International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 08 Issue: 02 | Feb 2021 www.irjet.net p-ISSN: 2395-0072

# GREEN SYNTHESIS AND CHARACTERIZATION OF IRON OXIDE NANOPARTICLES USING HIBISCUS ROSA-SINENSIS LEAF EXTRACT

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**Abstract -** *Synthesis of metallic nanoparticles was performed by variety of physical and chemical methods. However, these methods may use toxic chemicals and are harmful to the environment. Recently, biological or green synthesis of nanoparticles (NPs) received enormous attention over the physical and chemical synthesis, as it is clean, non-toxic and an eco-friendly approach. Present research work was focused on the synthesis of Iron oxide nanoparticles using Hibiscus Rosa-Sinensis (HR) leaf extract as a plant resource. The collected samples are preprocessed by drying, grinding and boiling with distilled water to obtain the HR extract. The $Fe_3O_4$-NPs were synthesized by adding 0.01 M $FeCl_3.6H_2O$ solution to the HR extract in 1:1 volume ratio. The synthesized $Fe_3O_4$-NPs were tested by using UV-Visible spectrum. The average particle size of synthesized NPs was 45 nm as estimated by Zeta potential analyzer. The zeta potential of $Fe_3O_4$-NPs was -30 meV, which indicated that the synthesized NPs are highly stable due to the strong negative surface charge. The various functional groups in the bio-synthesized NPs were identified at 3440 $cm^{-1}$ (-OH bond), 1633 $cm^{-1}$ (C=O) and 533 $cm^{-1}$ ($Fe_3O_4$-NPs) by FTIR analysis. The pore size of surfaces on samples were characterized by surface area analyzer was 4.5 nm. The structural and Morphological features of synthesized NPs were characterized using XRD, AFM and SEM analysis. The present work will be used to provide an alternative approach for treating petroleum pollutants in an ecofriendly, economic and efficient mode.*

**Keywords:** Hibiscus rosa-sinensis, Co-precipitation method, Iron Oxide nanoparticles.

## 1. INTRODUCTION

Nanotechnology is the ability to measure, see, manipulate and manufacture things on an atomic or molecular scale, usually between one and 100 nanometers. These tiny products also have a large surface area to volume ratio, which is their most important feature responsible for the widespread use of nanomaterial in mechanics, optics, electronics, biotechnology, microbiology, Synthesis of metallic nanoparticles was performed by a variety of physical and chemical methods.

## 2. MATERIALS AND METHODS

In this paper, it is involved that the fresh leaves of *Hibiscus rosa-sinensis* was collected from the GCT campus, Coimbatore. The leaves of the plant were used for extract preparation. Each leaves were thoroughly washed several times with distilled water. Then dried in shades, cut into pieces, ground and powdered

### 2.1 Preparation of Hibiscus leaf extract

The leaf extract was prepared by taking 5 g of thoroughly washed, air dried and finely ground *Hibiscus* leaves in a 250 ml Erlenmeyer flask with 100 ml of deionized water and then boiling the mixture continuously for 15 min(as shown in fig 1). The extract was filtered using Whatman Filter Paper No.1 and the filtered extract was then stored at room temperature.

**Fig 1**: Boiling of samples

### 2.2 co-precipitation method

In this study, $Fe_3O_4$ nanoparticles were synthesized by co-precipitation method. Iron oxide nanoparticles were synthesized by adding 0.01 M $FeCl_3 \cdot 6H_2O$ solution to the HR extract in 1:1 volume ratio. $Fe_3O_4$-NPs were immediately obtained with the reduction

process. Then the mixture was stirred for 60 min and then allowed to stand at room temperature for another 30 min to obtain colloidal suspension. After the complete, addition of NaOH, the solution was added to increase the pH of the solution. The mixture was centrifuged and washed several times with ethanol and then dried at 40 °C under vacuum to obtain the $Fe_3O_4$-NPs. Finally, reddish brown precipitate was formed, it was filtered and calcined for 24 hours at 80 degree Celsius. Thus iron oxide nanoparticles were obtained. After drying those precipitated particles with drying oven at 80 degree Celsius for 12 hours, the dried $Fe_3O_4$-NPs were calcinated in muffle furnace.

## 3. CHARACTERISTICS

The synthesized nanoparticles were characterized by using

- Zeta potential and particle size analyzer
- Surface area analyzer
- FTIR analyzer
- Scanning Electron Microscope (SEM)
- UV-Vis Spectroscopy
- X-ray Diffraction(XRD)
- Energy dispersive X-ray Spectroscopy
- Atomic Force Microscopy

## 4. RESULT AND DISCUSSION

The synthesized $Fe_3O_4$ -NPs were subjected to various characterization studies to understand the specific properties such as optical, structural, morphological, elemental composition, particle size, functional groups studies which could be made precisely using sophisticated techniques such as UV-Visible spectroscopy, Zeta size, FT-IR, XRD, AFM, surface area analysis and SEM instruments. These techniques were helpful to verify our method is well optimized and meeting the requirements.

### 4.1 UV-Visible spectroscopy

The phytochemicals include hydroxyl, carboxyl, and amino functional groups, which can serve both as effective metal-reducing agents and as capping agents to provide a robust coating on the metal nanoparticles in a single step and leads to the colour change Yellow to brown. This colour change gave the confirmation of the synthesis of $Fe_3O_4$ -NPs. The synthesized iron oxide nanoparticle was confirmed using UV-Visible spectrum

### 4.2 Zeta potential and particle size analysis

The molecular size, particle concentration, particle size and zeta potential were determined.

The average size of the Iron oxide nanoparticle is 45 nm. The zeta potential of $Fe_3O_4$-NPs was -30 meV and 30 meV proved that synthesized NPs are highly stable due to the strong negative surface charge.

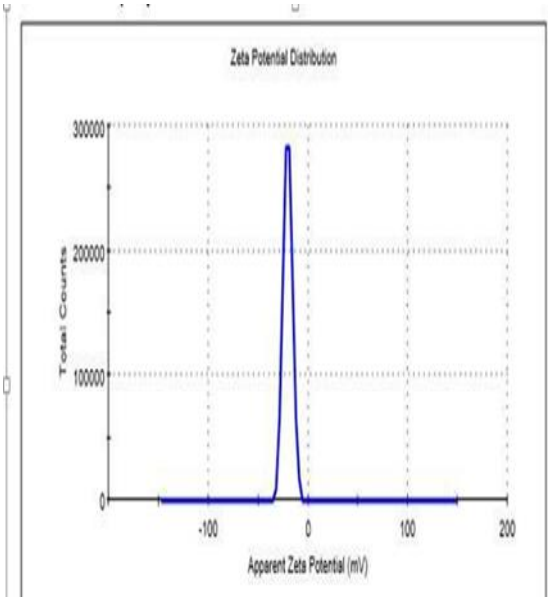


**Fig 2**: Particle size analysis

### 4.3 *XRD analysis*

The X-ray diffraction patterns obtained for the $Fe_3O_4$ -NPs synthesized using HR extract. It is found that the strong diffraction peaks with 2θ values of $28.26^0$, $32.28^0$ Using the Scherer equation the average crystallite sizes of the $Fe_3O_4$ -NPs are found to be in the range of 14 nm - 18 nm. The results indicated that all the nanoparticles were in spinel structure with face- centered cubic phase

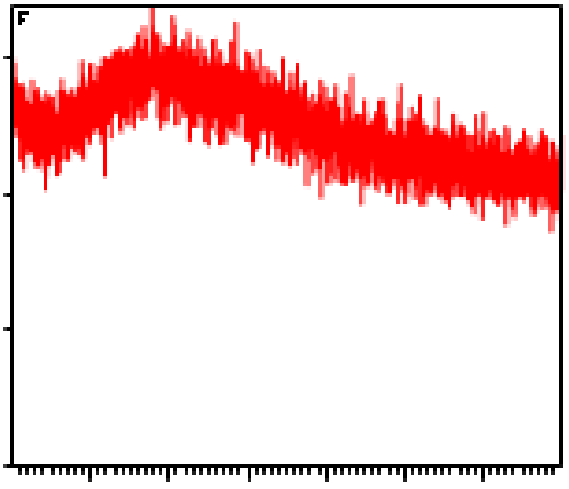


**Fig 3**: XRD Analysis

### 4.4 FTIR Analysis

FT-IR analysis gave the stretching vibrations at 3444 $cm^{-1}$, 1633 $cm^{-1}$and 533 $cm^{-1}$with in the region of 400 - 4000 $cm^{-1}$. These peaks represent the following bonding in the sample confirms the reducing agent role in the formation of $Fe_3O_4$ -NPs. The peak at 3444 $cm^{-1}$ corresponds to the -OH bond stretching denotes the aqueous phase as well as the reduction of the Ferric chloride, 1633 $cm^{-1}$ corresponds to the C=O bond stretching denotes the phytochemicals present in the plant extract and amino acids which stabilize as well as act as a capping agents. The strong peak at 533 $cm^{-1}$corresponds to the inorganic stretching indicates the $Fe_3O_4$ -NPs. Further, the strong vibration was observed at 466 $cm^{-1}$ corresponds to Fe-O vibration.

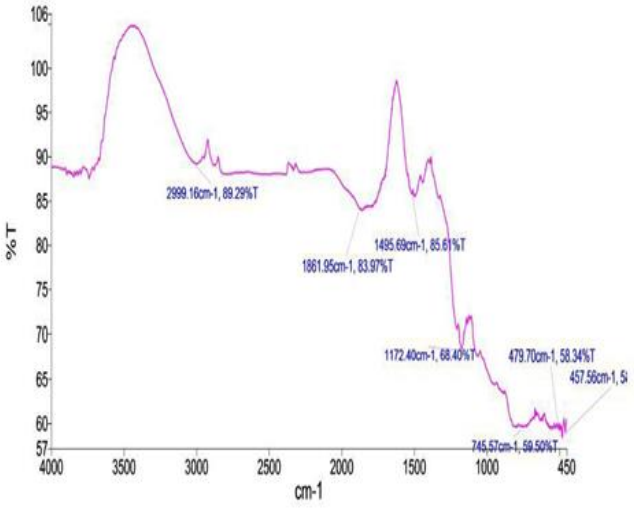


**Fig 4:** FTIR Analysis

### 4.5 SEM Analysis

Formation of $Fe_3O_4$ -NPs and its morphological dimensions were studied using the SEM. The observations demonstrated that the average size of the NPs was in the range of 30 nm – 100 nm similar phenomenon was reported in the previous studies. That also exhibits the formation of needle shape of iron nanoparticles as. The needle shaped nanoparticles formation has induced by phytochemicals present in the extract have an influence in the morphology of the nanoparticles. Due to the very narrow electron beam, SEM micrographs have a large depth of field yielding a characteristic three dimensional appearance useful for understanding the surface structure of a sample.

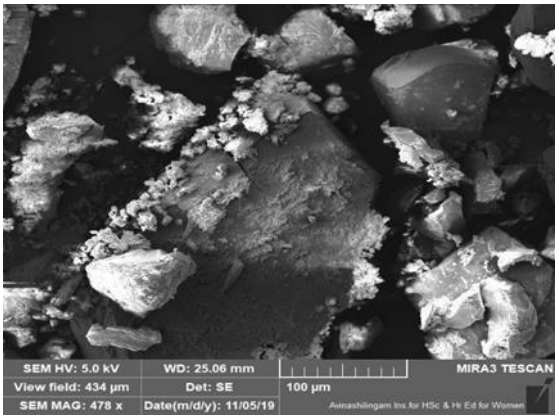


**Fig 5**: SEM Analysis

### 4.6 AFM Analysis

The structure of biological molecules and cellular components are analyzed by using Atomic Force Microscope. The morphology and the average size of $Fe_3O_4$ - NPs were evaluated by atomic force microscopy (AFM). Topography and phase contrast images from different regions over the sample surface were obtained. The nanoparticles are spherical in shape with average diameter of 31.2 nm.

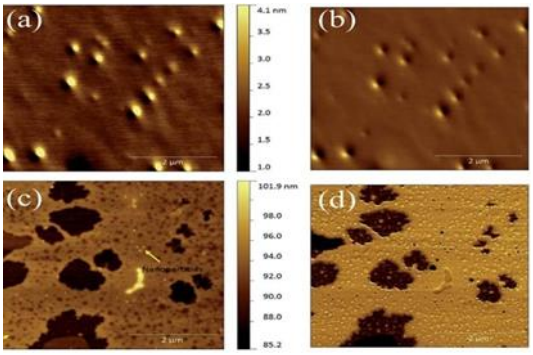


**Fig 6**: AFM Analysis

### 4.7 Surface Area Analysis

The specific surface area and porosity of the Iron Oxide were determined by using the $N_2$ adsorption and desorption isotherm. The isotherm of the sample reveals the step wise adsorption and desorption plot of type IV isotherm, typical mesoporous materials. A hysteresis loop with a step wise adsorption and desorption is observed at wide range of pressure. From the BET graph, plot the surface area of the Iron oxide adsorbent was observed to be 3.14 $m^2$/gm. In addition, the pore volume and pore diameter was measured to be 0.9 $cm^3$/g and 45 nm respectively.

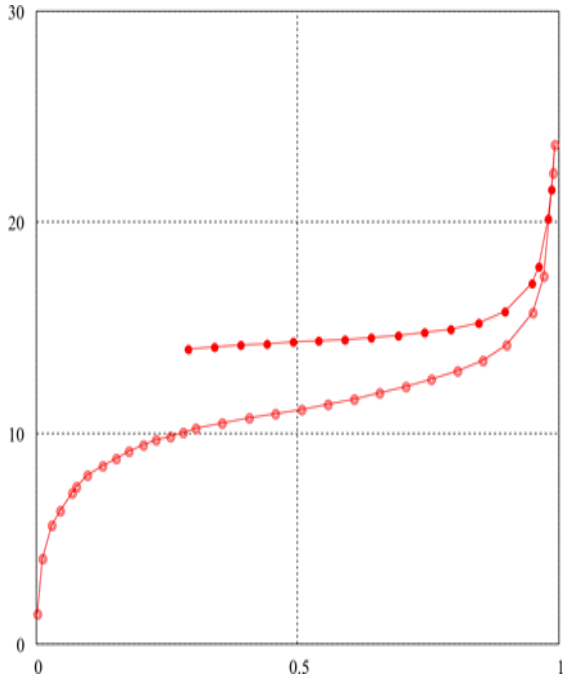


**Fig 7**: Surface area analysis

## 5. CONCLUSION

The Iron Oxide ($Fe_3O_4$) nanoparticles were synthesized by co-precipitation method. In this method, ferric hexahydrate was added with the leaf extract. The mean diameter of the $Fe_3O_4$ nanoparticle was found to be 45 nm. The zeta potential of $Fe_3O_4$ -NPs was -30 meV, which indicated that the synthesized NPs are highly stable due to the strong negative surface charge. The various functional groups in the bio-synthesized NPs were identified at 3440 $cm^{-1}$ (-OH bond), 1633 $cm^{-1}$ (C=O) and 533 $cm^{-1}$ ($Fe_3O_4$ -NPs) by FTIR analysis. The pore size of surfaces on samples were characterized by surface area analyzer was 45 nm. The optical characterization was determined by UV-VIS spectrum. The maximum peak obtained (λmax) is 335.4 nm at 0.9 (A°). The morphology and chemical composition were characterized by SEM and XRD. The present work will be used to provide an alternative approach for treating pharmaceutical pollutants in an ecofriendly, economic and efficient mode. The treatment efficiency of the $Fe_3O_4$ was determined using the photo-catalytic reaction under the UV light condition.

## 6. REFERENCES

1) Christian, P.; Von der Kammer, F.; Baalousha, M.; Hofmann, T. Nanoparticles: Structure, properties, preparation and behaviour in environmental media. Ecotoxicology 2008, 17, 326–343.
2) Osaka T, Matsunaga T, Nakanishi T, Arkaki A, Niwa D, Iida H. Anal Bioanal Chem 2006, 84, 593–600.
3) Song HT, Choi JS, Hum YM, Kim S, Jun YW, Suh JS, Suh J. Surface, modulation of magnetic nanocrystals in the development of highly efficient magnetic resonance probes for intera cellular labeling. J Am Chem Soc 2005, 9992–3.
4) Ankamwar B, Chaudhary M, Mural S. Synth React Inorg Met-Org Nano-Met Chem 2005, 35, 19–26.
5) Osaka T, Matsunaga T, Nakanishi T, Arkaki A, Niwa D, Iida H. Anal Bioanal Chem 2006, 384, 593–600.
6) Tyner KM, Schiffman SR, Giannelis EP, Nanobiohybrids as delivery vehicles for camptothecin. J Control Release., 2004, 95, 501-514.
7) Gupta AK, Gupta M, Synthesis and surface engineering of iron oxide nanoparticles for biomedical applications. Biomater 2005,
8) Faraji M, Yamini Y, Rezaee M, Magnetic nanoparticles: synthesis, stabilization, functionalization, characterization, and applications. J Iran Chem Soc., 2010, 7, 1-37.
9) Horváth E, Oszlánczi G, Máthé Zs, Szabó A, Kozma G, Sápi A, et al. 2011. Nervous system effects of dissolved and nanoparticulate cadmium in rats in subacute exposure. J Appl Toxicol., 2011, 31, 471-476.
10) Mittal AK, Chisti Y, Banerjee UC. Synthesis of metallic nanoparticles using plant extracts. Biotechnol Adv. 2013, 31(2), 346–356.
11) Kumar V, Yadav SK. Plant-mediated synthesis of silver and gold nanoparticles and their applications. J Chem Technol Biotechnol. 2009, 84, 151–157.
12) Kharissova OV, Rasika Dias HV, Kharisov BI, et al. The greener synthesis of nanoparticles. Trends Biotech., 2013.