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Fenugreek Mediated Synthesis of Silver Nanoparticles and its Characterization

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***Abstract* - Generally, nanoparticles are prepared by a variety of chemical methods which are not environmentally friendly. Use of biological method (plant seeds) in the synthesis of nanoparticles is quite novel leading to truly green synthesis which provides advancement over chemical and physical method, as it is cost effective and environment friendly. The stable, green synthesis of silver nanoparticles through biological methods has a wide range of applications and is very gratifying in the medical field because of their low toxicity. Therefore, the demands of silver nanoparticles synthesized biologically are on the rise. The present work aims at Biosynthesis of *Trigonella foenum-gracum L* seed nanoparticles and their characterization. During the synthesis of silver nanoparticles, the reduction reaction takes place i.e., Ag+ to Ag0 during exposure to the sunlight. The confirmation of synthesized nanoparticles from *Trigonella foenum-gracum L* seed extract was confirmed visibly by color change of the solution from light yellow to deep brown within 30 min of exposure. The separation of these nanoparticles was performed by centrifugation. The pellet of silver nanoparticles was further converted to powder by drying. From the characterization studies conducted by UV-Visible spectroscopy, it is observed that surface Plasmon resonance peaks have shown the maximum absorbance of silver- nanoparticles at 425 nm, indicating that silver nanoparticles were synthesized. Further characterization was conducted by distinct types of methods mentioned such as scanning electron microscope (SEM) analysis, Fourier Transform Infrared Spectroscopy (FTIR) analysis and Elemental detection sensor (EDS) analysis. The average size was found to be 100 nm and sphere shaped evaluated by Scanning electron microscope (SEM). The Fourier Transform Infrared Spectroscopy (FTIR) spectrum revealed that the absorption bands at 3282 cm1 and 1639 cm1 indicated the presence of -OH or -COOH and amide group stretching in the silver nanoparticles. The Elemental detection sensor (EDS) images are characterized by spectrum process with number of iteration 2 and peak are omitted at 2.145, 5.740keV represents the presence of elements like Silver, Carbon and Oxygen.**

Keywords: Fenugreek, Mediated Synthesis, Silver, Nanoparticles.

1. Introduction

Fenugreek (*Trigonella foenum graecum*) is an annual plant belongs to the family Leguminosae. It is the famous spices in human food. The seeds and green leaves of fenugreek are used in food as well as in medicinal application that is the old practice of human history. It has been used to increase the flavoring and color and modifies the texture of food materials. Seeds of fenugreek spice have medicinal properties such as hypocholesterolemic, lactation aid, antibacterial, gastric stimulant, for anorexia, antidiabetic agent, galactogogue, hepatoprotective effect and anticancer. These beneficial physiological effects including the antidiabetic and hypocholesterolemia effects of fenugreek are mainly attributable to the intrinsic dietary fiber constituent which has promising nutraceutical value. It is well known for its fiber, gum, other chemical constituents, and volatile contents. The dietary fiber of fenugreek seed is about 25% which changes the texture of food. These days it is used as food stabilizer, adhesive and emulsifying agent due to its high fiber, protein, and gum content. The protein of fenugreek is found to be more soluble at alkaline pH. Fenugreek is having beneficial influence on digestion and can modify food. Fenugreek is an annual herb of Leguminosae, is being used as spice with its seeds and as vegetable with its leaves. Fenugreek seeds have a strong aroma and are somewhat bitter in taste. Fenugreek is native to Southern Europe, the Mediterranean region, and Western Asia. It is cultivated from Western Europe to China for the aromatic seeds and is still grown for 11 fodders in parts of Europe and Northern Africa. The seeds are extremely hard, and difficult to grind.

About 76% of the seeds have a length ranging from 3.44 to 4.75 mm, about 80% a width ranging from 2.02 to 2.73 mm, about 77% a thickness ranging from 1.27 to 1.73 mm and about 74% a seed mass ranging from 0.0122 to 0.0197 g at 8.9% moisture content on dry basis. The average length, width and thickness of fenugreek seeds and single seed mass and geometric mean diameter ranged from 4.01 to 4.19, 2.35 to

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2.61, 1.49 to 1.74 mm, 0.0157 to 0.0164 g and 2.40 to2.66 mm as the moisture content.

1.1 Fenugreek chemical constituents

Fenugreek seeds are rich sources of fiber and protein. The fiber may be further classed as gum (gel fiber) and neutral detergent fiber. Whole fenugreek seeds also contain 4.8 % saponins.

Table 1: Proximate compositions % of Fenugreek seeds (Reference's: ISSN 0975-8585)

Component	Whole Seeds	Defatted Seeds
Moisture	9.0	9.0
Ash	3.0	3.5
Lipids	8.0	Negligible
Protein	26.0	28.3
Starch	6.0	6.5
Total Fiber	48.0	51.7
Gum	20.0	19.2
Neutral Detergent Fiber	28.0	32.5

1.2 Nutraceutical properties of Fenugreek

Fenugreek has a beneficial effect on cleansing the blood and as a diaphoretic it can bring on sweat and help detox the body. Fenugreek is also known for its lymphatic cleansing activity though its vital role is to irrigate the cells with nutrients and to remove toxic wastes, dead cells and trapped proteins from the body. Blocking in the lymphatic system can mean poor circulation of fluid, fluid retention, pain, energy loss and disease, anywhere in the body of a person. Fenugreek maintains mucus conditions. of the body, mostly the lungs, by helping to clear congestion. It also acts throat cleanser and mucus solvent that also eases the urge to cough. Drinking water in which seeds of fenugreek have soaked helps in softening and dissolving, accumulating, and hardening the masses of cellular debris. Fenugreek has been. used to relieve colds, bronchial complaints, influenza, asthma, catarrh, constipation, sinusitis, pleurisy, pneumonia, sore throat, laryngitis, hay fever tuberculosis and emphysema.

1.3 General overview about Nanotechnology

Nanotechnology involves research and technology development at the atomic, molecular or macromolecular levels in the range of approximately 1-100 Nanometers to provide fundamental understanding of phenomena and materials at the Nanoscale. The nanometer scale is about a billionth of a meter. In comparison, Fenugreek seeds saponins are of steroidal nature (type Furostanol Saponins) with Diosgenin as the principal steroidal Saponin. Human hair is about10,000 nanometers in diameter. Basically, nanotechnology is used to create structures, devices and systems that have novel properties and functions because of their minute size. The matter shows unusual physical and chemical properties due to increase in surface area compared to volume as particles get smaller in size and this is called quantum size effect. This means the bulk properties of materials at Nano-scale can be very different from those at larger scale. Taking the advantage of these characteristic of material, scientist designs and produces devices by manipulating the shape and size at Nano-scale with wide-range of implications which could include medicine, electronics, military applications, computing, space science and many more. Nanoparticles can be widely differentiated into organic carbon containing NPs and inorganic NPs include magnetic NPs, coinage metal NPs (like gold and silver) and semi-conductor nanoparticles (titanium oxide and zinc oxide). Synthesis of noble nanoparticles for the applications such as catalysis, electronics, environmental and biotechnology is an-area of sustained interest.

1.4 Green synthesis

Traditional methods are used from past many years, but research have manifest that the green methods are more effective for the generation of nanoparticles with the benefit of less chances of failure, low cost and ease of characterization. Plant based synthesis of Nanoparticles is certainly not a inconvenient procedure, a metal salt is synthesized with plant extract and the reaction is completed in minutes to couple of hours at typical room temperature. This grand design has fascinated much more attention among the most current decade predominantly for silver (Ag) and gold (Au) Nanoparticles, which are more secure as contrasted with other metallic NPs. Generation of Nanoparticles from green techniques can be intensified effortlessly and they are economically smart too. Considering their exceptional properties, the greenly orchestrated Nanoparticles are currently favored over the traditionally delivered Nanoparticles. Green methods of synthesis are significantly attractive because of their potential to reduce the toxicity of NPs. Accordingly, the use of vitamins, amino acids, plants extracts is being greatly popularized nowadays. Fenugreek plant is globally known for its medicinal significance. It is having a significant property such as hypocholesterolemic, lactation aid, antibacterial, gastric stimulant, for anorexia, antidiabetic agent, galactogogue, hepatoprotective effect and anticancer anti-inflammatory, anti- helmentic, lowering blood pressure antipyretic properties etc.

The major objective of this study is to present an eco-friendly, cost effective and biological method for the synthesis of Silver nanoparticles, using fenugreek seed plant extract.

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2. Objectives

- Synthesis of Silver Nanoparticles from Fenugreek seed by biological method.
- Characterization of Fenugreek Silver Nanoparticles using SEM, FTIR and EDS.

3. Methodology

Biological synthesis of Fenugreek silver nanoparticles

3.1 Materials used

- Fenugreek seeds
- Silver Nitrate ($AgNO_3$)
- Sterile Distilled water

3.2 Synthesis of fenugreek seeds extract

5g Fenugreek Seeds were weighed using weighing balance and then washed with distilled water to remove any contaminant or dust particles. The seeds were taken in a sterile container and 100ml of sterile distilled water was added. Further the seeds were soaked overnight. The soaked seeds were ground. The paste was centrifuged at 10000 rpm for 15 min to collect the upper supernatant. The supernatant was transferred into the brown bottle and kept in the dark room for 24 h.

3.3 Biosynthesis of Silver Nanoparticles

The fenugreek seed extract was mixed with equal proportion of freshly prepared silver nitrate (1 mM) and further solution is exposed to sunlight for 30 minutes. The transformation of the color of the solution was observed during the exposure process within 10–15 min. The silver particle solution was centrifuged at.10,000 rpm for 10 min to remove the debris. The color changes indicate the formation of Silver nanoparticles (AgNPs) from the fenugreek seed extract.

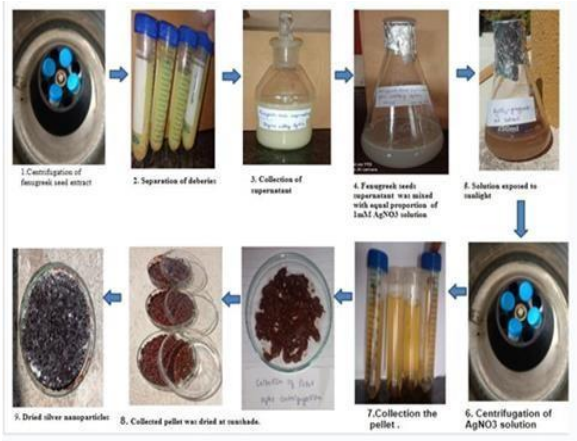

Figure 1: Overall process of Synthesis of silver nanoparticles

3.4 Characterization of synthesized Silver Nanoparticles

Characterization of the nanoparticles were done by using UV-VIS spectra analysis, scanning electron microscope (SEM) with element detection sensor (EDS) and Fourier transform infrared spectroscope (FT-IR).

3.4.1 UV-VIS spectra Analysis

The reduction of pure Ag+ ions was detected by measuring the Ultraviolet-visible spectroscopy of each reaction mixtures at different time intervals within the range of 400-480nm in the UV- VIS spectrophotometer.

3.4.2 Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups were matched with library (lab solution- IR) by giving Peak-peak search. Functional group in the Fenugreek mediated synthesized silver nanoparticles synthesized nanoparticles was analyzed by using (FTIR). The instrument was initialized with lab solution software (Version, 100). The method was created by Selecting per cent transmittance, number of scans (20 to 45), Resolution (4 cm-1) and scan range from 3282 to 470 cm-1. Mid IR beam splitter and DLATGS (Deuterated Lanthanum α Alanine doped Triglycine) detector was used for sample and background scan. Accurately, 0.5 g synthesized nanoparticles was placed on the Specac Quest Diamond ATR (Attenuated Total reflection) and sample scan was given in the software. After the sample scan IR spectra was obtained and the wave number range (400 to 4000 cm-1) for FT-IR analysis was selected according to the procedure given by Alam et.al, (2017).

3.4.3 Scanning Electron microscope (SEM)

The surface morphology was studied by using scanning Electron microscope (SEM) (Carl Zeiss, EVO 10, Germany). The aluminum stub (~1 cm diameter) was cleaned to remove Surface oils or dirt by using acetone and blowing with Compressed gas. The double coated conductive carbon tape was used as adhesives and pasted on stub. The thin layer of Nano adsorbents (~0.2 mg) was placed on adhesive surface, then they were coated with palladium for 90 s to make them Conductive using sputter coater (Quorum technologies, OM-SC7620, United Kingdom). The sample holder was removed from the sputter coater and placed in vacuum chamber of SEM and magnification was (1 to 30,000 times) carried out to get clear morphology of nanoparticles at an accelerating Voltage of 1 to 30 kv with a working distance of sample at 10Mm.

3.4.4 Elemental detection sensor (EDS)

The elemental detection of synthesized nanoparticles was Studied by using element detection sensor (EDS) (Oxford Instruments, X- maxn 80, United Kingdom. After obtaining

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the images from scanning electron microscopy (SEM), the images were loaded into Inca-software (Oxford-7ROJ6VOT) and confirmed the elements present in the synthesized nanoparticles. The images were analyzed in the Inca-software and per cent weight of elements present nanoparticles.

4. Results

4.1 Green synthesis of nanoparticles

After the extraction, the Fenugreek seeds supernatant was mixed with equal proportion of 1mM $AgNO_3$ solution and then exposed to sunlight. There is a change in color of the sample from colorless to dark brownish.

Figure 2: Change in color (Reduction)

By the observation of change in color in the sample by exposing to sunlight for 10 -15 minutes. The sample centrifuged at 10,000 rpm to remove debris and collected in petri plates and kept for drying to obtain powder form of $AgNO_3$ (Fenugreek Sliver nanoparticles).

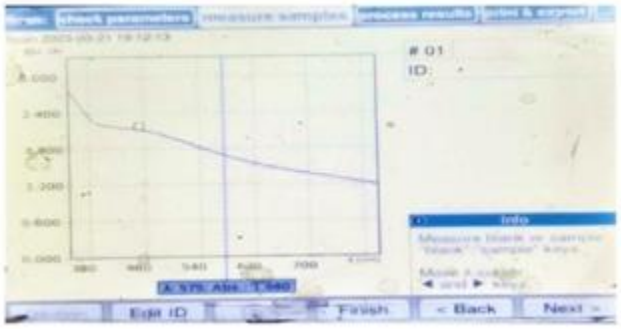
Figure 3: Powder form of fenugreek sliver nanoparticles

4.2 Characterization of nanoparticles

Characterization of nanoparticles is important to understand and control nanoparticles synthesis and applications. Characterization is performed using a variety of different techniques such as Ultraviolet–visible spectroscopy, scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) and Element dispersive spectra (EDS). These techniques are used for determination of different parameters such as particle size, shape, crystallinity, and pore size. For instance, the morphology and particle size could be determined by SEM.

4.3 UV–visible spectroscopy

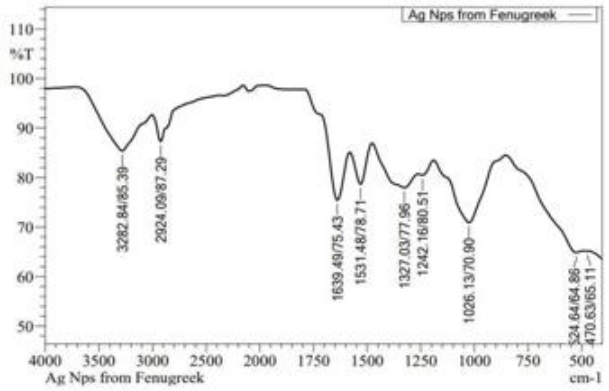
The Fenugreek seed extract were mixed with 1mM AgNO3 resulted in the formation of brownish red color revealed the synthesis of the AgNO3 and further confirmed by Ultraviolet–visible spectroscopy at 420 nm with the absorbance at 0.182. The results reported that the absorbance of the solution was increased with respect to the increase in the time periods because of the surface plasmon resonance of the nanoparticles of silver element (Dhas et al., 2014) Reports claimed that the reaction conditions such as temperature, light intensity and buffering conditions of the samples and the concentrations of the $AgNO_3$ play a major role in the formation of nanoparticles.



Graph 1: UV–visible absorption spectra of the $AgNO_3$ synthesized using the seed extract of fenugreek

4.4 Fourier transform infrared spectroscopy (FTIR)

The functional group of the $AgNO_3$ were analyzed by FTIR spectra. The synthesized $AgNO_3$ showed absorption bands at 3282cm-1, 2924cm-1, 1639cm-1, 1531cm-1. The peak at 3282cm-1 and 1639 cm-1 showed the presence of -OH or -COOH and amide group stretching in the $AgNO_3$. These absorbance bands were also associated with an O-H stretch of phenol or alcohol group primary amine, N=O bond of nitro group, $-NO_2$ of aliphatic group. Presence of the peak level and the peak point is X-ray of different elements.



Graph 2: FTIR spectra of the AGNPs synthesized using the seed extract of fenugreek

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4.5 Scanning electron microscopy (SEM)

Scanning electron microscope images are characterized by high resolution up to 1-100nm and large depth of field. Some information about relative heights in surface texture can be obtained from single SEM images but some impressions can also be misleading. SEM analyses the measurement of nanoparticles and morphological behavior shape and size of nanoparticles.

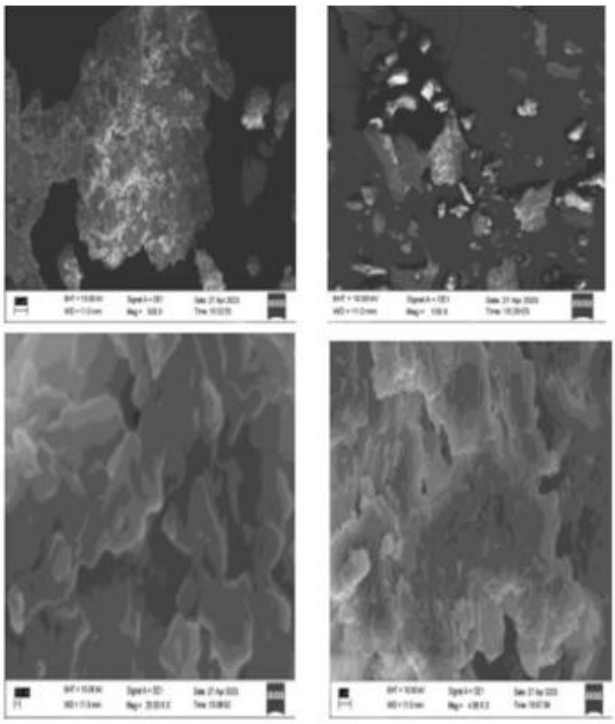
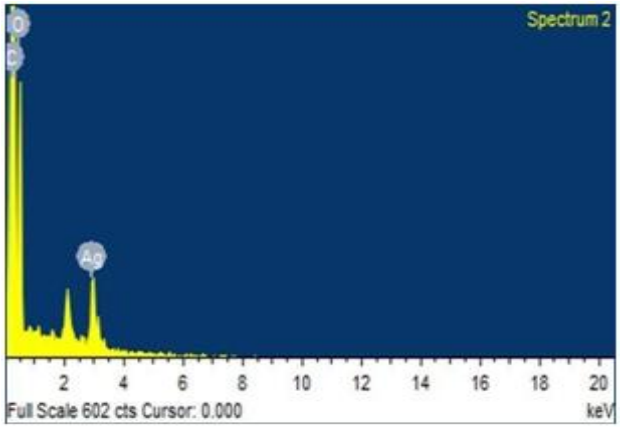


Figure 4: Scanning electron microscopic analysis of the reaction mixture

4.6 Element dispersive spectra (EDS):

EDS images are characterized by spectrum process with number of iterations 2 and possible peaks are omitted at 2.145 5.740 keV. This analyses the element detection, sensors, qualitative, quantification of nanoparticles.



Graph 3: EDS spectra of the AgNPs synthesized using the seed extract

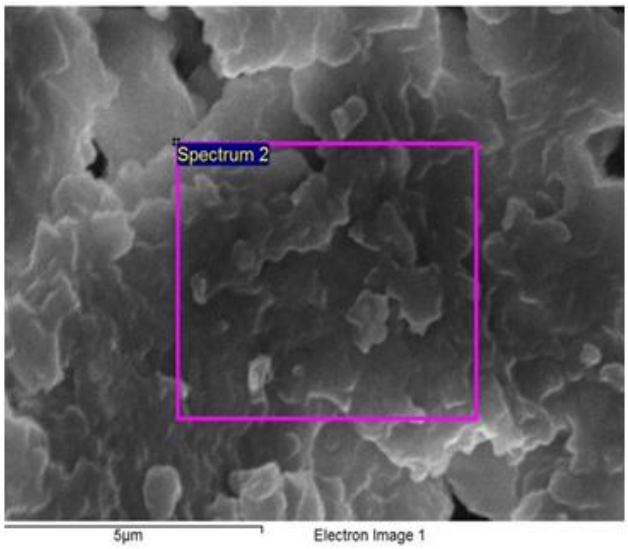


Figure 5: EDS analysis of synthesized nanoparticles

5. Conclusion

In this report, we conclude that we were synthesized silver nanoparticles from the seeds of fenugreek were confirmed by UV– VIS spectrophotometer, SEM, XRD, FTIR and EDX analysis. The results showed that the extracts of fenugreek seeds were capable of synthesizing silver nanoparticles.

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