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Green Synthesis of Silver Nanoparticles Using *Terminalia Bellirica* Leaves Extract

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Abstract: Nowadays, for green synthesis, different parts of plant extracts are being used for the synthesis of metallic nanoparticles. Green routes for the synthesis of metallic nanoparticles are most favorable method over conventional methods. In this study, we have synthesized Ag nanoparticles from *Terminalia bellirica* aqueous leaves extract with $AgNO_3$ solution. Recently, silver nanoparticles are being used for their beneficial effects on human health. It has been reported that it helps in lowering in cholesterol, blood pressure, thirst, pile, skin diseases and treatment of diabetes. Ag nanoparticles were characterized by UV-Vis spectrophotometer, XRD, TEM and FTIR techniques. It was found that Ag^+ ion reduced into Ag^0 and showed absorption band at 447 nm. X-Ray Diffraction for structural determination confirms the crystalline Ag nanoparticles. TEM analysis confirms the average particle size is less than 30 nm with spherical shape. FTIR spectra confirms the presence of active biomolecules (alcohols, phenols, proteins and nitro compounds) of plant leaves extract which played a key role in formation of Ag nanoparticles.

Keywords: silver nanoparticles, *Terminalia bellirica*, XRD, TEM, crystalline

Introduction

Terminalia bellirica Roxb is an important medicinal plant of Himalaya which belongs to Combretaceae family and is popularly known as “bahera’’. *Terminalia bellirica* is a deciduous tree, growing on wild throughout indian subcontinent, Srilanka and SE Asia, upto 1200 m in elevation (Bardul 2011; Motamarri et.al., 2012). It helps in lowering cholesterol, blood pressure, thrist, pile, skin diseases, worm, reduces ageing and in loss of appetite (Sabu et.al., 2009; Devi et.al., 2014). The fruits pieces are baked and chewed for cough, cold, hoarseness of voice and asthma. The unripe fruits are mild laxative and ripe fruits are astringent and antidiarrheal (Motamarri et.al., 2012; Deb et.al., 2016). Oil extract from the seed pulp is used in leucoderma and alopecia. The bark is mildly diuretic and is useful in Anaemia and Leucoderma (Sabu et.al., 2009; Bardul 2011). It exhibited a variety of biological activities, such as antioxidant, antimicrobial, antidiarrheal, anticancer, analgesic, antipyretic, antidiabetic and antihypertensive (Elizabeth et.al; 2005; Sabu et.al., 2009; Sharma et.al., 2010; Motamarri et.al., 2012; Deb et.al., 2016). Nanoscaled materials is a material in which at least one dimension of structural component is less than 100 nm (Awwad et.al., 2013; Khan et.al., 2018). Metallic nanoparticles are being widely used in different fields such as medicine, agriculture, cosmetic industry, drug delivery, catalysis and waste water treatment (Thakkar et.al., 2010; Awwad et.al., 2013; Chintamani et.al., 2018; Ovais et.al., 2018). The nanosized metallic nanoparticles (Au, Ag and Pt) are broadly being used commercially in shampoos, soaps, detergents, anti-ageing creams and perfumes for personal and beauty care (Iravani 2011; Jemel et.al., 2017). In the current scenario, the synthesis of nanoparticles in the research area is being done more biologically than chemical method because it is eco-friendly, cost effective, single step, easy to handle, safe and non-toxic (Daizy 2010; Mile et.al., 2013; Khan et.al., 2018). In biological method, microorganism (bacteria, fungi and yeast) and plant parts (leaf, bark, seeds, roots and fruits) are used for the synthesis of metallic nanoparticles (Ag, Au, Pt, ZnO and CuO) (Daizy 2010; Mason et.al., 2012; Reddy et.al., 2012). Phytoconstituents (polyphenolic acid, flavones, alkaloids, tannins, terpenoids, essential oils etc) present in different plant parts extracts act as reducing, capping and stabilizing agent of metallic nanoparticles (Iravani 2011; Noruzi 2015; Sati et.al., 2020). Many research work has been reported for the synthesis of Ag nanoparticles using different plants and their parts (Iravani et.al., 2011; Zargar et.al., 2011). The objective of present study is to synthesize Ag nanoparticles from leaves extract of *Terminalia bellirica* and its characterization by XRD, UV-VIS spectrometry, TEM and FTIR analysis. This green approach is convenient, cheap, environmentally advantageous over toxic chemicals.

Methodology

Preparation of aqueous plant extract

Fresh leaves of *T. bellirica* were collected from Advani forest region, Pauri Garhwal (Uttarakhand). Leaves were washed with double distilled water to remove filth/dirt. Then, washed leaves kept for drying under shade. Dried leaves were mashed to fine powder with mortar-pestle. There after 12 g of mashed leaves were boiled with 250 mL distilled water for 20 min at 65^0 C. Then, aqueous leaves extract was prepared after filtration with Whatman no. 1 filter paper. Extract was stored for the Ag NP's synthesis. The plant has been authenticated in the department of Botany, FRI, Dehradun.

Synthesis of Ag nanoparticles

Initially, 2 mM $AgNO_3$ (2L) aqueous solution was prepared. 200 mL aqueous leaves extract of *Terminalia Bellirica* was mixed with 1800 mL aqueous $AgNO_3$ solution (2mM) with 1:9 in a 2 L Erlenmeyer conical flask. The solution colour changed from light yellow to greyish black within 3 days indicating the formation of

Ag nanoparticles. Then, solution was centrifuged for 15 minutes at 7500 RPM. There after it was washed with distilled water followed by acetone to remove impurities. The gained materials dried at 45°C for 1 day in oven and using mortar-pestle for grinding dried materials to get fine powdered greyish black Ag nanoparticles. Finely powdered greyish black nanoparticles were stored in air locked bottles for the characterization. Silver nitrate is obtained from Blulux Laboratories (P), Limited, Faridabad-India.

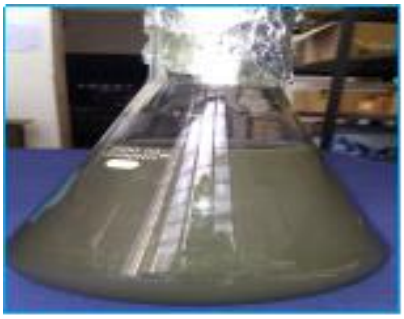

Fig .1: Synthesized Ag nanoparticles

Characterization of synthesized Ag Nps

Elite double beam UV-Vis spectrophotometer was used to investigate the formation of Ag nanoparticles. The spectra of the mixtures ($AgNO_3$ solution and leaves extract) were taken between 300 and 900 nm.

XRD Analysis. X-ray Diffraction (XRD) (PAN Analytical, X-pert pro, Diffractometer) was used for the structural and phase determination of Ag nanoparticles and its spectra was taken in the range of 2ɵ from 0^0 to 80^0. The average particle size was calculated by using Scherrer's equation.

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

FTIR Analysis. FTIR spectrometer (Perkin Elmer Model RZX) was used to analyze functional groups of the synthesized Ag nanoparticles in the range 4000-400 cm^{-1} using KBr pellet method.

TEM Analysis. Transmission electron microscope (JEOL JEM 1400) analysis was used to determine the surface morphology of synthesized silver nanoparticles.

Result and Discussion

In the present work, aqueous leaves extract of *Terminalia bellirica* was used to synthesize Ag nanoparticles.

UV-Visible Analysis.

It was observed that solution color changes from light yellow to dark brown or greyish black. UV-Vis spectra of the solution was taken in the range 300-900 nm. The color change is due to the surface plasmon resonance phenomenon in Ag nanoparticles (Awwad et.al., 2013). Several studies showed the similar results (Mason et.al., 2012; Jemel et.al., 2017; Sati et.al., 2020). The Surface Plasmon Resonance band observed at 447 nm which indicated the formation of Ag nanoparticles. Silver nanoparticles possesses absorption maximum band in the range of 400-500 nm (Kandwal et.al., 2019). (Fig. 2)

XRD Analysis. X-Ray diffraction studies confirms the crystalline nature of Ag nanoparticles. The XRD analysis of synthesized Ag nanoparticles from leaves extract of *T. bellirica* showed sharp diffraction peaks at $2\Theta = 36.98^0$, 43.04^0 and 63.33^0 which are indexed to (111), (200) and (220) lattice planes respectively (Table 1) of face centered cubic nano crystals (Fig. 3). Comparing with the standard data, which confirm the nanocrystalline synthesized Ag nanoparticles. The average size of synthesized nanoparticles from leaves extract of *T. bellirica* is found to be less than 5 nm. Similar results of synthesized Ag nanoparticles using guava leaves extract (Bose; 2016), Carob leaves extract (Awwad et.al., 2013), *mulberry* leaves extract (Awwad et.al., 2012) and *Ocimum sanctum* leaves extract (Singhal et.al., 2011; Garima et.al., 2011) were reported.

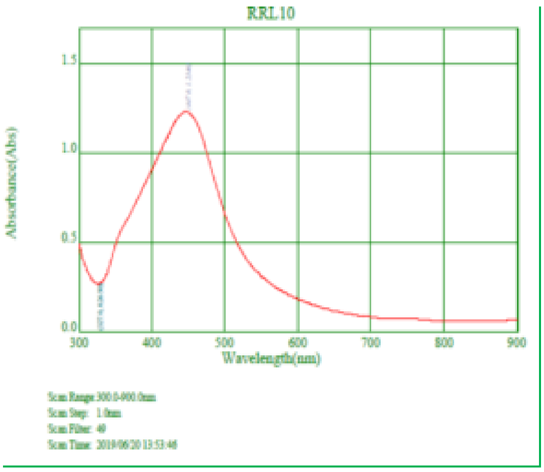

Fig. 2: UV-visible spectrum of silver nanoparticles

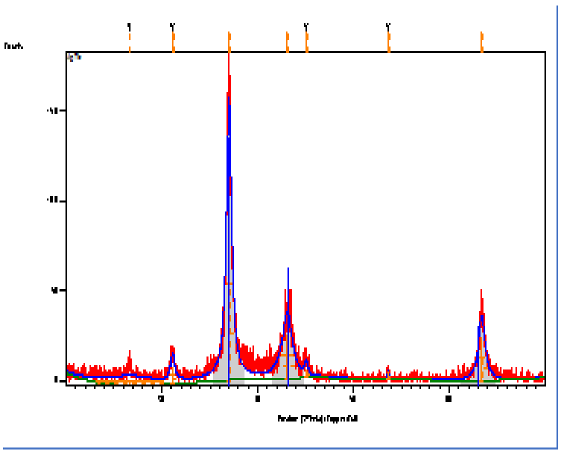

Fig. 3: XRD Spectra of silver nanoparticles

Table 1: Peak list for average size calculation of nanoparticles

2ϴ	HKL	FWHM LEFT	D-SPACING (Å)	REL. INT. (%)
36.98	111	0.64	2.42	100
43.04	200	1.21	2.09	22.99
63.33	220	0.78	1.46	23.7

Where ϴ is the Bragg's angle, FWHM is full width at half maxima, (hkl) are miller indices and d-spacing is interplanar distance.

FT-IR analysis

Green synthesized Ag nanoparticles of leaves extract of *T. bellirica* were characterized by FT-IR technique. The FT-IR spectra of *T. bellirica* leaves extract synthesized Ag nanoparticles in Fig. 4 shows peaks at 3429.1 cm^{-1},1620.2 cm^{-1} and 1384.2 cm^{-1}. The absorption peaks at 3429.1 cm^{-1} in the FTIR spectra depicts the O-H stretching vibration of alcohol and phenol. The peak at 1620.2 cm^{-1} corresponds to C-N and C-C stretching indicating the presence of proteins (Jemel et.al., 2017). The band at 1384.2 cm^{-1} corresponds to N=O symmetry stretching of the nitro compound which may reveals that it acts as reducing and capping agents for the synthesis of silver nanoparticles.

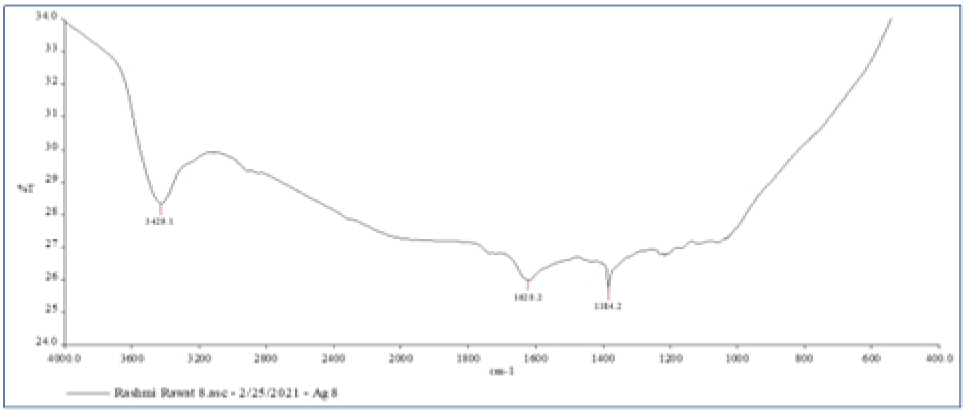

Fig. 4: FTIR spectrum of silver nanoparticles

TEM analysis

TEM analysis was used to study the surface morphology of Ag nanoparticles (Fig. 5). These results confirmed that silver nanoparticles were spherical in shape with their average size less than 30 nm. Similar results in the synthesis of Ag nanoparticles using guava leaves extract (Bose; 2016), Carob leaves extract (Awwad et.al., 2013), *mulberry* leaves extract (Awwad et.al., 2012), and *Ocimum sanctum* leaves extract (Singhal et.al., 2011; Garima et.al., 2011) were reported.

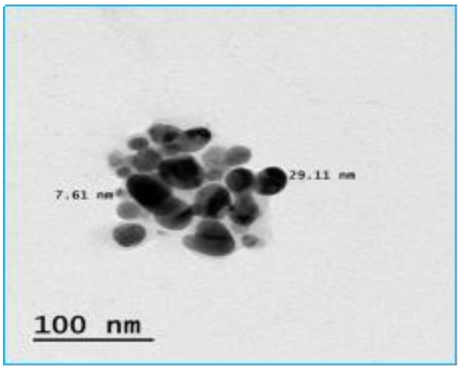

Fig. 5: Transmission electron micro-image of silver nanoparticles

Conclusion

In this study, we synthesized green Ag nanoparticles using leaves extract of *T. bellirica*. Characterization techniques (XRD, FTIR, TEM) confirmed that the Ag nanoparticles are stable, crystalline in nature, spherical in shape and crystallite size is less than 30 nm. This green route of Ag nanoparticles is simple, cost effective, safe to handle and advantageous to the environment over hazardous chemicals.

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Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of this article.

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