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# Green Synthesis of Zinc Nanoparticles using *Trigonella foenum-graecum*

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## Abstract

In the present study, biological synthesis of zinc nanoparticles was performed using hydroalcoholic extract of fenugreek. The synthesized zinc nanopariticles were investigated through the use of methods such as FTIR, UVvis, SEM, TEM, XRD. The SEM image of the prepared ZnNPs demonstrated a 3D nanostructure with an average diameter of 1.7 µm. The elemental composition of the prepared sample was analyzed by Energy-dispersive X-ray. The XRD pattern of the synthesized ZnNPs revealed various peaks at 2θ values of 31.92°, 34.42°, 36.31°, 47.52°, 56.68°, 62.93° and 67.98° which corresponded to (100), (002), (101), (102), (110), (103) and (112) crystal planes respectively. The average crystalline size of ZnNPs, derived from the extract of a more intense peak corresponding to the (101) plane (located at 36.31°) was estimated to be 15.41 nm using Scherrer's formula. UV-Vis spectroscopy initially confirmed the formation of ZnNPs within a range of 300 – 600 nm. The absorption spectrum of the nanostructure of Zn showed a distinctive band at 375 nm. FTIR was recorded to identify the most important functional groups on the fenugreek extract to examine their role in the manufacture and capping of Zn 3D structure. TEM of the synthesized ZnNPs was conducted to further investigate the topography structure of the prepared nanoparticles. TEM image revealed a flower-like shape which is in good agreement with the findings of SEM. The study successfully applied a simple and eco-friendly method for synthesizing efficient multifunctional zinc nanoparticles using green synthetic approach.

***Key words***: Zinc nanopariticles, Fenugreek, *Trigonella foenum-graecum,* Biosynthesis ZnNPs, ZnNPs characterization

The synthesis of metal nanoparticles (NPs) has been the topic of intense research over the past decade mainly owing to their unique properties and potential applications from a technological point of view. ZnO is a unique functional metal oxide nanoparticle because of its dual semiconducting and piezoelectric properties. This nanoparticle has a wide range of applications in optics, optoelectronics, sensors, actuators, energy and biomedical sciences, and spintronics. It is a direct wide-bandgap semiconductor with a high exciton binding energy of 60 meV. The high chemical stability and low toxicity of ZnO has rendered it suitable for UV screening applications. Furthermore, as a bactericide, it is effective in inhibiting both Gram-positive and Gram-negative bacteria. It is also a photocatalyst. Scientists have been interested in modifying and coating semiconductor nanoparticles such as ZnO via metallic particles such as Ag and Au, hence the development of nanocomposites. In metal oxide nanocomposites such as Ag-ZnO nanocomposites, Ag deposited on the metal oxide surface is able to significantly enhance the charge transfer kinetics between metal and semiconductor. Therefore, it significantly increases the photocatalytic activity of silver-coated nanostructures compared to non-coated metal oxide nanoparticles. Several physical and chemical methods have been conducted to synthesize Ag-ZnO nanocomposites. However, most of these methods require high-risk and toxic materials, expensive equipment, and harsh laboratory conditions, including intense heat and pressure and long reaction times. Consequently, there has always been a need for a simple, environment-friendly method for the synthesis of high-performance, low-cost Ag-ZnO nanocomposites [1].

Zinc nanoparticles (ZnNPs) are one of the most important inorganic metal oxide nanoparticles, which are employed in different biomedical applications such as cancer therapy due to its impressive properties, nontoxicity and biocompatibility [2]. Despite there being many approaches used in the manufacture of Zn nanostructure, they are related to certain constraints such as toxic chemicals, high operating costs and energy needs [3]. Therefore, replacing these approaches with simple, cost-effective and environmentally sustainable systems is becoming increasingly important. The green synthesis of ZnONPs using natural plant extracts attracted the attention of the researchers because plant-mediated synthesis is environmentally friendly, inexpensive, readily available, yields the highest quantity of NPs and has a wide range of metabolites to help effectively produce and cap NPs [4].

Fenugreek seed (*Trigonella foenum graecum*) is an annual plant belonging to the family of Leguminosae. Such seeds have therapeutic properties for anorexia, antidiabetic, hepatoprotective influence, anticancerial, antibacterial and gastric stimulant. Fenugreek seeds contain various components such as diosgenin, saponin, coumarin, a variety of alkaloids such astrigonellin, gentianin and carpaine, polyphenol

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compounds such as rhaponticin and isovitexin, and flavonoids [5]. Such biomolecules contain different functional groups capable of forming nanoparticles. The resulting nanoparticles are then safe against further reactions and aggregations, which increases its steadiness. Previous studies suggested that fenugreek seeds extract is rich in galactomannan polysaccharides that have reducing functional groups which may have an important role in nanoparticles preparation, in addition to, containing phenolic compounds that may act as capping and stabilizing agents for the prepared nanoparticles [6]. The objective of this study is to introduce a new and simple green synthesis method for preparing the nanostructure of zinc nanoparticles using fenugreek extract as a reducing agent and moreover, the synthesized Zn nanoparticles were assessed for their pharmacological activities.

## MATERIALS AND METHODS

Seeds of *Trigonella foenum-graecum* were purchased and collected from the local market. The seeds were dried and crushed into coarse powder, which was used for extraction with alcohol (95% v/v) using Soxhlet apparatus. The extracts were evaporated to dryness under controlled temperature (35-40°C). The extracts were stored in air tight containers under refrigeration. These dried extracts were dissolved in respective solvents and used for further analysis.

*Biosynthesis and characterization of zinc nanoparticle*

*Preparation of chemicals*

The chemicals used for synthesis are Zinc nitrate 2.1%, Ammonium carbonate 0.96% and polyethylene glycol 5% solution was prepared and stored in amber coloured bottle.

*Preparation of extract*

A quantity of 100 g of finely grounded *Trigonella foenum-graecum* was dissolved in 1000 ml of water and boiled for around 3 hr. After cooling at room temperature, it was centrifuged for 15 min and filtered. The filtrate was stored at 5–10$^0$C for further experiments.

*Preparation of Zn nanoparticles using leaf extract*

Zinc nitrate was used as precursor for the synthesis of Zn nanoparticles. A ratio of 1:3 of zinc nitrate and *Trigonella foenum-graecum* seed extract was mixed and the solution was subjected to microwave irradiation at 540 W, which produced brownish-black precipitate after 7 min. The precipitate was filtered and then dried at hot air oven for 4 to 5 h. The possible mechanism of the formation of Zinc nanoparticles from *T. foenum-graecum* extract.

*Synthesis of Zn nanoparticles*

Zn nanoparticles were synthesized by the precipitation method using zinc nitrate hexahydrate and sodium hydroxide as precursors. Zinc nanoparticles were produced by mixing aqueous solutions of zinc nitrate and sodium hydrate.

The aqueous solution was prepared by mixing zinc nitrate hexahydrate and sodium hydroxide aqueous solutions. In a typical procedure, 2.28 g of zinc nitrate hexahydrate was dissolved in 75 ml of deionized water and then, 0.6 g of NaOH in 150 ml of deionized water was added drop wise under magnetic stirring. After the addition was completed, the stirring was continued for 30 min and then cooled with cold water. The precipitates were filtered and washed by pure water several times. Then they obtained precipitates were dried at 60$^0$C for 24 h and calcined at 200$^0$C for 2 h. The crystalline and phase structure of the synthesized ZnO was studied by an X-ray diffracto meter. FTIR spectra were studied in the range of 400–4000 cm−1. Morphology and primary sizes of the synthesized ZnO were observed by a SEM. The particle size distribution was measured by a laser light scattering technique. Morphology, particle size and specific surface area of the conventional ZnO were studied by SEM and laser light scattering techniques respectively.

*Characterization of zinc nanoparticles by UV-Visible spectroscopy*

UV-Visible spectroscopy is used for monitoring the signature of zinc nanoparticles. UV-Visible spectroscopy is a powerful tool for the characterization of colloidal particles. Noble metal particles are ideal candidates for study with UV-Vis, since they exhibit strong surface plasmon resonance absorption in the visible region and are highly sensitive to the surface modification.

*Characterization of zinc nanoparticles by SEM*

This study was undertaken to know the size and shape of the zinc nanoparticles biosynthesized using *Trigonella foenum-graecum*. SEM analysis was done using FEI Quanta 200 SEM machine. Thin films of the sample were prepared on a carbon coated copper grid by just dropping a very small amount of the sample on the grid, extra solution was removed using a blotting paper. Then the film on the SEM grid was allowed to dry and the images of nanoparticles were taken.

*Characterization of zinc nanoparticles by AFM*

The surface morphology of the zinc nanoparticles was visualized by AFM under normal atmospheric conditions. The zinc nanopartcle samples dispersed on small slide and explored on the instrument were examined.

*Characterization of zinc nanoparticles by FTIR*

The dried zinc nanoparticles were subjected to FTIR analysis by Potassium Bromide pellet (FTIR grade) method in 1: 100 ratios and spectrum was recorded in Nicolet Impact 400 FT-IR Spectrophotometer using diffuse reflectance mode.

*Characterization of silver nanoparticles by XRD*

The air-dried nanoparticles were coated onto XRD grid and analyzed for the formation of zinc nanoparticle by Philips X-Ray Diffracto meter with Philips PW 1830 X-Ray Generator operated at a voltage of 40kV and a current of 30mA with Copper Potassium alpha radiation. The diffracted intensities were recorded from 10' to 80' of 20 angles.

## RESULTS AND DISCUSSION

*Biosynthesis and characterization of zinc nanoparticles*

To the ethanol extract, Zinc nitrate 2.1%, Ammonium carbonate 0.96% and polyethylene glycol solution was added slowly drop wise in a molar ratio of 1:2 under vigorous stirring, and the stirring was continued for 12 hours. The precipitate obtained was filtered and washed thoroughly with deionized water. The precipitate was dried in an oven at 100°C and ground to fine powder.

Synthesis of zinc nanoparticles by using seed extract of *Trigonella foenum-graecum* medicinal plant has been demonstrated in present investigation. The reduction of zinc ions and their capping were achieved by the organic molecules present in the leaf extract. The UV-Vis, X-ray diffraction graph, SEM, AFM, FTIR results revealed that the zinc nanoparticles were spherical in shape and ranging from 30 to 63 nm in size.

Scanning electron microscope was used to decide size, location and shape of the zinc oxide nanoparticles.

UV-visible spectroscopy is usually conducted to confirm the synthesis of zinc nanoparticles. Conducting electrons start oscillating at a certain wavelength range due to surface plasmon resonance effect. The occurrence of the peak at 380 nm is due to the phenomenon of surface Plasmon resonance, which occurs due to the excitation of the surface plasmons present on the outer surface of the zinc nanoparticles which gets excited due to the applied electromagnetic field. Initial peak obtained at range of 420 nm got further raised due to oscillation of more electrons after 5 hours which depicts the continuous synthesis of zinc nanoparticles (Fig 1).

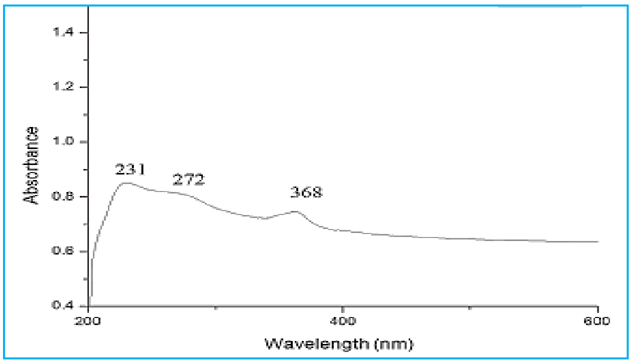


Fig 1 UV-visible spectrum peak of *T. foenum-graecum* synthesized zinc nanoparticle at 420 nm

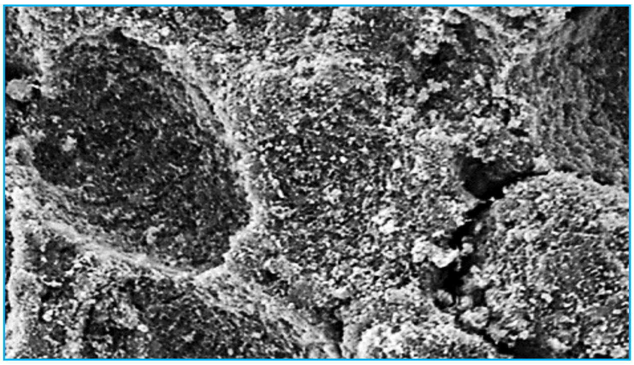

Fig 2 SEM image of synthesized zinc nanoparticle from *Trigonella foenum-graecum*

SEM analysis is done to visualize shape and size of nanoparticle. A scanning electron microscope was employed to analyze the shape of the zinc nanoparticles that were synthesized by green method. SEM analysis shows that the *Trigonella foenum-graecum* have tremendous capability to synthesize zinc nanoparticles which were roughly spherical in shaped nanoparticle with average diameter of 70 nm and were uniformly distributed (Fig 2).

FTIR gives the information about functional groups present in the synthesized zinc nanoparticles for understanding their transformation from simple inorganic zinc nitrate to elemental zinc by the action of different phytochemicals which would act simultaneously as reducing, stabilizing and capping agent. FTIR spectrum clearly illustrates the biofabrication of zinc nanoparticles mediated by the *Trigonella foenum-graecum* extracts (Fig 3).

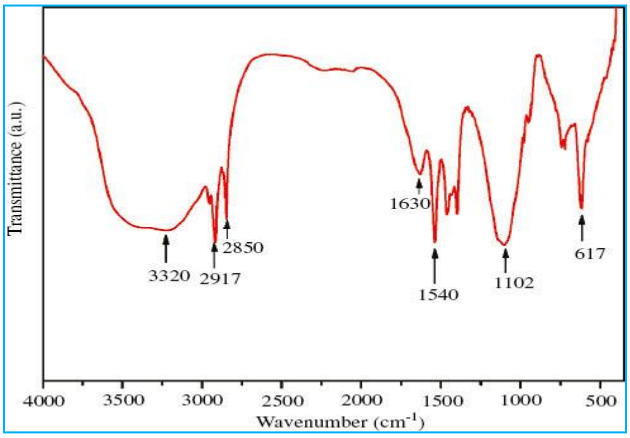


Fig 3 FTIR spectrum of synthesized zinc nanoparticle from *Trigonella foenum-graecum*

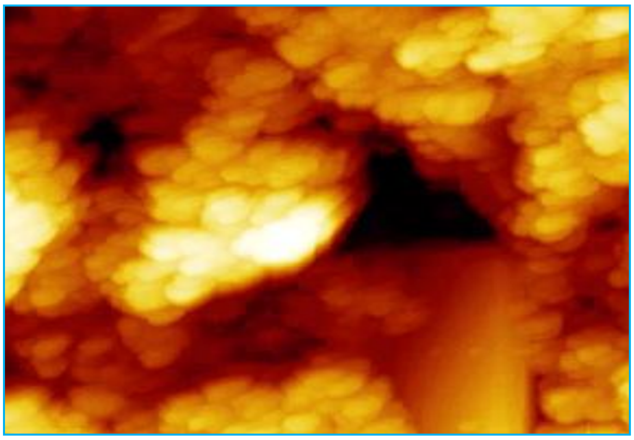

Fig 4 AFM image of synthesized zinc nanoparticle from *Trigonella foenum-graecum*

AFM was used to analyze the particle morphology (shape, size). AFM image of *Trigonella foenum-graecum* mediated synthesis of zinc nanoparticle shows that they have a uniformly packed surface and AFM imaging was conducted in different magnification ranges of 1, 2, 5 and 25 µm. Figure 30 shows the 3D AFM images of the plant extract mediated synthesis of nanoparticles. AFM (Fig 4) image clearly demonstrate smooth nanoparticle with capping of phyto-chemicals over the surface of nanoparticle.

XRD analysis is used to determine the phase distribution, crystallinity and purity of the synthesized nanoparticles. (Fig 5) shows the XRD patterns of *Tinospora cordifolia*. The XRD pattern of undoped zinc nanoparticles was uniform and no impurities were observed in the spectrum. Detailed structural characterizations demonstrate that the synthesized products are spherical and crystalline in structure and their diameter was about 30nm.

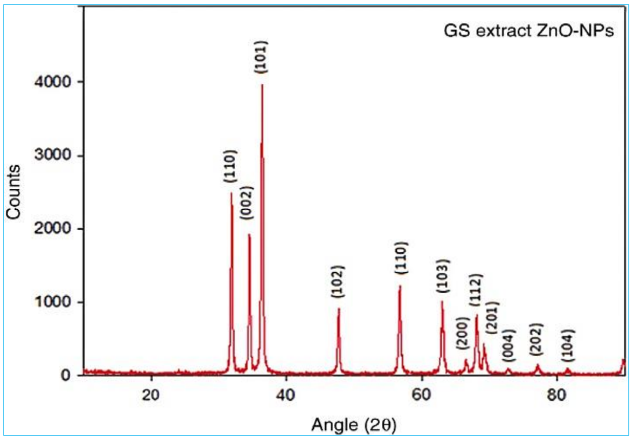


Fig 5 XRD patterns of synthesized zinc nanoparticle from *Trigonella foenum-graecum*

To the ethanol extract, Zinc nitrate 2.1%, Ammonium carbonate 0.96% and polyethylene glycol solution was added slowly drop wise in a molar ratio of 1:2 under vigorous stirring, and the stirring was continued for 12 hrs. The precipitate obtained was filtered and washed thoroughly with deionized water. The precipitate was dried in an oven at 100°C and ground to fine powder. Synthesis of zinc nanoparticles by using seed extract of *Trigonella foenum-graecum* medicinal plant has been demonstrated in present investigation. The reduction of zinc ions and their capping were achieved by the organic molecules present in the leaf extract. The UV-Vis, X-ray diffraction graph, SEM, AFM, FTIR results revealed that the zinc nanoparticles were spherical in shape and ranging from 30 to 63 nm in size. Scanning electron microscope was used to decide size, location and shape of the zinc oxide nanoparticles.

UV-visible spectroscopy is usually conducted to confirm the synthesis of zinc nanoparticles. Conducting electrons start oscillating at a certain wavelength range due to surface plasmon resonance effect. The occurrence of the peak at 380 nm is due to the phenomenon of surface Plasmon resonance, which occurs due to the excitation of the surface plasmons present on the outer surface of the zinc nanoparticles which gets excited due to the applied electromagnetic field. Myint Myint Khaing [7] reported that the zinc nanoparticles prepared using the extract obtained from the leaves of Neem and Golden shower are subjected to record UV-Vis spectroscopy. The absorption peaks is obtained at the wavelength 375 nm and 376 nm for Zn particles prepared from leaves of Neem and Golden shower.

SEM analysis is done to visualize shape and size of nanoparticle. A scanning electron microscope was employed to analyze the shape of the zinc nanoparticles that were synthesized by green method. SEM analysis shows that the *Trigonella foenum-graecum* have tremendous capability to synthesize zinc nanoparticles which were roughly spherical in shaped nanoparticle with average diameter of 70 nm and were uniformly distributed. The size of the Zn nanoparticles synthesized using neem leaf extracts was recorded to be 50 µm [8]. Amit Singha [9] reported that the SEM analyzes the comprehensive high-resolution images of the sample by detecting the secondary electron signal from the surface, which arises due to focusing of the electron beam over the surface. SEM is used to study the surface morphology of the resulting powder of zinc nanoparticles.

Noorjahan [10] reported that the FTIR spectrum of hot and cold methods of neem (*Azadirachta indica*) extracts of zinc oxide nanoparticles. The band at 437-445 cm-1 and 509-511cm-1 is attributed to ZnO nanoparticles. These bands are indicative of plant group of compounds present in aqueous neem (*Azadirachta indica*) extract [11-12]. From FTIR analysis, it can be inferred that alcohols, terpenoids ketones, aldehydes and carboxylic acid were surrounded by synthesized nanoparticles. Phenolic compounds flavonoids, lignans, coumarins, tannins, quercetin, alkaloids, cynogenic glycosides present in the leaves formed a strong capping on the nanoparticles [13]. The prominent doublet absorption at 2927-2931cm-1 indicates C-H stretching vibration of an aromatic aldehyde.

AFM was used to analyze the particle morphology (shape, size). AFM image of *Trigonella foenum-graecum* mediated synthesis of zinc nanoparticle shows that they have a uniformly packed surface and AFM imaging was conducted in different magnification ranges of 1, 2, 5 and 25 µm. The 3D AFM images of the plant extract mediated synthesis of nanoparticles. Prasanta Sutradhar and Mitali Saha [14] reported that the AFM showed the topo-graphic image of well-dispersed Zn nanoparticles. The luminescence of Zn nanoparticles is of particular interest from viewpoints of both physical and applied aspects. The excitation peaks corresponded to the band to band transition, which also confirmed the blue shift in the bandgap of Zn nanoparticles.

XRD analysis is used to determine the phase distribution, crystallinity and purity of the synthesized nanoparticles. Figure 5 shows the XRD patterns of *Trigonella foenum-graecum*. Detailed structural characterizations demonstrate that the synthesized products are spherical and crystalline in structure and their diameter was about 30nm. The crystallite size obtained was 23.59 nm [15]. The results confirmed the formation of nanoflowers of 51 nm. These zinc nanoparticles could be used as an inexpensive and effective adsorbent for the removal of arsenic ions from aqueous solution [8]. ZnO nanotubes were characterized by SEM and XRD. The final product was highly crystalline with the size of 25 nm. ZnO is non-toxic and used in industrial sectors including environmental, synthetic textiles, food, packaging, medical care, healthcare, as well as construction and decoration [10].

## CONCLUSION

*Trigonella foenum-graecum* is a herbaceous crop having a common name (Fenugreek). Seed extract of (Fenugreek) herb used for the biosynthesis of Zinc nanoparticles by green chemistry approach which is a cost effective and ecofriendly procedure. UV-Vis spectra was observed around 370 nm which confirmed presence of Zn nanoparticles. SEM revealed triangular shaped particles with size of 30nm, EDX along with SEM were also performed which confirmed the elemental composition to be of zinc metal. XRD confirmed the crystalline nature of the particles. Furthermore, these particles were also found pharmacological and cytotoxic in nature. So it was observed that biologically synthesized nanoparticles by Trigonella foenum can be used in different fields like medicinal, drug delivery, industrial and cosmetics industry in future.

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