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Synthesis and Thermo Acoustical Dynamics of PMMA/Fe2O3 Nanocomposites

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ABSTRACT
Inorganic metallic nanoparticle's incorporation into polymer matrices allows the modification of physico-chemical properties and its specific implementation. This work put forth the conventional co-precipitation method for synthesis of polymethyl methacrylate enriched in ferric oxide at nanoscale. The synthesized matrix is then structurally determined using XRD. The magnetic behavior is analyzed by VSM. The molecular interaction study is carried out using ultrasonic pulse echo technique. The specific concentration of metal ion in polymer matrix with fixed geometry has optimum magnetic property.
Keywords : polymer matrix, nanocluster, XRD, PMMA, VSM

I. INTRODUCTION

The mixing of polymer and nanoparticles is opening pathway for engineering flexible composite that exhibit advantageous magnetic, electrical, optical and mechanical properties. As part of this renewed interest in nanocomposite, researchers began seeking new strategies to engineer materials that combine the desirable properties of nanoparticles and polymers for the formation of polymer composite materials. The particle must integrate in a way leading to isolated, well-dispersed primary nanoparticles inside the matrix. There is a need for establishing processing techniques that are effective on the nanoscale yet are applicable to macroscopic processing. There have been several attempts for the synthesis of polymer composite material that can be classified under two major categories: as physical and chemical methods [1-5].

The composites are defined as materials that consist of two or more chemically and physically different phases separated by a distinct interface. The different systems are combined to achieve a system with more useful structural or functional properties not attainable by any of their constituents. The composites are wonder materials becoming an essential part of today's materials due to the advantages such as low weight, corrosion resistance, high fatigue strength, and faster assembly. They are extensively used as materials in making aircraft structures, electronic packaging, medical equipment, and space vehicle to home building [6-8]. The predominant useful materials used in our day-to-day life are wood, concrete, ceramics, and so on are surprisingly, the most important polymeric composites [1-3,6]. The composites are combinations of materials differing in composition, where the individual constituents retain their separate identities.

These separate constituents act together to give the necessary mechanical strength or stiffness to the composite part. Composite material is a material composed of two or more distinct phases [matrix phase and dispersed phase] and having bulk properties significantly different from those of any of the constituents. The matrix phase is primary phase having a continuous character. Matrix is usually more ductile and less hard phase. It holds the dispersed phase and shares a load with it. Dispersed [reinforcing] phase is embedded in the matrix in a discontinuous form. This secondary phase is called the dispersed phase. The dispersed phase is usually stronger than the matrix, therefore, it is sometimes called reinforcing phase. In structural applications composites have the following characteristics:

They have superior mechanical properties and in some cases uniquely different from the properties of their constituents

1. Among the many unique attributes of non materials especially noteworthy are their large surface to volume ratios and outstanding mechanical properties.
2. They generally consist of two or more physically distinct and mechanically separable materials.
3. They are made by mixing of the separate materials in such a way as to achieve controlled and uniform dispersion of the constituents

These properties offer venues for exciting areas of research as well as for technological innovations. Thus an important use of non materials is in reinforcing polymer matrices taking advantages of the ultra-high stiffness and hardness exhibited by them. Recent research has shown that small additions [upto ~ 5 wt%] of certain nano materials such as carbon nano tubes enhance the mechanical properties markedly, sometimes by as much as 100%. The precise mechanism responsible for this dramatic enhancement is not entirely understood. It is generally believed that molecular level interactions between the nano materials and polymer matrices play a major role. The large interfaces are available for such interactions clearly hold the key for the dramatic enhancement of mechanical properties [9-12]. The electrical properties constitute one of the most convenient and sensitive method of studying the polymer structure. They are affected not only by the structure and nature of dopant but also by the doping procedure [13].

PMMA is an excellent host for functional particles[2,4-8]. The various types of metal oxide fillers such as $Bi4Ti_3O_4$, SiO_2, TiO_2, Nb_2O_5, and Ta_2O_5/SiO_2 have been incorporated into polyacrylates to modify the optical properties of these polymers. For *e.g.,* the refractive indices of PMMA matrix can be increased or decreased by addition of SiO_2, Al_2O_3, or ZrO_2 filler particles[5-8,14-15]. A noteworthy property of nanoparticles is their ability to become dispersible in liquids by appropriate modification of their surface. This property makes nanophase materials very attractive since it helps their manipulation. Such as in thin film formation or homogeneous dispersion into matrices. The nano-composite polymers have been designed in a similar manner by incorporation of the nanoparticles in a polymer matrix alters, as expected the mechanical, thermal and the other characteristics of the polymer. The usual method for the preparation of such polymer composites is the dispersion of nanoparticles into a melted polymer or dissolving them in a solvent polymer [16-17]. Hence research in the field of such polymers is mainly at some suitable modifications of existing polymers. The properties of the polymer composites can be improved by involvement of organic materials and inorganic oxides or salts of different metals *viz.* SnO_2 [18-19], CeO_2 [20-21], TiO_2 [7, 22], fly ash composites [23-24], Fe_3O_4 [1-4], ZrO_2 [25] etc. Therefore PMMA and its composite with Fe_2O_3 nanoparticles were studied in this work. These were characterized for structural properties using XRD. The magnetic behavior is analyzed by VSM. The molecular interaction study is carried out using

ultrasonic pulse echo technique. The specific concentration of metal ion in polymer matrix with fixed geometry has optimum magnetic property.

II. METHODS AND MATERIAL

2.1 Synthesis

Synthesis of Fe_2O_3 NPs

The Fe_2O_3 NPs were synthesized by co-precipitation method. In a typical procedure 0.2 M aqueous solution of $FeCl_3.6H_2O$ was heated at 60 ^{0}C under mild stirring. A solution of 2 M sodium hydroxide was added drop wise into the ferric chloride solution until pH reaches 8. At this point red precipitate appeared in the solution. The slurry was heated at 100 ^{0}C for 5 h in a vacuum oven. The dried iron hydroxide nanoparticles were then finally heated at 400 ^{0}C in conventional muffle furnace for 4 h to obtain brick red Fe_2O_3 NPs.

Synthesis of PMMA/ Fe_2O_3 nanocomposites NCs

For the synthesis of binary composite of PMMA/ Fe_2O_3, particular amounts of Fe_2O_3 NPs were added, respectively, to 20 mL methyl methacrylate monomer (MMA) in weight fractions of 1 wt%, 2 wt% , 3 wt%, 4 wt% and 5 wt% and sonicated for 12 h in the presence of azobisisobutyronitrile (AIBN) as initiator. The reaction mixture was converted into a thick paste and dissolved in acetone to form uniform sheets of composites.

2.2 Characterization

The structural properties of PMMA and its composite with Fe_2O_3 nanoparticles were identified by X-ray powder diffraction (XRD) with a X'Pert PRO advanced diffractometer using Cu (K_α) radiation (λ=1.5406 Å). Ultrasonic measurements were performed at 4MHz by pulse echo method. The ultrasonic velocity (U) was determined with the experimental density and viscosity at various temperature. The magnetic measurements were made by using vibrating sample magnetometer (VSM) at room temperature.

III. RESULTS AND DISCUSSION

3.1 Structural Characterization

The XRD spectrum for polymer composite of PMMA with Fe_2O_3 is given in Fig 1. It is noticed the broader intensity peak occurred at lower angle of 2θ say 14^0, But the sharp peaks are occurred at 2θ angles of 33.14^0, 35.59^0, 40.83^0, 49.42^0, 54.04^0, 57.39^0, 62.38^0, 63.93^0, 71.93^0 and 72.25^0 correspond to the (1 0 4), (1 1 0), (1 1 3), (0 2 4), (1 1 6), (1 2 2), (2 1 4), (3 0 0), (1 0 10), (1 1 9), (0 2 10) were well matched with JCPDS No.79 -892810.

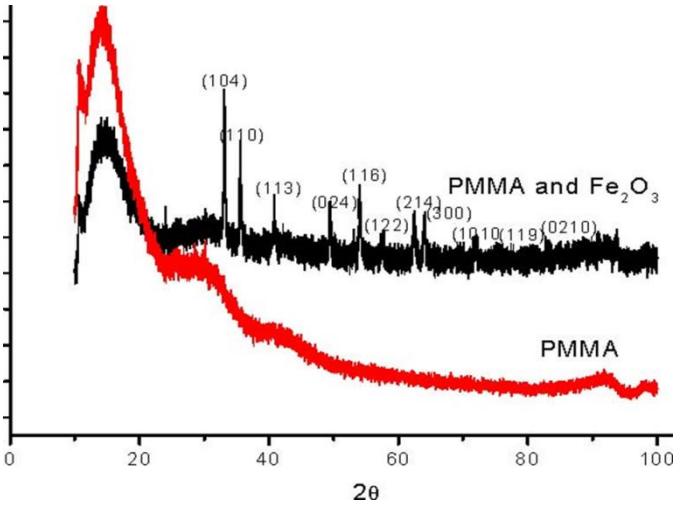

Fig. 1. The XRD Spectra of the PMMA and PMMA/Fe2O3 nanocomposites[1]

The particle size of the samples has been calculated by employing the Scherres equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where, θ is the angle between the incident and diffracted beams (degree), β is the full with half maximum (rad.), D is the particle size of the sample (nm) and λ is the wavelength of the X-ray. The average grain size of the prepared PMMA/ Fe_2O_3 nanocomposite (3 wt%) was found to be 28 nm.

3.2 Acoustical Characterization

Ultrasonic wave produces molecular vibration in the samples [26]. The acoustic study provides intermolecular interactions present in nanocomposites. The temperature dependence

ultrasonic velocity of PMMA/ Fe_2O_3 is elaborated here. For the interpretation of PMMA matrix with the Fe_2O_3 nanoparticles, ultrasonic velocity is the most significant propriety. The variation of ultrasonic velocity with Fe_2O_3 NPs conc (wt%) and temperature are plotted as shown in fig 2a and 2b.

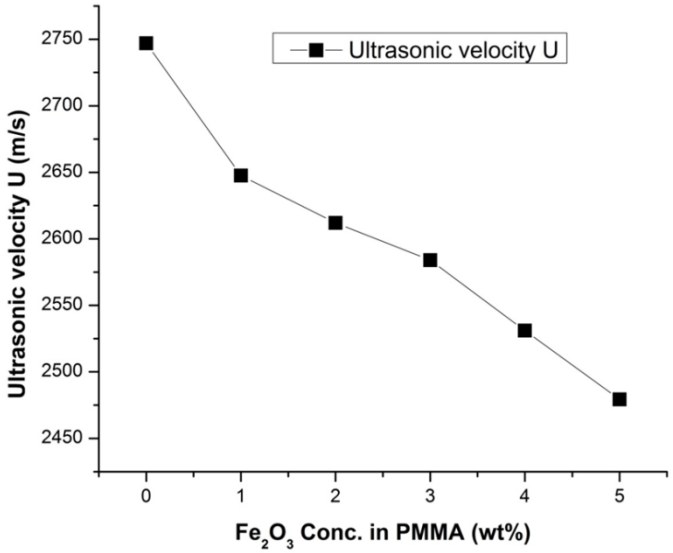

Fig.2a Variation of Ultrasonic velocity with Fe2O3 NP conc. in PMMA matrix

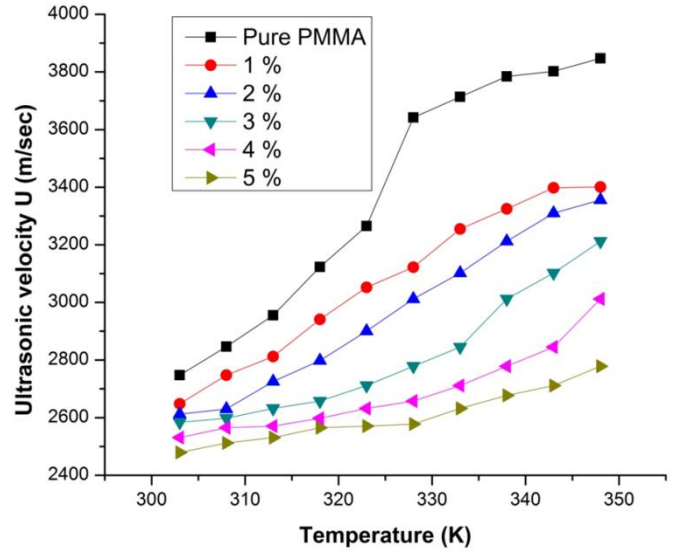

Fig.2b Temperature-dependent ultrasonic velocity of PMMA/ Fe_2O_3 matrix

From the Fig. 2a it is observed that the ultrasonic velocity was sensitive to the concentration of the nanoparticles PMMA matrix. The ultrasonic velocity of the PMMA composite decreases with the increase of nanoparticles concentration. This may be due to agglomeration of Fe_2O_3 NP in PMMA matrix. This decrease in ultrasonic velocity with an increase of concentration is recognized by metal oxide nanoparticles – PMMA interactions, and it further confirms the dominance of intramolecular interactions over the inter-molecular interactions[27-29]. Fig. 2b also reveals the changes in ultrasonic velocity with respect to temperature. From the observed results, it is clear that the ultrasonic velocity of PMMA and its Fe_2O_3 composites also increases with increasing the temperature. it is due to nonaqueous behavior of the liquids i. e. as the temperature of fluid increases, the average speed of the molecules rises with temperature and hence ultrasonic velocity increases. it also indicates the weakening of the intermolecular forces[1].

3.3 Magnetic measurements

The magnetic properties of the synthesized nanocomposites were analyzed using a Magnetometer (VSM) at room temperature.

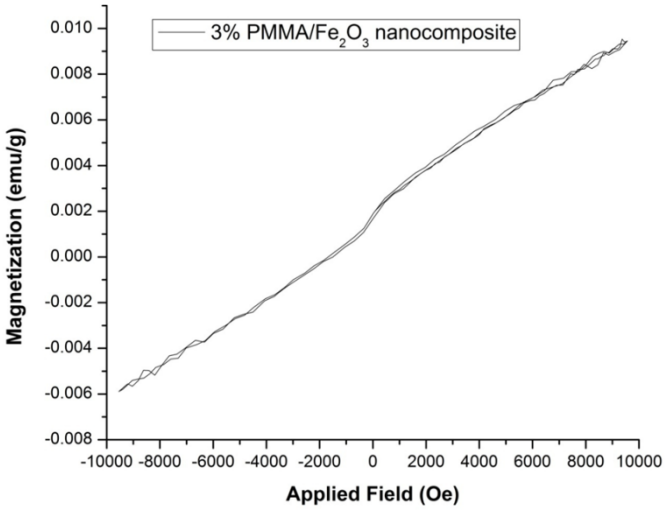

Fig.3 Hysteresis curve of PMMA/Fe_2O_3 nanocomposite (3 wt%)

Figure 3 shows the M-H curves of the prepared PMMA/ Fe_2O_3 nanocomposite (3 wt%). The saturation magnetization (Ms) values is found to be (14 emu/g) which is much greater than PMMA matrix (2 emu/g). This leads to the application of PMMA/ Fe_2O_3 nanocomposite for the synthesis of soft magnetic

materials with reasonable flexibility and moderate dielectric properties[1]

IV.CONCLUSION

The Fe_2O_3 nanoparticles of orthorhombic structure were synthesized successfully by co-precipitation method and were confirmed by XRD. PMMA and PMMA/ Fe_2O_3 nanocomposites of (1%, 2%, 3%, 4% and 5%) were characterized further for their structural, acoustical and magnetic properties. The acoustical studies were carried out at different temperatures ranging from 303 K to 348 K of the nanocomposites. . The ultrasonic velocity of the PMMA composite decreases with the increase of Fe_2O_3 nanoparticles concentration due to agglomeration whereas it increases with temperature due to intermolecular interaction. The saturation magnetization (Ms) of PMMA/ Fe_2O_3 (3 wt%) is found to be much greater than that of PMMA matrix makes it potential candidate for the fabrication of soft magnetic materials with reasonable flexibility.

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