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STABILITY STUDIES ON SILVER NANOPARTICLES SYNTHESIZED BY GREEN METHOD USING PHYLLANTHUS NIRURI AND SOLANUM NIGRUM LEAF EXTRACTS

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Abstract-- The present work was aimed to study the stability of silver nanoparticles synthesized from silver nitrate solution by green synthesis using binary mixture of extracts from the fresh leaves of plants of PhyllanthusNiruri and Solanum Nigrumcollected from in and around Kancheepuram district. The collected leaves were washed thoroughly by distilled water and extract mixture was prepared by boiling the leaves in distilled water. The binary extract mixture was mixed with silver nitrate solution kept at various concentrations. The silver nitrate solution showed various brown colour for various concentrations of the solution. The change in colour indicated the formation of silver nanoparticles. The preliminary confirmation on silver nanoparticles was done by Ultraviolet-Visible Spectroscopy (UV-Vis). Fourier Transform Infrared Spectroscopy (FTIR) analysis on the solution-extract mixture identified various reducing and capping agents present in the extract mixture. The synthesized silver nanoparticles in the solution were kept undisturbed for four weeks. Stability of silver nanoparticles was observed through analyzing their existence in the solution using Scanning Electron Microscope (SEM), Dynamic Light Scattering (DLS) and Zeta potential analyses. The nanoparticles showed stability to an optimum level.

I. INTRODUCTION

Nanoparticles are the particles which have the grain size lying between 1 to 100 nanometer roughly(1). Nanoparticles have high surface to volume ratio due to their nanoscale sizes and this in turn give rise to enhanced physical and chemical properties. Nanotechnology is an emerging field of science and it creates impact in solving many issues related to health and energy towards the need of the society. This is due to the fact that metal nanoparticles find applications in medicine (2), sensing and electronics (3). Nanotechnology defines a path to design, produce devices and systems through controlling the size and shape of the particle in nanoscale (4). Metal nanoparticles draw more attention due to their wide applications in various industries (5). One of the remarkable properties of nanomaterials is their anti-bacterial activity towards micro-organism (6,7). Silver nanoparticles have recently gained importance because of their good conductivity, stability, catalytic activity and mainly find applications in industries especially in medical industry in treating virus and as an anti-bacterial agent.

Silver nanoparticles have unique electrical and optical qualities. The nano particles can be synthesized by various physical and chemical methods and these methods sometimes lay basis for toxic substances to get adsorbed on to nanomaterials. In order to avoid this, green synthesis methods are now employed in nano synthesis. Green synthesis methods utilize certain principles which reduce the usage of hazardous substances during the synthesis [8].

II. SCOPE OF THE STUDY

The scope of this study is to synthesize silver nano particles using bi-component mixture of extracts from two different plant leaves and to study the stability if silver nanoparticles after 4 weeks from the day of preparation. The above goals were achieved by using FTIR, UV-Vis, SEM, DLS and Zeta potential analyses.

MATERIALS AND METHODS

Materials

Plant materials

Plants contain various bio active ingredients which act as reducing and capping agents during the synthesis of silver nanoparticles from silver nitrate solution(2-4). The bio active ingredients are available in various parts of the plant such as plant stem, root, leave and flowers, etc. In this study plant leaves were used for the synthesis of silver nanoparticles.

Sample Collection

Fresh leaves of plants of PhyllanthusNiruri (Keezhanelli) and Solanum Nigrum (Manathakkali) were collected from in and around Kancheepuram district.

Sample preparation

The fresh leaves were washed several times with plain water and then finally with distilled water. 10g of leaves of Keezhanelli and Manathakkaliare weighed and boiled for 30 minutes in200 ml of distilled water and it is filteredusing Whattman filter paper. Then the binary extract is stored in refrigerator for further use. This extract is used as reducing and stabilizing agent. Aqueous solutions of silver nitrate at various concentrations ((0.001, 0.015, 0.002, 0.0025, 0.003M) were prepared by adding required amounts of silver nitrate in 200 ml of double distilled water and it is stirred for 1 hour. Further 20 ml of silver nitrate solutions are taken from each concentration to synthesize silver nanoparticles.

III. METHODS

Ultraviolet-visible (UV-vis) spectroscopy analysis

The formation of silver nanoparticles was confirmed by Ultraviolet – Visible (UV-Vis) Spectroscopy using UV-Vis spectrophotometer facility at SCSVMV University, Enathur, Kancheepuram. UV-vis spectroscopy has been proved to be a very useful technique for nanoparticle study as peak positions and shapes in spectra are sensitive to particle size.

Fourier Transform Infrared (FTIR) Spectroscopy

The silver nitrate solution mixed with the extract was subjected to FTIR spectroscopy analysis using FTIR Spectrometer facility at SCSVMV University, Enathur, Kancheepuram. The FTIR absorption peaks indicated various bio-active components present in the extract.

Scanning Electron Microscope (SEM)

The formation of silver nanoparticles was initially confirmed by the change in colour of the solution from transparent to brown. The solution becomes dark brown as it left for 4 weeks undisturbed. The aged solution was subjected to SEM analysis to study the stability of nanoparticles. SEM facility at St.Joseph's college, Trichy was utilized for this study.

Dynamic Light Scattering (DLS) and Zeta Potential analysis

DLS techniques is very much useful in detecting the silver nanoparticle size range and average particle size present in the solution, The silver nitrate solution mixed with the extract was subjected to DLS and Zeta potential analysis. Zeta potential is concerned with surface of nanomaterial and playing domination role in the stability of nanoparticles.

IV.RESULTS AND DISCUSSION

UV-Vis Spectroscopic Study

The addition of leaf extract to silver nitrate ($AgNO_3$) solution resulted in color change of the solution from transparent to brown due to the formation of silver nanoparticles. The color changes arise from the excitation of Surface Plasmon vibrations with the silver nanoparticles.UV-Vis Spectrum shows characteristic Surface Plasmon Resonance (SPR) peaks which confirm the presence of nanoparticles (5) and it is shown in figure 1. The Surface Plasmon Resonance (SPR) peak that appeared initially at the wavelength 430 nm for leaf extract added with 0.001M of silver nitrate solution shifted to 442 nm due to adding leaf extract with 0.003M of Silver nitrate solution.

The shift in wavelength is responsible for the change in colors of solutions (6). The red shift in SPR peak is due to the formation of larger grains in the solution. The shape of the peak also determines the shape of the nanoparticles. Sharp peaks and multiple peaks in the spectrum are the characteristics of the size of nanoparticles.

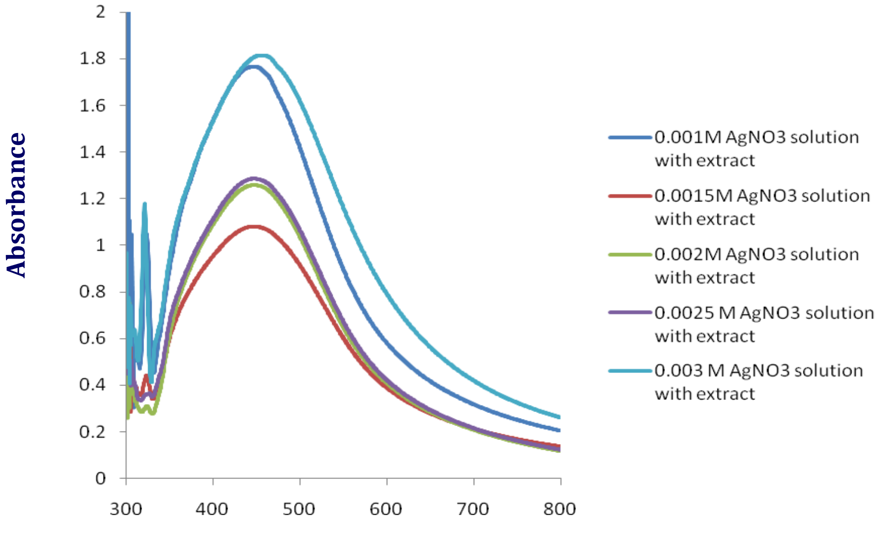


Fig.1. UV- Vis spectrum of Silver nitrate solutionsat different concentrations with extract

FTIR Spectroscopic study

FTIR measurements are carried out to identify the potential biomolecules in the plant leaf responsible for reduction and capping of the bio-reduced silver nanoparticles.The peaks observed are at 3269, 1635, 516, 464 and 413cm^{-1} as shown in figure 2. The peaks observed at 3269 cm^{-1} corresponds to O-H stretching with strong or broad intensities of alcohol or phenol. The absorption peak at 1635 cm^{-1} is close to native proteins present in the leaf extract which suggest that proteins are interacting with biosynthesized nanoparticles and also their secondary structure were not affected during reaction with silver ions or after binding with silver nanoparticles. The absorption peak at 516cm^{-1} is due to O-C-O in plane bending. The weak transitions at 464 cm^{-1} may be associated with water liberations. The UV – Vis spectroscopy results confirm the existence of silver nanoparticles through SPR peaks in the spectrum. Further FTIR spectroscopy confirmed the presence of reducing and stabilizing agents present in the leaf extract mixture.

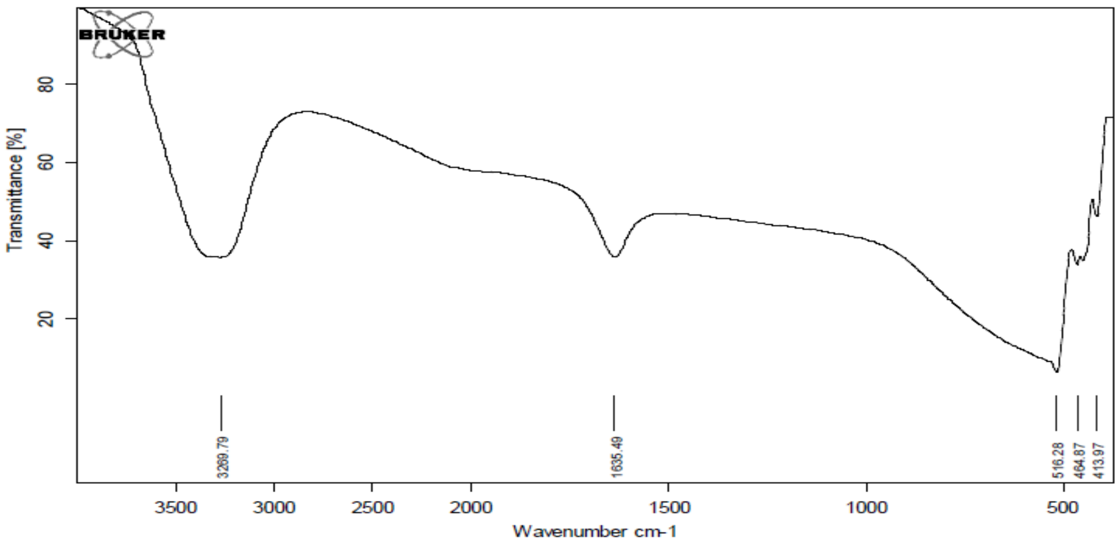
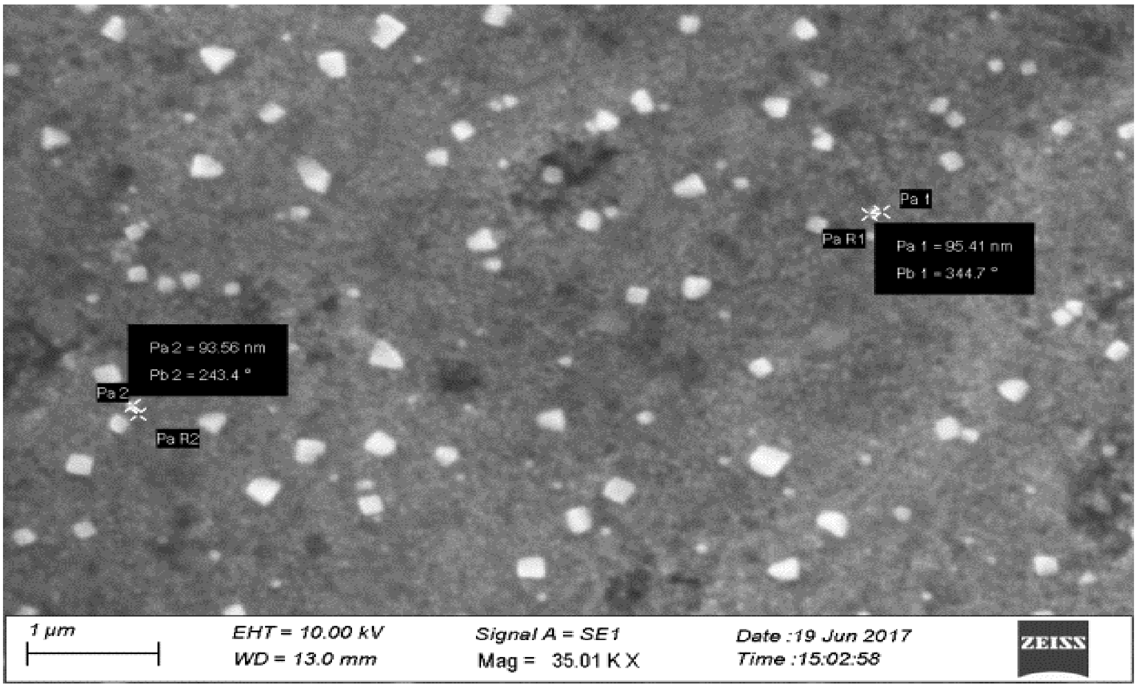


Fig.2. FTIR Spectrum for leaf extract with solution at 0 .001M concentration

Scanning Electron Microscope

The addition of leaf extract to silver nitrate ($AgNO_3$) solution resulted in color change of the solution from transparent to brown due to the formation of silver nanoparticles. The color changes arise from the excitation of Surface Plasmon vibrations with the silver nanoparticles.

The solution with leaf extract was subjected SEM analysis and the images are shown in the figure 3. The SEM images show clearly the presence of nanoparticles in the nanoscale range 52 – 94 nanometer. The majority portion of nanoparticle show cubical shape. But some of the nanoparticles are in triangular and spherical shapes, but not well defined. The SEM images show approximately 30 % of particles is in nanoscale while the other exceeds in size and some of the nanoparticles are in various shapes than cubical one.



From the above results, it can be concluded that the nanoparticles showed an optimum stability in retaining their size and this could be due to the bio-active agents of the extract mixture. The bi-component mixture may also be a reason for the formation of multi shaped nanoparticle formation.

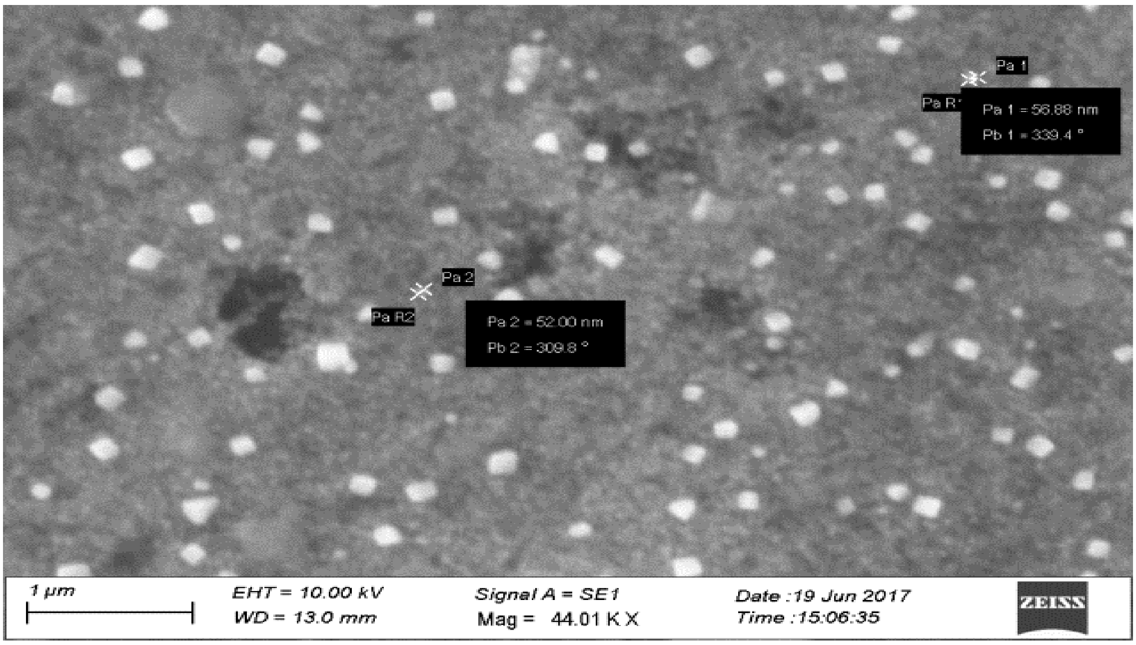


Fig.3.SEM images of silver nanoparticle synthesized from 0.001Msilvernitrate solution with leaf extract

Dynamic Light Scattering and Zeta Potential Analysis

The DLS and Zeta potential analyses results are shown in figure 4a for 0.001M solution after 4 weeks. From the figure 4a, the calculated z-average particle size distribution of silver nanoparticle is 407.88 with minimum peak at 44.22 nm. This confirms the results offered by SEM images where nanoparticles with a range 52 – 94 were detected.

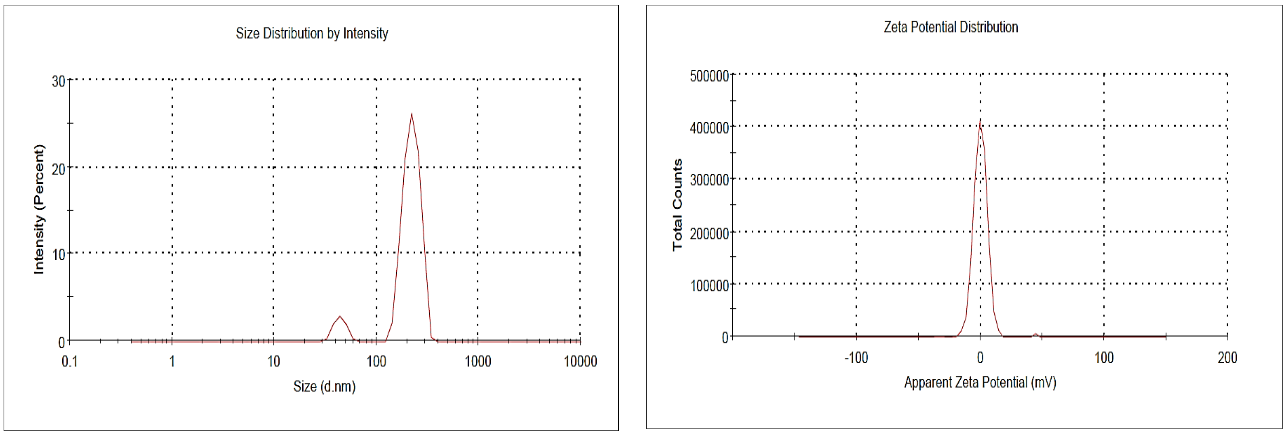


Fig.4a.DLS and Zeta potential of synthesized silver nanoparticles from 0.001M silver nitrate solution with leaf extract

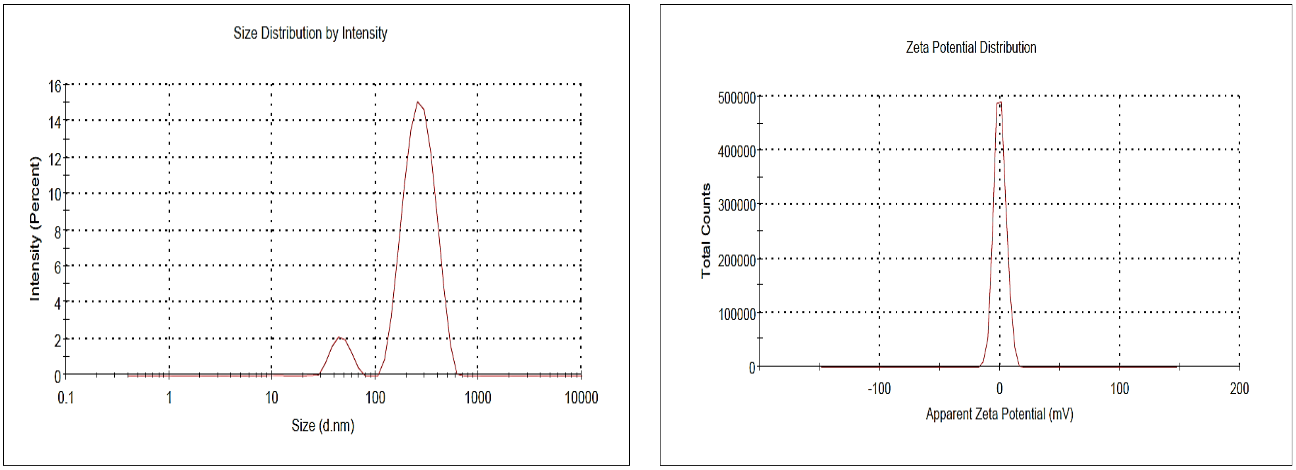


Fig.4b.DLS and Zeta potential of synthesized silver nano particles from 0.003M Silver nitrate solution with leaf extract

The DLS and Zeta potential analyses results are shown in figure4b for 0.003M solution after 4 weeks. From the figure 4b, the calculated z-average particle size distribution of silver nano particle is 288.7 nm with minimum peak at 46.98 nm. This confirms the results offered by SEM images where nanoparticles with a range range 52 – 94 were detected. The Z-Average size is intensity based and higher values show the presence of large size particles in more number. The corresponding average zeta potential value is -0.00453, and-0.0602mV (Fig.4a&4b) at 0.001, and 0.003M respectively. Since zeta potential decides the agglomeration action in nanoparticles, agglomeration will be less if zeta potential is more and vice versa. In this study, zeta potentials are low in value and this may due to the reason of longer duration of 4 week.

V.CONCLUSIONS

From the above, it may be concluded that the bi-component mixture attributes for multi-shaped nanoparticles because of the bio-active components present in the mixture, as revealed by SEM results. A minimum stability of nanoparticles (30% approx.) was observed in SEM study after 4 week period and this may be due to the bio active components of bi-component extract mixture. DLS results confirmed the nanoparticle presence in the solution as per the results of SEM. Further it gives a range of size of nanoparticles in which 30% of scale lies in nano range. Zeta potential analysis revealed the zeta (surface) potential of nanoparticles and which are found to be low. This may be due to the age of the solution in which potential decreases with respect to the time. This is already confirmed by the SEM results where most of the particles are grown beyond nanoscale due to agglomeration.

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