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Review article

ECOFRIENDLY GREEN SYNTHESIS OF IRON NANOPARTICLES FROM VARIOUS PLANTS AND SPICES EXTRACT

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ABSTRACT: Recently the green synthesis of nano particles involving plant extract has attracted the attention of researchers. In the present research program iron nano particles have been synthesized by using aqueous extract of ten plants and spices at room temperature. Synthesized nanoparticles were characterized using UV-visible spectrophotometer and SEM methods. The change in color and pH was observed significantly. Novelty of this present study is that the plant extract is very cost effective and eco friendly and thus can be economic and effective alternative for the large scale synthesis of iron nanoparticles.

Keywords: Iron nano particle; Plant extract; Characterization, Surface plasmon

INTRODUCTION

Matter can be broadly divided into two categories based on the size: Macroscopic and Mesoscopic. Macroscopic matter is visible to the naked eye whereas Mesoscopic particles such as bacteria and cells that have dimensions on the order of micron(s), can be observed with optical microscopes. Falling into the gap between the microscopic and the mesoscopic is another class of matter, the nanoscopic particles. The size of nanoparticles is compared to that of other “small” particles in Figure 1 below, where the bacterium is huge in comparison [1-10].

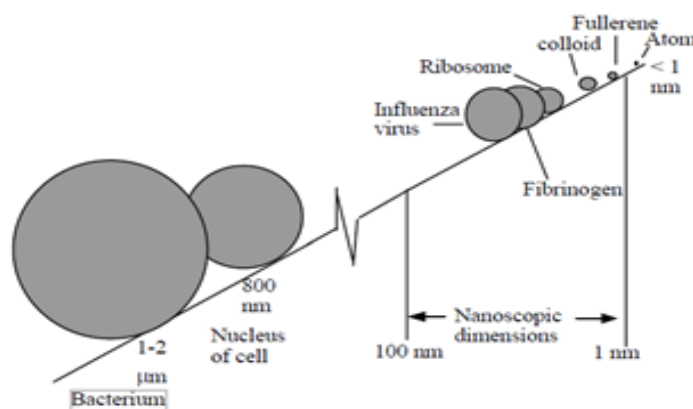


Figure1. Comparison of nano range with other sizes

Nanostructures usually range from 1-100nm in dimension. These particles have high surface to volume ratio and a high fraction of surface atoms. At nano level they have specific physicochemical properties such as optical property, magnetic property, catalytic property etc. [11-18].

With the emergence of new physical and chemical methods for the synthesis of nanoparticles, the concerns for environmental contamination have been increased. The synthetic procedures generate hazardous by products that could affect the environment directly. Thus there is a great requirement for green chemistry that includes methods which are environment friendly. In this method of green synthesis there is no requirement for high pressure, energy, temperature or toxic chemicals. Hence nowadays many researchers are diverting themselves from using synthetic methods. They are trying to turn themselves towards biological systems mostly plants for nanoparticle synthesis as it is cost effective and can be easily scaled up to be used for large scale production.[19-22].

Biological systems such as plants microorganisms produce inorganic materials and most of these are present in nanoscale dimensions. The cellular extracts from these biological organisms can be used to synthesize nanoparticles of different size and chemical compositions.

Biosynthesis of metal nanoparticles extracted from different parts (mostly leaf) of the plant is the most effective process of synthesis at a very affordable cost. During the synthesis bioreduction of metal ions takes place. According to the researchers the polyol components present in the plant extract are responsible for the reduction of iron ions whereas water soluble heterocyclic components stabilize the nanoparticles formed. Appropriate precursors such as Ferric Chloride can be used for the reduction of plant extracts. [23-31].

Here we report the synthesis of nanoparticles, reducing Ferric ions present in the aqueous solution of Ferric chloride by the help of different plant extracts. Through elaborate screening process involving about 45 plants, we selected 10 most suitable plants as the potential candidates for the synthesis of iron nanoparticles.

Experimental details

Materials

For the synthesis of Fe nanoparticles, we used extracts from 10 plant parts that include Mango leaves, Clove buds, Black Tea, Green tea leaves, Coffee seeds, Rose leaves, Cumin seeds, Origano leaves, Thymol seeds, Curry leaves for reducing. Ferric Chloride (FeCl_3) which was used as Fe precursor was purchased from Sigma Chemicals limited. Triple distilled water was prepared in the lab and used throughout the experiment.

Plants that were used in the experiment are described below:

1) **Bionomial Name-***Mangifera indica*

Common Name – Mango

Plant part taken- Leaves

Family Name-Anacardiaceae

Description-Mangiferin (a pharmacologically active flavonoid, a natural xanthone C-glycoside) is extracted from Mango at high concentrations from the young leaves. Mangiferin shows an exceptionally strong antioxidant capacity. It has a number of pharmacological actions and possible health benefits. These include antidiabetic, antioxidant, antifungal, antimicrobial, antiinflammatory, antiviral, hypoglycemic, anti-allergic and anticancer activity.



Fig 2: Mangifera indica

2 **Bionomial Name-** Syzygium aromaticum

Common Nam –Clove

Plant part taken- Buds

Family Name-Myrtaceae

Description: Cloves are the aromatic dried flower buds of a tree. It is used as a spice in cuisines all over the world. Cloves are used in Indian Ayurvedic medicine, Chinese medicine, and western herbalism and dentistry where the essential oil is used as an anodyne (painkiller) for dental emergencies. Cloves are used as a carminative, to increase hydrochloric acid in the stomach and to improve peristalsis.



Fig 3 Syzygium aromaticum

3. **Bionomial Name-**Rosa indica

Common Name – Rose

Plant part taken-Leaves

Family Name-Rosaceae

Description: Rose is a woody perennial. They form a group of erect shrubs, and climbing or trailing plants. Roses are best known as ornamental plants. Many roses have been used in herbal and folk medicines. Other species have been used for stomach problems, and are being investigated for controlling cancer growth.



Fig 4. *Rosa indica*

4. Bionomial Name- *Azadirachta indica*

Common Name – Neem

Plant part taken- Leaves

Family Name- Meliaceae

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.



Fig 5. *Azadirachta indica*

5. Bionomial Name- *Camellia sinensis*

Common Name – Black Tea

Plant part taken- Leaves

Family Name- Theaceae

Description: Tea is the second most commonly drunk liquid on earth after water. It has numerous medicinal benefits mainly due to its antibacterial and antioxidant properties. It has been known to inhibit the growth of cancer cells and support cardiovascular health.



Fig 6. *Camellia sinensis*

6. Bionomial name-*Camellia sinensis*

Common Name – Green Tea

Plant part taken-Leaves

Family Name-Theaceae

Description: Green tea originates in China. Green tea has purported health benefits, with some evidence suggesting that regular green tea drinkers may have a lower risk of developing heart disease and certain types of cancer. A green tea extract containing polyphenols and caffeine has been shown to induce thermogenesis and stimulate fat oxidation, boosting the metabolic rate 4% without increasing the heart rate. Flavonoids are a group of phytochemicals in most plant products that are responsible for such health effects as anti-oxidative and anticarcinogenic functions.



Fig 7. Green Tea

7. Bionomial name-*Coffea arabica*

Common Name – Coffee

Plant part taken-Seeds

Family Name-Rubiaceae

Description: Coffee is a genus of flowering plants whose seeds, called coffee beans, are used to make coffee. The caffeine in coffee "beans" is a natural plant defense against herbivory, i.e. a toxic substance that protects the seeds of the plant. Several insect pests affect coffee production, including the coffee borer and the coffee leafminer. Coffee is used as a food plant by the larvae of some Lepidoptera (butterfly and moth) species.



Fig 8. Coffea arabica

8. Bionomial name-*Trachyspermum ammi*

Common Name –Carom seeds

Plant part taken-Seeds

Family Name-Apiaceae

Description: The plant has a similarity to parsley. Because of their seed-like appearance, the fruit pods are sometimes called seeds. The raw fruit pod smells almost exactly like thyme because it also contains thymol. It is traditionally believed to be a digestive aid.



Fig 9. *Trachyspermum ammi*

9. Bionomial name-*Magnolia champaca*

Common Name –Joy Perfume Tree, Champa

Plant part taken-Leaves

Family Name- Magnoliaceae

Description: *Magnolia champaca* is a large evergreen tree. The flowers are used in Southeast Asia for several purposes. It is rarer and has a strong perfume, and is not that commonly or plentifully used. *Magnolia champaca* is cultivated and used as an ornamental tree in temperate climate gardens.



Fig 10. *Magnolia champaca*

10. Bionomial name: *Murraya koenigii*

Common Name – Curry Leaves

Plant part taken- Leaves

Family Name- Rutaceae

Description: It is a tropical to sub-tropical tree which is native to India. The leaves are highly valued as seasoning in southern and west-coast Indian cooking. The leaves are used as a herb in Ayurvedic medicine. It is valued as an anti-diabetic, antioxidant, antimicrobial, anti-inflammatory, anti-hypercholesterolemic, etc. It contains carbazolealkaloid that can induce apoptosis in cancerous cells in liver.



Fig 11. Murraya koenigii

Preparation of Plant Extract and the Precursor

For the synthesis of iron nanoparticles, 0.001 M Ferric Chloride was prepared by using triple distilled water. Plant extracts were prepared by taking approximately 25gms leaves/seeds/buds. These were of thoroughly washed with sterile distilled water, dried and finely crushed with the help of mortar and pestle by adding 5-10 ml of deionized water gradually. The mixture was poured in a flask and heated for 5-10 minutes at 70°C before finally decanting it. The mixture was then filtered using Whatman No. 1 filter paper. Wherever necessary the plant mixture was centrifuged at 5000 rpm for 5 minutes and the supernatant was collected as the plant extract and used for further process. Clean and aseptic condition was maintained throughout the process.

Synthesis of 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 plant extract was mixed to 5 ml of 0.001 M aqueous of FeCl₃ solution with constant stirring at 50-60°C.

Characterization

UV-Vis Spectroscopy

Ultraviolet-visible spectroscopy (UV-Vis) refers to absorption spectroscopy in the UV-Visible spectral region. This means it uses light in the visible and adjacent (near-UV and near-infrared (NIR)) ranges. The absorption in the visible range directly affects the perceived color of the chemicals involved. In this region of the electromagnetic spectrum, molecules undergo electronic transitions.

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 acidic. After reduction the pH of every sample was found to decrease and move towards the acidic range.

RESULTS AND DISCUSSION

Synthesis of iron nano particles

Ten plant extracts were used to produce iron nanoparticles (Table. 1 and 2), the reduction of iron ions into iron particles occurred after mixing ferric chloride with different plant and spices extract, followed by colour change and change in pH of solutions. As the plant extract was mixed in the aqueous solution of the iron ion complex, it started to change the color due to reduction of iron ion, which may be the indication of formation iron nanoparticles.

UV-Vis Spectroscopy

The bioreduction of Fe^{+3} in aqueous solutions was monitored by periodic sampling of aliquots of the mixture and subsequently measuring UV-Vis spectra. UV-Vis spectral analysis was done by using UV-Vis spectrophotometer Systronics 118 at the range of 300-500 nm and observed the **absorption peaks at 216-265 nm** regions due to the excitation of surface plasmon vibrations in the iron nanoparticles, which are identical to the characteristics UV-visible spectrum of metallic iron and it was recorded.

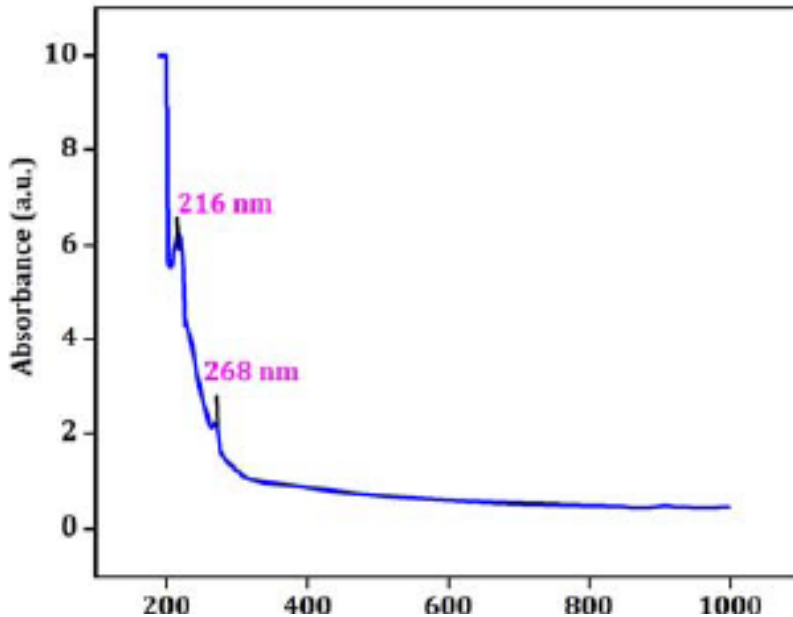


Fig 12 UN-vis of Iron Nano particles

The UV Visible spectrum of nano iron particle is shown in the above figure. The two absorption peaks at wavelengths of 216 nm and 268 nm indicates the formation of nano iron particles

Table 1: Indication of color change in green synthesis of iron nano particles

Sr. No	Nanoparticle Solution	Color change		Color intensity	Time
		Before	After		
1.	Mango	Turbid light yellow	Black	+++	Immediate
2.	Clove	Golden	Black	+++	Immediate
3.	Rose	Turbid green	Black	+++	Immediate
4.	Black Tea	Dark Brown	Black	+++	24 hours
5.	Green Tea	Golden	Pale black	++	24-48 hours
6.	Coffee	Brown	Brownish black	++	24-48 hours
7.	Carom Seeds	Light brown	Black	+++	24- hours
8.	Champa	Turbid brown	Dark Green	+++	Immediate
9.	Neem	Light green	Black	+++	24 hours
10.	Curry Leaves	Light brown	Black	+++	24-48 hours

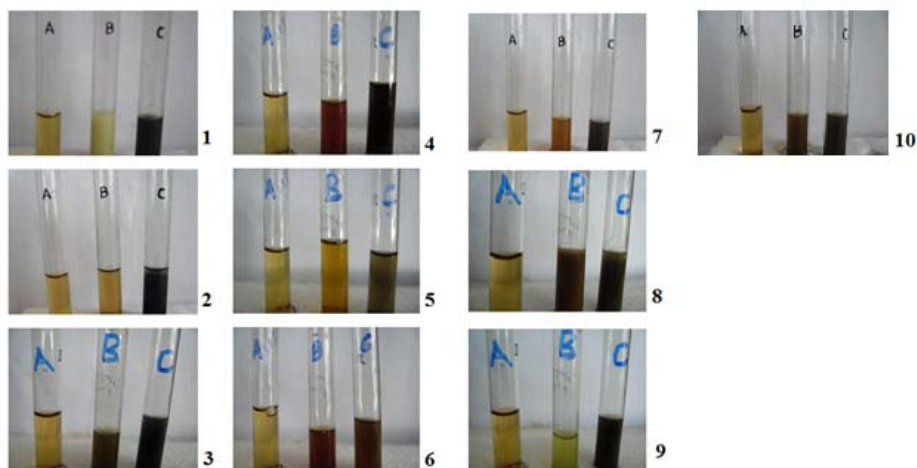


Figure 13. Color change seen in ten plant extracts taken for iron nanoparticle synthesis 1)Mango leaf extract 2) Clove bud extract 3) Rose leaf extract 4) Black Tea leaf extract 5) Green Tea leaf extract 6) Coffee seed extract 7) Carom Seeds extract 8) Champa leaf extract 9) Neem leaf extract 10) Curry leaf extract

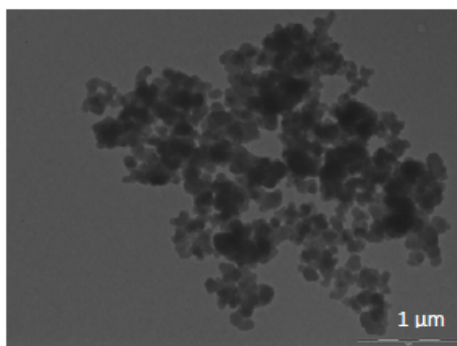


Fig 14. SEM of iron nano particle

Table 2: Indication of change in pH during green synthesis of iron nano particles

Sr. No	Nanoparticle solution	pH change		Result
		Before Reduction	After Reduction	
1.	Mango	5.12	2.16	+
2.	Clove	4.22	1.88	+
3.	Rose	5.75	2.75	+
4.	Black Tea	5.00	3.28	+
5.	Green Tea	5.37	2.65	+
6.	Coffee	4.90	2.20	+
7.	Carom Seeds	5.76	3.89	+
8.	Champa	4.86	3.22	+
9.	Neem	5.69	3.93	+
10.	Curry Leaves	4.50	3.23	+

The above figure shows the SEM image of freshly synthesized iron nanoparticles. It can be observed that the iron particles are in the form of nanospheres, which exist in contact with each other and form chains having diameters of 50-100 nm. This linear orientation is nearly due to the magnetic properties of iron species

CONCLUSION

The extracts of plants and spices may be capable of producing Iron nano particle. Under the UV-Visible wavelength Nanoparticles showed quiet good surface plasmon resonance behavior. The color change was also remarkable when Ferric chloride was mixed with reducing agent i.e plants and spices extract. As and when reduction occurred the color changed 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 nano particles. For more confirmation we can go for higher characterization techniques such as XRD, SEM, TEM, FTIR, etc.

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