

Alignment of CNTs in CNTs-Fe₃O₄ composites films and their characterization

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Abstract: The Fe₃O₄-CMC (carboxymethyl cellulose sodium) and CNTs- Fe₃O₄-CMC composites have been synthesized by chemical co-precipitation means. The examinations of properties of Fe₃O₄-CMC composites investigated that the dielectric properties of CNTs- Fe₃O₄-CMC composites have been shifted on adding the CNTs to the composites. The arrangement of CNTs inside the composites influencing dielectric properties has likewise been watched. The CNTs inside composites were aligned by applying the magnetic field amid film development. As electronic and dielectric properties of CNTs put together, composites discovered depending with respect to the orientation of CNTs, the impact of the alignment of CNTs on morphological and dielectric properties of composites has been explored. The dielectric studies were performed in 10²-10⁷ Hz frequency range.

Key words - Magnetic materials, Nanostructures, Composites, CNTs.

I. INTRODUCTION

In the field of research and nanotechnology, nano-materials have been assuming a ruling job because of their great mechanical, electrical and substance properties. CNTs with one dimension in nano-run have additionally been an alluring nano-material because of their large surface to volume proportion. Other than the great electrical, magnetic and optical properties [1-4], the CNTs based composites have additionally indicated changing properties in film formation. The composite films with well-dispersed CNTs have shown enhancement in electrical conductivity, mechanical strength and thermal stability [5]. The CNTs based magnetic (Fe₃O₄/Fe₂O₃) composites have broadly acknowledged as the adaptable material required in an electric gadget, magnetic information stockpiling, heterogeneous catalysis, medical diagnosis, high-density data stockpiling media and electromagnetic wave engrossing materials [6-7]. By one way or another, the SWNTs scattered in a dielectric polymer, for example, carboxymethyl cellulose have accompanied differed properties in terahertz frequency range [8]. At times, the outer magnetic field connected to CNTs has changed its electronic and bulk properties [9-10]. The composites films with adjusted CNTs have been considered before in numerous spots [11-12]. In the prior investigation, the CNTs based Fe₃O₄/Fe₂O₃ composites have been prepared with chemical co-precipitation ways [13-14]. Be that as it may, the magnetic composites with adjusted CNTs have been as yet a challenge. A similar way of research, the CNTs based magnetic composites have been synthesized. The uniform dissemination and arrangement of CNTs with CMC (carboxymethyl cellulose sodium, as the dispersive operator) has been accomplished. The dielectric properties of Fe₃O₄-CMC composite have been influenced with arrangements of CNTs. The CNTs-Fe₃O₄-CMC composites films were prepared on dried of a solution comprising of CNTs dispersed in ionic (Fe²⁺/Fe³⁺) and CMC. The CNTs inside the composites film were adjusted in the presence of a magnetic field connected in a vertical orientation with the plane of films. A set of arranged composites films have been recognized as composite A (Fe₃O₄-CMC, without CNTs), composite B (CNTs-Fe₃O₄-CMC, no magnetic field), composite C (CNTs-Fe₃O₄-CMC, field applied within a plane of the film) and composite D (CNTs- Fe₃O₄-CMC, field applied in vertical to film). The thicknesses of every single arranged film were found about 0.50 mm.

II. EXPERIMENTAL

2.1 Synthesis of Fe₃O₄ particles and CNTs

Magnetic Fe₃O₄ particles were synthesized from Aldrich AR grade FeCl₂.4H₂O and FeCl₃ (anhydrous) salts. The aqueous solution of 0.1mmolar FeCl₂.4H₂O and 0.2mmolar FeCl₃ was ready, and 5ml of oleic acid (OA) added to solution at 60°C. The co-precipitation was finished by including the 25% ammonium hydroxide solution at steady mixing at pH~10. In the wake of washing a few times with deionized miliQ water, the precipitate was kept on a hot plate keeping up the temperature at 75°C lastly accomplished the Fe₃O₄ particles. Then again, CNTs were prepared by chemical vapor statement (CVD) utilizing toluene and ferrocene as the forerunners in an inactive argon air at 750°-760°C [15]. The formless carbon-related with as-prepared CNTs was expelled after air oxidation at 450°C for 1hr.

2.2. Synthesis of CNTs-Fe₃O₄-CMC composite

For the amalgamation of the composite, the 60ml watery solution with 1% w/v of CMC was readied. The 0.1g Fe₃O₄ (prepared prior) powder was scattered in the solution. On legitimate ultrasonication, the solution was disseminated in two volumes of 15ml and 45ml. The 15ml solution was treated as a *solution without CNTs*. further, the 0.05g CNTs were added to this 45ml solution. After great ultrasonication, the 45ml solution having scattered CNTs was accomplished. The last solution was additionally disseminated into three 15ml solutions. Hence we had a set of four diverse 15ml solutions (for example three *solutions of 15ml each*

with dispersed CNTs and one 15ml solution without CNTs). Initial, a 15ml solution with scattered CNTs was kept without a magnetic field, while the second 15ml solution with scattered CNTs in the existing 4000G magnetic field (applied along the plane of the film) and third 15ml solution with scattered CNTs was put in a same magnetic field (applied in vertical plane of film). After dried at 50°C, four distinct composites film were accomplished. Three films were with CNTs scattered and one without CNTs. These composites films were demonstrated by composite A ($\text{Fe}_3\text{O}_4\text{-CMC}$, without CNTs), composite B (CNTs- $\text{Fe}_3\text{O}_4\text{-CMC}$, no connected attractive fields), composite C (CNTs- $\text{Fe}_3\text{O}_4\text{-CMC}$, field applied inside the plane of the film) and composite D (CNTs- $\text{Fe}_3\text{O}_4\text{-CMC}$, field applied vertically to film).

III. CHARACTERIZATION

The arrangement of CNTs has been seen in the SEM picture. The existence of CNTs and Fe_3O_4 in composites was affirmed by XRD. The dielectric properties of composites were examined by an impedance analyzer. The topographical examinations of composites were finished by scanning electron microscope instrument Model-LEO 440 with EDS connection Model-OXFORD-LINK ISIS-300 at 30-40 kV. The XRD pattern of composites to affirm the present phase of Fe_3O_4 and CNTs were analyzed by Rigaku make powder X-beam diffractometer. The $\text{CuK}\alpha$ radiation ($\lambda=1.54059 \text{ \AA}$) has been utilized for estimations. The pattern was recorded at $0.02^\circ/\text{s}$ scanning rate in the 2θ range from 20° to 70° at 40 kV, 30mA. The dielectric estimations of composites were examined by impedance analyzer at room temperatures in $10^2\text{-}10^7 \text{ Hz}$ frequency range

IV. RESULTS AND DISCUSSION

4.1 SEM

The SEM of composites B, C and D have appeared in fig.1. The image represented by 'a', 'b' and 'c' in the figure were composite B, composite C (with arrow) and composite D (round spot) separately. The arrow in the image of a composite C investigated that the CNTs were adjusted inside the plane of the composite film, while a round spot of composites D demonstrated that CNTs was adjusted by the field applied vertically to film. In this way, SEM pictures uncovered the uniform dispersion just as the arrangement of CNTs in CNTs- $\text{Fe}_3\text{O}_4\text{-CMC}$ composites films.

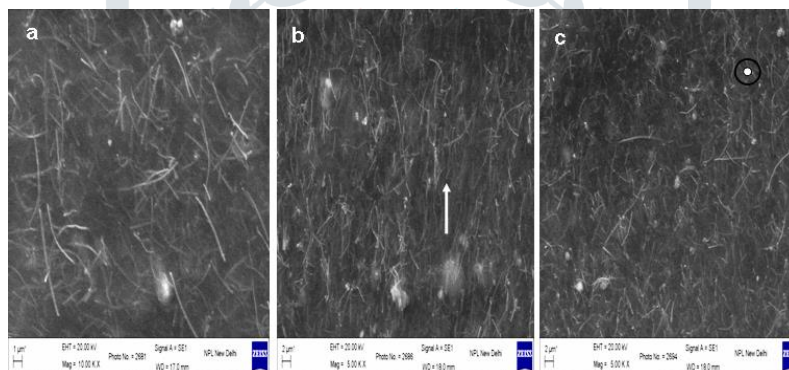


Figure 1. Shows the scanning electron micrograph of composite B, C and D. (where a is Composite B, b is composite C and c is composite D).

3.2 XRD

XRD patterns of CNTs alone and composite B, C and D have appeared in fig.2. The characteristics XRD peaks at $26^\circ(002)$ and $35^\circ(311)$ compared to CNTs and Fe_3O_4 (019-0629) individually. The d-values and crystallite measure (P) compares to (002) peak in XRD pattern of composites determined by $d=\lambda/2\sin\theta$ and $P=k\lambda/\beta\cos\theta$ (Alexander 1980; Chattopadhy et al 2001) are given in table 1. The normal crystallite estimate has fluctuated from 9 to 16nm. In the applied magnetic field to composites have demonstrated a shift in (002) pinnacle and changes d-values are watched. Additionally, the intensity of (002) top is diminished. Every one of these progressions has been seen with the introduction and arrangement of CNTs in composites.

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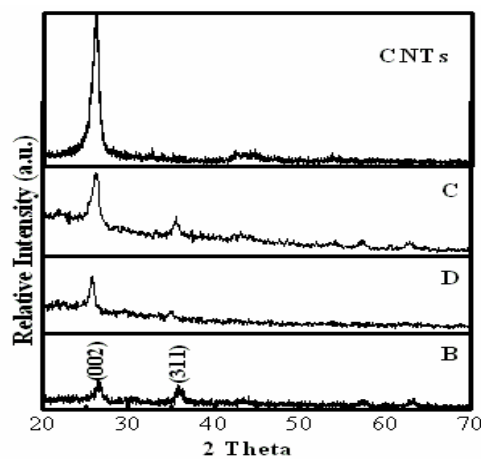


Figure 2. The XRD patterns of CNTs alone and composite *B*, *C* and *D*.

3.3 AC conductivity

The AC conductivity variation of composites with applied frequencies has appeared in fig. 3(a). The electrical estimations have demonstrated that the CNTs based composites (CNTs-Fe₃O₄-CMC composites) have higher conductivity than Fe₃O₄-CMC. At 100 Hz, the composite *D* uncovered the bigger quantities of adjusted CNTs along the flowing current over the contrary face of films. In this way composite *D* showed the bigger conductivity than composites *B* and *C*. At a frequency over 10⁶ Hz, composite *B* have bigger conductivity than composites *C* and *D*, which may be expected to bouncing taken placed set somewhere in the range of Fe⁺² and Fe⁺³ through oxygen [16].

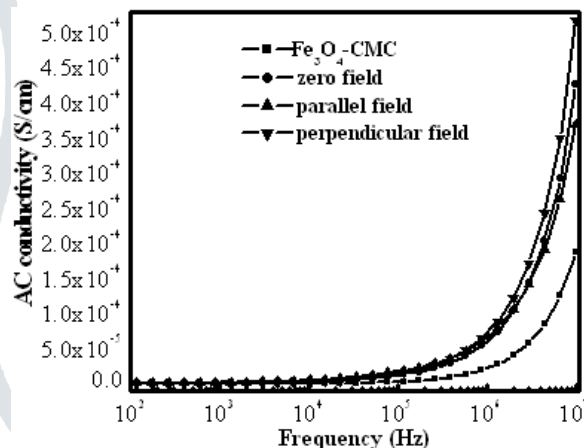


Figure 3(a). The Variation of AC conductivity of Fe₃O₄-CMC (-■-), composite *B* (-●-), composite *C* (-▲-) and composite *D* (-▼-) in 10²-10⁷ Hz.

3.4 Dielectric permittivity (ε')

The variety of dielectric permittivity of composites has appeared in Fig.4 (a). CNTs- Fe₃O₄-CMC composites have shown larger dielectric permittivity than composites Fe₃O₄-CMC. That may be because of imperfections made inside the gap between Fe₃O₄-CNT and CMC polymer and in this manner created prompted dipoles. Along these lines when connected frequency fluctuated, dipoles got arranged and decreased the trapping of space charge polarization [17-20]. That may characterize the character of substantial dielectric permittivity of composites underneath 10⁴ Hz. Further, CNTs- Fe₃O₄-CMC composites had comprised of haphazardly arranged CNTs demonstrated the dielectric permittivity at a higher frequency.

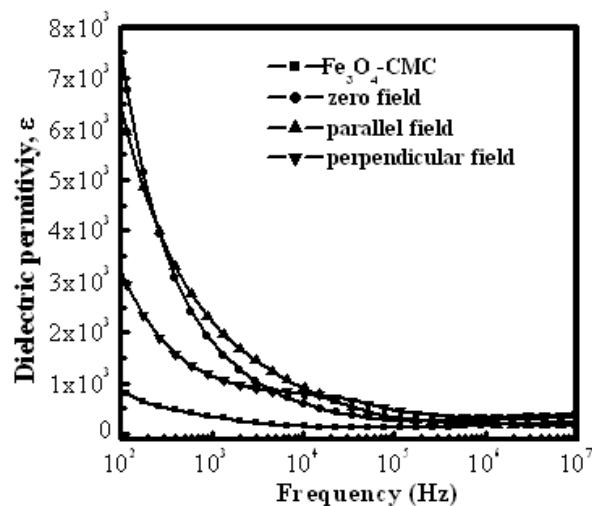


Figure 4(a). The variation of dielectric permittivity of Fe₃O₄-CMC (-■-), composite B (-●-), composite C (-▲-) and composite D (-▼-) in 10²-10⁷ Hz.

3.5 Dielectric Loss (ε'')

The dielectric loss was characterized as the absorption of energy from the alternating field by the materials. The electromagnetic energy, when connecting with conducting material comprising of countless electrons establishes current and for the most part, disseminate energy by absorbing the energy. The variation in dielectric loss of composites appeared in fig. 4(b). The CNTs- Fe₃O₄-CMC composite had a bigger dielectric loss than Fe₃O₄-CMC composite. The dielectric losses of Fe₃O₄-CMC and CNTs- Fe₃O₄-CMC composites have been diminishing with a frequency beneath 10⁴Hz. In the CNTs- Fe₃O₄-CMC composites, the alignment of CNTs have given the immediate directing way along CNTs and subsequently most extreme electromagnetic waves got transmitted resulting the littler dielectric loss of composite D, however in composites B and C, CNTs were haphazardly or vertically situated which caused the vast retention of electromagnetic energy and bigger dielectric loss.

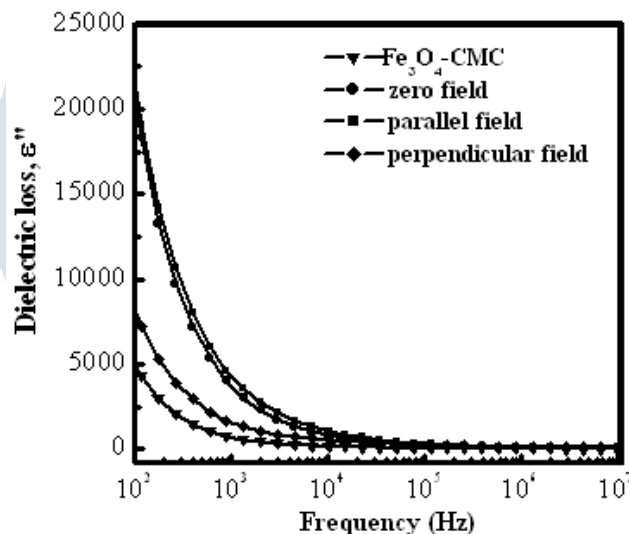


Figure 4(b). The variation of dielectric loss of Fe₃O₄-CMC (-▼-), composite B (-●-), composite (-■-) and composite (-◆-) in 10²-10⁷ Hz

Table 1: The XRD results corresponding to the (002) peaks.

| Samples | d-value (002) Å | Crystallite size(nm) |
|-------------|-----------------|----------------------|
| CNTs | 3.4189 | 9 |
| Composite B | 3.4678 | 11 |
| Composite C | 3.4102 | 9 |
| Composite D | 3.3787 | 4 |

The dielectric permittivity (ε') and dielectric misfortune (ε'') of all composites diminished with expanding frequency range. This conduct of a dielectric may be clarified subjectively by the component of the polarization procedure in ferrite was like that the conduction procedure in the CNTs based composites. Dielectric loss raised because of the limited movement of the charge bearers and this higher dielectric conduct behavior of composite makes them helpful workable for application in conductive paints, battery-powered batteries, sensors, and actuators, and so on.

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