SYNTHESIS AND CHARACTERIZATION OF POLYVINYL ALCOHOL (PVA) COATED FUNTIONALIZED γ-Fe2O3 NANOPARTICALS

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ABSTRACT

Superparamagnetic iron oxide nanoparticles have been intensively studied in the several years for various applications. The functionalized magnetic nanoparticles have many applications in site – specific drug delivery, MRI, magnetic gels, cancer treatment and other biomedical applications. In the present study we synthesized polyvinyl alcohol (PVA) coated functionalized γ-Fe2O3 nanoparticles (PCFNPs) in the weight ratio (10%). Further characterizations were carried out as the molecular structure through FT-IR spectroscopy, magnetic property by B-H loop tracer, thermal study by TGA, DSC and surface morphology by SEM. Through the mentioned characterizations we developed the well polymer coated functionalized γ-Fe2O3 nanoparticles

KEYWORDS: *Polyvinyl alcohol (PVA), Functionalized, Superparamagnetic, γ-Fe2O3 Nanoparticles, FT-IR, Thermal studies.*

I. INTRODUCTION

Nanoparticles with variable size showing diverse physical and chemical properties [1]. The γ-Fe_2O_3 Nanoparticles due to its high saturation magnetization, magnetic susceptibility are promising candidates for the applications such as electrical, optical, sensor, memory devices, contrast agent, ferro-fluids, magnetic resonance imaging (MRI), catalysis and biological separations [2,3]. General approach to tailor the surface property of the particles for many applications can be achieved by coating/functionalization [4]. The control of surface functionality is the key for controlling the nanoparticles interaction with biological species, dispersion in organic media, self-assembly and compatibility with polymeric materials [5]. There are two basic methods to synthesize functionalized nanoparticles 1) Grafting-from, in which the polymer react from a monolayer of a polymerization initiator on the nanoparticles surface. 2) Grafting-onto, in which the chain ends functionality of the polymer reacts with appropriately modified nanoparticles surface [6, 7]. The Polyvinyl alcohol (PVA) is biocompatible in nature due to its hydrophilic, biodegradability and a hydroxyl group that can react with many functional groups [8]. In views of the above said applications the aim of this work to synthesize PVA coated functionalized γ-Fe_2O_3 nanoparticles through grafting onto method. The functionalized γ-Fe_2O_3 nanoparticles have been thoroughly studied using characterization technique such as spectral, thermal, magnetic and morphological aspects.

II. EXPERIMENTAL

2.1. Synthesis of polyvinyl alcohol (PVA) coated functionalized γ-Fe_2O_3 nanoparticles (PCFNPs).

Polyvinyl alcohol (PVA) with molecular weight Approx. 1,25,000 was obtained commercially with AR grade, and γ-Fe_2O_3 was synthesized by combustion method as reported earlier [9]. Grafting onto

method adapted for the synthesis of PCFNPs as follows. A known weight (1.0) gram of PVA dissolved in distilled water and stir well for polymer gel. A known quantity of γ-Fe_2O_3 (10%) sonicated (Sonic Vibra cell) for 6 hours in separate container. The both solution is mixed in a rotary evaporated which was constantly maintained at 80°-90° C till the solvent becomes gel form. Later the gel was dried in a hot air oven 60^0 C for 1 hour, black brownish PCFNPs were obtained. The PCFNPs was then characterized for spectroscopy, 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 4000-400cm^{-1}. Thermal studies were carried out employing STA PT1600 Thermal Analyzer from Linseis under nitrogen atmosphere with a heating rate of 10°C/minute at a flow rate of 100 ml/min and temperature up to 600° C. Magnetic studies are carried out by B-H loop tracer at room temperature. The Scanning Electron Microscopy (SEM) images of the sample were obtained on a Leica 440 Cambridge steroscan operated at 20 kV.

III. RESULT AND DISCUSSION

3.1. FTIR studies

FTIR spectroscopy of PCFNPs is shown on Figure1. The spectrum of PCFNPs indicates that the major peaks are associated with alcohol (υ_{-OH}) strong stretching band observed at 3307cm^{-1}. υ_{-CH} alkyl stretching at 2936cm^{-1}, $\upsilon_{-C=O}$ stretch at 1732cm^{-1}, υ_{-CH} bending at 1373cm^{-1} , υ_{-C-O} stretch at 1242cm^{-1}, υ_{-C-O} stretch at 1086cm^{-1}, the below 604 peaks are assign H type interaction between γ-Fe_2O_3 and PVA. The peaks from 400cm^{-1} to 563cm^{-1} are the two specific peaks for ferrite sample along with a small red shift is observed [10]. The instrumental limitation did not allow the two peaks to be clearly shown. The peaks position of υ_{-OH} and ferrite clearly indicates the PVA coated functionalization of γ-Fe_2O_3 nanoparticles. This observation also collaborates from SEM images.

FTIR spectra

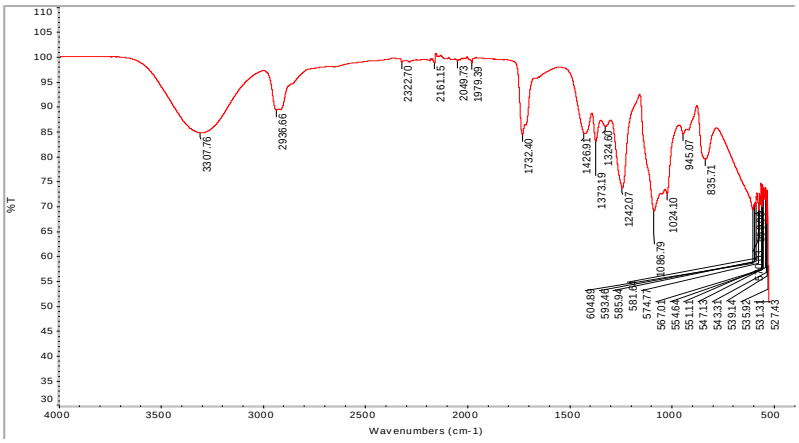


Fig.1 FTIR spectrum of PCFNPs.

3.2. Thermal analysis

The thermal decomposition of PCFNPs shown in figure 2 (a & b) shows two step weight loss. First weight loss is slow process and second was multi-step. Figure 2 (a) shows the first weight loss of 9.09 % from 69°C to 129°C, due to the loss of adsorbed water molecule present in PCFNPs. The enhancement of thermal stability of PVA to higher temperature showed clear indication for complete functionalization. A second weight loss of 39.21% ranging from 229°C to 489°C indicates the weight loss due to decomposition of PCFNPs. The figure 2(b) shows two endothermic peaks with two shoulders at 229°C and the second peak at 489°C. The DSC traces shown in figure 2(b) collaborates to TGA traces shown in figure 2(a).

Thermal Graphs

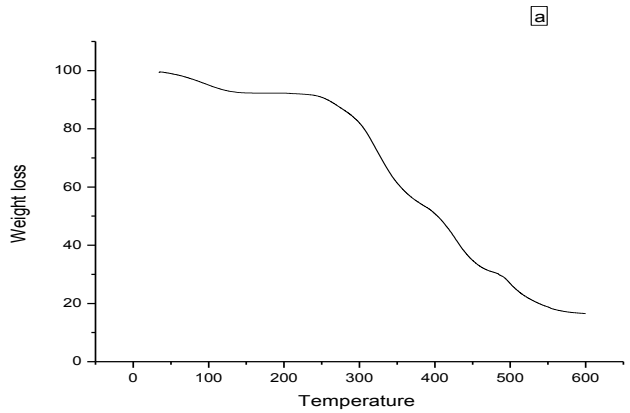


Fig. 2 (a) Showing the TGA graph of PCFNPs.

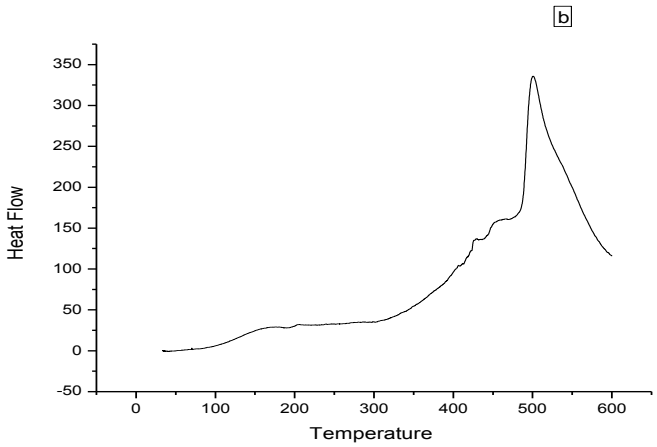


Fig 2(b) showing the DSC graph of PCFNPs.

3.3. Magnetic property

The magnetic hysteresis (MH) curve for the PCFNPs at room temperature given in the table 1. The values of saturation magnetization (M_s), remanence magnetization (M_r.) and coercivity (H_c) were 4.9 emu/g, 1.9 emu/g, and 90.0 Oe respectively. These values of pure γ-Fe_2O_3 were found to be11.0 emu/g, 3emu/g, and 165.0 Oe respectively, as reported [10]. The decreased low magnetic values indicate the superparamagnetic behavior of PCFNPs. The superparamagnetic behavior is essential for high density magnetic recording.

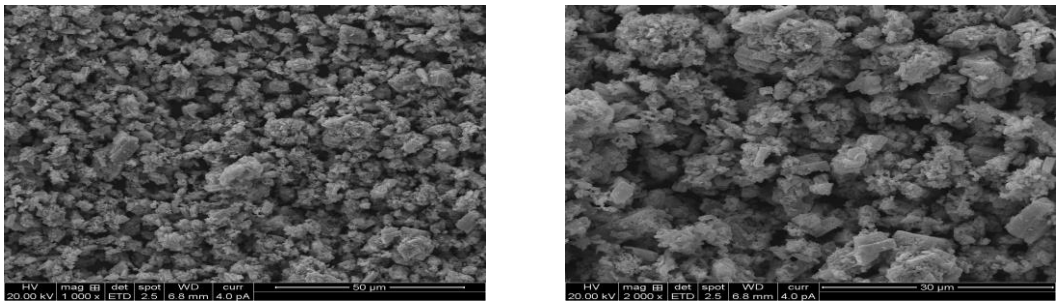
Table.1. Shows the hysteresis loop values of pure γ-Fe_2O_3 and PVA coated γ-Fe_2O_3 nanoparticles.

Sample	**Saturation Magnetization(M_s) emu/g**	**remanence magnetization (M_r.) emu/g**	**Coercivity (H_c) Oe**
PCFNPs	4.9	1.9	90.0
γ-Fe_2O_3	11	3.0	165

3.4. Scanning Electron Micrograph (SEM)

The SEM images shown in figure 3 (a & b), with low and high magnification, respectively. Figure 3(a) shows the PVA coated γ-Fe_2O_3 nanoparticles form globular aggregates of micro dimensions. These aggregates are almost similar throughout uniform particles dimensional shape. However in the higher magnification some smooth surface solid block observed it may be due to much closed packing of γ-Fe_2O_3 nanoparticles in the PVA.

Scanning Electron Micrographs



(a) (b)

Fig.3. (a-b) SEM images of PCFNPs.

IV. FUTURE DIRECTIONS

Looking into the processibility of functionalized nanoparticles and their thermal stability, Superparamagnetic PCFNPs can be further studied for the medical and electronic applications.

V. CONCLUSION

The PCFNPs showed superparamagnetic behavior. The FTIR spectroscopy showed several vibrational bands at various wave numbers. Several bands disappeared in the IR spectra of the coating due to formation of complexes. SEM images showed the PVA coated γ-Fe_2O_3 nanoparticles form globular aggregates are almost similar throughout uniform particles dimension and in the higher magnification some smooth surface solid block observed due to much closed packing of γ-Fe_2O_3 nanoparticles in the PVA. The thermal (TGA/DSC) studies showed an increase in thermal stability of the PCFNPs as compare to the pure polymer. As per the characterization we have obtained the functionalized γ-Fe_2O_3 nanoparticles with PVA.

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REFERENCES

[1] Jonsson T.,Mattsson J., Nordblad P.,Svedlindh P,(1997) " Energy barrier distribution of a nano- sized magnetic particle system"
[2] Arunkumar L, Vijayanand H, Basavaraja S, Balaji S.D.,Venkataraman A. (2007) "Microwave-assisted route for synthesis of nanosized metal oxides " Sci. Technol. Adv. Mater. V 8: pp.484-493
[3] Caruso F. (2001) **"**Nanoengineering of Particle Surfaces." Adv. Mater. V.13: pp. 11-22
[4] Bahadur D., Giri J.,Bibhuti B. Nanak., Sriharsha T, Pradhan P., Prasad N.K., Barick K.C and Ambashta R.D.(2005) " Processing, properties and some novel applications of magnetic nanoparticles." Parama J. Phy.V.65: pp.663-679
[5] Zhongbing Huang and Fangqiong Tang (2004) "Preparation, structure and magnetic properties of polystyrene coated by Fe_3O_4 nanoparticles" J. Coll.and Inter. Sci.V.275: pp. 142-147.
[6]Stefanie Gravano M., Randy Dumas, Kai Liu., Timothy Patten E. (2005) J. Poly. Sci, V.43:pp. 3675-3688.
[7] Gianina Dodi, Doina Hritcu, Gabriela Lisa, marcel I. Popa. " Core –Shell magnetic chitosan particles functionalized by grafting: Synthesis and characterization." (2012) Chem. Eng. J. V.203:pp.130-140.

[8] Chastellain M., Patri. A., Hofmann H. "Particle size investigation of multistep synthesis of PVA coated superparamagnetic nanoparticles." (2004) J.coll. and Inter. Sci.V.278:pp.353-360.
[9] Sharanabasava V.Ganachari, Venugopal K. Joshi, Ravishankar Bhat, Raghunandan Deshpande, Basavaraja Salimath, N. V. Srinivasa Rao, A. Venkataraman,(2011) "Large scale synthesis and characterization of γ-Fe_2O_3". Int. J. Sci. V.1, pp77 -79
[10] Mahesh D. Bedre, Raghunandan D, Basavaraja S, Balaji D.S, Arunkumar Lagashetty and Venkataraman A. (2010) "Preparation and characterization of magnetic Fe_2O_3" J.Metal. &
Mater. Sci., V.52 pp 209-214.

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