



GREEN SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES USING BARRINGTONIA ACUTANGULA LEAF EXTRACT

¹Renjusha S., ²Anjali R.

¹Assistant Professor

Department of Chemistry

N.S.S College, Pandalam

Pathanamthitta, Kerala, India

Abstract : Iron Nanoparticles were synthesized successfully using an unconventional, eco-friendly technique using *Barringtonia acutangula* leaves extract. The Polyphenols in *Barringtonia acutangula* extract may possess the properties of reducing the ferric cations and also act as capping agents. By adopting this biological method it was possible to conduct the synthesis in a simple and environment-friendly manner when compared to chemical methods. In recent years, iron oxide nanoparticles have been successfully synthesized from various plant species using green synthesis pathways and have been analyzed for different bioactivity properties. In this study, iron oxide nanoparticles were synthesized using a completely non-hazardous method using *Barringtonia acutangula* aqueous leaf extract. The synthesized product was characterized by SEM, XRD crystallography, FT-IR, and UV-Vis spectroscopy. Characterization methods have shown that the synthesized iron oxide nanoparticles have specific size and morphology. The studies concluded that the synthesis of iron oxide nanoparticles using plant extracts is more beneficial as it is an economical, energy efficient, low cost and environment-friendly process than the bio hazardous chemical synthesis.

Key Words: Nanotechnology, Green synthesis, Iron oxide, *Barringtonia acutangula*, Antibacterial activity

1. INTRODUCTION

With integrated technology and science, the orientation of research from the subsisting microscopic theme towards the nanoscopic system is materializing with scientific relevance¹. At nano scale, materials exhibit unique and enhanced properties which are not found in their bulk counterpart, these attributes arising from the effect of their size, shape, surface area to volume ratio, quantum confinement. Consequently, we see a myriad of applications of nanotechnology in pharmaceutical research, smart electronic materials, alternative energy generation, environmental remediation and allied fields [1,2]. Physical and chemical methods are being extensively used for production of metal and metal oxide nanoparticles which require the use of reactive and toxic agents which may cause death and adverse effects on the environment and organisms. In recent years, the development of efficient green chemistry approach attracted the attention of researchers to find out the non toxic method for the synthesis of nanoparticles. The metal oxide nanoparticles are synthesized using eco-friendly materials such as plant tissue. Bio synthesis is a biological method which includes a wide range of possible applications from nanotechnology enabled environmentally friendly manufacturing processes that reduce waste products and the use of toxic materials [3].

Iron oxide nanoparticles have Super Magnetic, and It is an exciting metal oxide because of its many applications in advanced technologies, as it is widely used in many critical medical and biological applications such as reversing images in magnetic resonance technology [4], tissue restoration [5], detoxification of living fluids [6], and as deliverables for drugs within living systems [7] and the removal of ions of toxic heavy metals from wastewater [8] and other uses. All of these applications require that iron oxide nanoparticles have high magnetization values, minimal particle dimensions, and scale distribution within a narrow range, and as a result, it has homogeneous properties. In this study, we report the synthesise of iron oxide nanoparticles using iron chloride tetrahydrate as a precursor. The various phytochemicals present in the *Barringtonia acutangula* leaves act as reducing and capping agents for the synthesis of NPs [9]. After mixing flower leaf extract with iron chloride tetrahydrate at certain reaction conditions. The phytochemicals that exist in the extract cannot reduce Fe²⁺ to Fe⁰; instead, they react with the iron ions to give iron oxide NPs,

as Fe is susceptible to oxidation. The brown colour formation occurred due to the interaction between these phytochemicals and metal ions, ensuring the formation of α -Fe₂O₃ nanoparticles [10].

2. EXPERIMENTAL PART

2.1. Preparation of *barringtonia acutangula* leaf extract

10 grams of *Barringtonia acutangula* leaves was weighed, cut into fine pieces. Then boiling them with 100mL of distilled water for 1 h at 60°C, and filtered through Whatman No.1 filter paper. The filtrate aqueous extract was used as a reducing agent.

2.2. Preparation of iron oxide Nanoparticles Using saffron extract

The preparation is made by adding 16.22 g of iron chloride to 100 ml of distilled water to form a solution with a concentration of (1M). 50mL ferric chloride solution is taken and poured into a clean stabilized flask. Equal amount of acutangula leaf extract is added to the flask. There was an immediate colour change to black, which indicates the presence of nano particles, after the addition of extract to the ferric chloride solution. The *pH* of the solution was adjusted with 0.01M NaOH solution. It was then centrifuged and the supernatant was discarded. The residue containing nanoparticles was washed with deionised water and again centrifuged to remove impurities. It was then filtered through whatmann No.41 filter paper. The precipitate along with the filter paper is incinerated in a silica crucible. The synthesized nano particles are calcinated at 400 °C in a muffle furnace for 2hrs to get uniform surface morphology of iron oxide nano particles.

3. RESULTS AND DISCUSSION

The synthesized Fe₂O₃ NPs were exposed to various characterization methods to identify their specific properties. Several techniques have been used to characterize the size, crystal structure, elemental composition and a variety of other physical properties of nanoparticles. In several cases, there are physical properties that can be evaluated by more than one technique. In this work we are concerned with UV – visible spectroscopy, IR spectroscopy, Scanning Electron microscopy (SEM), X-Ray Diffraction Technique (XRD). The absorbance spectra of sample were measured in wavelength within the range from 200-900 nm using a UV-Vis UVD-3500 spectrophotometer. Particle size of magnetic iron oxide nanoparticles was measured using SEM method. Structure and crystalline size of nanoparticles were determined by XRD. The functional groups of nanoparticles were recorded by FT-IR spectroscopy (Perking Elmer), with a scan range from 4000 to 400 *cm*⁻¹.

3.1. UV-VISIBLE SPECTRAL ANALYSIS

UV-Vis analysis is one of the most important characterization methods to study nanoparticles. The absorption of visible radiations due to the excitation of SPR, imparts various colours to nanoparticles. As the nanoparticles size changes, colour of the solution also changes. So UV-Vis absorption spectrum is quite sensitive to the formation of nanoparticles. The nanoparticle sample (Green method) is subjected to UV-Vis study. The nanoparticles synthesized by green method shows maximum absorption at 370 nm confirming that the synthesized product was iron oxide nanoparticles [11]. Iron oxide nanoparticles have been reported to exhibit a characteristic broad absorption band in the range of 330-400 nm. The spectra recorded for the samples synthesized by green method also shows an absorption band in the range of 330-360 nm, in addition to a small band around 400-450 nm that may be due the components present in the plants.

3.2.. INFRARED SPECTROSCOPY

The prepared iron oxide nanoparticles were subjected to FT-IR spectroscopy measurements. It was recorded by using a Perking Elmer, spectrometer in the range 400-4000 *cm*⁻¹. It was used to identify the possible biomolecules responsible for the reduction and capping of the nanoparticles.

Fig.1 shows the four strong bands around 3421 *cm*⁻¹ (br), 1633 *cm*⁻¹ (m), 1082 *cm*⁻¹ (m) and 468 *cm*⁻¹ (m). These four bands are also related to those reported for Fe₂O₃ [12]. The vibration bands are 468 *cm*⁻¹ (Fe-O), 1633 *cm*⁻¹ (H₂O bending vibration) and a broad peak at 3421 *cm*⁻¹ (H₂O str) [13,14].

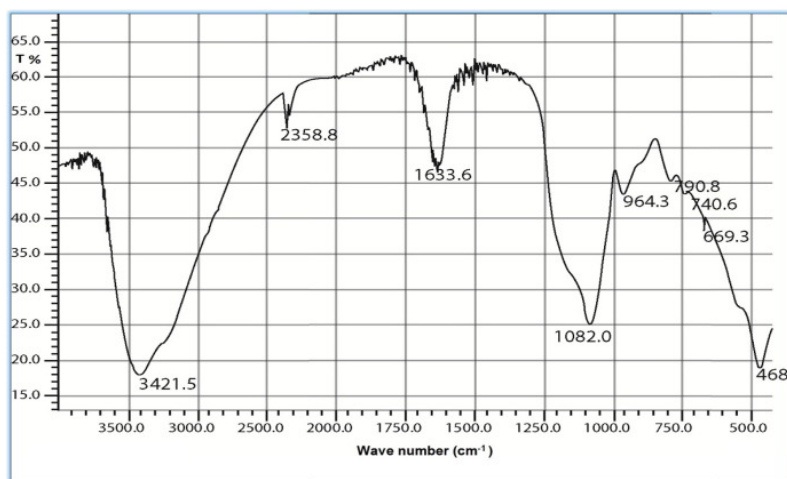


Fig.1 : IR Spectrum of nano particles

3.3. SEM ANALYSIS

The morphological dimensions of synthesized iron oxide nanoparticles were studied using the SEM. The image of SEM represents spherical morphology of iron oxide NPs. Mostly the NPs are in sphere form, except for few cubic in shape. The SEM also reveals that nanoparticles are agglomerated, which is due to the solution form of the sample and in-proper drying. The particles have a narrow size distribution with an average size of 10 nm (Fig. 2).

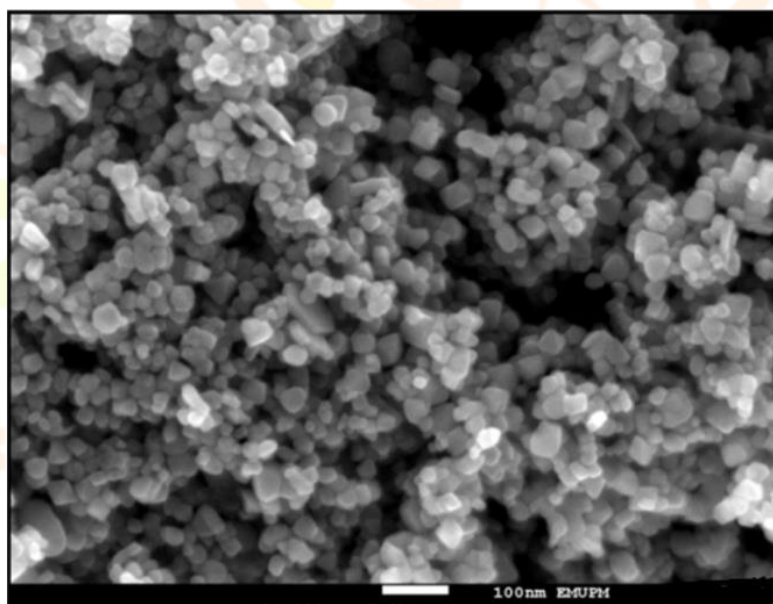


Fig 2: SEM images of **Fe₂O₃** nanoparticles synthesized from green method

3.4. X-RAY DIFFRACTION (XRD)

The samples prepared were subjected to X-ray Diffraction studies. Fig. 6 shows the XRD pattern of green synthesized Fe₂O₃ nanoparticles. The diffractions peaks could be indexed as that of cubic structure of Fe₂O₃ phase having lattice parameter $a=8.39\text{\AA}$. The particles size was calculated from the XRD data using Scherrer's equation $D = k\lambda/\beta\cos\theta$ where D is the particle size of the crystals, k is Scherer constant with value from 0.9 to 1, β is the full width at half-maximum value of XRD diffraction lines, λ is the X-ray wavelength and θ is the angle of Bragg diffraction. X-ray diffraction patterns for the iron oxide NPs synthesized using *Barringtonia acutangula* extract is shown in Fig.3. As found that there exist diffraction peaks with 2θ values of 30.2° , 36.5° , 38.2° , 42.91° , 53.8° , 57.3° , 63.21° , 71.9° and 73.78° , corresponding to the crystal planes of (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1), (4 4 0), (6 2 0) and (5 3 3) of crystalline iron oxide NPs. All of the diffraction peaks can be indexed to the magnetite structure phase of iron oxide by comparison with the data from JCPDS card (77- 1545)16. It is found that the average crystallite sizes associated with XRD data ranged between 21 and 82 nm.

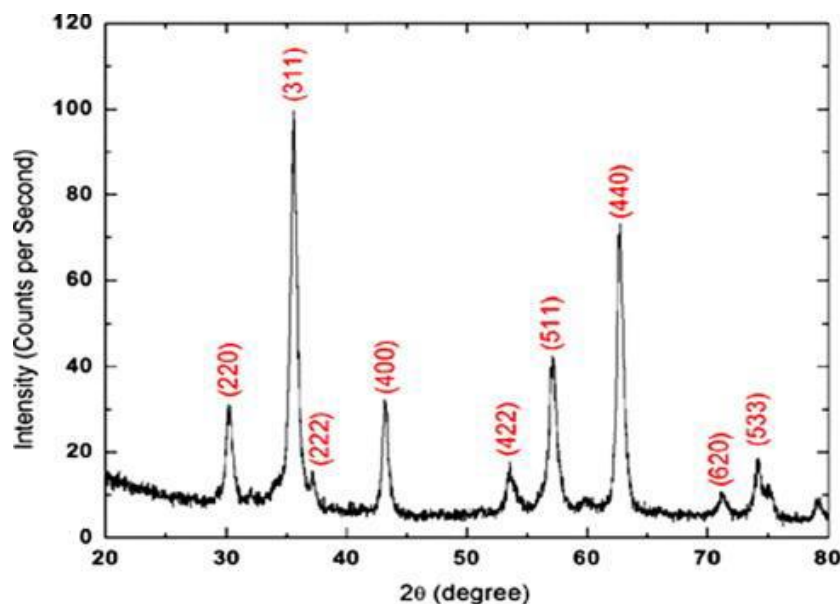


Fig 3: XRD pattern of green synthesized Fe_2O_3 nanoparticles

4. CONCLUSIONS

Iron oxide nanoparticles were prepared using the green synthesis method, using the extract of the saffron plant. Observing by eye, the change in the solution's color from light brown to dark brown indicates the formation of nanoparticles, and when it is deposited on a glass slide, it turns red, which is the colour of (Fe_2O_3). As the nanoparticles size changes, colour of the solution also changes. So UV-Vis absorption spectrum is quite sensitive to the formation of nanoparticles. The X-ray diffraction analysis confirmed that iron oxide is of type (Fe_2O_3) and that the data are identical to what is in the standard file (JCPDS card), which belongs to iron oxide. SEM result showed that the iron oxide particles were in the nano field and of high purity. Fourier spectroscopy showed the functional groups and the strength of chemical bonds of iron oxide particles.

ACKNOWLEDGMENT

The authors are grateful to the Department of Chemistry, N.S.S. College, Pandalam for providing the infrastructure to carry out the study.

REFERENCES

- [1] Khin M.M. , Nair A.S. , Babu V.J. , Murugana and S. Ramakrishna R., A review on nanomaterials for environmental remediation Energy Environ. Sci., 5 (2012), pp. 8075-8109
- [2] Zou Y. , Wang X. , Khan A., Wang P. , Liu Y., Alsaedi A., Hayat T. , and Wang X., Environmental remediation and application of nanoscale zero-valent iron and its composites for the removal of heavy metal ions: a review, Environ. Sci. Technol., 50 (14) (2016), pp. 7290-7304
- [3] Shameli K., Ahmad M.B, Zamanian A., Sangpour P., Shabanzadeh P., Abdollahi Y., Zargar M; "Green biosynthesis of silver nanoparticles using Curcuma longa tuber powder", Int. J. Nanomed. 7, (2012) 5603–5610
- [4] Laurent S, Boutry S, Mahieu I, Elst L Vander and Muller R N 2009 Iron Oxide Based MR Contrast Agents: from Chemistry to Cell Labeling Curr. Med. Chem. 16, 4712–27
- [5] Sharma R, Sharma A and Chen C J 2011 State of art on bioimaging by nanoparticles in hyperthermia and thermometry: Visualization of tissue protein targeting Open Nanomed. J. 3, 10–23
- [6] Souiri M, Gammoudi I, Ben Ouada H, Mora L, Jouenne T, Jaffrezic-Renault N, Dejous C, Othmane A and Duncan A C 2009 Escherichia coli-functionalized magnetic nanobeads as an ultrasensitive biosensor for heavy metals
- [7] Neuberger T, Schöpf B, Hofmann H, Hofmann M and Von Rechenberg B 2005, Superparamagnetic nanoparticles for biomedical applications: Possibilities and limitations of a new drug delivery system J. Magn. Magn. Mater. 293, 483–96
- [8] Hu J, Chen G and Lo I M C 2005 Removal and recovery of Cr(VI) from wastewater by maghemite nanoparticles Water Res. 39 4528–36

- [9] Salgado P., Márquez K., Rubilar O., Contreras D., and Vidal G., “The effect of phenolic compounds on the green synthesis of iron nanoparticles (Fe₃O₄-NPs) with photocatalytic activity,” *Applied Nanoscience*, vol. 9, pp. 371–385, 2019.
- [10] Bhuiyan S. H., Miah M. Y., Paul S. C. et al., “Green synthesis of iron oxide nanoparticle using *Carica papaya* leaf extract: application for photocatalytic degradation of remazol yellow RR dye and antibacterial activity,” *Heliyon*, vol. 6, Article ID e04603, 2020.
- [11] Behera S.S., Patra J.K., Pramanik K., Panda N. and Thatoi H. “Characterization and evaluation of antibacterial activities of chemically synthesized iron oxide nanoparticles” *World J Nano Sci Eng*, 2 (2012), p. 196
- [12] Kumar A. and Singhal A. “Surface modulation of magnetic nanocrystals in the development of highly efficient magnetic resonance probes for intera cellular labelling” *J Am Chem Soc* (2007), pp. 352-362
- [13] Haiss W., Nguyen T. K., Aveyard T. J., and Fernig D. G., “Determination of size and concentration of gold nanoparticles from U.V./vis spectra,” *Analytical Chemistry*, vol. 79, no. 11, pp. 4215–4221, 2007.
- [14] Demirezen D. A., Yilmaz S., and Yilmaz D. D., “Green synthesis and characterization of iron nanoparticles using aesculus hippocastanum seed extract,” *International Journal of Advances in Science Engineering and Technology*, vol. 62 pages, 2018.
- [15] Ansari S. A., Azam A., and Naqvi A. H., “Structural and morphological study of Fe₂O₃ nanoparticles,” *Asian Journal of Research in Chemistry*, vol. 4, no. 10, pp. 1638–1642, 2011.

