IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)
An International Open Access, Peer-reviewed, Refereed Journal

GREEN SYNTHESIS OF IRON NANOPARTICLES USING LEAVES EXTRACT AND THEIR CHARACTERIZATION ANALYSIS

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Abstract: Green synthesis of iron nanoparticles being cost effective and eco-friendly treatment technique, is gaining importance nowadays. The aim of the present study is to prepare leaf extracts, precursor and synthesis of iron nanoparticles. The green synthesis was done by using the leaves extract of abutilon indicum and plumeria alba grinded with Ferric chloride separately to synthesis Fe nanoparticles. The structural properties of the synthesized nanoparticles were investigated through advanced technique like Fourier Transform Infrared Spectroscopy (FT-IR), X-Ray Diffraction (XRD) and Particle Size Analysis (PSA). The Iron nanoparticles were synthesized by co-precipitation method using Ferric chloride precursor. The FT-IR analysis shows the interaction between the capping agent and the synthesized iron nanoparticle. The PSA results shows the nanoparticles was monitored in the method of dynamic light scattering indicate the average size of iron nanoparticles that was calculated by 141 nm and 614.6 nm. The XRD spectral analysis reveals the synthesized particles have orthorhombic phase with particle size in the 56 nm and 42 nm.

Index Terms **- Plant extract, abutilon indicum, plumeria alba, ferric chloride, nanoparticle.**

I. INTRODUCTION

Recently the Green synthesis of nanoparticles using plant extracts has created the attention of researchers. The progress of green chemistry in the synthesis of nanoparticles using plant has attracted a great attention nowadays due to its low cost, simple, non-toxic and environmental-friendly nature when compared to physical and chemical method. Recent advancement of green synthesis iron nanoparticle was using of vegetables, fruits, and organic waste extracts by mixing certain proportion of an iron compound without energy and any toxic chemical requirements [1]. Furthermore, using plant extracts as reducing agent offer suitability for biomedical and pharmaceutical applications since no toxic chemicals are used during synthesis [2]. Green Iron nanoparticles synthesis using plant extract is a very effective synthesis process at a very reasonable cost. Recently, many reports have discussed the successful synthesis of iron nanoparticles via green chemistry [3-4].

Nanotechnology is a reliable and enabling environment-friendly process for the synthesis of nano scale particles. Nano-size results in specific physicochemical characteristics such as high surface area to volume ratio, which potentially results in high reactivity [5]. It is multifaceted area, being considered as a novel and potential field of 21st century that deals with fabrication of materials at nano scale level. At present much ongoing advancement at nano scale level involves that nanotechnology will definitely have a very fascinating role in numerous main technologies, reorganization and exploitation of the materials composition in the range of 1 nm to 100 nm.

There are various studies that describe the synthesis of nanoparticles using plants. The biosynthesis of iron nanoparticles from Amaranthus dubius was reported [6]. Results showed the spherical surface structure of nanoparticles, size ranged from 43-220 nm. Due to the diverse applications in different areas green synthesized nanoparticles are receiving much interest. Leaf extract of Gardenia jasminoides and Lawsonia inermis were used to synthesize iron nanoparticles. Resulted nanoparticles were characterized through different characterization techniques [7].

Aim of the present study is to synthesize iron nanoparticles from the leaves extract of Abutilon Indicum and Plumeria Alba and developing potential green synthesized iron nanoparticles. Characterizations like PSA and XRD were employed to analysis the as-prepared material in terms of size, composition and structure. Furthermore, based on the FTIR analysis, a possible synthesis mechanism was proposed. Finally, they were used for the treatment of swine wastewater to evaluate their reactivity.

II. MATERIALS AND METHODS

Abutilon Indicum and Plumeria Alba leaves are collected from the rural areas of Madurai district, Tamilnadu, India. Ferric chloride and double distilled water of AR grade were purchased from Nice Chemicals Pvt. Ltd., Kerala, India. The collected leaves of Abutilon Indicum were cleaned using normal tap water and double distilled water. Then the cleaned leaves are cut into small pieces and dried for 15 minutes. The dried leaves are taken for weighted using in digital balance. Putting on 15g of dried leaves and add 100ml of double distilled water to boil in the range of 80^0c using boiler. The leaves are boiled well the water colour changes into light yellow. After the change of colour take the mixed water and using Whatman grade 1 filter paper to filter and get the leaves extract. Similarly this procedure is repeated for another leaves (Plumeria Alba).

For iron extract, first we take 5g of Ferric Chloride ($FeCl_3$) using digital balance and put in the beaker add 50 ml of double distilled water then mixed well. In magnetic stirrer the iron solution is stirred. The 25ml of both leaves extract is added in the iron solution were kept in the magnetic stirrer and half hour (30 minutes) stirred well. During the stirrer the leaves extract colour will changes into dark brown colour. After 30 minutes take the leaves extract and kept 5minutes at room temperature. Then the leaves extract is kept in the muffle furnaces for 15 to 20 minutes and get the iron nanoparticles.

III. RESULTS AND DISCUSSION

FTIR spectral analysis

Fourier Transform Infrared spectra analysis indicate the presence of functional groups in the Abutilon Indicum and Plumeria Alba leaves extract were responsible for the reduction and stabilization of Iron (Fe) nanoparticles. The frequency of the FTIR spectrum is recorded in the range of 4000 cm^{-1} to 400 cm^{-1}. The FTIR spectra for the two samples extract and synthesized Fe nanoparticles are shown in **Fig. 1 and 2**. The vibrational bands of analyzed Fe nanoparticles in the leaves extract of Abutilon Indicum and Plumeria Alba are 650.93 cm^{-1}, 689.5 cm^{-1}, 1624.92 cm^{-1}, 2337.56 cm^{-1}, 3381.95 cm^{-1} (sample 1) and 557.39 cm^{-1}, 668 cm^{-1}, 1623.95 cm^{-1}, 2360.71 cm^{-1}, 3380.02 cm^{-1} (sample 2). The two broad absorption peaks of the both samples in the region from 3381.95 cm^{-1} and 3380.02 cm^{-1} denotes the strong O-H stretching group vibration of water [8]. Then absorption bands of 2337.56 cm^{-1} and 2360.71 cm^{-1} represent the CO_2 stretching vibration [8] which was relieved from the extract leaves. The strong peaks at 1624.92 cm^{-1} and 1623.95 cm^{-1}, respectively have been assigned to C=C conjugated alkene stretching vibrations and C=O stretching vibrations of ketone group [8].

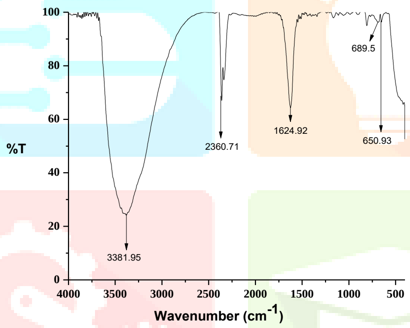

Fig. 1: FTIR spectrum of sample 1

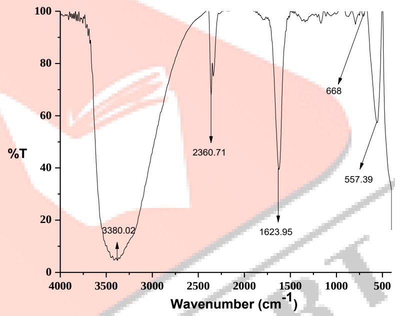

Fig. 2: FTIR spectrum of sample 2

The vibrational band at 557 cm^{-1} is correlated to the Iron Oxide (Fe_3O_4) nanoparticles [9]. The strongest band at 466 cm^{-1} was assigned to Fe-O stretching vibration of Fe nanoparticles [9]. According to Butt et. al [10], the vibrational band at 670 cm^{-1} confirms the Fe vibrations of synthesized nanoparticles. The band at 612.63 cm^{-1}, has been assigned from the analysis of FTIR spectral study leads to the conclusion that the fingerprint film consist of the Fe vibration [7]. The appearance of novel peak at less than 700 cm^{-1} for the ferric nanoparticle spectra which was correspond to the Fe-O vibrations of iron oxide [7]. Alshehri et al. [11], the two strongest absorption bands at 638 and 575 cm^{-1} which correspond to Fe-O vibrations and this peak was not observed in the parent compound of the Fe nanoparticle. So they [11] confirm the presence of the Fe nanoparticle in leaves extract. So in this present investigation the peaks at 650.93 cm^{-1}, 689.5 cm^{-1} and 688 cm^{-1} are attributed the Iron (Fe) nanoparticles as reported in literatures [9-11]. The stretching vibrations of O-H, C=O, C=C and Fe-O confirms the presence of the ferric nanoparticle in leaves extract [12]. Hence in this FTIR spectra are also confirmed the presence of the Fe nanoparticle from Abutilon Indicum and Plumeria Alba leaves extract.

Particle size spectral analysis

The size distribution and average particle size diameter of the synthesized iron nano particles was characterized through nano laser particle size analyzer (PSA). Dynamic light scattering or photon correlation spectroscopy is a technique used in material physics for measuring the size distribution of nano particles in suspension or polymers in solutions. The Abutilon Indicum and Plumeria Alba leaves extract was indicate the most suitable for generation of Fe nanoparticles. In **Fig. 3** and **Fig. 4** represents the particle size distribution analysis peaks of Fe nanoparticles synthesized by Abutilon Indicum and Plumeria Alba leaves extract. It is observed that the average diameter of iron (Fe) nanoparticles where within a range of 141 nm (sample 1) and 614.6 nm (sample 2). According to Butt et al. [10], the particle size result shows the average diameter of the iron nanoparticles was 266 and 108 nm. This result clearly indicates the plant extract concentrations deeply affect the size of the iron nanoparticles (NPs). The size distribution of the Fe NPs was probably due to the fact that non-thermal extracts in water contain various naturally derived compounds with different reducing properties and mean sizes of particles, variations in distribution, refractive index of the solvent used and nature of the dispersive medium used for characterization techniques [10]. So in this present investigation the average size of iron particles of both the samples are higher than the normal iron nanoparticles. This may due to the leaf extracts, bio-molecules affect the size of the nanoparticles and the various properties also influenced the size distribution of iron nanoparticles which was reported in literatures [10-12].

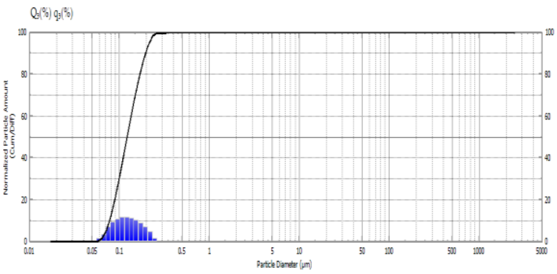

Fig. 3: Particle size analysis graph of sample 1

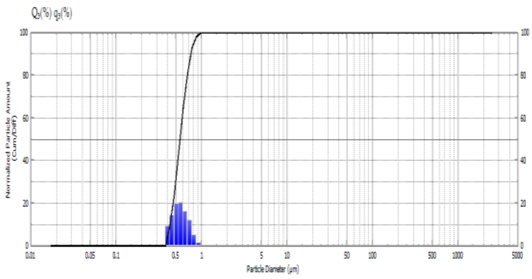

Fig. 4: Particle size analysis graph of sample 2

XRD spectral analysis

The X-Ray Diffraction technique was to identify the structure of Iron nanoparticles. The Green Synthesized Iron nanostructure by employing Abutilon Indicum and Plumeria Alba leaves extract was further demonstrated. A series of X-ray diffraction patterns were recorded in the range of 10-700. The XRD pattern indicates for the Fe nanoparticles stabilized using Abutilon Indicum and Plumeria Alba leaves extract shown in **Fig. 5 and Fig. 6**. The XRD analysis represent three intense diffraction patterns peaks in the whole spectrum of 2θ values at 32.266, 37.363, 43.121 (sample1) and 33.216, 35.709, 54.155 (sample2). The systematic planes having corresponding hkl values are (2 2 0), (2 2 2), (4 0 0) for sample1 and (1 0 4), (3 1 1), (4 2 2) for sample2 are indexed as cubic (JCPDS file no. 89-0950). The average crystallite size (D in nm) of zinc oxide nanoparticles was determined from XRD pattern from diffraction peak full width at half maximum (FWHM) according to the Debye - Scherer equation:

$$D = K\lambda/\beta cos\theta$$

Where K is the Scherer constant (0.9), it can be affected with the lattice direction and crystallite morphology. λ is the wavelength of the target Cu-Kα = 1.54060 Å. β refers to the peak width at half-maximum (FWHM) which determined in radians. θ is the diffraction angle. By using Debye-Scherer equation, the average crystalline size D of synthesized nanoparticles was calculated to be 56 nm for sample 1 and 42 nm for sample2 are shown in **Table 1** and **Table 2**.

Table 1: XRD data for sample 1

2θ (degree)	FWHM (β)	Crystalline Size (nm)	Plane
32.266	0.255	32.431	(2 2 0)
37.363	0.148	67.536	(2 2 2)
43.121	0.155	70.219	(4 0 0)
Average Size		56.728	

Table 2: XRD data for sample 2

2θ (degree)	FWHM (β)	Crystalline size (nm)	Plane
33.216	0.193	49.201	(1 0 4)
35.709	0.219	44.675	(3 1 1)
54.155	0.388	34.964	(4 2 2)
Average Size		42.945	

According to the reports of Alshehri et al. [11], the purity of the synthesized material was observed from the wide-angle XRD pattern, and peaks could easily be identified and corresponded to the pure cubic phase of Fe nanoparticles. Moreover the characteristic diffraction peaks were observed, which were assigned to the (220), (311), (400), (422), (511), and (440) Bragg reflections [10]. A broad hump appearing at about 2θ of 20°was observed, which could be bio-molecules forming a capping layer on the Fe NPs' surface resulting from the leaves extract [1]. The characteristic peak of zero-valent iron at at 2θ = 44.9° [6, 13]. There were two possible explanations for this result: that the content of Fe was insufficient, or that the nanoparticles were amorphous. So the iron nanoparticles were mainly amorphous in nature [13], which was consistent with the previously published data on synthetic nanoparticles using tea [14] and eucalyptus [15]. So in this present investigation, this can be interpreted by the fact that a thin layer of bio-molecules is capping on the iron nanoparticles surface in order to stabilize Fe NPs resistance to oxidation. And the content of Fe was insufficient, or that the nanoparticles were amorphous in nature which was discussed in literatures [12-15].

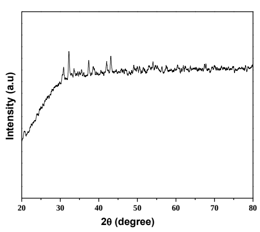

Fig. 5: XRD pattern of Fe nanoparticles of sample 1

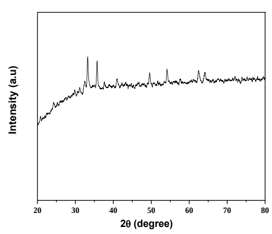

Fig. 6: XRD pattern of Fe nanoparticles of sample 2

IV. CONCLUSION

The rich biodiversity of leaves are due to the green world has potential for the synthesis of noble metal nanoparticles. The Green Synthesis of Iron (Fe) nanoparticles was carried out using Abutilon Indicum and Plumeria Alba leaves extract is available advantages of the natural reducing and capping ability of its compounds. The Fe nanoparticles are useful for nanotechnology applications. The formation Iron nanoparticle is characterized by technique such as FTIR spectroscopy, XRD and the particle size analysis methods. In FTIR spectra analysis result indicate the existence of Fe nanoparticles and stretches at different wavelengths capable of vibration due to functional groups were responsible in the process of Fe nanoparticles. The present stretching's O-H groups of water, CO_2, C=C conjugate alkene and C=O vibrations of ketone group, Iron Oxide and Iron nanoparticles. In the study of PSA, the mean size of Fe nanoparticles of the Abutilon Indicum and Plumeria Alba leaves extract are 141 nm and 614.6 nm. The XRD work represent, the average crystal of Iron nanoparticle was found to be 56 nm (sample 1) and 42 nm (sample 2). Overall these approaches are promising for the Green production of Fe nanoparticles.

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