International Journal Of Engineering Research And Development
e- ISSN: 2278-067X, p-ISSN: 2278-800X, www.ijerd.com
Volume 19, Issue 5 (Sep.-Oct. 2023), PP. 23-29

Synthesis of Iron Nanoparticles Using Moringa Oleifera Leaf Extract

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ABSTRACT
In this study, Iron nanoparticles (FeNPs) were synthesized using Moringa oleifera leaf extract as both reducing and stabilizing agent using green synthesis method and characterized using Ultraviolet (UV) spectroscopy, pH, Fourier Transform Infrared (FT-IR) spectroscopy. The determination of pH at different time interval shows a gradual decrease as a function of time and stabilizes from 14-24th hour and this confirm the stability of the nanoparticles. The UV spectroscopy results shows that the synthesized FeNPs varies in the range of 300-500nm. The FTIR spectrum reveal the functional groups responsible for the reduction and synthesis of the nanoparticles, this is obvious from the two spectra of biomass and Iron that the flavonoids and phenolic led to the bio-reduction. It can be concluded that iron nanoparticles synthesized using Moringa oleifera can be applied in medicine, as a curative agent and in domestic waste water treatment. The characterization results confirm the formation and presence of iron nanoparticles thus obtained is evaluated for simultaneous removal of total phosphates, nitrates and chemical oxygen demand.
Key words: *Iron nanoparticles; Moringa oleifera; Domestic waste water; Green synthesis*

Date of Submission: 10-09-2023 Date of Acceptance: 24-09-2023

I. INTRODUCTION

Oluwole Samuel Aremu et al.,2018 studied green synthesis of iron nanoparticles (FeNPs) using leaf and seed extracts of moringa oleifera were prepared by mixing different ratios of plant extracts with iron chloride solution. The batch adsorption results showed an enhanced removal of nitrate ion by 85% and 26% MOS-FeNPs and MOL-FeNPs respectively as compared to moringa extracts.

Carole Silveira et al.,2018studied andsynthesized iron-oxide nanoparticles via a green synthesis method, its adsorption capabilities and the shortest contact time to achieve equilibrium, the NPsFeo is a highly promising material for fluoride ion removal.

K.G.Ahila et al.,2018 investigated green synthesis of magnetic iron nanoparticle using moringa oleifera lam seeds and its application in textile effluent treatment.

T.Shahwan et al.,2011studied green synthesis of iron nanoparticles These indicates the complete removal of dyes from water over a wide range of concentrations, 10-200mgL-1, compared with iron nanoparticles produced by borohydride reduction.

C.P.Devatha et al.,2017studied the performance of COD removal(77%) and nitrate removal(74%) for 1:5 on 5th day is observed to be efficient. Size of FeNPs achieved for 1:2 ratio was 98-200 nm and for all proportions varying between 120 and 600 nm due to agglomeration enhanced bacterial decay. For the contact time of 48h, 1:5 found to be effective in inhibiting more number of bacterial cells compared to other proportions.

Crislaine Rodrigues Galan et al.,2018 investigated green synthesis of copper oxide nanoparticles impregnated on activated carbon using moringa oleifera leaves extract for the removal of nitrate from water.

S.Manishkanna et al.,2019 presented a facile biogenic synthesis of iron nanoparticles using aqueous extracts of three plant sources: Terminalia bellirica (TB); Moringa oleifera fruit (MOF) and Moringa oleifera leaves (MOL). The materials were characterized using TEM, SEM and XRD indicated particle size of 35-40 nm.

P.Karpagavinayagametal.,2018described green synthesis of iron oxide nanoparticles using Avicennia marina flower extract. UV-vis absorption spectrum of iron oxide nanoparticles display a peak in the region of 295-301 nm. The FTIR spectrum of Feo-NPs shows bands at 3354 cm-1,1630cm-1,1380cm-1 and 610cm-1.

Mohamed F.Alajmi et al.,2018 studied green synthesis of iron oxide nanoparticles using aqueous extracts of pandanus odoratissimus leaves for efficient bifunctional electro-catalytic activity. Iron oxide nanoparticles show uniform particle size distribution with an average diameter of ~5.0nm. Electrochemical water splitting reactions have been carried out using Fe_3O_4 NPs as electrocatalysts in 0.1 M KOH electrolyte solution.

S Amutha et al.,2018 studied the potential effect of Glycosmis mauritiana leaf extract for the formation of iron oxide nanoparticles and its application on antibacterial activity. The results revealed that iron oxide nanoparticles has the absorption peak at 404nm, spherical shaped and average size of particle is found to be below 100nm.

Thakur Deepa Singh et al.,2019studied green synthesis of iron, copper and sliver nanoparticles and their antibacterial activity on animal pathogens. The absorption values of Iron, copper and silver nanoparticles at 260nm, 340nm and 425nm respectively. The SEM analysis revealed the size of Iron, copper and silver nanoparticles as 58.9-78.0nm, 49.6-95.4nm and 63.6-88.8nm respectively.

Arun Kumar Thalla et al.,2016 investigated green synthesis of iron nanoparticles is done using various leaf extracts viz. Mangiferaindica, Murrayakoenigii, Azadiractaindica, The Among different plant mediated synthesized iron nanoparticles, Azadiractaindica showed 98.08% of phosphate, 84.32% of ammonia nitrogen and 82.35% of chemical oxygen demand.

K Sravanthi et al.,2018 presented synthesized zero-valent iron nanoparticles (ZVIN) using reproducible Calotropis gigantea (CG) flower extract served as both reducing and stabilizing agent completely by green approach. FT-IR and UV-Vis absorption spectra reveal that the polyphenols present in the CG flower extract responsible for the reduction and stabilization of the ZVIN.

Nasrin Beheshtkhoo et al.,2018 studied iron nanoparticles were synthesized by a simple bio-reduction method. The average diameter of the prepared NPs ranged from 6.5 to 14.9nm with a mean particle size of 9.2nm.

HelaleKaboliFarshchi et al.,2018 synthesized iron nanoparticles by rosemary extract and cytotoxicity effect evaluation on cancer cell lines. The mean size of the Rosemary-FeNPs were at about 100nm with PDI of less than 0.12, which indicates a homogeneous size distribution of the nanoparticles.

S Saranya et al.,2017 studied green synthesis of iron nanoparticles using aqueous extract of musaornata flower sheath against pathogenic bacteria. The optimum precursor salt concentration, pH of the reaction mixture, ratio between reducing agent and precursor salt and time for the synthesis of iron nanoparticles were found to be 5mM, 9.0, 3:7 and 0^{th} h respectively. X-ray diffraction method, it was found that the average particle size of magnetite nanoparticles was found to be 43.69 nm.

MallavarapuMegharaj et al.,2014 proposed that iron nanoparticles (Fe NPs) synthesized by green tea (GT-Fe) and eucalyptus leaves (EL-Fe) extracts, which regarded as cleaner productions can be used for the efficient removal of nitrate. Batch experiment showed that 59.7% and 41.4% of nitrate was removed by GT-Fe and EL-Fe NPs, compared to the 87.6% and 11.7% that was removed using zero-valent iron nanoparticles (nZVI) and Fe3O4 nanoparticles, respectively.

Monalisa Pattanayak et al.,2012synthesized iron nanoparticles using various plants and spices extract at room temperature. The color change occurred when ferric chloride was mixed with reducing agent i.e., plant and spices extract. The bioreduction of Fe3+ in aqueous solutions was monitored by periodic sampling of aliquots of the mixture.

Yufen Wei et al.,2016presented iron nanoparticles were synthesized using citrus maxima peel extracts to reduce Fe in aqueous solution. Based on the characterization results, irregular iron nanoparticles with diameters of 10-100 nm were synthesized.

Sara Hooshmand et al.,2016reported the synthesis and functionalization of magnetic iron nanoparticles using green chemistry for application of dispersive soli-liquid phase microextraction (DSLME) method for preconcentration and determination of nickel ions in soil, potato, red tea, white tea, mushroom, lettuce, cabbage, apple, urban water, purified drinking water through household water treatment device.

II. MATERIALS AND METHODS

Collection of plant material and preparation of extract:

Fresh leaves of plant (*Moringa oleifera*) was collected from farm in kammuru village, kuderumandal, Anantapur district. The collected leaves were washed with deionized water and chopped into pieces as shown in figure.1. A hot water extract of the leaf was prepared by taking 5 g of leaf in 100 mL of distilled water and boiled in an Erlenmeyer flask for 5 minutes. The clear extract was obtained by filtration using Whatman filter paper. The extract was stored at $4^{o}C$ for further use (it was used within one week). The filtrate acts as reducing and stabilizing agent for the synthesized iron nanoparticles.

Figure.1:Moringa oleifera dried leaves

2.1.Preparation of precursor:

Ferrous sulphate heptahydrate($FeSO_4.7H_2O$),which is used as a precursor, for the synthesis of FeNPs,0.10M of $FeSO_4.7H_2O$ is prepared using demineralised water.

2.2.Synthesis of Iron nanoparticles:

The desired concentrations of leaf extract was mixed with 0.10M of $FeSO_4.7H_2O$ solution with various volumes of 1:1,1:4,1:5 and at a temperature of 60^0C and constant stirring for 2hrs.A change in colour from reddish yellow to black after a certain period of time indicated the formation of FeNPs. The FeNPs solution was kept in centrifuge tubes about 60 min. The Iron nanoparticles were settled to the bottom of the tubes and the liquid on top of the tube. The particles slurry was taken into a glass plate which was kept in an electric oven for 6 hrs, The obtained FeNPs are removed carefully in sample bottle of air tight container.

2.3.Determination of physico-chemical properties of Iron nanoparticles:

UV-Visible spectrum of the reaction medium was used with model Spectrum lap 752 s to determine the reduction and stability of Iron nanoparticle. The pH was determined using pH meter, of model HI 2211 pH/ORP.

The FTIR spectrum of FeNPs was performed using FTIR with model to identify the possible bio-molecules responsible for capping and efficient stabilization of metal nanoparticles synthesis using extract of Moringa oleifera. FeNP pellet was washed 3 times with 20 mL of distilled water to get rid of all the free proteins/enzymes that were not capping the FeNP prior to the FTIR measurement. The Sample was dried and analyse with FTIR machine in the diffuse reflectance mode using Attenuated Total Reflectance (ATR).

III. Results

3.1.Leaf extract from Moringa leaf:

The leaf extract was prepared was prepared by taking 5 g of Moringa oleifera leaf in 100 mL of distilled water as shown in figure.2.

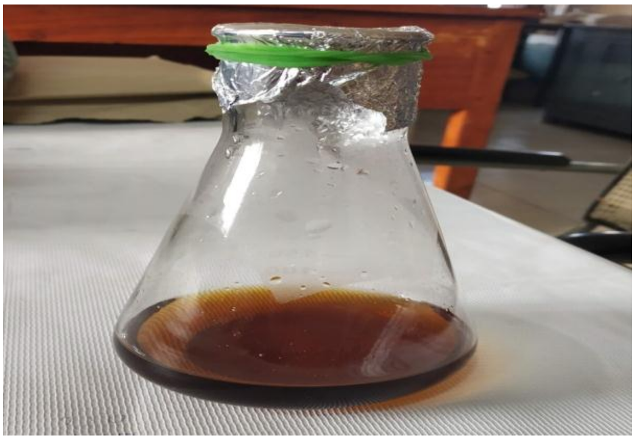

Figure**.2.**Moringa oleifera leaf extract

3.2.Synthesized iron oxide nanoparticles:

The Iron nanoparticles are synthesized as shown in figure.3 & 4The black powder obtained sample is stored in a sample bottle.

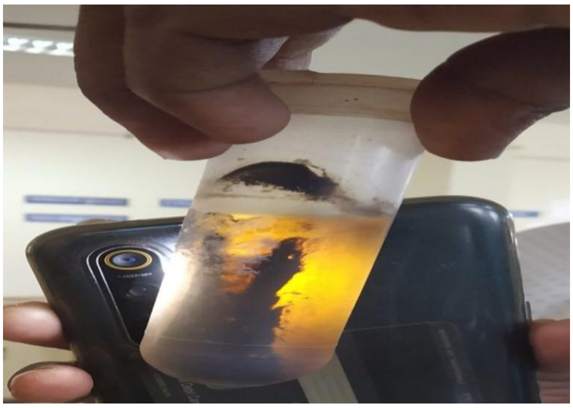

Figure.3. FeNPs attached to the centrifuge tube

Figure.4. Collected FeNPs of different volume ratios

3.3.Physicochemical properties:

The result of the pH analysis shows that Iron nanoparticles synthesized using *Moringa oleifera* was analyzed at relative humidity of 75% and room temperature 27°C, before the synthesis, the pH of Iron solution and moringa leaf extract were 3.33 and 5.91 respectively indicating that the iron salts are more acidic compared to moringa leaf extract, and after the synthesis, the pH of the iron nanoparticles at different time interval shows a gradual decrease as a function of time and stabilizes at 14-24th hour . The mean pH value for the Iron nanoparticles was 3.25 and was in the range of 3.15 to 3.45, shown in figure.5, this shows that the synthesized Iron nanoparticles is acidic .

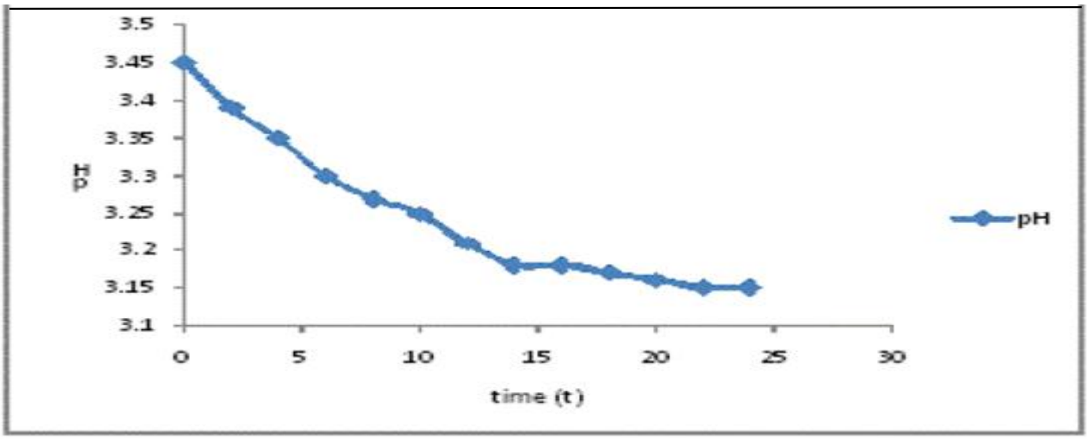

Figure.5.

3.4.UV spectroscopy:

UV-Vis spectroscopy is an important technique to establish the formation of nanoparticles. On adding the MOL extract to $FeSO_4.7H_2O$ solution, the mixture changed from greenish to black. The colour changes are due to the excitation of the surface plasmon resonance in the metal nanoparticles. An assessment of the UV analysis of MOL-FeNPs showed absorption peaks in the range of 210 and 240 nm, which are identical to the characteristic UV visible spectrum of metallic iron. The new peak on the MOL-FeNPs was formed at 240 nm. The peak show in figure.6. the interaction between $FeSO_4.7H_2O$ solution and the MOL extract.

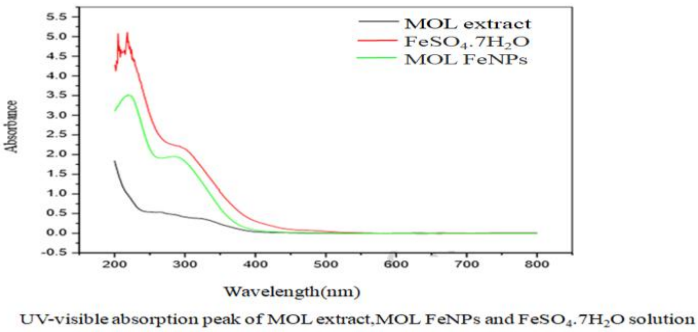

Figure.6. interaction between $FeSO_4.7H_2O$ solution and the MOL extract.

3.5.FTIR characterization of FeNPs:

The FTIR spectra indicated by following figure showed the bands present at 3412 cm-1, 2362 cm-1 and 1601 cm-1 due to O-H stretching, C-H, and C=O, respectively of MOL-FeNPs. These regions around 3270 cm-1 (O-H), 2900 cm-1 (C-H) and 1700 cm-1 (C=O) were also observed on the spectrum of MOL. *Moringa oleifera* has been reported to be enriched with phytochemicals such as amino acids, alkaloids, flavonoids and phenolic compounds, hence the presence of these peaks was observed. The peak in figure.7, at 565 cm-1 confirms that FeNPs were obtained.

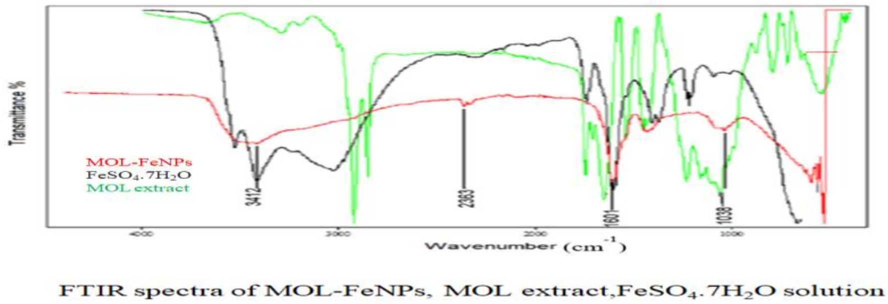

Figure.7. the spectrum of MOL

IV. Conclusion

A simple, rapid and ecofriendly procedure is adopted to synthesize FeNPs using Moringa oleifera leaves. The pH of the iron nanoparticles at different time interval shows a gradual decrease as a function of time and stabilizes at 14-24th hour and the mean pH of FeNPs was 3.25 and in the range of 3.15 to 3.45, this shows that the synthesized Iron nanoparticles is acidic.

The plant leaf extracts substantiate the presence of various phytochemicals such as phenols, flavonoids, alkaloids etc. which mediates the reduction process and helps in formation of capping agent on the synthesised FeNPs and thereby stabilising the nanoparticles. The reduction of Fe2+ by various leaf extracts is confirmed by characterization studies using UV-Visible spectrophotometer and FTIR results.

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