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RAPID GREEN SYNTHESIS OF SILVER NANOPARTICLES BY USING ACACIA CONCINNA PLANT EXTRACT

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ABSTRACT

Green Synthesis reduction method was employed to synthesise the silver nanoparticles (AgNPs) by aging the mixture of the aqueous extract of the Acacia concinna (AC) shikakai seeds and $AgNO_3$ solution at different time interval. Spherical shaped silver nanoparticles were characterized for their structural, optical and morphological properties. The Fourier transformer infrared (FTIR) spectroscopy reveals the presence of polyphenolic compounds in Acacia concinna leaves extract which acts as a reducing agent for the transformation of silver nitrate into silver nanoparticles. The x-ray diffraction (XRD) pattern substantiate the hexagonal system of silver nanoparticles, absence of impurities ascertains the higher order purity of the biosynthesized silver nanoparticles. The observed absorption peak at 419nm corresponds to surface plasmon resonance property of silver nanoparticles. The green synthesized silver nanoparticles with average size ranging below 40 nm imaged using (HRTEM) High Resolution Transmission Electron Microscopy.

Keywords: Silver Nitrate, Acacia Concinna Leaves, Nanotechnology, Silver Nanoparticles.

I INTRODUCTION

Nanotechnology plays a essential role in engineering and manipulating of the particles size at the nano rang from approximately 1-100 nm.[1] The significance of the nanotechnology in a multiplicity of fields such as biomedical science, environmental health, chemical industries, food, health care and feed, cosmetics, drug and gene delivery, electronics, mechanics, and space industries,[2] has been already reputable. It also has widely been used for the treatments of diabetes,[3] cancer,[4] allergy,[5] inflammation,[6] and infection,[7]etc. In current years the progress of the green synthesis of NPs is growing due to the number of its advantages greater than chemical synthesis methods such as single step, cleanness, mild synthesis reaction conditions and cost effectiveness.[8] Moreover, it is friendly for biomedical and food applications and this method eliminates the use of temperature, high pressure, energy and poisonous chemicals.[9, 10] the growing need of environmental friendly production of nanoparticles interacts of researchers to choose the green synthesis way for their fabrication. A variety of metallic nanoparticles,[11] because of their extraordinary properties over their bulk counter parts, used in the numbers of variety applications.[12] Green synthesis methods are more valuable than the other most popularly used chemical reduction methods, heat evaporation, photochemical reduction, electrochemical reduction, etc.[13] In biological method, the plant extract has been used as reducing surfactant and capping agent for the production of nanoparticles[14] due to their reducing properties.[15] Synthetic surfactants are used for different industrial purposes ranging from oil recovery, surface cleaning, froth floatation, etc. The artificial surfactants are not simply biodegradable and are reasonably costly, leaving surfactants extracted from microbial and plant as possible alternative sources to it. Plant surfactants on the other hand are cheaper and also appropriate for mass production compared to other chemical surfactants, but it has lesser wetting and surface tension properties compared to other chemical synthetic surfactant. The change in properties of the nanoparticles such as distribution, size and morphology of the nanoparticles are clearly experimental with biomaterial.[16] Various nanoparticles like copper, gold, iron, palladium, silver, zinc, quantum dots (CdS, ZnS,) are synthesized using variety of biochemical's. Silver nanoparticles are selected for the study among the above mentioned nanoparticles because of their several unbeatable properties such as optical, chemical, electronic, photo electro chemical, catalytic, magnetic, antibacterial, and antimicrobial activity. Silver nanoparticle like as antimicrobial agent can be used in medical applications such as coated capsules, band aids, blood collecting vessels, biological labelling, etc.[17] The silver nanoparticle is non-toxic for animal cells and highly toxic to bacterial cell, and other microorganisms (E-coli, Pseudomonas aeruginosa, Staphylococcus aureus, etc). Therefore, nanosilver is considered as a safe, effective and valuable bactericidal metal to be used for medical purpose.[18-20]

In present work, we used green synthesis method for silver nanoparticle, which are usually nontoxic, low expenditure, usage less amount of chemicals, environmental friendly workable at easy-going temperature and pressure conditions. Green synthesis of silver nanoparticles many reports have been published about the biogenesis of silver nanoparticles using several plant extracts like *Zea mays*[21], *Azadirachta indica* (Neem)[22], *Medicago sativa* (Alfa alfa)[23-24], *Aloevera*[25], *Emblica officinalis* (Amla)[26], *Capsicum annuum*[27], *Geranium* sp.[28-29], *Diopyros kaki*[30], *Magnolia kobus*[31] and *Coriandrum* sp.[32], etc. Different parts of plant like seed, flower, leaf, stem and skin of the fruits were previously used for the synthesis of AgNPs. Plant extract-coated nanoparticles, has medical advantageous, can be used in drugs synthesis and cosmetic applications.[33] this report mainly focuses on the synthesis of Silver nanoparticles. The objective of this study is to obtain AgNPs in presence of plant surfactants, Acacia concinna and also along with silver nanoparticles synthesized in-situ in acacia extract. *Acacia concinna* (AC) belongs to the family of Fabaceae plants, commonly known as shikakai. AC is a plant found in throughout India and has been served as an antibacterial and carminative drug in the traditional medicines.[34]AC consists of the lupeol, spinasterol, acacic acid, lactone, and the natural sugars glucose, arabinose and rhamnose. It also contains hexacosanol, spinasterone, oxalic acid, tartaric acid, citric acid, succinic acid, ascorbic acid, and the alkaloids calyctomine and nicotine. fatty acids, proteins, flavonoids and alkaloids that promotes antioxidant, antimicrobial, anti inflammatory and immune stimulant activity.[35] The current study focused on the synthesize of AgNPs, using the aqueous seeds extract of AC at different experimental conditions. Work will contribute in establishing the importance of plant sources and implementing green chemistry in synthesis of nano metal particles for the future research.

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II MATERIALS AND METHODS

2.1 Materials

Aqueous extract of *Acacia concinna* (shikakai) seeds was used to synthesize AgNPs, using the method mentioned elsewhere. Dry seeds were washed thoroughly with distilled water to make them free form dust particles and surface contamination and dried in sunlight for week long time.[36] Afterwards 5 gm dried seeds were soaked in to 100mL of pure deionised water for 24h. The extract was filtered using whatmann filter paper No.42. The filtrate was centrifuged at 2000 rpm for 30 min, and the suspended solid was used for further analysis.

2.2 Chemicals

Analytical grade chemicals from different suppliers such as $AgNO_3$ (silver nitrate, ≥99.0%, Merck) were used as purchased without any further purification. All solutions were prepared using deionised water (DIW).

2.3 Synthesis of Silver Nanoparticles

Silver nanoparticles were synthesized using the following procedure. Firstly, the aqueous 10^{-3} M $AgNO_3$ solution was prepared in 1000 mL of deionised water. Secondly, 2.5 mL of concentrated AC seeds extract was added drop wise into the 25 mL $AgNO_3$ solution that kept at room temperature for after 30minutes, 24h etc, respectively. During aging of $AgNO_3$ in AC extract, electron transfer from the solution to the Ag^+, was responsible for the electrical conduction in colloids and final conversion of Ag^+ ions into Ag nanoparticles. Reverse process occurs in oxidation process, where electrons get lost during reaction. The solution is colourless initially, but it turns to pale yellowish after 24 hours and then to dark reddish-brown colour after 48h and then it remains steady, indicating completion of reaction.

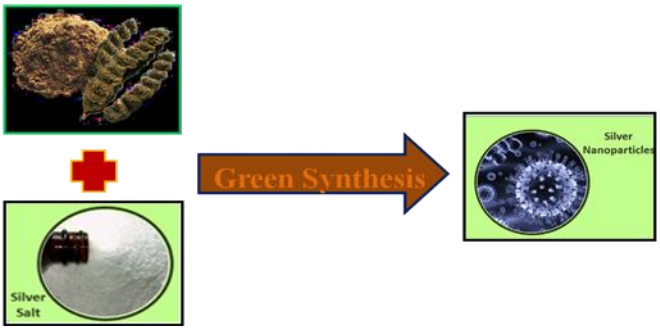

Fig.1 Synthetic Routes of Silver Nanoparticles

2.4 Optical Properties of Ag Nanoparticles

UV-Vis absorption spectrum was taken using a (LABINDIA UV-Visible 3000^+) spectrophotometer, where the cuvette path length was set to 1.0 cm and DIW was used for background subtraction. Ultraviolet-visible spectroscopy refers to the absorption of light in the UV-visible spectral region i.e. 200-800 nm that directly

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affects the perceived colour of the involved chemicals. In this region of the electromagnetic spectrum, molecules undergo electronic transitions. UV-VIS absorption spectra have been proven a sensitive tool to get the information about the formation of silver nanoparticles. Because an intense absorption peak of silver nanoparticles exhibited in certain region (419nm) that attributed to the surface plasmonic resonance (it describes the collective excitation of the conduction electrons in a metal) excitation. This technique can characterize the silver dendrites, colloids and surfaces.

2.5 pH Analysis

The pH was determined by using digital pH meter (Systronics; Model-ERMA). In making of the AgNPs process the pH of AC extract and $AgNO_3$ solution slowly became acidic with time that revealed in the form of the reduction in pH of studied sample.

2.6 XRD Analysis

As synthesized silver nanoparticles, were used for phase identification and the crystallinity check using powder XRD. The redispersed silver nanoparticles were kept in an oven at 60^{o}C for 24h in order to obtain the dry powdered AgNPs of high purity. The diffracted intensities were recorded from 10º to 70º of 2 theta angles. XRD analysis was performed using an X'Pert Pro X-ray diffractometer operated at a voltage of 40 kV and a current of 30 mA with Cu K_α radiation.

2.7 FTIR Analysis

In order to save solid biomass of the seed residue or synthesized compound, the resultant solution of 100 ml was centrifuged at 20,000 rpm for 30 min. This was followed by the drying of the AgNPs at 70^{o}C. As obtained sample of the synthesized silver nanoparticles, was subjected to FTTR analysis in the range of 400 to 4000 cm^{-1} using Bruker -Tensor Model FTIR spectrophotometer in the diffuse reflectance mode at a resolution of $4cm^{-1}$ in KBr pellets.

2.8 TEM Analysis

The TEM measurement was done using JEOL model 1200Ex microscope operated at an accelerating voltage of 80kV. Samples were prepared by placing 2-3 drops of the well dispersed Ag nanoparticles in alchohol on a 300-mesh carbon coated Cu grid (EM sciences) and allowing the liquid to disappear in air. For Ag nanoparticles the particle size distribution was based on randomly selected particles. The TEM image was taken with high resolution and MATLAB study gives the pixel depth of the image equal to bits and the image format as JPEG. The TEM Images have been taken at National Chemical Laboratory, Pune, India.

III RESULTS AND DISCUSSION

3.1 Optical Properties with UV Visible Spectroscopy

Green synthesis of the silver nanoparticles was confirmed on the basis of the colour change in solution during the reaction (transparent to dark reddish-brown) and change in pH (7 to 5.8) of solutions. The intensity of colour interprets the degree of bio-reduction of AgNPs due to addition of plant reducing agent. It is happening to

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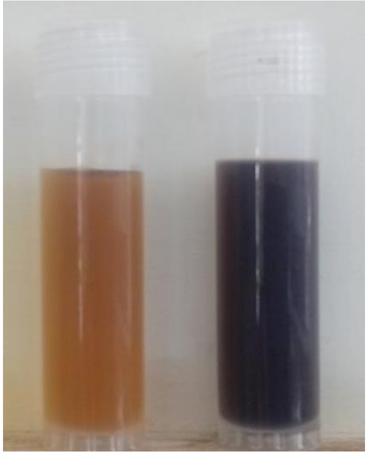
change the colour (from transparent to deep reddish brown) due to reduction of silver ion, which may be the indication of formation silver nanoparticles, as shown in below Fig.2 Colour of silver colloid is attributed to surface plasmon resonance (SPR) phenomena arising due to the collective oscillation of the free conduction electrons induced by an interacting electromagnetic field.[37] Therefore, the formation of AgNPs was primarily recorded on the basis of the surface plasmonic resonance first exitonic peak in UV-visible spectroscopy.



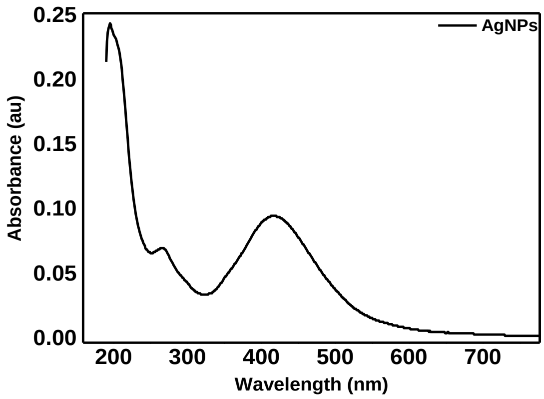


Fig.2 Synthesis of AC-Ag NPs at different aging time such as after 30 minutes and 24h, UV-Visible spectra of AgNPs

Surface plasmon resonance phenomena, attributed to the first exitonic peak at different wavelength for different nanoparticles solution. It is reported in the literature[38] that typical AgNPs shows the characteristic SPR at the wavelength in the range of 380-440nm. Fig 2 exhibited the SPR peak at the wavelength of 419 nm, which verified the presence of AgNPs in the solution. The SPR absorbance is sensitive to the nature, concentration; size and shape of the particles present in the solution and also depends upon their inner particle distance and the surrounding media.

3.2 Analysis of nanoparticle size by SEM and TEM:

The absorption spectra give solid evidence of nanoparticle formation and their growth kinetics, the size of the resultant particles were elucidated with the assist of SEM (Scanning electronic microscopy). After 24 hours, the analysis of Ag nanoparticle formed by the reduction $AgNO_3$ by acacia was done using Nano size particles, which gave the average particle size in the range of ~50nm.

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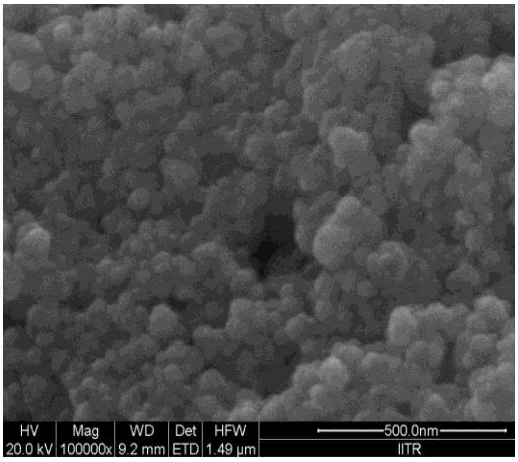

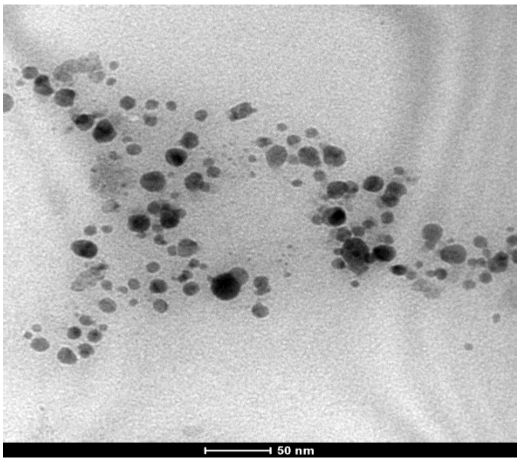

Fig.3 SEM images of AgNPs (A) **TEM images of Ag NPs (B)**

Shows the Fig. 3 (B) TEM images of silver nanoparticle synthesised in 1mM concentration Acaccia medium, which acts as both reducing as well as capping agent. From the figure it is clear that the silver nanoparticles are almost spherical and uniform in size. It can be clearly seen from the above figure that silver nanoparticle has a tendancy towards agglomeration, so it is difficult to get separate discrete silver image.

3.3 XRD Analysis

The XRD pattern of silver nanoparticles is shown in the figure represents the crystalline nature and purity of the silver nanoparticles. The peaks totally formed were coherent with standard data for silver (JCPDS 87 – 0720).

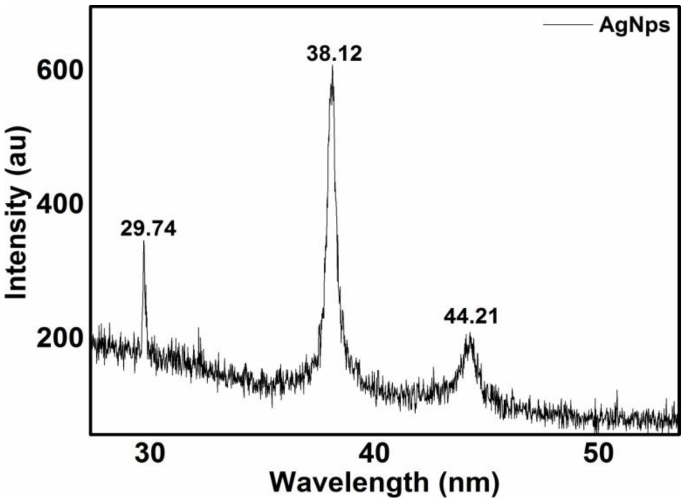

Fig.4 XRD plot of synthesized AgNPs

The XRD patterns of the powdered silver nanoparticles taken using CuKα radiation (λ= 1.5418 Å) gives major peaks at 38.09°, 44.12° and 64.37° that can be indexed to the (111), (200) and (220) planes of a cubic crystal system (a=4.0686Å) and corresponding well with the standard JCPDS file No.04- 0783 of pure silver metal.

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Broadness in patterns reflected the small particle size of the AgNPs and spin plasmonic resonance phenomena (Figure-4). The Debye-Scherrer formula (Dp = Kλ /β1/2 cosθ with K=0-89, λ=wavelength of the radiation, β=full width at half maximum, θ = incident angle of radiation beam, Dp=particle size) was also used for calculation of the particle size i.e. ~ 30-50 nm.

3.4 FTIR Analysis

FTIR investigation also gives a series of peaks for the AgNPs sample belong to the sequence of the phytochemicals present in the sample that acts as a reducing agent (Figure-5). FTIR investigations on the biosynthesized AgNPs was passed out in the range of 4000-500 cm-1 wave number to make out the most likely interactions between the Ag ions and phytochemicals present in AC seeds extract that used to stabilize the AgNPs. Figure-6 definite the existence of plant peptides visible due to the bending formed by amide bonds. AgNPs exhibits prominent peaks at 3388.73, 1646.45 and 1763.84 cm-1, representing the contribution of N-H stretching vibrations, N-H bending vibrations and C=O stretching vibrations that refers to the AC-AgNPs aggregates. The C-N and C-O-C stretching vibration suggested the existence of a lot of phytochemicals on the surface of the NPs.

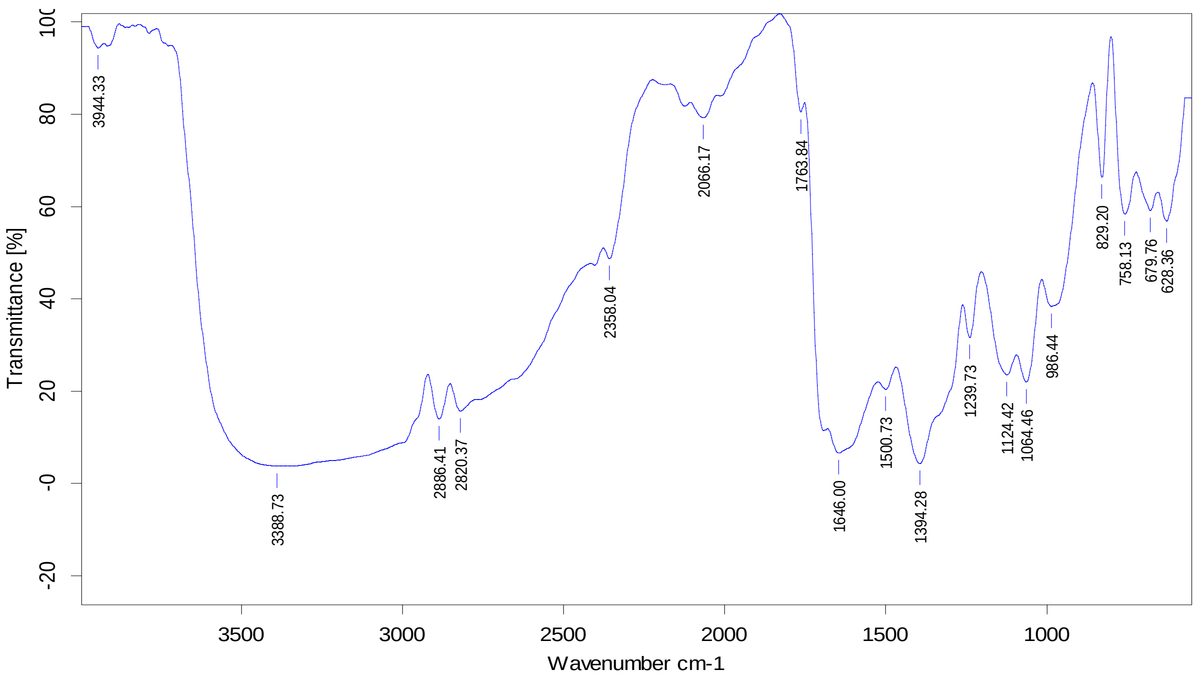

Fig.5 FTIR spectra of synthesized AgNPs

Green synthesis method of AgNPs is a supplementary way to chemical and physical synthesis methods and it is used to reduce metal ion of silver to AgNPs nano. Green Synthesis of the silver nanoparticles has been earlier reported using plant's seed extracts as well as different kind of plant body parts. The phytochemicals present in the seeds extract, which are responsible for reducing the silver salts and producing stable AgNPs without using any capping agent. Additionally, the antimicrobial, antifungal and antioxidant activities of the nanoparticles are also enhanced by the existence of the phytochemicals.

lV. CONCLUSION

In this current study we have designed and an ecofriendly synthesis of AgNPs using AC seed extracts via biological process, in which AC seed extract act as a reducing agent. In green synthesis method, AC Seeds extract are responsible for reduction of silver metal to nano size. The synthesized silver nanoparticles were subjected to advance analysis technique such as a TEM, XRD, Uv-Vis spectroscopy and FTIR in order to characterize them. The creation of nanoparticles as well as size was also confirmed by UV-Vis spectroscopy; while crystalline properties of the nanoparticles and average particle size is investigated by XRD analysis and the TEM image show visibly the formation of nanoparticles. To the best of our information this is the unique information of the explanation that lead the path to produce the AC mediated AgNPs. These research works are further extended to investigate the synthesis and optical properties of AgNPs formed by the other plant leaves' extracts.

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