



International Journal of Research in Agronomy

E-ISSN: 2618-0618
P-ISSN: 2618-060X
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NAAS Rating (2026): 5.20
www.agronomyjournals.com
2026; 9(5): 431-435
Received: 23-02-2026
Accepted: 26-03-2026

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Synthesis of nanoscale ZnO and boron and their spectral and surface morphological characterization

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DOI: <https://www.doi.org/10.33545/2618060X.2026.v9.i5f.5540>

Abstract

Micronutrient deficiencies, particularly zinc (Zn) and boron (B), are major constraints to sustainable agricultural productivity due to their critical roles in plant growth, metabolism and reproductive development. Conventional micronutrient fertilizers often exhibit low nutrient use efficiency because of fixation, leaching and poor synchronization with crop demand. Nanotechnology has emerged as a promising approach to enhance nutrient delivery and improve fertilizer efficiency through the development of nano fertilizers with superior physicochemical properties. The present study was undertaken to synthesize and characterize nanoscale zinc oxide (ZnO) and boron particles for potential agricultural applications. Nanoscale ZnO was synthesized using the oxalate decomposition method, while nanoscale boron was prepared through the encapsulation technique. The synthesized nanoparticles were characterized using UV-Visible spectroscopy, Transmission Electron Microscopy (TEM) and Dynamic Light Scattering (DLS) analysis. UV-Visible spectral analysis confirmed nanoparticle formation by exhibiting characteristic absorption peaks at approximately 370 nm for ZnO and 320 nm for boron nanoparticles. TEM analysis revealed that ZnO nanoparticles were predominantly irregular to nearly spherical, whereas boron nanoparticles were mostly irregular and partially spherical in shape, with particle sizes below 100 nm. DLS analysis showed uniform particle distribution with average particle sizes of 54.7 nm for nanoscale ZnO and 14.2 nm for nanoscale boron, indicating good dispersion and stability of the synthesized nanoparticles. The study confirmed the successful synthesis of stable nanoscale ZnO and boron particles with desired characteristics and points to their suitability for application in biological sectors, agriculture in particular.

Keywords: Nanoparticle synthesis, nanoscale ZnO, nanoscale boron, characterization

Introduction

Different synthesis approaches have been employed for the preparation of metal and metalloid nanoparticles, including chemical precipitation, sol-gel, hydrothermal, sonochemical, microemulsion and green synthesis methods (Namakka *et al.*, 2023) [3]. Among these, chemical synthesis methods are widely preferred because of their simplicity, cost-effectiveness, scalability and ability to produce nanoparticles with controlled size and morphology (Srinivasan and Rana, 2024) [5]. However, the synthesis conditions such as precursor concentration, pH, temperature, reaction time and stabilizing agents greatly influence nanoparticle formation, crystallinity, stability and physicochemical characteristics (Namakka *et al.*, 2023) [3]. Therefore, optimization and characterization of nanoparticles are critical for understanding their behavior and suitability for agricultural applications (Bayda *et al.*, 2020) [1].

Comprehensive characterization of nanoparticles is essential to confirm successful synthesis and to evaluate their structural, morphological and surface properties (Namakka *et al.*, 2023) [3]. Techniques such as transmission electron microscopy (TEM), dynamic light scattering (DLS) and ultraviolet-visible (UV-Vis) spectroscopy are commonly used for nanoparticle characterization. TEM reveals particle morphology and size distribution. DLS measurement is important for evaluating particle size distribution.

Although several studies have reported the synthesis of zinc or boron nanoparticles individually, limited information is available regarding their comparative spectral and surface morphological characterization and potential agricultural relevance under a unified experimental framework.

Moreover, inconsistencies in synthesis protocols, particle stability and characterization approach often make it difficult to compare findings across studies. Therefore, systematic synthesis and characterization of nanoscale zinc and boron particles are necessary to develop efficient and stable nano nutrient formulations for agricultural applications.

The present study was undertaken to synthesize nanoscale zinc and boron particles using a suitable synthesis approach and to characterize their physicochemical properties using advanced analytical techniques. The study aims to evaluate the structural, morphological and stability characteristics of the synthesized nanoparticles and to assess their potential suitability for use as nano fertilizers in sustainable crop production systems.

Materials and Methods

Synthesis of nanoscale ZnO and boron

Preparation of nanoscale ZnO: Nanoscale ZnO was prepared by using the oxalate decomposition technique as described by Prasad *et al.*, 2012 ^[4].

Preparation of nanoscale boron: Nanoscale boron was prepared by using the encapsulation method as described by Jangam *et al.*, 2023 ^[2].

Characterization of nanoscale ZnO and boron

UV – Vis (UV-Visible spectra) analysis: The UV-visible spectra of this solution were recorded in Spectrophotometer (UV-2450, SHIMADZU), from 200 to 800 nm. This analysis was useful to identify the presence of nutrients and its phase. Sample was prepared by suspending 1mg of the nanoparticles in 5 mL of water and then estimated using UV-Vis spectrophotometer.

TEM analysis: For TEM analysis, the samples were prepared by drop casting the nanoparticles suspension on the carbon coated Cu grids and allowed samples to dry in air. The nanoparticles surface morphology and size were studied by using transmission electron microscopy [JEOL (JEM-1010)] with an accelerating voltage of 80 kV.

DLS (Dynamic Light Scattering) analysis: The aqueous suspension of the synthesized nanoparticles was filtered through a 0.22 µm syringe driven filter unit and the size and zeta potential of the distributed nanoparticles were measured by using the principle of Dynamic Light Scattering (DLS) technique made in a Nanopartica (HORIBA, SZ-100) compact scattering spectrometer. This information is useful to know the dispersion capacity of nanoparticles and stability of nanoparticles by negative zeta potential values.

Results

Synthesis of nanoscale ZnO and boron

The nanoscale zinc oxide and boron were prepared as per the methodology mentioned in material and methods. The synthesis procedure is briefly illustrated in figure 1 and 2. By subjecting each nanoscale nutrient to different characterization studies, it was confirmed by the particle size, shape and dispersible nature that the synthesized nanoparticles were found suitable for foliar application and for further investigations in agriculture

Characterization of nanoscale ZnO and boron

UV – Vis (UV-Visible spectra) analysis for nanoscale ZnO and boron

The UV-Visible absorption spectrum of synthesized nanoscale

zinc oxide (ZnO) showed a prominent absorption peak at approximately 370 nm (Fig 3a). This characteristic absorption peak is attributed to the electronic transition from the valence band to the conduction band, confirming the formation of ZnO nanoparticles. The presence of this peak indicates the nanoscale nature and strong optical activity of the synthesized particles. Thus, the UV-Visible spectral analysis confirmed the successful synthesis of nanoscale ZnO particles.

The UV-Visible absorption spectrum of synthesized nanoscale boron showed a prominent absorption peak at approximately 320 nm (Fig 3b). This characteristic absorption peak is attributed to the electronic transition of boron-related species, confirming the formation of boron nanoparticles. The presence of this peak indicates the nanoscale nature and strong optical activity of the synthesized particles. Thus, the UV-Visible spectral analysis confirmed the successful synthesis of nanoscale boron particles.

Transmission electron microscopy (TEM) analysis of nanoscale ZnO and boron

The Transmission Electron Microscopy (TEM) analysis of synthesized nanoscale zinc oxide (ZnO) is presented in Fig. 4a. The TEM micrograph revealed that the ZnO nanoparticles were predominantly irregular to nearly spherical in shape with slight agglomeration of particles. The particles appeared well distributed with clear boundaries, confirming the formation of nanoscale ZnO. Based on the bar scale (100 nm), the particle size was found to be in the nanoscale range (below 100 nm). The agglomeration observed may be due to the high surface energy and strong inter-particle attraction among the nanoparticles and also may be due to the absence of protecting ligands on the surface. The TEM results clearly confirmed the successful synthesis of nanoscale ZnO particles with uniform morphology with nanoscale dimensions.

The TEM micrograph of synthesized nanoscale boron shown in Fig. 4b confirmed the successful formation of boron nanoparticles with nanoscale dimensions. The particles were observed to be mostly irregular, and partially spherical in appearance with slight clustering in some regions. This aggregation may be due to strong surface attraction between the fine particles and also may be due to the absence of protecting ligands on the surface. The nanoparticles were clearly visible and uniformly distributed throughout the image, indicating good synthesis efficiency. The particle size was observed to be below 100 nm based on the scale bar, which confirms their nano range. Thus, TEM analysis verified the morphology, size, and successful synthesis of nanoscale boron particles.

Dynamic Light scattering of nanoscale ZnO and boron

The Dynamic Light Scattering (DLS) analysis of synthesized nanoscale zinc oxide (ZnO) is presented in Fig. 5a. The particle size distribution graph showed a single prominent peak, indicating uniform distribution of ZnO nanoparticles. The average particle size (mean diameter) was observed to be 54.7 nm. The narrow size distribution suggests good homogeneity and stability of the synthesized nanoparticles. The particles were found to be well dispersed within the nanometer range, confirming successful synthesis of ZnO nanoparticles. The absence of multiple peaks indicates minimal aggregation and high uniformity in particle size. Thus, DLS analysis clearly confirmed the nanoscale size and stability of synthesized ZnO particles.

The Dynamic Light Scattering (DLS) analysis of synthesized nanoscale boron is presented in Fig. 5b. The particle size distribution graph showed a single prominent peak, indicating

uniform distribution of boron nanoparticles. The average particle size (mean diameter) was observed to be 14.2 nm. The particles were found to be well dispersed within the nanometer range, confirming successful synthesis of boron nanoparticles. The presence of a single major peak indicated relatively uniform

particle distribution, whereas the higher polydispersity index suggested slight aggregation of particles in the colloidal suspension. Thus, DLS analysis clearly confirmed the nanoscale size of the synthesized boron particles.

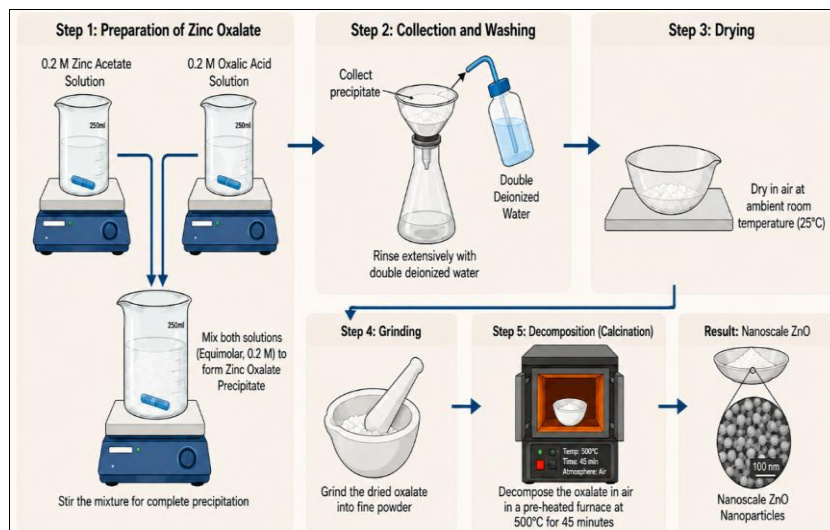


Fig 1: Synthesis of nanoscale ZnO by oxalate decomposition technique

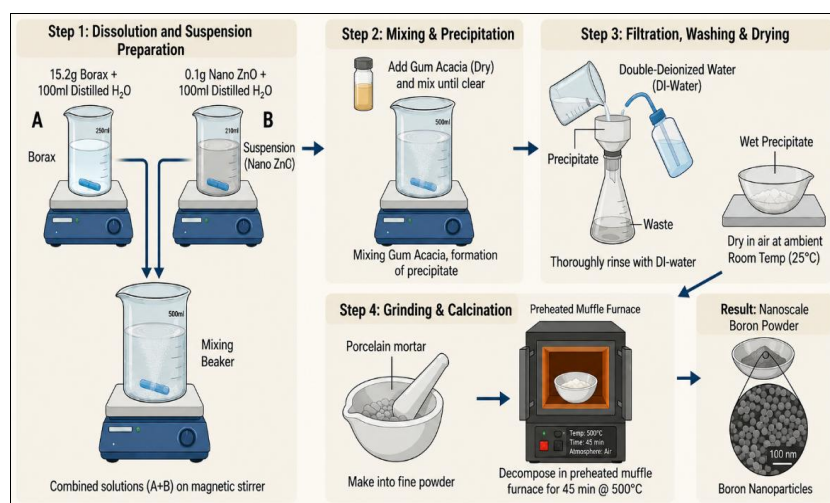


Fig 2: Synthesis of nanoscale boron by encapsulation method

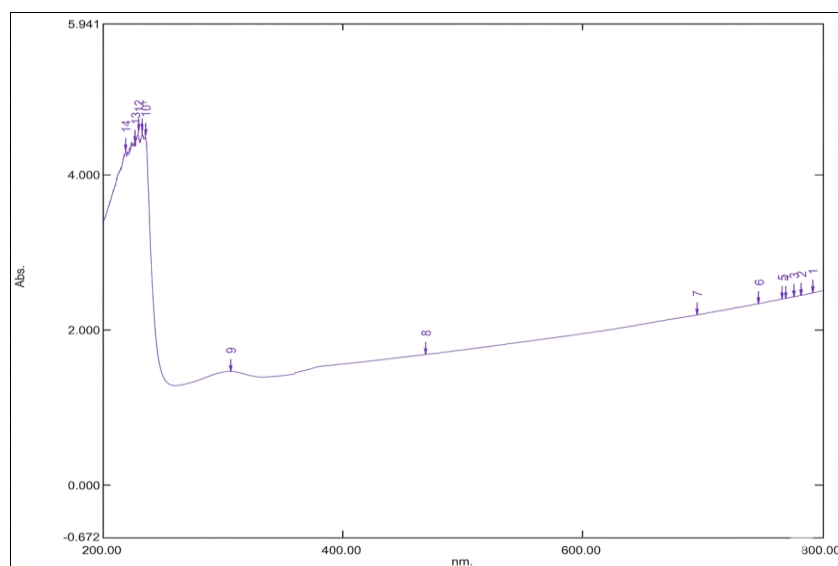


Fig 3a: UV-Vis absorption peak of nanoscale ZnO

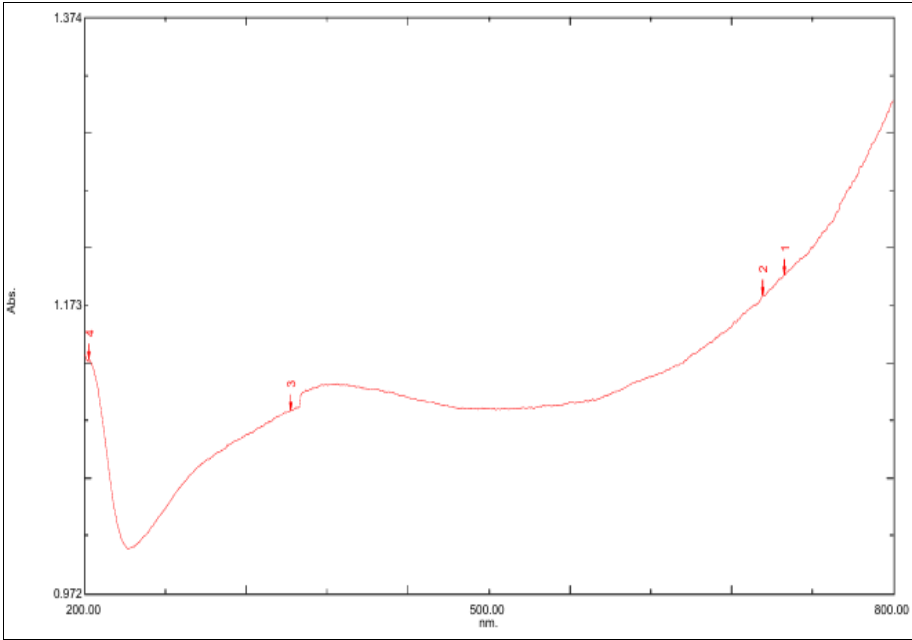


Fig 3b: UV-Vis absorption peak of nanoscale boron

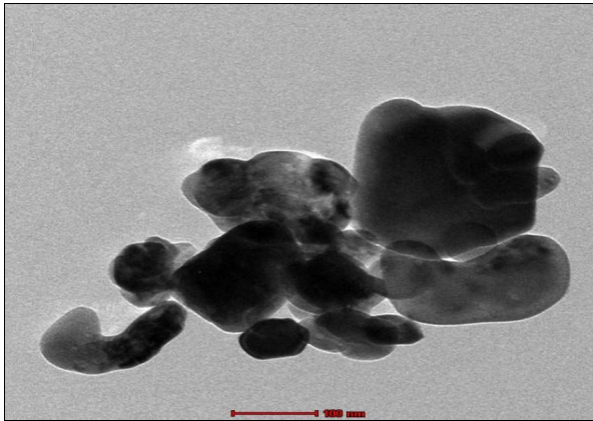


Fig 4a: TEM image of nanoscale ZnO (Bar scale 100 nm)

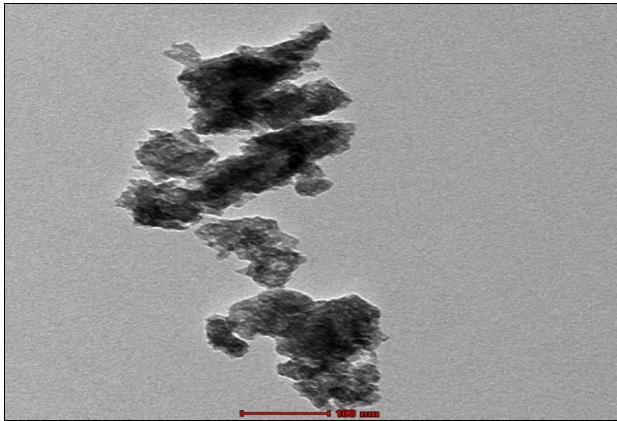


Fig 4b: TEM image of nanoscale boron (Bar scale 100 nm)

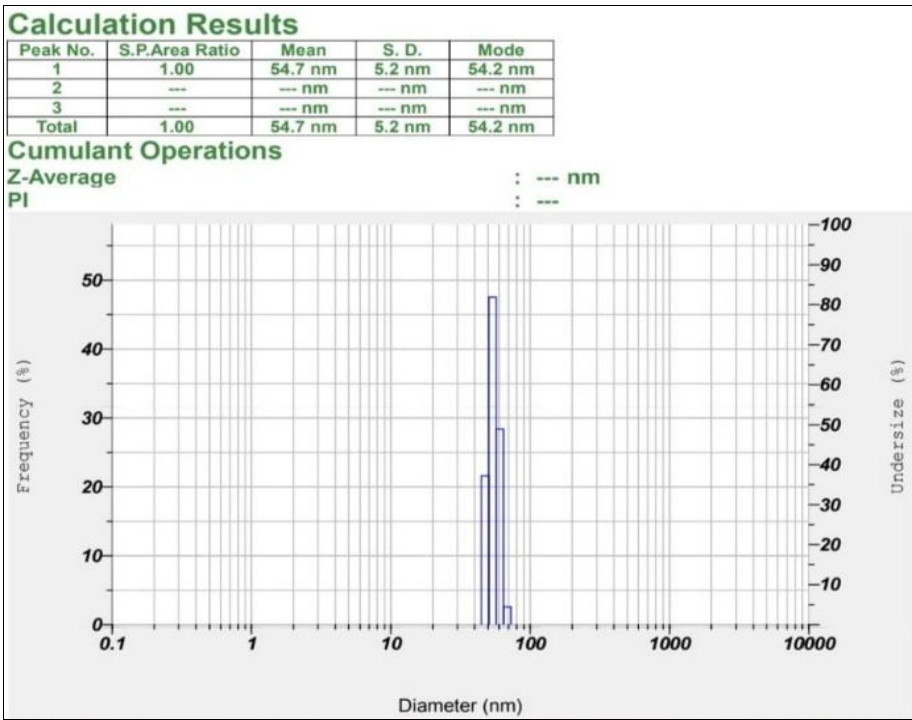


Fig 5a: Micrograph representing particle size distribution pattern of nanoscale ZnO (54.7 nm)

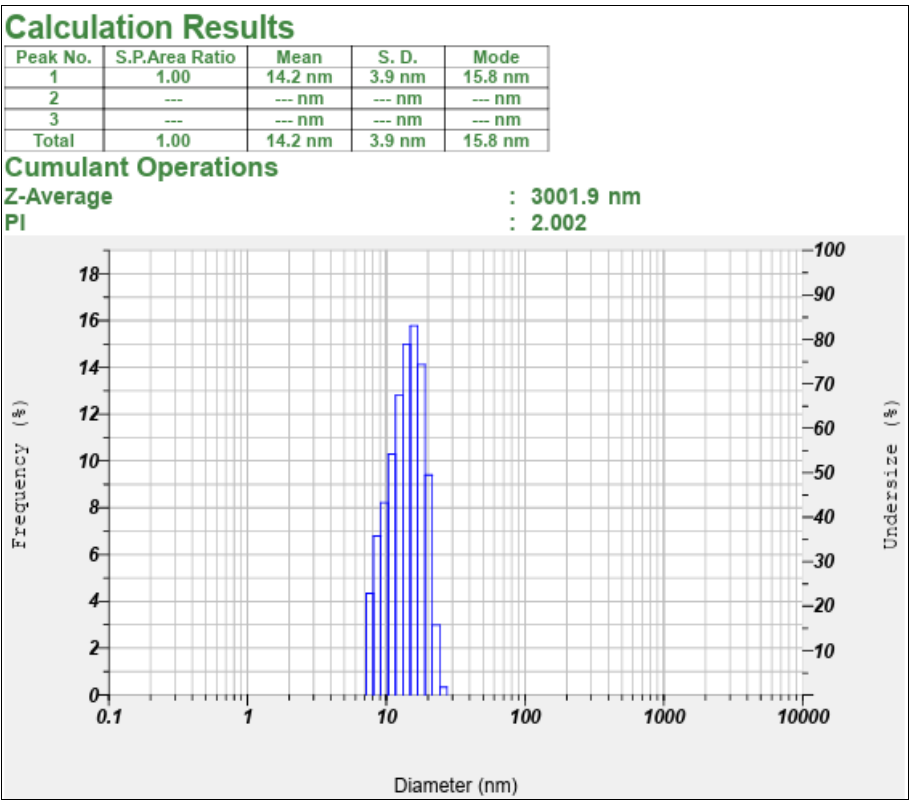


Fig 5b: Micrograph representing particle size distribution pattern of nanoscale boron (14.2 nm)

Conclusion

Successful synthesis of nanoscale zinc oxide (ZnO) and boron particles was achieved using oxalate decomposition and encapsulation methods, respectively. Characterization through UV–Visible spectroscopy, TEM and DLS analysis confirmed the formation of stable nanoparticles with desirable physicochemical properties. The characteristic absorption peaks at 370 nm for ZnO and 320 nm for boron verified successful nanoparticle formation. TEM analysis revealed predominantly irregular to nearly spherical nanoparticles with sizes below 100 nm, while DLS analysis confirmed uniform particle distribution and stability, with average particle sizes of 54.7 nm for ZnO and 14.2 nm for boron. The synthesized nanoparticles exhibited promising characteristics for use as nano fertilizer formulations and may enhance nutrient use efficiency and sustainable crop production.

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