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SYNTHESIS AND CHARACTERIZATION OF POLYSTYRENE COATED FUNCTIONALIZED γ -Fe_2O_3 NANOPARTICLES

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ABSTRACT

γ-Fe2O3 nanoparticles showing supermagnetic behaviour have been widely studied in recent years for various applications. Polymer coated functionalized γ-Fe2O3 nanoparticles have wide applications which attracted many researchers to study their different nature when they are supported with polymers. Functionalized γ-Fe2O3 nanoparticles has many applications in medical field such as MRI contrast, drug delivery, hyperthermia, magnetic gels, in cancer treatment and also used in memory devices, supermagnetic materials etc. In the present study we synthesized the polystyrene coated functionalized γ-Fe2O3 nanoparticles (PSCFNPs) in the weight ratio through grafting onto method. These nanoparticles were characterized and studied its magnetic property employing B-H loop tracer, molecular structure through FT-IR spectroscopy, thermal study employing TGA, DSC and morphology through Scanning Electron Microscopy (SEM) & transmission electron microscopy (TEM) techniques. Through the mentioned characterization techniques we obtained the formation of fine polystyrene coated functionalized γ-Fe2O3 nanoparticles. The medical applications of the nanoparticles with tailored surfaces by biocompatible polymers are envisioned.

Keywords: Coated, γ-Fe2O3, Functionalized, Nanoparticle, Polystyrene, SEM, TEM.

Contribution/ Originality

The present studies contribute for the investigation of cancerous cells, MRI and drug delivery system in the field of medical science and also for the low cost methodology for cancer treatments.

1. INTRODUCTION

The change in physical and chemical properties by reduction in size of nanoparticles has attracted many researchers [1]. Organic – inorganic nanocomposites are intensively studied for their applications in catalysis, sensing, electrical, optical, magnetic [2] and also in medical applications such as magnetic resonance imaging (MRI), drug delivery, hyperthermia and other

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biomedicines [3, 4]. Polymer supported nanosized metal oxides prepared by direct mixing in situ or by template method shows unique properties given by the combination of two components [5]. The use of magnetic nanocomposites as adsorbents in pollutant removal offers the easy separation through magnetic field [6].Tailoring the surface of nanoparticles can achieve desirable physical and chemical properties [7]. Hybrid nanoparticles composed of core shell can be formed by either through grafting onto and grafting from methods [8]. In grafting-from method the polymer reacts from the monolayer of a polymerization initiator on the surface of nanoparticles. Whereas in grafting – onto method the chain ending functionality of the polymer binds with appropriately modified nanoparticles surfaces [9]. The aim of this work to synthesized PS coated functionalized γ-Fe_2O_3 nanoparticles through grafting onto method. The synthesized nanoparticles were studied employing characterization techniques such as magnetic, spectroscopic, thermal, and morphological aspects.

2. EXPERIMENTAL PROCEDURE

2.1. Synthesis of Polystyrene Coated Functionalized γ-Fe_2O_3 Nanoparticles (PSCFNPs)

Polystyrene was commercially procured and γ-Fe_2O_3 was synthesized by combustion method as reported earlier [10]. Grafting onto method is adopted for the synthesis of PSCFNPs as follows: known weight (1.0 gram) of polystyrene is dissolved in benzene and stirred well to make polymer gel. A known quantity of γ-Fe_2O_3 (10%) is sonicated (Sonic Vibra cell) for 6 hours in separate container. Both the solutions were mixed in a rotary evaporator constantly maintained at 40^0 C till the solvent to form a gel. The gel was dried in hot air oven at 60^0 C for 1 hour, black brown powder obtained. The Powder was washed with distilled water and dried. The synthesized nanoparticles were studied employing characterization techniques.

2.2. Characterization

Magnetic studies are carried out employing B-H loop tracer at room temperature. FTIR Study carried out by Thermo Fisher ATR Nicolet diamond (iS5) the ranges from 4000-400cm^{-1} .Thermal Studies were carried out employing STA PT1600 Thermal Analyzer from Linseis under nitrogen atmosphere with a heating rate of 10^oC/minute at a flow rate of 100 ml/min and temperature up to 500^o C. The Scanning Electron Microscopy (SEM) images of the sample were obtained on a Leica 440 Cambridge steroscan operated at 20 kV. Transmission electron microscopy (TEM) obtained by Technai 20 Philip transmission electron microscope operated at 190 keV.

3. RESULTS AND DISCUSSIONS

3.1. Magnetic Property

The M (H) curve for the PSCFNPs at room temperature is given in the table 1. The values of saturation magnetization (M_s), remanent magnetization (M_r.) and coercivity (H_c) are 4.3emu/g, 0.9 emu/g and 69.0 Oe respectively. These values of pure γ-Fe_2O_3 are found to be11.0 emu/g,

3emu/g, and 165.0 Oe respectively, for our samples reported earlier [11]. The decrease in the low magnetic values indicates the superparamagnetic behavior of PSCFNPs.

Table-1. Shows the B-H loop values of pure γ-Fe_2O_3 and PSCFNPs.

Sample	Saturation Magnetization (M_s) (emu/g)	remanent magnetization (M_r) (emu/g)	Coercivity (H_c) (Oe)
PSCFNPs	4.3	0.9	69.0
γ-Fe_2O_3	11	3.0	165

3.2. FTIR Study

The figure 1 shows the FTIR spectrum of PSCFNPs. The spectrum indicates that major peaks are associated with styrene (υ_{-OH}), strong stretching band is observed at 3102 cm^{-1}o 3082cm^{-1}.The υ_{-CH} alkyl stretching at 290 cm^{-1}, ($\upsilon_{-C=O}$ stretch at 1745cm^{-1} 1802cm^{-1}, 1810 cm^{-1}, 1942, υ_{-CH} bending at 1372cm^{-1} , υ_{-C-O} stretch at 1154cm^{-1} , υ_{-C-O} stretch at 1181cm^{-1}, υ_{-CH} stretching at 965cm^{-1},756cm^{-1},the peaks below 697cm^{-1} peaks are associated to H type interaction between γ-Fe_2O_3 and polymer , the peaks from 451cm^{-1} to 539cm^{-1} are the two shift peaks for ferrite sample [11], along with a small red shift is observed. The limitations of the instrument denied the clarity in the vision of two peaks. The peaks position of polymer and ferrite clearly indicate a coating / functionalization this observation also collaborates with SEM image.

FTIR spectrum

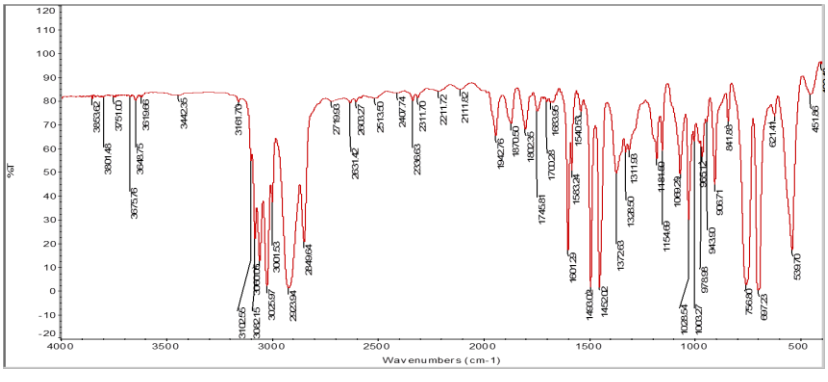

Fig-1. Shows FTIR spectrum of PSCFNPs

3.3. Thermal Analysis

The thermal decomposition of PSCFNPs 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 5.19 % from 69°C to 100°C is due to the loss of adsorbed water molecule present in the compound. The enhancement of thermal stability of the polystyrene to higher temperature is a clear consideration for the coating formation. A second weight loss of 73.99% ranging from 239°C to 439°C indicates the weight loss due to decomposition of PSCFNPs. This weight loss is a slow process and is a multistep one. The DSC traces shown in figure 2(b), two endothermic peaks are observed at

139°C, and 439°C are due to decomposition of PSCFNPs which are slow and multistep process. The DSC trace shown in figure 2(b) collaborates with TGA trace shown in figure 2(a).

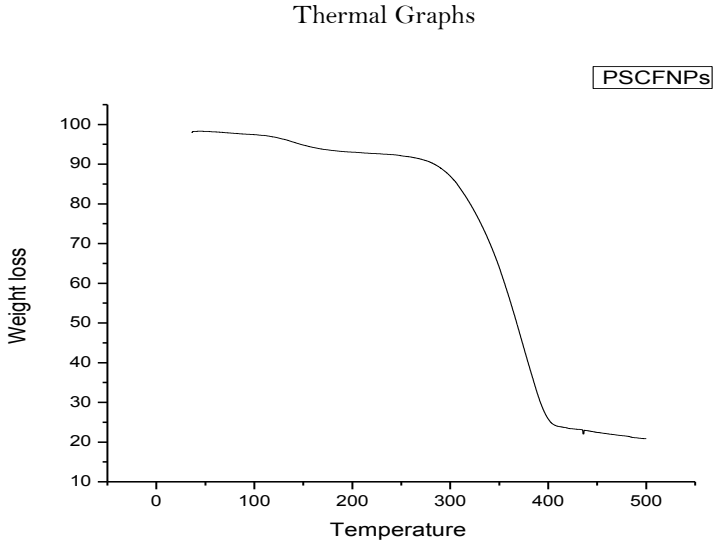

Fig-2(a). shows the TGA graph of PSCFNPs.

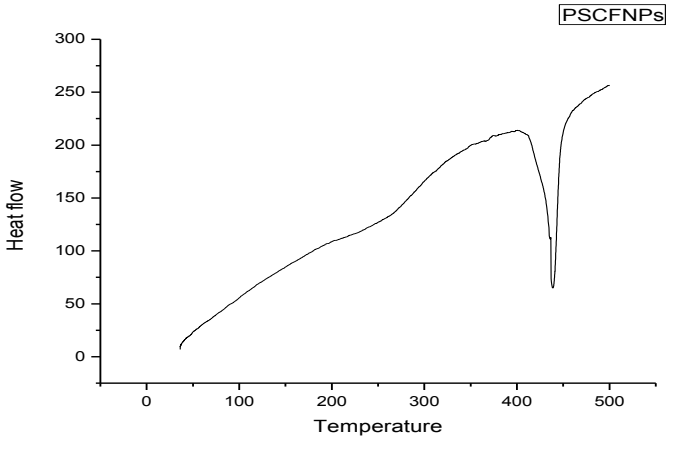

Fig-2(b). shows the DSC graph of PSCFNPs

3.4. Scanning Electron Microscopy

The figure 3 shows SEM image of PSCFNPs which shows the formation of globular aggregates in micron dimensions. The aggregates are almost similar throughout uniform particles dimensions. However in the higher magnification some smooth surface solid block observed it may be due to much closed packing of heterogeneous γ-Fe_2O_3 coated with PS.

Scanning Electron Micrographs

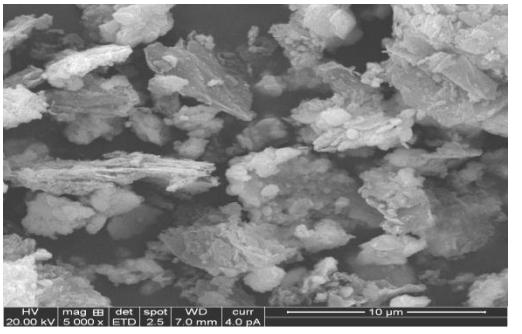

Fig-3. SEM image of PSCFNPs

3.5. Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) obtained by Technai 20 Philip transmission electron microscope operated at 190 keV, at the range of 200nm. The TEM image shows γ-Fe_2O_3 nanoparticles remained uniformly dispersed within the polystyrene matrix during grafting onto method. The granular shaped PS coated nanoparticles clearly visualized and confirms the functionalization of γ-Fe_2O_3 nanoparticles.

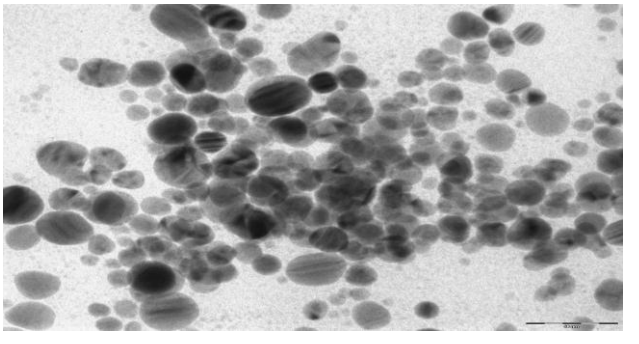

Fig-4. Shows TEM image of PSCFNPs

4. CONCLUSIONS

The method to synthesize PSCFNPs developed and optimized. Synthesized PSCFNPs showed a superparamagnetic behavior. The FTIR spectroscopy showed several vibration bands at various wave numbers and confirms the PS coating formation on γ-Fe_2O_3 nanoparticles. The thermal (TGA/DSC) study shows an increase thermal stability of the PSCFNPs as compare to the pure polymer. SEM image showed the PS coated γ-Fe_2O_3 nanoparticles form globular aggregates and some smooth surface solid block observed due to much closed packing of γ-Fe_2O_3 nanoparticles in the PS matrix, which also collaborates with TEM that we obtained PS coated functionalized γ-Fe_2O_3 nanoparticles.

5. ACKNOWLEDGMENT

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