Biosynthesized Silver Nanoparticles as Antimicrobial Finish over Cotton Fabric

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Abstract
This paper describes the use of the bioreduction process to synthesize silver nanoparticles (AgNPs). The aqueous extract of Tabernaemontana divaricta (pinwheel flower) and silver nitrate, which serve as a reducing agent and precursor salt, respectively, have been used in the synthesis of AgNPs. Using scanning electron microscopy and UV-visible spectroscopy, synthesized AgNPs have been characterized.The optical characteristics of AgNPs were assessed using UV-visible spectroscopy by means of the Surface Plasmon resonance (SPR) peak. This SPR peak appeared at 410 nm and suggested an average particle size of 40 to 60 nm. Using SEM examination, the AgNPs' morphology was investigated. Using the Continuous Pad-Dry-Cure technique, these synthesized AgNPs were applied to cotton fabric. AgNP-deposited fabricswere then evaluated in terms of antibacterial activity and wash durability. The developed AgNP-deposited cotton fabric showed excellent antibacterial activity up to 10 numbers of soap washes.

Key words:
Silver Nanoparticles, Tabernaemontana divaricta (Pinwheel) flower extract, bioreduction, cotton, antibacterial, wash durable.

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1.0 Introduction

The study of structures and substances with sizes between one and one hundred nanometres is known as nanotechnology. In nanometer size, structures have different properties (such as electrical, optical, thermal, and mechanical) than in macroscale or bulk scale [1,2]. In nanomaterials, properties differ based on the way molecules and atoms assemble on the nanoscale into larger structures based on quantum mechanical effects [2,3]. Nanotechnology can be usedin various fields such as nano-medicines [4,5], biomedical applications (to treat diseases or prevent health issues) [6], industrial applications (including construction materials, military goods, etc[7,8] , water purification, effluent treatment, etc [8]. Various physical and chemical methods can be used to prepare nanoscale materials having different sizes and shapes [9]. However high cost and involvement of toxic chemicals in the synthesis process limit the advantages and uses of these nanoparticles [10 ,11]. Biological procedures can overcome this problem by adopting the easy, nontoxic and environment-friendly methodology [12, 13]. Plant extract-based nanoparticles synthesis have drawn interest recently due to their ease of use and affordability. Sukumaran S et al. reported the antibacterial properties of Peltophorumpterocarpum (DC) flower extract [14]. Hassan Mahmoodi Esfanddarani et al. attained the biosynthesis of metal silver nanoparticles by using Malva sylvestris flower extract as reducing agent and nanoparticle antibacterial properties tested against Escherichia coli (E. coli), Staphylococcus aureus (S. aureus), Streptococcus pyogenes (S. pyogenes) using the disk diffusion assay [15]. The preparation of biosynthesized silver nanoparticles by lemon fruit extract and their potential application as antifungal finishes was explored by Vankar P. et al [16].

In this work, the water-based extract of Tabernaemontana divaricta flower (pinwheel flower) was utilized in the preparation of the silver nanoparticles. This extract acts as both an encapsulating agent and a reducing agent. UV-visible spectroscopy and SEM analysis were used to determine the size of synthesised nanoparticles. Prepared biosynthesized AgNPs applied on cotton fabric through pad-dry-cure process, to provide a wash durable antimicrobial finish. The treated textiles have shown remarkable antibacterial qualities. As far as we know, there haven't been any reports of using Tabernaemontana divaricta (pinwheel) flower extract for AgNP synthesis. Accordingly in this work synthesis, characterization, and application of AgNPs are reported using Tabernaemontana divaricta-(Pinwheel) Flowers extract.

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2.0 Experimental

2.1 Materials:

Silver Nitrate was used as the initial silver ion source in the synthesis and it was procured from Merck India PVT Ltd. Tabernaemontana divaricta-Pinwheel Flowers(Figure 1) were freshly collected from the campus of Bombay Textile Research Association (BTRA) Mumbai, Maharashtra, India from the Garden area. The synthesis used this flower extract as a reducing agent.

Figure.1 Tabernaemontana divaricta flower (pinwheel flower)

2.2 Preparation of flower extract

In a 250 ml glass beaker about 18 to 20 numbers of thoroughly washed pinwheel flowers(weighing approximately 4.5g), were taken and heated(up to boiling) for 10 minutes with hundred milliliters of deionized water. The resulting solution was then used as a bio extract for synthesis after being filtered via filter paper number 42.

2.3 Qualitative screening for phytochemicals

Standard techniques were used to qualitatively screen flower extract for the presence of alkaloids, phenolic chemicals, flavonoids, terpenoids, and tannins [12, 17]. Two millilitres of flower extract and one millilitre of Wagner's reagent were placed in a test tube for the purpose of detecting alkaloids. Alkaloids are present when a reddish-brown precipitate forms after adding Wagner's reagent. By adding a few drops of a 1% sodium hydroxide solution, flavonoids were found. The presence of flavonoids is indicated by the colour turning yellow immediately. Presence or absence of Tannins and Terpenoids were analysed using the ferric chloride test and Salkowaski test respectively. The formation of greenish black precipitate with ferric chloride addition in extract indicates the presence of Tannins. After carefully mixing 5 milliliters of extract with 2 millilitres of chloroform and 3 milliliters of strong sulphuric acid, the development of reddish-brown color indicates the presence of terpenoids. Presence of phenolic compounds was identified by ferric chloride test.

2.4 Synthesis of silver nanoparticles using bio extract:

With continuous stirring, five millilitres of the produced pinwheel flower extract were added to fifty millilitres of 0.001 millilitres of $AgNO3$ solution. To optimize the effects of the silver nanoparticles, the entire solution was kept at 90°C in a dark condition in water bath .The colour of the silver nitrate solution gradually changes from colourless to yellowish brown as the formation of silver nanoparticles proceeds (Figure 2).

Fig.2 Biosynthesized AgNPs using Pinwheel flower extract

2.5 Application of bio-synthesised silver nanoparticles to cotton fabric

The 3dip-3nip method is used to apply biosynthesized silver nanoparticles to cotton fabric, which are then dried and cured. Fabrics deposited with these nanoparticles were examined further for their antibacterial properties and washability. To analyse the wash durability of finished fabric, the ISO 6330 washing method is used and after washing ,durability measured in terms of antimicrobial activity. Antimicrobial activity of AgNP-deposited fabric was measured using AATCC-100 standard test method against staphylococcus aureus (SA) and Klebsiella pneumonia (Kp).

3.0 Characterisation of synthesized silver nanoparticles

3.1 Optical properties

The Schimadzu UV-visible Spectrophotometer model (UV-1800) was used to assess the optical characteristics of silver nanoparticles in the 200–800 nm range by observing the appearance of a surface resonance peak.

3.2 FTIR Investigation

The functional groups responsible in reducing Ag^+ ions to AgNPs were identified using FTIR analysis. The Pinwheel flower extract and related AgNPs' FTIR spectra were measured in the 600–4000 cm-1 range using the Perkin Elmer Miracle ATR-spectrum-2 FTIR spectrometer.

3.3 SEM Analysis

The size and shape of the generated silver nanoparticles were examined using a scanning electron microscope. The JEOL JSM IT 200 was used to capture scanning electron micrographs in order to examine the surface morphology of the biosynthesised AgNPs.

4. Results and discussion

4.1 Phytochemical Screening Evaluation

Table 1 displays the findings of the initial phytochemical screening of the pinwheel extract, which showed that phenols, flavonoids, tannis, and alkaloids were present but that terpenoids and saponins were not. The presence of alkaloids, flavonoids and phenolics phytonutrients in pinwheel flower extract is thought to be responsible for the bioreduction process. To study that FTIR investigation was carried out to see the involvement of related functional group from extract in AgNP synthesis.

Table 1 Qualitative Screening Results of Pinwheel Flower Extract

Alkaloids	Tannis	Flavonoids	Phenols	Terpenoids	Saponins
√	√	√	√	×	×

4.2 UV-Visible Spectroscopy:

It is commonly known that the activation of surface Plasmon vibration in silver nanoparticles causes them to appear yellowish brown in aqueous solution [18]. The colour of AgNPs produced from flower extract is shown in Fig. 2. UV-visible spectroscopy can be used to analyse the size and form of the nanoparticles in aqueous suspension. The UV-Vis absorption spectra of the biosynthesized AgNPs with 2, 3, 4, and 24 hours reaction time is displayed in Figure 3.

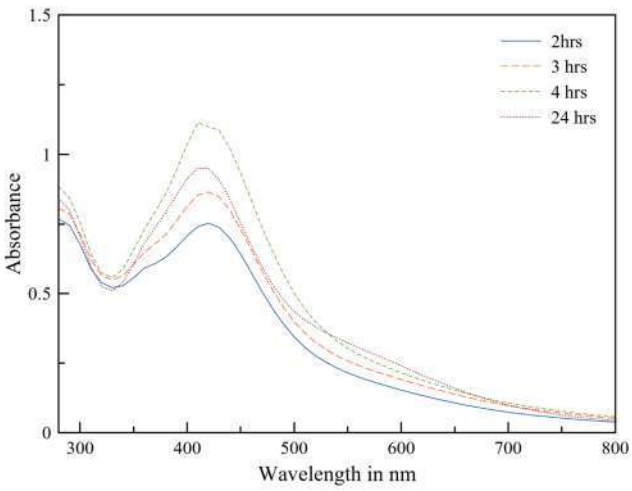


Figure 3 : UV-Visible spectrum of biosynthesized AgNPs with reaction duration of 2, 3, 4 and 24 hours

It reveals that the generation of AgNPs started within 2 hours. The maximum absorption peak of synthesized silver nanoparticles is achieved at 410 nm which confirms the formation of AgNPs in the size range of 60-80nm, based on published literatures [18]. UV-visible spectroscopy was used to verify the stability of the synthesised silver nanoparticles for a full day (up to 24 hours). This method confirmed a consistent SPR peak at 410 nm, although with a lower absorbance.

4.3 FTIR Analysis:

FTIR analysis was used to evaluate the data related to the chemical alteration of the functional group involved in bio reduction. Figure 4 displays the FTIR absorption spectra of the pinwheel flower extract and the biosynthesized AgNPs. The Pinwheel flower extract shows the strongest peak at 3340cm-1, 1637 cm-1 and 1043 cm-1 attributed to OH group, olefinic band and primary and secondary alcohol functionalities respectively. In case of AgNP synthesized using flower extract similar pattern of FTIR spectrum was achieved. However, it was discovered that the positioning and peak area of corresponding peaks slightly changed. FTIR Peaks at 3340, 1637, and 1043 corresponding OH, C=C, and C-O observed in flower extract were found to get narrowed and shifted to higher frequency regions. It confirms the involvement of OH and C-O groups from flower extract in the reduction of ionic silver to nanosilver. This further supports the hypothesis that the reduction and stabilisation of synthesised silver nanoparticles are caused by phytonutrients with OH and C-O functional groups in their structures, such as flavonoids, alkaloids, and phenolic chemicals.

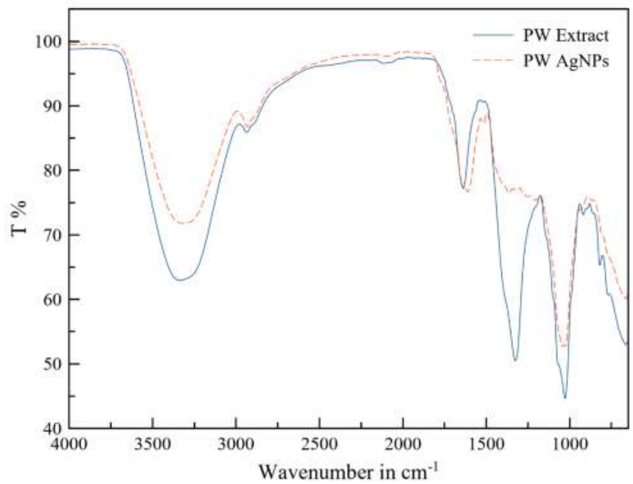
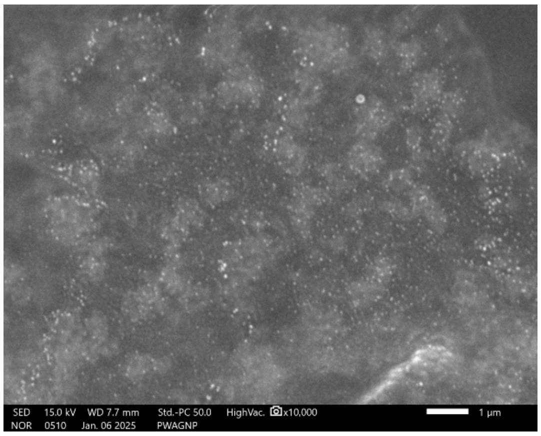


Figure 4: FTIR spectra between Tabernaemontana divaricta flower (pinwheel flower) extract and synthesized silver nanoparticles

AgNP generation occurs in high quantities and is smaller than 100 nm, as shown by the scanning electron micrograph (Fig. 5) of silver nanoparticles made using pinwheel flower extract. The particle size of the synthesized silver nanoparticles fall between 30 and 120 nm, with the largest particles measuring between 50 – 90nm, almost matching the values reported through UV-visible spectroscopy investigation.



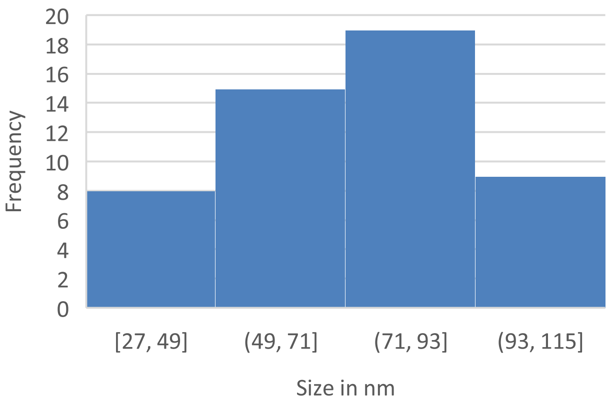


Figure 5: Scanning electron microscope image and size distribution histogram of synthesized silver nanoparticles

In comparison to their salt counterparts, silver nanoparticles have strong antibacterial properties due to their large surface area, which enables improved contact with microorganisms. Using the AATCC 100 standard test method, the antibacterial activity of textiles deposited with AgNPs was evaluated against Staphylococcus aureus and Klebsiella pneumoniae. AgNP-deposited fabric demonstrated outstanding antibacterial activity against both Gram-positive (Staphylococcus aureus) and Gram-negative (Klebsiella pneumoniae) bacteria; that is after 24 hours, the antibacterial rate increased to 99.80% against Klebsiella pneumoniae and 99.71% against Staphylococcus aureus. The cause might be that the small size of AgNP particles causes them to firmly adhere to the surface of bacterial cells, disrupting the cell membrane and ultimately killing the bacteria [19]. These biosynthesized AgNP-deposited samples then assessed for wash durability in terms of change of its related antimicrobial properties after 10 numbers of soap washes. After ten washes, antimicrobial properties of same fabric is 99.29 % for gram negative and for gram-positive, it is 98.90%. Excellent antibacterial activity and wash durability in cotton fabric are the results of the overall homogeneous deposition of biosynthesised AgNPs on cotton fabric, making them appropriate for commercial applications.

5. Conclusion

Using pinwheel flower extract as the reducing agent, a straightforward bioreduction synthesis technique for AgNP synthesis has been reported. These synthesized nanoparticles were examined using SEM, FTIR, and UV-visible spectroscopy. The particle size of AgNPs was theoretically obtained as 60 – 80nm by Uv-visible spectroscopy and SEM studies. These biosynthesized AgNPS were then applied over cotton fabric using a simple pad-dry-cure process and antibacterial activity, and wash durability were evaluated. AgNP deposited fabric showed excellent antimicrobial activity and wash durability against Klepsillaknumoniae and Staphylococcus aureus. This work can be further extended to the practical application of biosynthesized AgNPs in the pharmaceutical industry and wastewater treatment.

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