PRINT ISSN 1119-8362
Electronic ISSN 1119-8362

Full-text Available Online at
https://www.ajol.info/index.php/jasem
http://ww.bioline.org.br/ja

J. Appl. Sci. Environ. Manage.
Vol. 23 (4) 695-699 April 2019

# Green Synthesis and Characterization of Silver Nanoparticles Using Leaves Extracts of Neem (*Azadirachta indica*) and Bitter Leaf (*Vernonia amygdalina*)

**NZEKEKWU, AK; *ABOSEDE, OO**

*Department of Chemistry, Federal University Otuoke, P.M.B. 126, Yenagoa, Bayelsa State, Nigeria.*
**Corresponding Author Email: abosedeoo@fuotuoke.edu.ng*

**ABSTRACT:** Silver nanoparticles (AgNPs) were synthesized using *Azadirachta indica* and *Vernonia amygdalina* leaves extracts. Current nanotechnology research uses a lot of chemicals which are quite often toxic and flammable. In this research article, a simple and eco-friendly synthetic method for silver nanoparticles preparation was reported using the leaves extracts of *Azadirachta indica* (commonly called neem) and *Vernonia amygdalina* (commonly called bitter leaf) as reducing agents. The synthesis of AgNPs was monitored and confirmed with the use of UV-Visible spectrophotometer, Fourier Transform Infrared (FTIR) spectroscopy and Powder X-ray Diffraction (PXRD). The reduction process of $Ag^+$ to $Ag^0$ was observed by the change of color from yellow to brown for both leaves. The UV-Vis Spectra of AgNPs in aqueous solution showed absorbance peaks around 455 nm for *Azadirachta indica* and 460 nm for *Vernonia amygdalina* due to silver surface plasmon resonance. Crystallinity of the AgNPs was confirmed with PXRD. In addition, FTIR spectra showed that the AgNPs were capped with phytochemicals from the leaves extracts.

**DOI:** https://dx.doi.org/10.4314/jasem.v23i4.19



**Dates:** Received: 09 February 2019; Revised: 26 March 2019; Accepted 18 April 2019

**Keywords**: AgNPs, UV-Vis, FTIR, *Azadirachta indica*, *Vernonia amygdalina*

Nanotechnology is an important field of applied science and technology dealing with design, synthesis and manipulation of structure of particles ranging from approximately 1–100 nm. AgNPs are widely researched for diverse applications because of their fabulous superior characteristic features based on their properties such as size, morphology and other size-dependent properties (Smith *et al.*, 2016). Nanoparticles have drawn the attention of researchers because of their extensive applications in areas such as mechanics, optics, biomedical sciences, chemical industry, electronics, space industries, drug gene delivery, energy science and catalysis (Schmid *et al.*, 1992; Hoffman *et al.*, 1992). Nanoparticles can be synthesized by various methods such as chemical and photochemical reactions in reverse micelles, thermal decomposition, electrochemical, sonochemical, microwave assisted process, and also by biological methods (Iravani *et al.,* 2014). Among these methods, biological processes that are based on bacteria, fungi, bio-derived chemicals and plant extracts are considered as safe and economically viable for the nanomaterial fabrication (Valli and Vaseeharan, 2012). Plant extracts produce best capping material for the stabilization of silver nanoparticles. Researchers have discovered that phytochemicals present in plant extracts are responsible for metal ion reduction and capping of the newly formed particles during their growth processes (Smith *et al.,* 2006; Wei *et al.*, 2005). Alkaloids, flavonoids, terpenoids, ketones, amides, aldehydes, polyphenols and carboxylic acids present in plants are good promoters for bioreduction of metal ions into nanoparticles and also in supporting their subsequent stability. The objective of this work is to synthesize silver nanoparticles by simple, effective and eco-friendly method using leaves extracts of *Azadirachta indica* (commonly known as neem), a member of the Meliaceae family and *Vernonia amygdalina* (commonly known as bitter leaf) a tropical plant belonging to the family Compositae. The objective also includes the characterization of the as-synthesized nanoparticles. Both plants are known to be medicinal with various therapeutic benefits (Abay *et al.,* 2015; Asante *et al.,* 2016; Luo *et al.,* 2011; Sinisi *et al.,* 2016; Subarpriya, 2005). The synthesized silver nanoparticles were characterized by UV–Vis spectroscopy, FTIR and powder X-ray diffraction (PXRD).

## MATERIALS AND METHODS

$AgNO_3$ and 0.2 μm membrane filter paper are from VWR chemicals. Fresh leaf samples of *Vernonia Amygdalina* and *Azadirachta Indica* were purchased from Free Zone market at Aba, Abia State, Nigeria. UV-Visible spectra were taken on JASCO UV-Vis V-730 spectrophotometer with a resolution of 1 nm.

**Corresponding Author Email: abosedeoo@fuotuoke.edu.ng*

FTIR spectra analyses of the as-synthesized nanoparticles were taken on a Shimadzu FTIR spectrometer from 4000 $cm^{-1}$ to 400 $cm^{-1}$ at Redeemer University of Nigeria.

The fresh leaves were first dried under a shade until the moisture content was reduced. The plants were grounded to tiny particles and stored in polythene bags until used.


**Fig 1:** (a) Fresh leaves of *Vernonia Amygdalina* (b) Fresh leaves *Azadirachta Indica*

*Preparation of plant extracts:* The leaf extract was prepared by taking 10 g of the grounded dried leaves into a 500 mL beaker with the addition 100 mL of distilled water and then stirred for about 15 minutes. The mixture was incubated in a cupboard for 30 minutes at 25 ºC and was allowed to settle for another 30 minutes. Clear extract was collected by filtration using Whatman filter paper and then stored in the fridge at 4 ºC for further use. This method was used for both leaves separately.

*Synthesis of Silver Nanoparticles:* Aqueous solution of silver nitrate ($AgNO_3$) at concentration of 0.1 M was prepared and used for the synthesis of AgNPs. For the reduction of $Ag^+$ ions, 1 mL of *A. indica* extract was added into a clean test-tube and then 9 mL of 0.1 M aqueous $AgNO_3$ solution was added into the extract. On addition of aqueous $AgNO_3$ to the extract, colour change was noticed after about 30 minutes from yellow to brown. For the reduction of $Ag^+$ ions using *V. amygdalina*, 1 mL of the extract was added into a test-tube and then 9 mL of 0.1 M aqueous $AgNO_3$ solution was added into the extract. On addition of $AgNO_3$ to the extract, colour change was noticed after about 2 minutes from yellow to brown.

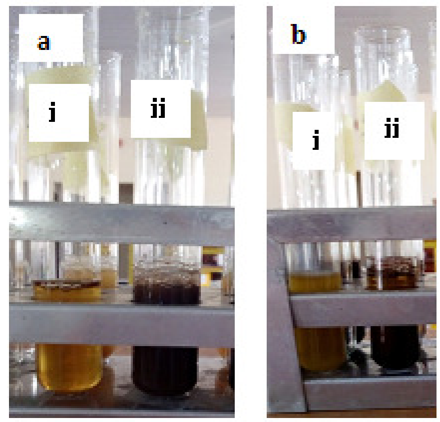


**Fig 2**: (a) Formation of silver nanoparticles by *Azadirachta indica*, (b) *Vernonia amygdalina* (i) plant extract without silver nitrate, (ii) plant extract with silver nitrate solution

Formation of AgNPs at different time intervals were monitored between 200 and 700 nm. Distilled water was used as blank. After the complete formation of the nanoparticles, the solution was then filtered using 0.2 µm membrane filter paper to separate out the nanosized particles from the suspension. The nanoparticles were then allowed to dry so as to be used for further analysis.

## RESULTS AND DISCUSSION

*Characterization of Silver Nanoparticles: UV-Vis Spectroscopy Analysis:* The aqueous extracts of leaves of both neem and bitter leaf were used for the green synthesis of silver nanoparticles. The silver nanoparticles (AgNPs) appear brownish in colour in aqueous medium as a result of surface plasmon vibrations (Arya *et al.*, 2017). As the different leaves extracts were added to aqueous silver nitrate solution, the colour of the extracts changed from yellow to brown for both *Azadirachta indica* and *Vernonia amygdalina* and finally with the formation of brownish precipitates indicating AgNP formation. Similar changes in colour have been observed in previous studies and hence confirmed the completion of reaction between leaf extract and $AgNO_3$ (Arya *et al.*, 2017). This was also confirmed by the UV-Vis spectrum of the precipitated silver nanoparticles which has been recorded as a function of time as shown in Figures 3 and 4.

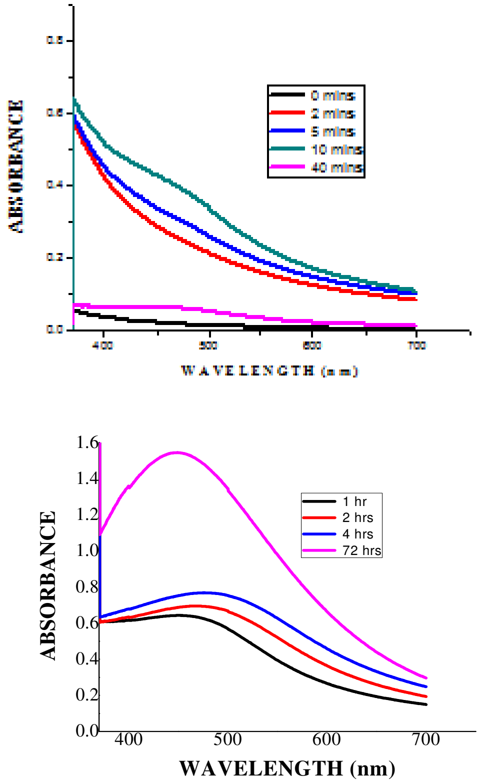


**Fig 3**: UV–Vis spectra recorded as a function of time of reaction of 0.1M aqueous solutions of silver nitrate with neem leaf broth

Generation of AgNPs exhibits unique and tunable optical properties resulting from its surface plasmon resonance (SPR) which is dependent on shape, size, and size distribution of the formed nanoparticles (Arya *et al.*, 2017). Typical SPR of AgNPs appear near 440nm and is due to the collective oscillation or vibration of free electrons in the conduction band after excitation by incident light of particular wavelength. Excitation of surface plasmonic vibrations due to the reduction of $Ag^+$ ions was measured spectrophotometrically at different time intervals. The UV-Vis spectra recorded showed that the bioreduction of silver ions was achieved using neem and bitter leaf extracts as reducing agent. After 72 hours, it was observed that the formation of AgNPs with neem extracts was completed and after 24 hours, the formation of AgNPs synthesized with Bitter leaf extracts was completed.

The sharp bands of silver nanoparticles were observed around 455 nm in case of *Azadirachta indica* (Figure 3), whereas the bands for *Vernonia amygdalina* were observed around 460 nm (Figure 4). From different literatures it was found that silver nanoparticles show SPR peak between 420 and 460 nm. The appearance of SPR at 455 nm and 460 nm and broadening of the peak indicated the formation of polydispersed large nanoparticles due to slow reduction rates (Arya *et al.*, 2017).

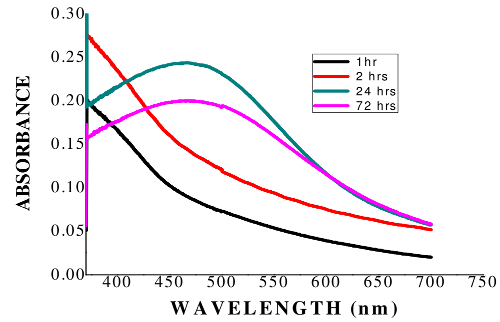


**Fig 4**: UV–Vis spectra recorded as a function of time of reaction of 0.1M aqueous solutions of silver nitrate with bitter leaf broth

*FTIR and PXRD Spectroscopy Analysis*: The FTIR spectra of the AgNPs synthesized with Neem and Bitter leaf extract (Figure 5) respectively showed absorption bands above 3390 $cm^{-1}$ attributed to the presence of N-H stretching vibrations of phytochemicals on the surface of the nanoparticles. A medium band at 2854 $cm^{-1}$ is due to C-H stretching vibration. Bands at 1741 $cm^{-1}$ and 1711 $cm^{-1}$ are characteristic stretching vibration of carbonyl functional group in acids and aldehydes.

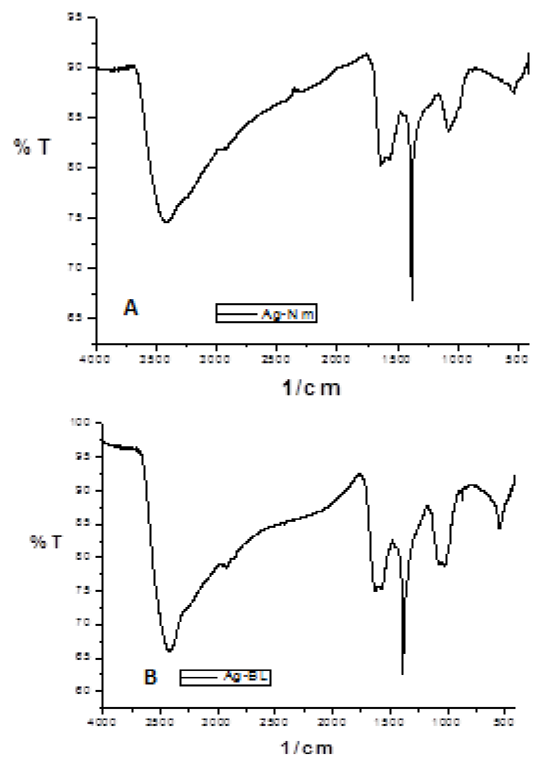


**Fig 5:** FTIR spectrum of Ag nanoparticles formed from (a) Neem and (b) bitter leaf extracts.

The medium bands at *ca* 1500 and 1380 $cm^{-1}$ are attributed to -C=C- stretching mode and C-N stretching vibrations of aromatic amines respectively. bands at 1050 and 850-900 $cm^{-1}$ are due to C-OH stretching of alcohols and C-O-C vibrations respectively. Therefore, FTIR analysis of the synthesized AgNPs using both neem and bitter leaves extracts respectively showed that these functional groups from the aqueous extracts of neem and bitter leaf were responsible for the reduction $Ag^+$ to $Ag^0$, the AgNPs synthesis and stabilization, and are present on the surface of the synthesized nanoparticles. The X-ray diffraction of the nanoparticles (Figure 6) reveal that the nanoparticles are crystalline and also confirms that they are capped by biomolecular compounds which were responsible for the reduction of silver ions.

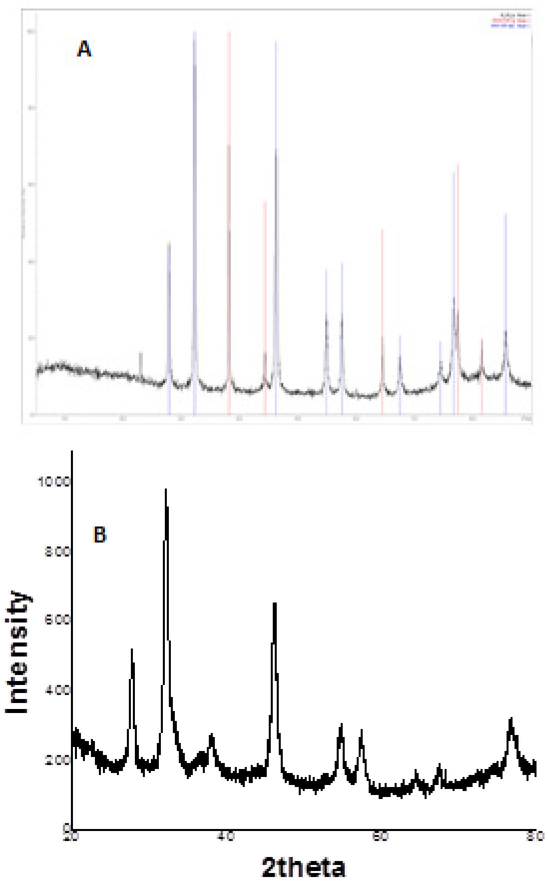


**Fig 6:** Powder XRD of Ag nanoparticles formed from (a) neem and (b) bitter leaf extracts

*Conclusion*: Silver nanoparticles have been synthesized using leaves extracts of neem and bitter leaf showing absorption peaks at 455 nm and 460 nm respectively. FT-IR spectra also showed that the silver nanoparticles were capped by biomolecular compounds which were responsible for the reduction of silver ions. From the technological point of view, these obtained silver nanoparticles have potential applications in the biomedical field and this simple procedure has several advantages such as cost-effectiveness, compatibility for medical and pharmaceutical applications.

*Acknowledgements*: Many thanks to Prof. Thomas Doert and Alex Zeugner at Technical University Dresden, Germany for measuring the X-ray diffraction pattern of the nanoparticles. JASCO UV-Visible V-730 spectrophotometer was a donation from the International Foundation for Science (IFS), Stockholm, Sweden and the Organization for the Prohibition of Chemical Weapons (OPCW) through a grant to Olufunso O. Abosede (IFS 5780-1).

## REFERENCES

Abay, SM; Lucantoni, L; Dahiya, N; Dori, G; Dembo, EG; Lupidi G (2015). Plasmodium transmission blocking activities of *Vernonia amygdalina* extracts and isolated compounds. *Malar. J.,* 14, 288

Arya, G; Sharma, N; Ahmed, J, Gupta, N; Kumar, A; Chandra, R; Nimesha S (2017). Degradation of anthropogenic pollutant and organic dyes by biosynthesized silver na.no-catalyst from *Cicer arietinum* leaves. *Journal of Photochemistry & Photobiology, B: Biology* 174, 90–96

Asante, D; Effah-Yeboah, E; Barnes, P; Abban HA, Ameyaw, EO; Boampong, JN; Ofori, EG; Dadzie, JB (2016). Antidiabetic effect of young and old ethanolic leaf extracts of *Vernonia amygdalina*: a comparative study. *J. Diabetes Res.* Doi: 10.1155/2017/5618548

Hoffman, AJ; Mills, G; Yee, H; Hoffmann, MR (1992). Q-sized CdS: Synthesis, Characterization and Efficiency of Photo-initiation of Polymerization of Several Vinylic Monomers. *Journal of Physical Chemistry*. 96(13), 5546–5552

Iravani, S; Korbekandi, H; Mirmohammadi, S; Zolfaghari, B (2014). Synthesis of silver nanoparticles: chemical, physical and biological methods. *Res. Pharm. Sci.* 9, 385

Luo, X; Jiang, Y; Fronczek, FR; Lin, C; Izevbigie, E; Lee KS (2011). Isolation and structure determination of a sesquiterpene lactone (vernodalinol) from *Vernonia amygdalina* extracts. *Pharmaceutical Biology* 49(5), 464-470

Schmid, G (1992). Large Clusters and Colloids. Metals in the Embryonic State. *Chemical Reviews* 92(8), 1709–1727

Sinisi, A; Munoz, E; Abay, SM; Habluetzel, A; Appendino, G; Millan E (2016). Poly-electrophilic sesquiterpene lactones from *Vernonia amygdalina*: new members and differences in their mechanism of thiol trapping and in bioactivity. *J. Nat. Prod.* 78(7), 1618-1623

Smith, AM; Duan, H; Rhyner, MN; Ruan, G; Nie, SA (2006). Systematic Examination of Surface Coatings on the Optical and Chemical Properties of Semiconductor Quantum Dots. *Physical Chemistry Chemical Physics*. 8(33), 3895–3903

Subarpriya, R; Bhuvaneswari, V; Ramesh, V; Nagini, S (2005). Ethanolic leaf extract of neem (*Azadirachta indica)* inhibits buccal pouch carcinogenesis in hamsters. *Cell Biochem Funct.* 23(4), 229-238

Valli, JS; Vaseeharan, B (2012). Biosynthesis of silver nanoparticles by *Cissus quadrangularis* extracts, *Mater. Lett.* 82,171-173

Wei, GH; Zhou, Z; Liu, Z (2005). A Simple Method for the Preparation of Ultrahigh Sensitivity Surface Enhanced Raman Scattering (SERS) Active Substrate. *Applied Surface Science.* 240, 260-267.