

Article

Microstructural Features of the Transition from Thermal Degradation to Initial Char Formation in Spruce Wood

Katarína Dúbravská ^{1,*} , Miroslava Mamoňová ^{2,*}  and Viera Kučerová ^{3,*} ¹ Department of Fire Protection, Faculty of Wood Sciences and Technology, Technical University in Zvolen, 96001 Zvolen, Slovakia² Department of Wood Science, Faculty of Wood Sciences and Technology, Technical University in Zvolen, 96001 Zvolen, Slovakia³ Department of Chemistry and Chemical Technology, Faculty of Wood Sciences and Technology, Technical University in Zvolen, 96001 Zvolen, Slovakia

* Correspondence: katarina.dubravska@tuzvo.sk (K.D.); mamonova@tuzvo.sk (M.M.); viera.kucerova@tuzvo.sk (V.K.)

Abstract

This study investigated microstructural, optical, and thermal changes in spruce wood (*Picea abies*) exposed to controlled laboratory heating to identify indicators associated with the transition from progressive thermal degradation to the initial char formation. Cubic specimens measuring 20 × 20 × 20 mm were exposed to selected temperatures between 240 and 300 °C under atmospheric conditions, with a 15 min isothermal exposure period. Microstructural changes were evaluated by scanning electron microscopy (SEM) and quantitative tracheid double cell wall measurements, supported by simultaneous thermal analysis (STA) and color and reflectance analyses. Simultaneous thermal analysis (TG/DTG/DSC) was performed on separate specimens from the same wood material to provide complementary thermal evidence. The most pronounced microstructural changes were observed between 250 and 260 °C, including substantial thinning of tracheid cell walls, degradation of bordered pits, increased brittleness, and localized structural collapse. Quantitative measurements showed reductions in double cell wall thickness exceeding 50% at 260 °C. TG/DTG analysis indicated the onset of intensive thermal degradation at 254.2 ± 1.48 °C, while optical measurements showed pronounced darkening and reduced differentiation of reflectance spectra above approximately 260 °C. The combined evaluation of complementary analytical methods indicates that the 250–260 °C interval represents a condition-dependent microstructural transition associated with accelerated thermal degradation and the early development of a charred structure under the applied experimental conditions. These findings provide complementary experimental evidence for interpreting the early stages of wood charring and may support the interpretation and future refinement of heat transfer and pyrolysis models. They complement, rather than replace, the conventional 300 °C engineering char line criterion used in structural fire design.



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Keywords: spruce wood; thermal degradation; initial char formation; microstructural features; scanning electron microscopy; tracheid cell wall thinning; spectrophotometry; TG/DTG/DSC; char line

1. Introduction

Wood is a lignocellulosic material composed primarily of cellulose, hemicelluloses, lignin, extractives, and inorganic constituents [1–4]. Its response to elevated temperature

is controlled by chemical composition, density, moisture content, anatomical structure, oxygen availability, heating rate, and heat transfer conditions [3,5,6]. During thermal exposure, these factors govern the progressive degradation of wood polymers, volatile release, structural shrinkage, cracking, and the gradual formation of a carbon-rich residue commonly referred to as char.

The thermal degradation of wood does not occur as a single sharply defined event. Hemicelluloses are generally the least thermally stable constituents and begin to degrade at relatively low temperatures, often around 160–180 °C [5,7,8], whereas cellulose decomposition becomes more intensive at higher temperatures, commonly within the range of approximately 250–350 °C [9]. Lignin degrades over a broader temperature interval and contributes to the formation of a carbonaceous residue during continued heating [9]. As a result, the transition from thermally modified wood to pyrolytically degraded and initially charred material is gradual rather than abrupt [10,11]. This gradual transition is particularly important because the formation of a charred zone affects heat transfer, oxygen diffusion, cracking, shrinkage, and the residual load-bearing cross-section of timber elements exposed to fire [12–16].

In structural fire engineering, the charred layer of a timber cross-section is generally assumed to have no effective load-bearing capacity, while the residual uncharred cross-section is used for load-bearing verification during fire exposure [3,16–22]. The boundary between these regions (the char line) is commonly approximated by a temperature isotherm. Eurocode 5 adopts the 300 °C isotherm as an engineering criterion for determining the position of the char line and for calculating the residual cross-section of timber members exposed to fire [22]. Although this criterion is widely used for structural design, it should not be interpreted as a direct microscopic or physicochemical definition of the first appearance of charred wood tissue.

Reported temperatures associated with the char front differ considerably in the literature. This variability reflects the different criteria and experimental approaches used to identify the char front. Values around 288 °C have been reported in experimental charring rate studies and are often linked to the base of the char layer under specific fire exposure conditions [13,23]. The 300 °C value is widely used as a practical engineering approximation, particularly in Eurocode-based structural fire design [22,23]. Higher values, up to approximately 360 °C, have also been reported in studies where the char front was defined using embedded thermocouples under cone calorimeter or other specific exposure conditions [7]. In contrast, lower temperatures around 250 °C are often associated with the onset of intensive thermal degradation, thermal modification limits, or early pyrolytic changes, rather than with a standardized structural char line definition.

These values are therefore not directly interchangeable. They originate from different methodological and engineering approaches used to define the char front, including visual assessment, thermocouple-based measurements, standard fire resistance testing, cone calorimeter exposure, thermal analysis, thermal modification studies, and engineering simplifications used for residual cross-section calculations. Therefore, temperatures associated with the char front should be interpreted within the methodological context in which they were determined rather than compared directly across different studies.

Figure 1 illustrates the influence of the selected temperature criterion on the interpreted position of the char front in a spruce CLT panel. In the cited example, the 300 °C isotherm represents the conventional engineering char line criterion, whereas the 120 °C isotherm indicates the zero-strength layer [24]. The figure is included to illustrate the engineering basis of char line determination rather than the microscopic transition interval investigated in the present study.

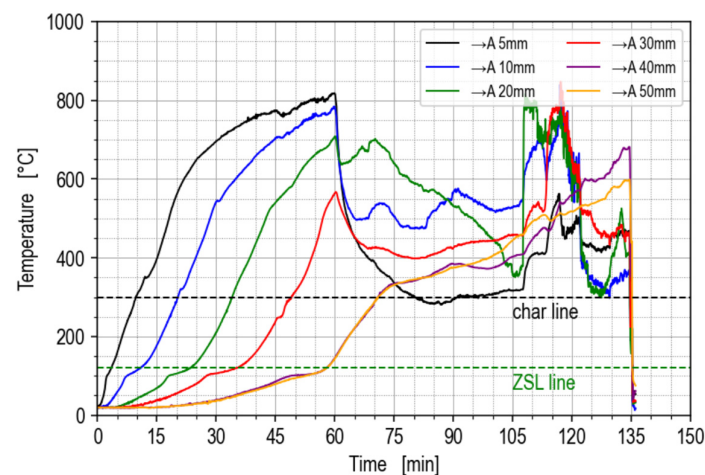


Figure 1. Heating and cooling phase temperature profiles for SIDE measuring locations. A (left)—depths indicated from the fire-exposed surface in the first lamella of a spruce CLT panel (thickness of the first layer = 40 mm). Char line = 300 °C isotherm; zero-strength layer (ZSL) line = 120 °C isotherm [24].

A similar approach was employed by [25] for spruce wood, where the charring rate was determined based on the char thickness corresponding to a temperature of 300 °C.

Although several methods have been proposed to determine charring temperature and charring rate, some approaches do not permit direct, continuous measurement of charring rate [26].

The choice of the charring temperature criterion directly affects the calculated char depth, apparent charring rate, residual cross-section, and predicted fire resistance of timber members. This is particularly relevant in numerical heat transfer and pyrolysis models, where the assumed degradation or charring boundary influences the predicted progression of the thermal degradation front and the assessment of the residual cross-section. However, although engineering criteria such as the 300 °C isotherm are well established for design purposes, they do not necessarily describe the earliest microscopic features of char formation.

The onset of initial char formation at the cellular level may be reflected by anatomical changes such as tracheid cell wall thinning, bordered pit degradation, torus disruption, increased brittleness, localized cracking, and structural collapse. These microscopic features may occur within a transition interval in which thermal degradation is already advanced, although the engineering char line, defined by a selected temperature isotherm, has not necessarily been reached.

Microscopic investigations of thermally degraded wood have been conducted for several decades. Using scanning electron microscopy, Fengel [27] examined spruce wood specimens exposed to temperatures of 180 °C and 200 °C for 24 h and reported drying-induced cracks along the middle lamella within the S1 layer. Structural alterations in bordered pits were already evident at 180 °C, including degradation of the torus–margo membrane, whereas exposure to 200 °C resulted in pronounced deformation and flow of the torus material. These observations provided insight into the anatomical response of wood to thermal degradation; however, the analyses were primarily qualitative and descriptive and were limited to temperatures not exceeding 200 °C. Further evidence of thermally induced microstructural deterioration was reported by McGinnes et al. [28], who examined the ultrastructure of pyrolyzed oak wood using electron microscopy. The earliest visible changes were observed in cell wall regions with a high lignin content. Upon reaching charring temperatures, the microfibrillar regions were completely destroyed, and the cell wall structure progressively transformed into an amorphous carbonaceous matrix.

Despite these early microscopic observations, quantitative assessment of microstructural damage, particularly under severe thermal degradation conditions, remains scarce.

The aim of this study is to identify microstructural and supporting analytical indicators associated with the transition from progressive thermal degradation to initial char formation in spruce wood under controlled laboratory exposure conditions. This study does not seek to establish a universal charring temperature or replace the conventional 300 °C engineering criterion used in structural fire design. Instead, it aims to provide microscopic evidence to support the interpretation of early char development in spruce wood.

It is hypothesized that pronounced microstructural degradation of spruce tracheids, characterized by cell wall thinning, bordered pit degradation, brittleness, and localized structural collapse, becomes evident within a narrower transition interval than the broad 250–300 °C range commonly associated with wood charring.

The novelty of this study lies in the combined evaluation of SEM-observed anatomical changes, quantitative tracheid cell wall thinning, optical surface changes, and TG/DTG/DSC degradation indicators within a single controlled exposure regime. This multi-method approach provides a comprehensive interpretation of the transition from thermal degradation to initial char formation rather than a single-temperature definition of charring.

2. Materials and Methods

2.1. Wood Material and Specimen Preparation

Norway spruce wood (*Picea abies* (L.) Karst.) originating from the University Forest Enterprise of the Technical University in Zvolen, central Slovakia, was used in this study. The material was harvested in 2025. Defect-free specimens with dimensions of 20 × 20 × 20 mm were prepared from the same wood batch, with attention given to fiber orientation and preservation of the main anatomical planes. The selected specimen size was chosen to enable controlled laboratory thermal exposure and subsequent preparation for scanning electron microscopy (SEM).

The average density of the spruce wood was $456 \text{ kg}\cdot\text{m}^{-3} \pm 5\%$, and the moisture content of the test specimens before thermal exposure ranged from 9.2% to 10.3%. The specimens were used without any surface treatment. Before thermal exposure, all specimens were conditioned at 21 ± 1 °C and $55 \pm 1\%$ relative humidity.

2.2. Experimental Design

The experimental design focused on identifying microstructural and supporting analytical indicators associated with the transition from progressive thermal degradation to initial char formation in spruce wood under fixed laboratory exposure conditions.

The thermal exposure program included seven target temperatures: 240, 250, 260, 270, 280, 290, and 300 °C. These temperatures were selected to cover the interval in which substantial physicochemical transformations of wood are expected, including the transition from thermal degradation toward initial char formation. The 300 °C level was included because this temperature is commonly used as an engineering criterion for the char line in structural fire design.

To improve the robustness of the SEM-based interpretation, additional independent specimens were included in the critical transition range. For the main quantitative SEM evaluation, three independent specimens were analyzed at each of the following temperature levels: reference, 240, 250, 260, and 270 °C. The 240–270 °C interval was selected because preliminary observations and previous literature indicate that this range captures the transition from early thermal degradation to more advanced microstructural deterioration.

This experimental structure was selected to distinguish between early thermal degradation, the microscopic transition interval, and more advanced thermal damage. This study does not aim to establish a universal charring temperature. Instead, it evaluates condition-dependent microstructural changes under the applied laboratory exposure regime.

2.3. Thermal Exposure of Test Specimens

Thermal exposure was performed in a laboratory muffle furnace under controlled atmospheric conditions. Each specimen was placed in a ceramic annealing dish positioned in the central region of the furnace chamber in order to reduce spatial temperature variation and to provide reproducible exposure conditions. The same specimen placement procedure was used for all temperature levels.

The thermal loading program consisted of two consecutive stages. In the first stage, the furnace temperature was increased from ambient temperature to the selected target temperature at a controlled heating rate of $2\text{--}3\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$. In the second stage, the specimens were maintained at the target temperature for 15 min. After the isothermal exposure, the specimens were removed from the furnace and allowed to cool under laboratory conditions at ambient temperature without forced convection.

Before the thermal exposure program, the furnace temperature field was checked using thermocouples positioned near the furnace sensor and at the specimen location inside the ceramic annealing dish. This procedure was used to minimize differences between the target furnace temperature and the actual temperature in the specimen exposure zone. Temperature verification was performed for the selected exposure levels before testing, and the same furnace program was used for all replicate specimens within each temperature group.

The selected temperature levels were 240, 250, 260, 270, 280, 290, and $300\text{ }^{\circ}\text{C}$, covering the range of significant physicochemical transformations in wood, including the transition from thermal decomposition to char formation. The upper temperature limit of $300\text{ }^{\circ}\text{C}$ was selected in accordance with the literature [22], where it is associated with the position of the char front.

The 15 min isothermal exposure period was selected based on preliminary experiments and previously published observations. Gosselink et al. [29] reported that thermal exposure for 15 min at comparable temperatures produced a relatively homogeneous carbonized structure, whereas shorter or longer exposure periods resulted in non-homogeneous carbonization or increased surface cracking and brittleness. Consistent with these observations, the thermally exposed spruce specimens in the present study exhibited a relatively uniform degradation pattern across the investigated cross-sections after splitting, without a pronounced visual transition between the surface and inner regions.

For each of the main SEM temperature groups, namely reference, 240, 250, 260, and $270\text{ }^{\circ}\text{C}$, three independent specimens were thermally exposed and analyzed.

2.4. Scanning Electron Microscopy

After thermal exposure and cooling, the specimens were prepared for SEM observation with respect to the anatomical orientation of spruce wood. The reference and thermally exposed specimens were split along a radial cleavage plane passing through the center of the specimen. The cleavage plane provided a radial observation surface. In addition, oblique transverse cuts were prepared at approximately 45° to improve the stability of the exposed anatomical surfaces [30,31].

The use of oblique sectioning was necessary because thermally degraded specimens were brittle and prone to surface disruption during conventional transverse preparation. Oblique sectioning provided smoother and more stable surfaces, reduced

preparation-induced damage and charge accumulation, and improved the visibility of cell wall boundaries.

Prepared wood sections were mounted on sample stubs using carbon tape and sputter-coated with gold in a Sputter Coater K650X (Quorum Technologies, Ashford, UK) under an argon atmosphere. The specimens were examined using high-vacuum scanning electron microscopy with a Tescan VEGA microscope (Tescan, Brno, Czech Republic) operated at 30 kV. The microscope was equipped with a tungsten filament electron source. Magnification, scale bar, accelerating voltage, and detector information were recorded directly in the SEM image data bar.

SEM observations were performed to identify microstructural features associated with progressive thermal degradation. Particular attention was paid to tracheid cell wall thinning, bordered pit degradation, torus disruption, increased brittleness, localized cracking, and structural collapse. These features were evaluated in both earlywood and latewood regions.

A backscattered electron detector was used to support the observation of thermally induced changes in tracheid walls and bordered pits. The presence, deformation, or absence of the torus in bordered pits of earlywood tracheids was used as one of the qualitative microscopic features of degradation.

SEM observations were performed in both near-surface regions and central regions of the sectioned specimens. The near-surface region was used to evaluate the earliest visible changes associated with thermal exposure, while the central region was examined to assess the degree of internal structural modification after the 15 min isothermal exposure. The same observation strategy was applied to the reference specimens and to all thermally exposed specimens.

2.5. Quantitative Measurement of Tracheid Double Cell Wall Thickness

Tracheid double cell wall thickness was measured from SEM micrographs using the length measurement tool. Measurements were performed separately for earlywood (Figure 2a) and latewood tracheids (Figure 2b). Since tracheid wall thickness in *Picea abies* varies systematically within an annual ring [32,33], the measurement region was defined to include both earlywood and latewood zones. The same measurement approach was applied to the reference specimens and to specimens exposed at each selected temperature.

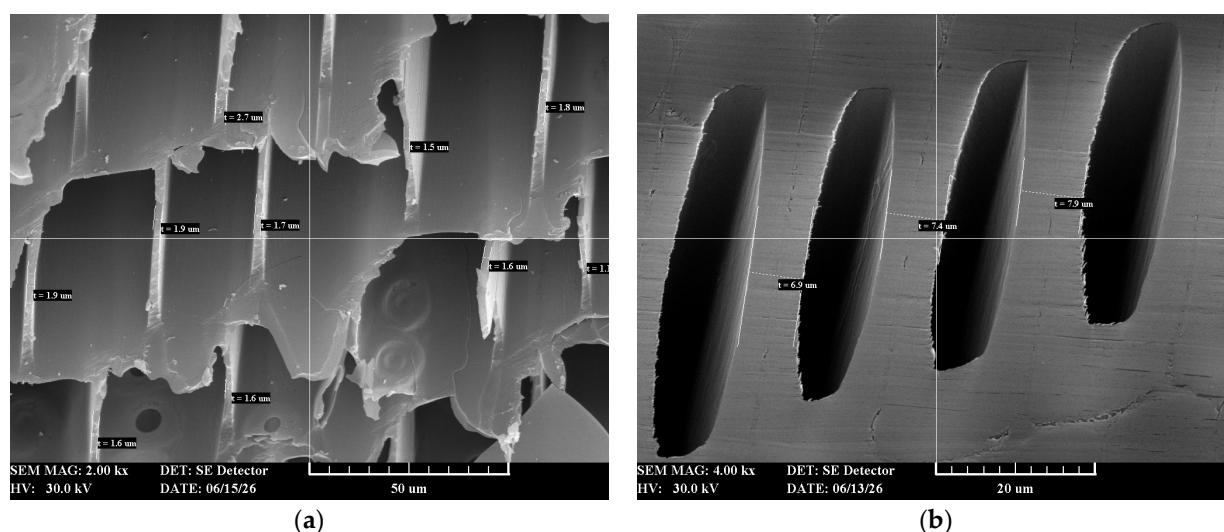


Figure 2. Measurement of tracheid double cell wall thickness in (a) earlywood and (b) latewood regions of spruce wood (*Picea abies*) as microstructural indicator of thermal degradation.

For the main quantitative evaluation, three independent specimens were analyzed for each of the following groups: reference, 240, 250, 260, and 270 °C. Each specimen contained approximately 8–10 annual rings within the selected observation region. For each temperature group, a total of 100 measurements were obtained from earlywood tracheids and 100 measurements from latewood tracheids, distributed across three specimens.

The expanded number of independent specimens was used to improve the robustness of the SEM-based interpretation and to reduce the risk that the observed transition would be based only on local anatomical variability. Although the number of cellular-level measurements was high, the statistical interpretation was based on the temperature groups and independent specimen replication. The cellular-level data were used to characterize within-group variability and to identify consistent microstructural trends.

The measured double cell wall thickness was used as a quantitative indicator of thermal degradation. In this study, reduction in double cell wall thickness was not interpreted as a direct mechanical measurement and was not used alone to define char formation. Instead, it was evaluated together with qualitative SEM observations, color and reflectance changes, and thermal analysis results.

2.6. Color and Spectrophotometric Analysis

Color and reflectance measurements were performed on the top surfaces of the reference specimens and thermally exposed spruce wood specimens. The measurements were carried out after conditioning of the specimens to $12 \pm 1\%$ equilibrium moisture content. The analyzed temperature range for color and spectrophotometric evaluation included the reference specimens and specimens exposed at 240, 250, 260, 270, 280, and 290 °C.

Measurements were performed using a Minolta CM-2600d spectrophotometer (Konica Minolta Sensing, Osaka, Japan) equipped with a xenon discharge lamp, D65 standard illuminant, and an 8 mm measuring aperture. The instrument was operated in specular component-included mode, and data acquisition was supported by SpectraMagic software (version 1.22).

Reflectance spectra were recorded across the visible wavelength range of 360–740 nm at a spectral resolution of 10 nm. Reflectance was determined as the ratio of the light intensity reflected from the wood surface to that from a white calibration standard, consisting of a high-purity aluminum oxide (Al_2O_3) ceramic tile, which served as the nominal 100% reflectance reference. Differential reflectance spectra were subsequently calculated by subtracting the spectra of the thermally treated specimens from those of the untreated reference specimens.

To ensure statistical rigor, the reflectance measurements were evaluated at a 95% confidence level for each individual wavelength.

Color changes were evaluated using the CIE $L^*a^*b^*$ color space. The measured coordinates— L^* (lightness), a^* (green–red chromaticity), and b^* (blue–yellow chromaticity)—were utilized to characterize surface darkening and chromatic shifts induced by the thermal exposure. Colorimetric measurements were performed exclusively on the radial surfaces of the specimens. For each specimen, three randomized measurements were taken per individual radial surface, resulting in a total of six independent measurements per single specimen.

Total color differences (ΔE^*_{ab}) relative to the untreated reference specimens were calculated using the standard Euclidean distance formula.

Color and reflectance measurements were used as complementary indicators of surface modification associated with thermal degradation. They were not used as independent criteria for defining the onset of char formation. In the interpretation of the results, optical changes were evaluated together with SEM observations and thermal analysis to determine whether they supported the same transition interval.

2.7. Simultaneous Thermal Analysis

The thermal stability and decomposition behavior of untreated spruce wood were evaluated using simultaneous thermal analysis, integrating thermogravimetry, derivative thermogravimetry, and differential scanning calorimetry. The analyzed material was untreated spruce wood from the same material batch as the furnace specimens. The STA samples were not taken from thermally pretreated cubes.

Measurements were carried out using a NETZSCH STA 509 series thermal analyzer (NETZSCH-Gerätebau GmbH, Selb, Germany) operated with Proteus software, version 9.4. Approximately 10 ± 0.5 mg of each sample was placed into an open Al_2O_3 crucible and heated from ambient temperature to 600°C at a constant heating rate of $10^\circ\text{C}\cdot\text{min}^{-1}$.

The measurements were performed under an oxidizing O_2/N_2 atmosphere with an additional protective N_2 flow. The gas flow rates were $39\text{ mL}\cdot\text{min}^{-1}$ for O_2 and $10.5\text{ mL}\cdot\text{min}^{-1}$ for N_2 , with an additional protective N_2 flow of $20\text{ mL}\cdot\text{min}^{-1}$. Calibration was performed using indium and alumina standards to ensure measurement accuracy.

The TG signal recorded the sample mass as a function of temperature, while the DTG curve, calculated as the first derivative of the TG signal, was used to identify the temperature of maximum decomposition rate and the onset of intensified mass loss. The DSC signal was recorded simultaneously and used to evaluate endothermic and exothermic processes associated with moisture release, decomposition, and oxidation-related reactions during thermal degradation. Exothermic transitions were evaluated according to the conventional instrument orientation.

The characteristic DTG onset temperature was defined as the temperature corresponding to a mass loss rate of $1\%\cdot\text{min}^{-1}$ [34]. The onset temperature was determined from exported DTG data using numerical procedures implemented in the analysis software and further processed in Statistica 14 (TIBCO Software Inc., Palo Alto, CA, USA).

The DTG onset temperature was interpreted as an indicator of intensified thermal degradation. It supports the identification of the temperature interval in which degradation processes accelerate, but it does not by itself prove the formation of a spatially defined char layer. Therefore, STA results are used in this study as supporting thermal evidence and not as an independent physicochemical boundary of char formation.

Simultaneous thermal analysis measurements were performed in repeated experiments ($n = 5$) to ensure reproducibility. This replicate number refers only to STA measurements and is independent of the number of furnace-exposed specimens used for SEM evaluation.

2.8. Statistical and Analytical Methods

Statistical analysis was performed to evaluate the effect of thermal exposure temperature on the measured double cell wall thickness of earlywood and latewood tracheids. The data are presented as the mean $\pm$ standard deviation. The coefficient of variation was calculated to assess variability within each temperature group.

For SEM-based cell wall measurements, three independent specimens were included for each main temperature group. Cellular-level measurements were first summarized at the specimen level in order to reduce the risk of pseudoreplication. The specimen-level means were then used for statistical comparison among temperature groups. This approach was selected because multiple cellular measurements obtained from the same specimen are not fully independent biological replicates.

One-way analysis of variance was applied separately to earlywood and latewood tracheid measurements to assess differences among temperature levels. When a statistically significant temperature effect was identified, Tukey's honestly significant difference post hoc test was used for pairwise comparisons between groups. Statistical significance was evaluated at $\alpha = 0.05$.

In addition to statistical significance, the magnitude of change between temperature levels was evaluated using percentage reduction in double cell wall thickness relative to the reference specimens. Particular attention was paid to the interval between 250 and 260 °C, where the most pronounced microstructural transition was expected based on preliminary observations.

Color and reflectance data were evaluated descriptively and comparatively across temperature groups. Changes in L^* , a^* , b^* , ΔE^*ab , and reflectance spectra were used to identify trends in surface darkening and optical modification. These data were interpreted as supporting evidence and not as independent proof of char formation.

STA data were evaluated using Proteus software and exported for additional processing in Statistica 14. The characteristic temperature corresponding to a DTG mass loss rate of $1\% \cdot \text{min}^{-1}$ was determined for each replicate. Repeated-measures analysis of variance was used to evaluate the effect of temperature on DTG values, with temperature treated as a within-subject factor.

The overall interpretation of the transition interval was based on the convergence of several indicators: SEM-observed anatomical degradation, quantitative reduction in tracheid double cell wall thickness, color and reflectance changes, and DTG onset of intensified thermal degradation. SEM observations and cell wall measurements were treated as the primary evidence, while color, reflectance, and STA results were treated as complementary supporting evidence.

3. Results and Discussion

3.1. Overview of Observed Thermal Changes

Thermal exposure of Norway spruce wood resulted in progressive changes in both microscopic structure and surface optical response. The specimens exposed at lower temperatures showed early signs of thermal modification, whereas specimens exposed at higher temperatures exhibited increasingly pronounced microstructural deterioration. The most evident changes were observed in the tracheid cell walls, bordered pits, torus structures, and the overall integrity of earlywood and latewood tissues.

The results are presented in the following order: first, SEM observations are used to describe the qualitative development of microstructural changes; second, quantitative measurements of tracheid double cell wall thickness are presented; third, optical surface changes are reported using CIE $L^*a^*b^*$ and reflectance data; and finally, TG/DTG/DSC results are presented as thermal evidence of degradation behavior. The interpretation of these results in relation to the transition from thermal degradation to initial char formation is provided separately in Section 4.

3.2. SEM Observations of Microstructural Changes

SEM observations revealed temperature-dependent changes in the anatomical structure of spruce wood. The reference specimens showed preserved tracheid walls, clearly identifiable bordered pits, and intact torus and margo structures. Earlywood and latewood regions were distinguishable by their typical anatomical characteristics, with thinner-walled earlywood tracheids and thicker-walled latewood tracheids.

Thermally loaded Norway spruce wood (*Picea abies* (L.) Karst.) at 240 °C is shown in Figure 3. The ray parenchyma at the surface of the sample is presented in Figure 3a. Parenchyma cell walls are preserved. The cell wall structures are fragile and slightly degraded. Figure 3b shows the ray parenchyma and latewood tracheids (LTs) located in the center of the sample. Parenchyma cell walls remain preserved, and fibrillation of the secondary cell walls of tracheids is observed. Figure 3c illustrates fibrillation and thermal wrinkling of the secondary cell walls of tracheids. Figure 3d shows earlywood tracheids (ET) with tori present in bordered pits without obvious degradation. Fibrillation and thermal wrinkling

of the secondary cell wall of ET are observed. The image was acquired using a BSE detector. Figure 3e presents fragile cell walls of ET. Thermal wrinkling and microcrack formation occur. Figure 3f provides a detailed view of bordered pits in ET, where the torus is present without apparent degradation. Thermal wrinkling of the secondary cell wall and microcrack formation indicate weakening of the S2 layer, and small spherical droplets (arrow) are present. Lignin migration through the secondary cell wall was further confirmed by SEM micrographs, which revealed the formation of spherical and droplet-like structures on the wood cell walls. These features are attributed to the thermal softening, migration, and subsequent condensation of lignin at elevated temperatures, consistent with previously reported observations of lignin droplet formation associated with lignin softening [35–38].

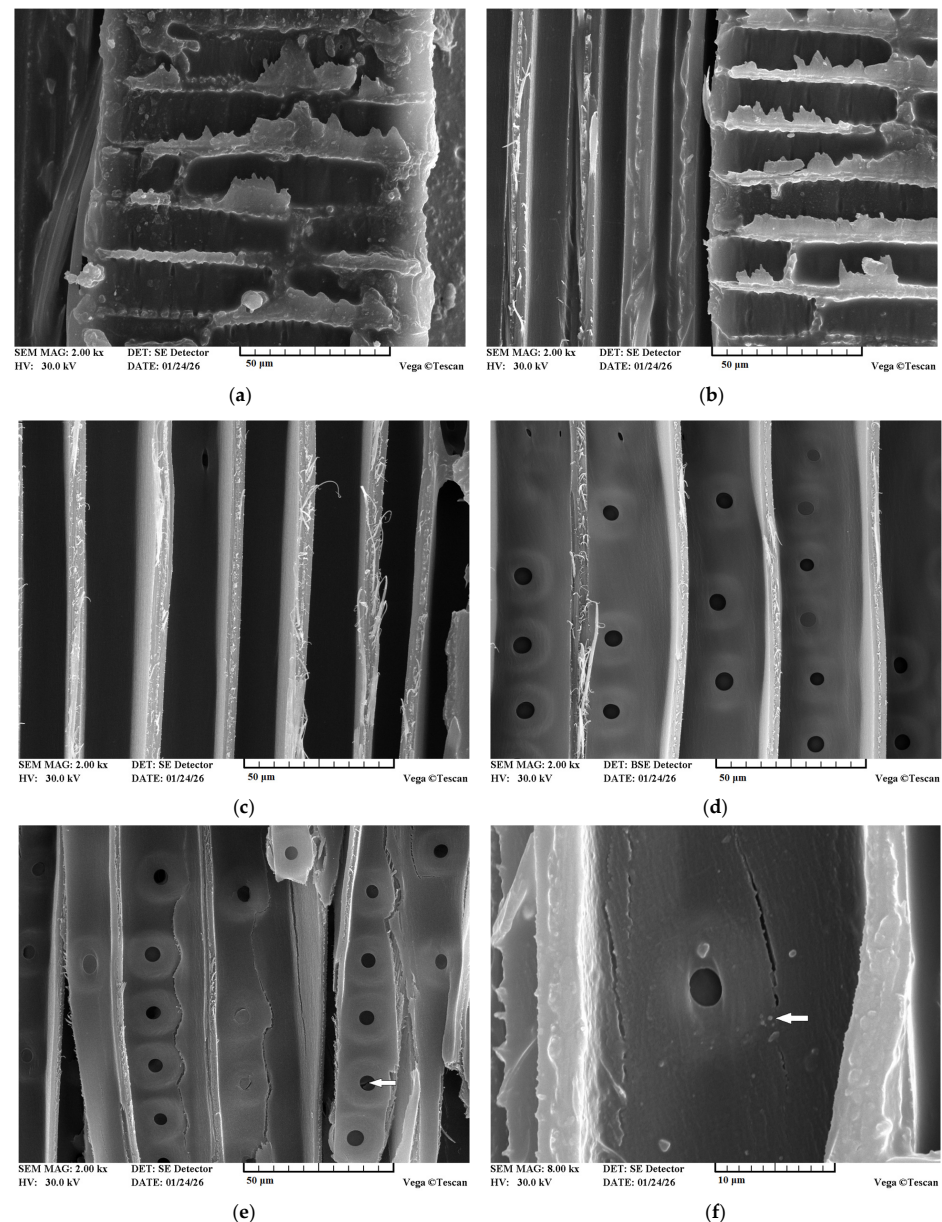


Figure 3. SEM micrographs of Norway spruce wood exposed at 240 °C for 15 min. (a) Ray parenchyma near the specimen surface with preserved cell wall structure. (b) Ray parenchyma and latewood tracheids in the central region of the specimen. (c) Fibrillation and thermal wrinkling of secondary tracheid walls. (d) Earlywood tracheids with identifiable bordered pits and preserved torus structures. (e) Fragile earlywood tracheid walls with localized microcracking, with localized disruption of the torus in bordered pits (arrow). (f) Detail of bordered pits in earlywood tracheids. Abbreviations: ET, earlywood tracheid; LT, latewood tracheid.

The structure of thermally loaded Norway spruce wood (*Picea abies*) at 250 °C is illustrated in Figure 4. Figure 4a shows a radial intercellular canal beneath the sample surface without apparent degradation of cellular structures. Figure 4b presents a section through the ray parenchyma of the thermally loaded sample. Piceoid cross-field pit membranes are preserved without obvious degradation. Figure 4c shows the center of the sample, where cell wall structures are fragile and brittle. Tori of earlywood tracheids (ETs) are present without apparent degradation. Slight thermal wrinkling of the secondary cell wall is observed without microcrack formation. Figure 4d shows that tori of bordered pits in ET are locally absent in the surface layers. Thermal wrinkling of the secondary cell wall and microcrack formation occur, and the cell walls are fragile. Small spherical droplets are present.

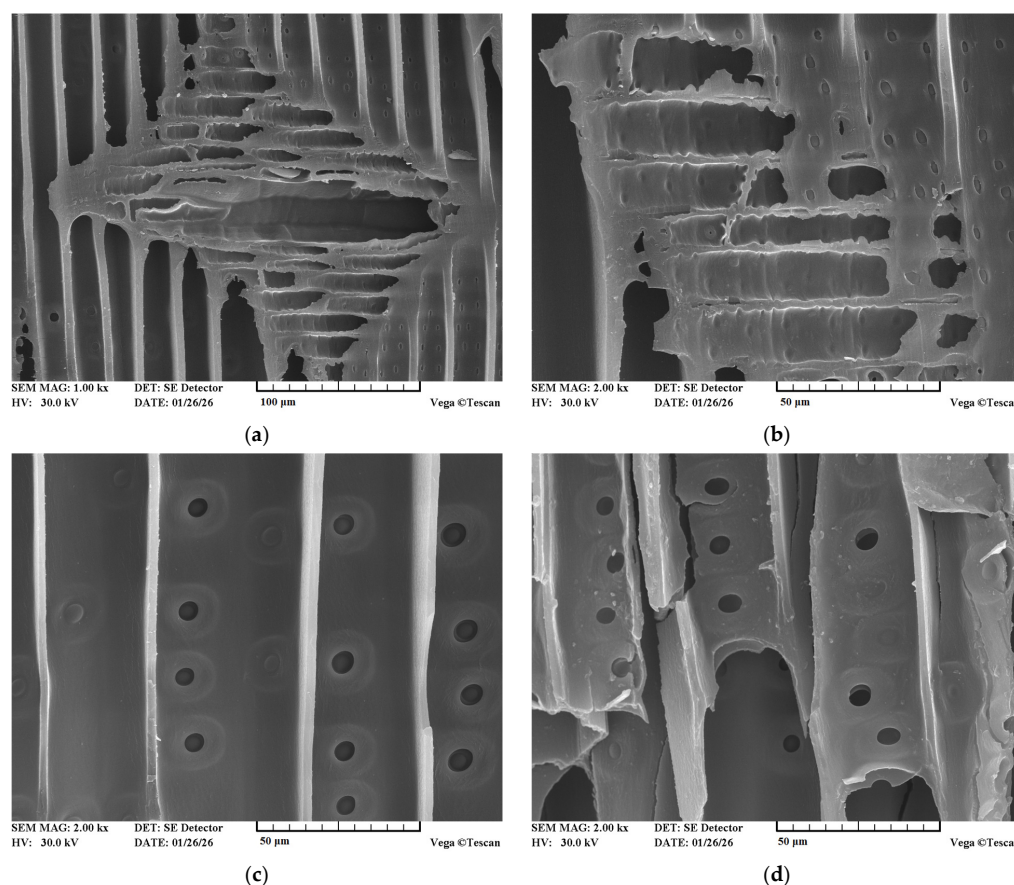


Figure 4. SEM micrographs of Norway spruce wood exposed at 250 °C for 15 min. (a) Radial intercellular canal with surrounding tracheid structure. (b) Ray parenchyma region with preserved piceoid cross-field pits. (c) The central region of the specimen showing fragile cell wall structures and identifiable bordered pits. (d) Earlywood tracheids in the near-surface region with localized alteration or absence of torus structures. Abbreviations: ET, earlywood tracheid; LT, latewood tracheid.

Thermally loaded Norway spruce wood at 260 °C is shown in Figure 5. Figure 5a illustrates a radial section of the thermally degraded sample. In the transverse plane, where the characteristic morphology of gradual tracheid decomposition is evident, with more resistant structures represented by a narrow band of latewood tracheids (LTs). Figure 5b depicts a radial intercellular canal, while the surrounding tracheids exhibit brittle fractures and pronounced cell wall fragility; tori of bordered pits are disrupted. Figure 5c shows that piceoid cross-field pits remain largely preserved, except for degraded tori in earlywood tracheids (ETs) and ray tracheids. Figure 5d presents ETs with complete degradation of the tori or localized cracking; the tracheid cell walls are characterized by thermal wrinkling and

the presence of small spherical droplets. Figure 5e shows the center of the sample, where cell wall structures are fragile and brittle, while the tori of ETs remain without apparent degradation; thermal wrinkling of the secondary cell wall and microcrack formation are observed. Figure 5f provides a detailed view of thermal wrinkling of the secondary cell wall of tracheids, with small spherical droplets present as a result of thermal degradation; the tracheid walls appear smooth and compact in section, with no visible middle lamella.

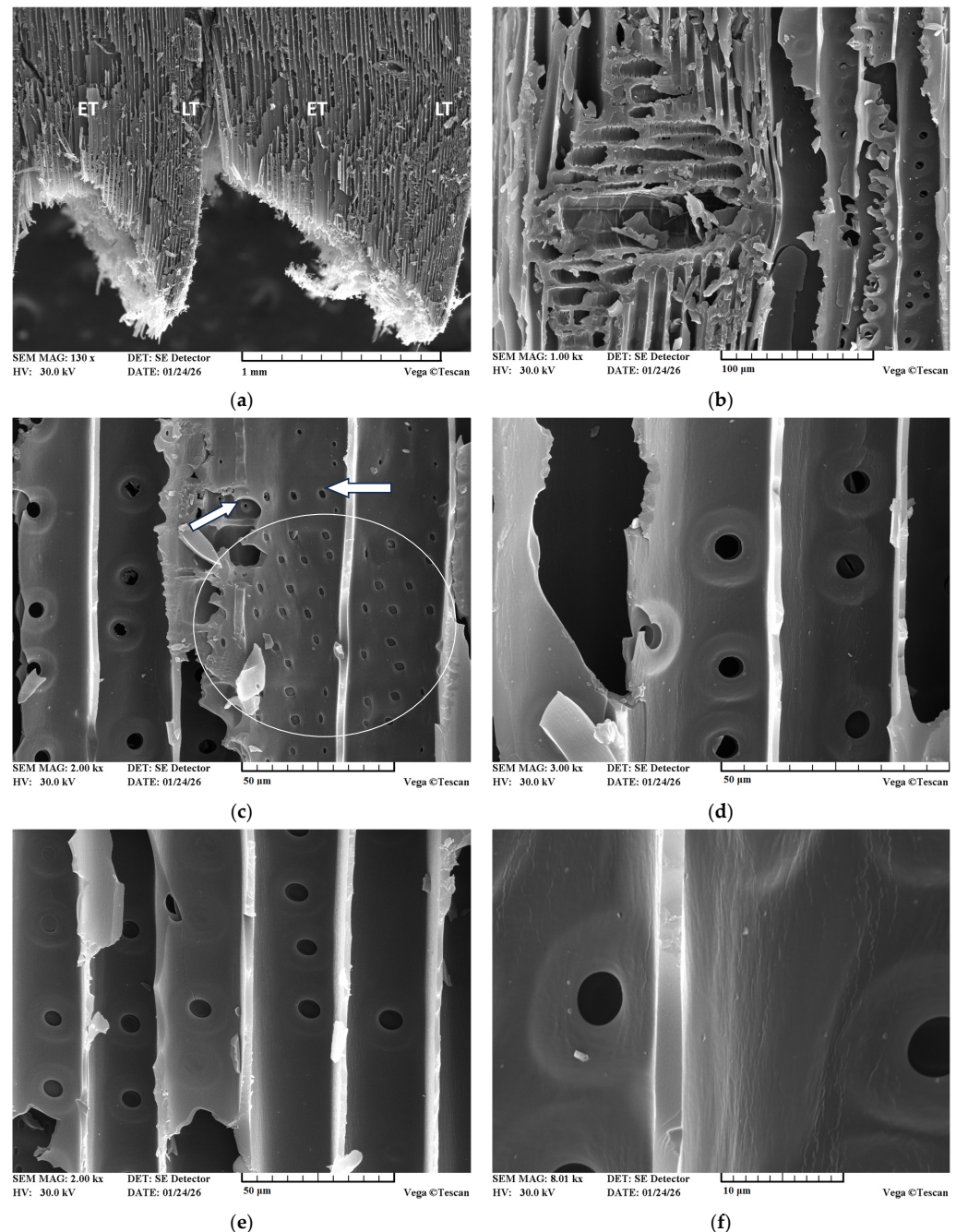


Figure 5. SEM micrographs of Norway spruce wood exposed at 260 °C for 15 min. (a) Transverse view showing earlywood and latewood regions and the development of degraded tissue structure. (b) Radial intercellular canal with brittle fractures in surrounding tracheids. (c) Piceoid cross-field pits (circle) and altered torus structures in ray tracheids (arrows). (d) Earlywood tracheids with degraded or cracked tori. (e) The central region of the specimen showing brittle cell wall structures. (f) Detail of secondary wall wrinkling and surface deposits. Abbreviations: ET, earlywood tracheid; LT, latewood tracheid.

The comparison of bordered pits in reference and thermally degraded Norway spruce wood (*Picea abies* (L.) Karst.) is presented in Figure 6. Figure 6a shows bordered pits in thermally degraded wood at 260 °C, where the margo of bordered pits appears sealed and the torus is degraded. Figure 6b illustrates a bordered pit in an earlywood tracheid (ET) in reference wood in the radial section; the torus appears compact and is anchored by a finely fibrous margo, and the pit surface is smooth.

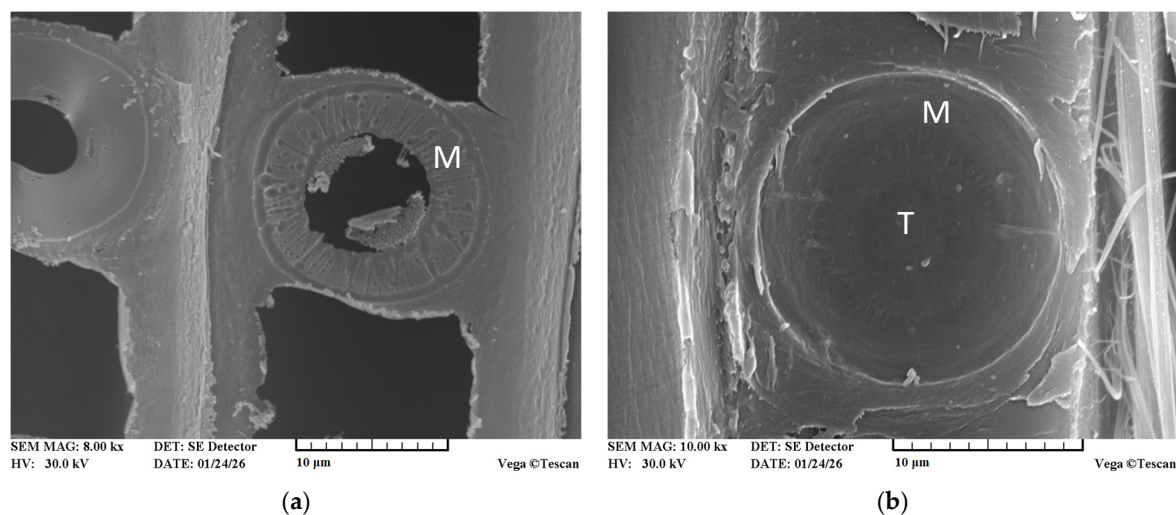


Figure 6. Comparison of bordered pits in reference and thermally exposed Norway spruce wood. (a) Bordered pits in wood exposed at 260 °C, showing altered pit structure and torus degradation. (b) Bordered pit of an earlywood tracheid in the reference specimen, showing a compact torus and fibrous margo. Abbreviations: T, torus; M, margo.

At 270 °C, the microstructural changes observed at 260 °C became more extensive. The tracheid walls showed more frequent cracking, increased brittleness, and more pronounced local collapse. Bordered pit structures were less frequently preserved, and earlywood tracheids showed more advanced deformation than latewood tracheids. The tissue structure remained recognizable in selected areas, but the degree of degradation was higher than in specimens exposed at 240–260 °C.

SEM observations indicated that the transition from progressive thermal degradation to the early development of charred structure occurred within the range of approximately 250–260 °C under the applied laboratory exposure regime. Similar but more advanced degradation features were subsequently observed at 270 °C, supporting the interpretation of this interval as a microstructural transition associated with the onset of char formation.

For this reason, quantitative SEM evaluation was concentrated on the reference condition and on specimens exposed to temperatures up to 270 °C. Higher temperature levels were not included in the main quantitative assessment because the principal transition interval had already been identified within the lower temperature range. Observations at temperatures above 270 °C served only to confirm the continued progression of thermal degradation toward increasingly developed charred structures.

3.3. Quantitative Changes in Tracheid Double Cell Wall Thickness

Quantitative SEM measurements showed a progressive decrease in double cell wall thickness with increasing exposure temperature. The decrease was observed in both earlywood and latewood tracheids. The reference specimens showed the highest mean double cell wall thickness values, while the thermally exposed specimens showed a gradual reduction with increasing temperature.

The recalculated specimen-level data obtained from three independent specimens per main temperature group are summarized in Table 1.

Table 1. Double cell wall thickness of earlywood (ETs) and latewood tracheids (LTs) in reference and thermally exposed Norway spruce specimens.

Tracheid Type	Temperature Group	n Specimens	n Cellular Measurements	Mean CWT (μm)	SD (μm)	CV (%)	Min–Max (μm)	Decrease Relative to Reference (%)
ET	Reference	3	100	3.78	0.49	12.89	2.7–5.0	—
ET	240 °C	3	100	2.88	0.40	13.92	1.8–3.7	−23.91
ET	250 °C	3	100	2.45	0.45	18.23	1.6–3.9	−35.28
ET	260 °C	3	100	1.85	0.38	20.38	1.1–2.9	−50.96
ET	270 °C	3	100	1.20	0.33	23.00	0.9–2.4	−68.25
LT	Reference	3	100	7.48	0.98	13.12	5.5–9.2	—
LT	240 °C	3	100	6.38	0.86	13.51	3.8–8.6	−14.66
LT	250 °C	3	100	5.10	0.41	8.04	3.6–7.3	−31.77
LT	260 °C	3	100	3.42	0.49	14.32	2.2–4.9	−54.25
LT	270 °C	3	100	1.50	0.35	20.00	1.1–2.8	−79.95

Notes: Values represent mean thickness (μm), standard deviation (SD), coefficient of variation (CV), and observed range (min–max) based on 100 measurements per group. The percentage decrease (%) expresses the reduction in mean thickness relative to the untreated reference samples.

The decrease in double cell wall thickness was gradual between the reference condition and 250 °C. A more pronounced reduction was observed between 250 and 260 °C in both earlywood and latewood tracheids (Figure 7). The 270 °C group showed further reduction or stabilization of highly degraded wall structures, depending on the anatomical region and local preservation of measurable cell wall boundaries.

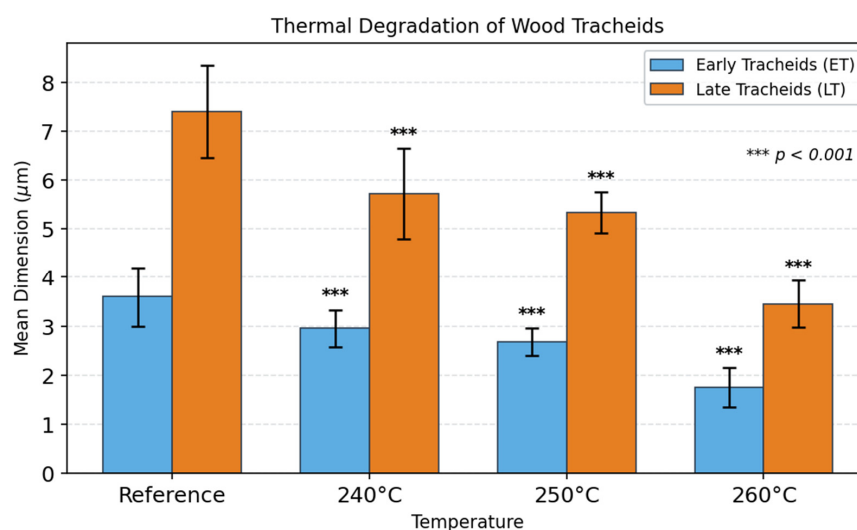


Figure 7. Effect of thermal exposure on measured double cell wall thickness of earlywood and latewood tracheids. Bars represent mean specimen-level values, and error bars represent standard deviation. Different letters indicate statistically significant differences among temperature groups according to Tukey’s HSD test at $\alpha = 0.05$. Abbreviations: ET, earlywood tracheid; LT, latewood tracheid. Note: *** Indicates a statistically significant difference compared to the reference group at the $p < 0.001$ level.

Earlywood tracheids showed greater local variability at higher temperatures, which was reflected in increased dispersion of measured values. Latewood tracheids retained higher absolute wall thickness values than earlywood tracheids, but the relative reduction with increasing temperature followed the same general trend.

One-way ANOVA applied to specimen-level means indicated a statistically significant effect of temperature on double cell wall thickness in both earlywood and latewood tracheids. Tukey’s HSD post hoc test showed significant differences between the reference

group and thermally exposed groups, with the largest pairwise change occurring between 250 and 260 °C. The 270 °C group remained significantly different from the reference and lower-temperature groups.

3.4. Color Changes and Spectrophotometric Results

Thermal exposure caused progressive darkening of the spruce wood surface. The reference specimens showed the highest lightness values and the typical pale yellowish color of spruce wood. Specimens exposed at 240 °C showed a visible decrease in lightness and chromaticity. Further exposure at higher temperatures resulted in darker and more achromatic surface appearance.

The CIE *Lab** values and color differences relative to the reference specimens are summarized in Figure 8. The strongest decrease in lightness occurred between the reference condition and the lower thermal exposure levels. Between 250 and 260 °C, a further reduction in chromatic coordinates was observed, indicating a shift toward darker and less saturated tones. At temperatures above 260 °C, the differences among color patches became less visually distinct.

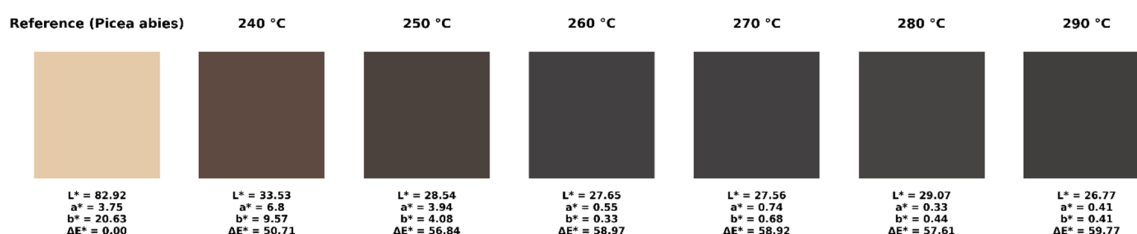


Figure 8. Reconstructed color patches of Norway spruce wood based on measured CIE *Lab** values after thermal exposure. The figure shows the visual progression from reference wood to specimens exposed at 240, 250, 260, 270, 280, and 290 °C.

Reflectance spectra showed a systematic decrease in reflected light after thermal exposure. The reference specimens showed the highest reflectance values across the visible range. Thermally exposed specimens showed lower reflectance, with the most evident differences observed between the reference specimens and the thermally exposed groups. The spectra of specimens exposed at 260–290 °C showed reduced differentiation compared with the difference between the reference and lower-temperature specimens (Figure 9).

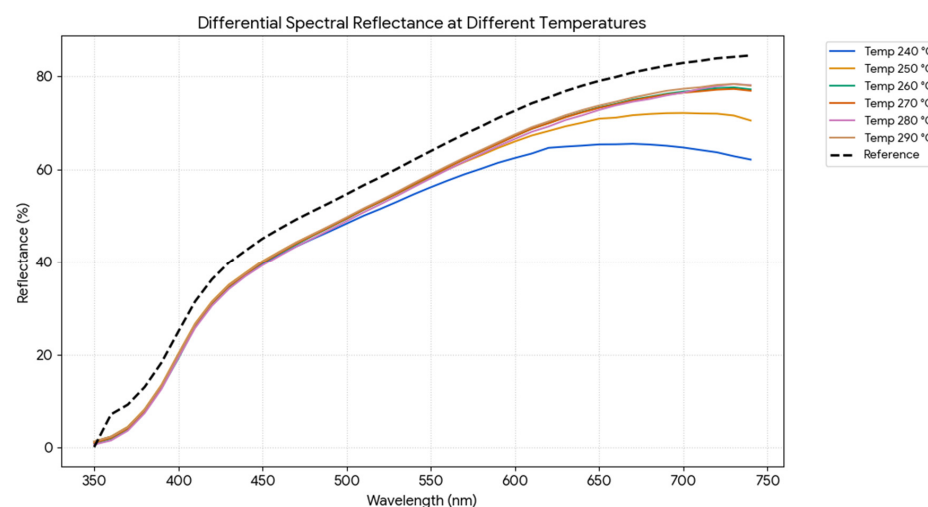


Figure 9. The spectral reflectance profiles of Norway spruce wood across the visible wavelength range after thermal exposure at selected temperatures. The profiles show reduced reflectance after thermal exposure and reduced differentiation among specimens exposed at 260–290 °C.

3.5. Simultaneous Thermal Analysis

Thermogravimetric analysis (TGA) is widely used to evaluate mass changes of a material as a function of temperature [39]. The TG and DSC curves obtained under an oxidizing O₂/N₂ atmosphere with an additional protective N₂ flow are presented in Figure 10. The TGA of spruce samples in air at a heating rate of 10 °C·min^{−1} (Figure 11) revealed multi-step decomposition, which is typical of lignocellulosic materials and consistent with previous studies [39,40].

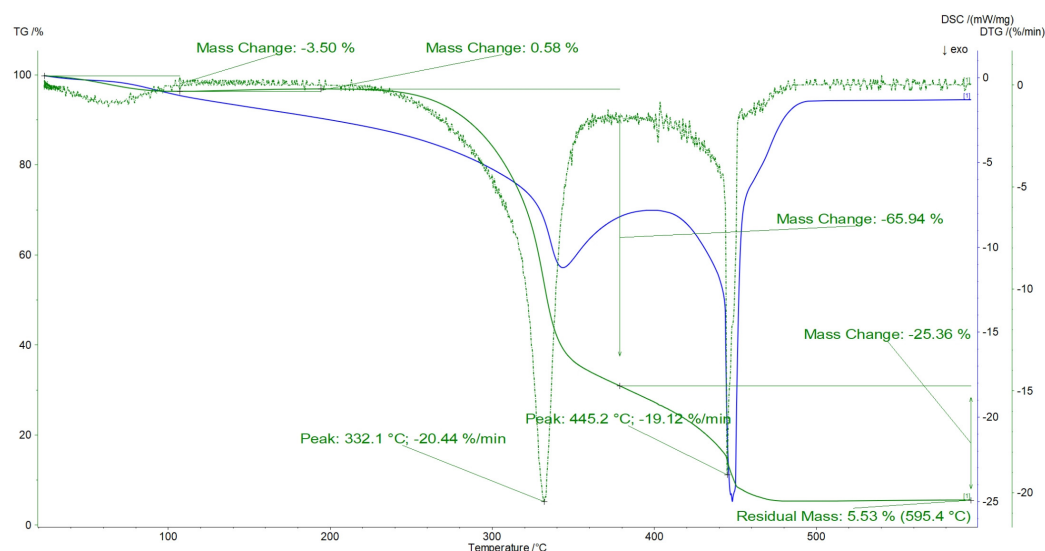


Figure 10. TG and DSC curves of untreated Norway spruce wood measured under oxidizing O₂/N₂ atmosphere with an additional protective N₂ flow. The TG curve (green line) shows mass loss as a function of temperature, while the DSC curve (blue line) shows thermal effects associated with moisture release, decomposition, and oxidation-related processes.

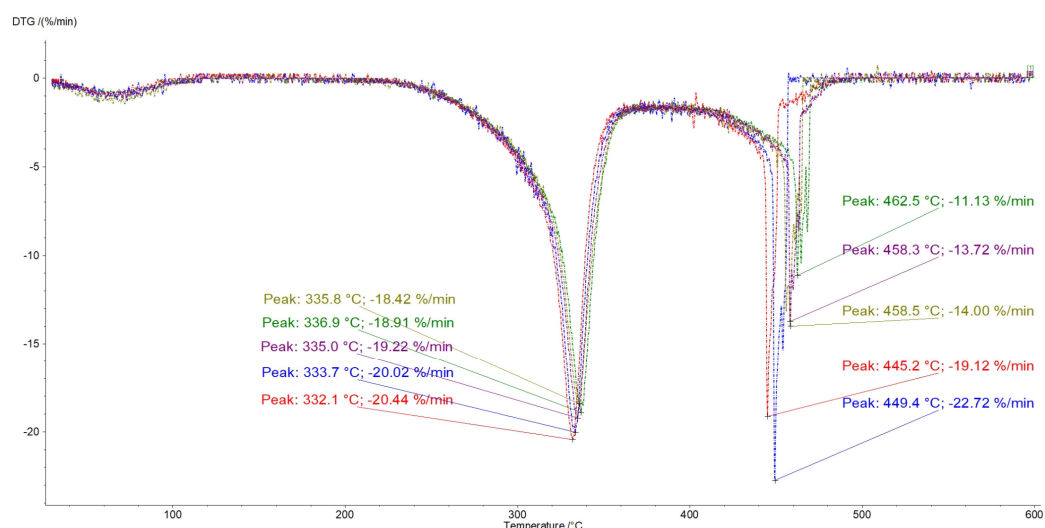


Figure 11. DTG curves of five untreated Norway spruce replicates measured over the temperature range of 30–600 °C. The curves show the onset of intensified degradation and two main degradation stages.

The thermal behavior of spruce sample 1 was evaluated using TG/DTG and DSC analyses. The thermal decomposition of sample 1 was monitored in air over the temperature range of 30 °C to 600 °C at a heating rate of 10 °C·min^{−1}. Three distinct stages of mass loss were identified, as shown in Figure 10.

The first peak, occurring up to approximately 109 °C, corresponds primarily to the removal of free moisture [41,42]. An endothermic effect associated with the evaporation of physically bound water and low-molecular-weight volatile compounds [40] was observed up to approximately 194 °C. This is consistent with the minor mass loss recorded on the TG curve within this temperature region. For sample 1, this stage corresponds to a mass loss of 0.58%.

The exothermic decomposition began at approximately 200 °C and continued up to 595 °C. The maximum decomposition temperature, defined as the temperature corresponding to the maximum rate of mass loss [43] was determined to be 332.1 °C. At this point, the maximum mass loss rate reached $-20.44\% \cdot \text{min}^{-1}$, which is mainly attributed to the decomposition of hemicellulose and cellulose [44,45]. A second peak was observed at 445.2 °C, with a maximum mass loss rate of $-19.12\% \cdot \text{min}^{-1}$.

TG curves were used to determine the onset temperature, defined as the temperature corresponding to $1\% \cdot \text{min}^{-1}$ mass loss [34]. Since thermal decomposition reactions of lignocellulosic materials, including devolatilization and carbonization processes, occur at very low degrees of conversion, this threshold can be regarded as a practical operational indicator of intensified thermal degradation associated with onset charring. This definition is consistent with recommendations for kinetic analysis at low conversion levels [46] and with studies describing the gradual and overlapping nature of biomass decomposition reactions.

Thermogravimetric (TG) curves were processed by calculating the first derivative, yielding DTG curves (Figure 10). The resulting DTG data were exported and subsequently analyzed using Statistica 14 (TIBCO Software Inc., Palo Alto, CA, USA).

The onset of thermal degradation was defined as the temperature corresponding to a mass loss of 1% [34]. As shown in Table 2, the characteristic DTG parameters of untreated Norway spruce wood were determined from five independent STA replicates.

Table 2. Characteristic DTG parameters of untreated Norway spruce wood determined from five STA replicates.

Parameter	Mean	SD	CV (%)
DTG onset at $1\% \cdot \text{min}^{-1}$ (°C)	254.2	1.48	0.58
Tmax ₁ (°C)	334.7	1.86	0.56
DTGmax ₁ ($\% \cdot \text{min}^{-1}$)	−19.40	0.78	4.02
Tmax ₂ (°C)	454.8	7.1	1.56
DTGmax ₂ ($\% \cdot \text{min}^{-1}$)	−16.14	4.46	27.63

Notes: DTG onset was defined as the temperature corresponding to a mass loss rate of $1\% \cdot \text{min}^{-1}$. Tmax₁ and Tmax₂ represent the temperatures of the first and second maximum degradation rates, respectively.

Repeated-measures ANOVA confirmed a statistically significant effect of temperature on DTG values. The DTG onset temperature occurred within the same general temperature range in which SEM observations and double cell wall measurements indicated pronounced microstructural deterioration. The interpretation of the relationship among these indicators is provided in Section 4.

3.6. Summary of Main Results

The main results are summarized in Table 3. SEM observations showed progressive microstructural degradation with increasing exposure temperature. Quantitative measurements confirmed a reduction in tracheid double cell wall thickness in both earlywood and latewood. Color and reflectance measurements showed surface darkening and reduced spectral differentiation at higher exposure temperatures. STA measurements identified the onset of intensified degradation at 254.2 ± 1.48 °C.

Table 3. A summary of the main experimental results obtained from SEM, cell wall measurements, optical analysis, and STA.

Method	Main Observed Result	Temperature Range or Value	Role in Results
SEM observation	Progressive cell wall thinning, pit degradation, brittleness, and localized collapse	Most pronounced between 250 and 260 °C	Primary morphological result
Double cell wall measurements	Progressive reduction in ET and LT wall thickness	Reference to 270 °C	Primary quantitative result
Color analysis	Decrease in L*, a*, and b* values	240–290 °C	Supporting optical result
Reflectance analysis	Reduced reflectance and reduced differentiation among spectra	260–290 °C	Supporting optical result
STA: DTG onset	Onset of intensified degradation	254.2 ± 1.48 °C	Supporting thermal result
STA: main DTG peak	Primary degradation stage	334.7 ± 1.9 °C	Thermal degradation result
STA: secondary DTG peak	Secondary degradation stage	454.8 ± 7.1 °C	Thermal degradation result

4. Discussion

4.1. Microstructural Meaning of 250–260 °C Transition Interval

The results of this study show that the most pronounced microstructural change in Norway spruce wood occurred between 250 and 260 °C under the applied laboratory exposure regime. SEM observations revealed a progressive sequence of anatomical degradation, beginning with slight wall fragility and secondary wall wrinkling at 240 °C, followed by increased brittleness and localized pit alteration at 250 °C. At 260 °C, these changes became more distinct and included substantial tracheid cell wall thinning, degradation or the absence of torus structures in bordered pits, localized cracking, and partial structural collapse.

These observations indicate that the transition from early thermal degradation to a more advanced degraded and initially charred structure is gradual rather than instantaneous. The 250–260 °C interval should therefore not be interpreted as a sharply defined charring temperature. Instead, it represents a condition-dependent microscopic transition interval in which several anatomical features become pronounced and consistent under the applied exposure conditions.

The quantitative measurements of double cell wall thickness supported the SEM observations. Both earlywood and latewood tracheids exhibited a progressive reduction in wall thickness with increasing temperature, indicating a continuous degradation process rather than an abrupt structural change. However, the most pronounced reduction occurred between 250 and 260 °C. The mean double cell wall thickness of earlywood tracheids decreased from 2.45 to 1.85 µm, whereas latewood tracheids decreased from 5.10 to 3.42 µm. These changes coincided with a substantial increase in the severity of anatomical damage observed by SEM, including cell wall thinning, pit deterioration, cracking, and localized structural collapse. Therefore, the interval between 250 and 260 °C is interpreted as a region of accelerated degradation rather than a discrete thermal threshold. Earlywood tracheids showed greater local variability at higher temperatures, which is consistent with their thinner cell walls and higher susceptibility to thermal degradation. Although latewood tracheids retained greater absolute wall thickness values, they followed the same general degradation trend.

A similar relationship between increasing thermal exposure and cell wall degradation was reported by [47], who quantitatively monitored tracheid morphology during the charcoalification of spruce and pine wood. Their results showed progressive thinning and homogenization of tracheid cell walls with increasing temperature and identified cell wall thickness as one of the most sensitive indicators of thermal degradation. The authors

reported reductions in tracheid wall thickness of approximately 20% following charcoal formation, confirming that quantitative anatomical measurements provide valuable evidence for assessing the progression of thermal damage and structural transformation in wood.

In the present study, this transition was quantitatively demonstrated by measurements of double tracheid cell wall thickness and volumetric shrinkage. The mean double cell wall thickness of earlywood tracheids decreased from 3.78 μm in the reference specimens to 1.85 μm at 260 $^{\circ}\text{C}$ (−51.0%), while latewood tracheids decreased from 7.48 μm to 3.42 μm (−54.3%).

A limitation of the present study is the use of three independent specimens per temperature level. However, the quantitative anatomical evaluation was based on 100 tracheid measurements per treatment group, exceeding the minimum sample size required based on the observed variability and a measurement error of 5%. Nevertheless, natural variability associated with wood density, annual ring width, and anatomical heterogeneity cannot be completely excluded. Future studies involving a larger number of biological replicates would further strengthen the general applicability of the findings.

4.2. Relationship Between SEM Observations and Thermal Analysis

The STA results provided supporting thermal evidence for the temperature interval identified by SEM observations. The DTG onset temperature, defined as the temperature corresponding to a mass loss rate of $1\% \cdot \text{min}^{-1}$, was 254.2 ± 1.48 $^{\circ}\text{C}$. This value lies within the same general interval in which the SEM observations and double cell wall measurements showed pronounced microstructural deterioration.

Since thermal decomposition reactions of lignocellulosic materials begin at very low degrees of conversion, the DTG onset can be regarded as an indicator of the onset of intensified thermal degradation rather than as direct evidence of char layer formation. This interpretation is consistent with recommendations for kinetic analysis at low conversion levels [46] and with studies describing the gradual and overlapping nature of lignocellulosic biomass decomposition reactions [48]. However, TG/DTG analysis and SEM characterize different aspects of the degradation process. TG/DTG records mass loss and the rate of thermal decomposition under the conditions of thermal analysis, while SEM provides spatial and anatomical information on the wood structure after controlled furnace exposure. Therefore, the consistency between the DTG onset temperature and the temperature interval in which SEM revealed pronounced microstructural deterioration should be regarded as complementary evidence supporting the identification of the 250–260 $^{\circ}\text{C}$ transition interval, rather than as direct proof of the formation of a spatially defined char layer.

The main DTG degradation stage occurred at a higher temperature than the microscopic transition interval identified by SEM. This finding is expected because the maximum decomposition rate represents the stage of the most intensive mass loss, whereas the earliest irreversible anatomical changes become detectable before the maximum rate of thermal decomposition is reached. Consequently, SEM is particularly suitable for identifying the earliest observable anatomical manifestations of thermal degradation, while STA provides the broader thermal context of the degradation process.

4.3. Role and Limitation of Color and Reflectance Indicators

Color and reflectance measurements showed progressive optical modification of the wood surface with increasing exposure temperature. Thermal exposure resulted in darkening, reduction in lightness, and a shift toward more achromatic tones. Reflectance spectra also showed reduced differentiation among specimens exposed at 260–290 $^{\circ}\text{C}$, indicating that the optical response became less sensitive to further increases in exposure temperature.

These optical changes are consistent with progressive thermal degradation of wood. Although the black appearance of the wood surface is widely recognized as a macroscopic manifestation associated with charring, color change alone cannot be considered a definitive criterion for the onset of charring. Surface color is affected by several factors, including chemical changes in extractives and lignocellulosic polymers, oxidation, surface roughness, exposure duration, and local variation in heat transfer. Therefore, color and reflectance data should be interpreted only as supportive indicators of thermal degradation and evaluated together with complementary analytical technique.

In the present study, the value of color and reflectance analysis lies in its consistency with the results observed by SEM and STA. The optical response changed in the same temperature range in which the wood structure showed pronounced microscopic deterioration and STA indicated the onset of intensified thermal degradation, supporting the interpretation that accelerated thermal degradation occurs within the 250–260 °C interval. However, the optical response alone does not permit independent identification or localization of the char front.

4.4. Comparison with Engineering Char Line Criteria

The results of this study should be clearly distinguished from the engineering char line definitions used in structural fire design. In Eurocode-based design, the 300 °C isotherm is commonly used as a practical criterion for determining the char line and calculating the residual cross-section of timber members exposed to fire. This criterion is intended for standardized engineering calculations and does not necessarily represent the first microscopic appearance of thermally degraded or initially charred tissue.

The present study does not propose replacing the 300 °C engineering criterion. Instead, it shows that pronounced microstructural degradation can be observed below this value under the applied laboratory conditions. The 250–260 °C interval identified in this study should therefore be interpreted as a microscopic transition interval associated with accelerated thermal degradation rather than as a new structural design criterion.

This distinction is fundamental because engineering char line is a simplified boundary used for residual cross-section calculations, whereas the microscopic transition interval describes anatomical changes at the cellular level. Although these concepts are related, they represent different aspects of the charring process. A wood specimen may therefore exhibit pronounced microstructural degradation before reaching the temperature criterion used for engineering char line determination.

4.5. Relevance for Fire Engineering and Modeling

From a fire engineering perspective, the main contribution of this study is not the definition of a new charring temperature, but the identification of microscopic features that help describe the early development of charred structure. These indicators may be relevant for the interpretation of fire-exposed wood, forensic assessment of thermally damaged timber, and future validation of heat transfer and pyrolysis models.

In numerical models, the assumed degradation or charring boundary influences the predicted progression of thermal degradation. Although the present findings provide additional physical insight into this process, further experimental validation is required before the identified temperature interval can be considered for engineering applications or numerical model calibration.

4.6. Integrated Interpretation

The integrated results indicate that the transition from progressive thermal degradation to initial char formation in spruce wood is not represented by a single temperature.

Instead, it occurs over a temperature interval in which several physical, anatomical, optical, and thermal changes become increasingly pronounced.

SEM observations and quantitative double cell wall measurements showed the clearest evidence of microstructural transition between 250 and 260 °C. Color and reflectance measurements showed surface darkening and reduced spectral differentiation at higher temperatures. STA identified the onset of intensified degradation at 254.2 ± 1.48 °C.

Together, these findings support the interpretation that, under the applied laboratory exposure regime, the 250–260 °C interval represents a meaningful transition region between progressive thermal degradation and the early development of charred structure in Norway spruce wood. This interval should be understood as condition-dependent and complementary to existing engineering criteria, not as a universal charring temperature.

4.7. Novelty and Scientific Contribution

The present study contributes to the current understanding of the early stages of wood thermal degradation by integrating complementary analytical techniques within a single experimental framework. SEM observations, quantitative cell wall measurements, simultaneous thermal analysis (STA), and color and reflectance analyses provided independent microstructural, thermal, and optical evidence that was interpreted collectively to characterize the microscopic transition associated with the early development of charred structure under controlled laboratory conditions.

The identified temperature interval of 250–260 °C should therefore be regarded as a refinement of the current understanding of the transition between thermally degraded and initially charred wood under the applied laboratory conditions, rather than as a new charring temperature or a replacement for existing engineering concepts. The findings demonstrate that combining complementary analytical techniques provides additional experimental evidence for interpreting this microscopic transition beyond that obtained from any individual analytical method alone. This integrated approach contributes to a more comprehensive physical interpretation of the early stages of wood thermal degradation while remaining fully consistent with the engineering concepts used in structural fire design.

5. Conclusions

This study identified the microstructural features associated with the transition from progressive thermal degradation to the early development of charred structure in spruce wood under controlled laboratory conditions. Rather than defining a universal charring temperature, this study focused on identifying a condition-dependent microstructural transition using complementary analytical techniques.

The results consistently indicated that the most pronounced changes occurred within the 250–260 °C interval. SEM observations revealed substantial tracheid cell wall thinning, degradation of bordered pits, increased brittleness, and localized structural collapse. Quantitative measurements showed a reduction in double cell wall thickness exceeding 50% at 260 °C in both earlywood and latewood tracheids.

The microstructural observations were supported by independent analytical methods. The DTG onset temperature (254.2 ± 1.48 °C) indicated the onset of intensified thermal degradation, while color and reflectance analyses showed pronounced optical changes above approximately 260 °C. Color and reflectance measurements showed progressive surface darkening and reduced differentiation among reflectance spectra at higher exposure temperatures. The agreement among these methods strengthens the interpretation of the identified transition interval under the applied experimental conditions.

The principal contribution of this study lies in the combined evaluation of microstructural, thermal, and optical indicators within a single experimental framework. This integrated approach provides complementary experimental evidence that may support the interpretation and future refinement of heat transfer and pyrolysis models describing the early stages of wood charring.

The identified transition interval should be interpreted within the context of the applied laboratory conditions and is intended to complement, rather than replace, the conventional 300 °C engineering char line criterion used in structural fire design.

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