

Synthesis and Characterization of Silver Nanoparticles by *Murraya Koenigii* Leaves

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ABSTRACT

Nanoparticles have a size of 1nm-100nm in any one of the three dimensions. Smaller nanoparticles have different physical, chemical and biological properties than atoms and molecules. Metals, non-oxide ceramic materials, metal oxides, silicates, and polymers, and organic and biomolecular components can be used to create material nanoparticles. Nanoparticles come in various shapes, like spheres, platelets, cylinders, and tubes. Green synthesized nanoparticles are not costly due to unavailability of toxic and hazardous compounds. Plants are widely spread, freely accessible, and safe to touch. They also supply a variety of metabolic compounds which are advantageous in reducing, capping and stabilizing process throughout in synthesis process. The reduction mechanism is based on the phytochemicals present in plant extract. In present work we synthesize silver nanoparticles by using *Murraya Koenigii* leaves through ecofriendly method. For synthesis of Silver nanoparticles, Silver nitrate ($AgNO_3$) used as metal precursor salt and green extract of *Murraya Koenigii* used as reducing and capping agent for formation of nanoparticles. The nanoparticles then formed characterized by X-ray diffraction, Scanning electron microscope, Energy dispersive spectroscopy, Dynamic light scattering, Fourier transform infrared spectroscopy and optical properties by UV-Visible spectroscopy. XRD revealed the crystalline structure of silver nanoparticles, FESEM and Dynamic light scattering revealed the particle size of 60 nm, FTIR revealed the presence of different functional groups which are attached with sample and Optical properties of sample revealed by UV-Visible spectroscopy that also satisfies different experimental results.

Keywords: Silver nitrate; Nanoparticle; Green extract; *Murraya Koenigii*

INTRODUCTION

Nanotechnology deals with the particles of size 1-100 nm, which plays an important role in the field of environment, medical, agriculture, electronics and several fields also. For synthesis of nanoparticles top down and bottom up approaches can be used (Boutinguiza et al. 2015; Marquestaut et al. 2014; Rafique et al. 2017; Chan et al. 2020; Goudarzi et al. 2016). Green synthesis is a part of bottom up approach in which activation, nucleation and termination phases are crucial for the formation of nanoparticles. Green method not only provides the desired shape and size of nanoparticles but also not produce toxic or hazardous substances for environment. The environment benign process, abundance of raw material, low cost, no toxic byproducts, large scale production, easy methodology are the key points for the success of Green method over different physical and chemical methods (Hashemi et al. 2020; Chakraborty et al. 2019; Pelaz et al. 2017). In this paper our research group synthesize silver nanoparticles by *Murraya koenigii* or sweet neem which is also known as curry plant in India. *Murraya koenigii* also used as a herb in ayurveda, spices,

vomiting, inflammation, antiulcer and many more (Handral et al. 2012). The phytochemicals present in this plant are alkaloids, calcium, phosphorous, iron, Vitamin C and Oxalic acid (Ajay et al. 2011). The synthesized nanoparticles have potential to detect heavy metal ions (Aragay et al. 2011). Alsammarraie et al., Established cost effective method for silver nanoparticles by green synthesis from the extract of turmeric powder (Alsammarraie et al. 2018). The synthesis of gold and silver nanoparticles of different size and shape by leaf extract of *Hibiscus rosa* have been reported by (Philip et al. 2010). Karthiga et al. studied the colorimetric sensing of heavy metal ions in aqueous solution by using silver nanoparticles synthesized from green extract of neem, mango, tea leaves across a wide range of PH values (2.0-11) and successfully reported the colorimetric sensors for toxic metal ions of Hg^{2+} , Pb^{2+} and Zn^{2+} (Karthiga et al. 2013). Singh et al. reported a eco-friendly approach to synthesize Silver nanoparticles of size 10-15 nm by using extract of mulberry and reveal the antimicrobial properties as well as the potential applications in waste water treatment (Singh et al. 2017).

SYNTHESIS PROCESS

SYNTHESIS OF NANOPARTICLES

MATERIALS AND METHODS

For synthesis process silver nitrate (AgNO_3) purchased from Glaxo Smithkline pharmaceuticals Limited Mumbai used as metal precursor salt and *Murraya Koenigii* leaves collected from the local area of Shekhawati Rajasthan. The collection and authentication of samples are the key factors for synthesis. The *Murraya Koenigii* leaves collected first washed with tap water then washed with double distilled water to remove the dust and other contaminating particles. After washing leaves cut in to fine pieces and leave them to dry for 15 days. After 15 days leaves crushed with grinder to make fine powder and stored in an air tight jar for future experimental purpose.

EXTRACT PREPARATION

25 gm of *Murraya Koenigii* leaves powder mixed with 200 ml D.I. water and heated for 45 minutes after that the mixture was filtered with whatman paper to find the extract. The extract stored at 4°C for further experimental process. To prepare the metal precursor salt solution of 0.1 M; 1.698 gm

AgNO_3 mixed in 100 ml DI added with *Murraya Koenigii* leave extract (20 ml) and placed for stirring for one hour. The colour changed from pale yellow to reddish brown can be observed with necked eyes (shown in Fig 2) which were the first indication of formation of nanoparticles. The flowchart of synthesis is shown in Fig 1.

EXPERIMENTAL TECHNIQUES

The different structural, morphological and optical properties were revealed by various characterization techniques. The crystalline nature of sample characterized by XRD analysis (Bruker-D8 advance instrument), Surface morphology revealed by FESEM, Elemental composition of sample revealed by EDS spectra, which is monitored by Nova Nano FE-SEM 450 with EDS facility. The formation of nanoparticles confirmed by DLS measurement using Malvern Zetasizer ZEN 3600 instrument. The functional group attached with sample revealed by FTIR Instrument of PerkinElmer spectrum version (Frontier) and optical properties like SPR absorption peak and band gap revealed by UV-Visible spectra provided by LAMBDA 750 (Perkin Elmer) UV-Vis NIR Spectrophotometer. The baseline correction will be done using Deionized water in UV- Visible spectra.

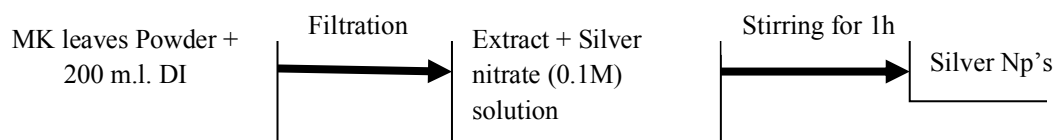


FIGURE 1. Flow Chart for Synthesis of Silver nanoparticles



FIGURE 2. Colour changing process from pale yellow to Red-Brown

RESULT AND DISCUSSION

X-ray diffraction: XRD technique revealed the presence of different crystalline plane and the crystallite size can be found with the help of Debye-Scherrer formula $D = (0.94\lambda / \beta \cos \theta)$, which depends upon the wavelength of incident radiation, angle of diffraction and FWHM. The XRD Pattern of Silver nanoparticle was recorded in a wide range of angle from 10° to 80° as shown in Figure 3. The diffraction maxima found at 27.90° , 32.33° , 38.23° , 44.42° , 46.38° , 64.59° , 77.47° . The most intense peak found at 38.23° . The other intense peaks at 38.23° , 44.42° , 64.59° , 77.47° represents the presence of diffraction planes at (111), (200), (220) and (311) which is found in good agreement with data provided by JCPDS No.

04-0783. There exists some planes other than JCPDS file may be due to the presence of some impurity in sample or presence of some other molecules (Kirtiwar et al. 2019). The XRD pattern revealed the crystalline nature of the samples with planes.

FESEM and EDS: FESEM revealed the surface morphology of Ag nanoparticles of irregular shape of as shown in Figure 4. Nanoparticles aggregates due to large surface area. The average crystalline size found around 60nm by FESEM and EDS result show the elemental composition of the sample (shown in Figure 5). This sample consists of Ag, Au, O and Cl. There percentages are in shown in Table 1 for normal and atomic %.

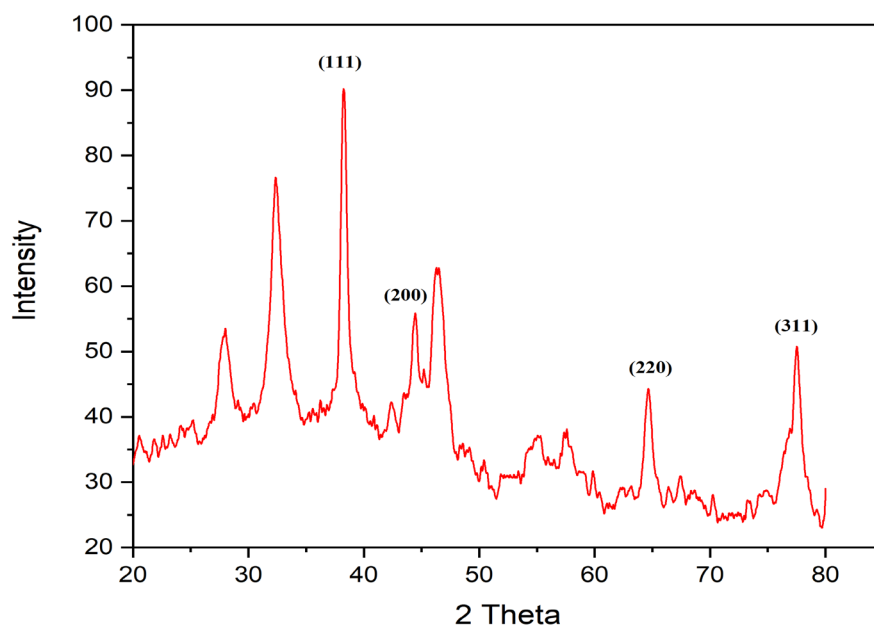


FIGURE 3. XRD Pattern of silver nanoparticles

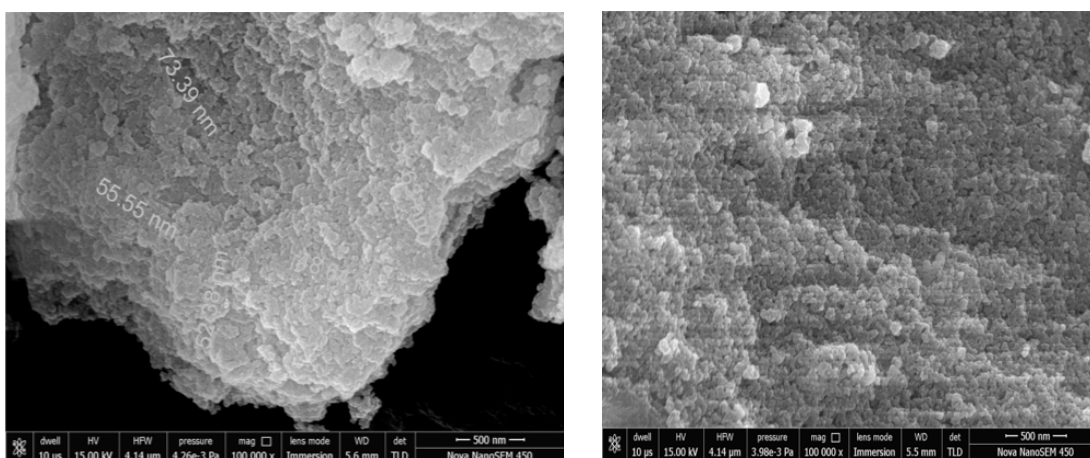


FIGURE 4. SEM Pattern of silver nanoparticles

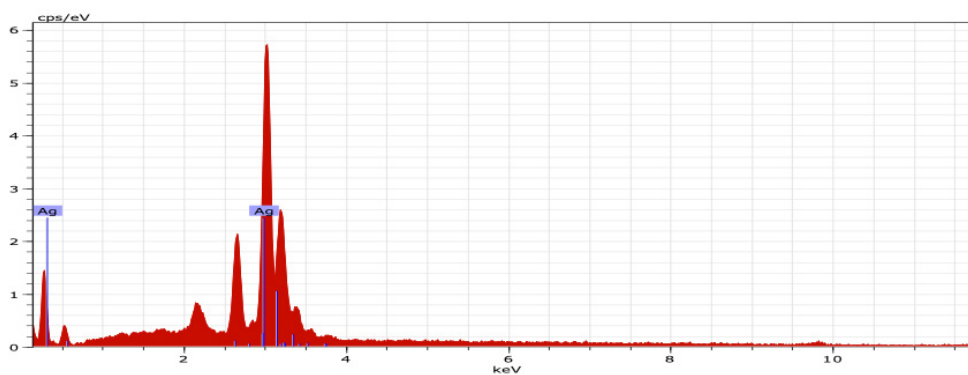


FIGURE 5. EDS Pattern of silver nanoparticles

TABLE 1. Elemental analysis of Silver nanoparticles

| Elements | Atomic No. | Series | norm.[wt%] | Atom. [at.%] |
|----------|------------|----------|------------|--------------|
| Ag | 47 | L-series | 71.14 | 49.64 |
| Au | 79 | L-series | 15.91 | 6.08 |
| O | 8 | K-series | 6.50 | 30.60 |
| Cl | 17 | K-series | 6.44 | 13.68 |
| Total: | | | 100.00 | 100.00 |

DLS measurements: Dynamic light scattering results revealed the particle size range from 0.3nm to 10000 nm. This technique capable for characterization of particles size based on Brownian motion and Stokes-Einstein relationship, which explained that the particles size is inversely proportional to the diffusion coefficients. This relation also show that the smaller particles travelled with faster than heavier particles. The light scattered from the particles

contains the information about the diffusion coefficients or the particle size. The particle size histogram and behavior of correlation coefficient are shown in Fig 6(a). The decay of correlation coefficient with time [Figure 6(b)] explains the size of particles, and results revealed that the correlation coefficient falls more rapidly with time for smaller size of particles.

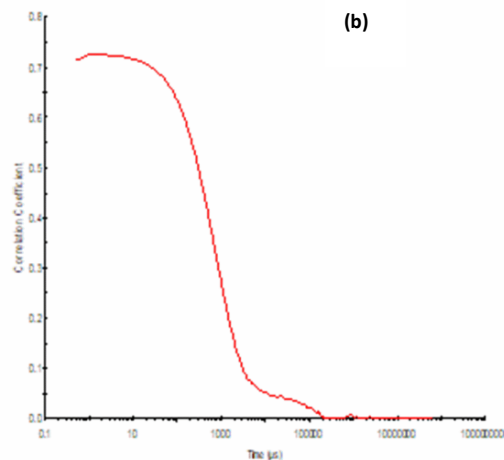
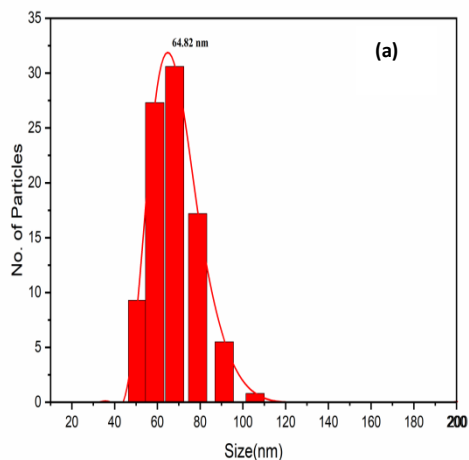


FIGURE 6. (a) Particle size histogram and (b) Correlation Coefficient for the Silver nanoparticles

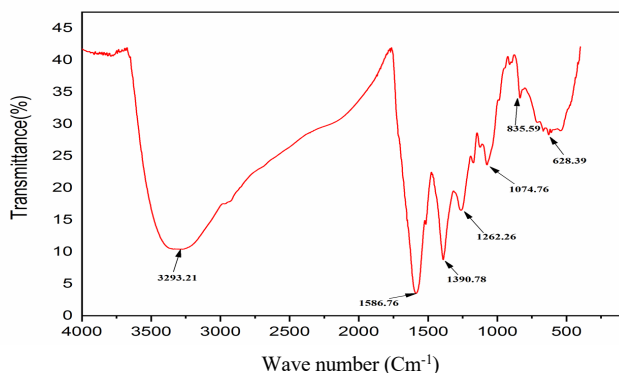


FIGURE 7. FTIR Spectra of silver nanoparticles

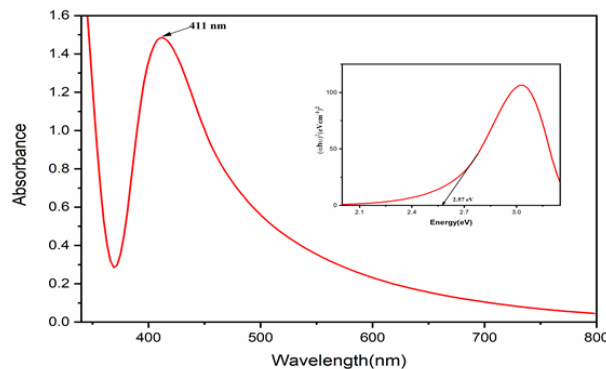


FIGURE 8. UV-Visible Spectra of silver nanoparticles

FTIR measurements: Fourier transform infrared spectroscopy show the different functional groups attached with the sample. FTIR spectrum measured over a wide range of wave number 4000 cm^{-1} to 400 cm^{-1} as shown in Figure 7. A broad signal at 3293 cm^{-1} revealed the presence of hydroxyl group (-OH) stretching. The peak at 1586 cm^{-1} show the strong presence of C=C stretching due to Nitro group. Various peaks from 1070 cm^{-1} to 1350 cm^{-1} found due to the C-O stretching of carboxylic acid, alcohols and ethers. The bending vibration of C-H bonds of aromatic compounds is appeared at 835 cm^{-1} and 628 cm^{-1} (Hashemi et al. 2020; Qais et al. 2019).

UV-Visible Spectroscopy: With the help of UV-Visible spectroscopy absorbance of sample were recorded over a wide range of wavelength of electromagnetic radiation. The absorbance spectra show absorbance peak at 411 nm with blue shift of 19 nm with respect to the bulk sample. The direct band gap of silver nanoparticles found about 2.57 eV, which is calculated by Tauc's plot as shown in Figure 8. This result is very close to the band gap of silver nanoparticles which is about 2.51 eV. The results provided by UV-Vis spectra are similar with the reported results (Qais et al. 2019). The band gap of silver nanoparticles also depends upon the synthesis process because the particle size reduces as band gap increases (Gharibshahi et al. 2017; Anuja et al. 2020; Rafique et al. 2019). We can impart nanoparticles of different size with different process. The optical band gap of the particles decreases with the increase in grain size. The barrier height of the grain boundary increases with the increase in grain size resulting in a decrease in the band gap of the particles.

CONCLUSIONS

Silver nanoparticles were successfully synthesized by *Murraya koenigii* leaves. The phytochemicals present in leaves extract plays crucial role in reducing the metal precursor salt. The crystalline structure of synthesized nanoparticles disclosed by XRD, FESEM with EDS confirmed the presence of silver in sample. The DLS results revealed the particle size of nanoparticles which was about 60 nm, while FTIR Results also favorable for Ag nanoparticles with surrounding OH stretching, C=C stretching C-O stretching, vibration of C-H bonds. The optical properties have been revealed a blue shift of 19 nm in absorbance peak and confirmed the formation of silver nanoparticles. The direct band gap of silver nanoparticle is calculated about 2.57 eV using Tauc's plot. The blue shift of the fundamental absorption peak of Silver nanoparticle gives the information about particle size and their distribution, which is also favorable to the theoretical determination of absorbance of the nanoparticles.

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DECLARATION OF COMPETING INTEREST

None

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