Development of high-performance ZnO nanoparticles using *Olea europaea* leaf extract: potential for multifunctional textile finishing and dye degradation

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ABSTRACT – REZUMAT

Development of high-performance ZnO nanoparticles using *Olea europaea* leaf extract: potential for multifunctional textile finishing and dye degradation

Zinc oxide nanoparticles were sustainably synthesised using Olea europaea leaf extract as a high-performance candidate for functional textile finishing. These green-synthesised nanoparticles were compared to conventional chemical ZnO to evaluate their suitability for textile-related applications. SEM analysis revealed that Green ZnO consists of well-defined hexagonal prisms with a primary size of 200–500 nm, providing a high density of active surface sites. The material exhibited a reduced band gap (2.6 eV) and enhanced visible-light absorption attributed to carbon incorporation and induced oxygen vacancies during thermal treatment. These characteristics are particularly advantageous for the development of self-cleaning textiles under natural sunlight. Green ZnO exhibited superior methylene blue adsorption (52 mg/g, 21% higher than chemical ZnO) and achieved 94% photocatalytic degradation within 3 h. The results indicate that these nanoparticles can effectively degrade textile dye effluents and provide self-cleaning properties to treated substrates. Furthermore, antimicrobial testing showed significant inhibition against Staphylococcus aureus (16 mm) and Candida albicans (28 mm). This eco-friendly, scalable ZnO presents a multifunctional finishing agent capable of providing simultaneous dye removal, self-cleaning, and disinfection, offering a sustainable pathway for the production of smart, bioactive textiles.

Keywords: *green ZnO, textile dye, adsorption, antimicrobial, photocatalytic activity*

Dezvoltarea unor nanoparticule de ZnO de înaltă performanță folosind extractul din frunze de *Olea europaea*: potențial pentru finisarea textilelor multifuncționale și degradarea coloranților

Nanoparticulele de oxid de zinc au fost sintetizate în mod durabil folosind extractul din frunze de Olea europaea ca material cu performanțe ridicate pentru finisarea textilelor funcționale. Aceste nanoparticule sintetizate ecologic au fost comparate cu ZnO chimic convențional pentru a evalua utilizarea lor în aplicații din domeniul textil. Analiza SEM a relevat că ZnO ecologic este format din prisme hexagonale bine definite, cu o dimensiune primară de 200–500 nm, oferind o densitate ridicată de zone active la suprafață. Materialul a prezentat o bandă interzisă redusă (2,6 eV) și o absorbție îmbunătățită a luminii vizibile, atribuite încorporării de carbon și apariției de lacune de oxigen induse în timpul tratamentului termic. Aceste caracteristici sunt deosebit de avantajoase pentru dezvoltarea de materiale textile cu autocurățare sub lumina naturală a soarelui. ZnO ecologic a prezentat o adsorbție superioară a albastrului de metilen (52 mg/g, cu 21% mai mare decât ZnO chimic) și a atins o degradare fotocatalitică de 94% în decurs de 3 ore. Rezultatele indică faptul că aceste nanoparticule pot degrada eficient efluenții de coloranți textili și pot conferi proprietăți de autocurățare substraturilor tratate. În plus, testele antimicrobiene au evidențiat o inhibare semnificativă împotriva Staphylococcus aureus (16 mm) și Candida albicans (28 mm). Acest ZnO ecologic și scalabil reprezintă un agent de finisare multifuncțional capabil să asigure simultan îndepărtarea coloranților, autocurățarea și dezinfectarea, oferind o cale durabilă pentru producția de textile inteligente și bioactive.

Cuvinte-cheie*: ZnO ecologic, colorant textil, adsorbție, proprietăți antimicrobiene, activitate fotocatalitică*

INTRODUCTION

The textile industry is currently undergoing a paradigm shift toward sustainable functionalization, driven by the global demand for “smart” fabrics that possess self-cleaning, antimicrobial, and UV-protective properties [1, 2]. Conventional textile finishing often relies on synthetic binders and hazardous chemical agents that contribute to environmental degradation and toxic dye effluents [3, 4].

Consequently, the development of multifunctional, eco-friendly nanomaterials that can facilitate both the remediation of dye-polluted water and the bio-activation of textile surfaces has become a primary research frontier [5, 6].

Among various metal oxides, Zinc Oxide (ZnO) nanoparticles have emerged as a leading candidate for textile applications due to their high chemical stability, non-toxicity, and significant photocatalytic potential [7]. Traditional chemical synthesis routes for

ZnO, such as sol-gel and hydrothermal methods, frequently involve the use of toxic reducing agents like sodium borohydride or hydrazine, which limit their "green" credentials [8–11].

To address this, plant-mediated "green" synthesis has gained traction, utilising the bioactive secondary metabolites of flora such as polyphenols, flavonoids, and terpenoids to act as natural reducing and capping agents [2, 12, 13]. Specifically, Olea europaea (olive) leaf extract is a promising bio-resource due to its high concentration of oleuropein and hydroxytyrosol, which have been shown to influence the morphology and optical properties of metallic nanoparticles [14–16].

However, a critical analysis of current literature reveals a significant research gap. Most green synthesis protocols for ZnO utilise low-temperature calcination (below 600°C), which often results in unstable organic surface residues that may leach or degrade, potentially compromising the long-term durability required for industrial textile finishing [17]. Furthermore, while the narrowing of the ZnO band gap via plant extracts is well-documented [18, 19], there is a scarcity of research investigating how high-temperature thermal treatment (900°C), while removing primary organic fractions, can induce stable, in situ carbon-doped defect states. These states are vital for extending the photocatalytic response into the visible light spectrum, which is a prerequisite for efficient self-cleaning functionality under natural sunlight [20, 21].

This study addresses these limitations by reporting the green synthesis of ZnO nanoparticles using Olea europaea leaf extract at a high calcination temperature of 900°C. The work provides a rigorous comparison between the green-synthesised ZnO and conventional chemical ZnO regarding their structural, optical, and morphological characteristics.

The performance of these nanoparticles is evaluated through two critical lenses for the textile industry: the adsorption and photocatalytic degradation of Methylene Blue (MB) dye under natural sunlight, and broad-spectrum antimicrobial efficacy against *Staphylococcus aureus* and *Candida albicans*. By establishing the superior visible-light activity and adsorption capacity of this carbon-modified green ZnO, this research provides a sustainable pathway for the next generation of bioactive and self-cleaning textile finishes.

METHODS AND MATERIALS

ZnO synthesis

Olive (*Olea europaea*) leaves were collected, rinsed repeatedly with tap and distilled water to remove dust, shade-dried at room temperature for 10–12 days, and ground into coarse powder. The aqueous extract was prepared by boiling 10 g of powder in 100 mL double-distilled water at 80°C for 60 min, followed by filtration and storage at –4°C. Green ZnO nanoparticles were synthesised by dissolving 10 g zinc acetate dihydrate (Sigma-Aldrich) in 100 mL olive leaf extract and stirring at 80°C for 2 h, forming a pale yellow precipitate. The product was centrifuged, washed thoroughly with deionised water, dried at 80°C for 24 h, and calcined at 900°C for 2 h to yield fine ZnO powder. For comparison, conventional ZnO was prepared via NaOH precipitation. A 0.1 M zinc acetate dihydrate solution was heated to 80°C under stirring, and 0.2 M NaOH (Novachem) solution was added dropwise to form a zinc hydroxide precipitate. After 1 h of stirring, the precipitate was cooled, repeatedly washed with distilled water, dried, and calcined at 450°C for 2 h to obtain ZnO powder. Both synthesis routes produced white ZnO nanoparticles suitable for textile finishing applications.

Antimicrobial activity

The antimicrobial activity of green-synthesised ZnO nanoparticles was assessed against *Staphylococcus aureus* (ATCC 25923) and *Candida albicans* (ATCC 10231) using the disk diffusion method. Overnight cultures were adjusted to 0.5 McFarland standard (~1.5×10^8 CFU/mL) in nutrient broth. Microbial suspensions were lawn-spread on Mueller-Hinton agar plates. Sterile 6-mm filter paper disks were loaded with ZnO suspension (50 µL) and placed on the agar. Distilled water served as a negative control, while ampicillin (10 µg) and fluconazole (25 µg) were positive controls for bacteria and fungus, respectively. Plates were incubated at 37°C for 24 h (bacteria) or 48 h (*C. albicans*). Inhibition zone diameters were measured in millimetres using a digital calliper. Clear zones indicated effective antimicrobial action of the green ZnO nanoparticles.

Adsorption and photocatalytic properties

Methylene blue (MB, Sigma-Aldrich) was selected as a model textile dye due to its widespread use and environmental relevance. Adsorption isotherms were studied by contacting 80 mg of ZnO adsorbent (green or chemical) with 20 mL MB solutions (10–500 mg/L) at natural pH. Suspensions were agitated at 150 rpm for 24 h to reach equilibrium. Kinetic studies used 25 mg of adsorbent in 50 mL of 25 mg/L MB, with sampling from 15 to 240 min. Photocatalytic degradation was evaluated under natural sunlight in Borj Cedria, Tunisia (36°43′04″N, 10°25′41″E) on 22 July 2025, between 12:00–15:00 CET (GHI 800–1000 W/m^2). 50 mg of ZnO was dispersed in 50 mL of 25 mg/L MB solution. After 24 h of dark equilibration to achieve adsorption–desorption balance, suspensions were exposed to direct sunlight without stirring. Aliquots (3 mL) were collected periodically, centrifuged (10,000 rpm, 5 min), and analysed by UV–Vis spectrophotometry at λmax = 664 nm.

Characterization techniques

X-ray diffraction (XRD) patterns were recorded using an X'Pert Pro Panalytical diffractometer and a Bruker D8 ADVANCE system (USA), with a 0.02° step size and analysed via X'Pert HighScore Plus software. Functional groups were identified by attenuated total reflection Fourier-transform infrared (ATR-FTIR)

spectroscopy (PerkinElmer Spectrum, 400–4000 cm^{-1}) equipped with an ATR accessory.

Dynamic Light Scattering was determined using a Zeta meter (Zetasizer ZS90, Malvern, UK). 0.1 g of SA was introduced into 100 mL of ultrapure water under ultrasonic stirring at 500 rpm for 5 min. Then, 0.75 mL was injected into the ZP measuring cell in the zeta meter instrument.

Surface morphology was examined by scanning electron microscopy (SEM) using a Quanta 650 microscope (30× to 200,000× magnification, 0.2–30 kV accelerating voltage). Optical properties were investigated with a Shimadzu UV-1800 spectrophotometer (Kyoto, Japan). A scanning electron microscope (SEM), specifically the FEI Quanta 650, was used for surface examination.

RESULTS AND DISCUSSION

XRD

The X-ray diffraction (XRD) pattern of the synthesised Zn nanoparticles, prepared using olive leaf extract, was recorded to investigate their crystallographic structure. The pattern exhibits characteristic peaks corresponding to the wurtzite (hexagonal) phase of ZnO, confirming the successful synthesis of crystalline zinc oxide. Prominent reflection peaks were observed at 2θ = 31.3° (100), 34° (002), and 35.8° (101), which are consistent with the hexagonal wurtzite structure of ZnO [29, 30]. These findings are in agreement with previous studies on plant-mediated synthesis of ZnO nanoparticles [22]. Additional peaks, if present, at 47.1° (102), 56.2° (103), or 62.5° (110), further support the wurtzite phase, though their intensities may vary depending on synthesis conditions such as the 80°C extraction temperature used in this study. No significant peaks corresponding to impurities ($Zn(OH)_2$ or unreacted Zn precursors) were observed, indicating a high purity of the ZnO phase.

FTIR

The successful formation of zinc oxide was further confirmed by the Fourier transform infrared spectrum, as illustrated in figure 2. The spectrum revealed characteristic absorption peaks at 3376, 1582, 1400, 1020, 747, 664, and 483 cm^{-1}. The broad bands observed at 3376 cm^{-1} are attributed to O-H stretching vibrations, indicating the presence of adsorbed water molecules and/or surface hydroxyl groups on the material [23], which serve as active sites for photocatalytic radical generation. Crucially, the peaks at 1582 cm^{-1} and 1400 cm^{-1} are assigned to the asymmetric stretching (v3) of carbonate (CO_3^{2-}) species [24]. These inorganic carbonates, along with the C–O stretching at 1020 cm^{-1}, represent a stable carbonaceous framework resulting from the high-temperature (900°C) thermal decomposition of the Olea europaea extract [25]. Finally, the strong absorption bands at 664 cm^{-1} and 483 cm^{-1} are characteristic of the metal-oxygen (Zn-O) stretching vibrations in the hexagonal wurtzite structure of zinc oxide [22].

DLS

The Dynamic Light Scattering (DLS) analysis of Zinc Oxide nanoparticles synthesised using Olive Leaf Extract provides crucial insight into their colloidal characteristics (figure 3). The analysis shows a bimodal size distribution characterised by two distinct peaks: the primary peak at 973.1 nm representing 66.5% of the intensity distribution, and a secondary

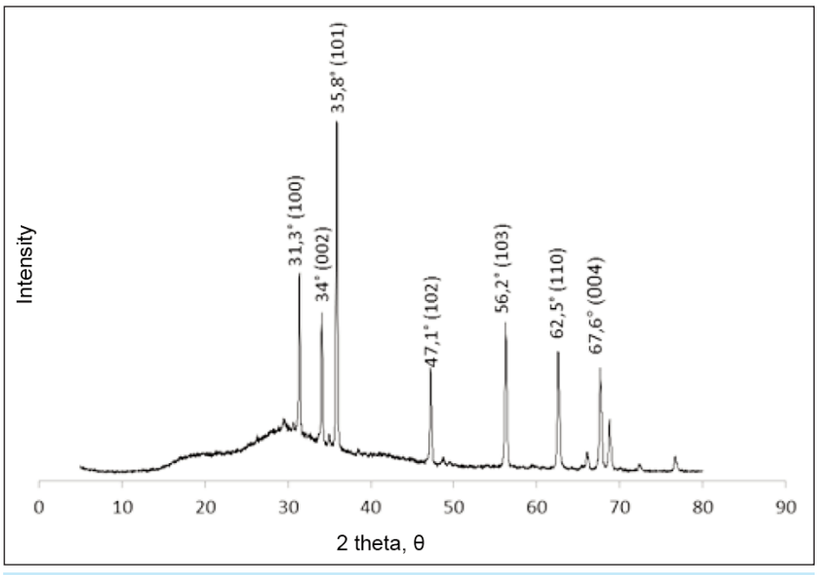

Fig. 1. XRD pattern of Zn nanoparticles synthesised using olive leaf extract

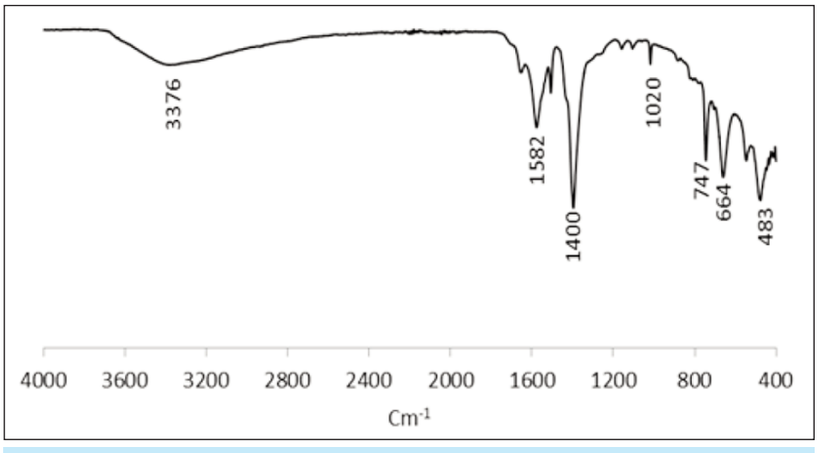

Fig. 2. FTIR spectrum of the synthesised ZnO nanoparticles

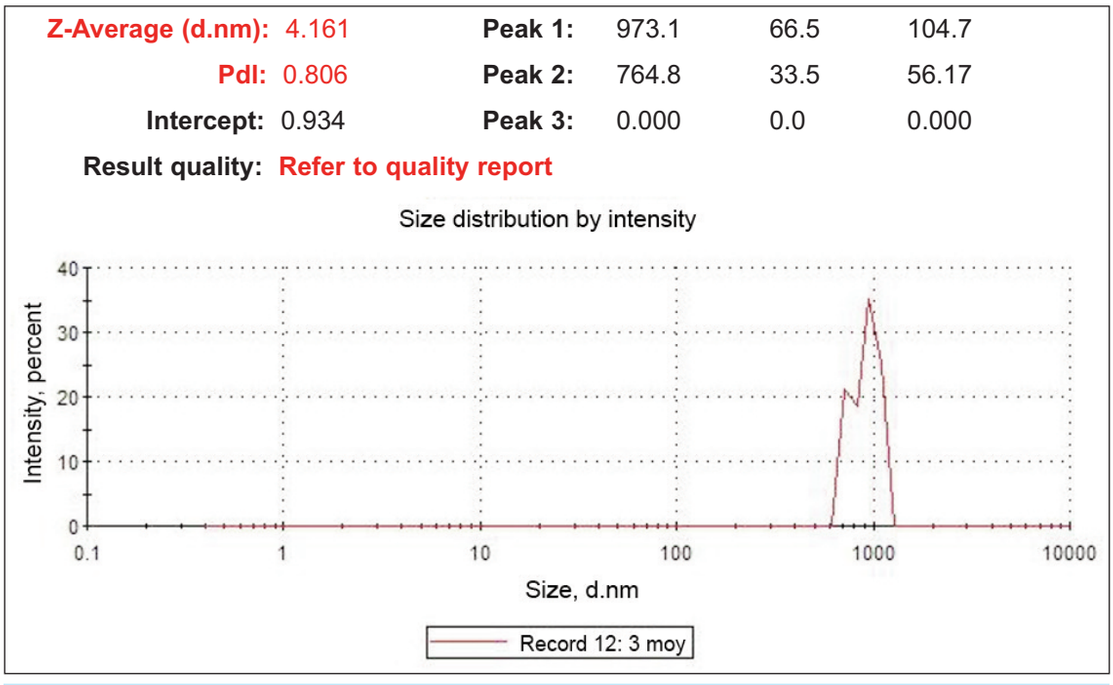

Fig. 3. Dynamic Light Scattering (DLS) analysis of Zinc Oxide nanoparticles synthesised using olive leaf extract

peak at 764.8 nm accounting for 33.5% of the intensity. This bimodal pattern suggests the coexistence of two different populations of particle aggregates in the suspension.

SEM

Figure 4 displays SEM images of ZnO nanostructures synthesised via conventional chemical precipitation and green synthesis using Olea europaea leaf extract. Conventional ZnO (figure 4, *a*) exhibits irregularly shaped, highly agglomerated particles forming rough, coral-like porous clusters (200–800 nm). The heterogeneous surface, covered with fused granules lacking defined facets, results from rapid NaOH-induced supersaturation, homogeneous nucleation, and uncontrolled dehydration of $Zn(OH)_2$ without capping agents. This morphology is typical of chemical routes favouring bulk growth over shape control [24].

The morphological characteristics were further investigated using SEM and DLS to distinguish between primary crystal size and colloidal behaviour. SEM analysis (figure 4, *b*) reveals that green ZnO consists of well-defined hexagonal plates with primary lateral dimensions of 200–500 nm [25] with thicknesses estimated between 100 and 200 nanometers. DLS showed a Z-average diameter of 4161 nm with a bimodal distribution (peaks at 973.1 nm and 764.8 nm). This discrepancy is common in green synthesis; while SEM captures the “dry” inorganic core, DLS measures the hydrodynamic diameter, which includes the solvation shell and the carbonaceous framework surrounding the particles. The larger DLS values indicate that in aqueous suspension, these hexagonal plates form stable, loosely bound agglomerated clusters. For textile applications, such clusters are often beneficial as they facilitate better entrapment within the microfiber networks of cotton during the dip-coating process.

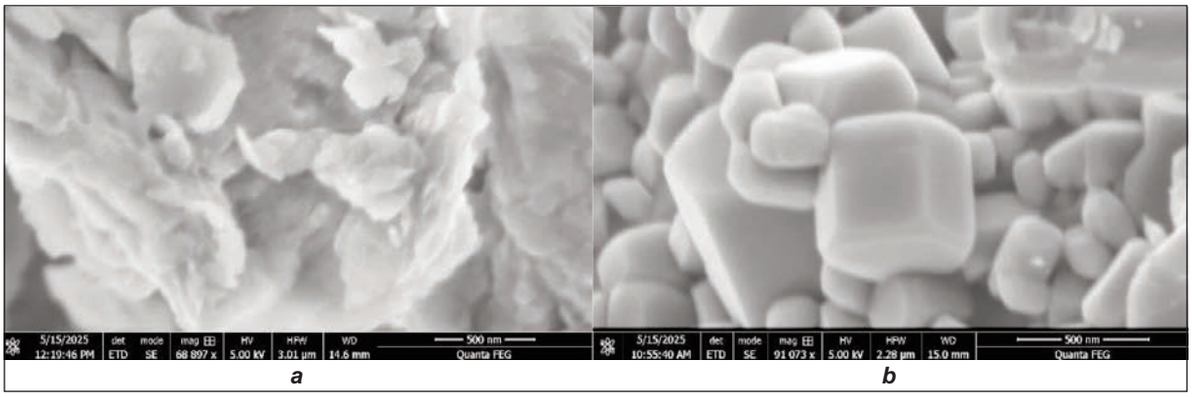

Fig. 4. SEM image of: *a* – chemically synthesised ZnO; *b* – olive leaf extract mediated ZnO

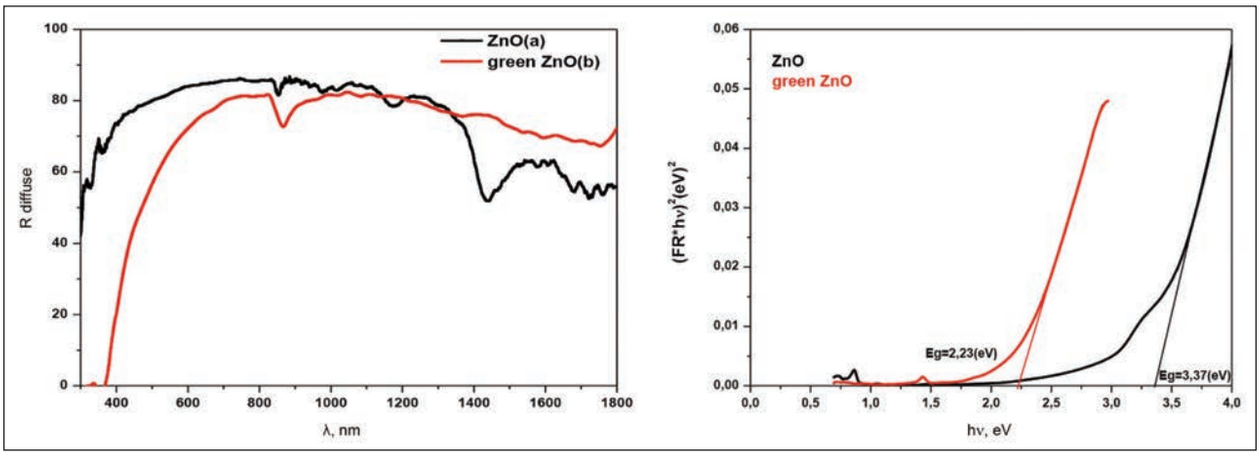

Fig. 5. Reflectance spectra and determination of the band gap of green and chemically synthesised ZnO

Reflectance spectroscopy and determination of band gap using Kubelka-Munk model

Figure 5 presents the reflectance spectra of green-synthesised and conventional ZnO across the UV-Vis-NIR range, revealing distinct optical behaviours. Conventional ZnO exhibits high reflectance (~80%) in the visible region, characteristic of high crystalline quality and low defect density. In contrast, green ZnO displays significantly lower reflectance and a pronounced absorption tail extending into the visible region, indicating enhanced light-harvesting capability. Band gap energies were determined using the Kubelka-Munk function combined with Tauc plots (figure 5).

Conventional ZnO shows a direct band gap of 3.37 eV, typical for pure ZnO. Remarkably, green ZnO exhibits a reduced band gap of 2.26 eV, representing a 33% narrowing. This substantial red-shift arises from the in situ incorporation of carbon and heteroatoms (N, O) resulting from the thermal decomposition of *Olea europaea* leaf extract during the 900°C calcination process. These species, along with induced oxygen vacancies, introduce mid-gap states and carbon-based defect levels within the ZnO lattice. The lowered band gap enables green ZnO to absorb a broader portion of the solar spectrum, particularly in the visible range, making it far more efficient for sunlight-driven photocatalytic applications compared to conventional ZnO, which is primarily UV-active. This optical tuning, achieved solely through eco-friendly precursors, eliminates the need for doping with rare-earth or transition metals, preserving the non-toxic nature of the material.

Antimicrobial activity

The antimicrobial activity of green-synthesised ZnO nanoparticles was investigated against two microorganisms: *Candida albicans* (C.a) and *Staphylococcus aureus* (S.a), employing the standard disk diffusion method as presented in figure 6. As shown, both microbial strains revealed clear zones of inhibition around the ZnO-loaded disk, confirming the broad-spectrum antimicrobial potential of the biosynthesised nanoparticles. *C. albicans* demonstrated a substantially larger inhibition zone with a diameter of 28 mm compared to *S. aureus* (16 mm in diameter), suggesting greater susceptibility of the fungal pathogen to ZnO nanoparticles. The observed inhibition zones suggest that the green-synthesised ZnO nanoparticles exert their antimicrobial effects through multiple mechanisms, including membrane disruption and interference with cellular metabolic processes. These results show the promising potential of eco-friendly synthesised ZnO nanoparticles as effective antimicrobial agents against both fungal and bacterial pathogens, investigation for biomedical and pharmaceutical applications.

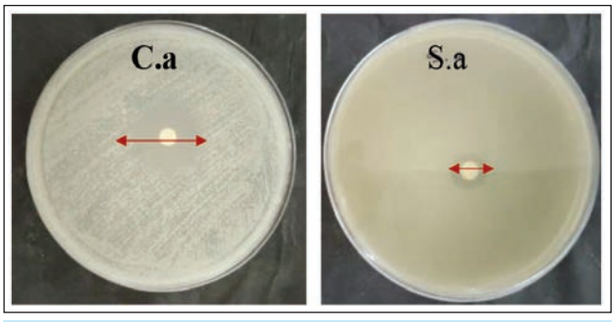

Fig. 6. Antimicrobial activity for *Candida albicans* (C.a) and *Staphylococcus aureus* (S.a)

Adsorption and photocatalytic properties

Figure 7 compares the adsorption performance of green-synthesised ZnO and chemically synthesised ZnO toward MB. The kinetic profile reveals rapid initial uptake within 50 min for both materials, reaching equilibrium by ~60 min. Green ZnO achieves a significantly higher equilibrium capacity ($q_e \approx 52$ mg/g) than chemical ZnO ($q_e \approx 43$ mg/g), representing a 21% enhancement. This superiority stems from polyphenolic capping layers on green ZnO, which provide additional hydroxyl and aromatic groups for electrostatic attraction, hydrogen bonding, and π–π stacking with cationic MB. The adsorption isotherm, conducted at equilibrium, shows that green ZnO maintains higher uptake across the entire concentration range (5–60 mg/L). At 50 mg/L initial MB, green

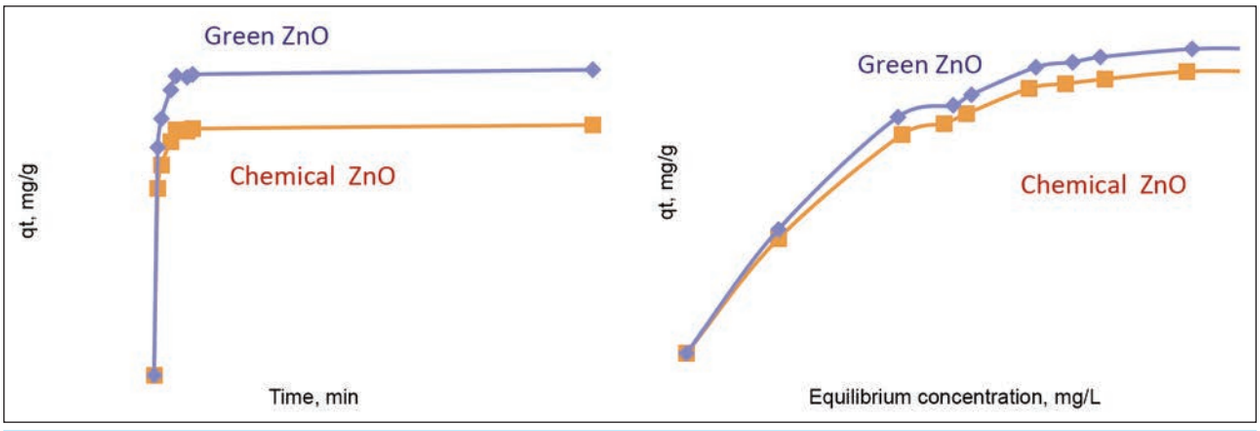

Fig. 7. Kinetic and adsorption isotherm for MB of green and chemical synthetized ZnO

ZnO removes ~94% of the dye, compared to ~78% by chemical ZnO. The steeper initial slope for green ZnO indicates stronger affinity, while the plateau suggests monolayer coverage consistent with Langmuir behaviour. The organic residues not only increase active binding sites but also create surface defects that improve dispersibility and prevent aggregation, ensuring more accessible surface area. Figure 8 compares the photocatalytic degradation of MB under natural sunlight using green-synthesised and conventional ZnO nanoparticles. The process includes a 24-h dark adsorption phase followed by solar exposure, ensuring that observed decolourisation reflects true photocatalysis rather than mere adsorption. Green ZnO demonstrates superior performance, achieving 94% MB degradation within 3 h under static conditions, compared to 88% for chemical ZnO. This enhanced activity stems from its narrowed band gap (2.6 eV) caused by phytochemical-induced defects and mid-gap states, extending absorption into the visible region (up to ~477 nm) and enabling efficient utilisation of the solar spectrum. Conversely, chemical ZnO, with a wider band gap (3.37 eV), relies primarily on UV light (<368 nm). Although its highly crystalline structure promotes charge separation and ROS generation, limited visible-light absorption restricts overall efficiency.

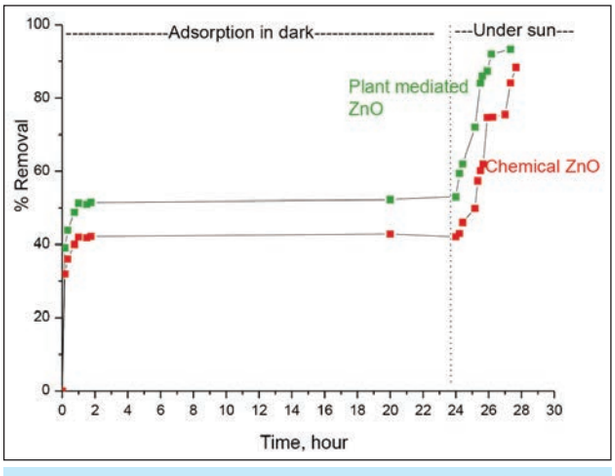

Fig. 8. MB removal using chemical ZnO and plant-mediated ZnO adsorption in dark and photocatalytic degradation under sunlight

Proposed mechanism

Figure 9 presents sunlight photocatalytic activity enhancement and mechanism of green ZnO prepared with olive leaf extract. The green synthesis method yields ZnO with superior photocatalytic performance compared to conventional ZnO's wurtzite structure. Chemical synthesis using NaOH yields near-stoichiometric ZnO with band gaps around 3.37 eV, due to minimal defects and high crystallinity. In contrast, green synthesis employing olive leaf extract results in a reduced band gap of ~2.6 eV, enhancing photocatalytic and antimicrobial applications by extending absorption into the visible spectrum. The proposed mechanism centres on the multifaceted role of olive leaf phytochemicals, primarily polyphenols like oleuropein, hydroxytyrosol, and flavonoids, which serve as bioreductants, stabilisers, and inadvertent dopants. During synthesis, Zn^{2+} ions from zinc acetate are reduced to ZnO nuclei via electron donation from phenolic -OH groups, forming

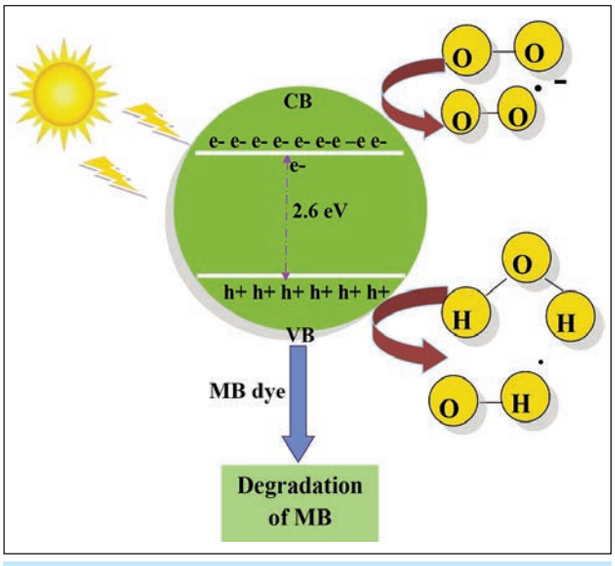

Fig. 9. Sunlight photodegradation of MB by green ZnO prepared with olive leaf extract

Zn-O-C bonds at the surface. This capping prevents agglomeration but introduces organic residues. Upon partial decomposition of these organics, oxygen vacancies and interstitial oxygen defects are generated, which contribute to narrowing the band gap by ~0.7 eV. Simultaneously, carbon from decomposed phenolics forms impurity bands that overlap with valence/conduction bands.

Implications for textile functionalization

While this study primarily focuses on the synthesis and physicochemical optimisation of green-synthesised ZnO, the findings have significant implications for the development of bioactive textiles. The hexagonal prismatic morphology (200–500 nm) and the resulting high surface area to volume ratio are critical for increasing the density of active sites available for dye adsorption and bacterial interaction [26].

Furthermore, the in situ carbon-doped framework and stable carbonate species products of the 900 °C thermal treatment enable high photocatalytic efficiency (94%) under natural solar irradiation.

From a processing perspective, the bimodal cluster distribution observed in DLS is expected to facilitate better mechanical interlocking within the porous cotton microfiber matrix during standard dip-dry-cure applications [27, 28]. This provides a distinct advantage over conventional chemical ZnO, which lacks these specific surface modifications. Future research will investigate the wash-fastness and laundering durability of these particles when immobilised using green cross-linking agents, such as citric acid, to ensure long-term functionality in smart textile substrates [29].

CONCLUSION

This study successfully demonstrates a fully green, scalable synthesis of ZnO nanoparticles using *Olea europaea* leaf extract as a precursor and template, offering a sustainable alternative to conventional chemical routes. The resulting green ZnO exhibits superior physicochemical properties, hexagonal prismatic morphology, a narrowed band gap (2.6 eV), and abundant surface defects, marking it as a highly promising multifunctional textile finishing agent. Compared to chemical ZnO, the green variant achieves 21% higher methylene blue adsorption (52 mg/g), 94% photocatalytic degradation within 3 h under natural sunlight, and stronger antimicrobial activity against *Staphylococcus aureus* and *Candida albicans*. These improvements stem from the in situ carbon modification and induced oxygen vacancies resulting from the thermal treatment of the plant-based precursors, which increase active sites and extend visible-light responsiveness. By proposing the replacement of multiple toxic finishes with a single bio-derived nanomaterial, this approach significantly reduces environmental impact, aligns with circular economy principles, and meets the textile industry's demand for eco-friendly, high-performance solutions for future smart fabric development. By bridging the gap between waste-derived green synthesis and high-performance textile finishing, this study demonstrates a sustainable pathway for producing multifunctional, bioactive fabrics with significant industrial potential.

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