Biosynthesis of Silver Nanoparticles using Histiopteris Incisa leaf extract and their Characterization and Thermophysical study

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Abstract:
Nanofluids are nanoparticle suspensions in base fluids such as water oil. Nanoparticles can alter the thermophysical, mechanical, and other characteristics of base fluids. These nanofluids exhibit significantly greater thermal properties than conventional working fluids, such as increased thermal conductivity by increasing the temperature and percentage volume in the base fluid. The significance of silver nanoparticles in enhancing coolant characteristics in heat transfer applications has been established. One of the synthesis techniques is the biosynthesis technique, which is environmentally friendly, energy-efficient, large-scale, and low-cost. The biosynthesis of silver nanoparticles (AgNPs) utilizing Histiopteris incisa (water fern) leaf extract was the focus of this study. The silver nanoparticles were prepared at room temperature at different concentrations, such as 1 mM, 3 mM, and 5 mM, using a stirrer for 1 h. Plant extract was combined with different concentrations of silver nitrate solutions and analyzed for nanoparticles using UV-Vis spectroscopy. High-resolution transmission electron microscopy (HRTEM), X-ray Powder Diffraction (XRD), Dynamic light scattering (DLS), Zeta potential, and Fourier Transform Infrared Spectroscopy (FT-IR) were employed to analyse the silver nanoparticles. The AgNPs synthesized were spherical, having particle sizes ranging from 98 nm to 69 nm. Thermal conductivity increased with increasing temperature and volume concentration.

Keywords: Silver Nanoparticles (AgNPs), Histiopteris Incisa leaf extract, Biosynthesis, Thermal conductivity, Heat transfer
1. Introduction:

Transferring thermal energy from one stream to another by adding, removing, or transferring between them is a difficult task for industrial requirements in a variety of process industries. Since global energy consumption is increasing, these processes provide a platform for energy retrieval. Most researchers are now fascinated by the application of nanotechnology to heat transfer. Water, oil, and other common base fluids have been used in heat exchangers for many years. As Table 1 shows [1–6], these fluids possess lower thermal conductivity than metals and metal oxides. Because of the existence of nano-sized particles (smaller than 100 nm), nanoscience technology has generated a revolutionary heat transfer fluid known as "nanofluid" that has been prepared, and its thermal properties are superior to those of standard base fluids [7]. As nanofluids have better thermal characteristics than conventional or base fluids, they can be employed as a covert alternative to the existing coolants [8]. Adding nanoparticles (NPs) to a base fluid boosts its conductivity and film coefficient significantly. As a result, utilizing nanofluids in heat exchangers to exchange considerable energy is a cost-effective option than using conventional coolants.

In recent decades, substantial progress has been made in the synthesis and characterization of nanoparticles (NPs) using various methods to achieve diverse sizes and morphologies. The imperative now is to advance manufacturing processes for cleaner, safer, and more intelligent goods, particularly in agriculture and medicine. The primary objective of NP synthesis is to be eco-friendly, energy-efficient, and cost-effective. Utilizing biological entities like plants, bacteria, algae, yeast, fungi, and waste materials, which serve as bio-laboratories due to their composition of biomolecules, presents an efficient method to prepare nanoscale metals and metal oxides with distinct shapes. This green technology approach not only aligns with environmental sustainability but also addresses the need for biocompatible and non-toxic nanoparticles, making them applicable in diverse fields. Overall, the emphasis on green synthesis is crucial in contemporary research and applications, fostering the development of nanomaterials with reduced ecological impact and heightened compatibility for safe utilization in various technological and biomedical sectors [9].
In recent years, dispersed NPs in base fluids have gained increased attention in the field of heat transfer to improve heat transfer rates. Previous works have mainly discussed the advancement of nanofluids in experimental and theoretical studies, and they are concerned with thermal and physical characteristics, conductivity, or convective coefficient [10-11]. Several chemical, physical, and biological techniques are used to produce and synthesise NPs. Among them, biological methods, which use plants, bacteria, yeast, waste materials, and so on, for synthesising specific NPs, have an upper hand over physical and chemical methods as they are eco-friendly, conserve energy, and have mass production [12-14]. The biological approach of synthesizing NPs with extraordinary shapes and sizes, such as silver (Ag) NPs with extremely small diameter and Ag+ ion in the form of an aqueous solution, has been established, and the newly generated NPs are non-hazardous [15-18]. Synthesized NPs, which are plant extracts from diverse sections, have also been employed as reducing, stabilising, and capping agents.

Throughout the decades, two approaches have been used: the top-down approach and the bottom-up approach. In the top-down technique, bulk materials are turned into small particles by physical and chemical conversion, whereas in the bottom-up approach, extremely small particles such as atoms are assembled individually to form NPs [19]. However, conventional procedures include harmful physical and chemical processes, whereas the green synthesis method is non-toxic and follows the bottom-up strategy.

In this work, synthesis of AgNPs from various components of plant extracts and microorganisms (Figure 1). It has various ecological and non-toxic benefits, and it is extensively employed in many industrial and medicinal industries because no chemicals are used in the production of the NPs. The NPs synthesis technique entailed collecting a plant component from any place of interest and thoroughly washing it with distilled water; dust particles were eliminated using distilled water. The cleaned, fresh plant pieces were dried in the shade for a week before being crushed using a household grinder. 10 g of the dry powder was heated in 100 mL of deionized distilled water. This was used to prepare plant extracts, after which the mixture was properly filtered. The silver nitrate solution was formed by reducing pure silver ions to silver nanoparticles, which could then be analyzed using UV-visible spectroscopy.
2. Materials and methods

2.1 Plants and Chemicals

Fresh Histiopteris \textit{incisa} leaves in good physical condition were taken from terrain near a tea estate in Udagamandalam, Nilgiris district, India. For the preparation of silver nanoparticles (AgNPs), silver nitrate (AgNO$_3$) [LR Grade, LOBE CHEMIE] was used as received without any purification. The glassware used in this experiment was thoroughly washed in distilled water and dried. In this experiment, distilled water was employed. All of the tests were carried out in triplicate.

2.2 Leaf extract preparation

To eliminate any dust particles, the newly gathered leaves of Histiopteris incisa were carefully washed with deionized water. Furthermore, distilled water was used to rinse the leaves, which were then dried in the shade for one week before being milled into a fine powder. 10 g of leaves powder was suspended in a conical flask containing 100 mL distilled water. The suspension was heated at 40°C for 30–40 min under continuous magnetic stirring. The leaf extract was then poured and filtered using Whatmann's filter paper, and the filtrate solution was collected and kept at 4 °C in an Erlenmeyer flask for later use. The extract plays a pivotal role in the synthesis process of nanoparticles, particularly in green synthesis methods. Acting as a bio-reducing and stabilizing agent, the extract, derived from various biological sources such as plants, bacteria, or fungi, facilitates the reduction of metal ions to form nanoparticles. The biomolecules present in the extract, such as phytochemicals or enzymes, act as reducing agents, converting metal precursors into nanoparticles. Additionally, these biomolecules may serve as capping agents, preventing the agglomeration or undesired growth of nanoparticles and contributing to their stability. The unique composition of the extract imparts a green and sustainable aspect to the synthesis, avoiding the use of harsh chemicals and promoting environmentally friendly practices. Overall, the extract not only acts as a bioreactor for nanoparticle formation but also influences the size, shape, and surface properties of the nanoparticles produced [20].

2.3 Preparation of silver nanoparticles (AgNPs)

AgNO$_3$ solutions of concentrations 1, 3, and 5 mM were prepared and utilized for the synthesis of AgNPs. 90 mL of AgNO$_3$ solution of 1 mM concentration was taken in a beaker and 10 mL of \textit{Histiopteris incisa} aqueous extract under magnetic stirring was added to it. A
mixture was continuously stirred until the color changed to yellowish-brown, illustrated in. Similar procedure was repeated to prepared AgNPs with AgNO$_3$ solutions of 3 and 5 mM concentrations. \textbf{(Figure 2)}.

The study elucidates the reduction potential of both extracts and metals, showcasing their crucial role in nanoparticle synthesis. The interplay between the extracts and metals is explored, emphasizing their contribution to the reduction process. This investigation not only unveils the mechanisms governing nanoparticle formation but also provides valuable insights into optimizing the reduction potential for enhanced synthesis efficiency. The quantification and analysis of reduction potentials offer a comprehensive understanding of the intricate dynamics involved in the synthesis of nanoparticles, contributing to the advancement of nanoparticle science and technology [21].

\section*{2.4 Characterization of Silver nanoparticles}

A combination of UV-Vis spectroscopy with Surface Plasmon Resonance (SPR) in the 300-800 nm range was used to investigate Ag$^+$ reduction and the formation of AgNPs.

Fourier transform infra-red (FT-IR) spectroscopy was also used to determine the functional biomolecules of Ag$^+$ ions. The XRD technique was used to examine the phase and particle size. Furthermore, HRTEM is an important tool for investigating AgNPs in terms of particle size and form. Smaller colloidal particles are described using DLS analysis. This approach is commonly used in nanomaterial size distribution and particle sizing. Furthermore, the Zeta potential was measured to determine the stability of AgNPs. The conductivity of water-based AgNPs was determined using the KD2 pro thermal analyzer (transient hot-wire technique).

\section*{3. Results and Discussion}

\subsection*{3.1 Visual and UV-Vis analysis}

Histiopteris incisa leaf extract was visually examined when it was mixed with colorless AgNO$_3$ solution of varied concentrations (1-5 mM). According to the literature, a solution of yellowish-brown colour is observed as proof of AgNPs synthesis [22]. The synthesised AgNPs in the mix were verified by UV-Vis analysis for the aforementioned concentrations after the completion of i.e after an hour \textbf{(Figure 3)}. As the metabolites present in Histiopteris incisa leaf extract function as the reducing, capping, and stabilising agents in the process of creating
the AgNPs, the colour change occurred owing to the reaction of Ag\(^+\) into Ag\(^0\) [23]. When scanned across wavelengths spanning from 300 to 800 nm, the strong peaks of each combination were observed at 427, 429, and 425 nm for 1, 3, and 5 mM concentrations, respectively. The detected peak was due to Surface Plasmon Resonance (SPR), and a single and broad peak at each concentration implies that the AgNPs were modest in size [24, 25].

3.2 Fourier Transform Infrared (FT-IR) spectroscopy analysis

With varied doses of AgNO\(_3\), such as 1 mM, 3 mM, and 5 mM, there are numerous physiologically plausible potential functional groups that serve as reducing, capping, and stabilising agents (Figure 4). This investigation was conducted on biologically synthesized AgNPs to identify NPs in the wave number range of 4000-500 cm\(^{-1}\) at 4 cm\(^{-1}\) resolution that were recognised by the FT-IR analyzer peaks. In all concentrations, the study gives almost the same value range. At 1 mM concentration, the FT-IR spectra revealed strong absorption bands at 3367, 2980, 2887, 1641, and 721 cm\(^{-1}\) [26]. O\(-\)H was assigned the U-shaped absorption band at 3367 cm\(^{-1}\), which matched the O\(-\)H stretching of the alcohol group (Intermolecular Bonded). Sharp bands of medium intensity, allocated at 2980 and 2887 cm\(^{-1}\), were related to the C-H stretching alkane group. C=C was allocated the medium sharp band at 1641 cm\(^{-1}\), which corresponds to the C=C stretching of alkane group (distributed). C=C was assigned the sharp strong band at 721 cm\(^{-1}\), which matched the C=C bending alkane group (distributed). For a concentration of 3 mM, it shows the significant absorption bands at 3369, 2980, 2887, 1641, and 725 cm\(^{-1}\). O\(-\)H was assigned the U-shaped absorption band at 3369 cm\(^{-1}\), which matched the O\(-\)H stretching of the alcohol group (Intermolecular Bonded). C-H was allocated medium-sharp bands at 2980 cm\(^{-1}\) and 2887 cm\(^{-1}\), which related to the C-H stretching alkane group. C=C was allocated the medium sharp band at 1641 cm\(^{-1}\), which corresponds to the C=C stretching alkane group (distributed). C=C was assigned the sharp strong band at 725 cm\(^{-1}\), which matched the C=C bending alkane group (distributed). It shows that the strong absorption bands for a 5 mM concentration at 3346, 2980, 2887, 1641, and 732 cm\(^{-1}\). O\(-\)H was assigned the U-shaped absorption band at 3369 cm\(^{-1}\), which matched the O\(-\)H stretching of the alcohol group (Intermolecular Bonded). C-H was allocated the medium sharp bands at 2980 cm\(^{-1}\) and 2887 cm\(^{-1}\), which related to the C-H stretching alkane group. C=C was allocated the medium sharp band at 1641 cm\(^{-1}\), which corresponds to the C=C stretching alkane group (distributed). C=C was assigned the sharp strong band at 732 cm\(^{-1}\), which matched the C=C bending alkane group (distributed). These band values show that the
leaf extract's functional group was responsible for the synthesis, capping, and stabilisation of nanoparticles.

3.3 X-ray Powder Diffraction (XRD) studies
The XRD technique was used to validate the phase and crystallinity of AgNPs produced from Histiopteris incisa leaf extract. XRD analysis of AgNPs reveals a crystalline structure corresponding to the peaks (111), (200), (220), and (311), at 1, 3, and 5 mM concentrations (Figure 5 a, b, c). The broadening section's Bragg's peaks show the development of NPs. AgNPs display a similar diffraction characteristic in XRD, with intensity peaks at 20 of 37°, 46°, 68°, and 76°, respectively [27]. The crystalline character of biosynthesized AgNPs is demonstrated by the greatest intensity peak (111), which has a fine full width at half maximum. The lattice parameter of silver nanoparticles is face-centered cubic (FCC) crystalline structure. The significant characteristic peak intensities of the AgNPs suggest that the NPs are pure, as there are no extra diffraction peaks [28].

3.4 Dynamic Light Scattering (DLS) and Zeta potential analysis
The particle size distribution of synthesized AgNPs were determined using DLS analysis. Figure 6 depicts the DLS particle size distribution plot spanning from 0.1 to 10000 nm. The average particle size distribution of AgNPs at 1 mM concentration concentration, the particle size distribution was 98.01 nm (Figure 6 a), with a polydispersiveindex (PDI) of 0.158. Similarly, the particle size distribution at 3 mM (Figure 6 b) was 71.74 nm with PDI- of 0.352, and at 5 mM (Figure 6 c) was 69.49 with PDI-0.355 [29]. According to this research, when the concentration was raised, the particle size distribution was good. PDI was used to estimate the average homogeneity of a particle solution as well as the effectiveness of NPsaggregation and particle structural alterations throughout the sample. The Zeta potential study showed thatAgNPs stability and larger negative values presented by particle surface charges, with values of 20 mV, -31 mV, and -14 mV for 1 mM, 3 mM, and 5 mM, respectively (Figure 6d), suggesting stronger AgNPs stability. The findings suggested that AgNPs had stabilized [30].

3.5 High-Resolution Transmission Electron Microscopy (HRTEM) analysis
To comprehend the morphology and particle size of AgNPs, HRTEM imaging analysis with different magnifications was carried out. The photos clearly reveal that the AgNPs were spherical, with just a few particles of varied sizes and very few agglomerations. The HRTEM
images (Figure 7 a, b, c) showed particles with diameters ranging from 20 to 50 nm, with an average particle size of around 25 nm. These results are consistent when compared to previous researches in the literature [31]. The crystalline form of the AgNPs was explained by the area picked in the electron diffraction pattern (Figure 7d). The particles' round brilliant spots suggest that they are solitary crystalline.

3.6 Thermal Conductivity measurement

Thermal conductivity is a significant feature in heat transfer applications for enhancing the rate of heat transmission. Base fluid, surfactant, percent volume, temperature, particle size, particle shape, pH, and particle composition have all been shown to impact thermal conductivity. The KD2 pro thermal analyzer was utilized in this investigation to test the thermal conductivity of biosynthesized water-based AgNPs at 1, 3, and 5 mM concentrations. Thermal conductivity is calculated using the Transient Hot-Wire (THW) equipment. The thermal conductivity of the NPs was measured by varying the temperature from 30 to 70 °C, and it was discovered that raising the temperature increased the conductivity (Figure 8a). The conductivity was then evaluated by increasing the volume concentrations by adjusting the volume from 0.5 to 2 %. Figure 8 (b) illustrates that conductivity rises with increasing volume and temperature [32-33].

4. Application

We investigate the heat transfer and fluid flow characteristics of a helically coiled heat exchanger (Figure 9) employing a coiled pipe with two loops: the outer coil circulates hot water, while the inner loop circulates Biosynthesized aqua-based silver nanofluids [34]. The laminar flow conditions are maintained throughout the experiment. The experimental setup includes a 50-litre hot water tank connected to the outer coil, heated by a thermostatically regulated 1500 W immersion water heater, and a 25-litre tank connected to the inner coil, holding the Biosynthesized aqua-based silver nanofluids. The double helically coiled pipe test section, wound around a wooden pattern with equal space between the pipes filled with fine sand particles, is subjected to consistent heat flux, and temperatures are measured at various points using eight K-type thermocouples. Metal paste is applied to prevent leaks, pressure gauges measure the nanofluid loop pressure, and flow rates are controlled by rotometers.

To ensure uniform conditions, the test area is insulated with cotton, and a radiator attachment is used to cool the nanofluids. The experiment involves circulating hot water initially to
achieve a steady state, followed by varying concentrations of Biosynthesized aqua-based silver nanofluids flowing through the inner loop. Measurements are taken for temperature, flow rates, pressure drop, and density. The specific heat capacity is determined using a heat-flux-type differential scanning calorimeter, revealing distinct peaks corresponding to the metallic silver melting point, indicating nanofluid purity. Density measurements are conducted using a transient hot-wire apparatus, with some minor deviations attributed to instrument uncertainties.

Overall, the experimental setup and procedures described contribute valuable insights into the heat transfer and fluid flow characteristics of helically coiled heat exchangers, especially when using Biosynthesized aqua-based silver nanofluids. The detailed measurements and analyses performed enhance the understanding of the system's performance and lay the foundation for potential applications in various thermal management scenarios.

5. Conclusions

The use of Histiopteris incisa leaves extract in a facile green synthesis of AgNPs was demonstrated. According to the findings, Histiopterisincisa leaves extract was an effective reducing, capping, and stabilising agent in the production of AgNPs. The yellowish-brown hue in aqueous solution was caused by the excitation energy of surface plasmon vibration, and it is believed that the components in the leaves extract were reduced. UV-Vis spectroscopy, FTIR, XRD, DLS, Zeta potential, and HRTEM were used to analyse the AgNPs. The UV-Vis spectroscopy shows strong peaks at 427, 429, and 425nm for 1, 3, and 5mM concentrations respectively, indicating the successful production of AgNPs. In the process of biosynthesized AgNPs, FT-IR identified the most effective function groups. XRD analysis was used to identify the crystalline structure FCC geometry corresponding to the peaks (111), (200), (220), and (311) at various concentrations. DLS investigations showed that the particle size and dispersion of aqueous produced AgNPs range between 98 and 69 nm for various concentrations. The Zeta potential measurement demonstrates the stability of AgNPs, with a greater negative value indicating the particles' surface charges. According to the HRTEM, the AgNPS were primarily spherical with diameters ranging from 20 to 50 nm, with an average particle size of 25nm. The thermal conductivity increased with percentage volume concentration and temperature. As a result, the biosynthesis technique of AgNPs employing Histiopteris incisa leaves extract can be used to establish a large scale
manufacturing. These synthesized NPs can be employed in heat transfer applications to increase the rate of heat transfer.

**Author Contributions**

The study's inception and design were helped by all of the writers. Arun M. and I. Rajendran prepared the material, collected the data, and carried out the analysis. All writers provided feedback on earlier drafts of the text after Arun M wrote the initial draft. The final draft was read by all writers and got their approval.

**Data Availability**

Due to Ph.D. work, the datasets created and/or analysed during the current study are not publicly available, but they are available from the corresponding author upon justifiable request.

**Disclosures and declarations**

The authors declare that none of their known financial conflicts of interest or close personal ties might have seemed to have influenced the research presented in this study.

The authors affirm that they did not accept any money, grants, or other assistance for the creation of this manuscript.

No humans and/or animals were involved.

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Tables:

Table 1. Thermal conductivity of metals, metal oxides, and working fluids at room temperature

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal conductivity W/mK</th>
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<tbody>
<tr>
<td><strong>Working Fluids</strong></td>
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<tr>
<td>Water</td>
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<td>Cupric oxide</td>
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Arun M, born on May 18, 1982, in Ooty, is an accomplished Assistant Professor in the Department of Mechanical Engineering at V.S.B. Engineering College, Karur, Tamil Nadu. Holding a Bachelor's degree in Mechanical Engineering and a Master's degree in Thermal
Engineering from Anna University, Chennai, he has dedicated 18 years to a distinguished career in teaching. Arun M has been a mentor and guide to numerous students, with two currently pursuing their M.E. under his supervision and nine successfully completing their postgraduate degrees. Beyond his contributions to academia, Arun M is actively involved in cutting-edge research, particularly in the field of Biosynthesized Nanofluids, showcasing his commitment to advancing knowledge and innovation in mechanical engineering.

Dr. I. Rajendran is a distinguished scholar and educator with a rich background in Mechanical Engineering and Engineering Design. He earned his bachelor's and master's degrees from the prestigious College of Engineering-Guindy, Anna University, Chennai, in 1991 and 1994, respectively. His academic journey culminated in a doctoral degree from PSG College of Technology, Bharathiar University, Coimbatore, in 2001. With a remarkable career spanning 31 years, Dr. Rajendran boasts 25 years of research experience and has contributed significantly to the field. He has authored over 55 papers in national and international journals and presented 30 papers at conferences. Driven by a passion for mentoring, he has guided numerous research scholars and supervised a multitude of undergraduate and postgraduate projects. Additionally, Dr. Rajendran is renowned for his organizational skills, having orchestrated several International and National conferences in the realm of Materials Product Design and Mechanical Engineering. His outstanding contributions have earned him accolades such as the "Innovative Potential of Students Projects Award at Doctoral Level" from the Indian National Academy of Engineering (INAE) and "The N. K. Iyengar Memorial Medal for the Best Practice-Oriented Paper on Machine Design" from The Institution of Engineers (India), Kolkata. In 2019, he was honored with the Outstanding Engineer Award in Mechanical Engineering from the Institution of Engineers (India) Coimbatore Centre, solidifying his legacy in the field.

Dr. A.P. Senthil Kumar, born on January 18, 1975, in Pollachi, is an accomplished Professor in the (Govt. Aided) Department of Mechanical Engineering at P.S.G College of Technology, Coimbatore. He earned his Bachelor's degree in Mechanical Engineering and further pursued a Master's degree in Thermal Engineering, culminating in a Ph.D. from Anna University, Coimbatore. With an illustrious career spanning 25 years in teaching, he was honored with the Best Faculty Award in 2003 from Maharaja Institution, a testament to his exceptional teaching prowess. Dr. Senthil Kumar has mentored and guided numerous students, with one currently pursuing a Ph.D. under his supervision and nine successfully completing their doctoral studies. Beyond academia, he has made significant contributions to research, having authored and published 78 International Journal papers, showcasing his commitment to advancing the field of mechanical engineering. Dr. A.P. Senthil Kumar's dedication to education and research has undoubtedly left an indelible mark on the academic landscape.