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Characterization of Iron Oxide Nanoparticles from the Leaf Extract of Piper Betle by Green Synthesis Method

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Abstract

In this present study, Iron Oxide nano particles were synthesized by using Green method. For this synthesis on Iron oxide, the leaf extract of piper betle was used as a reducing agent and $FeCl_3$ as a precursor. Thus, they were characterized by XRD, SEM, EDX and FTIR. The parity of FeO nano particles was confirmed by EDX. The crystalline size of Iron Oxide nano particles was analyzed using X-ray Diffraction (XRD) spectrum. The functional groups are identified in Fourier Transform Infrared Spectroscopy (FTIR). The surface morphology of the Iron Oxide Nano particles is found from Scanning Electron Microscopy (SEM). The optical properties are determined by using UV-Visible Spectroscopy. Thus, the so-formed nano particles were FeO..

Keywords: Iron oxide, Nano particles, Green synthesis, XRD.

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1. Introduction

Nanotechnology is one of the important and exciting area of science. It is the manipulation of matter on an atomic, molecular and supramolecular scales that ranging from (1 to 100nm).[3] The preparation of most essential metal oxide nanoparticles have gained concentration in physical, chemical, medical, biological, optical and so on to explore and handle single atoms and molecules. [1] Nanotechnology has numerous applications in the field nano medicine, nano electronics, biomaterials, and energy production and consumer products.[4] It ignores the use of poisonous chemical solution, for the preparation of nanomaterials has been examined by referring the prospect of manufacturing nano products in the help of capping agent.[2]

Iron Oxide nanoparticles are an inorganic transition metal oxide with many applications. The band gap energy of the iron oxide nanoparticles is always in the range of (2.2 to 2.7) eV. FeO has a wide range of applications like low-friction seals, dampening and cooling agents in loudspeakers, magnetically active membrane biological reactor, regenerate solution, recovery of hazardous wastes and controlled micro fluidic flow. Various methods have been estimated for the synthesis of Iron Oxide Nanoparticles that involves both physical and chemical method like sol-gel method, hydro thermal method, Electro- deposition, Bio synthesis method, chemical vapor deposition, sono-chemical synthesis, microwave method.[5-9]

The growing need of environmentally friendly nano particles, researches are using green synthesis method for the export of various metal nano particles. By using plant or leaf extract. The synthesis of metal oxide nanoparticles were suggested as valuable alternative for chemical method .[18, 19] In the presence study green synthesis of iron oxide carried out using the leaf extract of piper betle leaves.

2. Experimental Procedure

2.1 Materials Required

Various synthesis techniques have been applied to prepare iron oxide nanoparticles such as chemical precipitation method, micro emulsion, hydrothermal synthesis, Co-precipitation method, Thermal decomposition method, Sol-gel method. The advantages of green synthesis method over the other methods because it is simple, cost-effective, and relatively reproducible, and often result in more stable materials. There is no requirement for high pressure, energy, temperature, toxic chemical[10-19]

2.1.1 *Preparation of piper betle leaf extract*

About 50 gm of fresh healthy leaves of piper betle leaf were collected from Erode, Tamil Nadu. They were washed thoroughly with de-ionized water, they are cut into fine pieces and boiled with 100ml de-ionized water until the water changes to golden yellow color and the extract was filtered through filter paper.100ml of distilled water and 4 mm of ferric chloride is stirred using a magnetic stirrer.

2.1.2 *Properties and uses of Iron Oxide*

Fe_2O_3 nanoparticles find great attention in biomedical applications due to their super paramagnetic properties arising from its biocompatibility and non-toxicity. They have the basic composition of Fe, o but differ in the valency of iron and overall the structure of crystal some of the important iron oxides are goethite, akaganeite, lepidocrocite, magnetite and hermatite.[20,-22] The chemical properties of iron oxide nano particles are having the chemical symbol Fe_2O_3 and its electronic configuration iron [Ar] 3d 4s oxygen [He]2s 2p and its physical properties are molar mass of 159.69g/mol and its melting point 1566°C and 2851°F. There properties provide additional stability for magnetic nanoparticles in solutions. There are many properties of iron oxide such as it is odorless and appearance in reddish brown color solid. It has various melting points which depend upon the medium used to melt it. Density of iron oxide is 5.25g/cm^3 which are Insoluble in water. And the iron oxides are used in iron ores, pigments, catalysts, and in thermite, and occur in hemoglobin. The iron oxides are inexpensive and durable pigments in paints, coating and colored concretes.

2.1.3 *Synthesis of Iron Oxide Nanoparticles*

The prepared ferric chloride solution was mixed with piper betle leaf extract in 1:1 ratio in a beaker. The mixture was heated at 70° to 80°C for 30 minutes. The formed solid product was collected by centrifugation and washed with deionized water for three to 4 times and dried at room temperature. The dried product was calcinated at 400°C for 3 hours. The prepared samples were characterized by UV-Visible spectroscopy, Fourier Transform Infrared (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX).

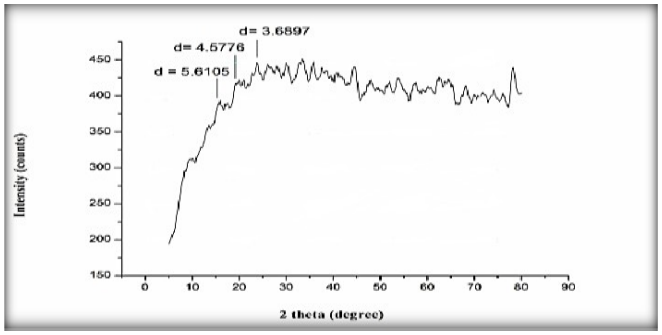

Fig.1: XRD Pattern for Fe2o3 Nanoparticles

3. Results and Discussions

3.1 X-ray Diffraction analysis

The average size, the crystalline nature of the particles and quality of compounds were determined by X-Ray diffraction (XRD) spectrum with CuKα radiation λ =1.5406*10 $^{-10}$ m. The X-ray diffraction patterns in Fig.1. The XRD spectrum shows different diffraction peaks at corresponding to the crystal plan. The strongest peaks of Fe_2O_3 with 2θ values are (15.7828), (19.3750) and (24.1000). The Crystalline-size of the synthesized iron oxide nano particles using the Scherer equation (d = k λ / β cos θ) by determining the width of the Bragg's reflection, where k is the Scherer constant (0.94), is the wavelength of X-Ray (1.5406*10^{-10}), is the half width of the peak and is the Bragg's angle. Those values are tabulated it shown in Table.1. The average crystalline size is 9.9171 nm.

3.1.1 *Scanning Electron Microscope (SEM) analysis*

The powered sample was analyzed for the structure and morphology of the synthesized iron oxide nano particles using SEM at different magnification levels are shown in Fig.3.2. SEM images revealed that the synthesized iron oxide nano particles were aggregated as tiny spherical shapes. The morphology of the nano particles mostly appeared to be a porous and spongy.

3.1.2 *Fourier Transform Infrared Spectroscopy (FTIR) studies*

FTIR was employed to identify the functional group of the synthesized samples. The wave length region was recorded in the range of (400-4000) cm^{-1}. The FTIR spectrum of iron oxide nano particles are shown in Fig. 3. The absorption band is observed at 3084.18 cm^{-1} which is

Table.1: Structural Parameters of Fe_2o_3 Nanoparticles

S. No.	2θ (degree)	Inter- planar Distance(d)	Crystalline Size [D*10^{-9} m]	Dislocation Density (δ* 10^15)	Micro- strain (ε *10^-3)
1.	15.7828	5.6105	9.91666	15.5676	4.2375
2.	19.3750	4.5776			
3.	24.1000	3.6897			

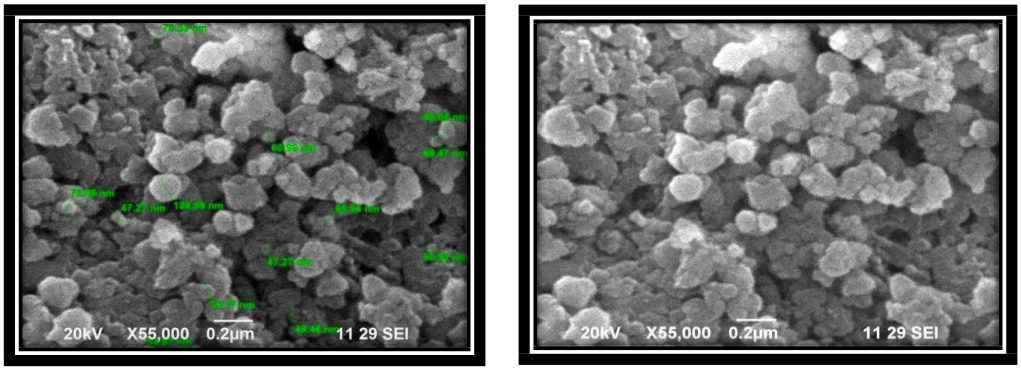

Fig. 2 SEM Images of Fe2o3 Nanoparticles

assigned to O-H in alcohol O-H bending. The absorption band is observed 2779.42 cm^{-1} which is assigned to C-H in ALKYNE bending. The band at 2358.94cm^{-1} was assigned to O=C=O ISOCYNATE. The band at 1612.49 cm^{-1} was assigned to C-Cin α, β unsaturated. The band at 1546 cm^{-1} as assigned to nitro compound N-O stretching. The band at 1400cm^{-1} was assigned to carboxylic acid O-H bending. The band at 1058.92 cm^{-1} was assigned to aliphatic ether C-O bending. The band at 788.89cm-1 was assigned to 1,2,3 tri substituted C-H bending. The band at 565.14cm^{-1} was assigned to halo compound C-Br bending and so on. The Table.2 shows the functional groups of synthesized iron oxide. The Characteristics Absorption of functional groups and type of vibrations with intensity were listed in the Table.2.

Table 2: Functional Groups in IR Spectra of Fe_2O_3

BAND ASSIGNMENT	WAVE NUMBER	INTENSITY
ALCOHOL O-H	3429.43	Medium, sharp
ALKYNE C-H	3084.18	Medium
ALDEHYDE C-H	2779.42	Medium
ISOCYNATE O=C=O	2358.94	Strong
$\alpha.\beta$ UNSATURATEDKETONE C-C	1612.49	Strong

3.1.4 *Ultra –Violet Visible Spectroscopy (UV) analysis*

UV-visible spectroscopy is most widely used technique to investigate the optical properties of the particles. The color change from colorless to black indicated the formation of iron oxide nanoparticles UV-visible spectroscopy analysis was done in the range of (200- 1200) nm is shown in Fig. 3.4. The maximum absorbance was observed at 229 nm for the formation of iron oxide nanoparticles due to the excitation

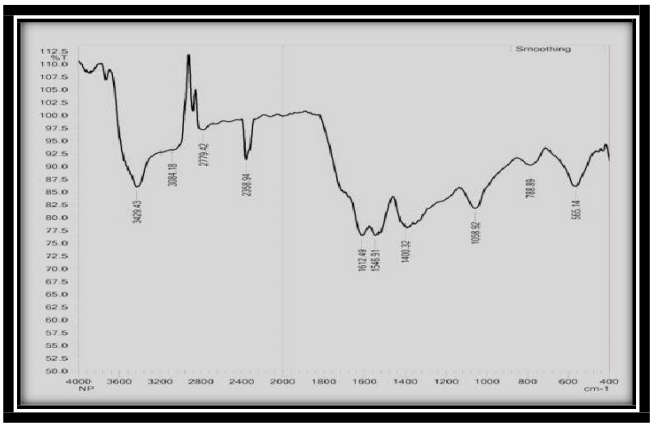

Fig. 3: FTIR Spectrum of Fe_2O_3

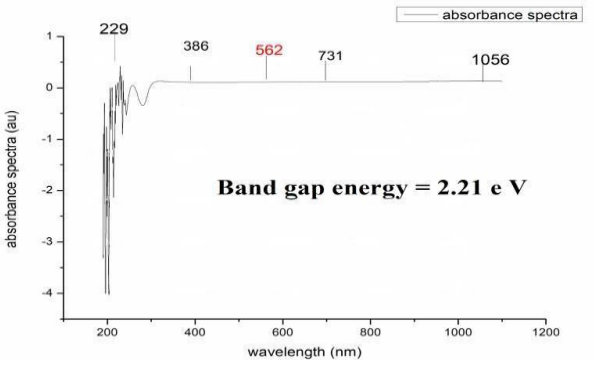

Fig. 4: UV Visible Spectra of Iron Oxide

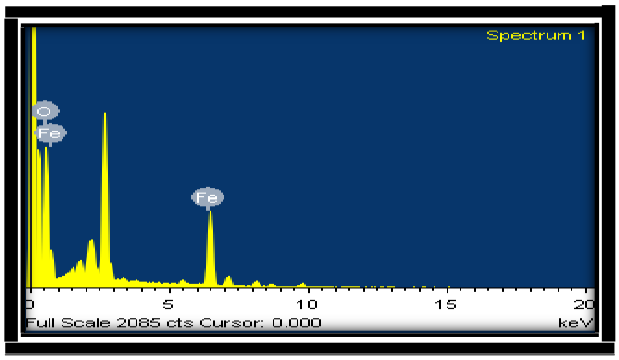

Fig. 5: EDAX of Fe_2O_3 Nanoparticles

of surface plasma on vibration. The UV spectra for the synthesized iron oxide nanoparticles are shown in Fig.4. Using the absorbance lambda value of iron oxide of λ=562 nm, the band gap energy of the iron oxide nano particles sample was calculated to be 2.21 eV.

3.1.5 *Energy Dispersive X-Ray Spectroscopy (EDAX) analysis*

Energy Dispersive X-Ray Spectroscopy is used to determine composition of synthesized nanoparticles. The EDAX Graph for iron oxide nanoparticles is shown in the Fig. 5. Since from the figure Fe - content is 16.37% and O – content is 83.63%.

4. Conclusion

Iron oxide nanoparticles were successfully synthesized by a green synthesis method. XRD reveals that crystalline size of Fe_2O_3 was found to be 9.9166nm. SEM estimated the morphology of Synthesizing Fe_2O_3, which are tiny-spherical shapes. FTIR shows that functional groups present in the repaired iron oxide. UV determined the band gap energy of 2.21eV for the explored iron oxide EDX shows the composition synthesized iron oxide i.e.; Fe content is 16.37% & O- content is 83.63%.

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