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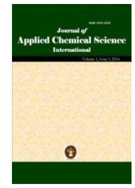

GREEN SYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES USING *Calotropis procera* EXTRACT

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AUTHORS' CONTRIBUTIONS

This work was carried out in collaboration between all authors. Author AOD designed the study, wrote the protocol and interpreted the data, gathered the initial data and performed data analysis. Authors OJO and APO initiated the speedy accomplishment of the study. Author FED anchored the field study. Authors FED and OJA managed the literature searches and produced the initial draft. All authors read and approved the final manuscript.

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ABSTRACT

The green synthesis of silver nanoparticles (Ag-np) using leaves extract from *Calotropis Porocera* has been investigated. Extract of *Calotropis procera* was used as both reducing and stabilizing agent instead of the general convention of the use of sodium borohydride solution. Ag-np was characterized using a combination of spectroscopic techniques: Ultraviolet-Visible (UV-VIS) Spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), Scanning and Transmission Electron Microscopy (SEM and TEM), and Energy Dispersive X-ray (EDX). The spectrum from UV-VIS depicted the wavelength for the formation of Ag-np at 420 nm. Both SEM and TEM depicted the morphology and size of the nanoparticles while the elemental analysis and the relative abundance of each element in the nanoparticles were revealed through the EDX. This research gives a clear efficacy and reality of the use of low cost and environmentally benign extract a main precursor in the synthesis of Ag nanoparticles.

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Keywords: *Calotropis procera;* green synthesis; Ag nanoparticle; UV-Vis; FTIR; SEM-EDX; TEM.

1. INTRODUCTION

Nanotechnology is as aspect of modern synthetic chemistry mainly concerned with growing and development of nanoparticles and their hybrid with variable sizes, shapes, chemical structure which find relevance and application in several fields such as medicine benefiting human being [1-3]. However, the current trend is the use of green chemistry approach in the synthesis of nanoparticles. Although, the use of chemical (Sodium borohydride) and physical (mechanical attrition) methods may successfully produce pure, well defined nanoparticles but these are quite expensive and potentially dangerous to the environment if disposed carelessly. Therefore, green synthesis as the alternative method of preparing nanoparticles involves the use of plant extract. Polyphenols, the active agent in the plant extract possesses both reducing and capping characteristics [3]. A number of researchers have worked on the green synthesis of silver different plant extracts but the use of *calotropis procera* extract has not been totally explored. So far, the green synthesis of Ag nanoparticles using the following plants' extracts have been reported: *D. carota* extract [4], *Punica granatum* peel [5], *Sinorhizobial octasaccharide* isolated from *sinorhizobium meliloti* [6], *Polyalthia longifolia* leaf extract along with D-Sorbitol [7], aqueous extract of *Azadirachta indica* leaves [8], bamboo hemicelluloses and glucose [9] and sucrose and maltose [10], *Malva parviflora* [11], and *Ocimum sanctum* leaf extract [12]. The objective of this research is to develop reliable, simple, reproducible and eco-friendly green approach for synthesis of Ag nanoparticles biologically as an important step in the field of application of nano-science and nanotechnology using *Calotropis procera* leaves extract.

2. MATERIALS AND METHODS

2.1 Preparation of *Calotropis procera* Leaves Extract

The *Calotropis procera* (Sodom apple) leaves were collected outside Landmark University Campus in Omu-Aran, Kwara State, Nigeria. All reagent used were of analytical grades obtained from Sigma-Aldrich, USA and all experiments were carried out using distilled-deionized water. Fresh leaves of *Calotropis procera* were collected, washed thoroughly with distilled-deionized water, and incised into small pieces. About 10 g of finely cut *Calotropis procera* leaves were weighed and transferred into a 250 cm^3 beaker containing 100 cm^3 deionized water, mixed well, and boiled for 5 minutes. The extract obtained was filtered through Whatman No. 1 filter paper, the filtrate was collected in 250 cm^3 Erlenmeyer flask and used thereafter [13].

2.2 Synthesis of Silver Nanoparticles (Ag-np)

Shown in Fig. 1 is the typical picture of *Calotropis procera* (C.P) plant. The green synthesis of Ag-np was prepared following the method reported in the literature [5,6]. Preparation was done by reacting 10 mL of the C.P extract with 90 mL $AgNO_3$ solution (1 mM) and was agitated on the air bath magnetic stirrer for 15 minutes at room temperature. A colour change was observed from colourless to pink. The mixture was centrifuged and dried in the oven at temperature between 50°C – 60°C overnight. Silver nanoparticles was characterized using a combination of spectroscopic techniques: UV-Vis, FTIR, SEM, EDX and TEM.

2.3 Characterization of *Calotropis* Silver Nanoparticles (C-Ag-np)

2.3.1 Ultraviolet-visible spectroscopy (UV-Vis)

The Absorption bands arising from the Surface Plasmon Resonance in the C-Ag–np were measured using a Beckmann Coulter DU 730 Life Science UV-VIS spectrophotometer. The absorbance is taken between the range of 200 nm – 800 nm to determine the maximum wavelength or peak where for Surface Plasmon Resonance (SPR). The absorbance of silver nanoparticle dispersed in a quartz cuvette with a 1 cm optical path was measured by withdrawing small aliquot from the reaction mixture and wavelength scan was taken at 60 minutes intervals from 200 nm to 800 nm until a stable absorbance was obtained at maximum wavelength [14].

2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

The information on the molecular environment and functional groups in the extract were investigated using Schimadzu FTIR model IR 8400S.

2.3.3 Scanning Electron Microscopy and Energy Dispersive X-ray (SEM-EDX)

The surface morphological characterization and elemental analysis were carried using a scanning electron microscopy (SEM) integrated with Energy Dispersive X-ray (EDX) analyzer. SEM images and EDX spectra were obtained using a TESCAN Vega TS 5136LM typically at 20 kV at a working distance of 20 mm. Samples for SEM analysis were prepared by coating them in gold using a Balzers' Spluttering device.

2.3.4 Transmission Electron Microscopy (TEM)

The transmission electron microscopy (TEM) was carried out using A Zeiss Libra 120 transmission electron microscope at 80 kV. This measurement was useful to determine the size and shape of the nanostructures. Samples were well dispersed, supported on a copper grid and dried at ambient temperature.

3. RESULTS AND DISCUSSION

3.1 UV-VIS Spectroscopy

The Ag-np was synthesized using aqueous extract of *Calotropis procera*. Before addition of *Calotropis* extract, the Ag salt solution was colourless. Hence, after addition of the *Calotropis procera extract*, the colour changed to pink which was an indication of the formation of silver nanoparticle (Ag-np). This finding was congruent the report of other researchers [6,15- 20]. In metal nanoparticles, the conduction band and valence band lies very close to each other in which electrons move freely. These free electrons gave rise to a Surface Plasmon Resonance absorption band, occurring due to collective oscillation of electrons in resonance with light wave. The absorption spectrum showed a Surface Plasmon Resonance (SPR) and peak was observed at 420 nm for C-Ag-np as observed in Fig. 2.

Fig. 1. Typical plant of *Calotropis Procera*

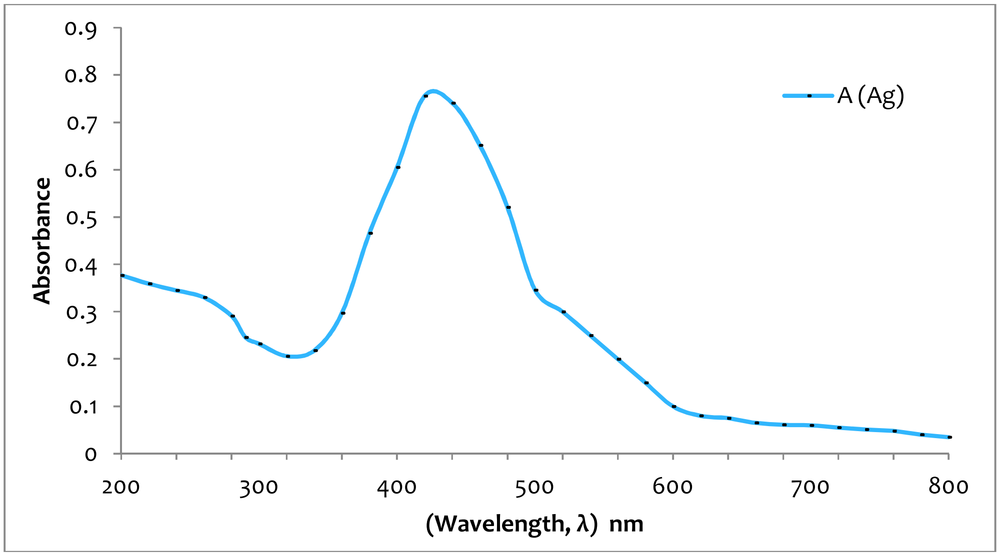

Fig. 2. UV-Vis spectrum of C-Ag-np

3.2 FTIR Analysis

The FTIR analysis was carried out to identify the potential biomolecules in *Calotropis procera* responsible for reducing and capping the silver nanoparticles. FTIR signal at 1635 cm^{-1} was attributed to C=O stretching of amide group which is responsible for the reduction of Ag^{+} to Ag^{0} [21,22]. The band at 3445 cm^{-1} is attributed to N-H band of amine [23], peak at 1386 cm^{-1} may be attributed to C–H bending of carboxylic acid. The peaks at 572.88 cm^{-1}, 516.94 cm^{-1} and 420.50 cm^{-1} may be attributed to C-H bending of Alkyne. The absence of peak at 1414 cm−1 may be due to the capping action by C–O group in the synthesis of AgNPs. These various peaks indicated the presence of biomolecules which are responsible for the reduction and stabilization of Ag–np [23 - 25].

3.3 SEM-EDX Analyses

Fig. 4 shows the SEM micrograph of C-Ag-np prepared by bio-reduction of Ag^{+} using extract of *Calotropis procera*. The image revealed some irregular dispersion and pores indicating that the Ag nanoparticles could be used as adsorbent as well as anti-bacteria material.

The EDX analysis gave qualitative as well as quantitative status of the constituents of a nanoparticle [26,27]. Fig. 5 shows elemental profile of C-Ag-np synthesized using extract of *Calotropis procera*. As shown in Table 1, the EDX elemental percentage composition of Ag –np synthesized using leaf extract of *Calotropis procera* revealed highest proportion by weight of silver (68.78%) in nanoparticle. Other elements such as carbon (28.85%), nitrogen (10.13%), oxygen (11.91%) and e.t.c. may arise from the *Calotropis procera* extract (Plant broth), the copper grid or other additives used during the analysis. The Tellurium may arise from the plant broth which contains some biomolecules. The strongest peak observed (Fig. 5) was from Ag-np peculiar at 3keV. Generally, metallic silver nanocrystals show typical optical absorption peak approximately at 3 keV due to their Surface Plasmon Resonance**.** One of the advantages of green synthesis is the presence of other phytochemical elements from the EDX spectrum which may possible arise from the *C. procera* extra serving as the capping agents. This is supported by the findings of other researchers [8,16–20, 24,25,28,29].

3.4 TEM Analysis

The TEM analysis was further employed to characterize the size and shape of C-Ag-np. Small samples of C-Ag-np was dropped on the copper grid and allowed to dry before carrying out the TEM analysis on Zeiss Libra 120 @ 80kV**.** Fig. 6 shows a spherical shape and size variation 3 – 26 nm due to some agglomeration of C-Ag-np which was totally dispersed on the surface. This finding was supported by the report in literature [5, 22–29].

Table 1. EDX Elemental percentage composition of C-Ag-np

Element	Weight %	Atomic %
Ag	67.78	12.58
C	28.85	48.08
N	10.13	14.48
O	11.91	14.90
Mg	0.14	0.11
P	0.51	0.33
S	0.82	0.51
Cl	15.54	8.77
K	0.31	0.16
Te	0.43	0.07
Total	136.43	100

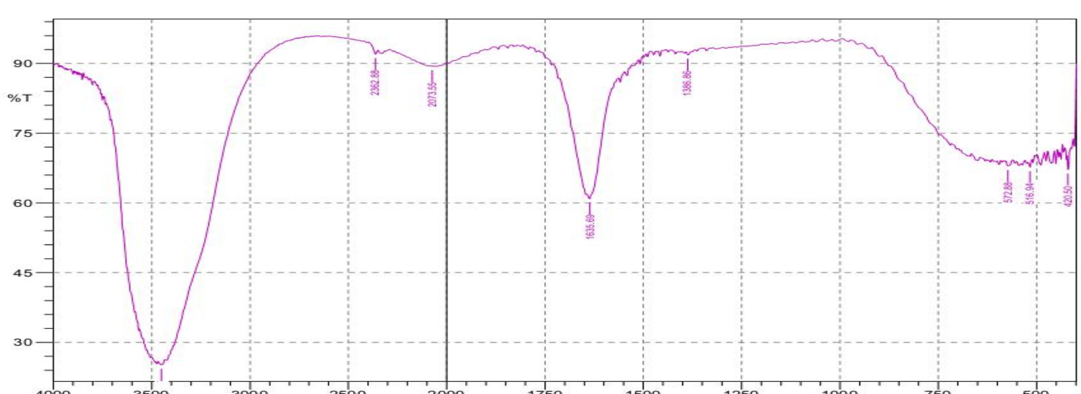

Fig. 3. FTIR Spectrum of *Calotropis procera* leaf extract

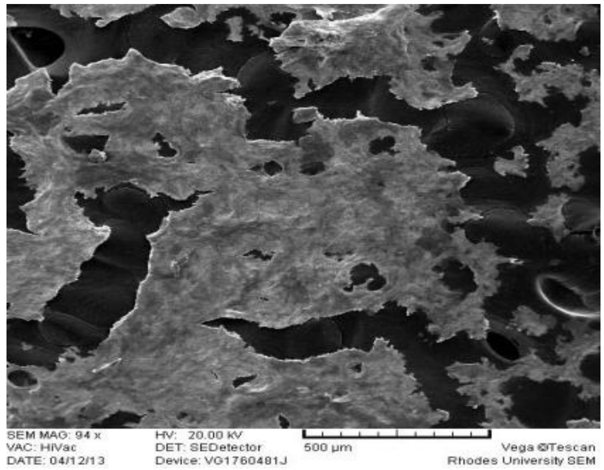

Fig. 4. SEM image of C-Ag-np

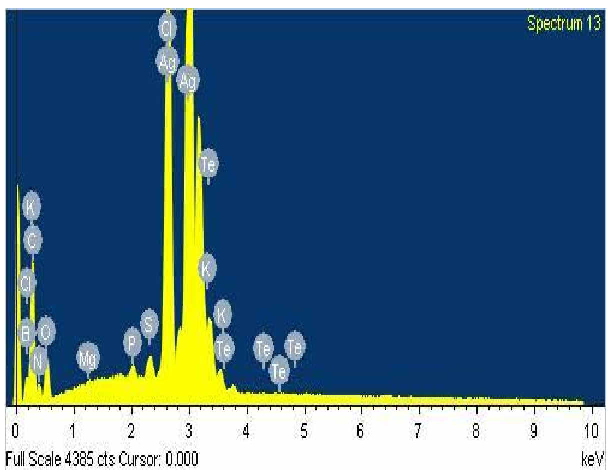

Fig. 5. EDX Spectrum of C-Ag-np

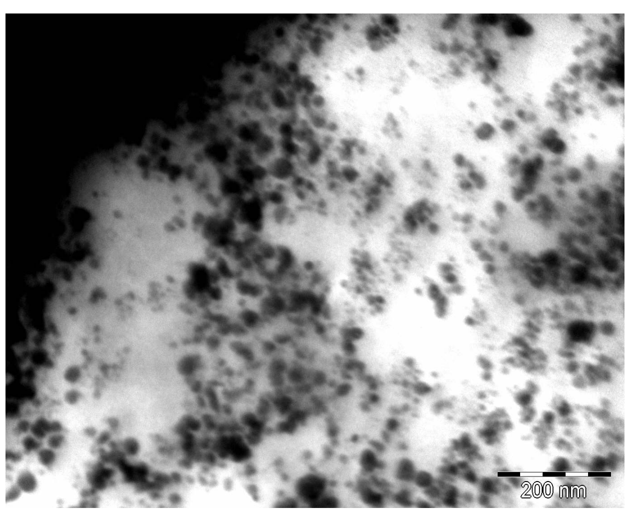

Fig. 6. TEM image of C-Ag-np

4. CONCLUSION

Green synthesis of silver nanoparticles was prepared from green leaves extract of *Calotropis procera.* A combination of analytical techniques: *Ultraviolet-Visible (UV-VIS) Spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Energy Dispersive X-ray (EDX)* justified the formation of C-Ag-np. The characteristic Surface Plasmon Resonance absorption band was recorded at 420 nm. The research has truly shown the feasibility of replacing sodium borohydride with plant extract which serves as both reducing agent and capping agent. These properties are considered as the advantage of the green plants over the common toxic sodium borohydride.

COMPETING INTERESTS

No potential conflict of interest was reported by the authors.

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