RESEARCH PAPER

A study on Synthesis and Characterization of Ag/ Fenugreek Nanostructures Using Green Method

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ABSTRACT

A method for producing nanostructures of silver and fenugreek (Ag/fenugreek NSs) has been enhanced for cost-effectiveness and environmental sustainability by using non-toxic and renewable fenugreek as a capping and reducing agent. This makes it unnecessary to use chemical reducing agents in order to transform silver ions into silver nanoparticles. X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), field emission-scanning electron microscopy (FE-SEM), and Fourier transform infrared spectra (FT-IR), and ultraviolet visible spectra were used to analyze the resulting silver/Ag-fenugreek nanoparticles.

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INTRODUCTION

Globally, research and study in the field of nanotechnology have advanced quickly. Discussions concerning Nanoparticles' potential hazards and effects on the environment and human health are still being investigated, despite the fact that this field has a lot of opportunity to grow [1]. Because of their numerous uses in cancer treatment, biomarkers, cell labeling, antimicrobial agents, medication transport, and diagnostics, nanoparticles have attracted a lot of attention recently.[2]. The ability to comprehend, manage, create, and work with matter at the atomic and molecular level, similar to the abilities of a master craftsman, has been made possible by nanotechnology. The main goal of this technique

is to work with particles with a nanometer (nm) scale measurement that are smaller than 100 nm are called nanoparticles. Physical techniques used to produce these particles include vapor deposition, pulsed electrochemical etching, sputtering deposition, laser pyrolysis, laser ablation, lithography, and plasma or flame spraying synthesis. Sol-gel processing is one of the chemical methods used in this synthesis. Concerns over the safety of nanoparticles for As their manufacture and use in a variety of consumer goods gain more notice, the environment and human health are evolving [3-4].

Numerous issues plague the processes used in their manufacture, including the usage of hazardous chemicals that are expensive, energy-

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intensive, and harmful to the environment and living creatures.[5-6] The drawbacks of commonly used physical and chemical processes have prompted the development of green synthesis, a highly effective, economical, and ecologically safe substitute for producing nanoparticles. Recently, biosynthetic methods—also known as "green chemistry"—that use biological organisms like bacteria, fungi, and plant extracts along with naturally occurring reducing agents like polysaccharides have become simple and efficient substitutes for conventional chemical synthesis in the production of nanoparticles. The need for efficient and ecologically friendly techniques for creating nanoparticles is growing as green chemistry gains more attention. Among the most promising techniques for creating monodispersed inorganic nanoparticles is template synthesis, in which the produced nanoparticles are encapsulated in uniform voids of porous materials [7].

The production of nanoparticles from biological systems which might include complete species like bacteria, plants, and animals, or their metabolites is referred to as "green synthesis." Enzymes, proteins and their derivatives, phytochemicals, and structural templates like membranes or DNA are examples of these metabolites. Numerous studies carried out in recent years have demonstrated the effective production of metal nanoparticles by a

variety of biological systems, including bacteria, fungi, and plant metabolites. [8-15].

The numerous sources cited include cobwebs, honey [16–17], earthworms [18], insects and their byproducts, and more. The biological systems used, the many green synthesis techniques, and the distinctive properties of the particles produced have all been carefully investigated [16] Silver nanoparticles have drawn a great deal of focus in recent decades from scholars due to their numerous uses in medical diagnostics and antimicrobial coatings. They can also be utilized in an extensive variety of goods, such as cosmetics and textiles, due to their antibacterial qualities. Additionally, the oscillation of valence electrons at the surface of silver nanoparticles is known as surface plasmon resonance, or SPR [17,18].

In recent decades, researchers have focused a lot of attention on silver nanoparticles due to their many applications in antimicrobial coatings and medical diagnostics. Because of their antibacterial properties, they can also be utilized in a range of goods, such as clothing and makeup. Furthermore, surface plasmon resonance, or SPR, is the term used to describe the oscillation of valence electrons at the surface of silver nanoparticles. [20,21] Because of their function in photosynthesis and the greater availability of H⁺ ions, which aid in the reduction of silver nitrate during the creation of silver nanoparticles, green

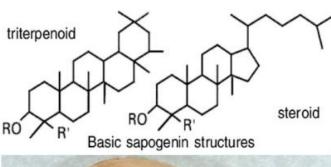




Fig. 1. Bioactive chemical constituents of fenugreek [24].

leaves are preferred for the synthesis of silver nanoparticles in this context over other plant kinds [1,22]. Trigonella foenum graecum, or fenugreek, was utilized to create silver nanoparticles. One of the oldest traditional medicinal plants, fenugreek (Trigonella foenum graecum) is grown throughout the Indian subcontinent as well as in West Asia, the Middle East, North Africa, the United Kingdom, Russia, Mediterranean Europe, Australia, and North America [23]. Fenugreek, a member of the Fabaceae family, is used as a vegetable (fresh leaves), a spice (seeds), and a herb (dried or fresh leaves). Additionally, it contributes to the flavoring of artificial maple syrup and is used in the manufacturing of steroids and other hormones for the functional food, pharmaceutical, and nutraceutical industries. Conventionally (Fig. 1) [24].

In this work, we produced silver particles by extracting *fenugreek*. Both chemical and physical methods could be used to create metal nanoparticles as an alternative. This technique is inexpensive, simple to apply, and eco-friendly because it uses plants as a natural stabilizing and capping ingredient. The production of silver nanoparticles is confirmed by X-ray diffraction (XRD), field emission-scanning electron microscopy (FE-SEM), energy dispersive X-ray dispersive

spectroscopy (EDS), Fourier transform infrared spectra (FT-IR), and ultraviolet visible spectra (UV-Visible) were used to analyze the resulting silver/Ag-fenugreek nanoparticles.

MATERIAL AND METHODS

Silver nanoparticle synthesis:

In this work, distilled water was used to dissolve the materials used. silver nitrate (AgNO₃) (2 x10⁻⁵ M). The solution was kept away from light to prevent oxidation. To prepare fenugreek extract:

- 1-) 5 g(of dried and ground fenugreek 100 milliliters of distilled water were used to dissolve the powder.
- 2- Stir the mixture constantly while heating it for five minutes.
 - 3- The solution was filtered using filter paper.

After that, 80 milliliters of silver nitrate and 20 milliliters of fenugreek extract were mixed, and the combination was left in a dark place for a full day. A centrifuge was used to remove the precipitate from the solution, and After that, it was dried at 50 °C in an oven [25]. Next, the precipitate was crushed and subjected to XRD, FE-SEM, EDS, FTIR and UV-visible analysis (Fig. 2).

RESULTS AND DISCUSSIONS

Using an XRD model (PanalyticalX'pert) with

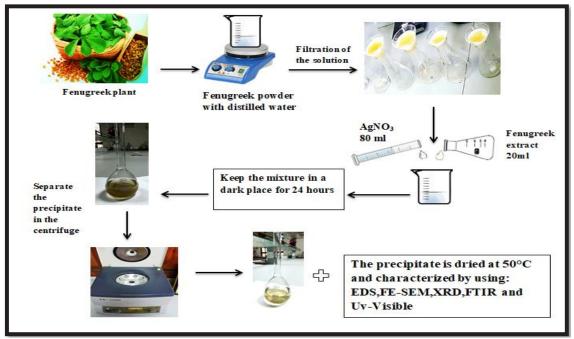


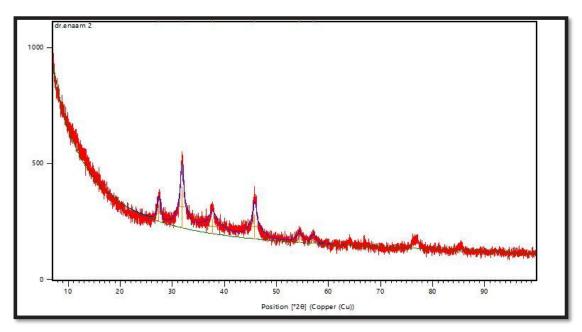
Fig. 2. Working scheme for Ag -fenugreek NPs synthesis.

CuKαradiation of λ =1.5405 A°, the crystal structure, phase identification, and crystallite size of the produced powder samples were investigated. XRD analysis was used to look at the crystalline composition of the biosynthesized Ag/Fenugreek nanoparticles is seen in Fig. 3, The XRD shows nine peaks at 27.769°, 31.698°, 37.856°, 45.897°, 54.675°, 57.294°, 64.025°, 76.731°, and 85.508°, which propose the Ag/Fenugreek nanoparticles' FCC phase structure. Based on the XRD data, the size of the silver/fenugreek nanoparticles is calculated using Scherrer's equation:

Crystalline size (D) =
$$\frac{K\lambda}{\beta \cos \theta}$$
 (1)

where the Cu-K α X-ray's wavelength (λ = 1.5406 Å), diffraction angle (θ), and full breadth at half maximum (β) are all expressed in radians. K is the symbol for the Debye Scherrer constant [26].

Fourier transform infrared spectroscopy (FT-IR) measurements were performed using to identify the biomolecules that reduced Ag ions to Ag-fenugreek NPs. The peaks observed at (1693,56,1624.12, 1516,10,1450.52) cm⁻¹, which



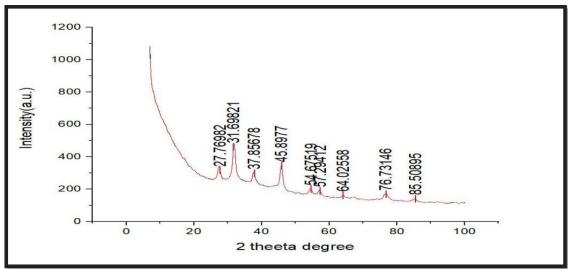


Fig. 3. XRD pattern for Ag/fenugreek NPs.

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are attributed to (-C=C-, C=O- aromatic), and – COOH stretching vibrations, respectively , (-C=O) at (1273.06,1226.77, 1134.18,) cm $^{\text{-}1}$, and (-OH, stretching)) at (3263.66,3132.50) cm $^{\text{-}1}$, [26],the band at 2970.48 cm $^{\text{-}1}$ corresponds to the (CH $_2$) asymmetric stretching vibrations. The absorption peaks appeared at (987.59, 671.25, 516.94, 408. 92) cm $^{\text{-}1}$ due to medium C-C stretching of disubstituted alkene medium are displayed in Fig. 4.

In Fig. 5, (a,b) using FE-SEM analysis, the produced materials' average size and surface morphology were determined [27-28]. The produced Ag/fenugreek nanoparticles have diameters of 79.66, 65.97, and 75.32 nm, according

to the FE-SEM pictures. This is because the Ag/fenugreek extract works as a capping agent and prevent the Ag/fenugreek NPs from aggregation. The average diameter of Ag/fenugreek particles was estimated from the FE-SEM images and it was (~73.65) nm.

The presence of the Ag/fenugreekNPs was confirmed by energy dispersive spectroscopy, or EDS. The Ag/fenugreek NPs nanocrystal line exhibits strong and precise diffraction peaks, indicating good crystallinities. The EDX spectra of the pure Ag-fenugreek NPs reveal the presence of only C, O, P, S,Cl and Ag [29]. Table 1 evaluates the atomic concentration in a semi-quantitative manner (atom %). It indicates that

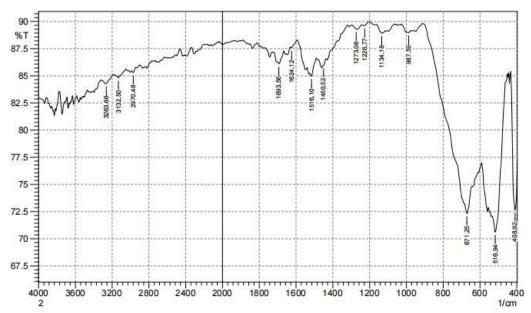


Fig. 4. FT-IR spectrum for Ag/fenugreek nanoparticles.

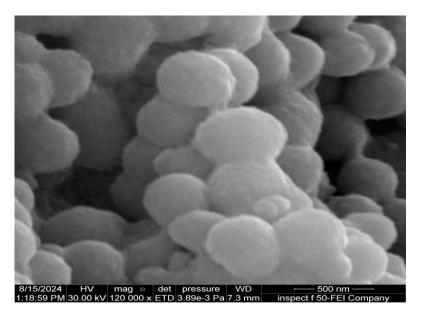
Table 1. Ag/fenugreek NPs composition was examined using EDS

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
С	57.3	0.3	33.0	0.2
0	32.0	0.4	24.5	0.3
Р	1.0	0.0	1.5	0.0
S	0.3	0.0	0.4	0.0
Cl	2.3	0.0	3.8	0.0
Ag	7.1	0.0	36.8	0.2

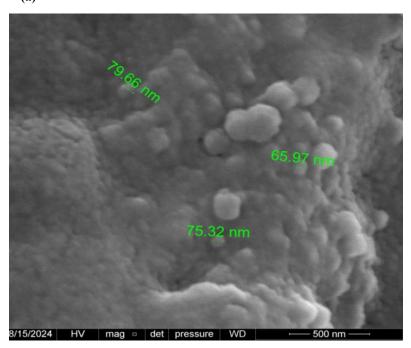
the products' Ag-fenugreek NPs elements content is (33.0,24.5, 1.5,0.4,3.8, and 36.8) for Carbon (C), Oxygen (O), Phosphor (P), Sulphur (S), Chlorine (Cl), and Silver (Ag).

The EDS results are shown in Fig. 6, where it is evident that the weight ratios of Ag-fenugreek

NPs were, respectively, (79.66, 65.97, and 75.32) %. The EDS examination showed that the sample contained the necessary phases of Ag that the produced Ag-fenugreekNPs were highly pure. Similar C, O, P, S, Cl and Ag ratios that are near to the theoretical values are seen in the EDS results



(a)



(b)

Fig. 5. FE-SEM spectrum (a,b) for Ag/Ag-Triticum aestivum NPs

from the current investigation [30].

Ag- fenugreek NPs.samples' chemical composition is revealed by EDS analysis. The produced samples are mostly made up of C,

O, P,and Ag, with very little S and Cl present, as shown in Fig. 7.

To bolster the synthesis of Ag-fenugreek NPs, the 8110SP, a Japanese spectrophotometer, was used

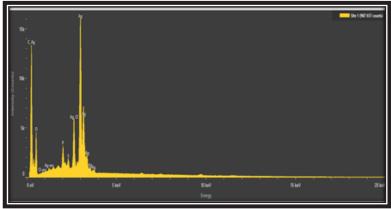


Fig. 6. Pure Ag-Triticum aestivfenugreek NPs elemental mapping and EDS analysis.

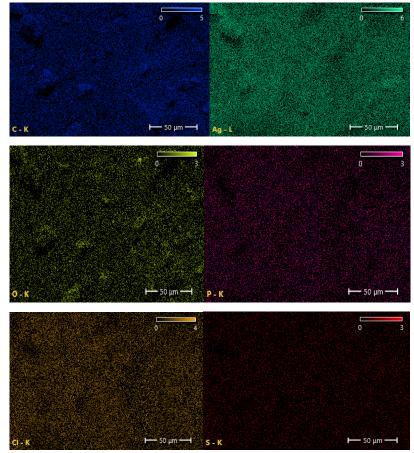


Fig. 7. EDS analysis for Ag-fenugreek NPs.

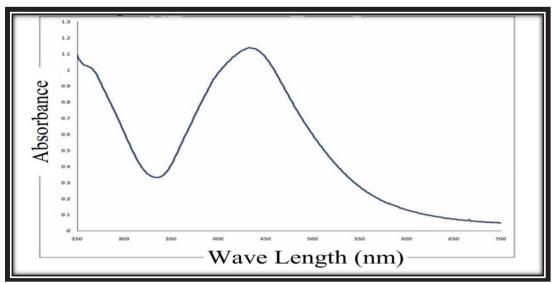


Fig. 8. Ag-fenugreek NPs' UV-Vis absorption spectrum, which was produced by employing fenugreek extract as a reducing agent. The produced Ag-fenugreek NPs' SPR absorption band is indicated by the red arrow at about 457 nm.

to record the UV-Vis absorption spectrum. The produced solution's surface plasmon resonance (SPR) absorption band is roughly 457 nm, as seen in Fig. 8. This is a red shift from the silver metal's SPR band, which is at 425 nm [31]. This red shift is caused by the synthesis of Ag-fenugreek (NPs) when Ag - fenugreek extract is present.

CONCLUSION

An environmentally friendly method has been successfully used to manufacture silver-fenugreek nanoparticles. The manufacture of Ag-fenugreek nanoparticles using plant extract as an agent for reduction.is the basis of this method, which could result in applications for the particles in the biological, pharmacological, and medical domains. Ag-fenugreek nanoparticle manufacturing has been verified by FTIR, XRD, FE-SEM, EDX, and UVvisible.

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CONFLICT OF INTEREST

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

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