

Article

Sustainably Synthesized CeO₂ Nanoparticles from Lemon Juice and Sucrose for Antibacterial Applications

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Abstract

Green synthesis of metal oxide nanoparticles is a promising route to reduce toxic reagents and energy consumption while enabling biocompatible nanomaterials for biomedical use. In this work, cerium oxide (CeO₂) nanoparticles were synthesized using lemon juice and sucrose as bio-based chelating, capping and stabilizing agents. Three synthesis routes were designed by varying the use of lemon juice, sucrose, or their combination. The synthesized materials were characterized using thermal analysis (DSC—Differential Scanning Calorimetry and TGA—Thermogravimetric Analysis), X-ray diffraction (XRD), Raman spectroscopy, and scanning electron microscopy (SEM). Additionally, their antibacterial activity was assessed against Gram positive bacterium *Staphylococcus aureus* (*S. aureus*). Thermal analysis showed that heat treatment at 600 °C promotes high crystallinity, as evidenced by the development of sharp diffraction peaks associated with the cubic fluorite CeO₂ structure, and a dominant F_{2g} Raman mode at 463 cm^{−1}. SEM micrographs revealed nanometric particles and highlighted that combining lemon juice and sucrose effectively suppresses coalesced structures, yielding more homogeneous morphologies. Crystallite size calculations gave average sizes of 17.2 nm, with the lemon juice-only route producing the largest crystallites. Antibacterial tests revealed a clear dose-dependent inhibition of *S. aureus*, with marked inhibition of bacterial growth at concentrations ≥5 mg/mL and a plateau effect above 25 mg/mL. This study confirms the feasibility of using plant-based extracts as sustainable reagents for CeO₂ nanoparticle synthesis, with promising structural and biological performance for potential biomedical applications.

Keywords: green synthesis; cerium oxide nanoparticles; lemon juice; sucrose; antibacterial activity



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1. Introduction

Nanotechnology has enabled the design of nanomaterials with size dependent physicochemical properties that are unattainable in their bulk counterparts, opening opportunities in biomedicine, catalysis, environmental remediation, and energy systems [1–4]. Metal and metal oxide nanoparticles are particularly attractive due to their high surface-to-volume ratio, tunable surface chemistry, and versatile optical, electronic, and catalytic behavior, which can be engineered through precise control of size, morphology, and crystallinity [5,6].

However, conventional synthetic routes rely on harsh conditions, toxic precursors, and energy-intensive processing, raising concerns about environmental impact and biocompatibility in biomedical applications [7].

Green synthesis has therefore emerged as a sustainable alternative that aligns with the principles of green chemistry by minimizing hazardous reagents and reducing waste [8]. In this approach, bio-based materials serve as chelating, stabilizing, and capping agents, offering a cost-effective and nontoxic platform for nanoparticle production [9,10]. Compared with purely chemical methods, plant-mediated syntheses frequently improve biocompatibility through the adsorption of functional phytochemicals onto the nanoparticle surface, which is advantageous for antimicrobial applications [11]. Recent studies have highlighted citrus-derived extracts as particularly promising green reagents, since peels and juices are rich in phenolic compounds, flavonoids, and organic acids capable of driving nucleation and growth of metal oxide nanoparticles while valorizing agri-food residues [12,13].

Among metal oxides, cerium oxide (CeO_2) has attracted growing interest due to its redox behavior associated with the $\text{Ce}^{3+}/\text{Ce}^{4+}$ couple and its high density of oxygen vacancies [14]. These characteristics confer enzyme-mimetic catalytic activities, including superoxide dismutase- and catalase-like behavior, enabling CeO_2 nanoparticles to scavenge reactive oxygen species (ROS) [15–17]. Conversely, under specific physicochemical and environmental conditions, CeO_2 nanoparticles may also exhibit oxidase- and peroxidase-like activities, promoting ROS generation and contributing to their antibacterial properties [18]. In the last few years, a variety of plant-mediated strategies have been reported for the synthesis of CeO_2 nanoparticles using fruit juices and peels, including watermelon and different citrus species, with demonstrated antibacterial or antioxidant performance [19–22]. Nevertheless, most works focus either on a single type of extract or on generic citrus peels, with limited systematic comparison of how specific bio-based additives and their combinations influence CeO_2 phase formation, crystallinity, and morphology.

Lemon juice is an abundant, low-cost source of citric acid and polyphenols that simultaneously act as chelating [23] and complexing agents [24]. Similarly, sucrose is a widely available carbohydrate that can function as a benign capping and structure-directing agent in green syntheses [23,24].

Although carbohydrate sugars (fructose, lactose and glucose) have been used to produce CeO_2 nanostructures with acceptable cytocompatibility [25], the potential synergy between crude lemon juice and sucrose in a single synthesis route has not been explored in detail. Specifically, there is a need to correlate the combined action of these natural agents with the evolution of phase purity and morphology.

The novelty of this study lies in the systematic evaluation of a multi-component green precursor system. We report the synthesis of CeO_2 nanoparticles using lemon juice (LJ), sucrose (S), and their combination (LJ+S) as bio-based chelating, capping, and stabilizing agents, followed by thermal treatments at 350 °C and 600 °C. By systematically comparing these three formulations, we elucidate how the precursor chemistry and calcination temperature govern crystallinity, secondary phase formation, and morphology using DSC/TGA, XRD, Raman spectroscopy, SEM, and Scherrer-based crystallite size analysis. To the best of our knowledge, this is the first study combining lemon juice and sucrose in a single green synthesis route for CeO_2 . The role of these bio-based reagents in controlling nanoscale structural and morphological features was examined, and antibacterial activity against *Staphylococcus aureus* was assessed for the sample exhibiting the most favorable morphology.

2. Materials and Methods

2.1. Materials

The synthesis of CeO₂ nanoparticles involved the use of Cerium (III) Nitrate Hexahydrate (Ce(NO₃)₃·6H₂O, ≥99%, St. Louis, MO, USA), sodium hydroxide (NaOH) and commercial-grade sucrose (99.7% purity). The lemons used for the preparation of the lemon juice were collected in Coimbra, Portugal, in March. All aqueous solutions were prepared using deionized ultrapure water, self-produced using ultrapure water equipment.

2.2. Extract Preparation and Nanoparticle Synthesis

Lemons were peeled, and the flesh was homogenized in a food processor (during 10 min) to produce a crude extract. To retain the full profile of natural chelating, capping and stabilizing agents, the unpurified whole-fruit homogenate was utilized directly for the synthesis.

Three synthesis conditions were evaluated: lemon juice alone (LJ), sucrose alone (S), and a combination of both (LJ+S). For each condition, 8.0 g (0.0184 mol) of the cerium precursor (Ce(NO₃)₃·6H₂O) was used. The reaction mixtures were designed around a 1:3 molar ratio of cerium precursor to the natural stabilizing agents [25]. Specifically, the LJ sample contained 18.9 g of crude lemon extract in 100 mL of water; the S sample contained 18.9 g (0.055 mol) of sucrose in 100 mL of water; and the LJ+S sample combined 18.9 g of extract and 18.9 g of sucrose in 100 mL of water.

The prepared mixtures were heated to 60 °C under continuous magnetic stirring. The cerium precursor was gradually added over 10 min. A 20 g/L NaOH solution was subsequently added dropwise to adjust the pH until stabilization, resulting in observable changes in the viscosity and color of the systems, consistent with the formation of a viscous sol that gradually evolved into a gel-like network (Table 1). Stirring was maintained for 10 min post-stabilization, after which the mixtures were allowed to settle overnight.

Table 1. Initial and final pH values, along with the corresponding visual evolution (color and viscosity changes) of the reaction mixtures during the sol–gel–derived synthesis of CeO₂ nanoparticles for the different compositions (S, LJ, and LJ+S).

Sample	pH (Initial–Final)	Visual Evolution
S	4.8–11.2	Transparent → golden → gray coloration with increased viscosity Increase in yellow coloration and viscosity Increase in yellow coloration and viscosity
LJ	0.98–7.6	
LJ+S	1.1–5.3	

The resulting materials were washed twice with 200 mL of deionized water, aided by 30 min of ultrasonication per wash. Finally, the samples were dried in an oven at 120 °C for 5 h at a heating rate of 5 °C/min. This drying cycle was performed twice for the LJ and LJ+S samples, and three times for the S sample. The green synthesis workflow is illustrated in Figure 1.

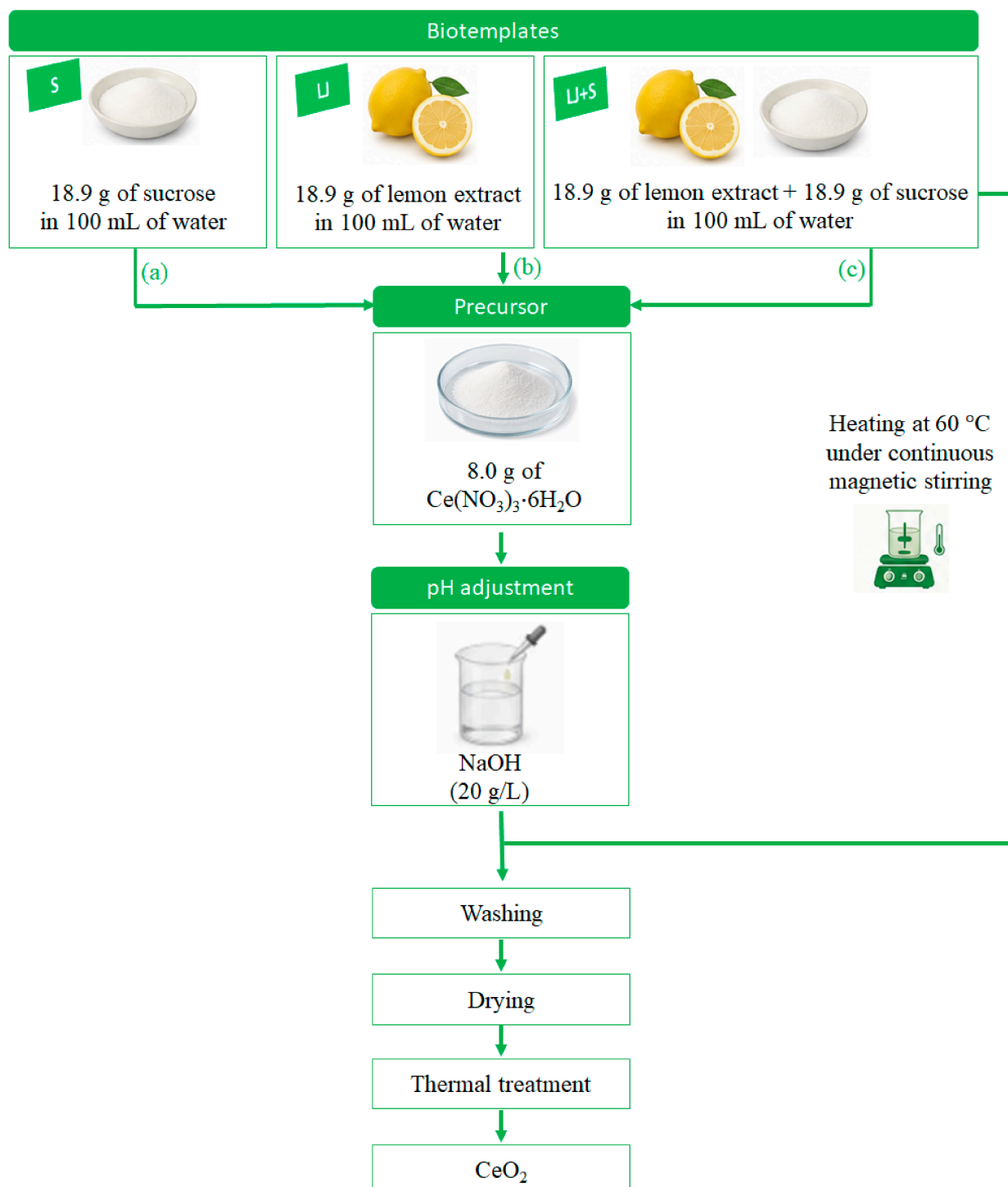


Figure 1. Schematic representation of the synthesis routes for the (a) S, (b) LJ, and (c) LJ+S samples.

2.3. Characterization Techniques

Thermogravimetric analysis (TGA) was performed using a Netzsch-Gerätebau GmbH TG 209 F1 Libra (Selb, Germany) under a nitrogen atmosphere (30–1095 °C, 5 °C/min). Differential scanning calorimetry (DSC) was conducted using a Netzsch-Gerätebau GmbH DSC 204 F1 Phoenix (Selb, Germany) under nitrogen (25–500 °C, 5 °C/min).

Raman spectroscopy was performed using a Renishaw plc inVia™ (Wotton-under-Edge, UK) confocal spectrometer with a 532 nm green laser. X-ray diffraction (XRD) patterns were acquired using a Rigaku Corporation Smartlab diffractometer (Tokyo, Japan) with Cu

K α radiation ($\lambda = 1.54060 \text{ \AA}$) operating at 40 kV and 50 mA, with Bragg-Brentano geometry and a step size of 0.02° .

Scanning electron microscopy (SEM) was performed in secondary electron mode using a Zeiss Merlin microscope (Zeiss, Oberkochen, Germany), at a magnification of 10.00 and 35.00 k $\times$ and an accelerating voltage of 1.00 kV.

2.4. Antibacterial Activity Assay

The antibacterial activity of CeO₂ nanoparticles from the LJ+S sample was evaluated against the Gram-positive bacterium *Staphylococcus aureus* (ATCC 6538).

S. aureus cells were stored at -80°C in Tryptic Soy Broth (TSB, Frilabo, Milheirós, Portugal) supplemented with 15% (*v/v*) glycerol. Prior to experimentation, the cells were streaked onto Tryptic Soy Agar (TSA, Frilabo, Milheirós, Portugal) plates and incubated at 37°C for 36 h. A single colony was then selected, inoculated into TSB, and incubated at 37°C for 18 h with agitation at 120 rpm. The resulting culture was diluted in TSB to achieve a final concentration of 7×10^6 CFU/mL.

CeO₂ NP suspensions were prepared at concentrations ranging from 5 to 60 mg/mL by weighing the appropriate mass of nanoparticles (10–120 mg) under sterile conditions. Subsequently, 1 mL of the bacterial suspension (7×10^6 CFU/mL) was added to each well of a sterile 24-well culture plate. In parallel, 1 mL of the same bacterial suspension was added to microcentrifuge tubes containing the pre-weighed CeO₂ NPs and thoroughly mixed to ensure homogeneous dispersion. The resulting mixtures (total volume of 2 mL) were then transferred to the corresponding wells of the 24-well plate. A control consisting of 2 mL of bacterial suspension without nanoparticles was included. Plates were incubated at 37°C for 24 h under orbital shaking (120 rpm). The pH of the untreated control and CeO₂ nanoparticle suspensions was measured after 24 h of incubation using a calibrated pH meter to evaluate whether changes in medium pH could contribute to the observed antibacterial effects.

Following incubation, bacterial viability was quantified using the colony-forming unit (CFU) counting method. Serial dilutions (10^{-1} to 10^{-6}) were prepared in phosphate-buffered saline (PBS), and 10 μL aliquots of each dilution were plated onto TSA. After incubation at 37°C for 24 h, visible colonies were enumerated. The bacterial concentration was calculated using the following equation [26]:

$$\text{CFU/mL} = \frac{\text{Number of colonies} \times \text{Dilution factor}}{\text{Volume plated (mL)}} \quad (1)$$

All measurements were performed in duplicate, and the entire experiment was repeated in at least two independent assays to ensure reproducibility. Results were analyzed using two-way analysis of variance (ANOVA), followed by Dunnett's multiple comparisons test to compare each treated group with the untreated control. Analyses were performed using GraphPad Prism software (GraphPad Prism 8.0.2). A *p*-value < 0.05 was considered statistically significant.

3. Results and Discussion

3.1. DSC/TGA Analysis

The thermal behavior of the S, LJ, and LJ+S samples was evaluated to identify appropriate calcination temperatures based on their thermal decomposition profiles. The DSC and TGA results are presented in Figure 2.

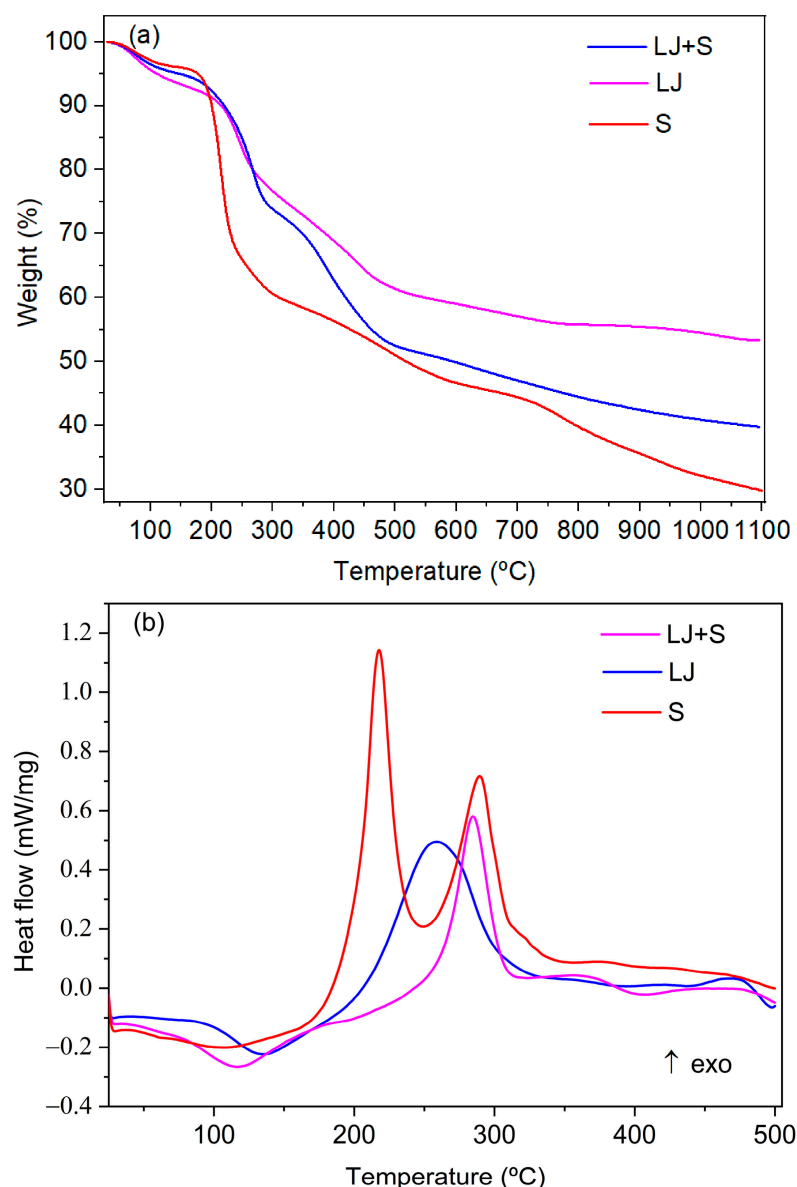


Figure 2. (a) TGA and (b) DSC analysis of the three samples prepared (S, LJ, and LJ+S). The upward arrow in (b) denotes the exothermic direction.

For the S sample, the DSC reveals a slight endothermic peak around 100 °C, which can be attributed to the evaporation of residual water and volatile organic components [27], followed by two exothermic peaks at 220–250 °C and 300 °C, indicating significant exothermic reactions, potentially linked to the formation of the CeO₂ phase. The TGA corroborates these findings, showing an initial mass loss between 50–150 °C, followed by a substantial mass decrease between 200–400 °C, which aligns with the exothermic peaks in the DSC. Beyond this range, mass loss continues gradually up to 1000 °C, albeit at a lower rate.

In the DSC of LJ, an endothermic peak is observed between 100–150 °C, possibly associated with the loss of residual water and volatile organic components, followed by a well-defined exothermic peak at 250 °C, initially suggesting the formation of CeO₂. However, the TGA profile indicates a continuous mass loss extending up to 600 °C, which signifies that chemical decomposition and organic burnout remain ongoing beyond this temperature. When correlated with XRD data discussed later, these results refute the hypothesis that stable, crystalline CeO₂ is fully formed at 250 °C.

For the LJ+S sample, the DSC exhibits an initial decline at 100–150 °C, consistent with water loss, followed by an exothermic peak at 250–300 °C, suggesting the formation of CeO₂. The TGA supports this assumption, displaying a significant mass loss between 200–400 °C, coinciding with the endothermic peak in the DSC, further reinforcing the likelihood of chemical reactions occurring within this temperature range. Above 600 °C, mass stabilization indicates that the material reaches a stable state.

Based on these observations, 350 °C was selected for the initial thermal treatment. Although mass loss continued beyond this threshold in all cases, the DSC analysis was limited to a maximum temperature of 500 °C. Consequently, 350 °C was identified as a critical point where the gradual mass loss likely corresponds to the thermal decomposition and the onset of crystallization associated with the formation of the CeO₂ phase [28]. However, literature indicates that increasing the temperature to 600 °C is necessary to remove residual carbon-based impurities from the nanoparticle surface, although this may increase the risk of particle agglomeration [29]. The two temperatures are evaluated to determine their influence on phase crystallinity, purity, and morphology, thus establishing the justification for the selection of the sample used in subsequent biological analyses.

3.2. XRD Analysis

The XRD patterns for the three samples treated at 350 °C and 600 °C are shown in Figures 3a and 3b, respectively. A comparative analysis reveals significant structural evolution as a function of the calcination temperature. In all samples (S, LJ, and LJ+S), the diffraction peaks align with the characteristic indices of the face-centred cubic fluorite-type structure [30] of CeO₂ (ICCD 01-001-0800) shown in Figure 3b.

For the first calcination temperature (350 °C), the diffraction patterns exhibit broad and poorly defined peaks, indicating low crystallinity and/or very small crystallite size, with a possible contribution from amorphous material. In contrast, increasing the thermal treatment to 600 °C (Figure 3b) resulted in a dramatic improvement in the diffraction profiles. The peaks for all three samples remain centered at the same 2θ positions but appear significantly narrower and sharper. This transformation reflects enhanced crystallinity and the promotion of crystallite growth as the thermal energy facilitates the reorganization of the Ce-O lattice and the removal of surface defects [31].

A notable deviation was observed in the S sample at 600 °C, where a new diffraction peak (2θ ≈ 34°) emerged that was not present at the lower temperature. This auxiliary peak indicates the formation of a secondary crystalline phase. Given the synthesis conditions, specifically the use of NaOH for pH adjustment and the carbon-rich nature of the sucrose precursor, this phase is tentatively identified as sodium bicarbonate (NaHCO₃). The position of this secondary reflection aligns with the most intense diffraction peaks reported for NaHCO₃ in the literature [32]. At 350 °C, this phase was likely either amorphous or masked by the broad reflections of the poorly crystallized CeO₂ matrix [33,34]. These results demonstrate that the thermal treatment at 600 °C is significantly more effective than 350 °C for producing highly crystalline CeO₂ nanostructures.

To quantify the influence of the various green precursors on the resulting nanostructure, the average crystallite size (*D*) of the samples calcined at 600 °C was estimated using the Scherrer equation [35]:

$$D = \frac{K \lambda}{\beta \cos \theta} \quad (2)$$

where *K* is the shape factor (0.94), *λ* is the X-ray wavelength (1.5406 Å), *β* is the full width at half maximum (FWHM) of the (111), (200), and (220) planes, and *θ* is the Bragg angle.

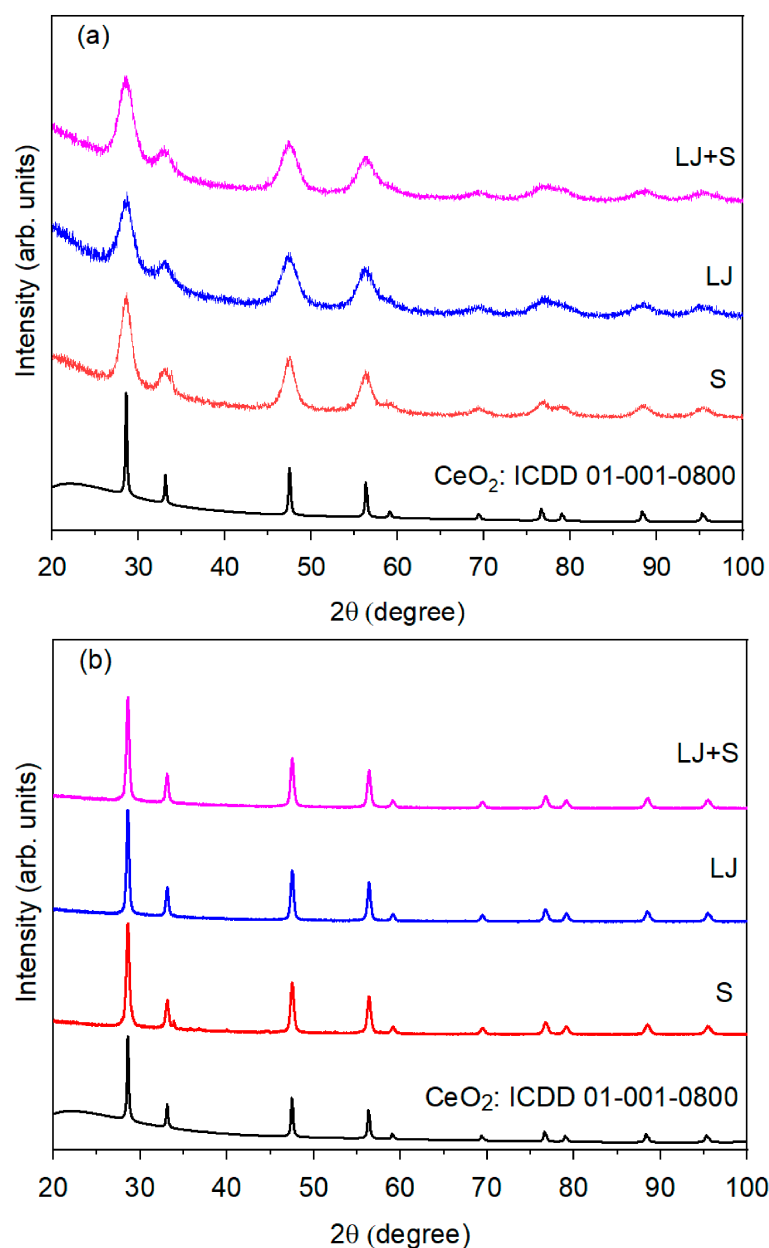


Figure 3. X-ray diffraction patterns of the three samples (S, LJ, and LJ+S) after heat treatment at (a) 350 °C and (b) 600 °C, with a reference CeO₂ diffraction pattern (ICDD 01-001-0800) included for comparison.

The average crystallite sizes ranged from 15.2 nm to 17.2 nm. The LJ sample yielded the largest crystallites (17.2 nm), while the addition of sucrose in the LJ+S (15.8 nm) and S (15.2 nm) samples resulted in a slight reduction in size. This narrow distribution suggests that while the lemon juice extract facilitates crystallite growth, sucrose acts as a mild capping agent that stabilizes the nanostructure. This aligns with previous reports on carbohydrate sugars and sucrose as green capping/stabilizing agents for CeO₂ nanoparticles [25,36].

3.3. Raman Spectroscopy

Raman spectroscopy was employed to investigate the structural properties and phase purity of the synthesized CeO₂ nanoparticles. Figure 4 displays the Raman spectra for the S, LJ, and LJ+S samples calcined at 350 °C and 600 °C.

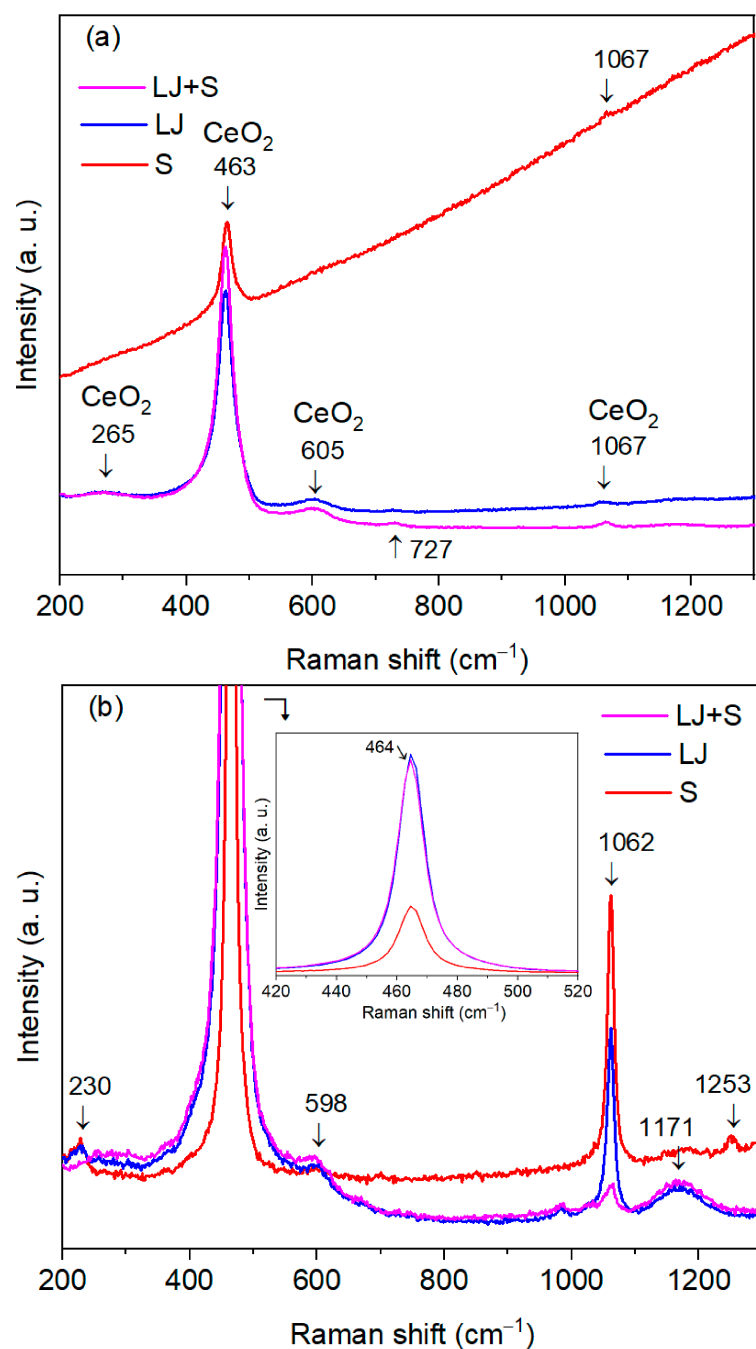


Figure 4. Raman spectra of the three samples (S, LJ, and LJ+S) under heat treatment at (a) 350 °C and (b) 600 °C.

Raman spectra for the three samples calcined at 350 °C are presented in Figure 4a. The spectra are dominated by a prominent peak at 463 cm⁻¹, which corresponds to Ce–O lattice vibrations, specifically the F_{2g} mode of the cubic fluorite phase of CeO₂ [37]. Additionally, weaker bands are observed at 265, 605, and 1067 cm⁻¹. The band at 265 cm⁻¹ is commonly ascribed to a second order, doubly degenerate transverse acoustic (2TA type) vibration of the CeO₂ lattice, whereas the 605 cm⁻¹ feature is attributed to a defect related longitudinal optical (LO) mode associated with oxygen vacancy formation and the accompanying Ce⁴⁺/Ce³⁺ redox process [38]. The band around 1067 cm⁻¹ is assigned to a higher order overtone/combination vibration of the cubic fluorite lattice, in line with previous reports on nanostructured CeO₂ [39]. A weak additional feature at 727 cm⁻¹ is observed exclusively in the LJ+S sample. Since this band is not characteristic of fluorite

CeO₂, it most likely arises from a minor secondary phase. The corresponding XRD pattern at 350 °C (Figure 3a) does not show extra crystalline reflections, suggesting that this phase is either amorphous or is masked by the broad reflections of the poorly crystallized CeO₂ matrix. Notably, the S sample also displays a pronounced elevation of the Raman baseline across the entire spectral range. This behaviour is characteristic of fluorescence, most likely originating from residual carbonaceous species that absorb the excitation light and re-emit at longer wavelengths, thereby superimposing a broad background on the Raman spectrum. This fluorescent contribution partly obscures weaker vibrational modes and complicates the reliable identification of low intensity Raman bands [40].

After thermal treatment at 600 °C, Raman spectra were again recorded for the three samples (Figure 4b). The characteristic bands of CeO₂ at approximately 265, 463, 605, and 1067 cm⁻¹ are still observed, confirming that the fluorite crystalline phase is preserved after this calcination. The sharp and intense F_{2g} mode at 463 cm⁻¹ remains the dominant feature, consistent with the formation of nanometric CeO₂ particles. In addition, a distinct band appears at 1171 cm⁻¹, which is only detected in the samples containing lemon juice (LJ and LJ+S) and is absent in the sucrose only sample (S). The band at 1171 cm⁻¹ is characteristic of CeO₂ and has been assigned to the second order (2LO) vibration associated with the bulk structure of the fluorite lattice [39]. This feature is reported to remain essentially unchanged under reduction treatments and is therefore frequently used as an internal reference in Raman analyses of ceria. Its presence in the LJ and SLJ samples indicates that the CeO₂ crystalline framework remains well defined after calcination at 600 °C [41].

A further Raman band emerges at 1253 cm⁻¹ exclusively in the S sample after heat treatment at 600 °C, which is not present in the corresponding spectrum at 350 °C. Since this band is not typical of CeO₂, it can be associated with a secondary phase and is plausibly linked to the additional diffraction peak observed in the XRD pattern of the S sample at 600 °C, tentatively assigned to sodium bicarbonate (NaHCO₃). Thomas Rainer [42] reported that the Raman spectrum of NaHCO₃ exhibits several characteristic vibrational modes, including an intense band at about 1045 cm⁻¹ and a weaker band near 1266 cm⁻¹. The strong 1045 cm⁻¹ band can overlap with the CeO₂ s order signal close to 1060 cm⁻¹ and thus remain unresolved in our spectra, whereas the band detected at 1253 cm⁻¹ in the S sample can be correlated with the weaker NaHCO₃ vibration typically reported around 1260 cm⁻¹, supporting the presence of NaHCO₃ as a secondary phase even when its most intense Raman features are not clearly separated from those of CeO₂.

3.4. SEM Analysis

SEM was used to evaluate the morphological evolution of the CeO₂ nanoparticles synthesized via three distinct green routes (S, LJ, and LJ+S) under controlled thermal treatments at 350 °C and 600 °C, as shown in Figure 5.

Regarding the micrograph analysis of the samples calcinated at 350 °C, the morphology at this stage is characterized by large, dense clusters, likely due to the presence of residual amorphous organic matter that prevents the formation of well-defined primary nanoparticles. A notable feature in the S and LJ samples is the presence of large structures (Figure 5a,c) resulting from the coalescence and agglomeration of particles [43]. Interestingly, these large formations persisted even after increasing the thermal treatment to 600 °C (Figure 5b,d). However, the LJ+S samples presented a distinct exception (Figure 5e,f). Although some degree of coalescence is still observed, the nanoparticles exhibit significantly improved dispersion and a more refined morphology, characterized by a homogeneous distribution of predominantly spherical particles. This enhanced morphological outcome is attributed to the synergistic stabilizing effects of the dual precursor system. The citric acid and various phytochemicals in the lemon juice act as effective chelating agents, which help

stabilize the initial nuclei and prevent early stage coalescence [44]. Meanwhile, sucrose acts as both a structure-directing and capping agent, promoting the formation of smaller and more uniformly dispersed nanoparticles by limiting agglomeration and enhancing colloidal stability [25].

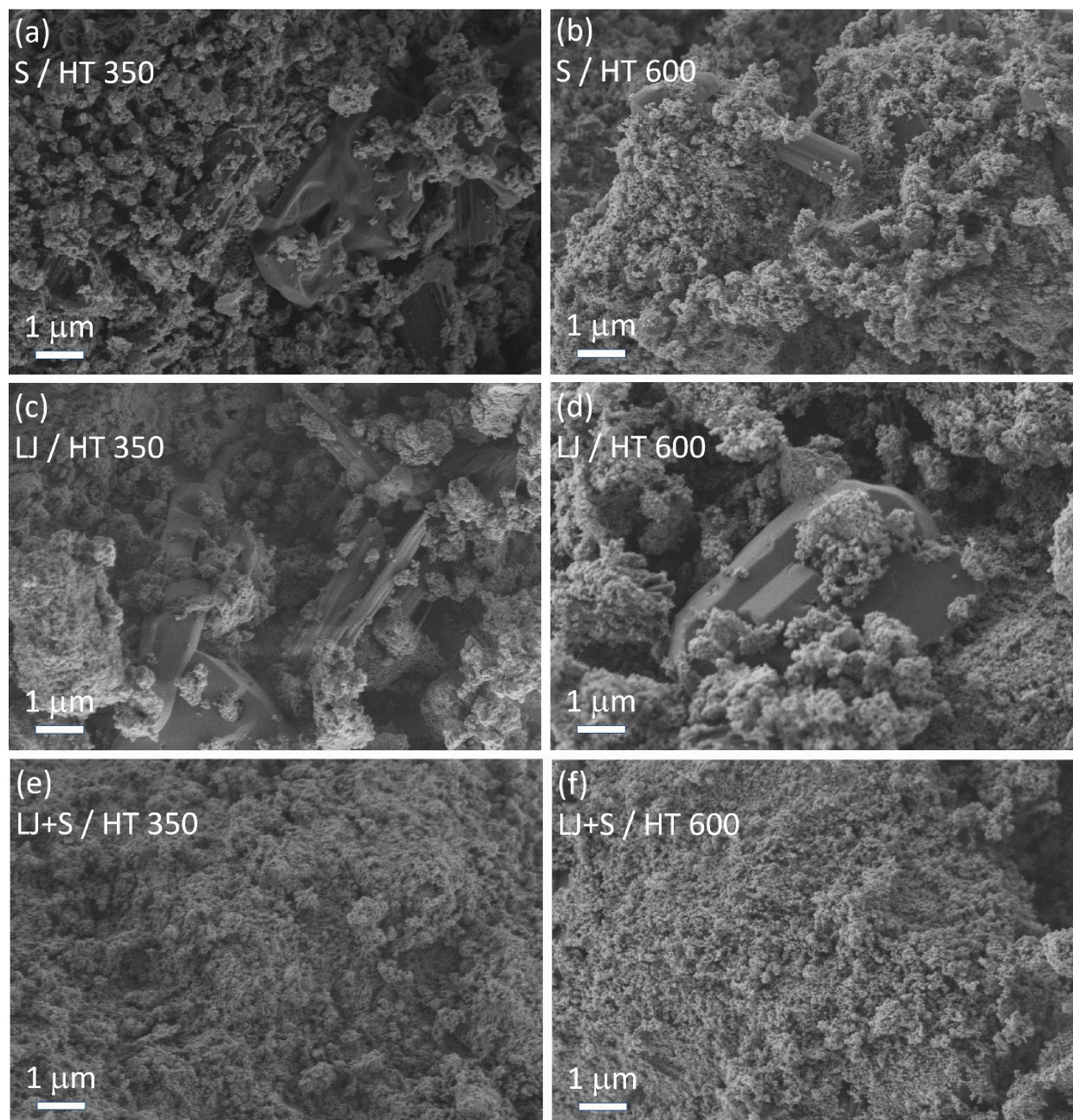


Figure 5. SEM micrographs of CeO_2 samples synthesized using sucrose (S), lemon juice (LJ), and their combination (LJ+S), after heat treatment at 350 °C and 600 °C: (a) S/HT350, (b) S/HT600, (c) LJ/HT350, (d) LJ/HT600, (e) LJ+S/HT350, and (f) LJ+S/HT600.

Under the acidic conditions provided by the citric acid present in lemon juice, sucrose can hydrolyze into glucose and fructose. Upon heating, glucose and fructose may undergo oxidation and thermal degradation reactions, yielding oxygenated intermediates rich in hydroxyl (–OH) and carboxyl (–COOH) functional groups. These species can coordinate Ce^{3+} ions derived from $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and contribute to the formation of an organic–inorganic network, playing a role analogous to that of polyols such as ethylene glycol in polymeric precursor methods. During calcination, the organic matrix decomposes

while the coordinated cerium species undergo condensation and crystallization, leading to the formation of stoichiometric CeO_2 nanoparticles. Although CeO_2 formation occurs in all formulations, the combined presence of lemon juice and sucrose appears to provide improved control over nanoparticle development, resulting in enhanced dispersion and morphological homogeneity [45–47].

To further examine the particle morphology and size distribution, a higher-magnification micrograph of the LJ+S sample and the corresponding particle size distribution histogram are presented in Figure 6.

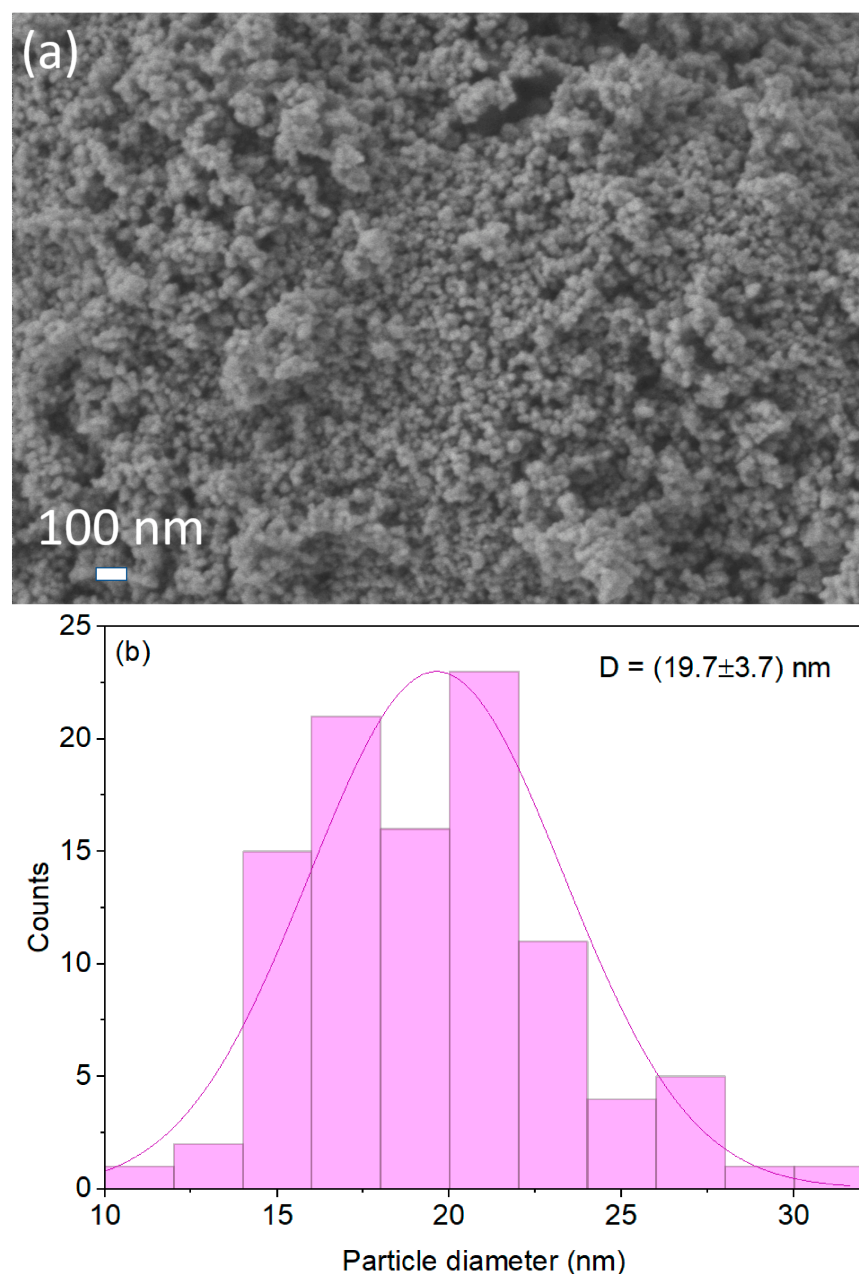


Figure 6. (a) High-magnification SEM micrograph of the CeO_2 sample synthesized using sucrose and lemon juice (LJ+S), after heat treatment at 600 °C; (b) Particle size distribution obtained from measurements of 100 nanoparticles. The pink columns and solid curve represent the experimental data histogram and the corresponding Gaussian fit, respectively.

Particle size analysis was carried out using ImageJ software (version 1.53m). The diameters of 100 nanoparticles randomly selected from different regions of the micrograph were measured to obtain a representative size distribution. As the particles displayed an

approximately spherical morphology, their size was determined from a single diameter measurement. The resulting average particle diameter was (19.3 ± 3.7) nm.

To better contextualize the physicochemical characteristics of the CeO₂ nanoparticles synthesized in this work and highlight the novelty of the proposed green synthesis route, Table 2 compares the present approach with selected studies reporting the green synthesis of CeO₂ nanoparticles using different natural extracts. The comparison includes the bio-based agent, synthesis method, crystallite size, particle size, and particle morphology. This overview provides a broader perspective on the structural and morphological characteristics of CeO₂ nanoparticles obtained through various green synthesis strategies and highlights the position of the proposed lemon juice–sucrose route within the current state of the art.

Table 2. Comparison of CeO₂ nanoparticles synthesized using different green routes, including crystallite size, particle size, morphology, and key distinguishing features.

Bio-Based Agent	Synthesis Method	Crystallite Size (nm)	Particle Size (nm)	Morphology	Ref.
<i>Acacia concinna</i> fruit extract	Sol-gel	22.7	-	Porous network of roughly spherical nanoparticles; uniform distribution with limited agglomeration	[18]
<i>Citrus nobilis</i> peel extract	Co-precipitation	5.1	25	Spherical to quasi-spherical nanoparticles; uniform distribution with moderate agglomeration	[19]
<i>Olea europaea</i> leaf extract	Precipitation	6	24	Highly homogeneous distribution of spherical nanoparticles	[48]
<i>Magnolia kobus</i> leaf extract	Precipitation	20	50	Nearly spherical, well-dispersed nanoparticles	[49]
<i>Solanum nigrum</i> leaf extract	Precipitation	10.08	20	Spherical to quasi-cubic, uniformly dispersed nanoparticles with reduced agglomeration	[50]
<i>Crocus sativus</i> by-product extract	Precipitation	16.71	-	Spherical nanoparticles with a homogeneous distribution	[51]
<i>Abelmoschus esculentus</i> fruit extract	Precipitation	30	36	Spherical nanoparticles with a homogeneous distribution	[52]
Lemon juice + sucrose	Sol gel based	15.8	19.7	Quasi-spherical nanoparticles with improved dispersion and homogeneity	This work

3.5. Antibacterial Activity

The antibacterial effect of the biosynthesized CeO₂ nanoparticles (LJ+S sample calcined at 600 °C) against *Staphylococcus aureus* (*S. aureus*) was evaluated by monitoring the logarithmic reduction in CFU/mL after 24 h of exposure. The LJ+S sample was chosen based on its superior physicochemical characteristics, including a more homogeneous morphology, reduced presence of large secondary structures, and higher phase purity, as evidenced by SEM, XRD, and Raman analyses. As illustrated in Figure 7, the nanoparticles exhibited a clear dose dependent inhibitory effect.

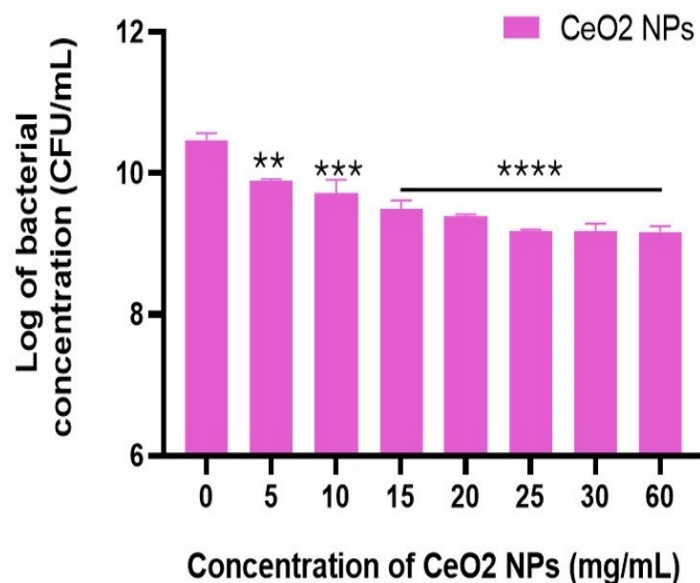


Figure 7. Logarithm of bacterial concentration after 24-h exposure to increasing concentrations of CeO₂ nanoparticles (LJ+S sample calcined at 600 °C). Results are expressed as mean $\pm$ standard deviation (SD). Statistically significant differences compared to the untreated control group are indicated by asterisks (**** $p < 0.0001$, *** $p < 0.001$ and ** $p < 0.01$).

A reduction in bacterial viability was observed at 5 mg/mL (** $p < 0.01$), indicating the onset of antibacterial activity. This effect became more pronounced at 10 mg/mL (*** $p < 0.001$), confirming a concentration-dependent response. Highly significant antibacterial activity (**** $p < 0.0001$) was detected at concentrations ≥ 15 mg/mL. Notably, the antibacterial effect reached a plateau at approximately 25 mg/mL, beyond which further increases in CeO₂ NP concentration did not result in a proportional reduction in CFU counts, suggesting that the maximum effective dose under the tested conditions had been attained [53].

Several physicochemical factors may contribute to this observed saturation. A primary explanation involves the concentration dependent aggregation of CeO₂ nanoparticles. At elevated concentrations, the increase in particle-particle collisions often leads to the formation of larger agglomerates, which significantly reduces the effective surface-area-to-volume ratio available for interaction with the bacterial cells. This reduction in active surface area diminishes the density of reactive sites available for oxidative stress induction and direct membrane disruption, two primary drivers of nanoceria toxicity [54,55]. Furthermore, the observed tendency of the nanoparticles to sediment at higher concentrations may limit the bioavailability of the particles in the liquid phase, thereby reducing the effective contact frequency with the suspended bacteria.

The pH of the nanoparticle suspensions was also monitored after 24 h of incubation under the same conditions used for the antibacterial assays. The untreated bacterial control exhibited a pH of 6.68, while the CeO₂-treated bacterial suspensions showed pH values ranging from 6.49 to 7.38, depending on nanoparticle concentration. These results indicate that all conditions remained close to neutral pH throughout the experiment. Therefore, the observed reduction in bacterial viability cannot be attributed to major pH-induced stress and is more likely associated with the intrinsic antibacterial properties of the CeO₂ nanoparticles.

Several mechanisms may contribute to the antibacterial activity observed for the CeO₂ nanoparticles. Although the present study did not aim to elucidate the bactericidal mechanism in detail, previous reports suggest that CeO₂ nanoparticles can affect bacteria through multiple, possibly simultaneous, pathways. One of the main proposed mechanisms

involves direct interaction between nanoparticles and the bacterial cell envelope [56]. Due to electrostatic and physicochemical interactions, CeO₂ nanoparticles may adsorb onto the bacterial surface, promoting close contact with the cell wall and membrane. This interaction can disturb membrane organization, alter permeability, impair ion transport, and affect nutrient exchange between the bacterial cell and the surrounding medium. In addition, cerium species may interact with membrane-associated proteins, including thiol-containing groups, as well as transporters and porins, further compromising bacterial homeostasis [57]. Another important contribution may arise from the redox activity of CeO₂ nanoparticles. The reversible Ce³⁺/Ce⁴⁺ transition, together with the presence of oxygen vacancies, can promote catalytic reactions associated with the generation of reactive oxygen species (ROS), including superoxide radicals, hydroxyl radicals, and hydrogen peroxide, depending on the physicochemical properties of the nanoparticles and the surrounding environment [18,58]. These ROS can induce oxidative stress and damage essential bacterial components such as membrane lipids, proteins, polysaccharides, proteins, and nucleic acids, ultimately leading to loss of cellular integrity and bacterial death [59]. Furthermore, oxygen vacancies are recognized as key active sites that enhance the catalytic activity of CeO₂ nanoparticles and may contribute to ROS-mediated antibacterial effects. Particle size, morphology, surface defects, and oxygen vacancy concentration have also been reported to influence antibacterial performance by modulating surface reactivity, bacterial contact, and ROS generation [60].

Therefore, the reduction in viable *S. aureus* cells observed in this study is likely associated with the combined contribution of nanoparticle–cell envelope interactions and CeO₂-mediated redox activity.

In summary, the CeO₂ nanoparticles synthesized via the green LJ+S route demonstrate robust antibacterial efficacy against *S. aureus*. While aggregation and sedimentation present inherent limitations to dose escalation, the high activity observed at relatively low concentrations confirms the potential of these biosynthesized nanoparticles as effective agents for biomedical coatings or antibacterial formulations. It should be noted that this study represents an initial evaluation of antibacterial performance, focused exclusively on *S. aureus* as a model Gram-positive bacterium. Future work will extend this assessment to a broader range of clinically relevant bacterial strains, including Gram-negative species, and will further investigate the underlying antibacterial mechanisms through complementary physicochemical and biological analyses, including ROS quantification, membrane integrity assessment, and bacterial morphology characterization.

4. Conclusions

This work demonstrates a convenient green synthesis route for CeO₂ nanoparticles using lemon juice, sucrose, and their combination as bio-based chelating, capping, and stabilizing agents. Thermal, structural, and vibrational analyses collectively show that calcination at 600 °C is essential to obtain highly crystalline cubic fluorite CeO₂, while the precursor chemistry decisively governs phase purity and morphology.

XRD and Raman spectroscopy confirm that all samples exhibit nanocrystalline CeO₂ with average crystallite sizes in the 15–17 nm range, whereas sucrose-only processing promotes the formation of a NaHCO₃ secondary phase at high calcination temperature. The combined lemon juice/sucrose system effectively suppresses this secondary phase and stabilizes the fluorite lattice. SEM further reveals that this dual precursor route mitigates the formation of large coalesced aggregates, offering homogeneous, well dispersed, and spherical nanoparticles that are not achieved with either lemon juice or sucrose alone.

Biological assays against *Staphylococcus aureus* demonstrate a clear dose-dependent antibacterial response for the LJ+S-derived CeO₂ nanoparticles, with significant reductions

in CFU/mL at concentrations ≥ 5 mg/mL and a pronounced bactericidal effect up to a saturation regime around 25 mg/mL. The high activity at relatively low doses, combined with the green synthesis route and favorable nanoscale morphology, highlights these biosynthesized CeO₂ nanoparticles as promising candidates for integration into antimicrobial coatings and biomedical applications.

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References

1. Abdullah, M.; Obayedullah, M.; Shariful Islam Shuvo, M.; Abul Khair, M.; Hossain, D.; Nahidul Islam, M. A Review on Multifunctional Applications of Nanoparticles: Analyzing Their Multi-Physical Properties. *Results Surf. Interfaces* **2025**, *21*, 100635. [CrossRef]
2. Benzarti, Z.; Saadi, H.; Abdelmoula, N.; Hammami, I.; Fernandes Graça, M.P.; Alrasheedi, N.; Louhichi, B.; Seixas de Melo, J.S. Enhanced Dielectric and Photocatalytic Properties of Li-Doped ZnO Nanoparticles for Sustainable Methylene Blue Degradation with Reduced Lithium Environmental Impact. *Ceram. Int.* **2025**, *51*, 47170–47184. [CrossRef]
3. Neiva, J.; Benzarti, Z.; Carvalho, S.; Devesa, S. Green Synthesis of CuO Nanoparticles—Structural, Morphological, and Dielectric Characterization. *Materials* **2024**, *17*, 5709. [CrossRef] [PubMed]
4. Benzarti, Z.; Neiva, J.; Faia, P.; Silva, E.; Carvalho, S.; Devesa, S. Novel Green Synthesis Route of ZnO Nanoparticles for Dielectric Applications. *Nanomaterials* **2025**, *15*, 991. [CrossRef] [PubMed]
5. Saadi, H.; Khaldi, O.; Larbi, T.; Benzarti, Z. Correlations between Structural, Dielectric Properties, and Ab Initio Calculations in Co-Doped ZnO Nanoparticles: Effect of Co Concentration. *Appl. Phys. A* **2023**, *129*, 848. [CrossRef]
6. Aigbe, U.O.; Osibote, O.A. Green Synthesis of Metal Oxide Nanoparticles, and Their Various Applications. *J. Hazard. Mater. Adv.* **2024**, *13*, 100401. [CrossRef]
7. Saini, D.K.; Singh, M.; Pandey, J.K.; Rathore, S.; Singh, R.P.; Kumar, M. Green Synthesis of Nanoparticles for Biomedical Applications. *J. Mater. Environ. Sci.* **2025**, *16*, 1833–1859.
8. Gour, A.; Jain, N.K. Advances in Green Synthesis of Nanoparticles. *Artif. Cells Nanomed. Biotechnol.* **2019**, *47*, 844–851. [CrossRef] [PubMed]
9. Jadoun, S.; Arif, R.; Jangid, N.K.; Meena, R.K. Green Synthesis of Nanoparticles Using Plant Extracts: A Review. *Environ. Chem. Lett.* **2021**, *19*, 355–374. [CrossRef]
10. Antunes Filho, S.; dos Santos, M.S.; dos Santos, O.A.L.; Backx, B.P.; Soran, M.-L.; Opreș, O.; Lung, I.; Stegarescu, A.; Bououdina, M. Biosynthesis of Nanoparticles Using Plant Extracts and Essential Oils. *Molecules* **2023**, *28*, 3060. [CrossRef] [PubMed]
11. Kumar, A.; Shah, S.R.; Jayeoye, T.J.; Kumar, A.; Parihar, A.; Prajapati, B.; Singh, S.; Kapoor, D.U. Biogenic Metallic Nanoparticles: Biomedical, Analytical, Food Preservation, and Applications in Other Consumable Products. *Front. Nanotechnol.* **2023**, *5*, 1175149. [CrossRef]
12. Hossain, A.; Abdallah, Y.; Ali, M.A.; Masum, M.M.I.; Li, B.; Sun, G.; Meng, Y.; Wang, Y.; An, Q. Lemon-Fruit-Based Green Synthesis of Zinc Oxide Nanoparticles and Titanium Dioxide Nanoparticles against Soft Rot Bacterial Pathogen *Dickeya Dadantii*. *Biomolecules* **2019**, *9*, 863. [CrossRef] [PubMed]

13. AlZahabi, S.; Mamdouh, W. Valorization of Citrus Processing Waste into High-Performance Bionanomaterials: Green Synthesis, Biomedicine, and Environmental Remediation. *RSC Adv.* **2025**, *15*, 36534–36595. [[CrossRef](#)] [[PubMed](#)]
14. Mohajeri, M.; Momenai, R.; Karami-Mohajeri, S.; Ohadi, M.; Raeisi Estabragh, M.A. Cerium Oxide Nanoparticles, Physical and Chemical Properties, Applications and Toxicological Implications: A Review. *Results Chem.* **2025**, *15*, 102302. [[CrossRef](#)]
15. Rajwani, P.; Maddheshiya, S.; Nara, S. Exploring the Impact of CeO₂ Precursors on the Intrinsic ROS Generating Ability and Antibacterial Potential. *Mater. Sci. Eng. B* **2026**, *323*, 118721. [[CrossRef](#)]
16. Zhang, B.; Huyan, Y.; Wang, J.; Wang, W.; Zhang, Q.; Zhang, H. Synthesis of CeO₂ Nanoparticles with Different Morphologies and Their Properties as Peroxidase Mimic. *J. Am. Ceram. Soc.* **2019**, *102*, 2218–2227. [[CrossRef](#)]
17. Yim, G.; Kim, C.Y.; Kang, S.; Min, D.-H.; Kang, K.; Jang, H. Intrinsic Peroxidase-Mimicking Ir Nanoplates for Nanozymatic Anticancer and Antibacterial Treatment. *ACS Appl. Mater. Interfaces* **2020**, *12*, 41062–41070. [[CrossRef](#)]
18. Muduli, S.; Sekhar Behera, S.; Kumar Mohapatra, R.; Kumar Parhi, P.; Ranjan Sahoo, T. Efficient Adsorption and Antimicrobial Application of Bio-Synthesized Porous CeO₂ Nanoparticles. *Mater. Sci. Eng. B* **2023**, *290*, 116275. [[CrossRef](#)]
19. Putri, G.E.; Labanni, A.; Arief, S.; Dafriani, P.; Darma, I.Y.; Handayani, S.; Widyastuti; Jaffar, N.; Mahmud, M.; Ritonga, A.H. Green Synthesis of Cerium Oxide Nanoparticles Using Citrus Nobilis Lour. Peel Extract and Evaluation of Their Potential as Antibacterial and Antioxidant Agents. *Case Stud. Chem. Environ. Eng.* **2025**, *11*, 101062. [[CrossRef](#)]
20. Martha, R.; Gutierrez, P.; Celeste, A.; Rivera, T.; Jeronimo, F.M.; Tellez, J.; Carlos, J.; Ochoa, S.; Martha, R.; Gutierrez, P.; et al. Synthesis of Cerium Oxide Nanoparticles Using Citrus Paradisi Peel Aqueous Ex- Tract and Their Effect on Protein Aggregation, and Acetylcholinesterase Inhibi- Tion Citation. *J. Herb. Med. Med. Plants* **2024**, *2*, 1–19.
21. Samaru, M.; Sourav, M.; Ashwini, P.; Sharath Chandra, K.; Sagnik, N.; Rokeya, S.; Mohammed Gulzar, A. Sustainable Biogenic Synthesis of Cerium Oxide Nanoparticles (CeO₂ NPs) Using Cinnamomum Verum Bark: An in Vitro Physicochemical and Biological Assessment. *Discov. Mater.* **2025**, *5*, 226. [[CrossRef](#)]
22. Nosrati, H.; Heydari, M.; Khodaei, M. Cerium Oxide Nanoparticles: Synthesis Methods and Applications in Wound Healing. *Mater. Today Bio* **2023**, *23*, 100823. [[CrossRef](#)] [[PubMed](#)]
23. Irshad, M.; Baber, K.; Butt, M.S.; Hanif, M.B.; Asad, M.; Ghaffar, A.; Rafique, M.; Hussain, I.; Al-Anazy, M.M.; Makarov, H.; et al. Synergistic Role of Biomolecules and Bio-Chelating Agents in the Sustainable Development of an Efficient BaCe_{0.97}M_{0.03}O_{3-δ} (M = Sm, Gd) Perovskite Electrolyte for IT-SOFC. *Ceram. Int.* **2023**, *49*, 38360–38366. [[CrossRef](#)]
24. Khraibet, A.A.H.; Wahab, W.S.A. Green Synthesis of Silver Nanoparticles Using Lemon Juice Extract and Low Laser Irradiation. *Ann. Chim.-Sci. Des. Matériaux* **2025**, *49*, 281–290. [[CrossRef](#)]
25. Sangsefidi, F.S.; Nejati, M.; Verdi, J.; Salavati-Niasari, M. Green Synthesis and Characterization of Cerium Oxide Nanostructures in the Presence Carbohydrate Sugars as a Capping Agent and Investigation of Their Cytotoxicity on the Mesenchymal Stem Cell. *J. Clean. Prod.* **2017**, *156*, 741–749. [[CrossRef](#)]
26. Tripathi, K.; Srivastava, Y.; Kumar, N. Enumeration of Soil Microbiota: Calculation of Colony Forming Units (CFU) for Microbial Quantification. In *Biotechnology Lab Techniques: Culture Media, Microscopy, and Microbial Analysis*; Deep Science Publishing: Toronto, ON, Canada, 2025; pp. 89–91.
27. Naz, S.; Kazmi, S.T.B.; Zia, M. CeO₂ Nanoparticles Synthesized through Green Chemistry Are Biocompatible: In Vitro and in Vivo Assessment. *J. Biochem. Mol. Toxicol.* **2019**, *33*, e22291. [[CrossRef](#)] [[PubMed](#)]
28. Rana, A.; Hasan, I.; Koo, B.H.; Khan, R.A. Green Synthesized CeO₂ Nanowires Immobilized with Alginate-Ascorbic Acid Biopolymer for Advance Oxidative Degradation of Crystal Violet. *Colloids Surf. A Physicochem. Eng. Asp.* **2022**, *637*, 128225. [[CrossRef](#)]
29. Nyoka, M.; Choonara, Y.E.; Kumar, P.; Kondiah, P.P.D.; Pillay, V. Synthesis of Cerium Oxide Nanoparticles Using Various Methods: Implications for Biomedical Applications. *Nanomaterials* **2020**, *10*, 242. [[CrossRef](#)] [[PubMed](#)]
30. Mim, J.; Sultana, M.S.; Dhar, P.K.; Hasan, M.K.; Dutta, S.K. Green Mediated Synthesis of Cerium Oxide Nanoparticles by Using Oroxylum Indicum for Evaluation of Catalytic and Biomedical Activity. *RSC Adv.* **2024**, *14*, 25409–25424. [[CrossRef](#)] [[PubMed](#)]
31. Arooj, Y.; Karim, R.; Maqsood, S.; Farhan, A.; Kahlid, A.; Erum, A.; Qamar, M.A. Effect of Temperatures Variation on the Synthesis and Characterization of Cerium Oxide Nanoparticles by Co-Precipitation Method. *J. Ovonic Res.* **2025**, *21*, 307–318. [[CrossRef](#)]
32. Sanfilippo, C.; Fiore, V.; Calabrese, L.; Megna, B.; Valenza, A. Effect of Sodium Bicarbonate Treatment on the Properties of Sisal Fibers and Their Geopolymer Composites. *Case Stud. Constr. Mater.* **2024**, *21*, e03536. [[CrossRef](#)]
33. Wang, Y.; Cheng, Y.; Yu, M.; Li, Y.; Cao, J.; Zheng, L.; Yi, H. Methane Explosion Suppression Characteristics Based on the NaHCO₃/Red-Mud Composite Powders with Core-Shell Structure. *J. Hazard. Mater.* **2017**, *335*, 84–91. [[CrossRef](#)] [[PubMed](#)]
34. Kolev, K.; Dimitrov, O.; Dimitrova, M.; Shipochka, M.; Karashanova, D.; Petkova, T. Structural and Morphological Characterization of Gd-Doped Ceria (Ce_{1-x}Gd_xO_{2-x/2}) Synthesized by an Optimized Hydrothermal Method. *Materials* **2025**, *18*, 4957. [[CrossRef](#)] [[PubMed](#)]
35. Monshi, A.; Foroughi, M.R.; Monshi, M.R. Modified Scherrer Equation to Estimate More Accurately Nano-Crystallite Size Using XRD. *World J. Nano Sci. Eng.* **2012**, *2*, 154–160. [[CrossRef](#)]

36. Mobeen, A.; Sundaram, R. Synthesis and Characterization of Cerium Oxide Nanoparticles Using Sucrose as a Green Capping Agent and Its Application for Antibacterial and Humidity Sensor Studies. *Int. J. Res. Advent. Technol.* **2019**, *7*, 217–223. [\[CrossRef\]](#)
37. Gajbhiye, S.S.; Salve, M.V.; Gaikwad, M.D.; Rasage, A.R.; Khater, M.S.; Ghadage, V.H.; Chaure, N.B. Influence of Synthesis Conditions on the Physical Characteristics and Antibacterial Activities of Cerium Oxide Nanoparticles in Biomedical Applications. *Eng. Sci.* **2024**, *32*, 1254.
38. Jayakumar, G.; Albert Irudayaraj, A.; Dhayal Raj, A. A Comprehensive Investigation on the Properties of Nanostructured Cerium Oxide. *Opt. Quantum Electron.* **2019**, *51*, 312. [\[CrossRef\]](#)
39. Loridant, S. Raman Spectroscopy as a Powerful Tool to Characterize Ceria-Based Catalysts. *Catal. Today* **2021**, *373*, 98–111. [\[CrossRef\]](#)
40. Wei, D.; Chen, S.; Liu, Q. Review of Fluorescence Suppression Techniques in Raman Spectroscopy. *Appl. Spectrosc. Rev.* **2015**, *50*, 387–406. [\[CrossRef\]](#)
41. Pushkarev, V.V.; Kovalchuk, V.I.; D'Itri, J.L. Probing Defect Sites on the CeO₂ Surface with Dioxygen. *J. Phys. Chem. B* **2004**, *108*, 5341–5348. [\[CrossRef\]](#)
42. Rainer, T. NaHCO₃-NaDCO₃ and 13CO₂-Rich Fluid Inclusion in Pegmatite Quartz from Bornholm Island/Denmark. *Geol. Earth Mar. Sci.* **2024**, *6*, 1–5. [\[CrossRef\]](#)
43. Jiang, L.; Guo, Y.; Liu, Z.; Chen, S. Computational Understanding of the Coalescence of Metallic Nanoparticles: A Mini Review. *Nanoscale* **2024**, *16*, 5521–5536. [\[CrossRef\]](#) [\[PubMed\]](#)
44. Magalhães, D.; Vilas-Boas, A.A.; Teixeira, P.; Pintado, M. Functional Ingredients and Additives from Lemon By-Products and Their Applications in Food Preservation: A Review. *Foods* **2023**, *12*, 1095. [\[CrossRef\]](#) [\[PubMed\]](#)
45. Majedi, A.; Abbasi, A.; Davar, F. Green Synthesis of Zirconia Nanoparticles Using the Modified Pechini Method and Characterization of Its Optical and Electrical Properties. *J. Sol-Gel Sci. Technol.* **2016**, *77*, 542–552. [\[CrossRef\]](#)
46. Davar, F.; Majedi, A.; Mirzaei, A. Green Synthesis of ZnO Nanoparticles and Its Application in the Degradation of Some Dyes. *J. Am. Ceram. Soc.* **2015**, *98*, 1739–1746. [\[CrossRef\]](#)
47. Devesa, S.; da Silva, P.S.P.; Graça, M.P.; Costa, L.C.; Paixão, J.A. Impedance Spectroscopy Studies of ErNbO₄ Synthesised by the Sol–Gel Method. *J. Sol-Gel Sci. Technol.* **2020**, *96*, 143–152. [\[CrossRef\]](#)
48. Maqbool, Q.; Nazar, M.; Naz, S.; Hussain, T.; Jabeen, N.; Kausar, R.; Anwaar, S.; Abbas, F.; Jan, T. Antimicrobial Potential of Green Synthesized CeO₂ Nanoparticles from Olea Europaea Leaf Extract. *Int. J. Nanomed.* **2016**, *11*, 5015–5025. [\[CrossRef\]](#) [\[PubMed\]](#)
49. Fesenko, T.V.; Laguta, I.V.; Stavinskaya, O.M.; Kuzema, P.O.; Anishchenko, V.M.; Oranska, O.I.; Ivannikov, R.V.; Diyuk, O.A.; Skorochod, I.O. Green Synthesis of Antibacterial Cerium Oxide Nanoparticles Using Magnolia Kobus Leaves Extract. *Him. Fiz. Tehnol. Poverhni* **2023**, *14*, 546–554. [\[CrossRef\]](#)
50. Muthuvel, A.; Jothibas, M.; Manoharan, C.; Jayakumar, S.J. Synthesis of CeO₂-NPs by Chemical and Biological Methods and Their Photocatalytic, Antibacterial and in Vitro Antioxidant Activity. *Res. Chem. Intermed.* **2020**, *46*, 2705–2729. [\[CrossRef\]](#)
51. Khadfy, Z.; Mamouni, R.; Jadouali, S.M.; Atifi, H.; Saffaj, N.; Chartier, A.; Nehme, R.; Boussif, K.; Achemchem, F. A Novel Natural Reducing Agent for the Preparation of Cerium Oxide Nanoparticles Based on the Saffron By-Product: Characterization, Antioxidant and Antibacterial Activity for Nutritional Applications. *Nano-Struct. Nano-Objects* **2024**, *40*, 101381. [\[CrossRef\]](#)
52. Ahmed, H.E.; Iqbal, Y.; Aziz, M.H.; Atif, M.; Batool, Z.; Hanif, A.; Yaqub, N.; Farooq, W.A.; Ahmad, S.; Fatehmulla, A.; et al. Green Synthesis of CeO₂ Nanoparticles from the Abelmoschus Esculentus Extract: Evaluation of Antioxidant, Anticancer, Antibacterial, and Wound-Healing Activities. *Molecules* **2021**, *26*, 4659. [\[CrossRef\]](#) [\[PubMed\]](#)
53. Fiddaroini, S.; Indu, K.; Madaniyah, L.; Amalia, S.; Aulanni'am; Rahman, M.F.; Sabarudin, A. Different Types of Honey on the Synthesis of Silver Nanoparticles (AgNPs) and Their Antibacterial Activity: In-Vitro and in-Silico Studies. *Plant Nano Biol.* **2025**, *13*, 100188. [\[CrossRef\]](#)
54. Farias, I.A.P.; dos Santos, C.C.L.; Sampaio, F.C. Antimicrobial Activity of Cerium Oxide Nanoparticles on Opportunistic Microorganisms: A Systematic Review. *Biomed. Res. Int.* **2018**, *2018*, 1–14. [\[CrossRef\]](#) [\[PubMed\]](#)
55. Nisha, U.M.; Venkatesh, D.; Vasanthan, S.; Rajeswaran, P.; Balaji, J.; Karthik, P.S. Interfacial Coupling Effects of Chitosan Integrated ZrO₂/Bi₂O₃/CeO₂ Quaternary Composite for Efficient Wastewater Treatment and Antimicrobial Activity. *Ionics* **2025**, *31*, 2739–2756. [\[CrossRef\]](#)
56. Zheng, H.; Tang, J.; Wei, Y.; Deng, X.; Zhang, Y.; Ma, X.; Jiang, X.; Xu, Z.P.; Liao, H. Antibacterial Properties of Cerium Oxide Nanoparticles: Recent Progresses and Future Challenges. *Particuology* **2024**, *93*, 264–283. [\[CrossRef\]](#)
57. Rollin-Genetet, F.; Seidel, C.; Artells, E.; Auffan, M.; Thiéry, A.; Vidaud, C. Redox Reactivity of Cerium Oxide Nanoparticles Induces the Formation of Disulfide Bridges in Thiol-Containing Biomolecules. *Chem. Res. Toxicol.* **2015**, *28*, 2304–2312. [\[CrossRef\]](#) [\[PubMed\]](#)
58. Ravishankar, T.N.; Ramakrishnappa, T.; Nagaraju, G.; Rajanaika, H. Synthesis and Characterization of CeO₂ Nanoparticles via Solution Combustion Method for Photocatalytic and Antibacterial Activity Studies. *ChemistryOpen* **2015**, *4*, 146–154. [\[CrossRef\]](#) [\[PubMed\]](#)

59. Qi, M.; Li, W.; Zheng, X.; Li, X.; Sun, Y.; Wang, Y.; Li, C.; Wang, L. Cerium and Its Oxidant-Based Nanomaterials for Antibacterial Applications: A State-of-the-Art Review. *Front. Mater.* **2020**, *7*, 213. [[CrossRef](#)]
60. Gao, Z.; Liu, M.; Wang, Z.; Zhang, M.; Shen, G.; Gong, Y.; Zheng, K.; Yang, M.; Wang, Q. In Vitro Antibacterial Activity Evaluation and Mechanism of Morphology-Controlled Synthesis of Cerium Dioxide Nanoparticles. *Int. J. Mol. Sci.* **2025**, *26*, 10587. [[CrossRef](#)] [[PubMed](#)]

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