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# Green Synthesis of Silver Nano Particles (AgNPs) Mediated By Waste Peel Aqueous Extract of Lemon Fruits

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**ABSTRACT:** Ecofriendly or Green synthesis of metal nanoparticles has become an important branch of nanotechnology and there is an increasing commercial demand for nanoparticles due to their wide applications. In the present study, we report an eco-friendly and economical way for the synthesis of silver nanoparticles using waste peel extracts of lemon (*Citrus limon*). The lemon (*Citrus limon*) is a species of small evergreen trees in the flowering plant family Rutaceae. For the synthesis of silver nanoparticles (AgNPs) using the waste peel extract of lemon (*Citrus limon*) as a reducing agent from 1 mM silver nitrate ( $\text{AgNO}_3$ ) has been investigated. The resulting AgNPs are characterized using UV-Vis, FTIR, FL, DLS, SEM and CV study. Silver nanoparticles were synthesized within 24 hours of incubation period and synthesized AgNPs showed an absorption peak at around 483 nm in the UV-visible spectrum. The morphological study of Silver nanoparticles using TEM suggests that the nanoparticles are spherical in shape with a diameter around 45-nm. This route is rapid, simple without any hazardous chemicals as reducing or stabilizing agents and economical to synthesized AgNPs.

**KEYWORDS:** Green synthesis, *Citrus limon*, waste peel extract, UV-Vis, FTIR, FL, DLS, SEM and CV study

## I. INTRODUCTION

Nanotechnology, and alongside nanostructured materials, play an ever increasing role in science, research and development as well as also in every days life, as more and more products based on nanostructured materials are introduced to the market. The advance and very applicable technology is nanotechnology and it was derived from the term of nano it is the billionth of meter or  $10^{-9}$  m. The Nano come ultimately from the Greek word for dwarf, and is also related to the Spanish word Nino [1]. The synthesis of silver nanomaterials or nanoparticles extensively studied by using chemical and physical methods, but the development of reliable technology to produce nanoparticles is an important aspect of nanotechnology. Biological synthesis process provides a wide range of environmentally acceptable methodology, low cost production and minimum time required. At the same time the biologically synthesized silver nanoparticles has many applications includes catalysts in chemical reactions [2]. Microbial source to produce the silver nanoparticles shows the great interest towards the precipitation of nanoparticles due to its metabolic activity. Of course the precipitation of nanoparticles in external environment of a cell, it shows the extracellular activity of organism. Extracellular synthesis of nanoparticles using cell filtrate could be beneficial over intracellular synthesis, the fungi being extremely good candidates for extracellular process and also environmental friendly. There are few reports published in literature on the biosynthesis of silver nanoparticles using fungal as source [3]. The use of bacterial strain in the bio-manufacturing process has the advantage that ease of handling than the fungal sources [4-7]. Plant biotechnology has opened up unexpected new ways for finding new way for trapping their potential resources. Silver nanoparticles (AgNPs), are the noble metal nanoparticles that has being studied extensively due its various biological properties [8]. Silver is a nontoxic, safe inorganic antibacterial agent used for centuries and it has the capability of killing different type of diseases causing microorganisms [9]. Silver has been known to be a potent antibacterial, antifungal and antiviral agent, but in recent years, the use of silver as a biocide in solution, suspension, and especially in nano-particulate form has experienced a dramatic revival. Due to the properties of silver at the nano level, nanosilver is currently used in an increasing number of consumer and medical products. The remarkably strong antimicrobial activity is a major reason for the recent increase in the development of products that contain nanosilver. The main

challenge in nanomaterials synthesis is the control of their physical properties such as obtaining uniform particle size distribution, identical shape, morphology, chemical composition and crystal structure. For the production of nanoparticles, one needs to know the physical and chemical principles of nanoscale materials and also know how to commercialize them. Generally, metal nanoparticles can be prepared and stabilized by chemical, physical, and biological methods; the chemical approach, such as chemical reduction, electrochemical techniques, photochemical reduction [10-12], and pyrolysis [13], and physical methods, such as Arcdischarge and physical vapor condensation [14], are used. Living organisms have huge potentials for the production of nanoparticles of wide applications. A promising move towards to reach this objective is to develop the array of biological resources in nature. Indeed, over the past several years, plants, algae, fungi, bacteria and viruses have been used for production of inexpensive, energy-efficient and eco-friendly metallic nanoparticles. Many bacterial cultures were used for different kind of nanoparticles some are gold nanoparticles using *Shewanella algae*; it's a kind of marine bacterium [15], silver nanoparticles by *Cynobacteria Plectonema boryanum* [16], cadmium nanoparticles biosynthesis was done by *Clostridium thermoaceticum*, magnetite nanoparticles by *Actinobactor* sp., *Shewanella oneidensis* used for uranium nanoparticles [17].



Figure: 1 (a) Lemon fruit with their plant, (b) Sliced lemon, (C) collection of waste peel of lemon

Existing literature also reports successful synthesis of silver nanoparticles through a green route where the reducing and capping agent selected was the latex obtained from *Jatropha curcas* [18]. Ag NPs were also obtained using *Aloe vera* [19], *Acalypha indica* [20], *Garcinia mangostana* [21] leaf extracts. *Crataegus douglasii* fruit extract [22] as well as various other plant extracts [23] as reducing agent. Here we have developed a rapid, eco-friendly and convenient green method for the synthesis of silver nanoparticles from silver nitrate using leaf extracts of three Indian medicinal, namely, *Musa balbisiana* (banana), *A. indica* (neem) and *O. tenuiflorum* (black tuls), by microwave irradiation method. In this research, the plant mediated synthesized Ag NPs were characterized and studied in details with all of their properties significant to current science and prevailing technologies. However, the biosynthesis of silver nanoparticles by free cell system and culture filtrate has not been investigated yet. In this paper, we report on the synthesis of silver nanoparticles by the reduction of aqueous Ag<sup>+</sup> ion by simultaneous reduction of aqueous Ag<sup>+</sup> with the leaf extract of medicinal plant. Through our screening process involving a number of plants we observed that were potential candidate for rapid synthesis of silver nanoparticles.

## II. MATERIALS & METHODS

### 2.1 Sample collection

The waste lemon peels were collected from juice stall in Chidambaram town, the collected waste peels was washed thrice in the running tap water and again washing deionized water. Then sliced in small pieces, the pieces are dried in seven days in sun light; the dried peels were grained to powder. The powdered Lemon (*Citrus limon*) fruit peels stored and further green synthesis process.

### 2.2. Chemicals, Solvents and Starting Materials

De-ionized water, whatmann 1 $\mu$  and whatmann 41 $\mu$  filter papers, Potassium di chromate Ethyl alcohol, sodium hydroxide pellets, Hydrochloric acids, sulphuric acid and other chemicals were purchased from Merck (India) Ltd. All chemicals were used without further purification.

### 2.3 Instruments and equipment

Electric oven, Magnetic stirrer (REMI 2 MLH), E-1 portable TDS & EC meter, pH-009(I)A pen type pH meter, sterilized 250ml separating funnels, sterilized conical flasks, sterilized 400ml beakers, watch glasses, 7'' funnels, glass rods, and 10ml measuring cylinders,

### 2.4 Extraction of waste fruit peelsLemon (*Citrus limon*)

5 grams of Lemon (*Citrus limon*) fruit peelpowder with 50 mL of double-distilled water (DDW) taken in the 250 mL round bottomed flask, water condenser fitted and fix the running tap water then heated for 20 min at 80<sup>o</sup> C. Then the extract was filtered with Whatman 1 $\neq$  filter paper. The filtrate was used to the further green synthesis of process.

### 2.5 Green synthesis of Silver nanoparticle (AgNPs) by waste peel extracts of Lemon (*Citrus limon*)

Silver nitrate (AgNO<sub>3</sub>) solution of 0.1m M was prepared. Drop wise 10 ml of waste fruit peels extracts of Lemon (*Citrus limon*) has been added and color changes were observed after 30 min. and stirring at 500 rpm for 2 h at magnetic stirrer with heating instrument, to achieve precipitate containing final solution[92], The white colour reactant solution is turn to brownish black colour. The obtained precipitate was filtered whatmann 1 $\neq$  filter paper. The nanoparticles containing filter paper was dried hot air oven and collect the air tight click lock tube. The green synthesised silver nanoparticles were characterised by UV, FTIR, FL, DLS, SEM, EDS and CV analysis.



Figure:2 Extract of Lemon (*Citrus limon*) and AgNPs suspension

## III. RESULT AND DISCUSSION

### 3.1 UV-Vis spectral Analysis

It is well known that silver nanoparticles exhibit yellowish brown color in aqueous solution due to excitation of surface plasmon vibrations in silver nanoparticles. As the waste peel extract of lemon fruit was mixed with aqueous solution of the silver nitrate, it started to change the color from watery to brown due to reduction of silver ion; which indicated the formation of silver nanoparticles. It is generally recognized that UV-Vis spectroscopy could be used to examine size and shape-controlled nanoparticles in aqueous suspensions. **Fig. 3a**, shows the UV-Vis spectra recorded from the reaction medium after heating the solution at 75 °C for 60 min. Absorption spectra of silver nanoparticles formed in the reaction media has absorbance peak at 483 nm and broadening of peak indicated that the particles are polydispersed.

### 3.2 FTIR study of AgNPS by Peel extract of Lemon (*Citrus limon*)

The FTIR spectrum of AgNPs obtained in this study. FTIR measurement was carried out to identify the possible biomolecules responsible for capping and efficient stabilization of AgNPs synthesized using waste peel extract of lemon fruit. In the FTIR spectrum of waste peel extract of lemon fruit capped AgNPs, the spectrum showed absorptions at 3444 cm<sup>-1</sup> due to -OH group of polyphenolic compounds and the band observed at 1634 cm<sup>-1</sup> is due to C=O of -COOH respectively. The band Observed at 1350 carboxylic, aldehydes, ester, .etc phyto-compounds and also indicates carbonyl stretch in proteins. The strong peak at 1383.90cm<sup>-1</sup> corresponds to C-N stretching vibration of the amine with amide group. The peak of 1340.13 cm<sup>-1</sup>, implying the binding of silver ions with hydroxyl and carboxylate

groups of the extract, The peak near  $609.70\text{ cm}^{-1}$  is assigned to CH out of plane bending vibrations of substituted ethylene systems  $-\text{CH}_2\text{CH}_2-$ . In the case of nanoparticles, the peak of  $557.09\text{ cm}^{-1}$  reveals that Ag-O stretching vibrations. The spectra also illustrate a various functional groups in compounds of the Pomelo peel extract have interacted with AgNPs surface making AgNPs highly stable.

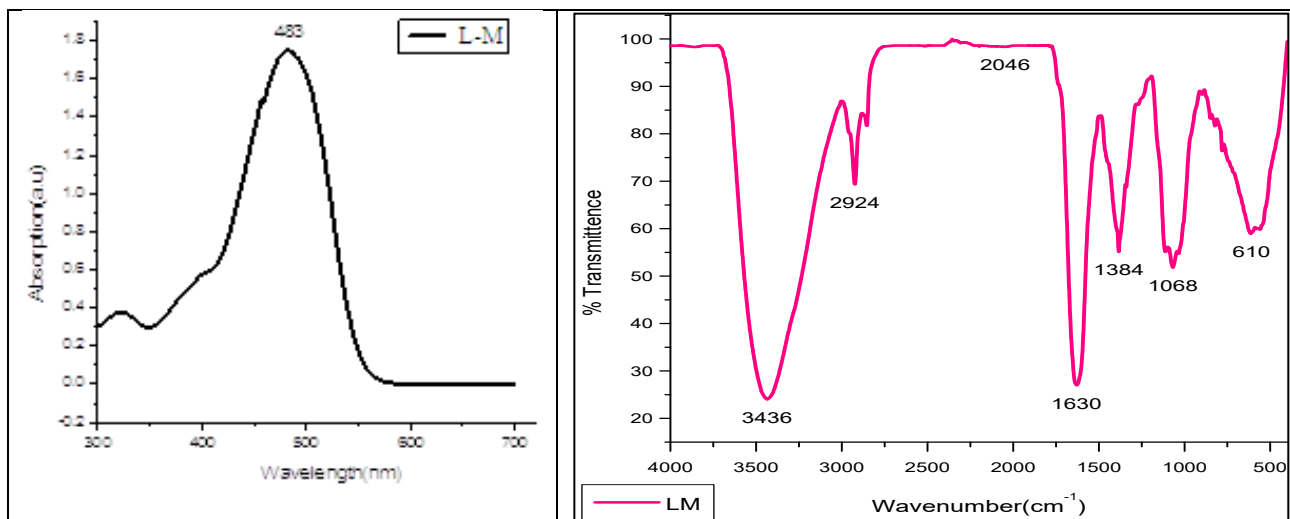
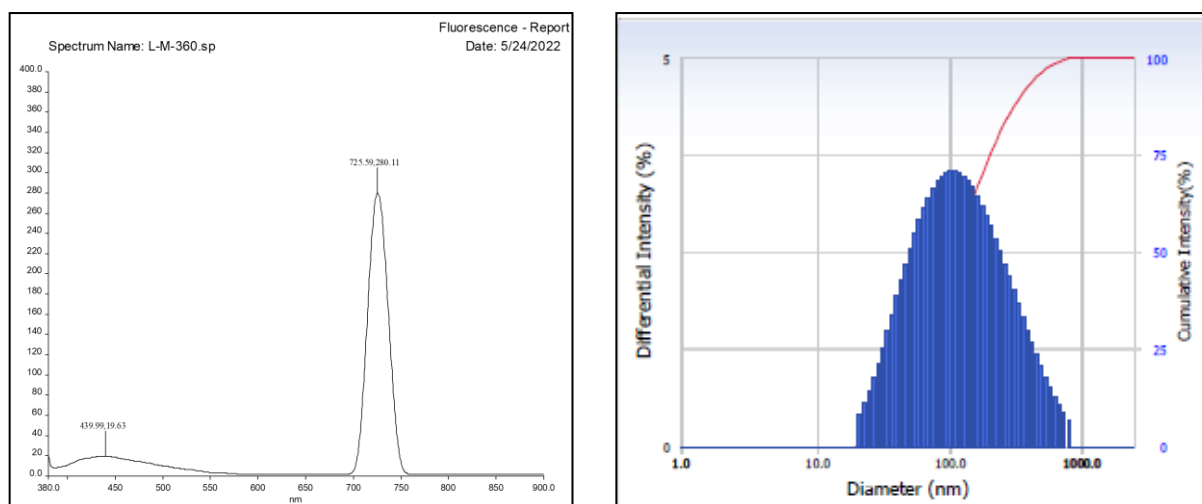


Figure:3a UV Spectrum of AgNPS by Orange peel extract Figure:3b FT-IR Spectrum of AgNPS by Lemon peel extract

### 3.3 Fluorescence spectroscopy

The fluorescence emission spectrum has been shown an obvious peak at  $725.59\text{ nm}$  in AgNPs sample, shows no trend in neither the Stern-Volmer quenching plot nor the direct fluorescence method (Figure 12). Overall, even in the presence of AgNPs nanoparticles by lemon waste peel aqueous extract.



### 3.4 DLS analysis

DLS is often interpreted to as a scattering of quasi-elastic light. It fulfills the role of size distribution and agglomeration of selective NPs. This process is quite sensitive, rapid and it can calculate the mean size of a particle on both macro and nano scale. The speed of the DLS technique is based on particle size. Small particles in suspension undergo random thermal motion known as Brownian motion. This random motion is modelled by the Stokes-Einstein equation. Below the equation is given in the form most often used for particle size analysis. The DLS analysis of green synthesized cobalt nanoparticle to shown the average size of the nanoparticle is  $99.08\text{ nm}$ . This result also shown in some of the nanoparticle having more than  $100\text{ nm}$ , this is due to growth of the nanoparticle and aggregation of the two is more nanoparticles.

### 3.5 SEM analysis of silver nanoparticles

The morphology determined by SEM analysis of green synthesized Ag-nanoparticle by waste peel of orange fruit aqueous extract has shown Figure; 14a and 14 b. It can be seen from SEM image that AgONPs are agglomerated and not well formed. One can vividly see well defined nanoparticles with distinguish shapes. Specifically, SEM image for AgONPs reveal distinguishable spherical shaped nanoparticles, elongated nanorods and highly agglomerated nanoparticles, AgONPs nanoparticles become well-formed revealing distinct shapes. To identify chemical elements and purity of synthesized samples Figure 14a shown in 2  $\mu\text{m}$  and 2  $\mu\text{m}$ , Figure 14 b has been shown 50  $\mu\text{m}$  and 1  $\mu\text{m}$ ,

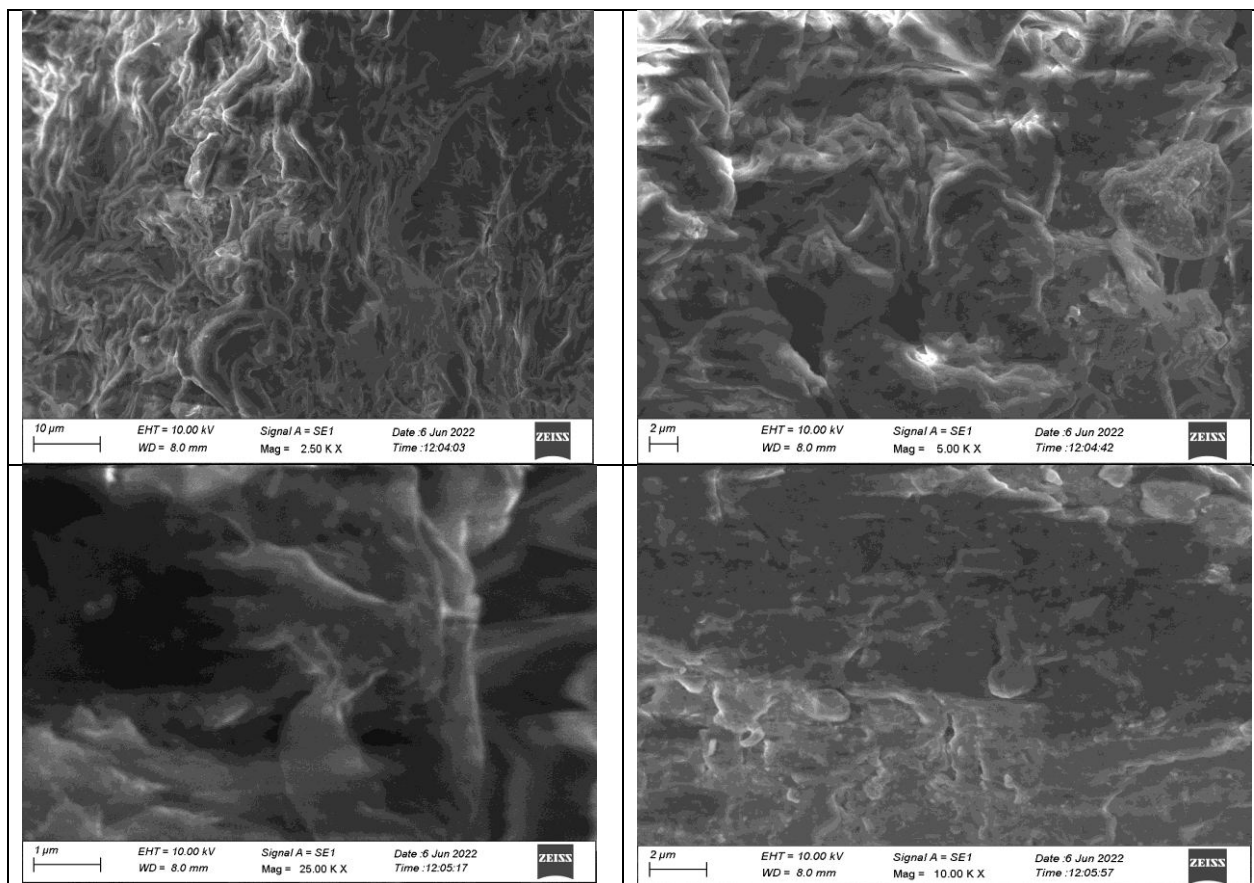


Figure:14 a SEM image of AgNPs by lemon peel extract

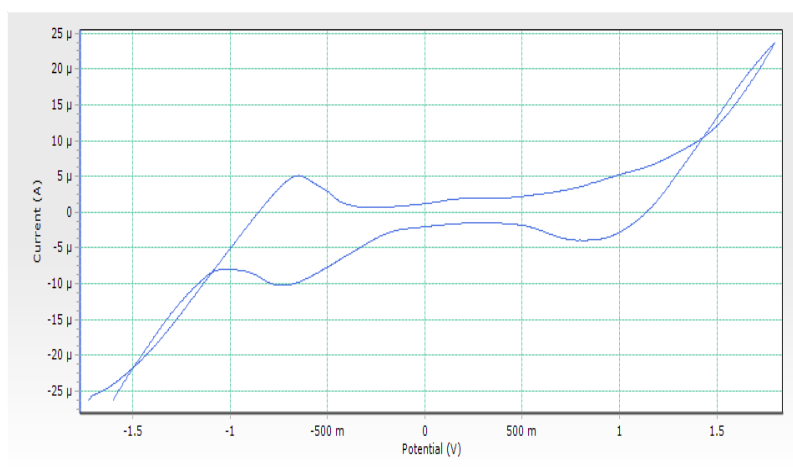
### 3.6 CV study of silver nanoparticles

The shape of the cyclic voltammetry curves is an ideal rectangular shape observed at 10 mV/s. Further increasing the scan rate the observed pattern of the CV curve is altered and it confirms the pseudo capacitive nature of the material.

The specific capacitance (SC) values of manganese ferrite electrode can be estimated by using the formula

$$C_s = Q / m\Delta V$$

Where,  $C_s$  is the specific capacitance,  $Q$  the anodic and cathodic charges on each scanning,  $m$  is the mass of the electrode material (mg) and  $\Delta V$  is the scan rate ( $\text{mVs}^{-1}$ ). Electrochemical measurements were performed in 0.2 M tetra butyl ammonium perchlorate with a standard three electrode configuration consisting of a sample (working electrode), an Ag/AgCl (reference electrode) and a high platinum wire (counter electrode). The scan rate increased in the range from 10 mV/s to 100 mV/s and its corresponding specific capacitance values depicted in Fig.15. Further, the specific capacitance values of 290.4 F/g observed in the scan rate of 10 mV/s for the sample annealed at 700°C. The reason for high specific capacitance at low scan rate is observed in the present study suggested that the ionic diffusion takes place both inner and outer surfaces.



The higher specific capacitance values observed in the present study confirm the good crystallinity of the cobalt nanoparticles.

#### IV. CONCLUSION

Nanotechnology will have large impact on rural development. Synthetic biology can revolutionize food production threatening traditional methods of agriculture. It is necessary to create international standards for nanotechnology and in addition special international organizations in the area of nanotechnologies to reduce national differences in assessing of nanotechnologies and risk governance practices. For these purposes it is necessary to create the research infrastructure for toxicology and risk assessment. In aspects of nanotechnology study courses it is necessary to define what kind of skills and knowledge are needed in a small, agricultural country to take advantage of nanotechnology and to manage risks that are likely to emerge with increasing commercialization of nanotechnology. Ultimately, nanotechnology innovations may enable the agricultural industry to precisely control and improve production by reducing the disease incidence and increasing the nutrient availability. The green synthesis of cobalt nanoparticles from Lemon (*Citrus limon*) peel aqueous extract was studied. The reduction of the metal ions led to the formation of silver nanoparticles of fairly well-defined dimensions using the extract. The synthesized nanoparticles are characterized by three spectroscopy such as UV, FTIR and FL spectroscopies, particle size measured by DLS and SEM analysis and electrical behaviour also carried out by Cyclic voltammetry study. This green chemistry approach towards the synthesis of silver nanoparticles has many advantages such as environmental friendly, cost effective and easily scaled up to large scale synthesis.

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