



Original Article

# Synthesis characterization and morphological study of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ Core-Shell Nanoparticles

S. P. Gawali<sup>1</sup> D.V. Mane<sup>2</sup>

<sup>1</sup>School of Science, Yashwantrao Chavan Maharashtra open University, Nashik, Maharashtra – India, <sup>2</sup>Department of Chemistry, Sundarrao More Arts, Commerce and Science College, Poladpur, Raigad, India

<sup>2</sup>Former Regional Director, YCMOU Nashik India. Department of Chemistry, Shri Chhatrapati Shivaji College Omerga, Dharashiv India

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Correspondence Address:  
S. P. Gawali  
School of Science, Yashwantrao  
Chavan Maharashtra open  
University, Nashik, Maharashtra  
– India, <sup>2</sup>Department of  
Chemistry, Sundarrao More Arts,  
Commerce and Science College,  
Poladpur, Raigad, India  
Email: [spgawali1986@gmail.com](mailto:spgawali1986@gmail.com)

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## Abstract

This study investigates the creation and analysis of core-shell nanoparticles composed of magnetite and silica ( $\text{Fe}_3\text{O}_4@\text{SiO}_2$ ) utilizing a modified Stöber process that incorporates microwave-assisted heating.  $\text{Fe}_3\text{O}_4$  nanoparticles were encapsulated with amorphous silica shells through the controlled hydrolysis and condensation of sodium metasilicate under optimized pH conditions. X-ray Diffraction confirmed the cubic spinel structure of magnetite with characteristic peaks at  $2\theta = 30.1^\circ, 35.5^\circ, 43.1^\circ, 57.0^\circ$ , and  $62.6^\circ$ , yielding an average crystallite size of 19.2 nm. FTIR spectroscopy revealed Si-O stretching peaks at  $1022\text{--}1034\text{ cm}^{-1}$ , confirming successful silica coating. TEM analysis demonstrated clear core-shell morphology with particle sizes increasing from 147.5 nm ( $\text{Fe}_3\text{O}_4$ ) to 157.7 nm ( $\text{Fe}_3\text{O}_4@\text{SiO}_2$ ). VSM measurements showed superparamagnetic behavior with enhanced saturation magnetization (41 emu/g for  $\text{Fe}_3\text{O}_4@\text{SiO}_2$  Vs. 28 emu/g for  $\text{Fe}_3\text{O}_4$ ). The nanoparticles exhibited excellent colloidal stability (zeta potential:  $-23.5$  to  $-38.4\text{ mV}$ ), making them promising candidates for biomedical applications, drug delivery, magnetic separation, and catalysis.

**Keywords:** Magnetite nanoparticles, Core-shell nanostructures, Silica coating, Nanocomposites

## Introduction

In recent years, magnetic nanoparticles have attracted considerable interest because of their distinctive physicochemical characteristics and diverse applications in fields such as biomedicine, environmental cleanup, catalysis, and materials science [1]. Among various magnetic nanomaterials, magnetite ( $\text{Fe}_3\text{O}_4$ ) nanoparticles have emerged as particularly promising candidates owing to their biocompatibility, superparamagnetic behavior, high saturation magnetization, and ease of synthesis [2].  $\text{Fe}_3\text{O}_4$  nanoparticles exhibit characteristics that make them exceptionally well-suited for a range of applications, including improving contrast in MRI scans, facilitating targeted drug delivery, treating hyperthermia, aiding in bioseparation, and eliminating environmental contaminants. Nevertheless, despite these advantageous traits, bare  $\text{Fe}_3\text{O}_4$  nanoparticles face significant obstacles that restrict their practical applications [3,4]. The high surface energy of magnetic nanoparticles promotes particle aggregation, leading to loss of colloidal stability and diminished functionality. Additionally, magnetite is susceptible to oxidation in air and acidic environments, converting to maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ) or dissolving entirely, which compromises its magnetic properties and biocompatibility. Furthermore, the absence of functional surface groups limits the ability to attach biomolecules or targeting ligands for advanced applications [5, 6].

## Synthesis of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ Core-Shell Nanoparticles:

The composite was synthesized by a slight modification of a method that involves microwave heating of a suspension of the as-prepared nanoparticles in distilled water at a higher temperature. Sodium silicate solution was then added dropwise with continuous stirring and heating. The pH of this mixture was adjusted to the appropriate range, and the reaction mixture was further heated in the microwave oven for a few minutes.

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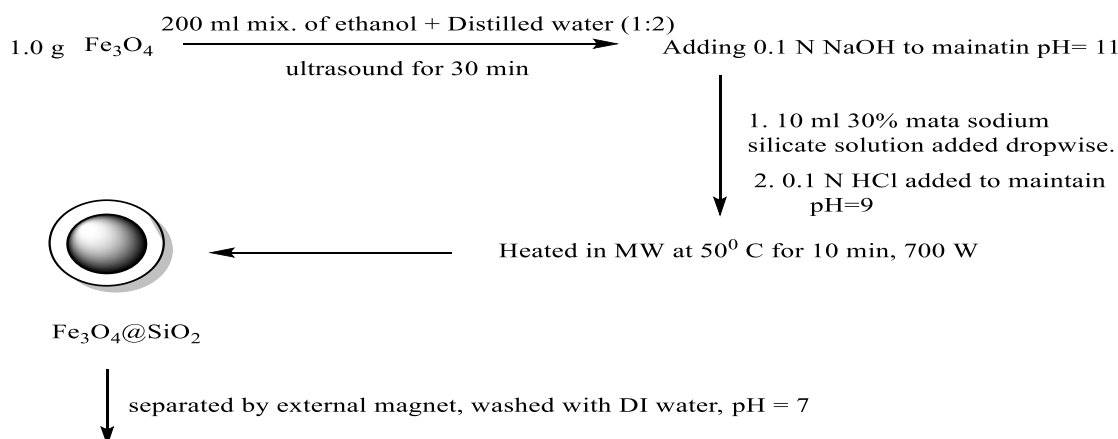
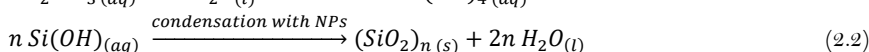
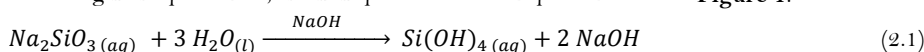
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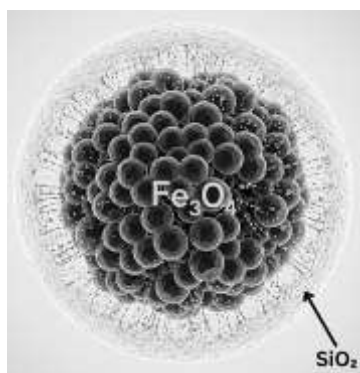
### Synthesis of $\text{Fe}_3\text{O}_4@\text{SiO}_2$ Core-Shell Nanoparticles:

The composite was synthesized by a slight modification of a method that involves microwave heating of a suspension of the as-prepared nanoparticles in distilled water at a higher temperature. Sodium silicate solution was then added dropwise with continuous stirring and heating. The pH of this mixture was adjusted to the appropriate range, and the reaction mixture was further heated in the microwave oven for a few minutes. The silica coating is crucial for preventing particle aggregation and enhancing the stability of the magnetic nanoparticles. The synthesis of magnetite@silica ( $\text{Fe}_3\text{O}_4@\text{SiO}_2$ ) core-shell nanoparticles was achieved through a modified Stöber method, where a uniform silica ( $\text{SiO}_2$ ) layer is deposited onto the surface of pre-synthesized magnetic nanoparticles as shown in **Figure 1**. To begin the procedure, the nanoparticles were dispersed



Dried at 80<sup>0</sup> C at hot air oven.

**Figure 1** A schematic diagram depicting the synthesis of core-shell nanoparticles using microwave assistance, highlighting the reaction conditions and sequence of processing steps.



**Figure 2** An illustrative representation of nanoparticles featuring a core-shell design, with a magnetic core encased by an outer shell.

The resulting mixture was stirred for one hour and then subjected to microwave irradiation to enhance the density and uniformity of the shell. After overnight aging, the core-shell product was repeatedly washed with deionised water until a neutral pH was achieved, followed by an ethanol wash. The nanoparticles were effectively separated from the solution using an external magnet and dried

in a solution composed of deionized water and ethanol in a 1:2 ratio. This was followed by a 30-minute ultrasonication process to achieve a uniform suspension and to prevent the particles from clumping together. To achieve the required basic conditions for the hydrolysis and condensation of the silica precursor, the pH of the mixture was increased to approximately 11 by adding 0.1 M sodium hydroxide (NaOH). Following pH adjustment, a 30% sodium metasilicate solution, which is the source of silica, is added dropwise to the suspension [7, 8]. The high pH catalyzes the hydrolysis of metasilicate ions ( $\text{SiO}_3^{2-}$ ) to form silicic acid ( $\text{Si}(\text{OH})_4$ ). The pH was then carefully lowered to 9 with 1.0 M hydrochloric acid (HCl) to promote the condensation of silicic acid molecules onto the surface of the  $\text{Fe}_3\text{O}_4$  nanoparticles, forming a coherent shell, as shown in **Figure 1**.

overnight in a hot air oven at 80<sup>0</sup>C to produce a fine powder.

### Results and Discussion:

#### X-ray Diffraction (XRD) Analysis:

XRD recorded on Panalytical's X'Pert Pro, which is indispensable for confirming the crystalline structure, phase purity, and lattice parameters of the synthesized

MOFs and their magnetic components. As depicted in Figure 3, the characteristic diffraction patterns of , can be identified. The base nanoparticles displayed a distinct cubic spinel structure, with well-defined diffraction peaks at  $2\theta$  values of approximately  $30.1^\circ$  (220),  $35.5^\circ$  (311),  $43.1^\circ$  (400),  $57.0^\circ$  (511), and  $62.6^\circ$  (440). These peaks corresponded to the standard magnetite phase, confirming the successful synthesis of crystalline magnetic nanoparticles. The

prominent diffraction peaks were indexed to align well with the standard JCPDS data for. When silica was coated to form, the magnetite peaks remained at similar positions and relative intensities, suggesting that the silica layer was amorphous and did not disrupt the crystalline structure of the magnetic core. The slight decrease in peak intensities indicates successful surface coating. [9,10].

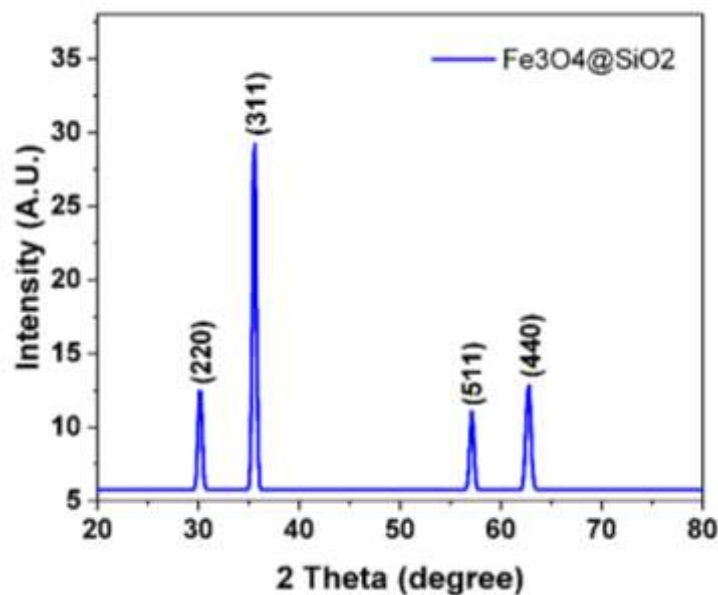


Figure 3 XRD patterns of nanocomposites.

**Table 2** X-ray diffraction (XRD) data for  $\text{Fe}_3\text{O}_4@\text{SiO}_2$ , including Miller indices (hkl), diffraction angles ( $2\theta$ ,  $\theta$ ), interplanar spacing (d), lattice parameter (a), and crystallite size (D) calculated using the Scherrer equation.

#### Fourier Transform Infrared Spectroscopy (FTIR):

FTIR was taken at SAIF Punjab University on Perkin Elmer - Spectrum RX-IFTIR. FTIR is a critical for identifying the functional groups present in the synthesized

materials, confirming the successful coordination of organic linkers to metal centers, the presence of amino and sulfonic groups, and the formation of Fe-O bonds in the magnetic core. Upon silica coating to form, additional Si-O stretching peaks emerge around 1022-1034, confirming successful surface modification while preserving the magnetic core integrity [11, 12]

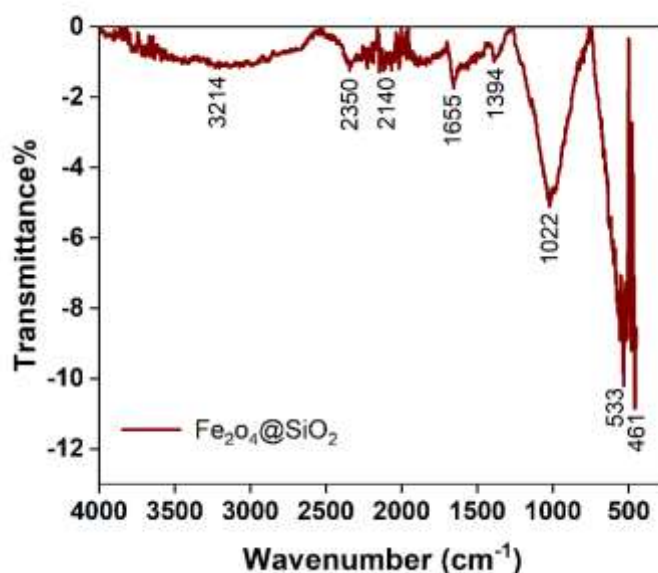


Figure 4 FTIR spectra of nanoparticles

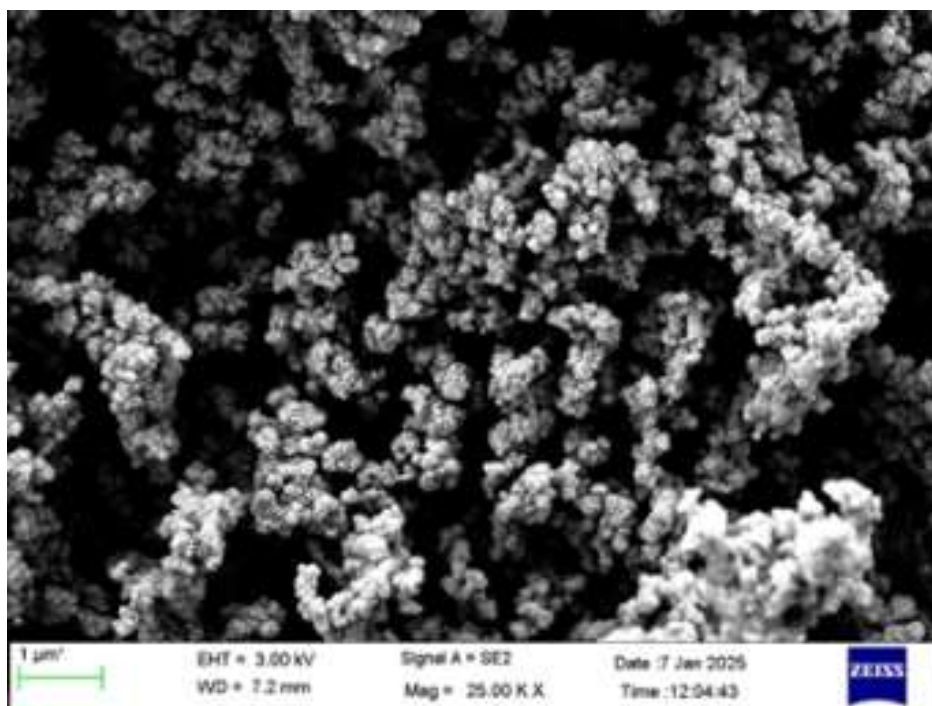


$Fe_3O_4@SiO_2$										
h	k	l	$2\theta$	$\theta$	$\sin\theta$	$2\sin\theta$	d	$(a/d)^2$	a	D nm
2	2	0	30.1	15.1	0.261	0.522	2.958	8.027	8.366	18.5
3	1	1	35.5	17.8	0.304	0.608	2.523	11.044	8.363	19.8
4	0	0	43.4	21.7	0.374	0.748	2.075	16.242	8.317	20.2
5	1	1	57.1	28.6	0.475	0.950	1.613	27.070	8.369	20.2
4	4	0	62.7	31.4	0.523	1.046	1.478	32.082	8.369	17.1
									8.357	19.2

#### Fourier Transform Infrared Spectroscopy (FTIR):

Scanning Electron Microscopy (SEM) was utilized to observe the surface morphology, particle size, and overall topography of the synthesized nanoparticles carried out at Jeol 6390LA/ OXFORD XMX N (DST-SAIF Cochin). It provides high-resolution, three-dimensional images, crucial for understanding the shape, size, and

arrangement of particles. As shown in **Figure 5**, the pristine nanoparticles exhibit a relatively uniform spherical morphology with smooth surfaces and particle sizes in the nanometer range. Upon silica coating to form, the particles maintain their spherical shape but show increased surface roughness and slightly larger dimensions due to the silica shell formation [13].

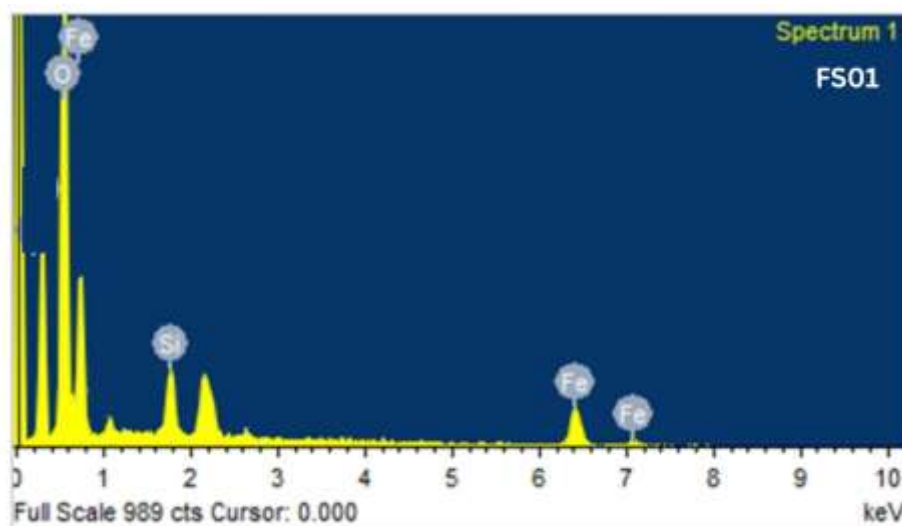


**Figure 5** SEM images of  $Fe_3O_4@SiO_2$  nanoparticles

#### Scanning Electron Microscopy (SEM):

Utilizing this technique, quantitative data regarding the elemental composition of the synthesized materials was obtained, verifying both the ratios of the constituent elements and the successful functionalization performed at Jeol 6390LA/ OXFORD XMX N (DST-SAIF Cochin), as illustrated in Figure 6. The pristine nanoparticles show characteristic peaks for iron (Fe) and oxygen (O) with the expected stoichiometric ratio, confirming the magnetite phase formation. Upon silica

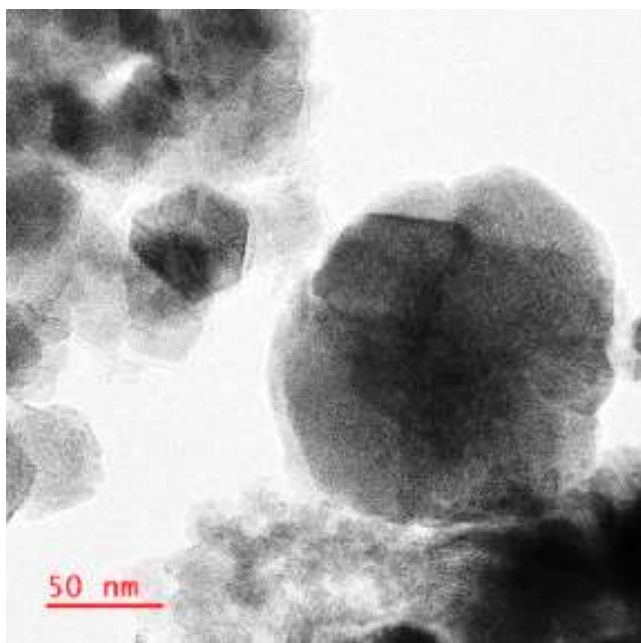
coating to form, additional silicon (Si) and oxygen peaks appear, indicating successful silica shell deposition while maintaining the underlying iron oxide core signals [14]. The relative peak intensities provide insights into the distribution and loading of each component, with the magnetic core signals being attenuated by the outer layers while maintaining detectability, indicating effective but not complete encapsulation that preserves the composite's multifunctional properties [15, 16].



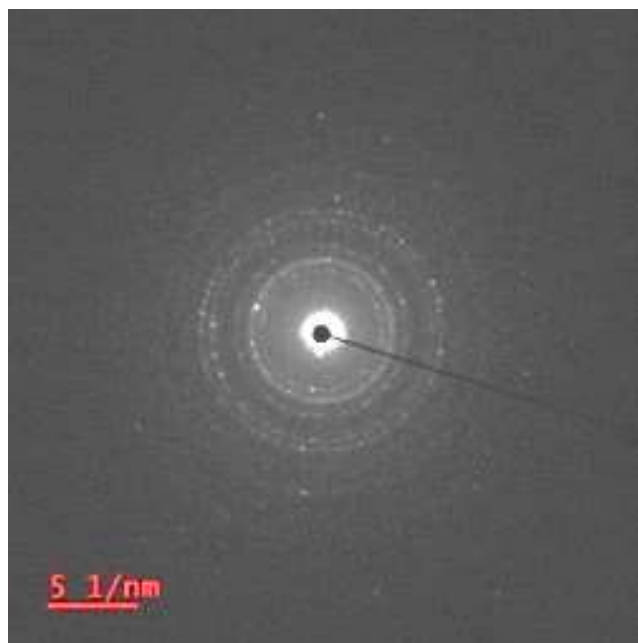
**Figure 6** EDX pattern of  $\text{Fe}_3\text{O}_4@\text{SiO}_2$  nanoparticles  
**Elemental Analysis (EDX)**

Transmission Electron Microscopy (TEM) was conducted at SAIF Mumbai, which provides high-resolution images of the internal structure, morphology, and core-shell formation of composite materials. This technique offers detailed insights into particle size and distribution at the nanoscale, distinguishing between surface and internal compositions [17], as illustrated in **Figure 7**. TEM reveals that  $\text{Fe}_3\text{O}_4$  appears as uniform, spherical nanoparticles,

generally measuring 10-15 nm. The selected area electron diffraction (SAED) patterns displayed distinct rings, confirming their crystalline cubic inverse spinel structure. For, TEM clearly shows a core-shell structure, with a dark core surrounded by a lighter, amorphous silica ( ) shell [18-20]. The SAED pattern supports this observation, showing the crystalline rings of the core along with a diffuse halo from the amorphous silica, as depicted in **Figure 8**.



**Figure 7.** HR-TEM of nanoparticles



**Figure 8.** SAED image of nanoparticles

The TEM-derived particle size values range widely across samples, reflecting the influence of different surface functionalizations and composite architectures. The bare  $\text{Fe}_3\text{O}_4$  nanoparticles exhibited a size of 147.5 nm, which slightly increases to 157.7 nm upon  $\text{SiO}_2$  coating, indicating successful shell formation [21, 22].

**Table 3** Particle Size (via TEM), zeta potential (mV), and electrophoretic mobility ( $\text{cm}^2/\text{Vs}$ ) of various synthesized samples including nanoparticles



Sample	Particle size (nm)	Zeta Potential (Mv)		Electrophoretic Mobility (cm <sup>2</sup> /Vs)	
Fe <sub>3</sub> O <sub>4</sub>	147.5	-41.4		-0.000321	
Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub>	157.7	-23.5	-38.4	-0.000182	-0.0003

#### Vibrating Sample Magnetograph (VSM):

VSM was done at SAIF Mumbai which offers high-resolution imaging of the internal structure, morphology, and core-shell formation of the composite materials, providing detailed information on particle size and distribution at the nanoscale. It can distinguish between surface and internal compositions. The Vibrating Sample Magnetometer (VSM) analysis provides insights into the

magnetic properties of the synthesized materials. At the outset, the nanoparticles demonstrated superparamagnetic behavior, which is indicated by a characteristic S-shaped hysteresis loop with very low remanence and coercivity, and a saturation magnetization ( $M_s$ ) close to 28 emu/g. Once coated with silica, the core-shell particles exhibit superparamagnetism, with an  $M_s$  value of approximately 41 emu/g [23, 24].

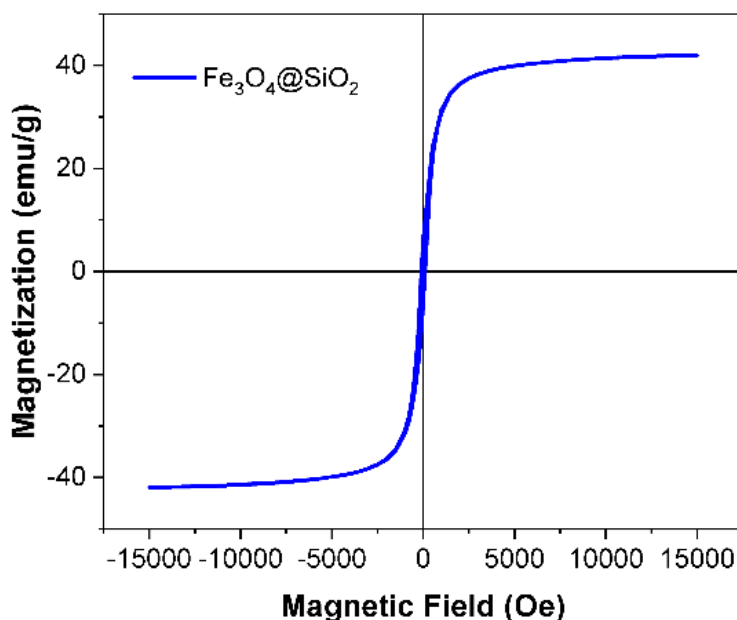


Figure 9 VSM images of nanoparticles

#### Conclusion:

This study effectively produced and analyzed Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub> core-shell nanoparticles through a modified Stöber method enhanced by microwave technology. XRD analysis verified the cubic spinel structure, displaying characteristic diffraction peaks at  $2\theta = 30.1^\circ$  (220),  $35.5^\circ$  (311),  $43.1^\circ$  (400),  $57.0^\circ$  (511), and  $62.6^\circ$  (440), with an average crystallite size of 19.2 nm. FTIR spectroscopy identified Si-O stretching peaks between 1022-1034 cm<sup>-1</sup>, indicating successful silica coating. TEM analysis showed a well-defined core-shell structure, with particle size increasing from 147.5 nm for Fe<sub>3</sub>O<sub>4</sub> to 157.7 nm for Fe<sub>3</sub>O<sub>4</sub>@SiO<sub>2</sub>. VSM measurements revealed improved superparamagnetic properties, with saturation magnetization rising from 28 emu/g to 41.0 emu/g following silica coating. The nanoparticles demonstrated excellent colloidal stability, with zeta potential values ranging from -23.5 to -38.4 mV. These findings confirm the successful creation of multifunctional magnetic nanocomposites that maintain crystallinity, exhibit enhanced magnetic properties, and offer superior stability, making them promising for applications in biomedicine, drug

delivery, magnetic separation, catalysis, and environmental clean-up.

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#### Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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