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Green Synthesis and Characterization of Zero Valent Iron Nanoparticles from the Leaf Extract of *Azadirachta indica* (*Neem*)

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Abstract: In the present work, nano scaled zero valent irons (nZVI) were synthesized from the plant extract of under atmospheric conditions. The obtained iron nanoparticles are mainly in zero valent oxidation state. A systematic characterization of nZVI was performed using UV, XRD and SEM studies. The diameter of iron nanoparticles was predominantly found within the range 50-100 nm.

Key words: Zero valent iron · Nanoparticles · Green Synthesis · UV · XRD · Transmission electron microscopy

INTRODUCTION

"Nano" is derived from the Greek word for dwarf. A nanometer is one billionth of a meter (10^{-9}) and might be represented by the length of ten hydrogen atoms lined up in a row [1] Nanotechnology implies the creation and utilization of materials, devices and systems through the control of matter on the nanometer-length scale i.e. at the level of atoms, molecules and supramolecular structures [2-4] Nanotechnology is mainly concerned with synthesis of nanoparticles of variable sizes, shapes, chemical compositions and controlled dispersity and their potential use for human benefits. Although chemical and physical methods may successfully produce pure, well-defined nanoparticles, these are quite expensive and potentially dangerous to the environment. Use of biological organisms such as microorganisms, plant extractor plant biomass could be an alternative to chemical and physical methods for the production of nanoparticles in an eco - friendly manner [5-7]. Nanotechnology is a reliable and enabling environment friendly process for the synthesis of nanoscale particles. Nanosize results in specific physicochemical characteristics such as high surface area to volume ratio, which potentially results in high reactivity [8]. Biosynthesis of nanoparticles is a kind of bottom up approach where the main reaction occurring is reduction/oxidation. With the antioxidant or reducing properties of plant extracts, they are usually responsible for the reduction of metal compounds into their respective nanoparticles. Green synthesis provides advancement over chemical and physical method as it is cost effective, environment friendly, easily scaled up for large scale synthesis and in this method there is no need to use high pressure, energy, temperature and toxic chemicals [9]. Green synthesis offer better manipulation, control over crystal growth and their stabilization. This has motivated an upsurge in research on the synthetic routes that allows better control of shape and size for various nanotechnological applications.

Here in the present work we have reported for the first time the synthesis of green iron nanoparticles using the leaf extract of the plant – (common name - Curry Leaves). Aqueous Ferric Chloride solution, after reacting with Curry Leaves extract, led to rapid formation of highly stable, crystalline Iron nanoparticles. The rate of nanoparticle synthesis was very high, which justifies use of plants over microorganisms in the biosynthesis of metal nanoparticles through greener and safer methods. In the subsequent sections we have described the synthesis of iron nanoparticles based upon the change in color, change in pH, change in absorbance and the particle size formed after reduction.

Plant Description:
Bionomial Name - *Azadirachta indica*
Common Name – Neem
Plant part taken - Leaves
Family Name – Meliaceae

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Fig. 1: *Azadirachta indica* (*Neem*)

Description: It is a tree in the mahogany family. The leaves are used in this manner that first they are washed thoroughly. Then 5-10 leaves along with the branch are boiled till the water turns green The water is then used for varying purposes. Elders find it useful in controlling high blood sugar level and is said to clean up the blood. The tender shoots and flowers of the neem tree are eaten as a vegetable in India. Neem gum is a rich source of protein. Products made from neem trees have been used in India for over two millennia for their medicinal properties: neem products are believed to be anthelmintic, antifungal, antidiabetic, antibacterial, antiviral, contraceptive and sedative.

MATERIALS AND METHODS

Reagents and Chemicals: 0.001 M Ferric Chloride was obtained from Sigma Aldrich. Freshly prepared triple distilled water was used throughout the experiment.

Collection of Extracts: Neem Leaves were collected from the local region. They were washed and cleaned with triple distilled water and dried with water absorbent paper. Then it was cut into small pieces with an ethanol sterilized knife and crushed with mortar and pestle dispensed in 10 ml of sterile distilled water and heated for 2-3 minutes at 70-80°C. The extract was then filtered using Whatman's No.1 filter paper. The filtrate was collected in a clean and dried conical flask by standard sterilized filtration method and was stored.

Synthesis of Zero Valeant Iron Nanoparticles: During the synthesis of Iron Nanoparticles both the precursor and the reducing agent were mixed in a clean sterilized flask in 1:1 proportion. For the reduction of Fe ions, 5ml of filtered Neem plant Leaves extract was mixed to 5 ml of freshly prepared 0.001 M aqueous of $FeCl_3$ solution with constant stirring at 50-60°C. Within a particular time change in colour from Light Green to Black color obtained by nanoparticles synthesis. The Iron Nanoparticles so prepared were stabilized by adding 1% of chitosan and 1% of PVA.

UV-Vis Spectra Analysis: The reduction of pure Fe^{+3} ions to Fe° was monitored by measuring the UV-Vis spectrum by sampling of aliquots (0.3 ml) of Fe Nanoparticle solution diluting the sample in 3 ml distilled water. UV-Vis spectral analysis was done by using UV-Vis spectrophotometer Systronics 118 at the range of 200-600 nm and observed the absorption peaks at 216-268 nm regions due to the excitation of surface plasmon vibrations in the FeNPs solution, which are identical to the characteristics UV-visible spectrum of metallic Iron and it was recorded.

pH Analysis: The pH was determined by using Digital pH meter Systronics. The pH of the reduced solution with Nanoparticle synthesized was found to be 2.16.

RESULTS AND DISCUSSION

Neem Plant Leaves extract is used to produce Iron Nanoparticles in this experiment. Fe^{+3} ions were reduced into Fe^0 nanoparticles when plant extract is mixed with $FeCl_3$ solution in 1:1 ratio. Reduction is followed by an immediate change in color from Light Green to Black and change in pH of the solution. It is well known that Ferric Chloride exhibit bright yellowish color in distilled water. On mixing the plant extract with the aqueous $FeCl_3$ solution it changed the color of the solution immediately and reducing the pH, which may be an indication of formation iron nanoparticles. In this experiment it was observed that the pH changed from high acidic to low acidic.

UV Visible Spectroscopy and Color Change for the Green Synthesized Iron Nanoparticles: The UV visible spectroscopy of the synthesized nanoparticles were in the range of 216-265 nm. Curry Leaves extract was shown to synthesize the iron nanoparticles by the indication of suitable surface Plasmon resonance (SPR) with high band intensities and peaks under visible spectrum.

XRD Pattern of Iron

SEM Images of Iron: The spheres having diameters of around 100 nm can be distinguished from each other and is in agreement with SEM results.

Table 1: Change in color of the solution during Iron Nanoparticle synthesis

		Color change			

Sr. No	Solution	Before Reduction	After Reduction	Color intensity	Time
1.	Neem Plant Leaves Extract	Light Green	Black	+++	24 hours
2.	0.001 M $FeCl_3$ Solution	Bright dark yellow			

Color intensity: - += Light color, ++= Dark color, +++= Very dark color

Table 2: Change in pH during iron nanoparticle synthesis

Plant Extract			Ph change			
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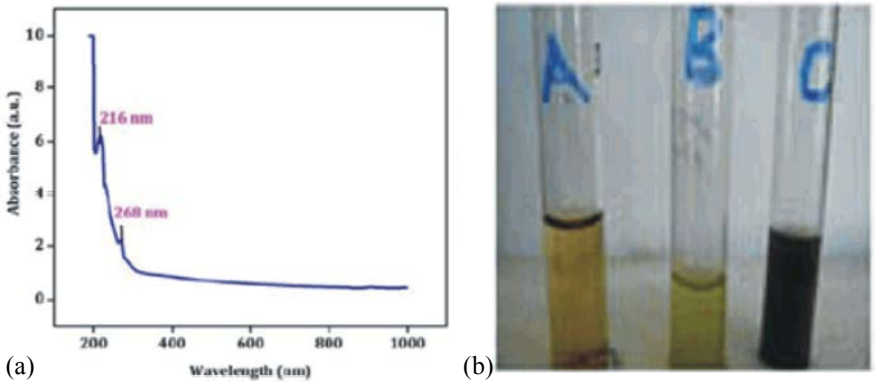
Binomial Name	Local name	Plant Part Taken	Before	After	UV range	Result
Azadirachta indica	Neem	Leaves	5.69	3.93	216-265nm	+

Result: - += Positive, -= Negative.



(a) (b)

Fig. 2: a) -Iron Nanoparticle at 216-268 nm, b) Mixing of plant Leaves extract with 0.001 M $FeCl_3$ Solution to get a reduced solution. Tube A- Ferric Chloride, Tube B- Neem Plant Leaves Extract, Tube C- Iron nanoparticle synthesized solution

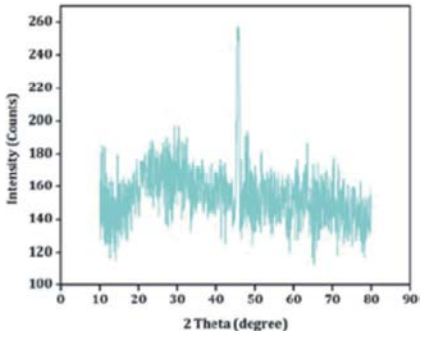


Fig. 3: XRD pattern of Iron Nanoparticles

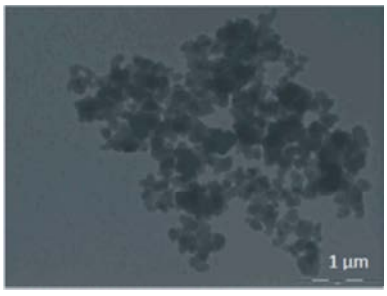


Fig. 4: SEM image of Iron Nanoparticles

CONCLUSION

It has been demonstrated that extract is capable of producing iron nanoparticles that shows good stability in solution, under the UV-Visible wavelength nanoparticles shown quiet good surface plasmon resonance behavior. Ferric Chloride with reducing agent i.e. Neem Plant Leaves extract has shown a remarkable color change with concerned change in pH of solution. Success of such a rapid time scale for synthesis of metallic nanoparticles is an alternative to chemical synthesis protocols and low cost reductant for synthesizing iron nanoparticles.

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