

## PREPARATION AND CHARACTERIZATION OF $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> DISPERSED POLYSTYRENE NANOCOMPOSITE FILM

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### ABSTRACT

Nanosized  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> particles showing supermagnetic behavior have been widely studied in recent years for various applications.  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> dispersed nanocomposites possessing wide applications have attracted many researchers to study their different nature when they are dispersed into polymers matrix.  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles dispersed polystyrene nanocomposite has many applications in medical field such as drug delivery, magnetic gels, in cancer treatment, and also in memory devices, supermagnetic materials and many more. In the present study we prepared the nano sized  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> dispersed polystyrene nanocomposite (FPSN) film, in the weight ratio 1%. These nanocomposites were characterized to understand the molecular structure through FT-IR spectroscopy, magnetic property employing B-H curve tracer, thermal study employing TGA, DSC and the morphology through Scanning Electron Microscopy (SEM) technique. Through the mentioned synthetic procedure and characterizations we obtained a well dispersed  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles polystyrene nanocomposite film having chemical homogeneity.

**KEYWORDS:**  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> Nanoparticles, polystyrene, Nanocomposites film, Magnetic Hysteresis, thermal study.

### I. INTRODUCTION

A wide research activity has occurred in producing polymer composites by incorporating inorganic nanoparticles [1]. The unique properties of nanoparticles are to quantum confinement or surface effect become operative on the nanoscale [2, 3]. Polymer nanocomposite material represents a new alternative to conventionally used nanoscopic inorganic material filled polymer. Because of their nanosize filler dispersion nanocomposite exhibit markedly improved properties when compared to the pure polymer or their traditional composite [4]. These include increased modulus and strength, outstanding barrier. Properties that improved heat resistance and decreased flammability. Researchers have attempted to enhance the desired properties of polymer nanocomposites and to extend their utility by reinforcing them with nanoparticles [5, 6].

Nanocomposite materials based on nanoscale inorganic materials have been of great interest to researchers due to their applications [7]. Metal oxide dispersed polymer nanocomposites have been extensively studied since they exhibit interesting properties with many application such as quantum electronics devices, magnetic materials, sensors, capacitors, conducting paints and rechargeable batteries [8-11]. In view of the said applications and vast utility of the above, aim of this work is to prepare  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles dispersed polystyrene polymer nanocomposite (FPSN) film through polymerization method. The composite film has been thoroughly studied using different characterizations like spectral, thermal, magnetic and morphological aspects.

### II. EXPERIMENTAL

#### 2.1. Preparation of $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> dispersed polystyrene nanocomposite (FPSN) film

Commercially polystyrene was procured and  $\gamma\text{-Fe}_2\text{O}_3$  was synthesized by combustion method as reported earlier [12]. Spin coating method is adapted for the preparation of nanocomposite film which is given as follows: A known weight (1.0 gram) of polystyrene is dissolved in benzene and stirred well to make polymer gel. A known quantity of  $\gamma\text{-Fe}_2\text{O}_3$  (1%) is sonicated (Sonic Vibra cell) for 0.5 hour in separate container. Both the solutions were mixed in a rotary evaporator constantly maintained at  $40^\circ\text{C}$  till the solvent was evaporated. Few drops of the solution was put on glass slide and kept on the spin coating unit (SCU 2007 A) at 800 RPM for about 15 min. A transparent film formation takes place. This film is detached from glass slide by using polar solvent such as distilled water. A uniform  $\gamma\text{-Fe}_2\text{O}_3$  dispersed polystyrene nanocomposite (FPSN) film was obtained. The Nanocomposites film was then characterized for structural morphology, thermal and magnetic behavior.

## 2.2. Characterization

FTIR Studies was undertaken employing Thermo Fisher ATR Nicolet model using diamond (iS5) in the range  $4000\text{-}400\text{cm}^{-1}$ . Thermal Studies were carried out employing STA PT1600 Thermal Analyzer from Linseis under nitrogen atmosphere with a heating rate of  $10^\circ\text{C}/\text{minute}$  at a flow rate of 100 ml/min and temperature up to  $600^\circ\text{C}$ . Magnetic studies are carried out by hysteresis loop tracer at room temperature. The Scanning Electron Microscopy (SEM) images of the sample were obtained on a Leica 440 Cambridge stereoscan operated at 20 kV.

## III. RESULT AND DISCUSSION

### 3.1. FTIR studies

FTIR spectroscopy of  $\gamma\text{-Fe}_2\text{O}_3$  dispersed polystyrene (FPSN) film is shown in figure 1. The spectrum of the nanocomposite film indicates that major peaks are associated with styrene ( $\nu\text{-OH}$ ), strong stretching band is observed at  $3025\text{cm}^{-1}$  to  $3082\text{cm}^{-1}$ . The  $\nu\text{-CH}$  alkyl stretching at  $2923\text{cm}^{-1}$ , ( $\nu\text{-C=O}$  stretch at  $1745\text{cm}^{-1}$ ,  $1802\text{cm}^{-1}$ ,  $1810\text{cm}^{-1}$ ,  $1942$ ,  $\nu\text{-CH}$  bending at  $1372\text{cm}^{-1}$ ,  $\nu\text{-C-O}$  stretch at  $1154\text{cm}^{-1}$ ,  $\nu\text{-C-O}$  stretch at  $1181\text{cm}^{-1}$ ,  $\nu\text{-CH}$  stretching at  $965\text{cm}^{-1}$ ,  $756\text{cm}^{-1}$ , the peaks below  $697\text{cm}^{-1}$  peaks are associated to H- type interaction between  $\gamma\text{-Fe}_2\text{O}_3$  and polymer, the peaks from  $451\text{cm}^{-1}$  to  $539\text{cm}^{-1}$  are the two shift peaks for ferrite sample [13], along with a small red shift was observed. The instrumental limitation did not allow the two peaks to be clearly resolved. The peak position of polymer and ferrite clearly indicate a composite formation and that the composites FPSN film is chemically homogeneous, transparent and well dispersed. This observation is also collaborated from SEM images (discussed later).

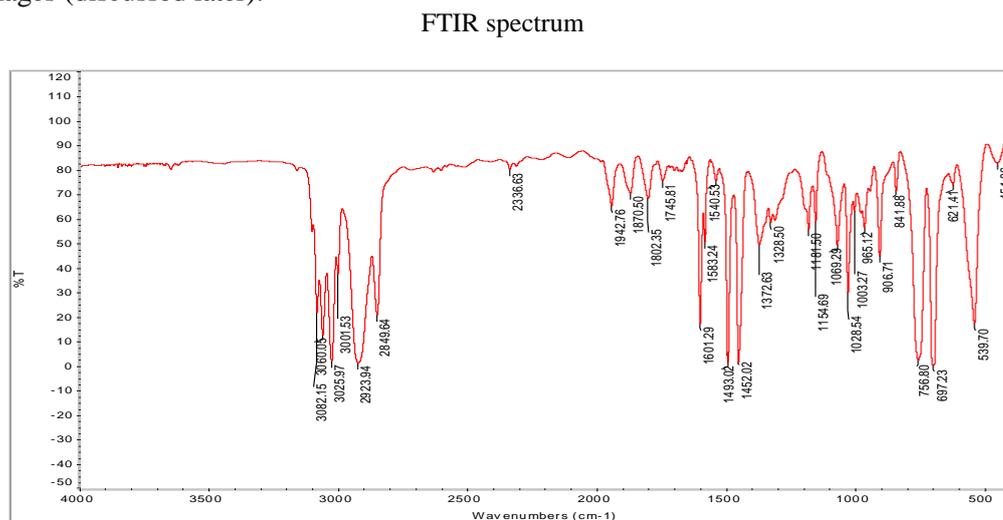


Fig.1 FTIR spectrum of FPSN film

### 3.2. Thermal analysis

The thermal decomposition of FPSN film is shown in Figure 2. (a & b). A two-step weight loss is shown on the thermal trace (Figure 2. (a)). The first weight loss of 4.39 % from 70°C to 100°C is due to the loss of adsorbed water molecule present in the film. The enhancement of thermal stability of the polystyrene to higher temperature is a clear consideration for the composite formation. A second weight loss of 72.19% ranging from 240°C to 430°C indicates the weight loss due to decomposition of FPSN film. This weight loss is a slow process and is a multistep one. The DSC trace is shown in figure 2(b) Two endothermic peaks with two shoulders for the main endothermic peaks are observed at 130°C, the second peak at 430°C are due to decomposition of the FPSN film which is a slow having a multistep process. The DSC trace shown in figure 2(b) collaborates with TGA trace shown in figure 2(a).

Thermal Graphs

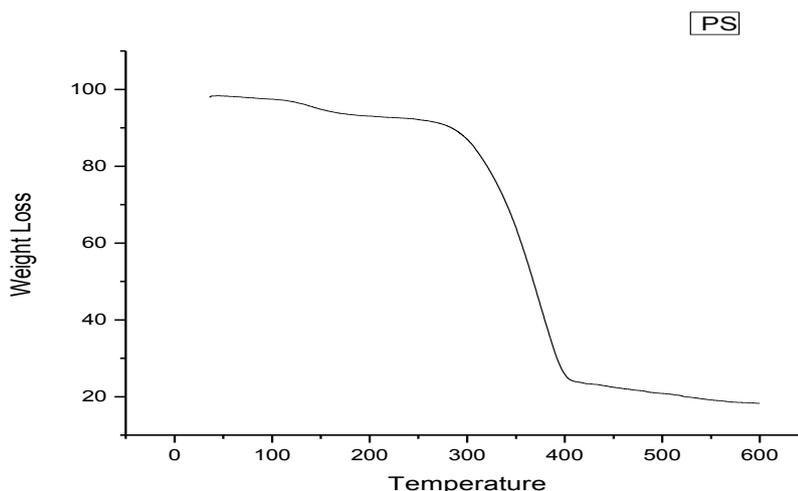


Fig. 2(a) Showing the TGA graph of FPSN film

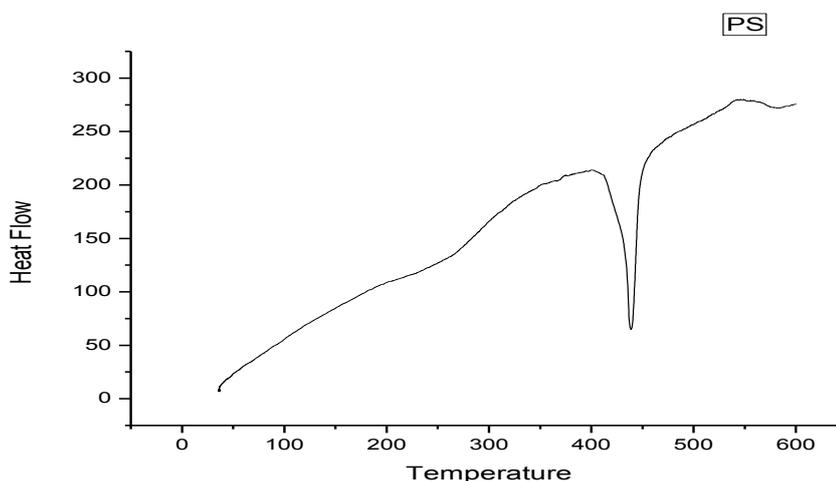


Fig 2(b) showing the DSC graph of FPSN film

### 3.3. Magnetic property

The M (H) curve for the FPSN film sample at room temperature is given in the table 1. The values of saturation magnetization ( $M_s$ ), remanence magnetization ( $M_r$ ) and coercivity ( $H_c$ ) are 0.2 emu/g, 0.1 emu/g, and 68.18 Oe respectively. These values of pure  $\gamma$ - $Fe_2O_3$  are found to be 11.0 emu/g, 3emu/g, and 165.0 Oe respectively, for our samples reported [13]. An drastic decrease to a very low magnetic

values indicates the superparamagnetic behavior of FPSN film. The superparamagnetic behavior is essential for high density magnetic recording.

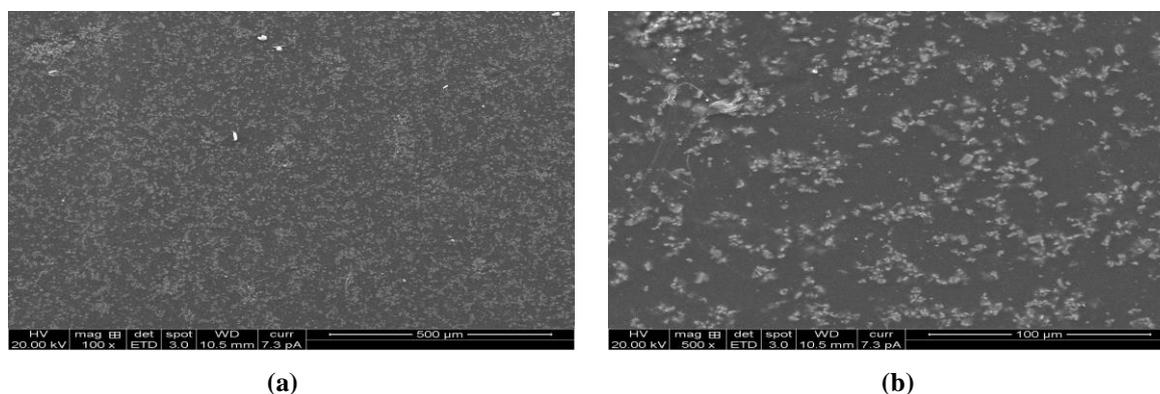
**Table.1.** Shows the hysteresis loop values of pure  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> and FPSN film

Sample	Saturation Magnetization(M <sub>s</sub> ) emu/g	remanence magnetization (M <sub>r</sub> ) emu/g	Coercivity (H <sub>c</sub> ) Oe
FPSN film	0.3	0.2	70.0
$\gamma$ -Fe <sub>2</sub> O <sub>3</sub>	11	3.0	165

### 3.4. Scanning Electron Micrograph

The images of FPSN film are shown in figure 3. (a,b). With low and high magnification. Figure 3(a) shows a well and uniform distribution of FPSN film. From this image it was not possible to calculate exact particle size of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> though the sample observed to possess nano dimension, i.e, 25-35 nms from powder X-Ray diffraction (PXRD) studies[12].

Scanning Electron Micrographs



**Fig.3. (a-b)** SEM images of FPSN film.

## IV. CONCLUSIONS

The Preparation and characterization FPSN film showed superparamagnetic behavior. The FTIR spectroscopy for the film showed several vibrational bands at various wave numbers. Several new bands disappeared in the IR spectra of the composite film due to formation of complexes by the addition of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> to the PS matrix. SEM images of the polystyrene  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> composites showed the particle morphology of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> self-assembled rod like particles embedded in spherical packets of the polymer matrix. The thermal (TGA/DSC) study showed an increase in thermal stability of the composite. The FPSN film was highly transparent, best suitable for processing and fabrication because of their flexibility. Use of such films as super capacitor and other medical devices is envisioned.

## V. FUTURE WORK

Looking into the processibility and chemical homogeneity of the magnetic polystyrene (FPSN) film obtained envisaged that these films will be of applications as supercapacitor and chemical sensors. The work in this direction is in progress.

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