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Research Paper

Preparation and Characterization of Magnetite Nanoparticle using Green Synthesis

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Abstract: *The studies show the synthesis of nanomagnetite, in lab by biological method as it does not involve any harmful chemicals and are easily available in the market. In the present work, magnetite (Fe_3O_4) nanoparticles were synthesized by using green biosynthetic method. The band- gap of the Fe_3O_4-NPs was investigated by UV-Vis Absorption & FTIR Spectroscopy are in range of 2.17-3.40 eV. The average particle diameter & its grain size are determined by the Effective Mass Approximation Formula and XRD technique, which came to be 1-2 nm and grain size ~4-14.3nm respectively. . Also conductivity of prepared samples was calculated using the TDS Conductivity Meter, the results shows that the prepared nanoparticle are conductive in nature. The nanoparticles synthesized by this biosynthesis method can potentially useful in various applications.*

Keywords: *Nanomagnetite, UV-Vis Absorption Spectroscopy, XRD, FTIR, nanoparticles, Effective Mass Approximation Formula, Conductivity.*

Introduction

Magnetite (Fe_3O_4), is a natural iron oxide magnet, has a distinguishing characteristic. Magnetite is the most magnetic of all the minerals on Earth. It has an inverse spinel structure with oxygen forming a face-centered cubic crystal system. In magnetite, all tetrahedral sites are occupied by Fe^{3+} and octahedral sites are occupied by both Fe^{3+} and Fe^{2+}. Magnetite is an adaptable material because of its physical-chemical properties such as mechanical, electrical, optical, magnetic properties [1-4]. It is used in various applications such as high gradient magnetic separation, magnetic resonance technology, drug delivery and various biomedical fields [5-7]. Magnetic properties of magnetic nanoparticles can be tailored by their particle sizes and size distributions. The particle sizes and size distributions of magnetic nanoparticles are, in turn, affected by the synthesis route. For these reasons, various synthesis approaches have been developed to produce Fe_3O_4 nanoparticles in order to obtain desired properties [8-11].

Different types of synthesis techniques are used for the synthesis of magnetite nanomaterials such as bottom-up approach, viz, chemical precipitation technique[12-15], thermal decomposition of organic iron precursor in organic solvents[16 -18], Polyol process[19], sol - gel method[18], sonochemical synthesis[20-21],solvothermal synthesis[22] hydrothermal synthesis [23-25], and emulsion technique[26,27] and in top-down, ball milling etc. Recently, great efforts were made to use green and ecofriendly method for synthesis of nanosize materials. These efforts include the use of plant or fruit extracts as surfactant [28,29]. Parts of plants such as leaf, latex, stem, seed, and root are being widely used for nanoparticle synthesis [30]. The greener & environment friendly processes are becoming very much popular, for preparing nanoparticle using naturally occurring reagents, which would be an attractive feature in the field of nanotechnology.The green synthesis ecological approach gives an excellent quality, high purity nano powder [31].In the present study characterization of magnetite nanoparticle has been done by Uv-Vis spectroscopy, FTIR & XRD technique and its particle size are estimated using effective mass approximation formula and also its conductivity is checked using Digital Conductivity Meter.

Material and Methods

Material Required

Edible oil: Canadian pride canola oil, pidilite klog remover, purchased from the market, water is used from laboratory tap water without purification, from rusted iron sheets, rust was obtained, Tops vinegar, ferrous sulphate

(purchase from merck's lab), ferric chloride(purchase from merck's lab), *Azadirachta Indica* (leaf extract). All chemicals used were of analytic grade.

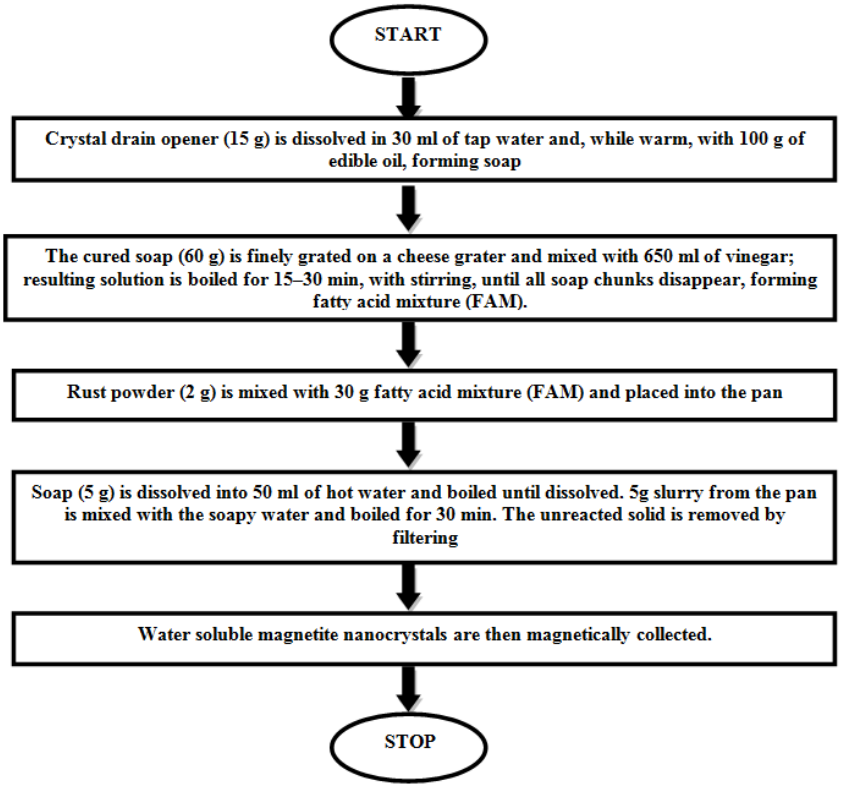


Figure 1: Flow chart of preparation of magnetite nanoparticles

Synthesis Technique

Using kitchen synthesis: In our work, Magnetite nanoparticles were synthesized using kitchen synthesis ecofriendly approach. The process is shown in flow chart given above:

Using green synthesis

In our exertion, Magnetite nanoparticle were synthesized using green synthesis (leaf extract), ecofriendly approach. Before going to the process first of all we must know how to prepare leaf extract of any plant sample:

Figure 2: *Azadirachta Indica* leaf extract

Azadirachta Indica leaves were collected from *Azadirachta Indica* plant at our college campus. The leaves were washed several times with distilled water to remove the dust particles and then dried in hot air oven to remove remaining moisture at 80℃. Now the dried leaves were grinded finely & measured and kept in glass beaker along with 500ml of double distilled water. the solution is placed in soxclet apparatus and wait for the extract to get prepared. The extract was cooled to room temperature, filtered, and stored at 20 °C before use.

The process is shown in flow chart given below.

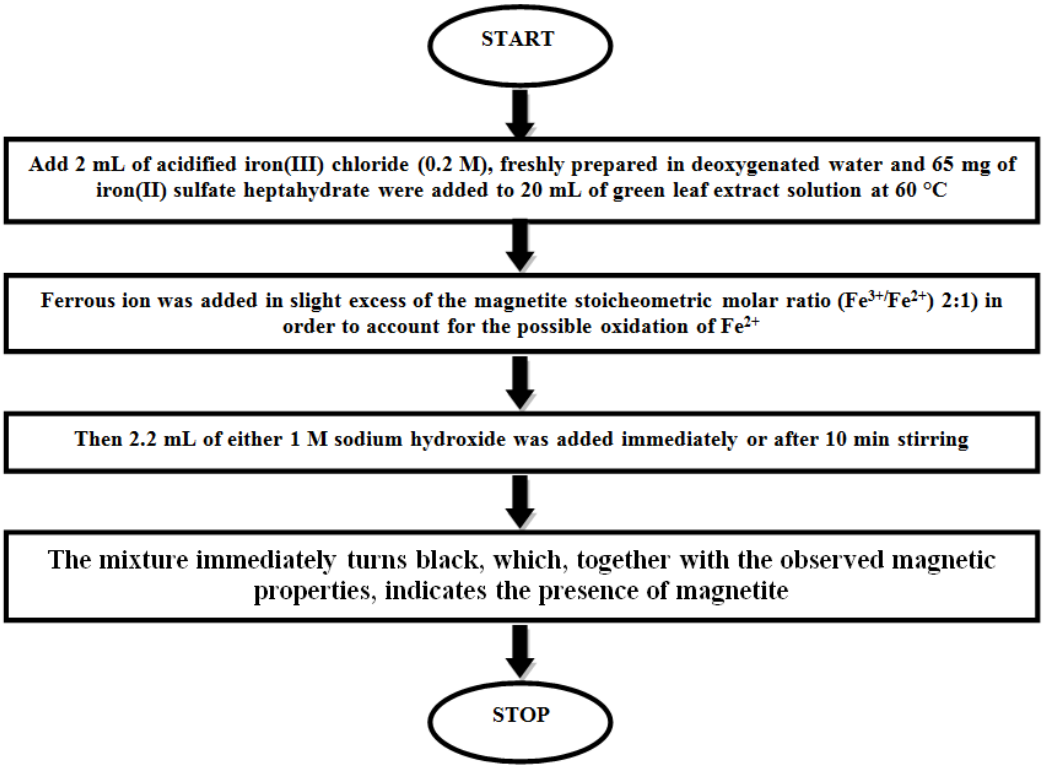
Figure 3: Synthesized Magnetite NPs

Characterization

UV-Vis absorption is first characterization technique for prepared nanoparticles, to check whether the prepared sample is in nano range or not as it gives information about

formation of nanoparicles, the band-gap, and its size distribution, from the absorption spectrum.

Absorbance spectrum of Magnetite nanoparticles are shown in graphs (Figure 4,5) was calculated usingdigital spectrometer in our lab itself digital spectrometer in our lab itself.



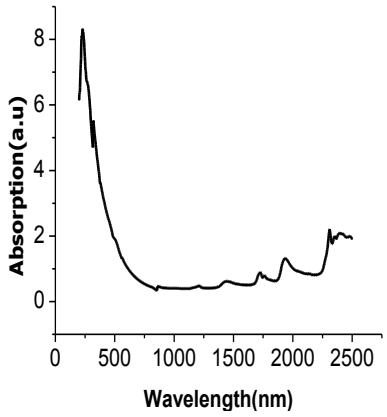


Figure 5: Absorbance spectrum of magnetite nanoparticles prepared using kitchen synthesis (Green Synthesis)

The absorbance peak is related to the band gap energy and hence using maximum absorbed wavelength, we can convert peak wavelength into band-gap energy. This is done by using famous Einstein-plank's relation:

$$E = \frac{hc}{\lambda}$$

Where,

E = band gap energy,

h = plank constant,

c = velocity of light,

λ = maximum absorbed wavelength.

By putting the respective values in above equation, we obtained energy of band gap to be 2.17eV (for kitchen synthesis) and 3.40 eV (for green synthesis) at wavelength, 580 nm, and 360 nm respectively.

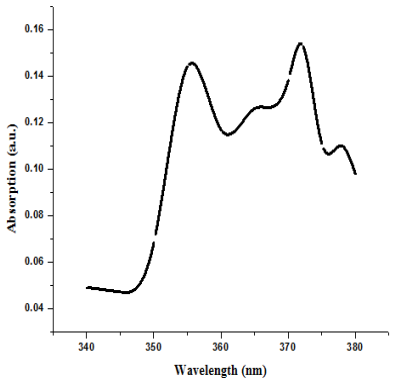


Figure 6: Absorbance spectrum of magnetite nanoparticle

The UV-Vis spectroscopy revealed that the material has turned into nanoparticle form showing enhancement in band gap, which is higher than the corresponding bulk magnetite.

X-Ray diffraction

X-ray diffraction is a rapid technique used for phase recognition of a crystalline material and can provide information on unit cell dimensions. Powder diffraction is often easier and more convenient than other crystal diffraction.

In powder XRD, the sample is usually in a powdery form, consisting of fine grains of crystalline material to be studied. Here we have used Brucker D-8 Diffractomter, which measures data in transmission mode, and is used mostly with solid sample.

In Brucker D-8 Diffractomter following instrumentation is used:

Source is CuK_α having a wavelength of, $\lambda K_\alpha = 0.154$ nm.
Sample: solid (powder sample).
Detector: Si/Li doped solid-state detector.
The value of its scanning range, i.e. $2\theta(°)$ value ranges from $20° \; to \; 100°$ as it is a standard scanning range for XRD.

XRD diffraction pattern of magnetite nanoparticles are shown in graph given below (Figure 6).

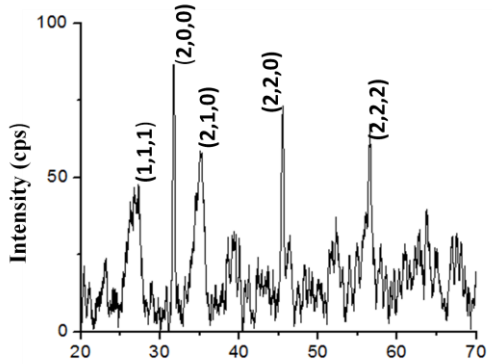


Figure 7: XRD graph of magnetite nanoparticle

The XRD data is used in interpretation of grain size of the sample using Scherrer formula

$$t = \frac{0.9\lambda}{B\cos\theta}$$

Where t is the particle size, λ is the wavelength of the incident X-ray beam, θ is the Bragg's diffraction angle, B is Full width at half maxima (FWHM) of the magnetite peak.

Fourier Transform Infrared (FTIR) Spectroscopy

It is a measurement technique for collecting infrared spectra. It is also cheaper than other conventional spectrometers, as a single spectrum measurement is faster than multiple one because it measures information at all frequencies simultaneously. An infrared spectrum (Figure 8) is obtained by passing IR radiation through the pallet sample, which determines which fraction of incident radiation is absorbed maximum at particular energy.

The absorption is in the range of 1500-4000 cm^{-1} corresponds to the face centered cubic crystal. The main absorption peak is at ~ 1500-2000 cm^{-1} (~2981x10^{-25}- 3975 x10^{-25} joule).

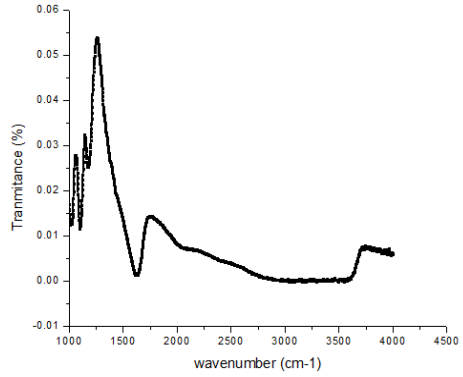


Figure 6: FTIR spectrum of magnetite nanoparticle

Theoretical Calculation

For determining particle size**,** Effective Approximation formula is used, this is a theoretical formula used to calculate particle size of prepared nanoparticles, the formula is derived from total energy concept i.e. the total energy of sample is basically sum-up of all internal energy, the formula is given below[32]:

$$E = E_g + \frac{\hbar^2 \pi^2}{2R^2}\left\{\frac{1}{m_e*} + \frac{1}{m_h*}\right\} - \frac{1.8e^2}{4\pi\varepsilon R}$$

Where,

E = band gap of the synthesized particle, Eg = bulk band gap, R = radius of the particle (0.1eV), m_e* = effective mass of electron (100m), m_h*= effective mass of hole (100m), m = mass of electron, ε is dielectric constant of the material.

On putting the respective values, in above equation:

➡

$$\text{E-Eg} = \frac{(1.054 * 10^{-34})^2 * 3.14^2}{2 * R^2 * (10^{-18})}$$

$$\left\{\frac{1}{100 * 9.1 * 10^{-31}} + \frac{1}{100 * 9.1 * 10^{-31}}\right\}$$

$$-\frac{1.8 * (1.6 * 10^{-19})^2}{\varepsilon * R * 10^{-9}}$$

➡

$$E - Eg = \frac{1.0964 * 10^{-49}}{R^2}\{2.197 * 10^{28}\} - \frac{5.4211 * 10^{-30}}{R}$$

$$-\frac{1.8 * (1.6 * 10^{-19})^2}{\varepsilon * R * 10^{-9}}$$

➡

$$(E - Eg)R^2 + 2.45 \times 10^{-29} R - 1.208 = 0$$

By putting the value of bulk band-gap and band-gap of prepared nanoparticle samples, we can calculate the particle size of respective nanoparticle samples.

Results and Discussion

1. Magnetite nanoparticles are successfully synthesized using ecofriendly approach through green route.

2. The band gap of prepared nanoparticle as calculated using UV- Visible absorption spectroscopy is :

Sample	Band gap (eV)
Magnetite Nanoparticle	
Using kitchen synthesis	2.17
Using green synthesis	3.40

3. By using XRD graph, the calculated size of nanocrystallites using the Scherrer formula was in the range of 4- 14.3 nm.

4. FTIR spectrum measurement shows the formation of magnetite bonds without any impurity, and this result can be interpreted as the change in energy level position due to quantum confinement effect.

5. The particle size was calculated theoretically by Effective mass approximation formula , using estimated value of band-gap which was obtained from UV-Vis spectroscopy.

Sample	Particle Size (nm)
Magnetite Nanoparticle	
Using kitchen synthesis	2.00
Using green synthesis	1.38

6. Conductivity of prepared sample is measured using TDS Conductivity meter came to be: 0.25 m-Siemens

7. The results obtained, proved that the particles synthesized were in nano range.

Conclusion

The kitchen synthesis is used for the magnetite preparation that has become a press forward and new technique reveals that, the synthesized nanoparticles are in nanorange, and are characterized using UV-Vis, XRD, and FTIR technique.

The particles size estimated theoretically were came to be approximately 2 nm using effective mass approximation formula and also studied that the prepared sample is conductive in nature showing 0.25milli-seimen conductivity.

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