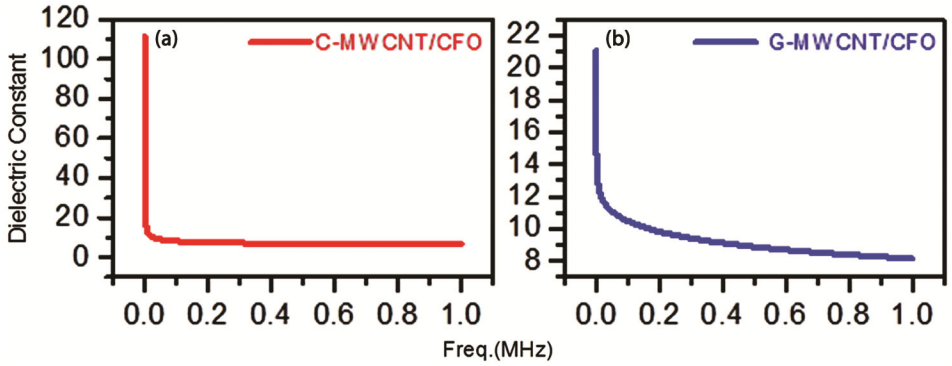

Fig. 4 — Dielectric constant as a function of frequency for (a) C-CFO/MWCNT and (b) G-CFO/MWCNT nanocomposites.

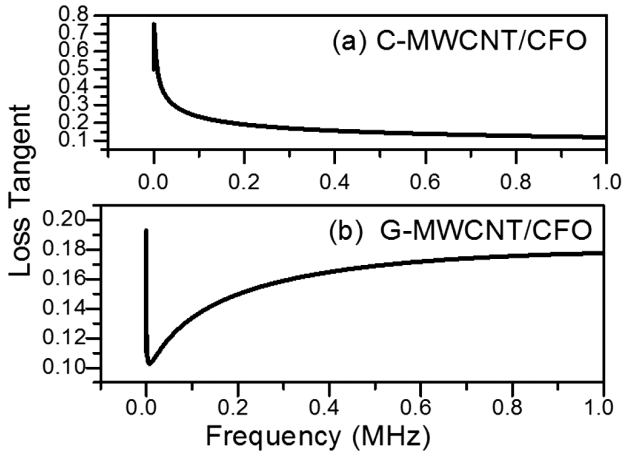

Fig. 5 — Variation of dielectric loss with frequency for (a) C-CFO/MWCNT and (b) G-CFO/MWCNT nanocomposites.

which is attributed to some structural changes that take place in the composite as result of filler addition. Therefore, addition of MWCNT with CFO/epoxy through solid state synthesis prevents the leakage of current in comparison to C-CFO/MWCNT epoxy nanocomposites.

4 Conclusions

The XRD analysis confirms the synthesis of pure CFO, C-CFO/MWCNT and GCFO/MWCNT nanocomposites of crystallite size 3 nm, 3.3 nm and 4 nm, respectively, using co-precipitation and solid state method. The aggregation of particles was also obtained on the surface of the devices. Impedance spectroscopy revealed that the dielectric constant decreases with increase in frequency and dielectric constant and loss both are more for C-CFO/MWCNT nanocomposites than that are for G-CFO/MWCNT nanocomposites. Consequently, G-CFO/MWCNT nanocomposites may prevent leakage current than that of C-CFO/MWCNT nanocomposites.

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