



## **Green Synthesis of Silver Nanoparticles Mediated by Waste Peel Extract of Pomelo (*Citrus Maxima*) Fruit**

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### **ABSTRACT**

There is an increasing commercial demand for nanoparticles due to their wide applicability in various areas such as electronics, catalysis, chemistry, energy and medicine. Metallic nanoparticles are traditionally synthesized by wet chemical techniques, where the chemicals used are quite often toxic and flammable. In this research article we present a simple and eco-friendly biosynthesis of silver nanoparticles using waste peel of Pomelo fruit aqueous extract as reducing agent. The aqueous silver ions when exposed to waste peel extract were reduced and resulted in silver nanoparticles whose average size was 35 nm. The silver nanoparticles were characterized by UV-Visible, Fourier transform infra-red spectroscopy (FT-IR), Florescence spectroscopy (FL), DLS analysis, SEM Analysis and also analyzed by cyclic voltammetry study of green synthesized silver nanoparticles

Keywords: Green synthesis, AgNPs, Citrus Maxima, UV, FT-IR, FL, DLS, SEM and CV

### **1. INTRODUCTION**

The field of nanotechnology is one of the most active areas of research in modern material science. Nanoparticles exhibit completely new or improved properties based on specific characteristics such as size, distribution and morphology. New applications of nanoparticles and nanomaterials are emerging rapidly[1-3] Nanocrystalline silver particles have found tremendous applications in the field of high sensitivity biomolecular detection and diagnostics[4], antimicrobials and therapeutics[5-6], catalysis[7] and micro-electronics[8]. However, there is still a need for economic, commercially viable as well environmentally clean route to synthesize silver nanoparticles. A number of approaches are available for the synthesis of silver nanoparticles for example, reduction in solutions[9], chemical and photochemical reactions in reverse micelles[10], thermal decomposition of silver compounds [11], radiation assisted[12], electrochemical[13], sonochemical[14], microwave assisted process[15] and recently via green chemistry route [16]. Unfortunately, many of the nanoparticle synthesis or production methods involve use of hazardous chemicals, low material conversions, high energy requirements, difficult and wasteful purifications. Biosynthetic methods employing either biological microorganisms or plant extracts have emerged as a simple and viable alternative to chemical synthetic procedures and physical methods. Most of the methods are still in the developmental stage and various problems are often experienced with the stability of nanoparticle preparations, control of crystal growth and aggregation of particles. Green synthesis provides advancement over chemical and physical method as it is cost effective, environment friendly, easily scaled up for large-scale synthesis and further there is no need to use high pressure, energy, temperature and toxic chemicals. Using plants for nanoparticle synthesis can be advantageous over other biological processes because it eliminates the elaborate process of maintaining cell cultures and can also be suitably scaled up for large-scale synthesis of nanoparticles under non- aseptic environment. Silver nanoparticles play a profound role in the field of biology and medicine due to their attractive physiochemical properties. Silver products have long been known to have strong inhibitory and bactericidal effects, as well as a broad spectrum of antimicrobial activities, which has been used for centuries to prevent and treat various diseases, most notably infections[17]. Silver nanoparticles are reported to possess anti-fungal[18], anti-inflammatory[19], anti-viral[20], anti-angiogenesis[21] and antiplatelet[22] activity. Here in, we report for the synthesis of silver nanoparticles, reducing the silver ions present in the solution of silver nitrate by the waste peel extract of pomelo fruit.

### **2.MATERIAL AND METHODS**

#### **2.1 Sample collection**

The *Pomelo (Citrus maxima)* fruit purchased from fruit stall in Chidambaram town, the purchased fruits after eaten peels are through to the dust pin. The collected waste peels was washed thrice in the running tap 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 *Pomelo (Citrus maxima)* 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 peels Pomelo (*Citrus maxima*)

5 grams of *Pomelo (Citrus maxima)* fruit peel powder 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 $\mu$  filter paper. The filtrate was used to the further green synthesis of process.

## 2.5 Green synthesis of Silver nanoparticle (AgNPs) from waste fruit peels Pomelo (*Citrus maxima*) extract

Silver nitrate (AgNO<sub>3</sub>) solution of 0.1m M was prepared. Drop wise 10 ml of waste fruit peels extracts of *Pomelo (Citrus maxima)* has been added and colour 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 block colour. The obtained precipitate was filtered whatmann 1 $\mu$  filter paper. The nanoparticles containing filter paper was dried hot air oven and collect the air tight click lock tube. The green synthesized silver nanoparticles were characterized by UV, FTIR, FL, DLS, SEM, and CV analysis.

## 3. CHARACTERIZATION

The UV-vis spectra were recorded at room temperature using a k-Helios SP Pye-Unicam spectrophotometer. Fourier transform infrared (FTIR) spectra were recorded at room temperature on a Nicolet 6700 FTIR spectrometer. For the FTIR measurements of capped silver nanoparticles, a small amount of silver nanoparticles (0.01 g) dried at 60<sup>o</sup>C for 4 h was mixed with KBr to form a round disk suitable for FTIR measurements. To obtain the FTIR Spectrum of the extract, an appropriate amount of the extract was mixed with KBr. Fluorecence spectra were recorded on a Perkin Elmer LS 50B fluorescence spectrophotometer. DLS Analysis was carried out by particle of the green synthesized nanoparticles. The size of the particle and exterior morphology of biosynthesized AgNPs were deliberated by SEM (Scanning electron microscope), Cyclic voltammetry have used to find the reduction potential of the green synthesized silver nanoparticles.

## 4. RESULT AND DISCUSSION



Figure:1 (a) and (b) *Pomelo (Citrus maxima)*, (c) Waste peel of *Pomelo (Citrus maxima)*

### 4.1 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 pomelo 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. 2, shows the UV-Vis spectra recorded from the reaction medium after heating the solution at 75 <sup>o</sup>C for 60 min. Absorption spectra of silver nanoparticles formed in the reaction media has absorbance peak at 478 nm and broadening of peak indicated that the particles are polydispersed.

#### 4.2 FTIR study of AgNPs by Pomelo peels extract

In the FTIR spectrum of Pomelo peel extract capped AgNPs (Fig:3). FTIR measurements were carried out to identify the possible biomolecules responsible for capping and efficient stabilization of the metal nanoparticles synthesized by waste peel broth. The peak IR bands observed at 3418.51 characteristic of the O–H stretching, 2923.46 and 2893.46  $\text{cm}^{-1}$  characterize alkane stretching vibrations, The band of 1632.81  $\text{cm}^{-1}$  have shown in and C=O stretching for carboxylic, aldehydic, ester, etc compounds and also indicates carbonyl stretch in proteins. Band at 1561.06  $\text{cm}^{-1}$  corresponds to amide rising due to. The strong peak at 1384.43  $\text{cm}^{-1}$  corresponds to C–N stretching vibration of the amine. The peak of 1070.61  $\text{cm}^{-1}$ , implying the binding of silver ions with hydroxyl and carboxylate groups of the extract, The peak near 574.48  $\text{cm}^{-1}$  is assigned to CH out of plane bending vibrations of substituted ethylene systems –CH<sub>2</sub>CH. In the case of nanoparticles, the peak of 490.34  $\text{cm}^{-1}$  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.

#### 4.3 Fluorescence spectroscopy

The fluorescence emission spectrum has been shown an obvious peak at 725.71 nm in AgNPs sample, shows no trend in neither the Stern-Volmer quenching plot nor the direct fluorescence method (Figure 4). Overall, even in the presence of AgNPs nanoparticles by pomelo waste peel aqueous extract.

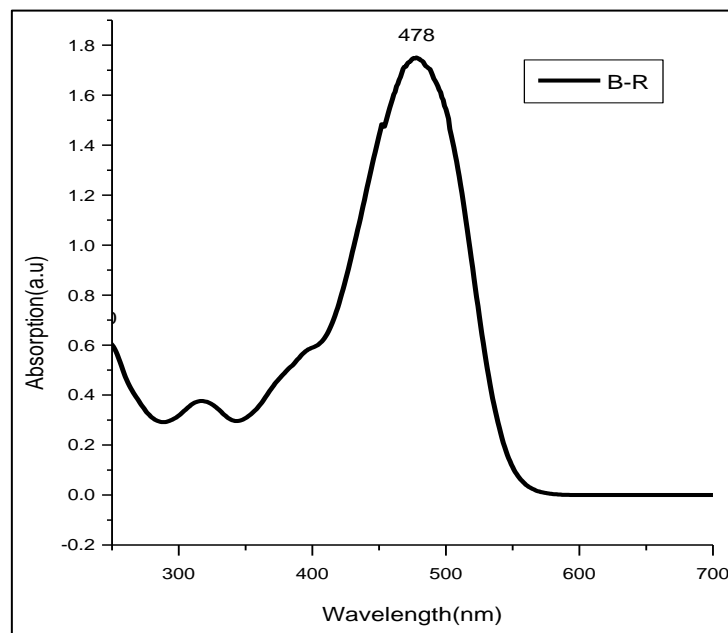


Figure:2 UV Spectrum of AgNPS by Pomelo peel extract

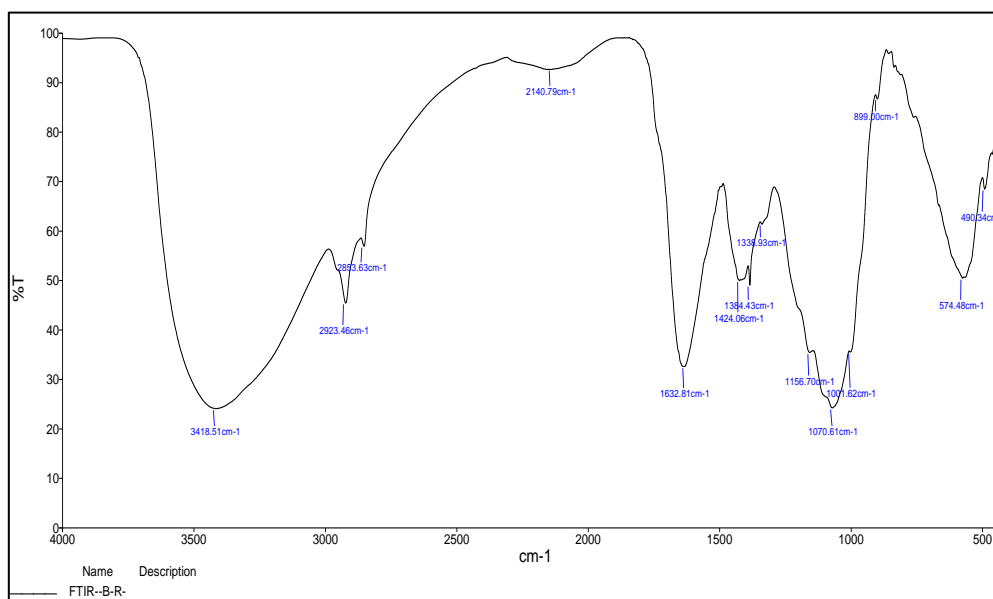


Figure:3 FTIR Spectrum of AgNPS by Pomelo peel extract

#### 4.4 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 (**Fig:5**). Small particles in suspension undergo random thermal motion known as Brownian motion. This random motion is modeled 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 96.08 nm. This result also shown in some of the nanoparticle having more than 100 nm, this is due to growth of the nanoparticle and aggregation of the two is more nanoparticles

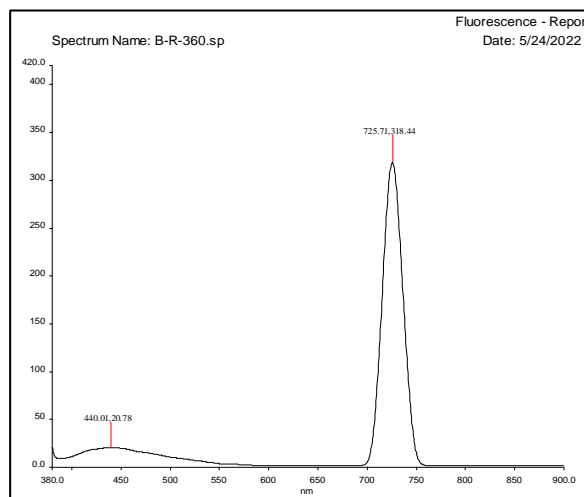


Figure:4 FL-Spectrum of AgNPS by Pomelo peel extract

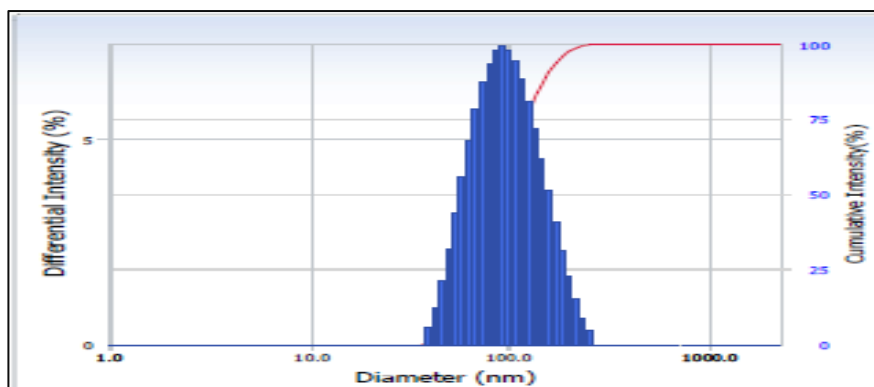


Figure:5 DLS spectrum of AgNPS by Pomelo peel extract.

#### 4.5 SEM analysis of silver nanoparticles

The morphology determined by SEM analysis of green synthesized AgO nanoparticle by the Pomelo peel aqueous extract has shown Figure:6. It can be seen from SEM image that AgO NPs are agglomerated and not well formed. One can vividly see well defined nanoparticles with distinguish shapes. Specifically, SEM image for AgO NPs reveal distinguishable rhomboid shaped nanoparticles, elongated nanorods and highly agglomerated nanoparticles, AgO nanoparticles become well-formed revealing distinct shapes

#### 4.6 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 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 100mV/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 silver nanoparticles.

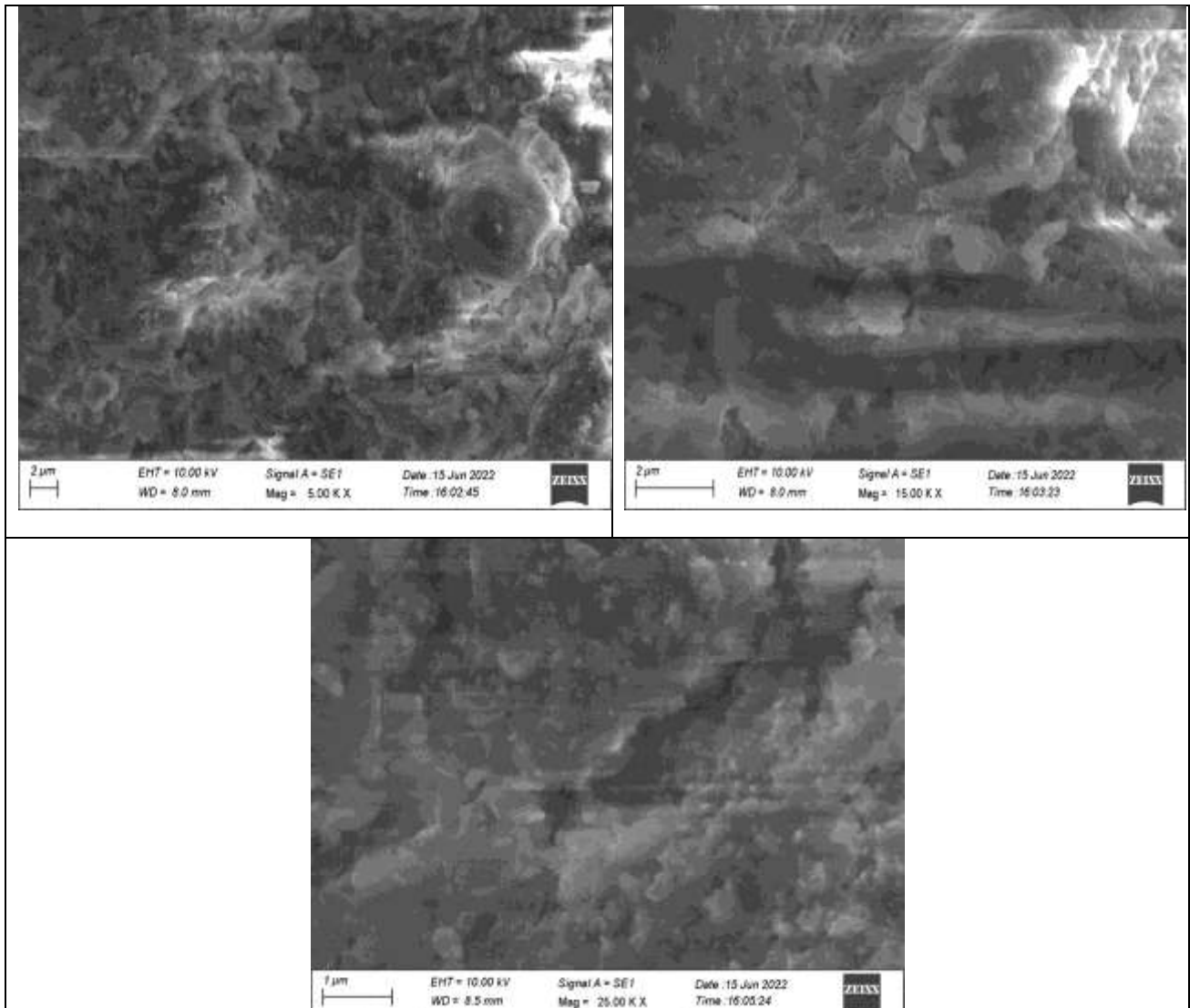


Figure:6 SEM image of AgNPs by Pomelo peel extract

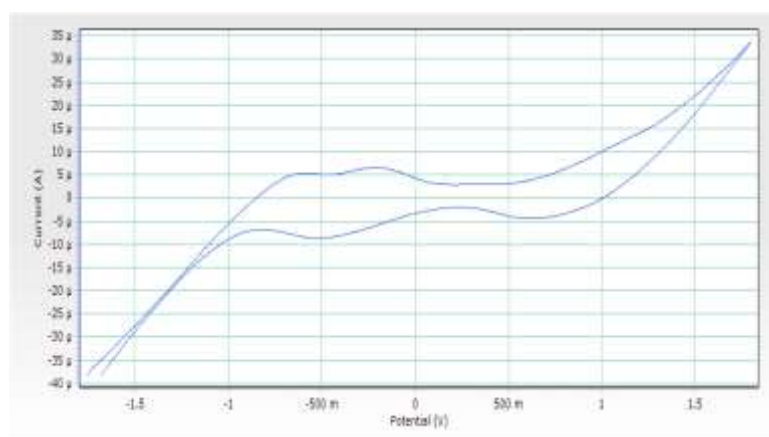


Figure:7 CV spectrum of AgNPs by Pomelo peel extract

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## 5. 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 Pomelo 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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