

Preparation and Characterization of Magnetite (Fe_3O_4) Nanoparticles with and Without Surfactant

Saranya G. M.Sc, M.Phil.,

Department of Physics,
JCT Polytechnic College, Coimbatore, Tamil Nadu, India.
saranya.gnanavelk@gmail.com

ABSTRACT:-Magnetite nanoparticles (Fe_3O_4) represent the most promising materials in medical applications. They have attracted extensive interest due to their super paramagnetic properties and their potential applications in many fields. Magnetite (Fe_3O_4) nanoparticles have found to be a potential material in several areas, such as biomedical [1–2], target drug delivery [3], tumor, cancer diagnosis and treatment [4] and as a magnetic resonance imaging (MRI) contrasting agent [5]. There are several techniques such as Chemical Bath Deposition, Hydrothermal method, Solvothermal, Co-precipitation method, Chemical Vapour Deposition and Sol-Gel method, Emulsion method, Arc discharge used for the preparation of Magnetite (Fe_3O_4) Nanoparticles. Among these methods, solvothermal method has been selected for the present work as it is simple, economic and non-polluting. This has been employed to synthesis Magnetite nanoparticles (Fe_3O_4) with and without the aid of surfactants, because there are several obvious advantages of the solvothermal technique over the traditional solid-state reactions. Also made investigate on structural, morphological and optical properties of the materials.

Key words- Magnetite, Biomedical, Target drug delivery, solvothermal.

1. INTRODUCTION

1. General

Iron oxide nanoparticles have been of great interest, not only for fundamental properties caused by their multivalent oxidation states but also for their super paramagnetic, high force, low Curie temperature, high magnetic susceptibility, etc. Release of iron oxide nanoparticles into the environment interact with air, water and soil often causes change in the surface properties of the particles which can result in particle aggregation or changes in particle charge and other surface properties.

Various surface modifications are being done for making these non-biodegradable nanoparticles more biocompatible. The iron oxide nanoparticles must be pre-coated with chitosan which is a biopolymer that increases their stability, bio degradability, and non-toxicity in the physiological medium and also to achieve combined properties of high magnetic saturation, biocompatibility and interactive functions on the surface.

The iron with 26 protons in nucleus is a component of transition elements group. Isotopes like ^{54}Fe , ^{56}Fe , ^{57}Fe and ^{58}Fe are extremely stable. It is the last element produced in the nucleus of stars (only in the stars bigger than five solar mass) through nuclear fusion, so that the iron is the heaviest element created outside the cataclysmic supernova processes. In abundance, is the

tenth in the Universe and the fourth on the Earth. The Earth comprises 80% Iron in a 2160 miles' radius sphere. Iron is widely spread in the nature in different chemical compounds. The interesting question about transition metals is that their valence electrons or the electrons they use to combine with other elements are to be found in more than one shell. This is the reason why; they often exhibit several common oxidation states. Normally, the iron has eight electrons on valence shield and due to oxygen's electro negativity it can form bivalent and trivalent combinations.

Iron oxides are chemical compounds composed of iron and oxygen. Altogether, there are sixteen known iron oxides and oxyhydroxides. The uses of these various oxides and hydroxides are tremendously diverse ranging from pigments in ceramic glaze, to use in thermite.

Oxides:

- Iron(II) oxide, wüstite (FeO)
- Iron (II, III) oxide, magnetite (Fe_3O_4)
- Iron(III) oxide (Fe_2O_3)
- Alpha phase, hematite (α - Fe_2O_3)
- Beta phase, (β - Fe_2O_3)
- Gamma phase, maghemite(γ - Fe_2O_3)
- Epsilon phase

Hydroxides:

- Iron(II) hydroxide ($\text{Fe}(\text{OH})_2$)
- Iron(III) hydroxide ($\text{Fe}(\text{OH})_3$), (bernalite)

Oxide/hydroxide:

- Goethite,

- Akaganéite,
- Lepidocrocite,
- Feroxyhyte,
- Ferrihydrite
- High-pressure FeOOH
- Schwertmannite

2. MAGNETITE (Fe₃O₄)

Iron (II, III) oxide is the chemical compound with formula Fe₃O₄ called as Magnetite. Iron (II, III) oxide contains both Fe²⁺ and Fe³⁺ ions and is sometimes formulated as FeO. Fe₂O₃. This iron oxide is encountered in the laboratory as a black powder. It exhibits permanent magnetism and is Ferrimagnetic, but is sometimes incorrectly described as ferromagnetic [6]. It's most extensive use is as a black pigment which is synthesized rather than being extracted from the naturally occurring mineral as the particle size and shape can be varied by the method of production [7].

Table 1: Properties of Magnetite (Fe₃O₄) nanoparticles

PROPERTIES	VALUES
Chemical formula	Fe ₃ O ₄
Molecular mass	231.533 g/mol
Appearance	Solid black powder
Density	5 g/cm ³
Melting point	1,597 °C (2,907 °F; 1,870 K)
Refractive index	2.42

3. APPLICATIONS OF MAGNETITE (Fe₃O₄) NANOPARTICLES

Due to the unique properties, Fe₃O₄ nanoparticles appear to be very promising for their applications in protein immobilization, bio-separation, environments treatment, biomedical and bioengineering usage, and food analysis.

3.1. Protein Immobilization

Protein immobilization serves as a very effective tool to solve the difficulties encountered in the catalytic application of free enzymes, such as poor stability and hard recovery. It is of vital importance to select proper immobilization basis for protein immobilization. Fe₃O₄ nanoparticles have been intensively utilized to realize this objective due to its unique magnetic performance, and various practical and economical biocatalysts with improved stability and reusability have been fabricated based on Fe₃O₄ nanoparticles, which could be easily separated from the reaction medium in the presence of external magnetic field. Proteins could be immobilized onto Fe₃O₄ nanoparticles in the manner of physical absorption.

3.2. Bio-separation

Magnetic separation is a commonly used technique for polypeptide/protein separation and cell separation. Magnetic separation possesses several advantages such as timesaving, gentle, easily automated, and can be directly used to remove target compounds from crude medium by the simple application of an external magnetic field. To construct Fe₃O₄ based composite nonmaterial's for separation, core/shell microspheres are generally fabricated with a Fe₃O₄ as a core and other functional materials as a shell.

Several functionalized Fe₃O₄ nanoparticles have been strategically developed for the purification of specific proteins utilizing the affinity interactions. The target protein is firstly captured by modified Fe₃O₄ nanoparticles from crude samples to form a complex, and the captured target protein on the particles is subsequently eluted from metal ion chelated magnetic nanoparticles by applying buffer solution with different pH or ion strength. This metal ion chelated magnetic nanoparticles were usually regenerated by using EDTA to strip the adsorbed protein and metal ion and then reloading with metal ion for recycling.

3.3. Environmental Treatment

As a result of rapid industrialization and urbanization, various pollutants particularly those entering aquatic systems have attracted worldwide concern. The development of efficient and cost-effective methods for environmental treatment is of primary concern for sustainable economic and social development. Due to the extremely small particle size, high surface-area-to-volume ratio, and more important the magnetism, Fe₃O₄ nanoparticles have been widely used and have shown promising performance in environments treatment, including pollutant removal and toxicity mitigation. Proper surface coating cannot only improve the removal capacity and affinity of the Fe₃O₄ nanoparticles, but also promote the desorption process. Pollutants generally adsorb to the surface of Fe₃O₄ nanoparticles through interactions including physical adsorption, ion-exchange, chemical bonding (complexation and/or chelation), hydrogen bonds, and van der Waal forces. Fe₃O₄/ZrO₂/chitosan composite was synthesized and employed for the removal of amaranth and tartrazine dyes removal, the adsorption capacities of which were 99.6 and 47.3 mg/g for amaranth and tartrazine dyes, respectively.

3.4. Biomedical Usage

Fe₃O₄ nanoparticles with appropriate surface properties have been widely used for numerous biomedical and bioengineering applications such as targeted drug delivery, biosensor, magnetic resonance imaging, hyperthermia, tissue engineering, magnetofection, etc. All these applications require that these nanoparticles not only possess high magnetization values, but also with

narrow particle size distribution and similar surface topography, so that the particles have uniform physical and chemical properties. Moreover, the magnetic nanoparticles for biomedical applications should be non-toxic and biocompatible. In a word, both of the nature and the geometric arrangement of surface coatings on the nanoparticles have apparent influence on bio-kinetics and bio-distribution of nanoparticles in the body.

3.5. Targeted Drug Delivery

Due to the unique capabilities (e.g., superparamagnetism and biocompatibility) and the negligible side effects, magnetic Fe_3O_4 nanoparticles with proper surface modification and conjugated targeting ligands/molecules have become a major research focus for drug delivery applications. Compare to the conventional, non-targeted methods of drug delivery, magnetic nanoparticles are promising drug carriers due to the better specificity to the target site and the reduced adverse effects. Drug carried by magnetic nanoparticles could be concentrated at the desired site to receive much high therapeutic efficiency. Since the drugs simply physically attached to the nanoparticle surface tend to release quickly before reaching the final destination, a core-shell structure consisting of a magnetic core and a shell is preferred in magnetic drug-delivery systems to achieve sufficient drug loading capacity and good transportation effect.

3.6. Biosensor

Fe_3O_4 nanoparticles based bioanalytical sensors could be fabricated by coating Fe_3O_4 nanoparticles with materials such as a fluorescent one, a metal, silica, or a polymer. Glucose oxidase was simply mixed with Fe_3O_4 nanoparticles and cross-linked on the Pt electrode with chitosan medium by glutaraldehyde, and then covered with a thin nafion film. The biosensor showed high sensitivity, low detection limit (6×10^{-6} M), and good storage stability.

3.7. Magnetic Resonance Imaging

Magnetic resonance imaging (MRI) is a commonly used non-invasive medical imaging technique in clinical medicine to visualize the structure and function of tissues, which is based on the behavior, alignment and interaction of protons in the presence of an applied magnetic field.

II. PREPARATION OF MAGNETITE (Fe_3O_4) NANOPARTICLES

1. Materials Required

The chemicals used in this experiment were all analytical grade and used without any further purification. Ferrous Sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$), Sodium Hydroxide (NaOH), Glucose, Ethylene Glycol (EG), EDTA, CTAB is the chemicals used in the reaction process.

2. Preparation of Magnetite (Fe_3O_4) Nanoparticles

In a typical procedure, 1 mole of Ferrous Sulfate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) and 2 mole of Sodium Hydroxide (NaOH) solution are prepared in deionized water. The solution was stirred continuously, until a clear solution is obtained. The pH of the solution is maintained at 10. Then the solution is transferred into a Teflon lined autoclave and it was maintained at 160°C for about 4 hours and then air cooled at room temperature. The precipitations were filtered out, washed with distilled water and absolute Ethanol. The final products were dried at 60°C for 3 hours.

- The same procedure was repeated with the addition of 0.5 ml of surfactants (CTAB, Glucose and EDTA).
- Also Ethylene Glycol was used as a solvent and surfactant to prepare Magnetite (Fe_3O_4) nanoparticles.

III. RESULT AND DISCUSSION

1. STRUCTURAL ANALYSIS

The structural properties of the Magnetite (Fe_3O_4) nanoparticles are measured by using X-ray Diffractometer with Cu-K α radiation (wavelength $\lambda = 1.54056 \text{ \AA}$) for the range of $2\theta = 20^\circ$ to 80° with a scan speed of $10^\circ/\text{minute}$.

1.1 Xrd Analysis of Magnetite (Fe_3O_4) Nanoparticles

Fig (1.1 (a) and (b)) shows the X-ray diffraction pattern of Magnetite (Fe_3O_4) nanoparticles prepared using water and Ethylene Glycol. Both the spectrum indicates sharp and well defined peaks corresponding the most prominent (311) orientation. Other peaks at (220), (400), (422), (333) and (440) of cubic Fe_3O_4 lattice respectively are also observed. These results are in good agreement with that of the standard JCPDS data (Card no: 82-1533) and also with that of the XRD pattern of Magnetite (Fe_3O_4) nanoparticles reported in the literature [8,9] confirming the cubic spinal structure of the Magnetite (Fe_3O_4) material.

The crystallite size of Magnetite (Fe_3O_4) nanoparticles obtained from the Scherrer's formula based on Full Width Half Maximum (FWHM) of major diffraction peaks are shown in Table (1.1). It is inferred from the table that the Magnetite (Fe_3O_4) nanoparticles prepared using water is smaller when compared to that of Ethylene Glycol. Though Ethylene Glycol is a strong reducing agent with a relatively high boiling point, producing Magnetite (Fe_3O_4) nanoparticles with acceptable size distribution without particle aggregation is a major problem.

During the formation process of Magnetite (Fe_3O_4) nanoparticles the charged groups which are present can coordinate with iron cation in solution and can effect the

nucleation and aggregation of the nanocrystals [10]. It has been reported that Fe³⁺ cannot be reduced by Ethylene Glycol alone. Because the coordination of water molecule to metal ions is stronger than that of Ethylene Glycol molecules and by adjusting the dosage of water, the particle size and size distribution of Magnetite (Fe₃O₄) nanoparticles can be controlled [11]. The lattice constant were also evaluated and presented in the Table (1).

Fig (2) shows the XRD pattern of Magnetite (Fe₃O₄) nanoparticles prepared CTAB, Glucose and EDTA as surfactant. The XRD patterns of three samples were compared with that of the standard JCPDS data (Card no: 82-1533). The position and relative intensity of the peaks obtained are in agreement with the standard powder diffraction with characteristics peak corresponding to (220), (400), (333) and (440) Bragg's reflection respectively.

This revealed that the resultant particles are the Magnetite (Fe₃O₄) iron oxide with a cubic spinel structure. No other diffraction peaks are observed indicating highly pure crystalline phase of magnetite (Fe₃O₄) nanoparticle. From the Fig (3): it evident that the intensity of the peaks decreases for the particle prepared using glucose as surfactant whereas, the intensity of the peak prepared using CTAB is almost same as that prepared in water without CTAB. The particle sizes are estimated using Scherrer's formula considering the major orientation at (311) plane and is shown in Table (3.1.2). From the table, it is observed that the Magnetite (Fe₃O₄) nanoparticles prepared using Glucose as surfactant is smaller when compared to that of CTAB and EDTA.

It has been reported [12] that, EDTA chelate precursors is an excellent method of preparing a powder with a controlled morphology at relatively low temperatures and CTAB modification effectively decreases it's particle size and enhances the dispersion of particle in solution [13], but the surfactant Glucose is an excellent reducing agent as it is possible to accomplish homogenous precipitation by the solvolysis of the Fe(III) complex which will eventually lead to the formation of large amount of nuclei instantaneously. The nucleation process can be followed by synergetic reduction with Glucose and water [14]. The lattice constant is evaluated for the most prominent orientation and is shown in Table [1.2].

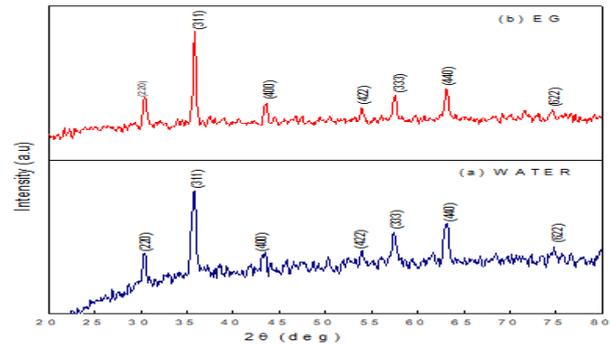


Fig 1: XRD spectrum of Magnetite (Fe₃O₄) nanoparticles prepared in(a) water (b) Ethylene Glycol.

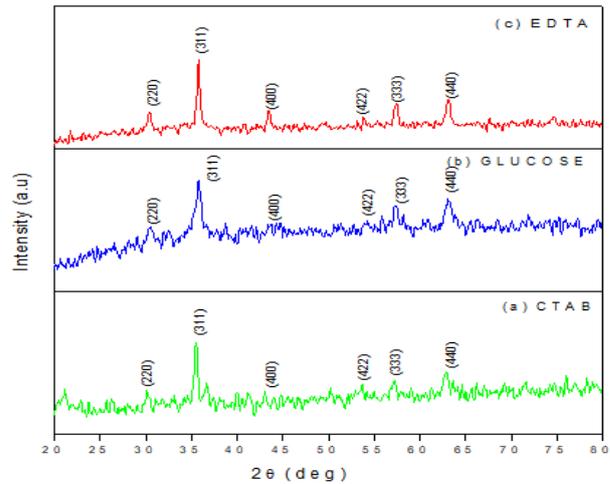


Fig .2: XRD spectrum of Magnetite (Fe₃O₄) nanoparticles with surfactant.

Table 1. Structural parameters of Magnetite (Fe₃O₄) nanoparticles without surfactant.

Sample	2θ (degree)		FWHM (β)	hkl	d-spacing (Å)		D (nm)	Lattice Parameter (Å)	
	Observed	standard			Observed	standard		Observed	Standard
Magnetite (Fe ₃ O ₄) with water	30.28	30.07	0.70	(220)	2.9509	2.9687	20	8.323	8.397
	35.77	35.42	0.68	(311)	2.5095	2.5317	20		
	43.37	43.05	0.56	(400)	2.0858	2.0992	25		
	57.49	56.93	0.81	(333)	1.6025	1.6160	17		
	63.07	62.52	0.81	(440)	1.4736	1.4843	17		
Magnetite (Fe ₃ O ₄) with EG	30.56	30.07	0.64	(220)	2.9245	2.9687	22	8.303	
	35.86	35.42	0.64	(311)	2.5035	2.5317	22		
	43.59	43.05	0.64	(400)	2.0757	2.0992	22		
	57.48	56.93	0.61	(333)	1.6028	1.6160	23		
	63.05	62.52	0.75	(440)	1.4739	1.4843	19		

Table 2: Structural parameters of Magnetite (Fe_3O_4) nanoparticles with surfactants

Sample	2θ (degree)		FWHM (β)	(hkl)	d-spacing (Å)		D (nm)	Lattice Parameter (Å)	
	Observed	Standard			Observed	Standard		Observed	Standard
Magnetite (Fe_3O_4) Without surfactant	30.28	30.07	0.70	(220)	2.9509	2.9687	20	8.332	
	35.77	35.42	0.68	(311)	2.5095	2.5317	20		
	43.37	43.05	0.56	(400)	2.0858	2.0992	25		
	57.49	56.93	0.81	(333)	1.6025	1.6160	17		
	63.07	62.52	0.81	(440)	1.4736	1.4843	17		
Magnetite (Fe_3O_4) With CTAB	30.28	30.07	0.68	(220)	2.9509	2.9687	20	8.346	8.397
	35.48	35.42	0.65	(311)	2.5294	2.5317	21		
	43.03	43.05	0.71	(400)	2.1015	2.0992	20		
	57.16	56.93	0.69	(333)	1.6110	1.6160	20		
	62.95	62.52	0.81	(440)	1.4761	1.4843	17		
Magnetite (Fe_3O_4) With Glucose	30.69	30.07	1.01	(220)	2.9123	2.9687	14	8.303	
	35.86	35.42	1.01	(311)	2.5035	2.5317	14		
	57.34	56.93	0.64	(333)	1.6064	1.6160	22		
	63.05	62.52	1.01	(440)	1.4739	1.4843	14		
Magnetite (Fe_3O_4) With EDTA	30.42	30.07	0.77	(220)	2.9376	2.9687	18	8.303	
	35.86	35.42	0.78	(311)	2.2531	2.5317	18		
	43.59	43.05	0.64	(400)	2.0758	2.0992	22		
	57.48	56.93	0.64	(333)	1.6028	1.6160	22		
	63.05	62.52	0.88	(440)	1.4739	1.4843	16		

2. MORPHOLOGICAL ANALYSIS

The morphology and particle distribution of the Magnetite (Fe_3O_4) nanoparticles were analyzed using Scanning Electron Microscopy (FESEM - BRUKER). Fig (2.1 to 2.5) shows the surface morphology of magnetite (Fe_3O_4) nanoparticles prepared in water, Ethylene Glycol, CTAB, Glucose and EDTA. Micrographs (2.1 to 2.3) reveal uniform spherical nanoparticles with an average diameter ranging from 50 to 200nm. The excess amount of Ethylene Glycol chains due to hydrogen bridging between adsorbed Ethylene Glycol molecules on Magnetite (Fe_3O_4) nanoparticles surfaces tending to increase in agglomeration above the critical concentration [15]. However, the micrographs (2.4 and 2.5) show spherical nanoparticles with high agglomeration and a significant increase in agglomeration is formed for nanoparticles prepared using EDTA.

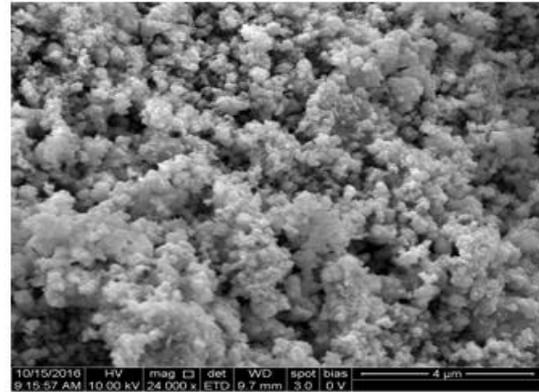


Fig 3: The FESEM Micrograph of Magnetite (Fe_3O_4) nanoparticles.

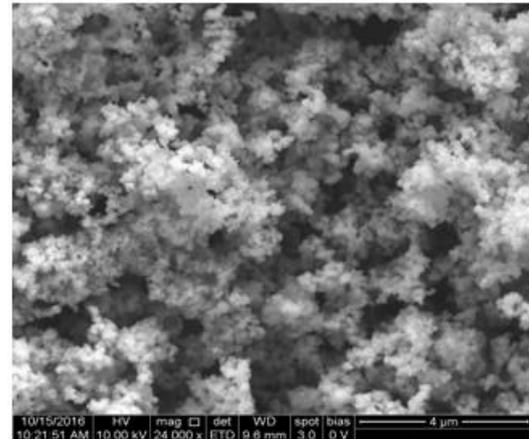


Fig 4 The FESEM Micrograph of Magnetite (Fe_3O_4) nanoparticles with EG.

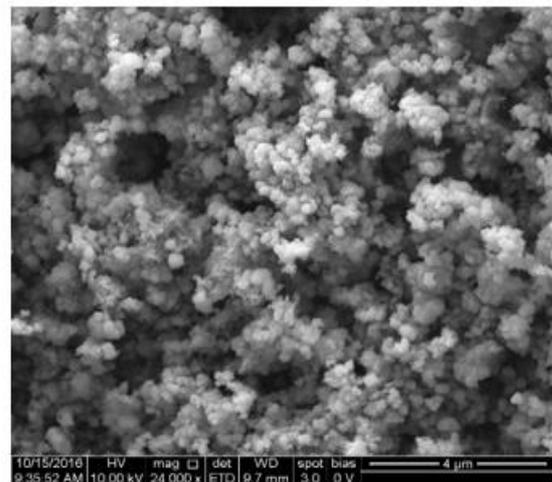


Fig 5 The FESEM Micrograph of Magnetite (Fe_3O_4) nanoparticles with CTAB.

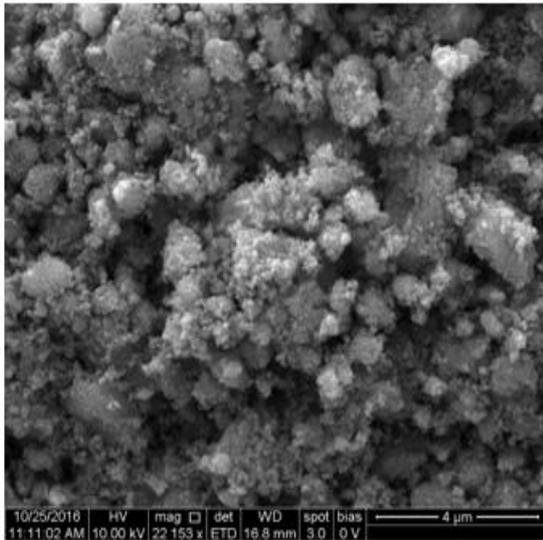


Fig 6 The FESEM Micrograph of Magnetite (Fe_3O_4) nanoparticles with Glucose.

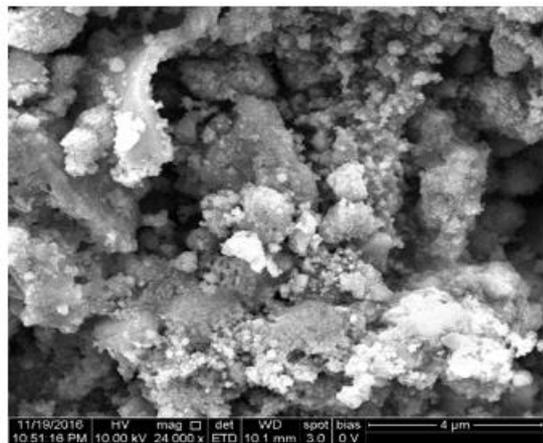


Fig 7 The FESEM Micrograph of Magnetite (Fe_3O_4) nanoparticles with EDTA.

3. COMPOSITIONAL ANALYSIS

The EDAX images (BRUKER - INSTRUMENT) of the prepared Magnetite (Fe_3O_4) nanoparticles with and without surfactants are shown in Fig (3.1 to 3.5). The corresponding atomic and weight percentage of all the prepared Magnetite (Fe_3O_4) nanoparticles are evaluated and tabulated [Table (3.1 and 3.2)]. All the prepared samples have Fe and O elements, without any traces of other elements in the sample. From the table, it is observed that Magnetite (Fe_3O_4) nanoparticles prepared using water and Glucose as surfactant are found to be nearly stoichiometric, whereas Magnetite (Fe_3O_4) nanoparticles prepared using CTAB, EDTA and Ethylene Glycol are nonstoichiometric. It is also observed that

there is large deviation in stoichiometry for the sample prepared using Ethylene Glycol.

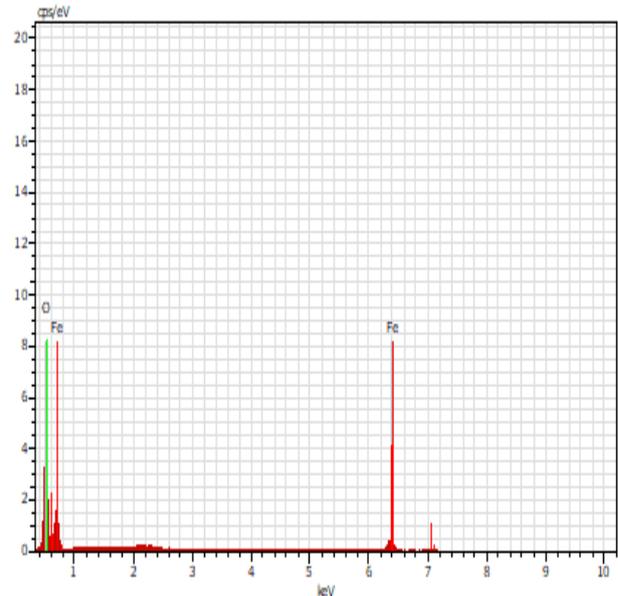


Fig 8 The EDAX Spectrum of Magnetite (Fe_3O_4) nanoparticles.

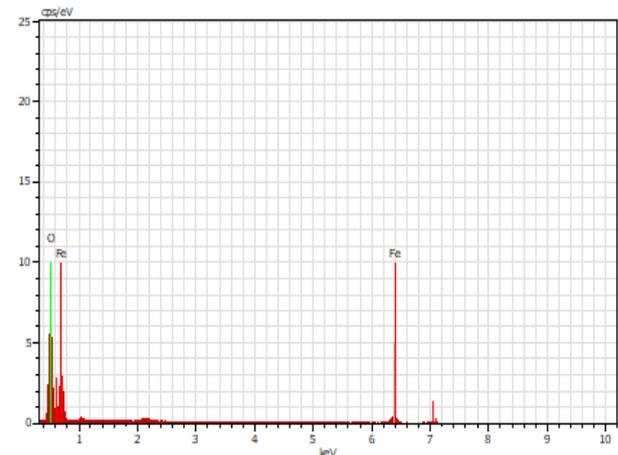


Fig 9 The EDAX Spectrum of Magnetite (Fe_3O_4) nanoparticles with EG.

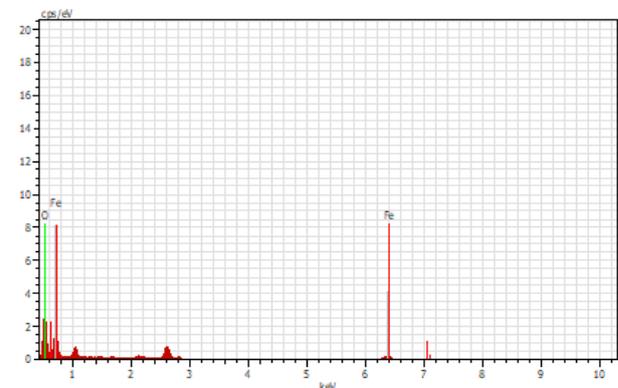


Fig 10 The EDAX Spectrum of Magnetite (Fe_3O_4) nanoparticles with CTAB.

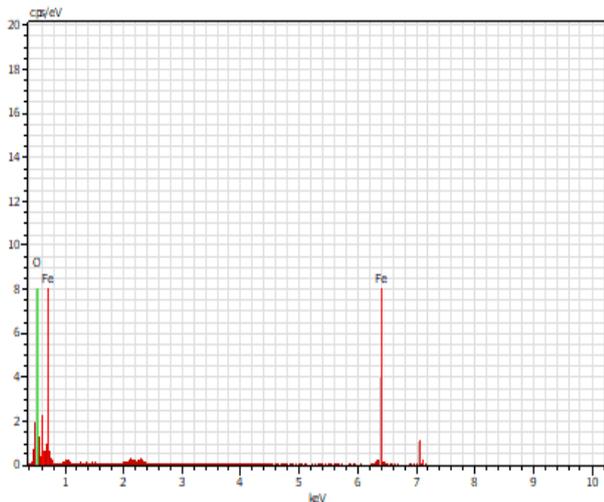


Fig 11 The EDAX Spectrum of Magnetite (Fe_3O_4) nanoparticles with Glucose.

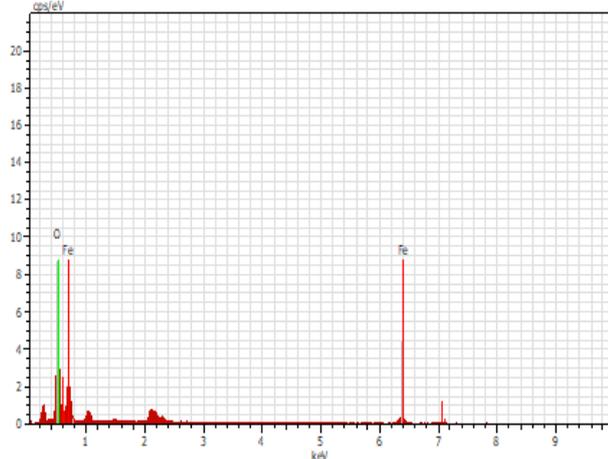


Fig 12 The EDAX Spectrum of Magnetite (Fe_3O_4) nanoparticles with EDTA.

Table 3 The Atomic and Weight % of Fe and O in Magnetite (Fe_3O_4) nanoparticles without surfactant.

Sample	Standard Values				Calculated Values			
	Atomic percentage		Weight Percentage		Atomic Percentage		Weight Percentage	
	Fe	O	Fe	O	Fe	O	Fe	O
Magnetite with water					41.06	58.94	70.86	29.14
Magnetite with EG	42.86	57.14	72.36	27.64	30.16	69.84	60.12	39.88

Table 4 The Atomic and weight % of Fe and O in Magnetite (Fe_3O_4) nanoparticles with surfactant.

Sample	Standard Values				Calculated Values			
	Atomic percentage		Weight Percentage		Atomic Percentage		Weight Percentage	
	Fe	O	Fe	O	Fe	O	Fe	O
Magnetite without surfactant					41.06	58.94	70.86	29.14
Magnetite with CTAB	42.86	57.14	72.36	27.64	38.23	61.77	68.36	31.64
Magnetite with Glucose					40.03	59.97	69.97	30.03
Magnetite with EDTA					34.44	65.56	64.71	35.29

4. OPTICAL ANALYSIS

The optical properties of Magnetite (Fe_3O_4) nanoparticles are studied using “Diffuse Reflectance Spectrometer”. Fig (4.1) and Fig (4.2) shows the absorbance spectra for Magnetite (Fe_3O_4) without and with surfactant. The optical spectra of the prepared samples are measured in the range of 400-1000nm. All the prepared samples exhibit the same pattern with higher absorption on the shorter wavelength side. The band gap energy was estimated from the plot of a graph between $[F(R) h] n$ and (h) . The best fit to the experimental data was obtained for $n=2$. This indicates that Magnetite (Fe_3O_4) nanoparticles are a semiconducting material with a direct-band gap.

Fig (4.1) reveals that, the maximum absorbance is obtained by using water as a solvent. The band gaps of Magnetite (Fe_3O_4) nanoparticles without surfactant are estimated to be 2.05 eV (water) and 1.95 eV (Ethylene Glycol). Optical band gap of Magnetite (Fe_3O_4) nanoparticles with surfactant are also calculated as 2.01 eV (CTAB), 2.09 eV (Glucose) and 2.02 eV (EDTA) respectively. The maximum absorbance of Magnetite (Fe_3O_4) nanoparticles was obtained for the sample prepared with surfactant. It has been reported earlier [16] that the band gap energy of Magnetite (Fe_3O_4) nanoparticles is in the range of 1.6 to 1.7 eV. Also H.El.Ghandoor et al [17] has reported a value of 2.81 eV. But in the present work band gaps are in the range 2.00 to 2.09 eV. Also the sizes of Magnetite (Fe_3O_4) nanoparticles were around 5nm but in the present work 20nm is observed. This means that, the energy band gaps of the nonmaterial's are inversely proportional to their sizes. From the above results it can be stated, that the energy band gaps of nonmaterial's can be controlled by controlling their sizes.

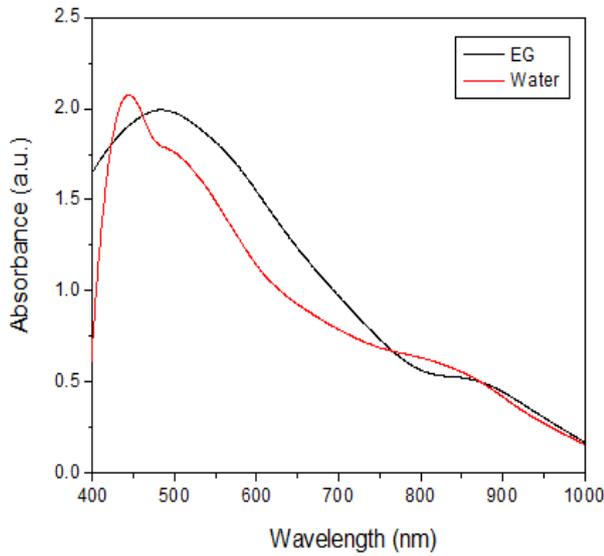


Fig 13 Wavelength Vs Absorbance of Magnetite (Fe_3O_4) without surfactant.

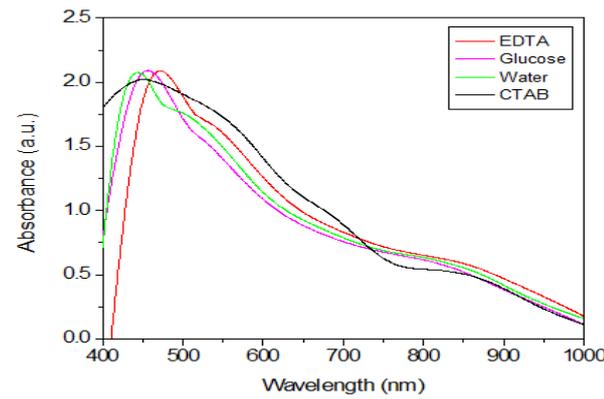


Fig 14: Wavelength Vs Absorbance of Magnetite (Fe_3O_4) with surfactant.

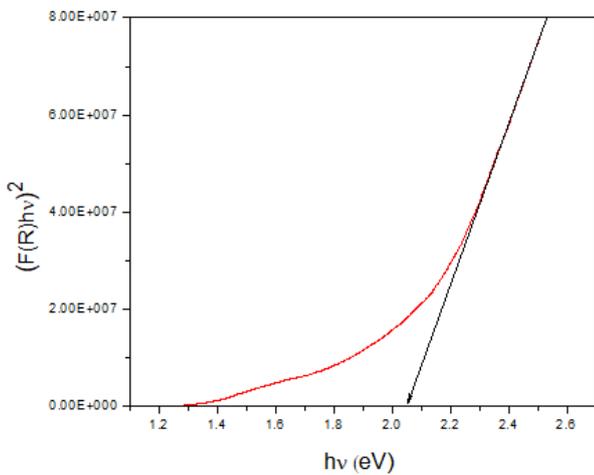


Fig 15 Plot of $(F(R)h)^2$ Vs (h) for Magnetite (Fe_3O_4) nanoparticles.

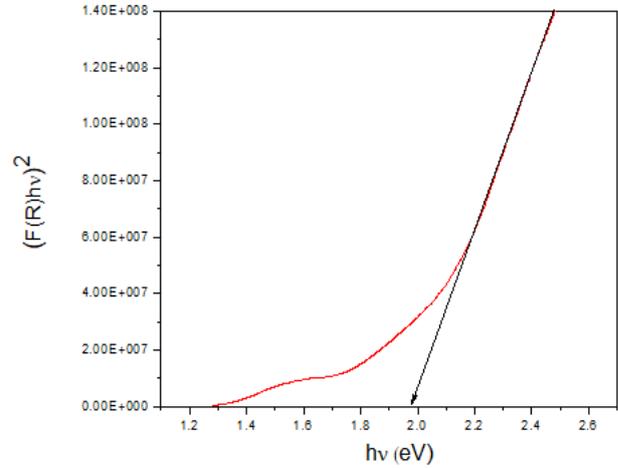


Fig 16 Plot of $(F(R)h)^2$ Vs (h) for Magnetite (Fe_3O_4) with EG.

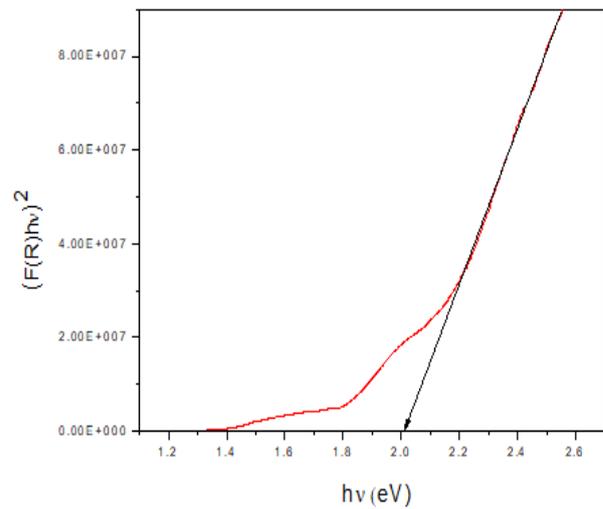


Fig 17 Plot of $(F(R)h)^2$ Vs (h) for Magnetite (Fe_3O_4) with CTAB.

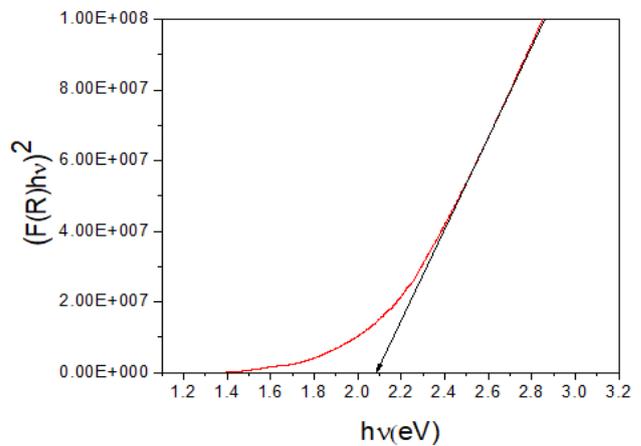


Fig 18 Plot of $(F(R)h)^2$ Vs (h) for Magnetite (Fe_3O_4) with Glucose.

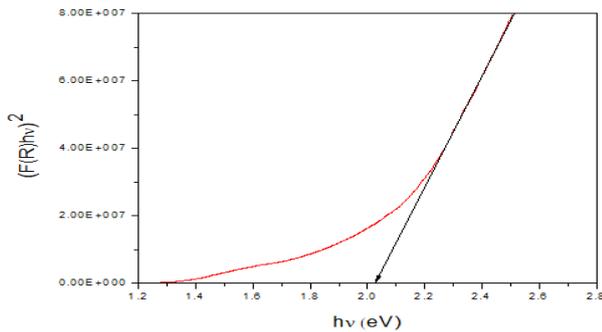


Fig . 19 Plot of $(F(R)h)^2$ Vs (h) for Magnetite (Fe_3O_4) with EDTA.

IV. CONCLUSION

Magnetic nanoparticles consisting of Magnetite (Fe_3O_4) possess unique characteristics that make them an excellent material for various applications. In this study, Magnetite (Fe_3O_4) nanoparticles were prepared using solvothermal technique. Ethylene Glycol and water were used as two reaction media in Magnetite (Fe_3O_4) nanoparticles, were obtained using anhydrous Ferrous Sulfate ($FeSO_4 \cdot 7H_2O$). Surfactants such as CTAB, Glucose and EDTA were also used for preparing the nanoparticle of sizes less than 20 nm. The nanoparticles were investigated for their structural, morphological, compositional and optical properties.

X-Ray Diffraction studies showed that Magnetite (Fe_3O_4) nanoparticles exhibited cubic spinel structure in nature. Also it is noted that Magnetite (Fe_3O_4) nanoparticles shows increase in particle size when Ethylene Glycol was used as a solvent. The sizes of the Magnetite (Fe_3O_4) nanoparticles prepared using surfactant CTAB was found to be higher (21nm) than EDTA (18 nm) and Glucose (14 nm). Morphological analysis of Magnetite (Fe_3O_4) nanoparticles with and without surfactant was found to be spherical like structure. Magnetite (Fe_3O_4) nanoparticles prepared using water as solvent were found to be nearly stoichiometric. Deviation in stoichiometry was observed for all other samples.

From the absorbance–wavelength measurement spectrum, it is found that the Magnetite (Fe_3O_4) nanoparticles exhibit high absorbing nature at the shorter wavelength indicating direct transition. The optical band gap values were also found to decrease with increase in particle size.

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