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Characterization of Zinc Nanoferrite Doped HPMC Polymers Using X-Ray Diffraction

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Abstract HPMC Polymer composites were prepared by doping various concentrations of Zinc ferrite nanoparticles using solution casting method. These polymer composites were characterized using X-Ray Diffraction and conductivity measurements. The addition of nanoferrites in the polymer matrix do change the structural and the AC conductivity properties of the film, which is supported by the results obtained and they are discussed briefly in this paper.

Keywords: XRD, Conductivity, Nano Ferrite.

1. INTRODUCTION

The preparation and characterization of nano-sized structures have attracted increasing attention to researchers and scientists in the last decade[1].This is because of nanotechnology which is emerged as the most promising field from the scientific and technological applications point of view[2]. The properties of nanostructured materials are deeply influenced by the chemical composition and microstructure of the materials, which are sensitive to the manufacturing process [3]. Nanoparticles of magnetic ferrites have attracted a great deal of research interest because of their applications in permanent magnets, drug delivery, microwave devices, ferro-fluids and high-density information storage [4,5]. Spinel ferrites have the general formula AFe_2O_4 (where A2+ = Fe, Co, Ni, Zn, Cr, Mn, etc) and the unit cell contains 32 oxygen atoms in cubic close packing with 8 tetrahedral (*T*d) and 16 octahedral (*O*h) occupied sites. By changing the type of divalent cation, it is possible to obtain a wide range of different physical and magnetic properties [6]. Several investigators have focused their attention on Ni-Cu ferrite because copper containing ferrites have interesting magnetic and electrical properties [7, 8]. Ni-Cu ferrites are preferred because of improved magnetic properties, high frequency response and high resistance. In the field of electronics, researchers continue to investigate the design of superstructures comprised of magnetically ordered arrays to enhance the sensitivity in magnetic sensors. The stability of electronic and high density data storage devices made by magnetic nano- composites is limited by their super paramagnetic transition. Thus, while current efforts are aimed at increasing the thermal energy barrier against the magnetic reversal in magnetic nanocomposites, there is strong interest to understand

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the magnetization processes in the super paramagnetic region [9]. Previously mixed spinel ferrites are prepared by conventional methods, which have disadvantages such as high period heating which may result in divalent volatilization and change the stoichiometry [10]. But nowadays numbers of physical and chemical techniques have been developed to prepare nanosized particles but chemical techniques has evolved as most common method due to its advantage of simplicity, energy saving and product homogeneity as compared with physical techniques.

2. EXPERIMENTAL

2.1. Preparation

Nano ferrites were taken from department of physics, Kuvempu University, Shimoga which is synthesized by solution combustion method. They were added to 5% HPMC solution, with different weight proportions of 0.01, 0.02 and 0.03gms which are taken separately in 3 beakers and were stirred for around one hour each and then were poured into casting plates to dry. So that to get doped HPMC film of 0.01, 0.02 and 0.03% along with pure HPMC film for our further comparisons and tests. These films were peeled after two weeks and used for characterizations. The desired sizes of these samples were cut and used for the XRD recording and Conductivity measurements of these doped HPMC polymers.

2.2. XRD Analysis

X-ray diffraction patterns of the polymer films were recorded using Rigaku Denki MiniFlex II Diffractometer available. It is Ni filtered CuKα radiation of wavelength 1.5406Å with a graphite monochromator. The specifications used for the recordings are 30kV and 15mA. The polymer films were scanned in continuous mode in the 2θ range $5^0 - 60^0$ with a scanning speed 5^0/ min and step size of 0.02^0. The XRD patterns obtained for Zinc ferrite nanoparticles and that of HPMC polymer composites are shown in figures 1 & 2 respectively.

2.3. Conductivity Measurements

The AC conductivity studies have been done for these samples using HIOKI LCR Hi-tester 3235-50 model available. For the frequency range of 50Hz to 5MHz and the results obtained was plotted as conductivity versus frequency for all the four samples and it is shown in the Figure 3.

3. RESULTS

3.1. X-Ray Diffraction Studies

From the obtained XRD recordings of the samples, we have determined the microstructural parameters like crystallite size and average crystallite strain by

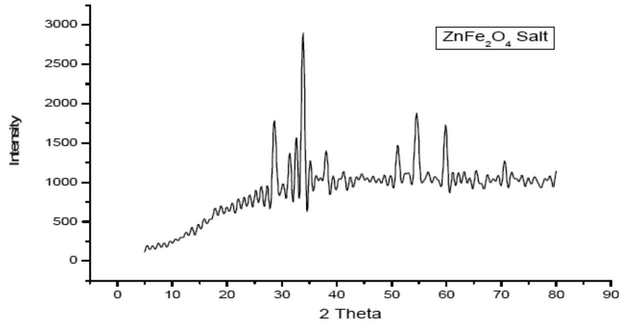

Figure 1: XRD plot of Zinc Ferrite nanoparticles.

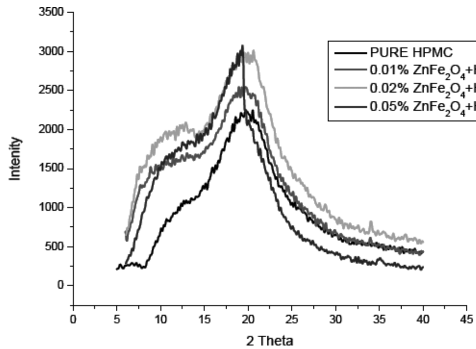

Figure 2: Consolidated XRD plot of HPMC composite films.

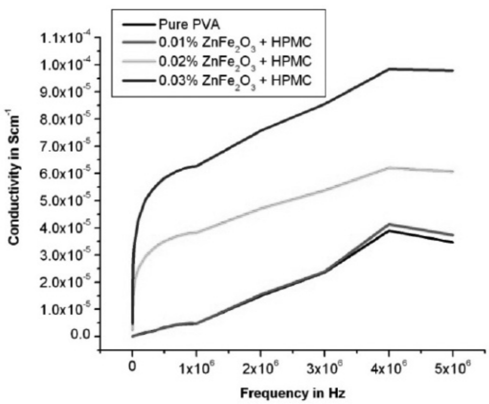

Figure 3: Consolidated conductivity plot.

Table 1: The Microstructural parameters calculated.

Sample	Crystallite Size in Å	Average Strain in %
Pure	11.44	1.90
0.01% $ZnFe_2O_4$ + HPMC	09.25	9.06
0.02% $ZnFe_2O_4$ + HPMC	16.31	7.45
0.03% $ZnFe_2O_4$ + HPMC	33.64	9.03

Williamson-Hall plot. The slope of the W-H plot represents the average strain in the crystal, whereas intercept with the y-axis gives the Crystallite size [11]. The Williamson-Hall relation is given by,

$$\beta\frac{\cos\theta}{\lambda}=\frac{1}{D}+4\,\varepsilon\frac{\sin\theta}{\lambda}. \tag{1}$$

Where 'β' is the full width at half maximum (FWHM) of the peak measured in radians, 'D' is average crystallite size and 'ε' is average lattice strain. The obtained XRD plots and the values of crystallite size and average strain are given in the Figure 1 and Table 1 respectively. From the obtained results it is seen that the crystallite size of the pure HPMC polymer decreases with the addition of nanoferrite initially, this is because of the nanoparticle which effects polymer network and hence the average crystallite size in polymer composite. For higher concentrations, the crystallite size of the nanoparticle dominates and hence increases the average crystallite size of the polymer composites.

3.2. Conductivity test

The conductivity measurements of samples were determined by using HIOKI LCR-Hi tester 3532-50 model available. Our samples were taken in the sample holder provided in the instrument. This instrument gives the transconductance (G) of the sample which is dependent of its thickness and the area. The conductivity (σ) of the sample can be obtained by the relation,

$$\sigma=\frac{\tau\times G}{A}. \tag{2}$$

Where 'τ' is the thickness and 'A' is the area of the sample.

This instrument gives the transconductance values for different frequencies from 50Hz to 5MHz. The obtained transconductance values for various frequencies were evaluated to get the conductivity results for the same corresponding frequencies. From the plot it is seen that the conductivity of the HPMC polymer increase with the increase in its dopant concentration. Though the conductivity for the first concentration does not show any considerable change, a drastic growth in the conductivity is observed for the next two concentrations. The plot of conductivity shown in Figure 3 is evident for this behavior.

CONCLUSIONS

From the obtained results it is evident that the crystallite sizes and the conductivity of the Zinc ferrite nanocomposites increases with the concentration. Moreover the conductivity varies with the crystallite size, proving that the enhancement in the conductance of the film is due to the presence of nanoparticle. Hence for the first concentration the change in the conductance is very less as the crystallite size is small. Therefore it is said from the results that the conductivity of these polymer composites changes proportionally with the crystallite size. It is pertinent to note that the conductivity in these polymers vary between 4.62×10^{-8} and 1.19×10^{-5} S/m with concentration of Zinc Ferrite which is an important observation.

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