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Synthesis and Characterization of Iron Oxide (Hematite) Nanoparticles by Sol-gel Method

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Abstract

Iron oxide nanoparticles have excellent biomedical applications because of its large surface area. It can be used in drug delivery, cell separation, tissue repair and MRI. A Fe_2O_3 nanoparticle is synthesized chemically by sol-gel method. This method uses Ammonium hydroxide and Ethanol as a precursor for forming Fe2O3 nanoparticles. Thus Fe_2O_3 nanoparticles are characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDAX) and UV Spectroscopy. All the structural parameters such as lattice constants, unit cell volume, density, crystalline size, micro strain are calculated from the XRD results using Debye Scherrer's formula. When annealing temperature increased from 400^{O}C to 1000^{O}C the average crystalline size of the Fe2O3 nanoparticles are increased from 18 nm to 22 nm. FTIR technique also confirms functional groups of the synthesized Fe2O3 nanoparticles. The SEM image indicates that Fe_2O_3 nanoparticles are approximately spherical in shape. Band gap of the Fe2O3 nanoparticles is founded using UV Spectroscopy and it is reported in paper.

Keywords: Sol-gel, XRD, SEM, FTIR, EDXAbstract.

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1. Introduction

Nanotechnology is the branch of science which deals with the examination of material in nano range generally between 1 to 100nm. It is a science that works at the nanoscale and gives various focal points to the delivers fields of science like bio-engineering.[1] Nanomaterials so far discovered were well in economic circulation which leads to the invention of many new products.[2, 3] The magnetic nanoparticles have many uses such as magnetic drug target, magnetic resonance imaging for clinical diagnosis, recording material and catalyst, environment, etc.[4-6]To ignore the use of poisonous chemical solutions and various reactions-conditions such as temperature, pressure and time[7,8], for the synthesis of nanomaterials the investigation have been introducing the prospect of manufacturing nano-products in aqueous medium with the help of capping agents. Magnetic nanoparticles have many unique magnetic properties such as super paramagnetic, high coercivity, low curie temperature, high magnetic susceptibility etc., Magnetic nanoparticles one of great interest for researchers from a broad range of disciplines, including magnetic fluids, data storage, catalysis and bio-applications.

Iron oxide nanoparticles play a major role in many areas of chemistry, physics, and material science.[9] For this application, certain parameters must be controlled during the synthesis, such as the size and shape of the nanoparticles.[10.11] The control of size, as well as size distribution is necessary because allows the control of the materials properties, such as super magnetism and hyperthermia.[13] Iron oxide exists in three forms in nature: Magnetite (Fe_2O_3), Maghemite (γ-Fe_2O_3) and Hematite (Fe_2O_3). Hematite is the oldest known of the iron oxide and is widespread in rocks and solids.

The important features of Hematite (α-Fe_2O_3) is its density 5.312g/cc and it is weakly ferromagnetic or antiferromagnetic , melting point is about 1350^{O}C and crystallographic system is Rhombohedral (or) hexagonal.[13] Iron oxide nanoparticles have a large surface to volume ratio and therefore possess high surface energies.[14]

2. Experimental Procedure

Various synthesis techniques have been applied to prepare Iron oxide nanoparticles such as Hydrothermal, solid- state reaction, Electron –chemistry, micro-emulsions, spray-pyrolysis, precipitation method and co-precipitation method.[15] Sol-gel is a wet-chemical process that involves the formation of an inorganic colloidal suspension (sol) and gelation of the sol in a continuous liquid phase (gel) to form a three- dimensional network structure. Hence, the sol-gel process has been employed for the synthesis of Iron oxide nanoparticles, because this process is a simple, low cost, more effective and low temperature method.

Iron oxide nanoparticles were synthesized using sol-gel method. In this sol-gel method, 1.30 grams of ferric chloride ($FeCl_3$) was taken and it was dissolved in 40 ml distilled water under vigorous stirring for 10minutes. Then ammonium hydroxide (NH4OH) is added drop wise until the brown colour was obtained. It was stirred for 10minutes and allowed it to settle for half an hour. Thus obtained solution was centrifuge for 10minutes to separate the gel and supertant. The gel was repeat washed with water and ethanol to remove impurities. It was dried in oven at 100°C for 10hours and calcinated at 400°C for 2 hours. The brown coloured iron oxide (Fe_2O_3) nanoparticles were obtained and it was grained and kept for further characteristics

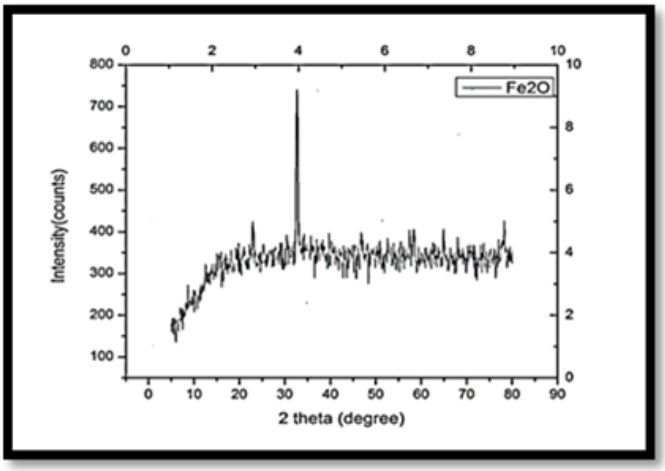

Fig. 1: XRD Spectrum of Iron Oxide

3. Results and Discussion

3.1 X-ray diffraction analysis

The crystallographic analysis was carried out by X-ray diffraction method. The XRD pattern of the prepared Hematite powder is shown in Figure 1. The powder sample was used by X-ray diffraction for confirming the presence of iron oxide and the structure. The XRD peaks confirm that the formation of Iron oxide nanoparticles. The characteristics peaks located at 2θ= 32.6648°, 22.9628°, 58.2814°. The presence of sharp peaks and straight line shows the synthesized powder containing crystalline in nature. The average crystalline size of synthesized Hematite nanoparticles was determined using Debye Scherrer formula. The average crystalline size for Fe_2O_3 is 18 to 22nm.

3.2 Scanning Electron Microscope (SEM) Analysis

SEM was used to investigate the surface morphology. The high resolution SEM analysis of the iron oxide nanoparticles was shown in figure. The Fig. 2 shows the scanned at different magnification. The synthesized iron oxide was formed spherical in shape.

3.3 Energy Dispersive X-Ray Analysis (EDX)

The chemical identity and purity of Fe_2O_3 were investigated with energy dispersive X-ray analysis (EDAX) and the pattern is shown in figure clearly.

Fig.3 shows the presence of elements like Iron & oxide. The presence of elements chloride can be removed by repeated washing using water and ethanol.

3.4 Fourier Transform Infrared Spectroscopy (FTIR) studies

The functional group analysis has been performed using Fourier Transform Infrared Analysis. The FTIR spectrum of the Fe2O3 nanoparticles was recorded. The FTIR spectra of iron oxide nanoparticles synthesized at 100 °C is shown in Figure 4. The peaks at 3730.33 cm^{-1} corresponds to the O-H stretching mode of hydroxyl groups were present on the surface due to moisture. The major peaks at 553.57cm^{-1} confirm the presence of Fe_2O_3 vibration.[16] Absorbance and transmittance studies of Fe_2O_3 nanoparticles for different wavelength range (400-4000 cm^{-1}) are studied using ultraviolet spectrophotometers.

Table 1: Structural parameters of Iron oxide Nano particles

Material	2θ (deg)	FWHM (deg)	Crystalline size D(nm)	Micro strain (ε)10^{-3} m	Dislocation density (δ)10^{15} m
Iron oxide (Fe_2O_3)	32.66	0.455	18.99	1.913	2.772
	22.96	0.452	18.73	1.932	2.848
	58.28	0.450	22.09	1.716	2.246

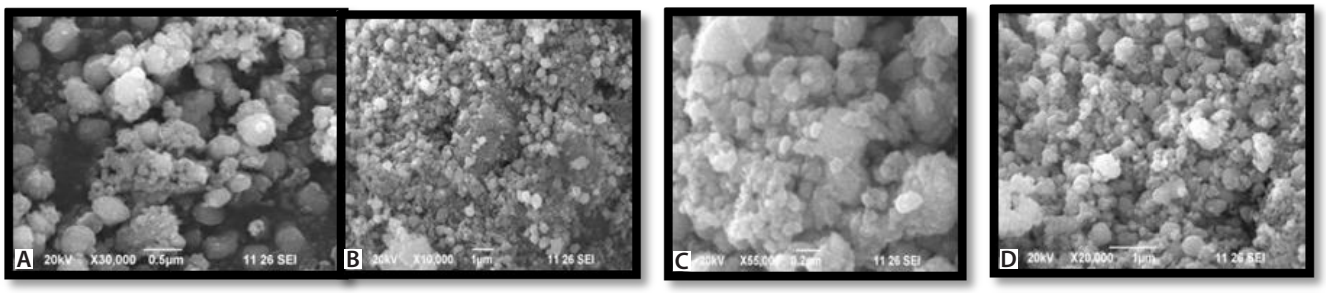

Fig. 2: SEM Images of Fe_2O_3 nanoparticles

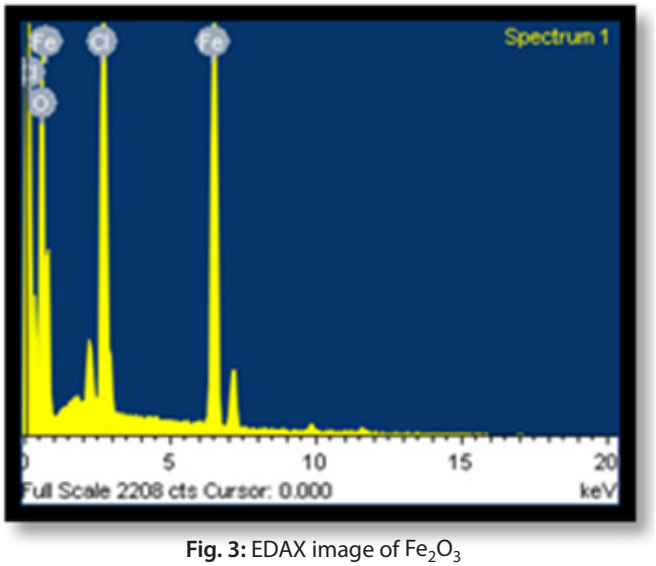

Fig. 3: EDAX image of Fe_2O_3

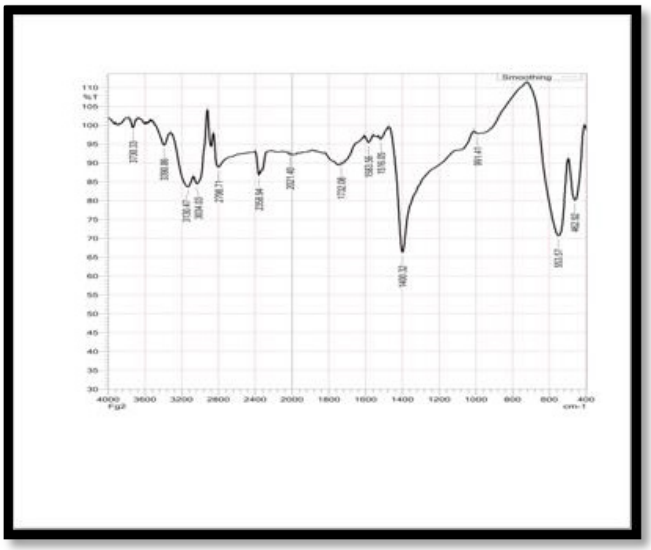

Fig. 4: FTIR vibration frequency of Iron oxide nanoparticles

Table 2: FTIR vibration frequency of iron oxide nanoparticles

Frequency (cm^{-1})	Bond stretching	Functional group
3730.33(m)	O-H stretch	Alcohol
3390.86(m)	N-H stretch	Aliphatic primary amine
3130.47(m)	C-H stretch	Alkene
3034.03(m)	N-H stretch	Amine salt
2798.71(s)	O-H stretch	Alcohol
2358.94(b)	O=C=O stretch	Carbon dioxide
1516.05(m)	N-O stretch	Nitro compound
1400.32(s)	C-F stretch	Fluoro compound
991.41(s)	C=C bend	Alkene
m- medium, s-strong, b-broad		

Table.3 UV analysis of Fe_2O_3 nanoparticles

Sample	Wavelength (nm)	Band gap (eV)
Iron oxide	258	4.80

3.5 Ultra-Violet Visible Spectroscopy Analysis

The optical properties of the behavior of the material synthesized were investigated using UV spectroscopy. The optical transmission wavelength of the iron oxide is 258nm.

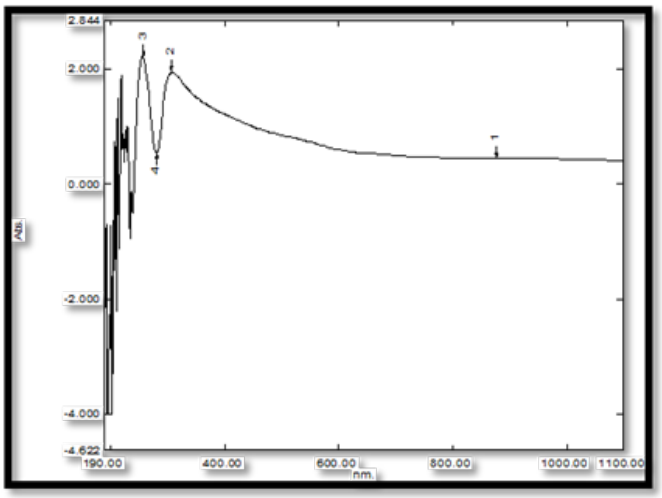

Fig. 5: UV analysis of Iron oxide nanoparticles

The band gap energy is calculated for maximum wavelength (λmax). Band gap energy formula, E = h*c/λ max Band gap energy of Fe_2O_3 nanoparticles is 4.80eV.

4. Conclusion

The Iron oxide nanoparticles with hematite phase were prepared using sol-gel process. The XRD pattern indicates

nano crystalline size of the particles. The SEM image confirms that $Fe2O_3$ nanoparticles have Spherical shape in nanoscale. The FTIR shows the functional group of Fe_2O3 nanoparticles. The UV spectrum is used to determine the band gap energy of Fe_2O_3 nanoparticles.

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