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Eco friendly Synthesis of Lemon Extract ZnO nanoparticles by using green synthesis method and its Antioxidant Activity

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ABSTRACT

Green nanotechnology, which involves nanoparticle synthesis using plants, has garnered significant attention among researchers due to its environmentally friendly approach that eliminates the use of toxic chemicals, making it an ecofriendly alternative to conventional physical and chemical methods. In this study, zinc oxide nanoparticles (ZnO-NPs) were synthesized using lemon extract and zinc nitrate. The Lemon extract-mediated synthesis of ZnO particles was thoroughly investigated through various analytical methods, such as Fourier Transform Infrared Spectroscopy (FTIR), FE-SEM, Transmission Electron Microscopy (TEM), and X-ray diffraction (XRD). The XRD analysis revealed that the ZnO-NPs possessed a hexagonal (wurtzite) shape with an average size of 49 nm, as indicated by changes in the intensity peaks. TEM studies showed that the ZnO NPs exhibited an agglomerated and spherical shape. Respectively. This ecofriendly synthesis method holds promise for a wide range of applications, including medical applications, as well as its potential use in the preparation of LEDs and solar cells.

Key words: ZnO NPs, Green synthesis,

Introduction

ZnO NPs, Green synthesis, Because of its unique optical and electrical properties, zinc oxide (ZnO), a versatile metal oxide, is highly regarded for its appropriateness for nano-scale applications (Sauvik Raha *et al.*, 2022). Because of their enormous specific surface area, high fraction of surface atoms, and wide gap between the valence and conduction bands, nanoparticles have atom-like behaviours when reduced to near-atomic sizes (Dieter Vollath *et al.*, 2018). When several metal oxides were investigated for their antibacterial activities, zinc oxide nanoparticles (ZnO NPs) shown outstanding toxicity. Their stability under rigorous processing conditions and relatively low toxicity have made them

favorable for use in agricultural and food industries (Sangeetha *et al.*, 2011). This positions ZnO as a versatile material with a broad range of applications, including optoelectronics, various industrial sectors, pharmaceutical and cosmetic industries, solar energy materials (for solar cells), gas sensors, varistors, and as a photocatalyst (Michal *et al.*, 2019). In line with existing literature, the green synthesis of ZnO NPs using plant-based approaches has been explored, and in this context, Lemon extract has been employed. This study represents the first-time synthesis and characterization of ZnO NPs using Lemon extract and zinc nitrate, offering promising prospects for environmentally friendly nanomaterial production.

Experimental

Preparation of leaf extracts

A total of 30 lemons were meticulously cleaned, dried, and then placed in a 50 ml beaker filled with deionized water. This beaker, containing the lemons, was subsequently heated on a hot plate for a duration of half an hour. Afterward, the resulting extract was allowed to cool and was subsequently utilized as the extract solution.

Synthesis of ZnO nanoparticles

The Lemon ZnO nanoparticles were synthesized using the Solution Combustion method. In this procedure, citric acid served as the fuel agent. The process involved adding the fuel agent to 5g of zinc nitrate, which was then dissolved in 50 ml of distilled water. Subsequently, 10 ml of lemon extract was slowly added drop by drop to the solution containing citric acid and zinc nitrate. The resulting mixture was stirred for 2 hours on a magnetic stirrer, and then the stirred solution was subjected to 20 minutes of sonication. The final solution was calcined at 80°C for 5 hours. The calcined material of interest was ground into a fine powder using an agate mortar. The powdered lemon ZnO nanoparticles were then subjected to an additional calcination step at 700°C for 2 hours in a furnace. The outcome of this process was the production of white-colored lemon ZnO nanoparticles. This synthesis method was replicated for different concentrations of lemon extracts (10 ml, 20ml, 30 ml, 40 ml). The green synthesis of ZnO nanoparticles using the Solution Combustion method is environmentally friendly, cost-effective, and results in crystalline nanoparticles.

Characterization of ZnO NPs

The ZnO nanoparticles (ZnONPs) were subjected to characterization through FTIR observation, TEM analysis, and XRD measurement. An X'Pert Pro X-ray diffractometer from PANalytical BV in the Netherlands was used for XRD analysis in order to ascertain the formation of ZnONPs and determine the size of the crystallites. The Scherrer equation was employed for this purpose. For a more detailed examination of the nanoparticles and Transmission Electron Microscope (TEM) analysis was performed to verify that zinc and oxygen were present in the particles and to find any additional components that might have been present. This analysis utilized a

JEOL JEM-2100 high-resolution transmission electron microscope.

To identify the functional groups in the samples, a Nicolet Avatar 3330 FT-IR spectrometer was employed. The samples were analyzed in KBr pellets, covering a wavelength range of 4000–500 cm^{-1} . This comprehensive characterization approach allowed for a thorough understanding of the ZnO nanoparticles and their properties.

Results and Discussion

XRD

The crystalline nature of the ZnO nanostructure was verified by the XRD pattern, as seen in Fig. 1. (100), (002), (101), (102), (110), (103), (200), (112), and (201) are the observed peaks. Exhibited a wurtzite structure, which closely resembled the patterns obtained through both wet chemical methods and green synthesis using Lemon extract (Sangeetha *et al.*, 2011). Crucially, the complete composition of the precursors was confirmed by the absence of any diffraction patterns suggesting the presence of any additional elemental species. The average particle aggregate size was calculated using the Scherrer equation and was found to be roughly 16.28 nm. The size of the nanoparticles is responsible for the XRD patterns' broadening, and the presence of strong, narrow peaks indicates that the product has good crystallinity (Bindu *et al.*, 2014). This comprehensive XRD analysis provides valuable insights into the structural properties of the synthesized ZnO nanoparticles.

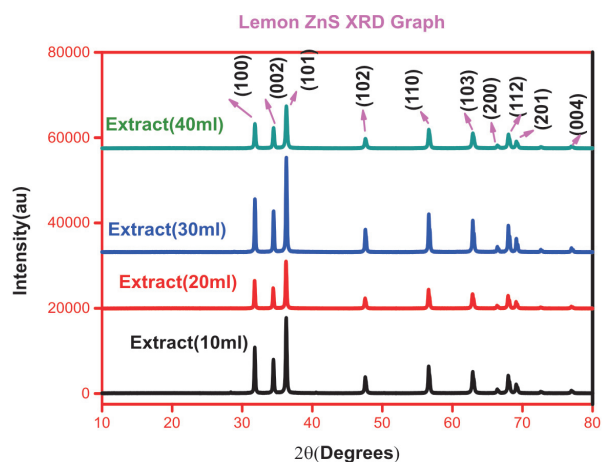


Fig. 1. XRD of Lemon ZnO nanoparticles

FE-SEM

The morphology of the synthesized Lemon ZnO was investigated using FESEM and the images at various magnifications are presented in Fig. 2. These images reveal that the ZnO particles predominantly exhibit a spherical shape. Furthermore, the particles were uniformly aggregated, leading to the formation of a hierarchical sheet-like appearance. This microstructural analysis offers valuable insights into the physical characteristics and arrangement of the ZnO particles.

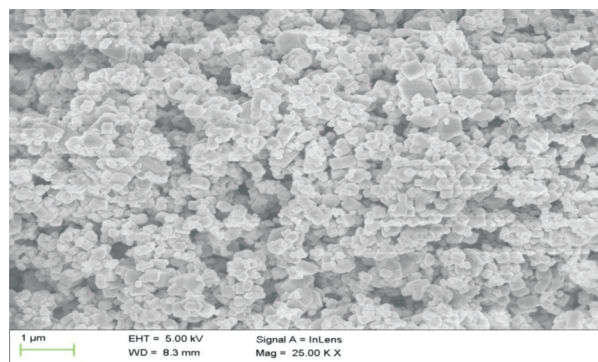


Fig. 2. FE-SEM image of Lemon(20 ml Extract) ZnO nanoparticles

TEM

The hexagonal (wurtzite) form and nanostructure of the ZnO nanoparticles (NPs) with a size of less than 20 nm were further validated by the TEM picture shown in Fig. 3. Because ZnO NPs have a high surface energy, they exhibit aggregation and spherical morphologies, which is a feature that usually happens when synthesis occurs in an aqueous medium

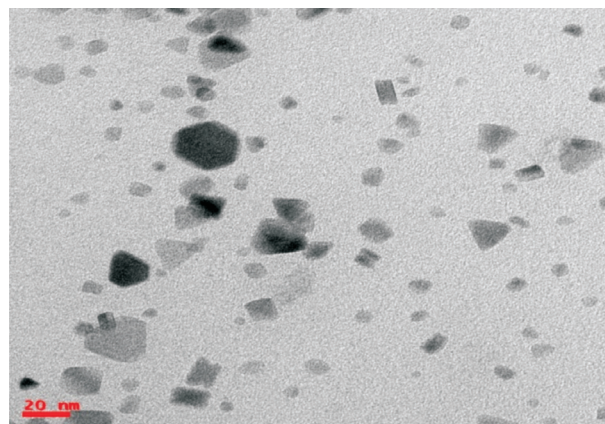


Fig. 3. TEM image of Lemon (20ml Extract) ZnO nanoparticles

(Alessio *et al.*, 2008). Additionally, this aggregation may be due to densification, resulting in a narrower space between the particles. These observations provide a detailed visual representation of the ZnO nanoparticles' morphology and structure.

FTIR Analysis

The ZnO nanoparticles (ZnONPs) and lemon extract FTIR spectra were acquired with an FT-IR device running in the percent transmittance mode at a resolution of 4000-500 cm^{-1} . Several absorption peaks and the functional groups that corresponded to them were found by the analysis: At 3001 cm^{-1} , weak absorption peaks were attributed to the N-H bend and O-H stretch functional groups. Additional, weaker bands seen at 2626 cm^{-1} and 886 cm^{-1} were identified as C-H (aromatics), C-H (alkanes), and CQC-H (alkynes). The aromatic rings' C-C stretching and CQC bending were linked to the absorption maxima at 1739 cm^{-1} and 1377 cm^{-1} , respectively. The aliphatic amines' C-N stretching mode was attributed to the peaks located at 1230 cm^{-1} and 1043 cm^{-1} . An absorption peak at 459 cm^{-1} indicated the presence of ZnO nanoparticles (Ramesh, *et al.*, 2015). These FTIR spectra provide insights into the chemical composition and functional groups present in both the Lemon extract and the synthesized ZnO nanoparticles, confirming the presence of ZnO in the latter.

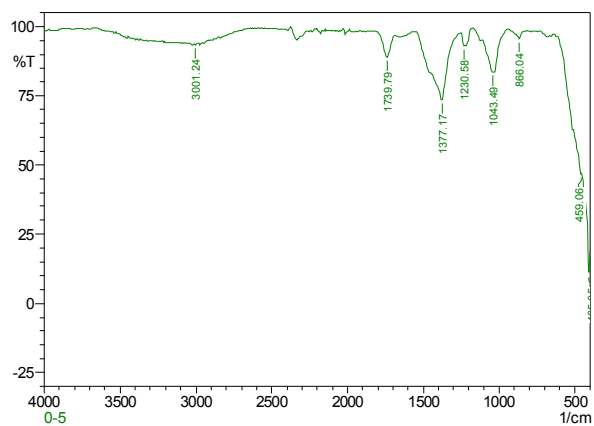


Fig. 4. FTIR Spectra of Lemon (20ml Extract) ZnO nanoparticles

Antioxidant activity

The produced ZnO NPs' antioxidant capacity was assessed in the manner listed below: Using the DPPH activity measurement method-which assumes the presence of an antioxidant compound-the

deep violet colour of DPPH turns yellow during the experiment. When hydrogen-donating substances are combined with DPPH, the result is a decrease in free radicals and a change in colour. Using the following formula, the DPPH free radical scavenging activity was calculated:

Inhibition percentage = (Control Absorbance - Sample Absorbance) $\times$ 100 / Control Absorbance

Lemons possess a natural, water-soluble pungent antioxidant property due to their high vitamin C content. This property helps enhance the body's immune system and combat free radicals and toxins. At low concentrations ranging from 1 to 5 mg/mL, the absorbance OD at 590 nm remains within the range of 0.0 to 0.3. This result contrasts with the high concentration of ZnO NPs.

Conclusion

Lemon extract was used in the green synthesis technology to create ZnO NPs, which is an easy and affordable process. The synthesised ZnO NPs were characterised by FTIR, TEM, and XRD analysis, among other methods. Agglomeration and sphericity were seen in the ZnO TEM examination, which was likely caused by the high surface energy of the ZnO NPs. Zinc oxide nanoparticle production was indicated by an absorption peak at 459 cm⁻¹ (Zn-O linkage) in the FT-IR investigations. The hexagonal zinc oxide structure with an average particle size of 49 nm was validated by XRD examination. The green synthesis approach is an efficient way to

synthesise ZnO nanoparticles, and the resultant ZnO NPs were found to exhibit a noteworthy antioxidant capability. They can also be used in a variety of sectors and to make solar cells and LEDs.

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