



International Journal of Research in Agronomy

E-ISSN: 2618-0618
P-ISSN: 2618-060X
© Agronomy
NAAS Rating (2026): 5.20
www.agronomyjournals.com
2026; 9(6): 1192-1195
Received: 24-04-2026
Accepted: 28-05-2026

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Synthesis of nanoscale calcium oxide and its spectral and surface morphological characterization

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DOI: <https://www.doi.org/10.33545/2618060X.2026.v9.i6o.5850>

Abstract

Calcium (Ca) is an essential secondary nutrient required for maintaining cell wall stability, membrane integrity, root development and overall plant physiological activity. Nanotechnology is a promising approach for improving nutrient use efficiency through the development of nanoscale fertilizers with enhanced surface area, reactivity and effective nutrient delivery. The present study was undertaken to synthesize and characterize nanoscale calcium oxide particles for potential agricultural applications. Nanoscale calcium oxide particles were synthesized using the sol-gel method and as synthesized nano calcium oxide particles were characterized using UV-Visible spectroscopy, X-ray diffraction (XRD), Fourier Transform Infrared spectroscopy (FT-IR) and Transmission Electron Microscopy (TEM). UV-Visible spectroscopic analysis confirmed the presence of nanoscale calcium oxide by exhibiting characteristic absorption peaks at approximately 283 nm and 303 nm. XRD analysis revealed the crystalline nature of the synthesized calcium oxide nanoparticles, with major diffraction peaks observed at 2θ values of 101° which confirmed the formation of crystalline phase with face centered cubic structure (FCC). FT-IR analysis indicated the presence of functional groups associated with calcium oxide nanoparticles. TEM analysis (bar scale 50 nm) revealed that the synthesized nano calcium oxide particles were mostly irregular to nearly spherical in shape, with particle size below 100 nm. The study confirmed the successful synthesis of well defined nanoscale calcium oxide particles which may be suitable for application in biological sectors, agriculture in particular as a potential nano calcium fertilizer.

Keywords: Nanoparticle synthesis, nanoscale CaO, characterization

Introduction

Nanotechnology has emerged as an important tool in modern agriculture due to its ability to develop materials with improved reactivity, higher surface area and better nutrient delivery efficiency. Nanoparticles are generally defined as atomic or molecular aggregates with particle size below 100 nm in at least one dimension, and exhibit distinct physical, chemical and biological properties compared with their bulk counterparts (Nel *et al.*, 2006) [6]. In recent years, nanomaterials have gained considerable attention in agriculture, particularly for improving nutrient use efficiency, reducing fertilizer losses and enhancing crop growth under different environmental conditions (Ghormade *et al.*, 2011, Prasad *et al.*, 2011) [3, 7].

Calcium is an essential secondary nutrient required for plant growth and development. It plays a vital role in cell wall formation, membrane stability, cell division, root growth, pollen tube development and regulation of several physiological processes (Thor, 2019) [8]. Calcium also improves structural rigidity of plant tissues by forming cross-links in the pectin matrix of the cell wall and contributes to better fruit and seed development. In groundnut, calcium is particularly important for peg penetration, pod development, kernel filling and production of mature kernels. However, calcium is relatively immobile in the phloem and is mainly transported through the xylem (Hockings *et al.*, 2016) [4]. As a result, its movement from older leaves to developing reproductive parts is restricted, which may lead to calcium deficiency in growing tissues and developing pods.

Conventional calcium fertilizers such as gypsum, calcium nitrate and bulk calcium oxide are commonly used to correct calcium deficiency. However, their efficiency is often limited due to

poor mobility, low translocation through phloem and dependence on soil moisture for nutrient availability and movement (Deepa *et al.*, 2015) [2]. In groundnut, calcium deficiency may cause restricted root growth, poor pod development, reduced kernel filling and lower yield. Therefore, development of an efficient calcium source with improved mobility and availability is important for enhancing calcium nutrition in crop plants.

Nanoscale calcium oxide (nano CaO) has gained attention as a potential nano calcium fertilizer because of its small particle size, high surface area and improved biological reactivity. The nanoscale form of CaO may facilitate better absorption and translocation in plants compared with bulk calcium sources. Nanoscale calcium oxide particles could move through phloem tissue in groundnut and correct calcium deficiency more effectively than bulk calcium sources (Deepa *et al.*, 2015) [2]. This indicates the potential use of nano CaO as an efficient foliar calcium source in agricultural crops.

Several methods are available for the synthesis of metal oxide nanoparticles, including sol-gel, chemical precipitation, hydrothermal, Sono chemical, microemulsion and green synthesis techniques. Among these methods, the sol-gel method is widely preferred for the preparation of calcium oxide nanoparticles because of its simplicity, cost effectiveness, better control over particle size and ability to produce stable nanoparticles (Naseeruddin *et al.*, 2018) [5]. In the sol-gel approach, factors such as precursor concentration, temperature, stirring time, drying condition and stabilizing agent greatly influence the formation, crystallinity, stability and morphology of nanoparticles.

Characterization of synthesized nanoparticles is essential to confirm their formation and to understand their structural, spectral, functional and morphological properties. UV-Visible spectroscopy is used to study optical absorption and confirm nanoparticle formation. X-ray diffraction (XRD) analysis provides information on crystalline nature, phase purity and structural properties of the synthesized particles. Fourier Transform Infrared spectroscopy (FT-IR) helps to identify functional groups associated with calcium oxide nanoparticles. Transmission Electron Microscopy (TEM) is used to study surface morphology, shape and size distribution.

Although nanoscale calcium oxide has been reported as a promising source of calcium for agricultural crops, limited information is available on its synthesis and characterization using spectral, structural and surface morphological techniques. Hence, the present study was therefore undertaken to synthesize nanoscale calcium oxide particles using the sol-gel method and to characterize them through UV-Visible spectroscopy, XRD, FT-IR and TEM techniques.

Materials and Methods

Synthesis of nanoscale CaO

Preparation of nanoscale CaO

Nanoscale CaO was prepared by using the oxalate decomposition technique as described by Deepa *et al.*, 2014 [1]. Briefly, Nanoscale calcium oxide particles (n-CaO) were prepared using sol gel method. 1% of Calcium nitrate (Tetrahydrate purified LR, Sd-fine chemicals Ltd, Mumbai, India) was mixed with 0.05% of sodium citrate tribasic dehydrate (extra pure AR, Sd-fine chemicals Ltd, Mumbai, India) and stirred at 60 °C for 3 hrs. Then the solution was filtered using filter paper (Whatman no.1) and dried at 100°C for 6 hrs. The collected powder was used for further characterization and experimental studies.

Characterization of nanoscale CaO

UV – Vis (UV-Visible spectra) analysis

The UV-visible spectra of this solution were recorded (UV-2450,

SHIMADZU), from 200 to 800 nm. Sample was prepared by suspending 1mg of the nanoparticles in 5 mL of water and then estimated using UV-Vis spectrophotometer in transmittance mode.

Transmission electron microscopic (TEM) analysis

TEM analysis was performed to study the surface morphology of prepared nanoscale calcium oxide particles. 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.

X-Ray Diffraction (XRD) analysis

X-ray diffraction (XRD) analysis was performed on an X-ray Diffractometer (JEOL, and Model: JPX-8030) using CuK α radiation (1.5418 Å) in the range of 40 kV and 20 A in order to record the reflections from the respective crystal planes and to identify the corresponding crystal phase. The built-in software (syn master 7935) program was used for the identification of XRD peaks corresponding to the Bragg's reflections.

Fourier Transform Infrared Spectroscopic Analysis (FT-IR)

Fourier Transform Infrared spectroscopic analysis was carried out to identify the functional groups that are present in the hydrosol containing synthesized nanoscale calcium oxide particles. A small quantity of the sample was mixed with KBr, finely ground and pressed into a transparent pellet. The FT-IR spectrum was recorded in the range of 400–4000 cm⁻¹ using ATR technique. The spectrum was recorded in transmittance mode using an FT-IR spectrometer.

Results

Synthesis of nanoscale CaO

The nanoscale Calcium oxide was prepared as per the methodology mentioned in material and methods. The synthesis procedure is briefly illustrated in (fig. 1) By subjecting nanoscale calcium oxide to different characterization studies, its size, shape and surface morphology and crystalline phase were determined.

Characterization of nanoscale CaO

UV – Vis (UV-Visible spectra) analysis for nanoscale CaO

The UV-Visible absorption spectrum of synthesized nanoscale calcium oxide (nano CaO) showed distinct characteristic absorption peaks at approximately 283 nm and 303 nm, along with minor absorption bands at (Fig. 2). The prominent absorption peaks in the UV region confirm the formation of nanoscale CaO particles and indicate their optical activity. The absorption bands observed around 283–303 nm may be attributed to electronic transitions associated with calcium oxide nanoparticles and surface-related interactions of the synthesized particles. The presence of these characteristic absorption bands confirmed the presence of nanoscale calcium oxide particles.

Transmission electron microscopy (TEM) analysis of nanoscale CaO

The Transmission Electron Microscopic (TEM) analysis of synthesized nanoscale calcium oxide (nano CaO) is presented in (Fig. 3) The TEM micrograph (bar scale 50 nm) revealed that the CaO nanoparticles were predominantly irregular and aggregated in nature, with some particles appearing nearly spherical in shape. The particles were observed as clustered masses with visible agglomeration, which could be attributed to the high surface

energy and strong inter-particle attraction among nanoscale CaO particles as there are no protecting ligands on the surface. The particle size was found to be within the nanoscale range (the measured average size is 50 nm), confirming the successful synthesis of nanoscale calcium oxide particles. The TEM results clearly indicated that the synthesized Nano CaO particles possessed nanoscale dimensions with irregular size and with moderate agglomeration.

Fourier Transform Infrared Spectroscopic Analysis (FT-IR)

The Fourier Transform Infrared (FT-IR) spectrum of synthesized nanoscale calcium oxide (n-CaO) is presented in (Fig.5). The spectrum showed major absorption bands at 3652, 1498 and 511 cm^{-1} . The sharp band observed at 3652 cm^{-1} could be attributed to O–H stretching vibrations of surface hydroxyl groups. The strong absorption band at 1498 cm^{-1} could be assigned to carbonate-related stretching vibrations, which may occur due to the interaction of CaO nanoparticles with atmospheric CO_2 during

sample handling. The band observed at 511 cm^{-1} corresponds to O stretching vibration, confirming the formation of oxide nanoparticles. Thus, FT-IR analysis confirmed the presence of characteristic functional groups and metal–oxygen bonding in the synthesized nanoscale calcium oxide particles.

XRD (X-Ray Diffraction) analysis

The X-ray diffraction (XRD) pattern of synthesized nanoscale calcium oxide (nano CaO) is presented in (Fig. 4). The XRD spectrum showed several sharp and well-defined diffraction peaks, indicating the crystalline nature of the synthesized nanoparticles. The major diffraction peak was observed at 101. The intense peak observed at indicated the dominant crystalline phase of the synthesized calcium oxide nanoparticles and which could be indexed to a cubic structure (FCC). Thus, XRD analysis confirmed the crystalline nature and successful formation of synthesized nanoscale calcium oxide particles.

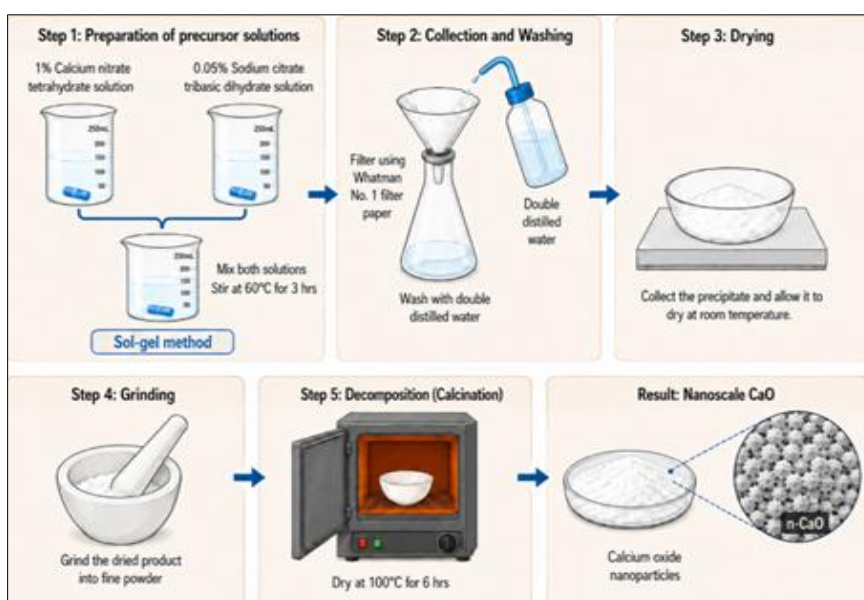


Fig 1: Synthesis of nanoscale CaO by Sol-gel method

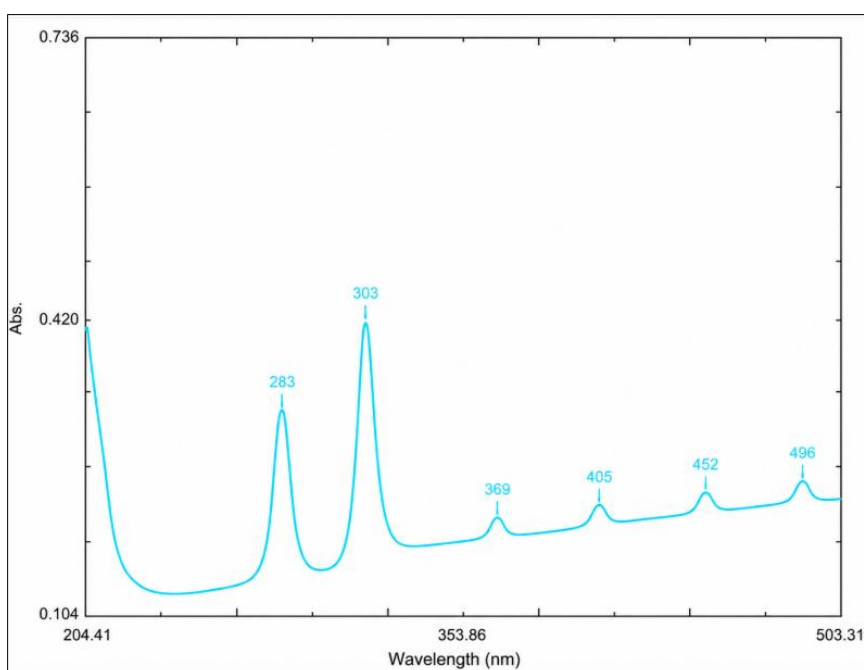


Fig 2: UV-Vis absorption peak of nanoscale Calcium

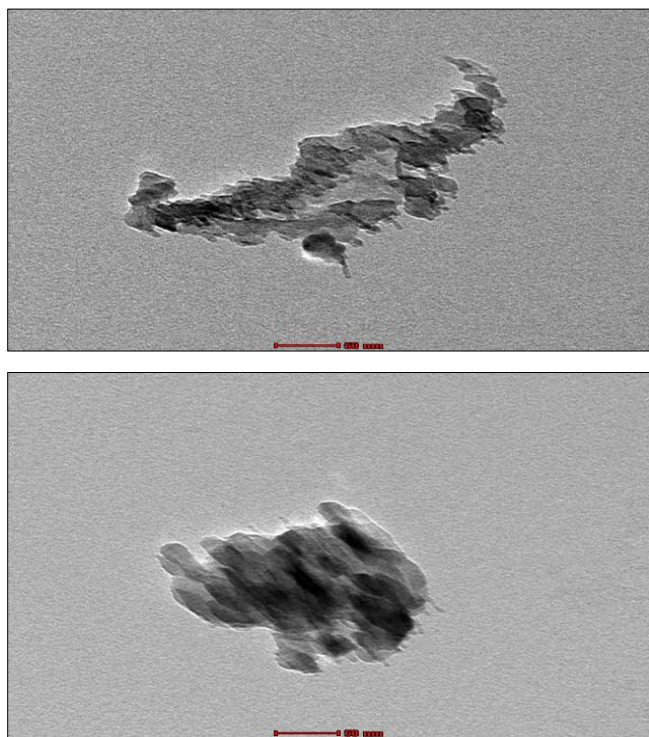


Fig 3: Both the TEM images of nanoscale CaO (Bar scale 50 nm)

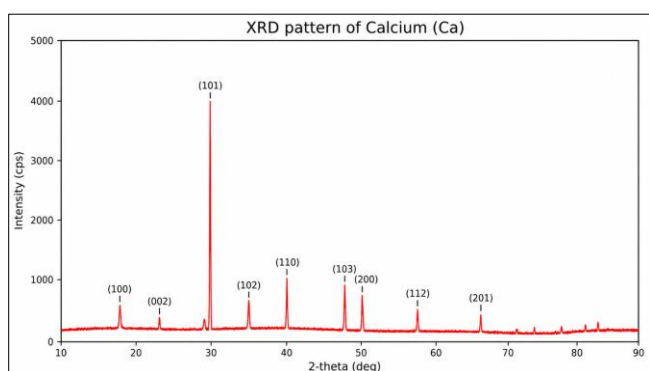


Fig 4: XRD 2-theta peaks of nanoscale Calcium

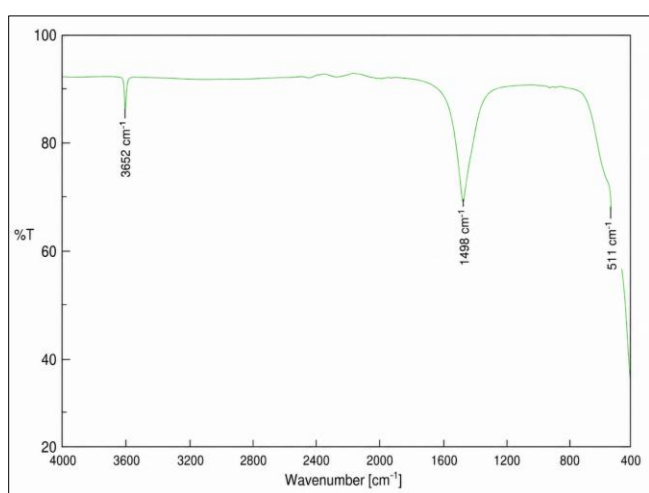


Fig 5: FT-IR wave length of nanoscale Calcium

Conclusion

In the present study, synthesis of nanoscale calcium oxide (n-CaO) particles was achieved using the sol-gel method. The synthesized particles were characterized using UV-Visible spectroscopy, X-ray diffraction (XRD), Fourier Transform

Infrared spectroscopy (FT-IR) and Transmission Electron Microscopic (TEM) techniques. The characterization results confirmed the successful formation of nanoscale calcium oxide particles with desirable spectral, structural, functional and morphological characteristics. Hence, the synthesized nano CaO particles may be considered for suitable applications in biological sectors, agriculture in particular as a potential nano calcium fertilizer.

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