Synthesis of ZnO flower-like structure into wood substrate to enhance thermal stability

Syntéza květovité struktury ZnO do dřevěného substrátu pro zvýšení tepelné stability

Paul D., Tesarova D.

Mendel University in Brno, Brno, Czech Republic, dabosmitapaul@gmail.com

Abstract

The development of self-coated bio-based composites with improved thermal resistance is an important step toward sustainable materials for construction and energy applications. In this study, flower-like zinc oxide (ZnO) micro/nanostructures were synthesised and deposited onto wood substrates to improve their thermal stability. The morphology and chemical features of the ZnO-coated wood were examined using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and Fourier transform infrared spectroscopy (FTIR). The findings show that ZnO-treated wood exhibits greater resistance to thermal degradation, underscoring the role of hierarchical micro/nanostructures in enhancing material performance.

Key words

ZnO; flower-like micro/nanostructures; wood substrate; thermal stability.

Introduction

Wood, as a naturally abundant, renewable, and lightweight material, is increasingly utilised in sustainable construction and composite design[1], [2], [3]. However, its intrinsic limitations, particularly low thermal stability and susceptibility to degradation at elevated temperatures, restrict its long-term performance in advanced applications[2].

Zinc oxide (ZnO), a wide-bandgap semiconductor (Eg ≈ 3.37 eV) with high thermal conductivity, ultraviolet (UV) absorption capacity, and chemical stability, is an attractive candidate for modifying wood [4]. Among the various nanostructures, flower-like ZnO assemblies comprising radially oriented nanosheets or nanorods offer a high surface area, anisotropic growth, and hierarchical porosity, which are expected to enhance coating efficiency and thermal barrier properties [5].

This work reports the synthesis of flower-like ZnO nanostructures via a wet chemical route and their subsequent impregnation into wood substrates. Structural characterisation using SEM, EDS, and FTIR provides insights into the morphology property relationship. As a result, interfacial mechanisms governing thermal stability enhancement are analysed in terms of bonding interactions and suppression of thermally activated degradation pathways.

Materials and Methods

Materials

Wood substrate: Scots pine (*Pinus sylvestris L.)* sapwood (dimensions: 20 × 20 × 5 mm), conditioned at 20 °C and 65% relative humidity.

Chemicals: Zinc nitrate hexahydrate [$Zn(NO_3)_2 \cdot 6H_2O$], ammonia solution (NH_3), starch, and deionised water. All reagents were of analytical grade.

Synthesis Methods of Flower-Like ZnO into Wood Substrate

The ZnO precursor solution was prepared by dissolving 8.94 g of $Zn(NO_3)_2 \cdot 6H_2O$ in 300 mL of distilled water and stirring for 20 min. Separately, 3 g of starch was dissolved in 150 mL of distilled water, then combined with 150 mL of warm water and stirred for an additional 10 min. The two solutions were mixed, and NH_3 was added gradually to adjust the pH to 8.5-9, promoting complete precipitation of $Zn(OH)_2$. The resulting mixture was stirred for 30 min at room temperature. Pre-dried wood specimens were then placed in the solution under vacuum conditions (0.8 bar for 20 min), followed by pressure treatment at 0.01 MPa for 1 h. After impregnation, the samples were kept in the solution for 24 h at 23˚C, dried at 80˚C for 24 h, and finally cured at 120˚C for 2 h to complete the ZnO incorporation process.

Characterisation Methods

SEM and EDS: Morphology of ZnO nanostructures and distribution on wood surface, Elemental mapping to confirm Zn and O presence. To observe the microstructural characteristics of the materials, the samples were prepared in longitudinal sections for both untreated and treated samples by cutting them with a sharp blade. These sections were then mounted onto conductive carbon tape and coated with a layer of 15 µm thick gold using a sputtering technique. Subsequently, the analysis was performed using a scanning electron microscope (Mira-STAN instrument from Tescan), with an acceleration voltage of 5 kV and a beam current of 10 pA to observe the samples in detail. EDS was carried out using an Oxford Instruments AZtecOne system to analyse the composition and distribution of elements within the samples.

FTIR: Chemical interactions between ZnO and wood constituents (cellulose, hemicellulose, lignin). ATR-FTIR spectroscopy was performed with a Bruker Invenio S. The sample and background scans were each conducted 32 scans, with data collected in the range of 4,000–400 cm^{-1}. The measurement time was 15 s in transmittance mode. OPUS software was used to analyse the results.

Thermal analysis: Thermogravimetric (TGA) evaluates the thermal stability by Discovery TGA, TA . Heating ramp from 30 to 700 °C (heating rate 20 °C/min, N2 atmosphere), isothermal 700 °C/5 min (air), 5-10 mg of sample

Results and Discussion

Morphology Analysis

The SEM micrograph of Figure 1(a) reveals the cross-sectional cellular structure of wood, with elongated cell lumina and layered cell walls visible, indicating that the sample preparation preserved its natural morphology without collapse. The image highlights the surface topography and arrangement of vessels, fibres, and pits, while showing no obvious deposition of foreign nanoparticles or coatings, thus serving mainly to confirm the ultrastructure of the untreated or gold-coated sample. The corresponding EDS spectrum of Figure 1(b) from the same region

identifies carbon (C) and oxygen (O) as the dominant elements, consistent with the cellulose, hemicellulose, and lignin composition of wood, while strong gold (Au) peaks arise from the conductive sputter coating applied to prevent charging during SEM analysis. The relative composition shows that C and O contribute approximately 80% by weight, reflecting the organic nature of the material.

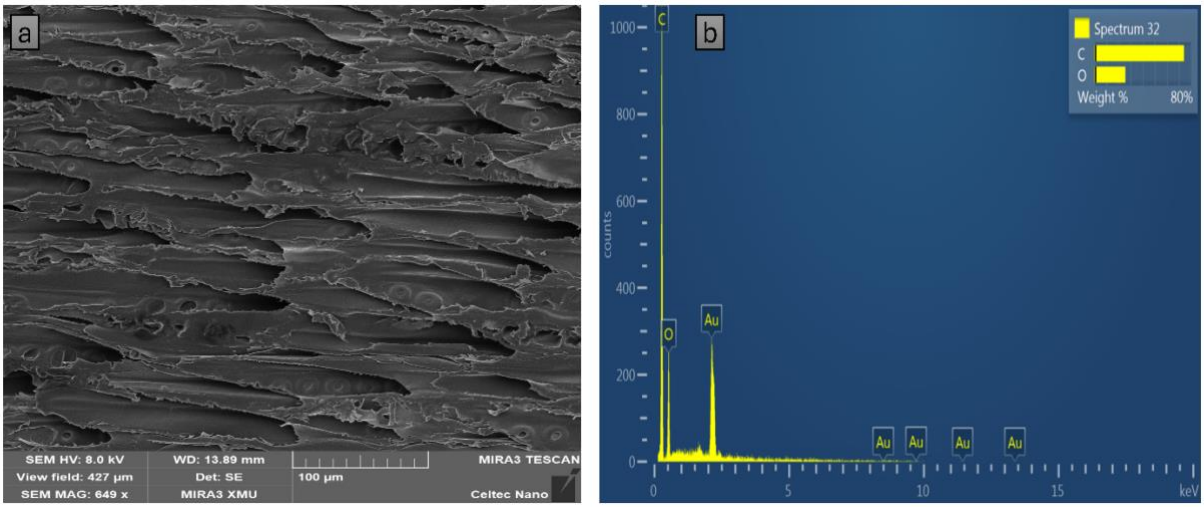

Figure 1. SEM image and EDS micrograph: (a) pine surface morphology; (b) pine elemental composition

The SEM micrograph Figure 2 (a) presents a high-magnification view of the wood surface after ZnO treatment. Distinct flower-like structures can be seen distributed across the cell wall surfaces. These are characteristic morphologies of ZnO formed through nucleation and growth, where nanosheets or nanoplates assemble radially to form petal-like architectures [6]. Such formations increase the surface roughness considerably and are known to enhance properties like hydrophobicity, UV shielding, and in some cases, antimicrobial activity [7], [8]. The underlying fibrous wood anatomy is still discernible, but it is partially covered by the dense deposition of these ZnO micro-/nanoflowers. The complementary EDS spectrum, Figure 2 (b), confirms the presence of zinc (Zn) as a major element, along with carbon (C) and oxygen (O) originating from both ZnO and the organic wood matrix. The Zn peak is dominant, with ~48.3 wt% contribution, supporting the effective loading and deposition of ZnO on the wood surface. Strong Au signals are also present, arising from the thin conductive gold coating applied before SEM imaging. Together, SEM and EDS demonstrate the successful formation and anchoring of ZnO flower-like nanostructures on the wood substrate.

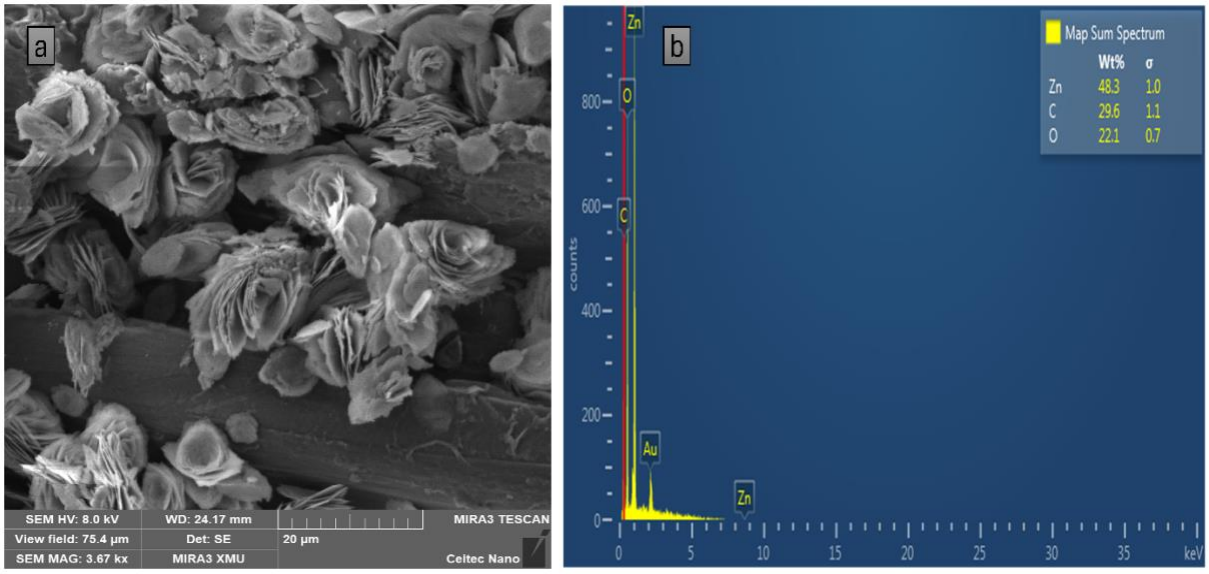

Figure 2. SEM image and EDS micrograph: (a) pine treated with ZnO surface morphology; (b) pine treated with ZnO elemental composition.

SEM and EDS analyses revealed clear structural and compositional differences between untreated and ZnO-treated wood. The untreated sample retained intact cellular morphology with carbon and oxygen as the dominant elements, confirming its organic composition. In contrast, ZnO-treated wood exhibited dense flower-like nanostructures on the surface, characteristic of ZnO crystal growth through nanosheet assembly. EDS confirmed high Zn content (~48 wt%), indicating effective deposition and strong anchoring of ZnO on the substrate. These nanostructures are expected to enhance hydrophobicity, UV resistance, and antimicrobial performance, demonstrating successful functionalisation of the wood surface. However, from the previous research, it was found that the synthesis of flower-like ZnO structures from self-assembled nanorods has been widely reported, with their hierarchical morphology shown to enhance surface area and stability [9]. On the other hand, from another research, it was observed that ZnO flower-like architectures also exhibit distinct photoluminescence properties, confirming their potential for multifunctional applications in optoelectronics and surface functionalisation [5].

Chemical Analysis

This FTIR spectrum Figure 3. compares the control pine sample (Pine_C, blue) with the treated pine sample (Pine_T, red), highlighting changes in functional groups associated with the treatment. Broad O–H stretching bands at ~3342 cm^{-1} (treated) and ~3330 cm^{-1} (control) correspond to hydroxyl groups in cellulose, hemicellulose, and absorbed water. The slight shift indicates altered hydrogen bonding due to treatment. The C=O stretching appears at ~1741 cm^{-1} (treated) and ~1734 cm^{-1} (control), associated with ester or carboxyl groups in hemicellulose. Reduced intensity in the treated sample suggests partial degradation or modification of hemicellulose. Aromatic skeletal vibrations from lignin occur at ~1516 cm^{-1} (treated) and ~1510 cm^{-1} (control). The difference in peak intensity indicates lignin modification during treatment. C–O stretching of cellulose and hemicellulose is observed around 1031 cm^{-1} in the control, but appears weakened in the treated sample, suggesting a chemical interaction or

reduction in polysaccharide content. Additional bands in the treated sample at ~513, 479, and 435 cm^{-1} correspond to Zn–O stretching vibrations, confirming the incorporation of ZnO nanostructures into the wood matrix.

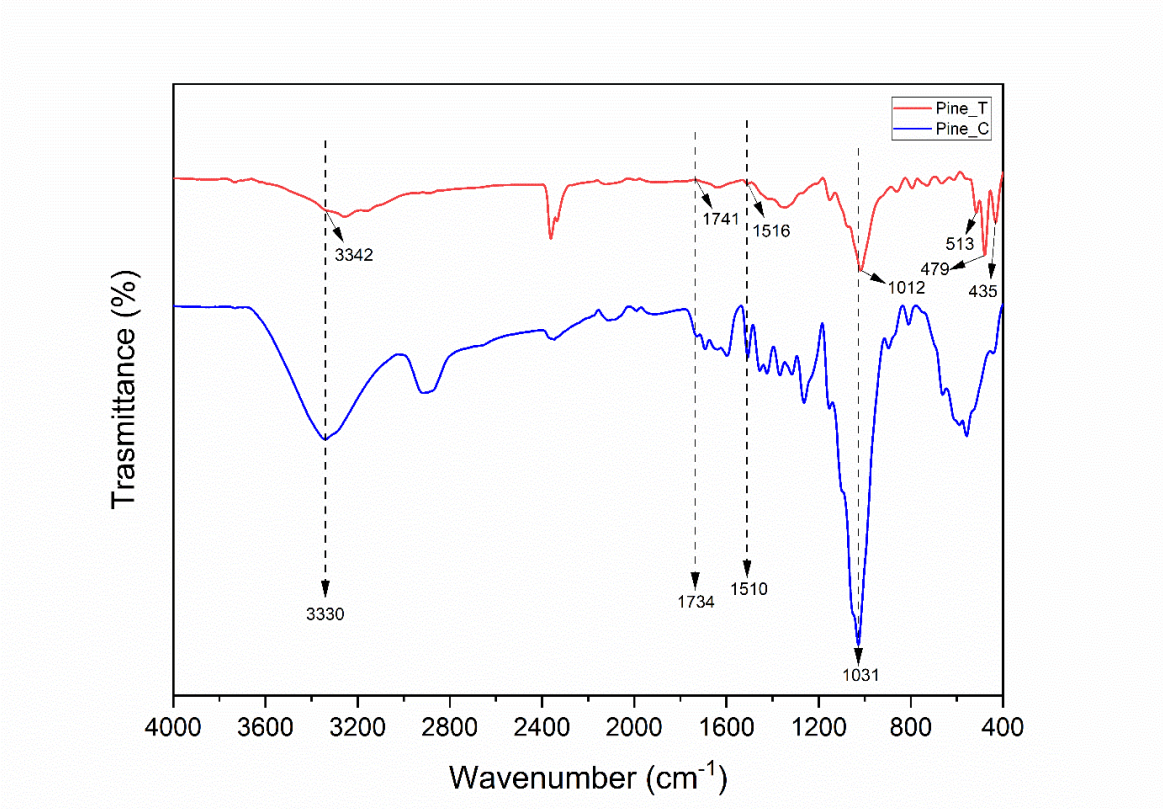

Figure 3. FTIR spectra of treated (Pine_T) and control (Pine_C).

The FTIR analysis demonstrates that treatment modifies the wood's chemical structure by affecting hemicellulose and lignin functionalities while introducing Zn–O bonds, which verifies the successful integration of ZnO into the pine substrate [9].

Thermal Stability Enhancement

TGA curve of Figure 4 compares the thermal behaviour of control pine (Pine_C, blue dashed line) and treated pine (Pine_T, red solid line). In the first stage (up to ~120 °C), both samples lose a small amount of weight due to the evaporation of free water, with Pine_T showing slightly lower mass loss, suggesting that treatment reduced moisture availability by modifying hydroxyl groups or pore structure. The second stage (120–250 °C) corresponds to the release of bound water and the onset of hemicellulose degradation; here, Pine_T shows delayed and reduced weight loss compared to Pine_C, indicating improved stability. The major decomposition phase (250–400 °C), mainly linked to cellulose degradation and partial lignin breakdown, is more gradual in Pine_T, while Pine_C undergoes faster weight reduction, confirming that treatment slows thermal decomposition. Beyond 400 °C, both samples stabilise, leaving char residue; Pine_T retains significantly more residue, reflecting the presence of thermally stable ZnO and enhanced char formation.

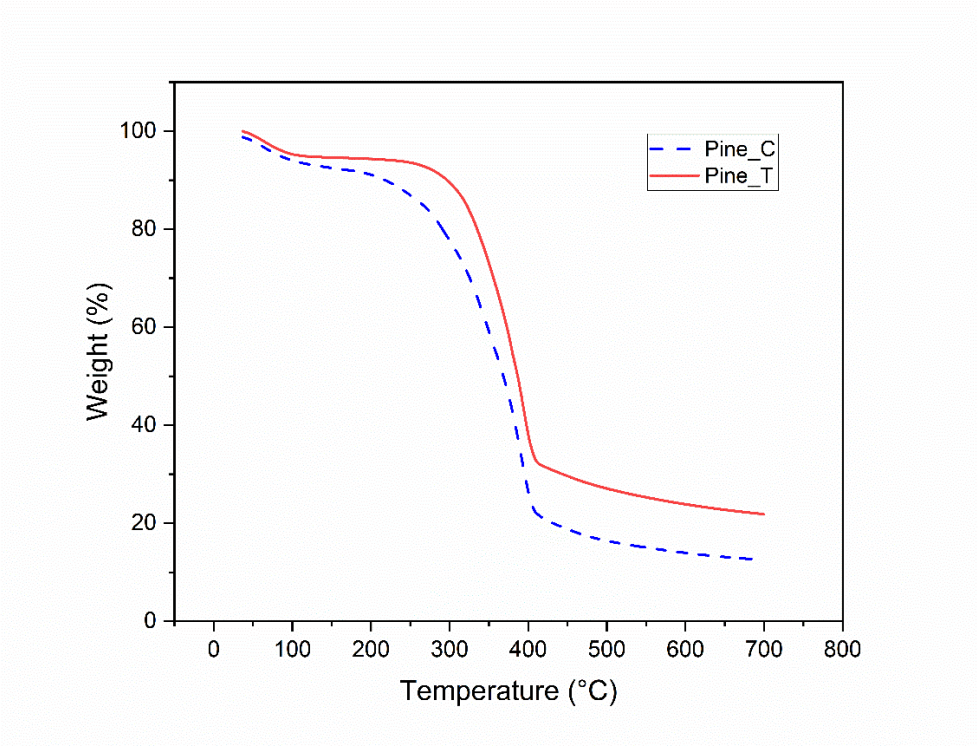

Figure 4. Shows TGA curve of treated (Pine_T) and control (Pine_C).

Overall, the treated sample demonstrates higher thermal stability, reduced moisture loss, delayed onset of thermal degradation, and greater residual mass than the control, indicating that the modification improves the physical resilience of wood under heating and allows it to retain its structure longer while resisting decomposition more effectively [10], [11].

Conclusions

In this study, ZnO flower-like nanostructures were successfully synthesised in situ within wood through vacuum-pressure impregnation using zinc nitrate and ammonia-based precursors. SEM and EDS confirmed the formation and anchoring of ZnO micro-/nanoflowers on the wood surface, while FTIR analysis verified chemical bonding through Zn-O incorporation. TGA results demonstrated that these structures enhanced thermal stability by reducing moisture loss, delaying degradation, and increasing char residue. Such functionalisation not only improves durability but also suggests potential applications in furniture, outdoor construction, and protective wood coatings. In addition, the semiconducting and photoluminescent properties of ZnO open opportunities for studies, including photocatalysis, optoelectronics, and the development of bio-based functional materials for advanced energy and sensing applications.

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