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Chemical Characterization of Synthesized Nanoparticles from Noni (*Morinda citrifolia*) Seed

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Abstract

Morinda citrifolia is an herbal medicine that is used for disease treatment as recommended in traditional medicine. The present study aimed to characterize silver nanoparticles from Noni seed. In this experimental study, green synthesis was carried out then the synthesized nanoparticles were characterized using SEM, EDX, TEM and FTIR. Findings showed that SEM images of the AgNPs of different shapes were obtained in the case of different seed extracts being used as reducing and capping agents. Seed extract formed approximately spherical, triangular, and cuboidal AgNPs, respectively. Elemental mapping of AgNPs by SEM-EDX shows the presence of 0.31% Ag and 40% oxides with 39% Carbon and other elements in trace amounts. TEM image demonstrates that the AgNPs were spherical. The image shows agglomerates of small grains and some dispersed nanoparticles, confirming the results obtained by SEM. FTIR result reveals the assignment of functional groups to 19 FTIR bands (690-3833 cm^{-1}). Bands were categorized based on possible functional group classes (alkene, aromatic, alcohol/phenol/ether, amine, etc.). Specific functional groups like alkenes, conjugated alkenes, ketones, aldehydes, nitriles, alkynes, and carboxylic acids were identified based on characteristic wavenumbers. The study presents a green synthesis approach to prepare silver nanoparticles using Noni seed extract. Reduction of silver nitrate with Noni seed extract is a simple, conducted at room temperature, efficient, and clean method to synthesize silver nanostructures.

Keywords: Nanoparticles, Characterization, Noni Seed, Medicinal Plants.

INTRODUCTION

The development of nanoparticles in recent years had evolved various methods to synthesize nanoparticles of various sizes and shapes of the metal. Metal nanoparticles possess unique chemical and physical properties from their bulk materials due to their high surface area to volume ratio (Murugan, and Natarajan, 2018). In recent medical applications, metal nanoparticle use has become standard. It has been observed that the use of silver nanoparticles is extensively used for medical purposes due to their better chemical stability and good antimicrobial activity (Ruddaraju *et al.*, 2019). Research interest in nanotechnology has increased significantly due to the exponential growth in nanomaterial production and marketing. Metallic nanoparticles can be shaped in different ways, spheres or rods. According to size, Nanomaterials are divided into different groups such as nanoparticles, dendrimers, nanotubes, and nanofilms. This further increases the variety of nanoscale materials (Han *et al.*, 2020). The silver nanoparticles were well known for their antibacterial, antioxidant property, and all other pharmacological potentials, which makes used for surgical prostheses and dental implants. In general, there are two approaches employed in the synthesis of nanoparticles, the first is the top-down approach (Physical methods), and the second is the bottom-down (chemical and biological methods) (Nakamura *et al.*, 2019). The physical method employs machining and etching to produce nanoscale structures whereas, other method employs chemicals such as sodium borohydride, ethanol, ethylene glycol, etc. as reducing agents (Ahmad, 2010). The chemical method is commonly used for producing silver particles. This is because of the simple equipment needed and the convenience to operate.

The use of herbal preparations as a form of therapy dates back many years ago. Their effectiveness in the general management of wellbeing has been attributed to the various phytochemical constituents commonly referred to as secondary metabolites. These compounds as used in phytomedicine can be considered to be the prime foundation of contemporary allopathic medicines today. The use and dependence on plants as medicines by man has been in existence since time immemorial and man continues to search for plants as a drug for a particular disease within his reach. Herbal medicines are safe than synthetic medicines because the phytochemicals in the plant extract target the biochemical pathway (Zaidan *et al.*, 2005). Plants are a good source of various functionally active secondary metabolites and a good source of essential nutrients (Khan *et al.* 2011). Screening active compounds from plants has led to the invention of new medicinal drugs which have efficient protection and treatment roles against various diseases including cancer and Alzheimers disease (Sheeja *et al*., 2007)

Presently, there has been an increasing interest in the study of traditional plants and their medicinal value in different parts of the world. The medicinal properties of plants have been investigated in the light of recent scientific development throughout the world, due to their strong pharmacological activities, economic viability, and low toxicity (Prashant *et al*., 2010). This tremendous interest in plant-derived drugs is mainly due to the current widespread belief that herbal medicine is safer and more reliable than the costly orthodox medicine, many of which may have adverse side effects (Jigna *et al*., 2006). Nanoparticles are being employed in drug delivery systems because of their unique property. There are large numbers of physical, chemical, biological, and hybrid methods available to synthesize metal nanoparticles (Jyoti *et al*., 2016). Generally, physical and chemical methods are non-eco-friendly, toxic, and low in yield. The nanoparticles synthesized from chemical methods are medically non-applicable because of contamination from precursor chemicals (Jyoti *et al*., 2016). Following this, a lack of standardization has placed a limitation on the general acceptance of herbal medicines. However, it is impossible to assay for a specific chemical entity when the bioactive ingredient is not known. In practice, assay procedures are not carried out even for those medicinal plant materials where there are known active ingredients (Yadav and Dixit, 2008). Hence, this problem has led to the present study. Although, natural compounds of plants have been utilized in the conventional system of medication to cure different infections and disorders. There are many plants whose total medicinal value is even now unknown and *Morinda citrofolia* is one among them. Screening active compounds from plants has to lead to the invention of new medicinal drugs which have efficient protection and treatment roles against various diseases (Anarado *et al*., 2021). A majority of the rich diversity of Nigerian medicinal plants is yet to be scientifically evaluated leading to its standardization and *Morinda citrofolia* is not left out. The study aimed at carrying out Chemical characterization of synthesized Nanoparticles from *Morinda citrofolia* seed.

MATERIALS AND METHOD

The present study entitled "Green synthesis of silver nanoparticles by Noni seed extract, their characterization" was performed in the Department of Pharmaceutical Technology "Federal Polytechnic Nekede Owerri". The details of materials used, methodologies employed, experiments, and techniques have been elaborated below. Silver nitrate of analytical grade was purchased from Sigma-Aldrich Chemicals and was used without any further purification. All the solutions and chemicals were prepared following standard procedures.

Sample Collection

Fresh seed of *Morinda citrofolia* was collected from Uli in Anambra State of Nigeria and was identified and authenticated by a Taxonomist from the University of Michael Okpara University of Agriculture Umudike.

Sample Preparation

The seeds were air-dried for fourteen days and then ground into powder form to increase its surface area.

Extraction Method

During the extraction process, 500g of *Morinda citrofolia* seed powder was measured and soaked in 1000 ml of methanol for 72hrs. The mixture was filtered. A distillation process was applied to separate the solvent from the extract by evaporating to dryness. The stock solution of the extract was weighed and stored under refrigeration for further study.

Synthesis of Silver Nanoparticles (Ag-np)

The green synthesis of Ag-np was prepared following the method reported in the literature (Khan *et al*., 2018). Preparation was done by reacting 10 mL of the *Morinda citrofolia* seed extract with 90 mL AgNO3 solution (1 mM) and was agitated on the air bath magnetic stirrer for 15 minutes at room temperature. A color change was observed from colourless to pink. The mixture was centrifuged and dried in the oven at a temperature between 50°C – 60°C overnight. The supernatant was used for Ag NP synthesis and stored in a refrigerator until further use.

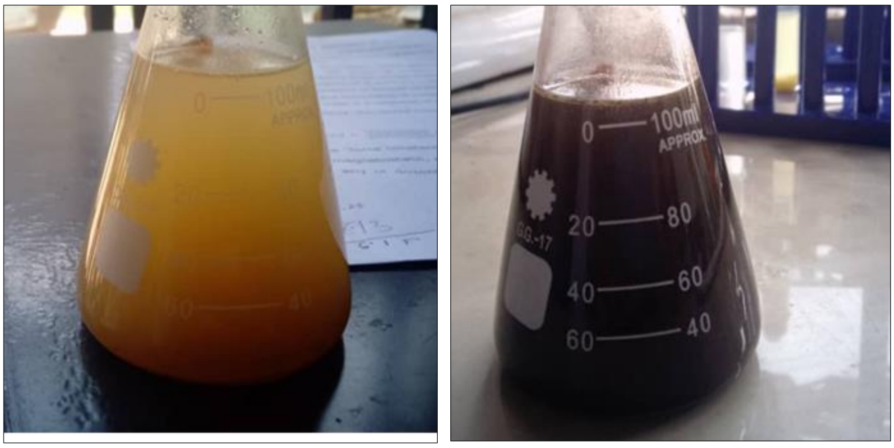

Figure 1: Steps of silver nanoparticle synthesis (a) AgNO3 and Noni seed extract mixture before reaction (b) reaction mixture after 1 hour

Characterizations

Characterization of materials was performed using Scanning Electron Microscopy (SEM), electron dispersive x-ray spectroscopy (EDX), Transmission Electron Microscopy (TEM) and and FTIR.

SEM Analysis: SEM analysis was done employing the method of Banerjee *et al*., (2014)

EDS Analysis

EDS analysis conducted using an EDAX-EDS spectrometer attached to the SEM. Scans were obtained using an accelerating voltage of 20.71keV with a 120 second count time per sample.

TEM Analysis: TEM analysis was done employing the method of Banerjee *et al*., (2014).

FTIR Analysis

Buck scientific M530 USA FTIR was used for the analysis. This instrument was equipped with a detector of deuterated triglycine sulphate and beam splitter of potassium bromide. The software of the Gram A1 was used to obtain the spectra and to manipulate them. An approximately of 1.0g of samples, 0.5ml of nujol was added, they were mixed properly and placed on a the salt pellet. During measurement, FTIR spectra was obtained at frequency regions of 4,000 – 600 cm-1 and co-added at 32 scans and at 4 cm-1 resolution. FTIR spectra were displayed as transmitter values (Odo *et al*., 2017).

RESULTS AND DISCUSSION

In the Present study a cost-effective and eco-friendly technique for the green synthesis of AgNPs has been developed; from the reduction of AgNO3 using Noni seed extract as reducing as well as capping agent. The extracts of Noni seed were used for both the reducing and stabilizing agent for the synthesis. The extract is considered ecofriendly over organic extracts and found more appropriate for the green synthesis of AgNPs. The synthesis was performed at room temperature as well as at high temperatures. Further AgNP studied by using different instrumental techniques which included EDX, SEM, TEM and FTIR techniques. Biologically inspired steps for the synthesis of nanoparticles have been evolving as an important branch of nanotechnology. The biosynthesis of nanoparticles via green route does not employ toxic chemicals and hence proving to become an environment-friendly processes. Several functional groups present in Noni seed extract such as flavonoids, citric acid, carotenoids, and aromatic compounds exhibit antioxidant properties, and consequently, can play an important role as reducing and/or capping agents in nanoparticle synthesis.

SEM and EDX Result of Silver nanoparticle from Noni seed Extract

SEM microscopy shows that the AgNPs are approximately 100 nm in size with a predominant spherical shape. In these images the particles are well dispersed, and the aggregation of the particles can be seen, as shown in Figure 2, 3 and 4. The EDX on the SEM analysis of the Silver Nanoparticles was performed for elemental study and are shown in Figures 5. EDX elemental analysis show the same elemental composition for all the samples.

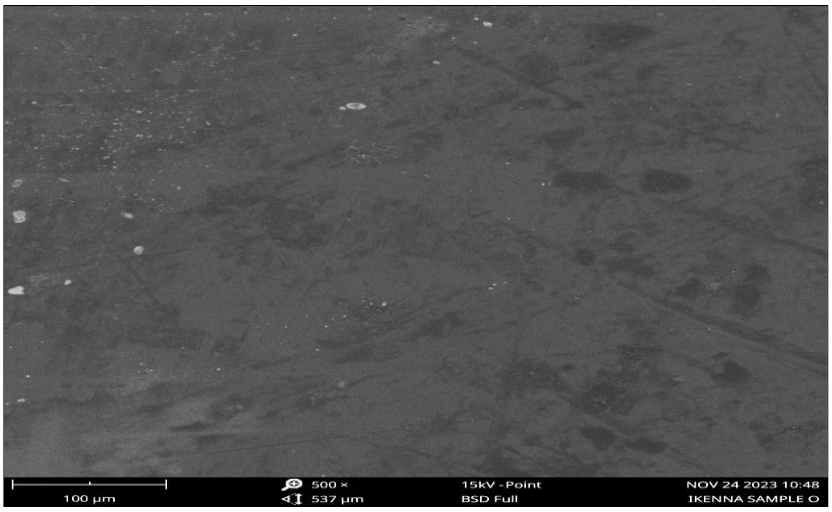

Figure 2: SEM micrograph of Silver nanoparticles synthesized from the Noni seed extract at Magnification of 500 x 537µm

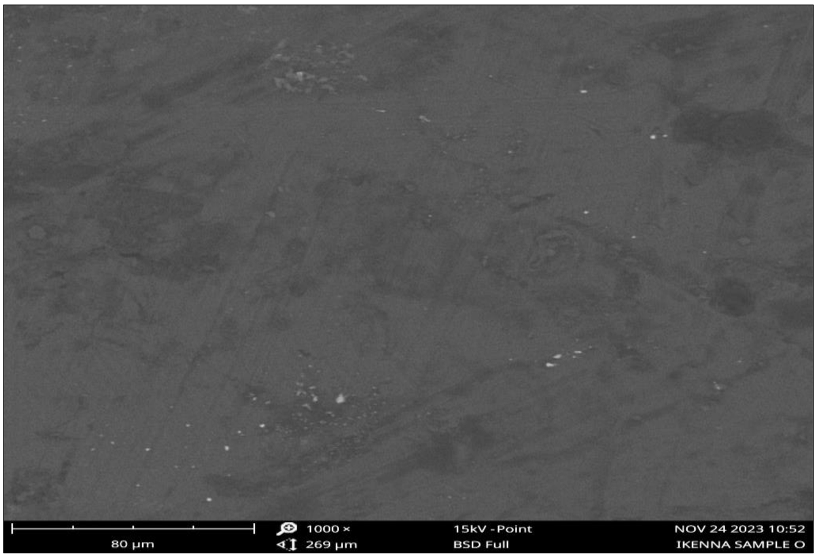

Figure 3: SEM micrograph of Silver nanoparticles synthesized from the Noni seed extract at Magnification of 1000 x 269µm

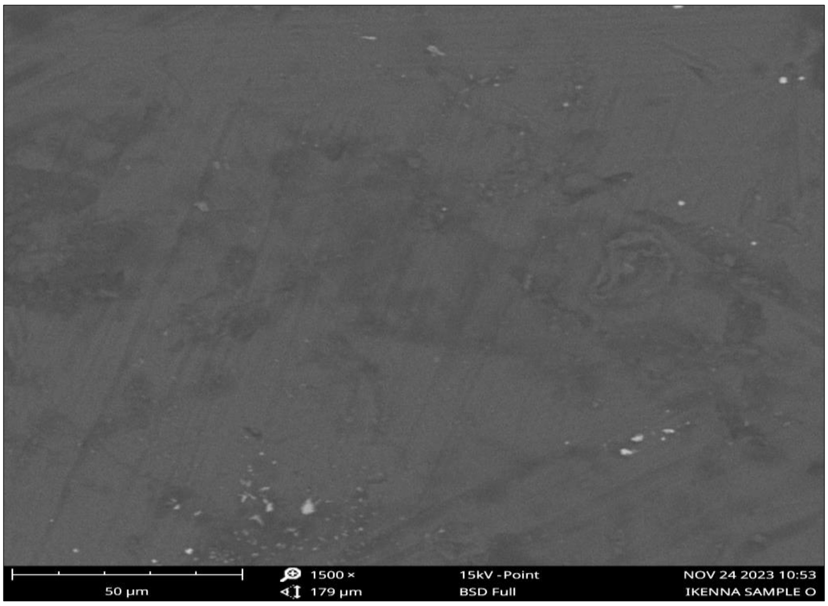

Figure 4: SEM micrograph of Silver nanoparticles synthesized from the Noni seed extract at Magnification of 1500 x 179µm

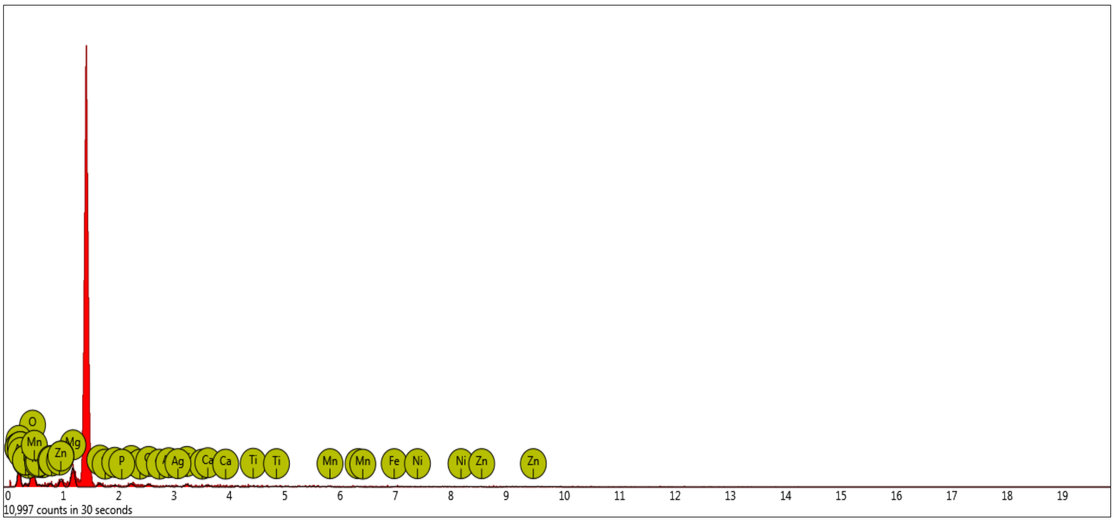

Figure 5: Energy dispersive spectrometer analysis of Silver nanoparticles synthesized from the Noni seed Extract

Table 1: EDAX analysis of Noni seed-AgNPs

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	39.99	40.01
6	C	Carbon	40.00	30.04
7	N	Nitrogen	8.81	7.71
12	Mg	Magnesium	4.33	6.58
11	Na	Sodium	2.34	3.36
47	Ag	Silver	0.31	2.06
19	K	Potassium	0.58	1.41
26	Fe	Iron	0.40	1.38
16	S	Sulfur	0.68	1.36
17	Cl	Chlorine	0.55	1.22
14	Si	Silicon	0.64	1.13
25	Mn	Manganese	0.31	1.05
20	Ca	Calcium	0.41	1.03
22	Ti	Titanium	0.34	1.02
15	P	Phosphorus	0.32	0.63

TEM Result of Silver nanoparticle from Noni seed Extract

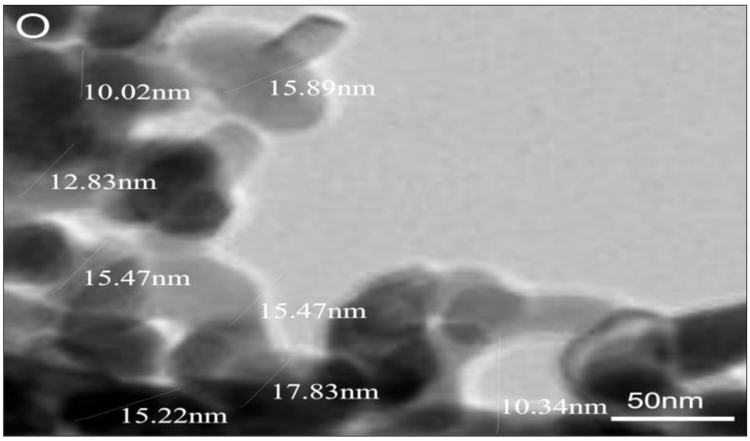

Figure 6: TEM images of silver nanoparticles. Formed by the reaction of 1 mM silver nitrate and 10% seed extract of Noni

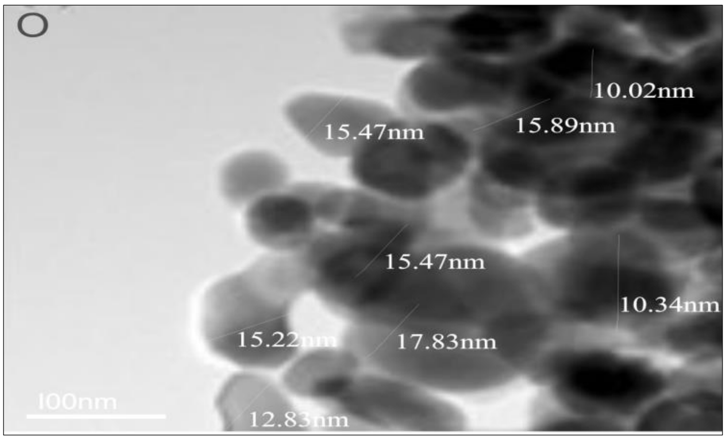

Figure 7: TEM images of silver nanoparticles. Formed by the reaction of 1 mM silver nitrate and 10% seed extract of Noni

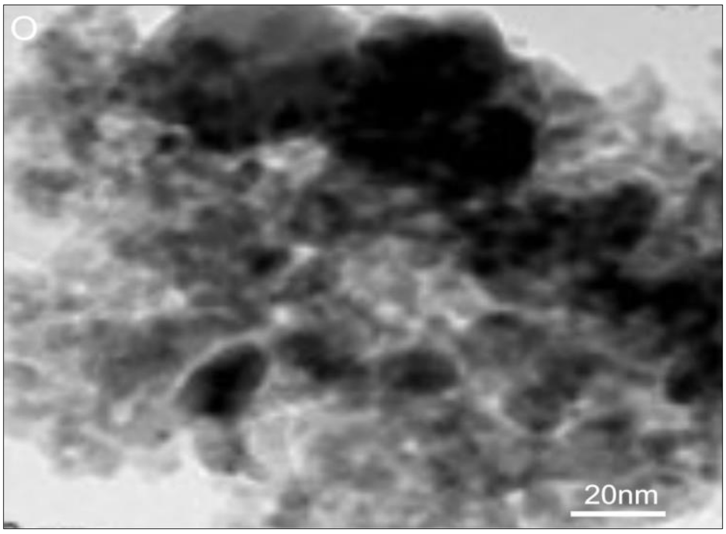

Figure 8: TEM images of silver nanoparticles. Formed by the reaction of 1 mM silver nitrate and 10% seed extract of Noni

FTIR Result of Silver nanoparticles from Noni seed Extract

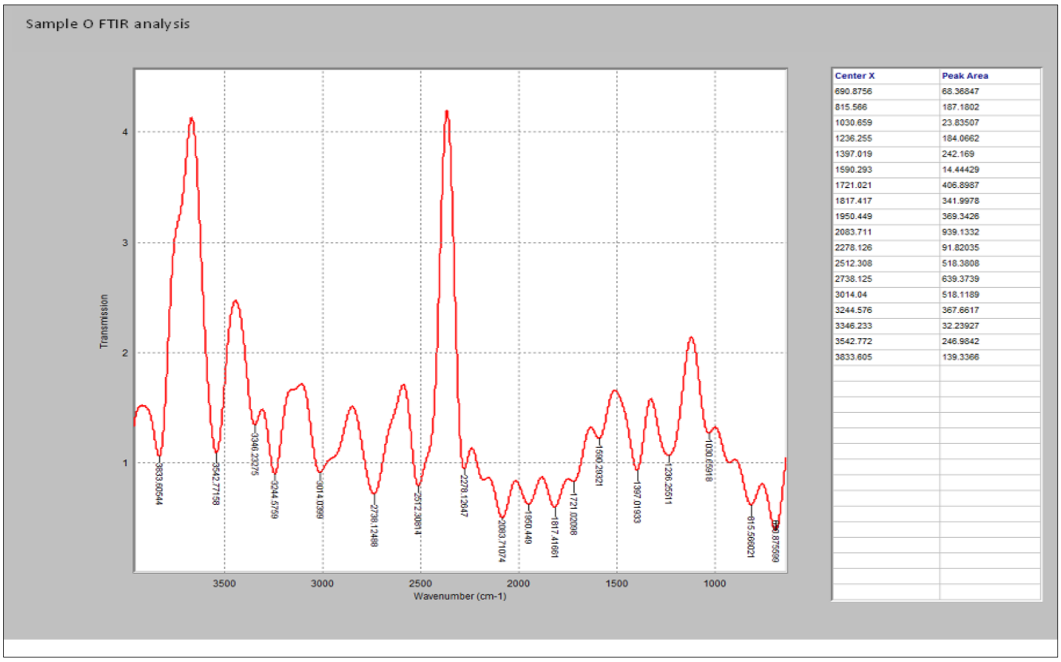

Figure 9: FTIR chromatogram of silver nanoparticles from Noni seed-AgNPs

Table 2: FTIR Band assignment of silver nanoparticles from Noni seed-AgNPs

Wavenumber	Functional Group Class	Possible Functional Groups
690	Alkene C=C stretch	Alkenes, conjugated alkenes
815	Aromatic C-H bend	Benzene, substituted benzenes
1030	C-O stretch (alcohol, phenol, ether)	Alcohols, phenols, ethers
1236	C-N stretch (amine)	Primary amines, secondary amines
1397	C-H stretch (methylene)	Alkanes, cycloalkanes
1590	C=C stretch (conjugated alkene)	Dienes, trienes
1721	C=O stretch (ketone, aldehyde)	Ketones, aldehydes
1817	C=O stretch (anhydride)	Anhydrides
1950	C≡N stretch	Nitriles
2208	C≡C stretch	Alkynes
2278	Ar-H bend	Aromatics
2512	O-H stretch (broad)	Carboxylic acids, alcohols, phenols
2738	N-H stretch (primary amine)	Primary amines
3014	C-H stretch (aromatic)	Aromatics
3244	O-H stretch (alcohol, phenol)	Alcohols, phenols
3346	N-H stretch (secondary amine)	Secondary amines
3542	O-H stretch (water)	Water
3833	O-H stretch (free carboxylic acid)	Carboxylic acids

DISCUSSION

A possible mechanism of AgNPs synthesis was to proceed via ionic interactions of functional groups and a silver salt. Several biomolecules present in the plant extract reduces the monovalent silver ion to uncharged atoms and these atoms aggregate to reach nano-size, other biomolecules of the plant extract act to the envelope to prevent their further aggregation (Ganaie *et al.*, 2014). The rich source of negatively charged functional groups may be responsible for the reduction of metal ions and efficient stabilization of synthesized NPs under natural conditions (Mittal *et al.*, 2013). In Noni seed tannins and flavonoids are main compounds responsible for bio reduction. These are water-soluble polyphenolic compounds and cause proteins coagulation and are responsible for the chelation of metal ions. Oxidation of phenols results in quinones or quinoid structure in case of tannins. The higher total phenolic content in seed extract facilitates the reduction of Ag+ ions to nanoscale size Ag0 particles due to electron donating ability of these phenolic compounds. Moreover, the quinoid compound produced due to the oxidation of phenol group in phenolics can be adsorbed on the surface of nanoparticles, accounting for their suspension and stabilization. Possibility of large and dispersed AgNP is obtained due to a slower rate of reaction between plant extract and AgNO3.

The SEM images of the AgNPs are shown in Figure 3-5. It is seen that AgNPs of different shapes were obtained in case of different seed extract being used as reducing and capping agents. Seed extract formed approximately spherical, triangular, and cuboidal AgNPs, respectively. This may be due to availability of different quantity and nature of capping agents present in the seed extracts. This is also supported by the shifts and difference in areas of the peaks obtained in the FTIR analysis. Elemental mapping of AgNPs by SEM-EDX shows the presence of 0.31% of Ag and 40% of oxides with 39% Carbon and other elements in trace amounts as shown in Fig. 5. Elemental analysis of AgNPs was confirmed by EDX as shown in Table 1. A strong signal of the peak that was observed at 3 KeV may reflect the absorption of another metallic nanoparticles. The presence of other elements confirms the lack of purity of prepared nanoparticles which maybe due to improper centrifugation. Reason is that centrifugation is a technique employed for purification. Also, this could be attributed to other elements present in the plant extract competing with silver ions for the available reducing agents, leading to the formation of non-silver nanoparticles or incomplete reduction of silver ions and sensitivity to light, and exposure to light during the reaction can affect the stability of the reducing agents or interfere with the reduction process, leading to lower silver quantities.

Further, an insight into the morphology and size details of AgNPs was provided by transmission electron microscopy. TEM image as shown in Fig. 6-8 clearly demonstrates that the AgNPs were spherical in shape. The image shows agglomerates of small grains and some dispersed nanoparticles, confirming the results obtained by SEM. The synthesized AgNPs in the TEM result was in the range of 10–20 nm. This size difference can be due to various factors such as temperature, pH, seed concentration or the reducing agent used.

FT-IR spectroscopy is used for characterizing chemical compounds involved in the formation of Noni seed-AgNPs. For the analysis, Noni seed-AgNPs were used in powdered form. This study reveals the assignment of functional groups to 19 FTIR bands (690-3833 cm^-1). Bands were categorized based on possible functional group classes (alkene, aromatic, alcohol/phenol/ether, amine, etc.). Specific functional groups like alkenes, conjugated alkenes, ketones, aldehydes, nitriles, alkynes, and carboxylic acids were identified based on characteristic wavenumbers. Tips for further assigning groups include spectral library comparison, characteristic group frequencies, band intensity, and considering the molecule's structure. This analysis facilitates functional group identification in unknown molecules using FTIR spectroscopy.

CONCLUSION

In this work, we present a green synthesis approach to prepare silver nanoparticles using Noni seed extract. Reduction of silver nitrate with Noni seed extract is a simple, conducted at room temperature, efficient, and a clean method to synthesize silver nanostructures. After extensive literature survey protocol has been optimized for rapid and high yield of AgNPs. Proteins, flavonoids and phenols play and important role in the green synthesis of AgNPs as well as the stabilization of the NPs. For biological applications such as medicine nanoparticles should be free from toxic compounds and have good adsorbitivity by biomolecules. So due to its huge potential in medicines these green or biolgically friendly synthesis are important. As well characterization plays a key factor for the potential applications. This simple, low cost, and green method for synthesizing silver nanoparticles may be valuable in future biological studies and may be extended into catalytic applications.

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