Green synthesis of silver nanoparticles and their characterization by XRD

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Green synthesis of silver nanoparticles and their characterization by XRD

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Abstract. A cost effective and environment friendly technique for green synthesis of silver nanoparticles has been reported. Silver nanoparticles have been synthesized using ethanol extract of fruits of *Santalum album* (Family Santalaceae), commonly known as East Indian sandalwood. Fruits of *S.album* were collected and crushed. Ethanol was added to the crushed fruits and mixture was exposed to microwave for few minutes. Extract was concentrated by Buchi rotavaporator. To this extract, 1mM aqueous solution of silver nitrate ($AgNO_3$) was added. After about 24 hr incubation Ag^+ ions in $AgNO_3$ solution were reduced to Ag atoms by the extract. Silver nanoparticles were obtained in powder form. X-ray diffraction (XRD) pattern of the prepared sample of silver nanoparticles was recorded The diffractogram has been compared with the standard powder diffraction card of JCPDS silver file. Four peaks have been identified corresponding to (hkl) values of silver. The XRD study confirms that the resultant particles are silver nanoparticles having FCC structure. The average crystalline size D, the value of the interplanar spacing between the atoms, d, lattice constant and cell volume have been estimated. Thus, silver nanoparticles with well-defined dimensions could be synthesized by reduction of metal ions due to fruit extract of *S.album.*

1. Introduction

The present paper deals with the synthesis of nanoparticles of silver using ethanol extract of the fruits of *Santalum album,* which is commonly known as East Indian sandalwood and belongs to the family Santalaceae. It is a mid-sized evergreen parasitic tree easily grown in India. The essential oil of sandalwood is usually prepared by steam distillation from the heartwood and is used in perfumes, cosmetics, and various medicines [1]. Though different parts of this plant can be used to prepare their extracts, only the extract of heartwood have been investigated and reported extensively in literature. There are hardly any reports about other extracts. In the present work we have synthesized the extract of the fruits and utilized it for the reduction of $AgNO_3$ to obtain silver nanoparticles.

Nanoparticles are atomic or molecular aggregates with at least one dimension between 1 and 100 nm, that can drastically modify their physico-chemical properties compared to the bulk material. Nanotechnology has opened up novel fundamental and applied frontiers in materials science. Nanoparticle production and applications have been extensively studied. Nanoparticles can be made from a variety of bulk materials and that they can explicate their actions depending on both the chemical composition and on the size and/or shape of the particles. In the last decade, biosynthesis of nanoparticles has received increasing attention due to a growing need to develop environmentally benign technologies in material syntheses. The significance of such a synthetic protocol has been well demonstrated. For instance, a great deal of effort has been put into the biosynthesis of inorganic

materials, especially metal nanoparticles, using microorganisms and plants**.** The formation of nanoparticles mediated by biological route, i.e., green process, is considered as better method than any other method [2].

In nanotechnology, silver nanoparticles are the most promising ones. Silver nanoparticles are nanoparticles of silver, i.e., particles size in range of between 1 and 100 nm and because of its nano size they have attracted intensive research interest. Silver nanoparticles are of interest because of the unique properties (*e.g.,* size and shape depending optical, electrical, and magnetic properties) which can be incorporated into antimicrobial applications, biosensor materials, composite fibers, cryogenic superconducting materials, cosmetic products, and electronic components. A variety of biological sources are able to produce silver nanoparticles of different shapes and nature. The biosynthetic method employing plant extracts has received some attention as a simple and reliable alternative to chemical procedures and physical methods synthesizing metal nanoparticles only in recent years [3]. Jose-Yacaman *et al* have reported the formation of gold and silver nanoparticles by living plants [4,5,6]. Sastry *et al* have extensively used the biosynthesis method of obtaining metal nanoparticles by plant leaf extracts and their potential applications [7-15]. They studied bioreduction of silver ions by a broth of geranium leaf [7,8] or Neem leaf. Very recently, they have demonstrated synthesis of silver nanoparticles using *Aloe vera* plant extracts.

Silver nanoparticles can be produced either intra or extra cellularly by using living organism like diatoms, fungi, bacteria, viruses etc. or mimicking biological systems using biomolecules isolated from them. While micro-organisms continue to be investigated for metal nanoparticle synthesis, the use of plant extracts in similar nanoparticle biosynthesis methodologies is an exciting possibility and is relatively unexplored and under-exploited. In the present paper, an attempt has been made to use plants for the extra-cellular synthesis of silver nanoparticles and demonstrate their capabilities as an alternative to chemical or micro-biological synthetic processes. To circumvent the problem of internalization of nanoparticles an attempt has been made to use the extract of a part of the plant, namely, fruit of *S.album*, instead of the live part.

2. Experimental

Extracts of different parts of *S.album* are commonly obtained by conventional distillation process which requires great energy, significant amount of solvents, and quite a long process time. Therefore, the use of new 'green technique' for extraction with minimum/low energy, solvents, and time is preferred. Hence, in the present work we have used microwave assisted extraction method for getting ethanol extract of fruits of *S.album.* All the reagents used for reaction were analytical grade chemicals and used without further purification. Redistilled deionized water was used for sample preparation.

The fruits of *S.album* are globose, fleshy drupe; red, purple to black when ripe, about 1 cm in diameter, with hard ribbed endocarp and crowned with a scar, almost stalkless, smooth, single seeded. Fruits were collected from the local area. They were washed and shed dried. These dried fruits were then crushed using a pestle and mortar. A measured amount of ethanol was added to the crushed sample. The mixture was kept in microwave for few minutes. Microwave ovens are generally operated at 2.45 GHz. Owing to their electromagnetic nature, microwaves possess electric and magnetic fields which are perpendicular to each other. The electric field causes heating via two simultaneous mechanisms, namely, dipolar rotation and ionic conduction. Dipolar rotation is due to the alignment on the electric field of the molecules possessing a dipole moment in both the solvent and the solid sample. This oscillation produces collisions with surrounding molecules and thus the liberation of thermal energy into the medium.

Extract was concentrated by Buchi rotavaporator. To 10 ml of this concentrated extract 90 ml of 1mM aqueous solution of silver nitrate ($AgNO_3$) was added. The mixture was kept in dark at room temperature. After about 24 hr incubation Ag^+ ions in silver nitrate solution were reduced to Ag atoms by the extract and the colour of the solution was observed to change from pale yellow to dark brown. A sufficient amount of dark brown precipitate was observed and it was separated by high speed centrifugation. The separated solid mass was washed with double distilled water for few times to remove impurities. After complete washing the solid mass was kept open to dry. The complete drying

of this solid mass resulted in a black colored material which was collected in powder form and sampled for characterization purpose.

3. Results and discussions

The X-ray diffraction (XRD) pattern of the prepared sample of silver nanoparticles was recorded at UGC-DAE Consortium for Scientific Research, Indore, by employing Bruker d8 Advance X-ray diffractometer, using CuKα radiation (λ = 1.5406 Å), 40 kV- 40mA, 2θ/θ scanning mode. Data was taken for the 2θ range of 30 to 80 degrees with a step of 0.0202 degree. The XRD data and it's analysis are given in table 1. The diffractogram (Fig. 1) has been compared with the standard powder diffraction card of JCPDS, silver file No. 04-0783. Four peaks at 2θ values of 38.2901, 44.5583, 64.8185, and 77.4383 degree in the experimental diffractogram have been identified to be due to silver metal and corresponding to (hkl) values - (111), (200), (220) and (311) planes of silver and are labeled as 1-4 in fig.1. The details of these peaks are given in table 1. The XRD study has thus confirmed that the resultant particles in the prepared sample are silver nanoparticles having face centered cubic crystal structure. There are four more peaks in the diffractogram at 32.35, 46.38, 54.03 and 57.66 degrees. These peaks have been identified to be due to $AgNO_3$, which might have not been reduced and hence remained in the sample in minute quantity. These peaks are not included in table 1.

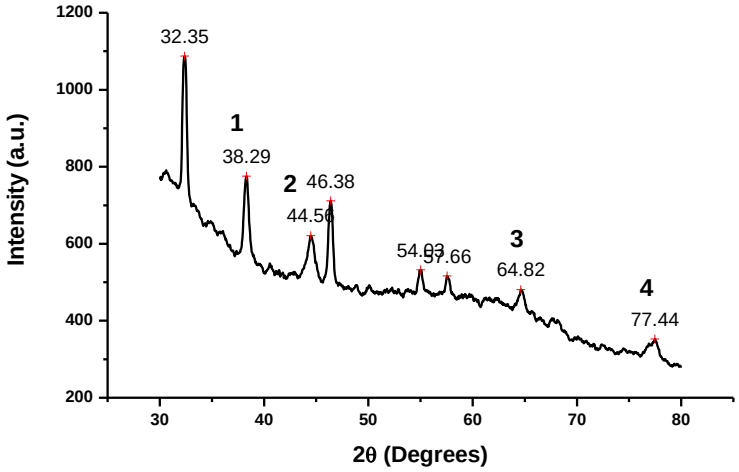

Figure 1. XRD of silver nanoparticles. Ag peaks are marked 1-4 and 2θ values are given.

The average crystalline size D of the silver nanoparticles have been estimated from the diffractogram by using Debye-Scherrer formula, $D = 0.9\lambda/ \beta \cos\theta$, where λ is the wavelength of the X-rays used for diffraction and β is full width at half maximum (FWHM) of a peak [16]. To estimate FWHM, each of the four peaks was fitted with a Gaussian function. The FWHM of the fitted gaussian curve is taken as FWHM of the peak. This could be done in the software *origin 6.1*. Four values of 'D'

Table 1. XRD results of silver nanoparticles.

P. No	2θ	Cos θ	Sin θ	FWHM degree	β radian	Cryst--alline size 'D' nm	Inter-planar spacing 'd' Å	h k l Identi-fied from peak	$h^2+k^2+l^2$ from identi--fied h k l	Lattice const. 'a' from d Å	Cell vol--ume Å^3
1	38.2901	0.9447	0.328	0.3407	0.0059	24.68	2.3485	1 1 1	3	4.0677	67.30
2	44.5583	0.9253	0.3791	0.6397	0.0111	13.41	2.0319	2 0 0	4	4.0638	67.11
3	64.8185	0.8442	0.536	0.6033	0.0105	15.59	1.4371	2 2 0	8	4.0648	67.16
4	77.4383	0.7789	0.6271	0.3607	0.0063	28.27	1.2284	3 1 1	11	4.0740	67.62

have thus been obtained from the data for the four peaks. It is seen from table 1 that the four values are nearly the same.

The average crystalline size of the silver nanoparticles is found to be 20 nm. The value of the interplanar spacing between the atoms, d, has been calculated using Bragg's Law, $2d\sin\theta = n\lambda$, where n is the order of diffraction pattern. In the present case n is equal to 1. From the four data sets for the four peaks, four values of 'd' are obtained and are given in table 1. It is seen that the four values are nearly the same.

Lattice constant has been estimated using the formula, $a = d*\sqrt{(h^2+k^2+l^2)}$. Average of the four values of 'a' calculated from the four values of 'd' as obtained from the data for the four peaks is found to be 4.0676 Å. This is in fair agreement with the standard value for silver that is 4.0857 Å.
Cell volume has also been estimated from the value of lattice constant and the values are given in table 1.

Thus, the XRD analysis has shown that silver nanoparticles with well-defined dimensions could be synthesized by reduction of metal ions due to fruit extract of *S.album.*

4. Conclusions

The present work shows that the reduction of silver ions from $AgNO_3$ to Ag occurs due to fruit extract of *S.album.* The synthesized silver particles have been characterized by XRD. The obtained XRD data for 2θ positions identifies the sample as silver crysatlline particles having (hkl) values corresponding to FCC silver. The d-spacing values, lattice constant and cell volume, all comfirm the sample to be silver crysatlline particles. The average crystalline size was estimated to be 20 nm. confirming the nanoparticle nature of the obtained sample. Thus, the present method leads to the formation of silver nanoparticles with well-defined dimensions.

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