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Green Synthesis of Silvernanoparticles using Fruit Extract and Its Characterization

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Abstract

Nanotechnology has emerged as a promising field with extensive applications in medicine, agriculture, environmental science, and biotechnology. The present study focuses on the green synthesis of silver nanoparticles (AgNPs) using fruit extract as a natural reducing and stabilizing agent. The use of fruit extracts offers an eco-friendly, cost-effective, and sustainable alternative to conventional chemical synthesis methods. Fresh fruit extract was prepared and mixed with an aqueous silver nitrate (AgNO_3) solution under controlled conditions. The formation of silver nanoparticles was indicated by a visible color change resulting from surface plasmon resonance. The synthesized nanoparticles were characterized using various analytical techniques such as UV–Visible spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM). Characterization results confirmed the successful formation of crystalline, stable, and predominantly spherical silver nanoparticles with nanoscale dimensions. The phytochemicals present in the fruit extract played a significant role in the reduction and stabilization of silver ions. The study demonstrates that fruit-mediated green synthesis is an efficient and environmentally benign approach for producing silver nanoparticles with potential applications in antimicrobial, biomedical, and environmental fields.

Keywords: Green Synthesis, Silver Nanoparticles, Fruit Extract, Nanotechnology, Silver Nitrate

I. INTRODUCTION

Nanotechnology is a sprouting interdisciplinary field of research interspersing material science, bionanoscience and technology. Remarkable advances are made in the field of biotechnology and nanotechnology to harness the benefit of life sciences, health care and industrial biotechnology. In nanotechnology, a particle is defined as a small object that behaves as a whole unit in terms of its transport and properties [1-2]. The use of nanoparticles is gaining impetus in the present century as they possess defined chemical, optical and mechanical properties. Metal nanoparticles are of importance due to their potential applications in catalysis, photonics, biomedicine, antimicrobial activity and optics.



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Nanotechnology is a reliable and enabling environment friendly process for the synthesis of nanoscale particles of structures ranging from approximately 1-100 nm nanoparticle. Nanosize results in specific physicochemical characteristics such as high surface area to volume ratio, which potentially results in high reactivity. Nanotechnology, a sub-classification of ultrafine particle with lengths in two or three dimensions greater than 0.001 micrometer (1 nanometer) and smaller than about 0.1 micrometer (100 nanometers) and which may or may not exhibit a size-related intensive property.

Particles are further classified according to size: in terms of diameter, coarse particles cover a range between 10,000 and 2,500 nanometers. Fine particles are sized between 2,500 and 100 nanometers. Ultrafine particles or nanoparticles are sized between 100 and 1 nanometer. The transition from micro particles to nanoparticles can lead to a number of changes in physical properties. At nano scale the properties of the materials are different from their same bulk size counterparts. These changes in properties may be due to quantum size effects [2-4].

Nanometer

A nanometer is one billionth of a meter (10^{-9} m). This is roughly ten times the size of an individual atom. A cube 2.5 nm on a side would contain about a thousand atoms. The smallest feature in an integrated circuit of today is 250 nm on a side, and contains one million atoms in a layer of atomic height. Proteins, the molecules that catalyze chemical transformations in cells, are 1 to 20 nm in size. For comparison, 10 nm is 1000 times smaller than the diameter of a human hair. There are as many nanometers in an inch as there are inches in 400 miles [5].

Importance of This Scale

There are five reasons why this length scale is so important:

- The wavelike properties of electrons inside matter are influenced by variations on the nanometer scale. By patterning matter on the nanometer length scale, it is possible to vary fundamental properties of materials (for instance, melting temperature, magnetization, charge capacity) without changing the chemical composition.
- The systematic organization of matter on the nanometer length scale is a key feature of biological systems. Nanotechnology promises to allow us to place artificial components and assemblies inside cells, and to make new materials using the self-assembly methods of nature. This is a powerful new combination of materials science and biotechnology.
- Nanoscale components have very high surface areas, making them ideal for use in composite materials, reacting systems, drug delivery, and energy storage.
- The finite size of material entities, as compared to the molecular scale, determine an increase of the relative importance of surface tension and local electromagnetic effects, making nanostructure materials harder and less brittle.



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- The interaction wavelength scales of various external wave phenomena become comparable to the material entity size, making materials suitable for various optoelectronic applications [5].



Figure 1 : Nanoscale material (hazema.com)

II. REVIEW OF LITERATURE

Over the past decade, nanomaterials have been the subject of enormous interest. The interest in nanoscale materials emerges from the fact that new properties are acquired at this scale and also that these properties change with size or shape of nanomaterials. Nanostructured materials have attracted a great deal of attention in the last few years for their unique characteristics that cannot be obtained from conventional macroscopic materials

However the credit of realizing the enormous potential of nanoparticles and their possible implications in different fields is given to Richard P. Feynman.

I would like to describe a field, in which little has been done, but in which an enormous amount can be done in principle. This field is not quite the same as the others in that it will not tell us much of fundamental physics (in the sense of, what are the strange particles?)

In the year 2000, when they look back at this age, they will wonder why it was not until the year 1960 that anybody began seriously to move in this direction.

When we get to the very, very small world---say circuits of seven atoms---we have a lot of new things that would happen that represent completely new opportunities for design. Atoms on a small scale behave like nothing on a large scale, for they satisfy the laws of quantum mechanics. So, as



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we go down and fiddle around with the atoms down there, we are working with different laws, and we can expect to do different things.

The principles of physics, as far as I can see, do not speak against the possibility of man everting things atom by atom. It is not an attempt to violate any laws; it is something, in principle, that can be done; but in practice, it has not been done because we are too big.

The problems of chemistry and biology can be greatly helped if our ability to see what we are doing, and to do things on an atomic level, is ultimately developed---a development which I think cannot be avoided.

The term nanoparticle is used to describe a wide variety of materials of submicron size. It is used differently, according to the context of material being described. According to a recent definition suggested by British Standards Institution "Nanoparticles are the particles with one or more dimensions at the nanoscale." They have defined the nanoscale as dimensions of the order of 100 nm or less. It is critical length scale at which certain novel size related properties develop and the materials start behaving differently than the molecules or bulk material (PAS71 2005). Here novel properties refer to optical magnetic and electrical properties which typically appear in materials below 100 nm size. However, many a times, besides "strictly nano" (1-100 nm) all submicron colloidal particles i.e. particles with at least one dimension in the scale of 1-1000 nm, also called mesoscale, are referred as nanoparticles, to include organic polymers and vesicles widely used in the area of drug delivery. (Gelperina et al. 2005)

Among the nano particles, the synthesis of silver nanoparticles is receiving increasing attention due to their applications in traditional medicines and culinary items. Several salts of silver and their derivatives are commercially employed as antimicrobial agents. Thus, nanoparticles of silver have aptly been investigated for their antibacterial property etc. To synthesize such particles several methods are used and nanoparticles formed using each method show specific properties, size and shape. Further, a number of studies have been reported on synthesis of silver nanoparticles indicated by the reports, papers and reviews on this subject. Review of literature has been carried out as given below to get an idea about work carried out by various scientists on synthesis and characterization of silver nanoparticles. (Ankamwar et al., 2005) Reported, synthesis and characterization of biologically synthesized nanomaterials have become an area of significant interest. In this paper, report the extracellular synthesis of gold and silver nanoparticles using *Emblca Officinalis* (amla, Indian Gooseberry) fruit extract as the reducing agent to synthesize Ag and Au nanoparticles, their subsequent phase transfer to an organic solution and the transmetallation reaction of hydrophobized silver nanoparticles with hydrophobized chloroaurate ions.



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III. EXPERIMENTAL WORK

Experimental Work

Nowadays, the attention of many scientists is focused on the development of novel techniques for green synthesis of nanoparticles. Different metals, metal oxides, sulfides, polymers, core-shell and composite nanoparticles can be prepared using a number of synthetic techniques. In the present silver nanoparticles have been synthesized by microwave method using fruit extract of *Ficus carica* (common fig) and pure silver nitrate in presence of sodium carbonate, ethylene glycol and cetyl trimethyl ammonium bromide.

Materials Used:

Figs are one of the highest plant sources of calcium and fiber. According to USDA data for the Mission variety, dried figs are richest in fiber, copper, manganese, magnesium, potassium, calcium, and vitamin K, relative to human needs. They have smaller amounts of many other nutrients. Figs have a laxative effect and contain many antioxidants. They are good source of flavonoids and polyphenols including gallic acid, chlorogenic acid, syringic acid, (+)-catechin, (-)-epicatechin and rutin. In one study, a 40-gram portion of dried figs (two medium size figs) produced a significant increase in plasma antioxidant capacity.

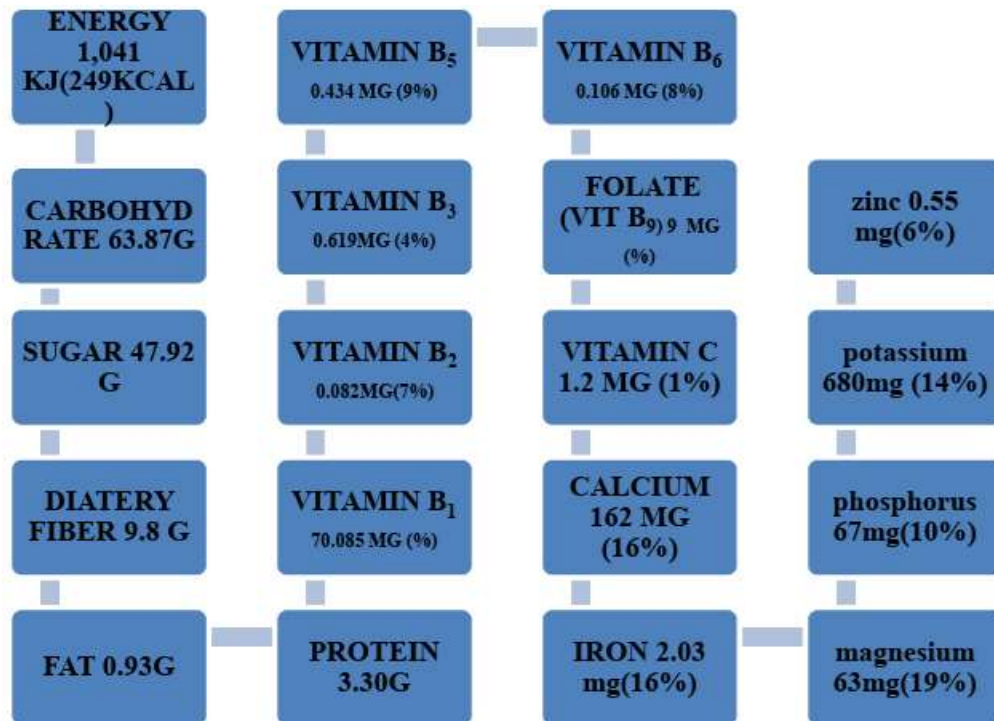


Figure 2: Nutrient values are given in bar chart



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SILVER NITRATE: Silver nitrate is an inorganic compound with chemical formula AgNO_3 . This compound is a versatile precursor to many other silver compounds, such as those used in photography. It is far less sensitive to light than the halides.



Figure 3:

Molecular Formula	= AgNO_3
Molar mass	= $169.87 \text{ g mol}^{-1}$
Appearance	= white solid
Density	= 4.35 g/cm^3
Melting point	= $212 \text{ }^\circ\text{C}$, 485 K , $414 \text{ }^\circ\text{F}$
Boiling point	= $444 \text{ }^\circ\text{C}$, 717 K , $831 \text{ }^\circ\text{F}$ (decomp.)
Solubility in water	= 1.22 kg/L ($0 \text{ }^\circ\text{C}$)
	2.16 kg/L ($20 \text{ }^\circ\text{C}$)
	4.40 kg/L ($60 \text{ }^\circ\text{C}$)
	7.33 kg/L ($100 \text{ }^\circ\text{C}$)
Solubility	= soluble in ethanol and acetone.

SODIUM CARBONATE: Na_2CO_3

Sodium carbonate is the name used to refer to the chemical compound Na_2CO_3 . Sodium carbonates have a white appearance. Solution prepared by sodium carbonate is colorless.



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Figure 4:

IV. CHARACTERIZATION TECHNIQUES

Characterization Techniques

The various characterization techniques like X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) and Disk Diffusion Method have been used for characterization of synthesized silver nanoparticles. The basic principles of these techniques have been discussed below in detail.

X-RAY DIFFRACTION (XRD)

XRD is a very important experimental technique that has long been used to address all issues related to the crystal structure of solids, including lattice constants and geometry, identification of unknown materials, orientation of single crystals, preferred orientation of polycrystals, defects, stresses, etc. In XRD, a collimated beam of X-rays, with a wavelength typically ranging from 0.7 to 2 Å, is incident on a specimen and is diffracted by the crystalline phases in the specimen according to Bragg's law

$$\lambda = 2d \sin\theta$$

Where,

d is the spacing between atomic planes in the crystalline phase and
 λ is the X-ray wavelength.

The intensity of the diffracted X-rays is measured as a function of the diffraction angle 2θ and the specimen's orientation. This diffraction pattern is used to identify the specimen's crystalline phases and to measure its structural properties. XRD is nondestructive and does not require elaborate sample preparation, which partly explains the wide usage of XRD method in materials characterization. Diffraction peak positions are accurately measured with XRD, which makes it the best method for characterizing homogeneous and inhomogeneous strains. If there is no



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inhomogeneous strain, the crystallite size, D , can be estimated from the peak width with the Scherrer's formula"

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

where λ is the X-ray wavelength

B is the full width of height maximum (FWHM) of a diffraction peak

θ is the diffraction angle

and K is the Scherrer's constant of the order of unity for usual crystal.

Scherrer's formula may produce results different from the true particle sizes. In addition, X-ray diffraction only provides the collective information of the particle sizes and usually requires a sizable amount of powder. This technique is very useful in characterizing nanoparticles. Similarly, the film thickness of epitaxial and highly textured thin films can also be estimated with XRD.



Figure 5: X-Ray Diffractometer at CSIR-AMPRI

Fourier Transform Infrared Spectrometry

FT-IR stands for Fourier Transform Infrared; the preferred method of infrared spectroscopy. Fourier transform infrared (FTIR) spectroscopy is a chemical analytical technique, which



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measures infrared intensity versus wavelength (wave number) of light. It is used to determine the nature of associated molecules of plants or their extracts with nanoparticles. Based upon the wave number, infrared light can be categorized as far infrared (4–400 cm^{-1}), mid infrared (400–4000 cm^{-1}) and near infrared (4000–14 000 cm^{-1}). Infrared spectroscopy detects the vibration characteristics of chemical functional groups in a sample. When an infrared light interacts with matter, chemical bonds will stretch, contract and bend. As a result, a chemical functional group tends to adsorb infrared radiation in a specific wave number range regardless of the structure of the rest of the molecule.

V. RESULT AND DISCUSSION

In present work, Ag nanoparticles were prepared by microwave method using Ficus carica extract, silver nitrate, sodium carbonate, and ethylene glycol and Cetyl trimethyl ammonium bromide. Characterization has been done by X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier Transform Infra Red Spectroscopy (FTIR), Thermal Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) and Bacterial assay by Disk diffusion method. It is well known that silver nanoparticles exhibit yellowish brown color in aqueous solution due to excitation of surface Plasmon vibrations in silver nanoparticles²⁷. As the fruit extract was mixed in the aqueous solution of the silver ion complex, it started to change the color from watery to yellowish brown. Reduction of silver ions was visually evident from the colour change and was completed within two hours with a stable brown colour indicating the formation of silver nanoparticles. Fig. 14 shows the color changes before (a), during (b) and (c) after the process of reduction of Ag^+ to Ag nanoparticle. The biosynthesized silver nanostructure by employing Ficus carica fruit extracts.



Figure 6: Image of solution before and after reaction



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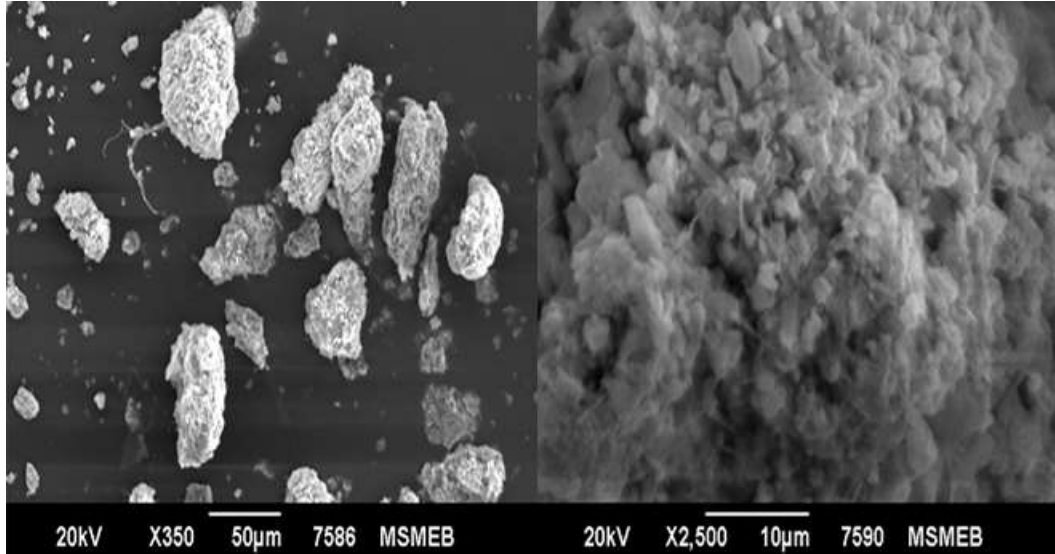


Figure 7: SEM image representing Silver Magnesium nanoparticle

Fig.7 shows representative SEM images recorded from drop-coated films of the silver nanoparticles synthesized by treating silver nitrate solution with *Ficus carica* fruit extract. The morphology of the nanoparticles was spherical in nature. Under careful observation, it is evident that the silver nanoparticles surrounded by a faint thin layer of other materials, which we suppose are capping organic material from *Ficus carica* fruit extract. [52-54]SEM image showing the high density silver nanoparticles synthesized by *Ficus carica* fruit extract further confirmed the development of silver nanostructures.

The band at 1387 cm^{-1} corresponds to O-H stretching H-bonded alcohols and phenols. The bands centered at 3846 , 3738 , 2918 , and 2815 cm^{-1} can be identified due to the -C-H-stretching and bending vibrations. The bands at 1016 , and 872 can be due to -P-O-C stretching alcohols, carboxylic acids, ethers and esters. Therefore the synthesized nanoparticles were surrounded by proteins and metabolites such as terpenoids having functional groups of alcohols, ketones, aldehydes and carboxylic acid. The bands observed at 3846 were due to C-H stretching and at 672 was due to OH and 1598 were due to NH bend primary amines. The peak due to C=O linkage was observed at 2355 cm^{-1} .

The peaks assignment in FTIR spectra is shown in Table below



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S. No	Wave number	Functional Group
1.	3846	-CH
2.	3738	- CH
3.	3410	- NH
4.	2918	-CH
5.	2851	-CH
6.	2355	C=O
7.	1598	-NH
8.	1387	-OH
9.	1016	P-O-C
10.	872	P-O-C
11.	672	-OH

VI. CONCLUSION

- X-ray diffraction studies revealed that the peaks observed in XRD pattern match well with those of Ag- Mg reported in JCPDS power diffraction data, and the size determined using Debye Scherrer equation is around 41 nm.
- SEM studies reveals microscopic image of synthesized silver nanoparticles.
- FTIR spectra showed the peaks due to stretching and bending vibrations of - CH -NH, -OH- and other characteristic groups.
- The TGA curves showed around 16 % weight loss in the sample by heating it from room temperature up to 16000c.
- DSC results display endothermic peaks in the temperature range of 90.21 to 100.850C and a very small endothermic peak in the temperature range of 784.78 to 791.560C and is associated with 819 mJ (35.66J/gm) and 349 mJ (15.19 J/gm) of energy respectively.
- Antibacterial assay was performed by disc diffusion method; result was found positive as zone of inhibition was found against bacillus species. Thus, the bio-reduction of aqueous Ag+ ions by the fruit extract of the Ficus carica has been demonstrated. The reduction of the metal ions through fruit extracts leading to the formation of silver nanoparticles of fairly well-defined dimensions. But the capabilities of the other plant part such as fruit as a capping and reducing agent is not tested and not well defined. In the present study we found that fruits can be also good source for synthesis of silver nanoparticles.



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