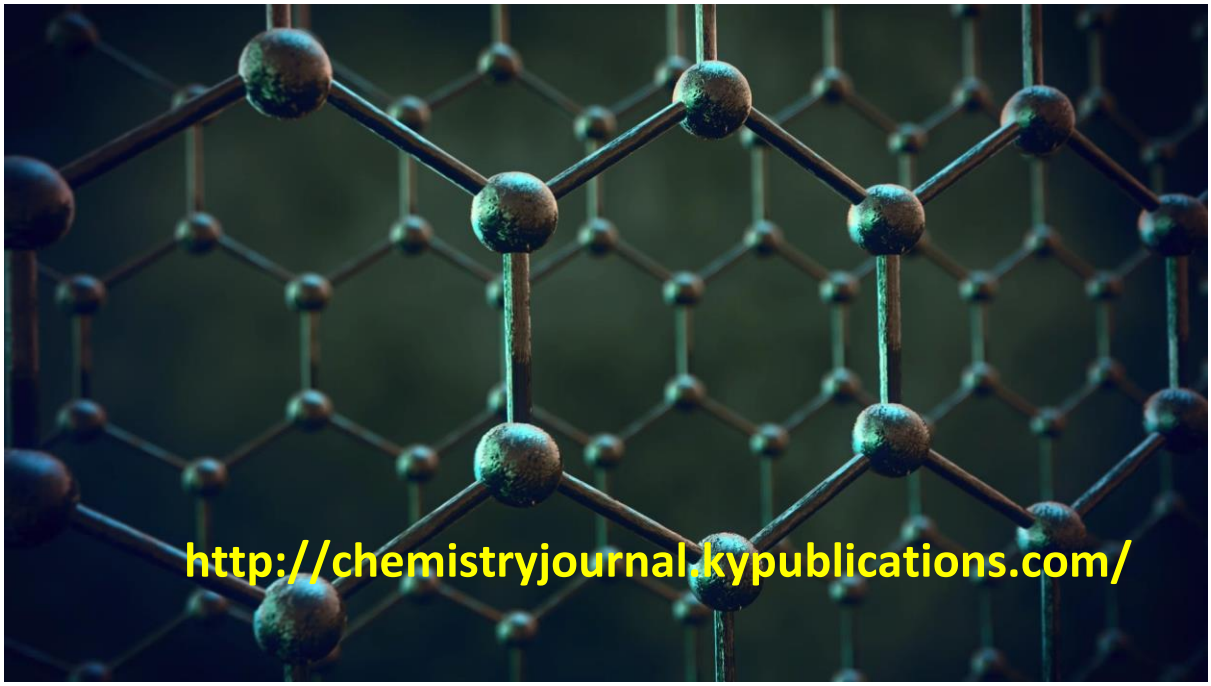

International Journal of Chemistry and Aquatic Sciences

(IJCA)

http://www.chemistryjournal.kypublications.com/

ISSN:2455-040X

www.kypublications.com

Editor-in-Chief

Dr.Y.H.Rao

Email:submitijca@gmail.com

©KY PUBLICATIONS
International Journal of Chemistry and Aquatic Sciences (IJCA)
Volume: 3, Issue 2, 2017 (March-April); Page 1-8
ISSN:2455-040X
Research Article

CHARACTERIZATION OF GREEN SILVER NANOPARTICLES SYNTHESISED FROM *ALBIZIA LEBBECK* (L.) BENTH ROOT BARK

KONGARA SHOBHA RANI
Lecturer in Chemistry, VSR & NVR College, Tenali, Guntur Dist. AP, India

ABSTRACT
This study aimed to synthesise silver nanoparticles with *Albizia lebbeck* root bark and prepared materials were characterised by using UV-visible absorption spectroscopy, scanning electron microscopy (SEM), EDX, TEM, X-ray diffraction (XRD) etc., techniques. Synthesis of silver nanoparticles was confirmed by the presence of an absorbance peak at 430-450 nm in UV-visible spectrum. Transmission electron microscopy (TEM) was used to characterize the morphology of the nanoparticles and confirm the size of nanoparticle with an average of 22nm obtained from plant extracts. Energy dispersive X-ray (EDX) spectrometer established the existence of elemental sign of the silver and other elements. Diffraction by using X ray (XRD) analysis for the formed AgNPs revealed spherical and cubical shapes structure with different planes ranged between 111 to 311 planes. Scanning electron microscopy (SEM) was used to characterize the morphology of the nanoparticles obtained from plant extracts. *Albizia lebbeck* root bark is a reducing agent suitable and compatible with environment for produce nanoparticles.
Key Words: *Albizia lebbeck* root bark, Silver nanoparticle synthesis, Characterization.

1.0 Introduction

Nanotechnology is the fastest growing area of manufacturing in the world today and there is an increasingly frantic search for new nanomaterials and methods to make them. It has been well known that living cells are the best examples of machines that operate at the nanolevel and perform a number of jobs ranging from generation of energy to extraction of targeted materials at very high efficiency.

Current nanotechnology developments have led to nanomedicine, a new field which includes many diagnostic and therapeutic applications involving nanomaterials and nanodevices [1]. Synthesis of nanoparticles using plant extracts supplies progression more than chemical and physical methods as it is cost effective, environmentally safe, simply scaled up for great range production and in this process there is no requirement to use high pressure, power, temperature and poisonous chemicals [2].

Silver has long been recognized as having inhibitory effect on microbes present in medical and industrial process [4]. The most important application of silver and silver nanoparticles is in medical industry such as topical ointments to prevent infection against burn and open wounds [5]. Further these biologically synthesized nanoparticles were found highly toxic against different multi-drug resistant human pathogens.

Nano chemistry has enhanced the production of minor AgNPs with little toxic effect to human and more effectiveness alongside bacteria. Furthermore, nanoparticles are alternatives to

antibiotics allowing better action against multidrug opposing bacteria and consequently plant-derived nanoparticles have been proved better than other methods [3].

Presently, the researchers are looking into the development of cost-effective procedures for producing reproducible, stable and biocompatible metal NPs. Aluminum, Gold, Zinc, Carbon, Titanium, Palladium, Iron and Copper have been routinely used for the synthesis of NPs. Metal nanoparticles are of use in various catalytic applications, electronics, biology and biomedical applications, material science, physics, environmental remediation fields

Albizia lebbeck (L) Benth (Family-Mimosoideae) (figure 1) is an important anti-poisoning herb of Ayurveda. Its use is even indicated in snake bite poisoning. It grows into a big tree, usually found in road sides of Southern India. According to Ayurveda Bark, seeds, leaves and flowers of *Albizia lebbeck* are used for medicinal purposes [6].

A. lebbeck is a member of this genus and used in folk medicine to treat inflammatory conditions as asthma, arthritis, burns allergic rhinitis, bronchitis and leprosy [7] and it have been claimed to be useful in treatment of Alzheimer`s and Parkinson`s diseases [8]. Moreover the extracts of *A. lebbeck* exhibited versatile biological effects as antioxidants [9], hepatoprotective, cardiotonic, lipid-lowering, hypoglycemic activities [10,11] antihistaminic [7] and antimicrobial [11]. Literature survey on *A. lebbeck* revealed the presence of sterols and triterpenes [12], phenolic compounds, flavonoids [13], isoflavone [14], alkaloids [12], miscellaneous compounds [15] and saponins [16]. But there is no report about *A. lebbeck* plant root bark powder growing in south India [17-20], this prompted us to investigate this plant. The present work deals with the synthesis of silver nanoparticles using n-hexane/ethyl acetate extract for the first time and characterise them with different techniques with the aim of development of Nano medicine.

2. Materials and Method

All chemicals and reagents had analytical grade. Silver nitrate, n-hexane with high purity purchased from Sd Fine/Merck India Chemicals, India.

2.1 Apparatus and Instruments: The conventional Soxhlet extraction apparatus was used, which consists of a condenser, a Soxhlet chamber, and an extraction flask. The extractor thimble was permeable one with 44 mm internal diameter and 200 mm external length. The rotary evaporator was used for evaporation of solvent of extracted material.

2.2 Sampling and extraction

***Plant Material*:** Fresh roots of *A. lebbeck* root bark in bulk collected in the month of May 2012 from agricultural fields of local area of Tenali revenue subdivision, Andhra Pradesh. 30x10 cm roots were collected and separated root bark manually, cut in to small pieces (figure 1), washed and dried in sunlight for one month completely to eliminate surface moisture. Then roots packed into envelop and kept in oven at 55^{o}C temperature for further dryness. Dried material was grinded separately in a mortar obtained fine powder and sieved; which was then kept in plastic bags for further use.

Preparation of plant extract: The dry root bark powder material of *A. lebbeck* passed through sieve (100□). The coarse powdered drug (200grams) was extracted in Soxhlet apparatus for 48 h with n-hexane and ethyl acetate (60:40) combination extract obtained was concentrated under reduced pressure in rotatory evaporator below 60°C temperature to get semisolid sticky brown residue (10.5 gm). Then the filtered extract was stored in refrigerator at 4°C for further use in synthesis of silver nanoparticles.

2.3 Synthesis of AgNPs (SNPs): The synthesis of silver nanoparticles was done by mixing *A. lebbeck* root bark extract and 1 mM of aqueous silver nitrate solution ($AgNO_3$) in the ratio 1:20 added to plant extract ethanolic solution and heated at 80 ± 2°C until the colour of the solution was changed from colour less to thick brown (Figure 2). Resulted solutions were settled for 24 hours in dark to avoid any further photochemical reactions, after that the solution was centrifuged at 5000 rpm for 10 minutes with magnetic shaker. The supernatant was discarded and the pellet was air dried in the incubator.

The bioreduction of Ag^+ ions was monitored by periodic sampling by the UV spectrophotometer. The AgNPs in the freeze-drying bottle were suspended in ultrahigh purity water for all characterization methods and antibacterial assays. During biosynthesis of silver nanoparticles when stem extract was added to 100 ml of 1 mM $AgNO_3$ salt, the ionization took place as follows:

$AgNO_3(aq) \leftrightarrow Ag^+(aq) + NO_3^-(aq)$

$e^- + Ag^+ \rightarrow Ag^o$

It is assumed that the silver ions enter inside the plant cell via the H^+ATPase protein embedded in the thylakoid membrane by an electro genic pump[4]. Synthesis of silver nanoparticles is a photochemical reduction reaction.

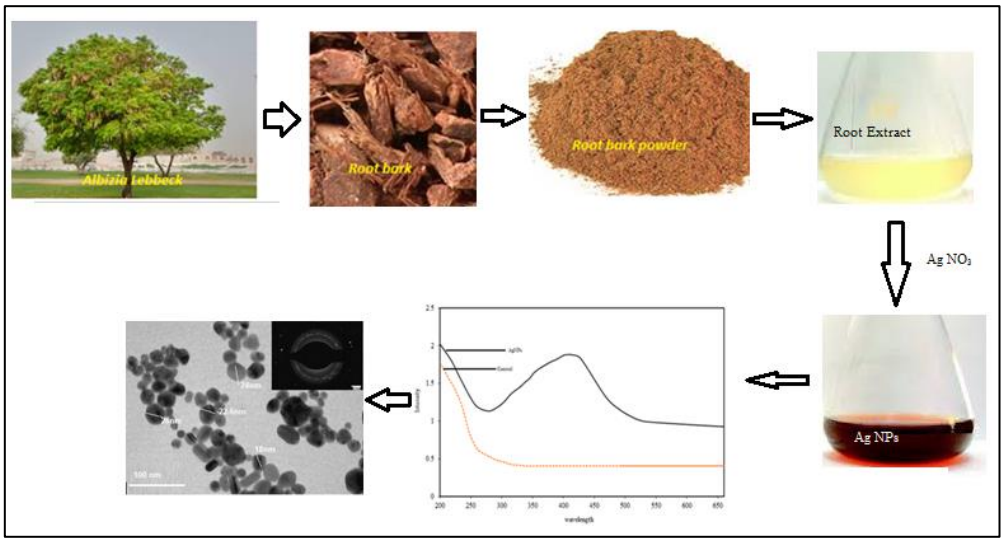

Figure 1. Shematic representation of green and chemical syntheses of silver nanoparticles (SNPs) using *A. lebbeck* root bark and evaluation of their surface characteristics

2.4 Characterization techniques

- UV–visible spectroscopy: The formation of dark brown color during the synthesis was confirmed as the formation of AgNPs. The reduction of the pure AgNPs was recorded under UV–visible spectroscopy using ELico model UV–visible spectrophotometer between 300 nm and 700 nm. The UV–visible spectra of the plant leaf extract and silver nitrate solution were also recorded.
- FTIR analysis was done using Perkin Elemer Spectrum-1, and was used to identify the chemical constituents in the region of 400-4000 cm^{-1} of the Ag-NPs
- XRD measurement: XRD measurements of Ag-NPs were cast into glass slides were done by Phillips PW 1830 instrument. The operating voltage of 40 kV and current of 30 mA with Cu kα radiation of 0.1541 nm wavelength, in the 2θ range 10- 80°, step size 0.02/θ.
- The morphology of the Ag-NPs was analyzed using an SEM. The powdered Ag-NPs were uniformly spread and sputter coated with platinum in an ion coater for 120 seconds, then observed by SEM JEOL-JSM 6360 MODEL, JAPAN). The size distribution of the nanoparticle was obtained by counting 150 particles from an enlarged SEM image.32 Elemental analysis of the powdered Ag-NPs was conducted using an EDX detector (EDS, EDAX Inc., Mahwah, NJ, USA) attached to the SEM machine.
- TEM analysis of Ag-NPs: Sample for TEM analysis was prepared, as mentioned in IR sample preparations. The sample was first sonicated (Vibronics VS 80) for 5 minutes. Ag-NPs were loaded on carbon coated copper grids, and solvent was allowed to evaporate under Infra light for 30 minutes. TEM measurements were performed on Phillips model CM 20 instrument, operated at an accelerating voltage at 200 kV.

3 RESULTS AND DISCUSSION

3.1 UV-Visible spectrophotometer studies

The light brown solutions of silver nanoparticles (figure 2 left) were analyzed by UV-visible spectroscopy to confirm the bio-reduction of silver ions (Ag^{2+}) into metallic silver nanoparticles by *A. lebbeck* root bark extract. The presence of a peak between 430-450 nm in UV-visible spectrum (Figure 2) confirms the synthesis of silver nanoparticles. Synthesis of the AgNPs was confirmed first by visual observation of the change in the color of $AgNO_3$ solution as a result of bio-reduction of Ag+ by plant extract.

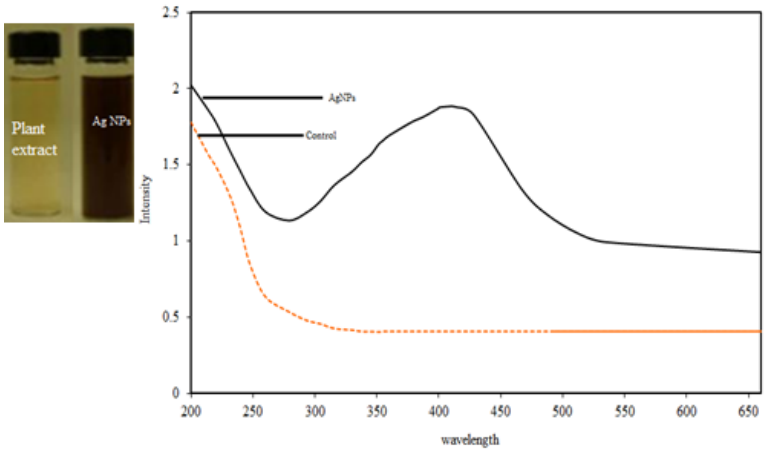

Figure 2: Photographs depicting synthesis of silver nanoparticles on the basis of visual observations and Ultraviolet-visible absorption spectra from *A. lebbeck* root bark extract

The Figure 2 shows the change of color from pale yellow to dark brown indicating the bio-reduction of Ag^{+} into AgNPs. Bio-reduction of Ag+ into metallic silver by leaf extracts begins after 30 minutes of incubation. In the control (without silver nitrate solution) no change in the colour was observed.

3.2 XRD studies

Analysis through X-ray diffraction was carried out to confirm the crystalline nature of the particles, and the XRD pattern showed numbers of Bragg's reflections that may be indexed on the basis of the face cantered cubic structure of silver. The XRD pattern of the biosynthesized AgNPs is depicted in Figure 3. It reveals the starch peak at 29.9 degree and four peaks at 2-theta values of 29.9, 40.2, 56.4 and 68.7 degrees corresponding to (111), (200), (220) and (311) planes of Silver respectively, according to the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), silver file No. 04–0783 [23]. The peak at 2-theta value of 29.9 degrees is the sharpest. The XRD study confirms / indicates that the resultant particles are the face cubic centre (fcc) silver nanoparticles. The average particle size of silver nanoparticle identified as 22.5nm in the present green method calculated using Debye-Scherrer equation [24].

$D = K\lambda / \beta \cos \theta$

Where D = the crystallite size of AgNPs particles

λ = the wavelength of x-ray source (0.1541 nm) used in XRD

β = the full width at half maximum of the diffraction peak.

K = the Scherer constant with value from 0.9 to 1.

θ = the Bragg angle.

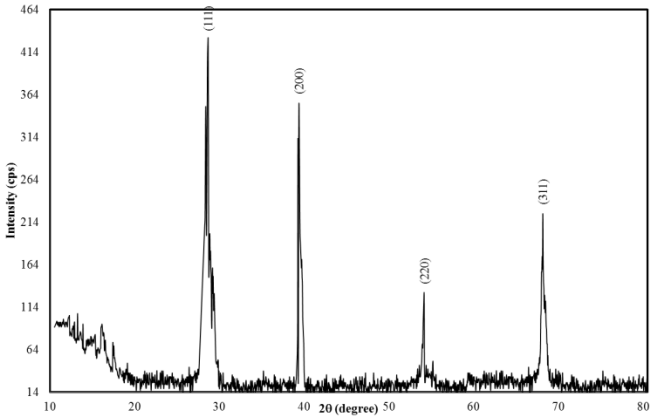

Figure 3. Showing the XRD pattern of *A. lebbeck* root bark extract silver nanoparticle

3.3 FTIR studies

FTIR has become an important tool in understanding the involvement of functional groups in relation between metal particles and biomolecules which is used to search the chemical composition of the surface of the silver nanoparticles and identify the biomolecules for capping and efficient stabilization of the metal nanoparticles [21]. There were many functional groups present which may have been responsible for the bio-reduction of Ag^+ ions.

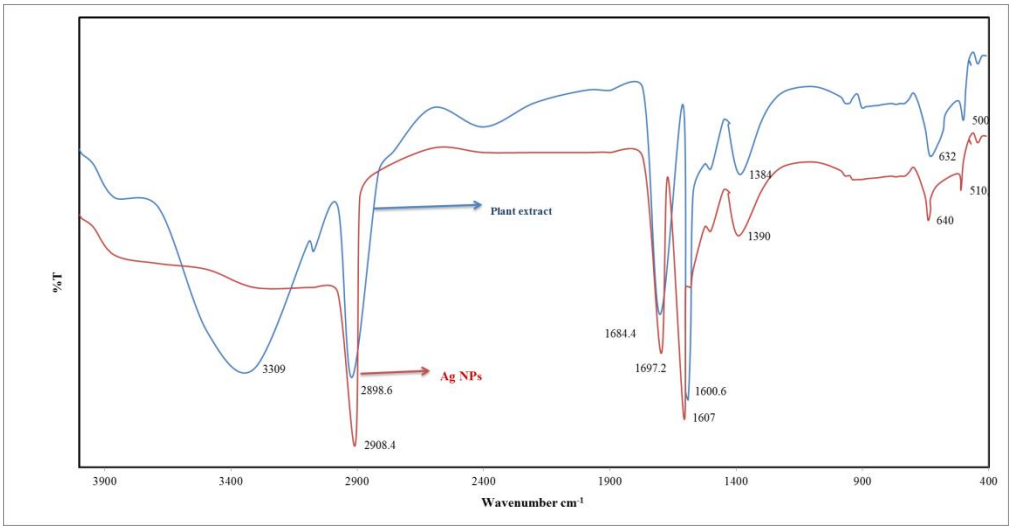

Figure 4: FTIR spectra of control and AgNPs of *A. lebbeck* root bark extract

As shown in Figure 3, the FT-IR spectrum of pure *A. lebbeck* root bark stem extract is remarkably similar to the FT-IR spectrum of AgNPs, except slight minimal shifts in a few peaks. This striking resemblance between these two spectra clearly suggests that some of the residual phytomolecules of the *A. lebbeck* root bark extract remained attached on the surface of the synthesized AgNPs. Therefore, the FT-IR spectrum of *A. lebbeck* root bark exhibit several absorption peaks at different locations including at, 3309 cm^{-1} (due to alcoholic or phenolic-OH), ~2898 cm^{-1} (-C–H, asymmetrical stretch), ~1684 cm^{-1} (due to C=O stretch), The narrow peaks at 1600 and 1384 regions clearly evince the presence of stretches of C=C-C and C-H bonds in aromatic rings that are found in Coumaric acid, Ferulic acid etc. 1384 cm^{-1} (C–O stretch) which correspond to various oxygen containing functional groups. The peak at 632 cm^{-1} is corresponds the alcohol and OH out-of-plane bending. Majority of these peaks are also present in the IR spectrum of AgNPs spectra with some minimal shifts. For instance, all the aforementioned peaks are slightly shifted in the FT IR spectrum of AgNPs and appeared at ~2908 cm^{-1}, ~1697 cm^{-1}, ~1607 cm^{-1}, 640 cm^{-1}, 510 cm^{-1}. Therefore, it proves that the reduction of the silver ions is matched to the oxidation of the hydroxyl and carbonyl groups.

Foundation of these band shifts, it can be concluded that both hydroxyl and carbonyl groups are included in the production of silver nanoparticles.

3.4 SEM-EDX studies

SEM technique was used to visualize the size and shape of silver nanoparticles obtained during the bioprocess. In Figure 5, SEM images were obtained with15 % of *A. lebbeck* root bark extract. The formation of silver nanoparticles as well as their morphological dimensions in the SEM study demonstrated that the average size was from 25–42 nm with inter-particle distance. The shapes of the silver nanoparticles proved to be spherical and some of the particles are cubical shape. EDX spectra recorded from the silver nanoparticles were shown in Figure 4. Energy dispersive spectrometry (EDX) micro-analysis is performed by measuring the energy and intensity distribution of X-ray signals generated by a focused electron beam on a specimen. EDX spectra were recorded from the silver nanoparticles. From EDX spectra (Figure 5 right side), it is clear that silver nanoparticles reduced by *A. lebbeck* root bark has the weight percentage of silver as 14.50%. From EDX spectra, it is clear that silver nanoparticles reduced by *A. lebbeck* root bark have the weight percentage of silver as 14.50%, carbon 74 %, 10 % of Oxygen element and 2 % of Nitrogen elements.

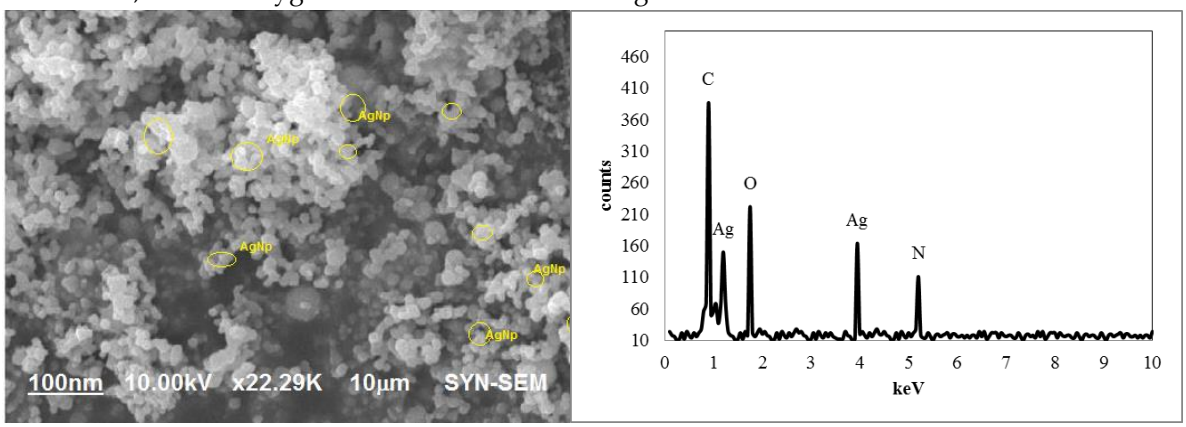

Figure 5: SEM image of silver nanoparticles formed by *A. lebbeck* root bark left & EDX spectra recorded for the AgNPs

3.5 TEM-Particle distribution studies

Figure 6 exhibits the TEM images of the silver nanoparticles produced *A. lebbeck* root bark extract and 1mM $AgNO_3$. It was detected that the nanoparticles are sometimes spherical and rarely cubical also determined abnormal distribution of particles. The size of the particles extended from 5 to 70 nm, and the mean particle size was around 22 nm (Fig. 6). A same result was described by Rahimi and et al 2014 applying *Ulva flexousa* reducing as well as capping agent [22].

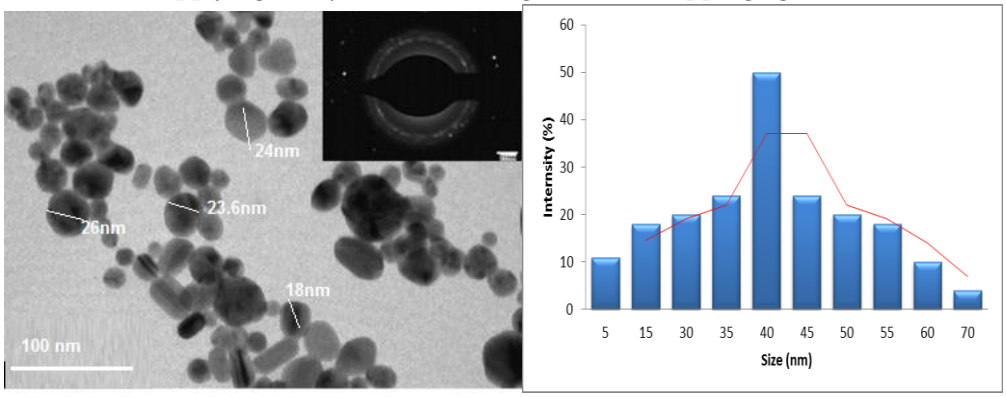

Figure 6: TEM image of synthesized silver nanoparticles (left), particle size distribution (right side) and inset picture is SAED pattern

The Selected-Area Electron Diffraction (SAED) patterns given reveal bright dots (figure 6), indicating that the nanoparticles are crystalline in nature. The data obtained corroborates other studies on green synthesis of metal nanoparticles

4. Conclusion

Green chemistry approach towards the synthesis of nanoparticles has many advantages such as, ease with which the process can be scaled up and economic viability. We have developed a fast, eco-friendly and convenient method for the synthesis of silver nanoparticles using *A. lebbeck* root bark extract with a diameter range of 22 nm. The silver nanoparticles synthesized from *A. lebbeck* root bark extract by bio-reduction method have exhibited all the characteristics features of the nanoparticles. In this study, we report rapid synthesis of silver nanoparticles in just 30 minutes by chemical method using bark extract as a reducing agent. Color change occurs due to surface Plasmon resonance during the reaction with the ingredients present in the plant leaves extract results in the formation of silver nanoparticles which is confirmed by UV–vis, XRD and SEM, having average mean size of 22nm had fcc structure. The synthesis developed in this study has distinct advantages over biological and chemical methods in being rapid, safe, and nontoxic, and moreover the synthesis of silver nanoparticles by this route is the appropriate way to develop green technology for the bulk synthesis of silver nanoparticles for industrial purpose.

Reference

[1]. Kagan VE, Bayir H, Shvedova AA. Nanomedicine and nanotoxicology: two sides of the same coin. Nanomedicine. 2005;1(4):313–6.

[2]. Saxena A, Tripathi RM, Zafar F, Singh P. Green synthesis of silver nanoparticles using aqueous solution of *Ficus benghalensis* leaf extract and characterization of their antibacterial activity. Mat Letters. 2012;67(1):91–4.

[3]. Alaraidh IA et al, Evaluation of Green Synthesis of Ag Nanoparticles Using Eruca sativa and Spinacia oleracea Leaf Extracts and Their Antimicrobial Activity, Iran J Biotech. 2014;12(1):e12392

[4]. Lok C, Ho C, Chen R, He Q, Yu W, Sun H, et al. et al. Silver nanoparticles: partial oxidation and antibacterial activities. J Biol Inorg Chem. 2007;12:527–534.

[5]. Ip M, Lui SL, Poon VKM, Lung I, Burd A. Antimicrobial activities of silver dressings: an in vitro comparison. J Med Microbiol. 2006;55:59–63.

[6]. http://easyayurveda.com/2012/12/26/shirisha-albizia-lebbeck-uses-medicinal-qualities-ayurveda-details/

[7]. M Farag; A El Gamal; A Kalil; A Al-Rehaily; O El Mirghany; K El Tahir, Pharmacology & Pharmacy, 2013,4, 473-477.

[8]. NP Babu; P Pandikumar; S Ignacimuthu, Journal of ethnopharmacology, 2009, 125, 356-360.

[9]. CR Resmi; MR Venukumar; MS Latha, Indian J Physiol Pharmacol., 2006, 50 (3), 297-302.

[10]. ZH Muhammad; A Shakeel; Q Mughal; E Sezai, Turkish Journal of Biology., 2013, 37, 25-32.

[11]. K Kokila; PS Deepika; V Sujatha, International Journal of Pharmacy & Pharmaceutical Sciences, 2013, 5 (3),70-73.

[12]. SM Seyydnejad; M Niknejad; I Darabpoor; H Motamedi, American J. of App. Sci., 2010, 7 (1), 13-16.

[13]. M Faisal; PP Singh; R Irchhariya, International Res Jour of Pharmacy, 2012, 3(5), 63-68.

[14]. S Verma, Research Journal of Pharmacy and Technology, 2013, 6, 1307-1313.

[15]. M Pandey; R Pandey; V Singh; V Pandey; U Singh, Mycobiology, 2002, 30, 55-56.

[16]. AK Dixit; LN Misra, Journal of natural products, 1997, 60, 1036-1037.

[17]. A Perwez; M Ali; A Vidhu. Der Pharmacia Lettre, 2011, 3 (6), 74-81.

[18]. ADP Barbosa, Journal of Pharmacy and Pharmaceutical Sciences, 2014, 6(11), 32-36.

[19]. A El-Mousallamy, Phytochemistry, 1998, 48, 759-761.

[20]. Ncube NS, Afolayan AJ, Okoh AI. Assessment techniques of antimicrobial properties of natural compounds of plant origin: current methods and future trends. African Journal of Biotechnology 2008; 7 (12): 1797-1806

[21]. Hemali Padalia, Pooja Moteriya, Sumitra Chanda, Arabian Journal of Chemistry, Volume 8, Issue 5, September 2015, Pages 732–741

[22]. Rahimi Z, Yousefzadi M, Noori A, Akbarzadeh A.Green Synthesis of silver nanoparticles using Ulvaflexousa from the Persian Gulf. J PG.2014; 5(15): 9-16.

[23]. Song JY and Kim BS, (2009). Rapid biological synthesis of silver nanoparticles using plant leaf extracts. Bioprocess and Biosystem Engineering, 32: 79-84.

[24]. Ahmad, N., Sharma S., AlamMd.K., V.N. Singh V.N., , Shamsi S.F., Mehta B.R., Fatma A. (2010). Rapid synthesis of silver nanoparticles sing dried medicinal plant of basil. Colloids and Surfaces B: Biointerfaces 81: 81-86.