

Assessing the Fire and Thermal Behavior of Thermally Modified, Acetylated, and One-sided Surface Charred Beech and Birch Wood

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The fire and thermal behavior of European beech (*Fagus sylvatica* L.) and silver birch (*Betula pendula* L.) woods were comparatively evaluated after being modified by thermal treatment (200 °C), acetylation, and one-sided surface charring. A small-flame ignition test (EN ISO 11925-2), gross heat of combustion *via* bomb calorimetry (EN ISO 1716), and thermogravimetric analysis (TG/DTG) were used for assessment. Thermal modification resulted in moderate mass loss (~3% to 4%), while acetylation achieved high weight percentage gain (>20%) and the strongest reduction in equilibrium moisture content; surface charring had only a negligible effect on bulk moisture properties. While none of the applied modifications altered the Euroclass-related reaction-to-fire classification, statistically significant differences in early-stage flame spread were observed. Acetylated wood exhibited increased flame spread, whereas surface-charred specimens showed a pronounced reduction, particularly for beech. All modified materials displayed higher gross heat of combustion compared with reference wood, reflecting increased carbon-rich constituents; however, this increase did not directly correspond to improved fire performance. TGA revealed similar degradation behavior for reference and thermally modified wood, whereas acetylated and surface-charred materials exhibited fundamentally different thermal responses because of chemical substitution and polysaccharide degradation with carbon-rich char formation, respectively. The study highlighted surface charring as an effective modification for reducing flame spread and demonstrates the necessity of combining complementary fire-testing methods to capture modification-specific fire behavior.

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INTRODUCTION

Wood is widely valued in construction for its renewability, low embodied energy, and carbon storage potential, which aligns well with sustainable development goals (Schlamadinger and Marland 1996; Gustavsson *et al.* 2006). Its high strength-to-weight ratio and aesthetic appeal have also contributed to its resurgence in modern architecture. However, despite these unquestionably positive benefits, its vulnerability to moisture, fungal decay, and fire limits its broader application in safety-critical or long-life structures

(White and Dietenberger 2010; Rowell 2013). Traditional preservative treatments can improve performance, but often they involve toxic compounds (Hill 2006). To address these limitations in an environmentally responsible way, wood modification technologies have been developed to improve dimensional stability, durability and, potentially, fire resistance (Esteves and Pereira 2009; Mantanis 2017; Sandberg *et al.* 2017; Jones and Sandberg 2020; Hill *et al.* 2021). According to Sandberg *et al.* (2017), wood modification includes chemical, thermo-hydro-mechanical, physical, and biological processes that alter the structure or composition of wood cell walls. Common techniques include acetylation, furfurylation, silane treatment, polymer impregnation, thermal modification, surface charring, *etc.* (Hill 2006; Sandberg *et al.* 2017). These modifications are typically non-toxic and result in wood products with improved service life and reduced maintenance needs. Among these, thermal modification, acetylation, and one-sided surface charring have attracted particular attention not only for enhancing dimensional stability, surface wettability, and decay resistance, but also for their potential impact on fire behavior (Rabe *et al.* 2020; Zelinka *et al.* 2022; Marino *et al.* 2024).

Thermal modification involves heating wood at elevated temperatures (typically 160 to 240 °C) in low-oxygen environments, leading to degradation of hemicelluloses and partial reorganization of lignin. These changes reduce the wood's hygroscopicity and improve its biological durability (Esteves and Pereira 2009; Čermák *et al.* 2022). However, their effect on fire behavior is more nuanced, with several studies reporting reduced burning intensity despite changes in ignition behaviour. For instance, Martinka *et al.* (2016) showed that Norway spruce (*Picea abies* L.) and English oak (*Quercus robur*) thermally modified at 200 °C exhibited a marked reduction in peak heat release rate (pHRR), decreasing from 268 and 243 to 218 kW·m⁻² at an external heat flux of 50 kW·m⁻², respectively. In open-flame fire resistance tests, Čekovská *et al.* (2017a) reported lower weight loss for thermally modified spruce, decreasing from 4.72% in untreated wood to the range 3.98% to 4.48% after treatment at 160 to 210 °C. Cone calorimeter measurements by Xing and Li (2014) demonstrated that thermally modified larch (*Larix* spp., 180 to 210 °C, nitrogen atmosphere) showed reduced burning intensity but a shorter ignition time (22 s to 13 s) and increased total smoke production (+4.8% to +43.3%). In contrast, for teak (*Tectona grandis* Linn), Čekovská *et al.* (2017b) observed an increase in weight loss after 10-min direct flame exposure, rising from 4.8% in untreated wood to the range 11.2% to 13.5% in thermally modified wood, highlighting that fire-related benefits of thermal modification are not universal.

Acetylation chemically modifies wood polymers by replacing the hydroxyl groups (–OH) with acetyl groups (–OCOCH₃), typically resulting in 18% to –25% weight percentage gain and substantially improved dimensional stability and durability (Popescu *et al.* 2014; Čermák *et al.* 2022). The effect of acetylation on flammability has been investigated in several studies with inconsistent outcomes. Cone calorimeter measurements on acetylated Monterey pine (*Pinus radiata*, WPG 20%) showed a shorter ignition time compared with unmodified wood (22 vs. 27 s) and a substantially higher first peak heat release rate (274 vs. 187 kW·m⁻²), indicating enhanced initial flammability (Rabe *et al.* 2020). In radiant panel tests on acetylated ash (*Fraxinus excelsior* L.), Morozovs and Bukšāns (2009) reported increased flame phase duration (1185 vs. 1675 s) and flame spread (437 vs. 600 mm) but decreased critical heat flux at extinguishment (4.8 vs. 2.6 kW·m⁻²). Small-flame fire tests conducted by Papadopoulos *et al.* (2010) showed that acetylated OSB (WPG 11.2%) and particleboard (WPG 12.2%) exhibited longer ignition and flaming times (up to 245 vs. 93 s), but substantially reduced glowing durations, reflecting altered

smoldering behaviour. These observations are consistent with findings by Hasburgh and Zelinka (2023), who attributed increased early heat release in acetylated wood to the emission of acetic acid and other volatile degradation products, which may act as additional combustible fuel under well-ventilated burning conditions.

Furthermore, surface charring is an age-old technique, traditionally known from the Japanese Yakisugi (Shou Sugi Ban) method, in which wood surfaces are intentionally burned to create a protective char layer (Kymäläinen *et al.* 2017; Ebner *et al.* 2024). The char layer acts as a carbonized barrier that is hydrophobic, fungal-resistant, and dimensionally stable, as most polysaccharides are decomposed during pyrolysis. Traditional flame charring exposes the wood surface to temperatures exceeding 800 °C, producing a thick (2 to 5 mm) but often cracked and brittle char layer (Ebner *et al.* 2022). In contrast, recent studies have explored low-temperature contact charring using hot plates at temperatures ranging between 250 and 400 °C (Kymäläinen *et al.* 2017; Šeda *et al.* 2021). Lin *et al.* (2023) demonstrated that pre-charred wood surfaces substantially improve fire performance when a continuous char layer is present. Increasing char thickness to 13.8 mm reduced the first peak heat release rate from 155 to 59 kW·m⁻², strongly suppressing early fire growth. Pre-charring also delayed ignition and increased ignition temperature to >650 °C. A minimum effective char thickness of ±6 mm was identified, below which fire performance improvements were negligible. Ebner *et al.* (2024) showed that one-sided surface charring improved fire resistance of silver fir (*Abies alba* Mill.) and European ash (*Fraxinus excelsior*), increasing burn-through time from 17.0 to 23.0 min and 22.0 to 25.0 min, with 6 and 3 mm char layer thickness, respectively. Contact charring using heated plate further confirmed the protective role of engineered char layers, as reported by Machová *et al.* (2021), who showed that one-sided charred beech wood (*Fagus sylvatica* L.) reduced weight loss during fire exposure from 16.6 % (untreated) to 6.8 % after surface charring at 250 °C for 6 min. It is evident that fire performance improvements are highly dependent on char uniformity and thickness and are not universally achieved across wood species (Hasburgh *et al.* 2021).

In addition to standardized fire tests, the fire response of wood is closely related to its thermal degradation behavior, which controls the onset of pyrolysis, volatile release, and char formation during heating. Thermogravimetric and calorimetric techniques are therefore widely used to describe the thermal stability and decomposition pathways of lignocellulosic materials, providing fundamental background information that complements flame-based fire testing (Grønli *et al.* 2002; Yang *et al.* 2007).

Although these modifications have been studied individually, comparative evaluations, especially on hardwoods like beech (*Fagus sylvatica* L.) and birch (*Betula pendula* L.), remain limited. Standardized testing using small-flame ignition (EN ISO 11925-2, 2020), gross heat of combustion (EN ISO 1716, 2018), and thermogravimetric analysis (TGA) can provide a clearer comprehensive picture of how these treatments influence flammability and combustion behavior. It is hypothesized that contact surface-charring will yield improved fire and thermal behavior, followed by thermal modification, while acetylation will provide either minimal or inconsistent benefits when compared to reference material.

EXPERIMENTAL

Material and Methods

European beech (*Fagus sylvatica* L.) and silver birch (*Betula pendula* L.) wood harvested in Training Forest Enterprise, Masaryk Forest Křtiny of Mendel University in Brno, Czech Republic, was studied. The tested materials were prepared from defect-free raw timber in accordance with the requirements of the applied test methods, with additional dimensional allowances to enable subsequent machining to the final specimen dimensions prior to testing.

Wood Modification

Thermal modification

Thermal modification at 200 °C (TM200) was carried out in a laboratory chamber (Katres spol. s r.o., CZ, volume 0.7 m³) in a superheated steam environment at atmospheric pressure. The treatment was performed in five stages: an initial heating ramp 10 °C/h to drying temperature (103 °C), 70 h at this temperature; a second ramp 4 °C/h to reach 130 °C; third ramp 30 °C/h until the maximum temperature (200 °C) was reached, maintained for 3 h; and natural cooling. The process intensity and degree of thermal modification was determined by mass loss (ML%), based on oven-dry (103 ±2 °C) mass before and after the thermal modification process. The ML was calculated according to Eq. 1:

$$ML (\%) = (Dry\ mass_{unmod} - Dry\ mass_{mod} / Dry\ mass_{unmod}) \quad (1)$$

Acetic anhydride modification – Acetylation

The oven-dried specimens were vacuum-impregnated at 10 kPa (absolute pressure) for 0.5 h with acetic anhydride (Acet) using a laboratory vacuum-pressure impregnation plant (J. Hradecký spol. s r.o., CZ, vessel volume 50 L). After vacuum impregnation, the specimens were kept in the chemical at ambient pressure for 0.5 h, then placed in stainless-steel containers and sealed during the acetylation. The acetylation reaction took place at 120 °C in a hot-air oven (Sanyo MOV-212P) for 24 h. On completion, the specimens were dried to 0% MC (12 h at 30 °C, 12 h at 60 °C and 48 h at 120 ±2 °C). The WPG values were calculated according to Eq. 2.

$$WPG (\%) = (Dry\ mass_{mod} - Dry\ mass_{unmod} / Dry\ mass_{unmod}) \quad (2)$$

One-sided surface charring

Specimens were one-sided surface charred by contact heating using a laboratory hot plate (CERAN 33SR) with a built-in Ni-Cr-Ni sensor and electronic temperature controller. The specimens were first dried to 0% MC (103 ±2 °C), then immediately charred to avoid any shape deformation during contact heating. Surface charring was carried out at 300 °C for 60 s at atmospheric pressure. No additional pressure was applied on specimens during the charring process. The char layer thickness was approximately 3 mm, following the methodology of Šeda *et al.* (2021)

For all studied wood modifications, the moisture exclusion efficiency (MEE) at 50% RH was determined according to Eq. 3.

$$MEE (\%) = (EMC_{unmod} - EMC_{mod}) / EMC_{unmod} \quad (3)$$

Fire-Resistance Properties

Single-flame source test

Fire performance related to ignitability and surface flame spread was evaluated using the single-flame source test in accordance with EN ISO 11925-2 (2020). Rectangular specimens with nominal dimensions of 250 × 90 × 20 mm (length × width × thickness) were prepared from reference and modified wood materials. Prior to testing, all specimens were conditioned at 23 ± 2 °C and 50 ± 5% RH until constant mass was achieved. During the test, the specimens were mounted vertically in the test frame and exposed to a propane flame applied to the specimen surface at an angle of 45°. The distance between the burner nozzle and the specimen surface was maintained at approximately 5 mm. The flame application time was 30 s, in accordance with the applied test procedure. For each material variant, ten repetitions were performed, exceeding the minimum requirement of six specimens specified by the standard. Before the fire tests, airflow conditions in the test environment were verified using a Trotec TA300 anemometer, and the air velocity was measured at approximately 0.7 m·s⁻¹ to ensure stable and reproducible testing conditions. During the test, ignition behavior, flame development on the specimen surface, and persistence of flaming after flame removal were visually observed and recorded in accordance with the standard. In addition to the standard observations, the height of the charred surface resulting from flame exposure and the weight loss of the specimens after the test were measured as supplementary parameters to further characterize fire-induced material degradation. Each material group was measured in ten replicates.

Gross heat of combustion

The gross heat of combustion (PCS) was determined in accordance with EN ISO 1716 (2018) using an oxygen bomb calorimeter to quantify the total potential heat release of the tested materials under complete combustion conditions. For this purpose, the wood specimens were mechanically milled to obtain a fine and homogeneous wood powder, from which small pellets were subsequently formed to ensure uniform combustion behavior. For the surface-charred specimens, the PCS was determined separately for two successive material layers: (i) an isolated outer charred surface layer obtained by milling the uppermost 0.5 mm of the charred surface, and (ii) a subsurface layer obtained by milling the subsequent 0.5 mm (*i.e.*, material taken from a depth of 0.5 to 1.0 mm). Approximately 1 g of wood powder was used for each test to prepare the pellets. All samples were oven-dried at 103 ± 2 °C to constant mass prior to testing. Each material group was analyzed in five independent repetitions. A known mass of the prepared pellet was combusted in a high-pressure oxygen atmosphere inside the calorimetric bomb, and the resulting temperature increase of the calorimetric system was continuously recorded. The gross heat of combustion was calculated according to Eq. 4,

$$PCS \text{ (MJ/kg)} = (E \cdot (T_m - T_i + c) - b) / m \quad (4)$$

where E is water equivalent (MJ/K), T_m is the maximum measured temperature after combustion (K), T_i is initial the temperature of the calorimetric system (K), c is temperature correction accounting for heat exchange with the surrounding (K), b is the correction for the heat contribution of auxiliary combustion material used during the test, such as cotton thread (MJ), and m is the dry mass of the test specimen (kg). Each material group was measured in five replicates.

Thermogravimetric analysis

Thermogravimetric measurements were carried out using a simultaneous thermal analysis instrument (Hitachi NEXTA STA200RV), combining thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) in a single unit. The instrument is equipped with RealView technology enabling optical monitoring of the sample during measurement. The balance weight resolution is 0.2 μg , with stability/drift/repeatability error $< 10 \mu\text{g}$. Wood dust (2 to 5 mg) was weighed into aluminium opened crucibles and measured against a blank reference. Experiments were performed under an inert nitrogen atmosphere ($100 \text{ mL} \cdot \text{min}^{-1}$). The temperature program started at 30°C (2 min stabilization) and increased to 500°C at a constant heating rate of $20 \text{ K} \cdot \text{min}^{-1}$. Data were recorded every 0.2 s, and sample images were acquired every 15 s. Each material group was measured in five replicates.

Data Processing and Statistical Analysis

Data were processed, evaluated, and graphed using OriginPro 2022 (OriginLab Corporation). Statistical differences among the studied groups were assessed using the nonparametric Kruskal–Wallis test. When significant differences were detected, pairwise comparisons were performed using Dunn's multiple comparison test with Holm adjustment. Statistical significance (α) was set to 0.05.

RESULTS AND DISCUSSION

Material Characteristics

Table 1 presents key indicators of modification intensity and moisture-related characteristics, such as mass loss (ML), weight percentage gain (WPG), equilibrium moisture content (EMC), and moisture exclusion efficiency (MEE), which together characterize the degree of chemical and structural alterations induced by the applied wood modifications and provide essential context for interpreting their performance.

Table 1. Wood Modification Groups, Process, and Moisture-related Characteristics

Group	ρ_0 (kg/m^3)	ML (%)	WPG (%)	EMC (%)	MEE (%)
BE-Ref	710.1 (13.5)	-	-	9.2 (0.3)	-
BE-TM ₂₀₀	696.5 (23.7)	3.8 (0.3)	-	4.7 (0.1)	48.9
BE-Acet	729.7 (22.7)	-	21.7 (1.4)	3.4 (0.3)	63.0
BE-Charred	707.8 (15.5)	-	-	9.0 (1.2)	2.2
BR-Ref	661.5 (29.8)	-	-	8.9 (%)	-
BR-TM ₂₀₀	648.5 (41.9)	3.2 (0.3)	-	4.9 .(%)	44.9
BR-Acet	683.2 (40.4)	-	24.6 (1.3)	3.1 (%)	65.2
BR-Charred	662.9 (34.9)	-	-	8.5 (%)	4.5

Thermal modification at 200°C resulted in a relatively low mass loss (ML) of 3.8% for beech and 3.2% for birch, indicating mild to moderate treatment severity, in which degradation is dominated by hemicellulose depolymerization and deacetylation, while cellulose degradation remains limited (Esteves and Pereira 2009). Such ML values are characteristic of thermally modified hardwoods processed under industrially relevant schedules aimed at improving hygroscopic behaviour while avoiding excessive

degradation of structural biopolymers. Comparable ML values for beech treated at 200 °C were reported by Čermák *et al.* (2021, 2022), who observed ML in the range of approximately 2.7% to 5.7%, depending on treatment duration. Olarescu *et al.* (2014) reported ML values ranging between 3.2% and 5.1% for beech wood thermally modified for at 200 °C for 2 h and 3 h, respectively. Similarly, Borůvka *et al.* (2021) reported ML values of 5% for beech and 8% for birch wood modified at 210 °C. Mass loss of 6.4% for birch (*Betula pendula*) wood modified at 200 °C for 4 h was reported by Zeman *et al.* (2000) and Esteves *et al.* (2008). These findings confirm that a wide range of ML values can be observed across different processing conditions, while both diffuse-porous hardwood species exhibit comparable degradation kinetics. The ML values obtained in this study indicate a relatively mild treatment severity when compared with other studies employing similar process conditions.

The moderate ML observed in both species is directly reflected in their moisture behavior. Thermal modification significantly reduced EMC, corresponding to MEE values of 48.9% and 44.9%, respectively. These reductions are consistent with literature data reporting EMC decreases of approximately 40% to 55% for wood thermally modified at 200 °C (Altgen *et al.* 2016; Čermák *et al.* 2021; Englund and Fredriksson 2021). Similar EMC reductions for thermally modified wood were also reported by Rautkari and Hill (2014), who attributed this behavior primarily to a reduced availability of accessible hydroxyl groups and partial collapse of the amorphous hemicellulose network. The close correspondence between ML and EMC reduction in the present study indicates that moderate thermal degradation is sufficient to substantially reduce hygroscopicity.

Acetylation resulted in a substantially higher degree of modification, with WPG values of 21.7% for beech and 24.6% for birch. These values are characteristic of strongly acetylated hardwoods and are consistent with results reported in laboratory- and pilot-scale acetylation studies. Čermák *et al.* (2022) reported a WPG of 24.9% for acetylated beech wood, accompanied by pronounced reductions in EMC. Similarly, Popescu *et al.* (2014) demonstrated that acetylated birch wood with WPG levels exceeding 16% exhibits a marked downward shift of the sorption isotherm, which can be attributed to chemical substitution of hydroxyl groups and permanent bulking of the cell wall. In the present study, acetylation led to a significant reduction in EMC, corresponding to MEE values of 63.0% and 65.2% for beech and birch, respectively. These reductions are in agreement with classical acetylation literature, where EMC decreases of approximately 60% to 70% are commonly reported for WPG levels in the range of 20% to 25% (Papadopoulos and Hill 2003; Rowell 2006; Čermák *et al.* 2022). Compared with thermal modification, acetylation therefore provides a more pronounced and stable reduction in wood hygroscopicity.

In contrast, one-sided surface charring induced only negligible changes in bulk moisture behaviour. The EMC values of charred wood remained close to those of the corresponding reference materials, resulting in low MEE values (2.2% to 4.5%). This response is characteristic of one-sided or superficial charring, where modification is confined to a thin surface layer and does not substantially affect the hygroscopic properties of the wood core. Similar findings were reported for one-sided charred beech by Čermák *et al.* (2019) and Šeda *et al.* (2021), who showed that although surface wettability and density profiles are markedly altered, the EMC of the bulk material remains largely unchanged. Comparable conclusions were also drawn by Kymäläinen *et al.* (2022), demonstrating that surface carbonization primarily affects surface moisture interactions rather than the equilibrium sorption of the entire specimen.

Fire Resistance Properties

The reaction-to-fire behavior of reference beech and birch wood, as well as modified wood was assessed using the EN ISO 11925-2 (2020) small flame test. All tested materials exhibited flame spread behavior comparable to their respective reference specimens, with no specimen reaching the critical flame spread height of 150 mm within the specified observation time and no visually significant occurrence of flaming droplets or sustained post-flame combustion. According to the classification framework of EN 13501-1, solid wood products of this type fall within Euroclass D when assessed by the small flame test alone, and from that perspective the applied modification treatments did not result in any observable change in classification (Östman *et al.* 2010).

Although the EN ISO 11925-2 (2020) assessment did not indicate any change in reaction-to-fire classification according to EN 13501-1 (2018), Fig. 1 clearly demonstrates statistically significant differences in flame spread height among the studied modifications. These results suggest that while the applied treatments do not affect compliance-based criteria, they do influence the early-stage flame propagation behaviour under small-flame exposure. For both beech and birch, reference wood and thermally modified wood at 200 °C (TM200) indicating comparable flame spread behaviour. This seems to be consistent with previous findings showing that moderate thermal modification temperatures around 160 to 200 °C result in no statistically significant changes to ignitability and flame spread, as hemicellulose degradation and char yield enhancement remain insufficient to alter flame propagation under small-flame conditions (Martinka *et al.* 2016; Čekovská *et al.* 2017a; Luptáková *et al.* 2019).

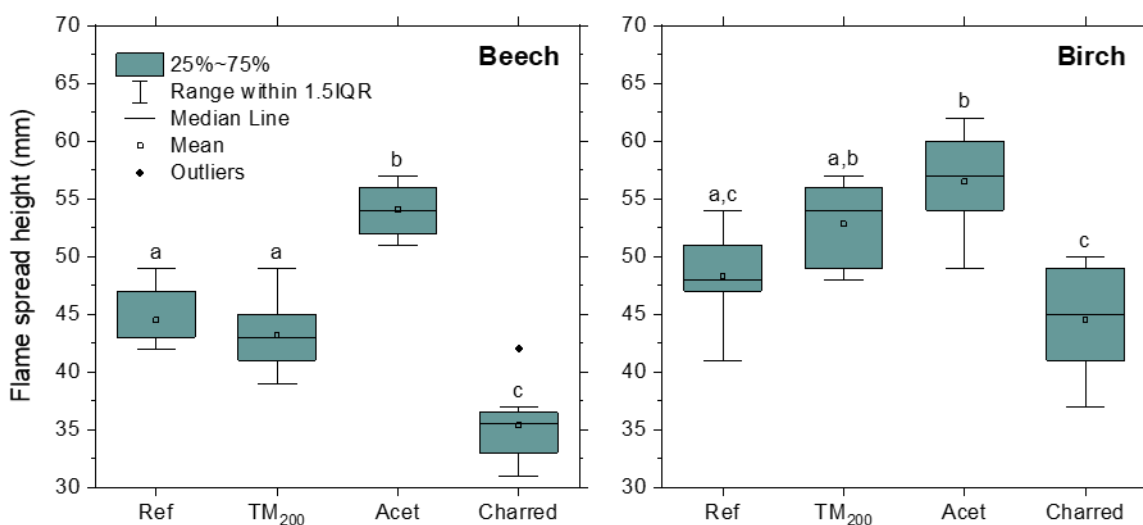


Fig. 1. Flame spread height (mm) measured during the small flame (n=10) test for beech (left) and birch (right) wood specimens

In contrast, acetylated wood exhibited significantly higher flame spread heights in both studied species. This observation is consistent with some studies showing that acetylation does not improve, and may locally increase, flammability indicators in small-scale and bench-scale fire tests (Mohebbi *et al.* 2007; Rabe *et al.* 2020). This behaviour can be explained by a combination of chemical and physical changes induced by acetylation. During thermal decomposition, acetylated wood releases increased amounts of acetyl-related volatile products, particularly acetic acid, originating from cleavage of ester

bonds. This has been detected in pyrolysis and combustion gases and is known to participate in flame chemistry under well-ventilated conditions (Schwarzinger and List 2010; Hasburgh and Zelinka 2023). In addition, acetylation substantially reduces EMC, thereby limiting the endothermic heat sink associated with water evaporation during heating. Reduced moisture buffering shortens the preheating phase prior to pyrolysis and ignition, facilitating earlier volatile release and flame propagation, a relationship that is well established for wood materials in ignition and flame-spread studies (Spearpoint and Quintiere 2000; Cuevas and Maluk 2023). Together, these effects explain why acetylated wood may display increased flame spread under small-flame exposure despite its improved material properties.

The one-sided surface-charred specimens exhibited a clear reduction in flame spread height, with the effect being most pronounced for beech. This behaviour can be attributed primarily to the formation of a continuous surface char layer, which acts as a physical and thermal barrier, reducing heat transfer into the underlying wood and thereby limiting the generation and release of flammable pyrolysis gases during small-flame exposure (Ebner *et al.* 2024). Although surface charring reduces the equilibrium moisture content, the protective effect of the char layer dominates, suppressing ignition and flame spread. In this sense, surface charring alters the initial fire–surface interaction, weakening flame attachment and slowing flame propagation along the specimen surface. The stronger response observed for beech compared with birch is likely related to species-specific differences in density, anatomical structure, and char formation behaviour. Beech has a higher average density (680 kg/m^3) and a more uniform diffuse-porous structure, which may promote the development of a denser and more cohesive char layer with better barrier properties. Birch, although also diffuse-porous, typically exhibits lower density (610 kg/m^3) and higher porosity, which likely result in a more fragmented or mechanically weaker char layer under identical charring conditions. Such species-dependent differences in char cohesion, density, porosity, and thermal response have been shown to influence flame spread and surface burning behaviour in wood (Lowden and Hull 2013; Bartlett *et al.* 2019; Mensah *et al.* 2023).

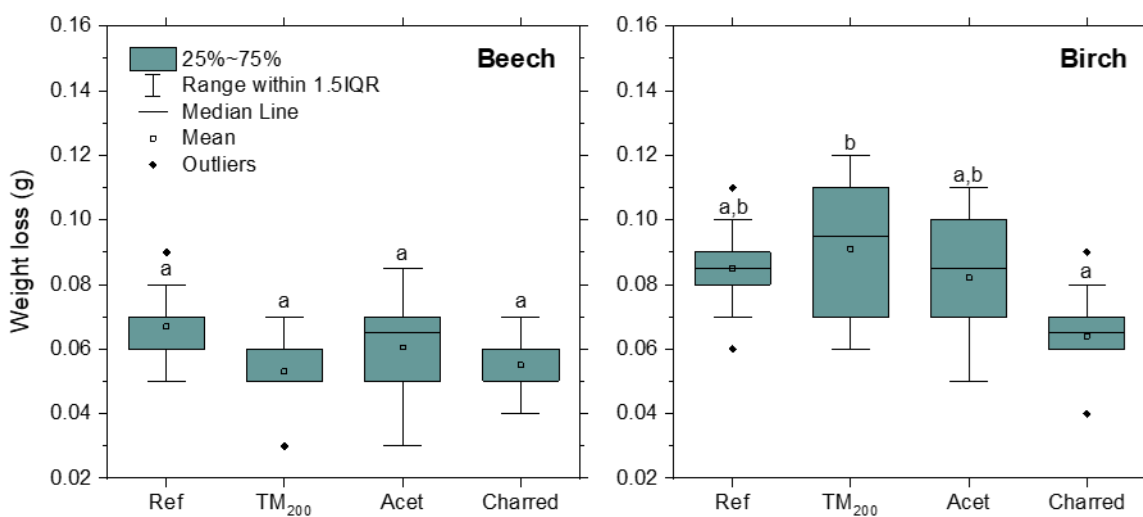


Fig. 2. Weight loss measured during the small flame test ($n=10$) for beech (*left*) and birch (*right*) wood specimens

Figure 2 shows the weight loss measured after small-flame exposure for beech and birch specimens. Overall, no statistically significant differences were observed among the studied modification types within each species, indicating that studied modifications did not substantially affect weight loss under the applied small-flame conditions. When comparing species, birch exhibited a slightly higher weight loss than beech, although the differences were small. This tendency can be attributed to species-related factors such as the lower density and higher porosity of birch, which may facilitate faster heat penetration and a more pronounced release of volatile decomposition products during short-term flame exposure.

Figure 3 presents the gross heat of combustion (PCS) determined for beech and birch after small-flame exposure. PCS represents the total amount of energy released by complete combustion of a unit mass of dry material, as measured by bomb calorimetry, and reflects the overall energy content of the solid material. For both wood species, all modified groups exhibited higher PCS values than the respective reference materials, indicating an increased energy density. This increase is consistent with modification-induced changes in chemical composition, such as a relative enrichment in lignin and carbon-rich structures due to hemicellulose degradation (thermal modification) or surface charring, as well as the incorporation of acetyl groups in acetylated wood. Lignin and carbonized structures are known to possess higher calorific values than polysaccharides, which explains the systematic increase in PCS observed here. Comparable PCS ranges and similar trends—higher calorific values for modified or carbon-enriched wood materials—have been reported for hardwoods using the same bomb-calorimetric approach (Demirbaş 2001; Telmo and Lousada 2011; Poletto *et al.* 2012).

Importantly, higher PCS does not directly imply improved fire performance. While materials with higher energy content can release more heat once fully involved in combustion, early-stage fire behaviour (such as flame spread height in EN ISO 11925-2 (2020)) is primarily governed by surface processes, heat transfer, and volatile release kinetics. This helps to explain why acetylated and charred specimens may show higher PCS yet contrasting flame spread behaviour, with acetylation promoting flame spread and surface charring suppressing it. Thus, the PCS results provide complementary information on material energy potential, rather than contradicting the flame-spread observations.

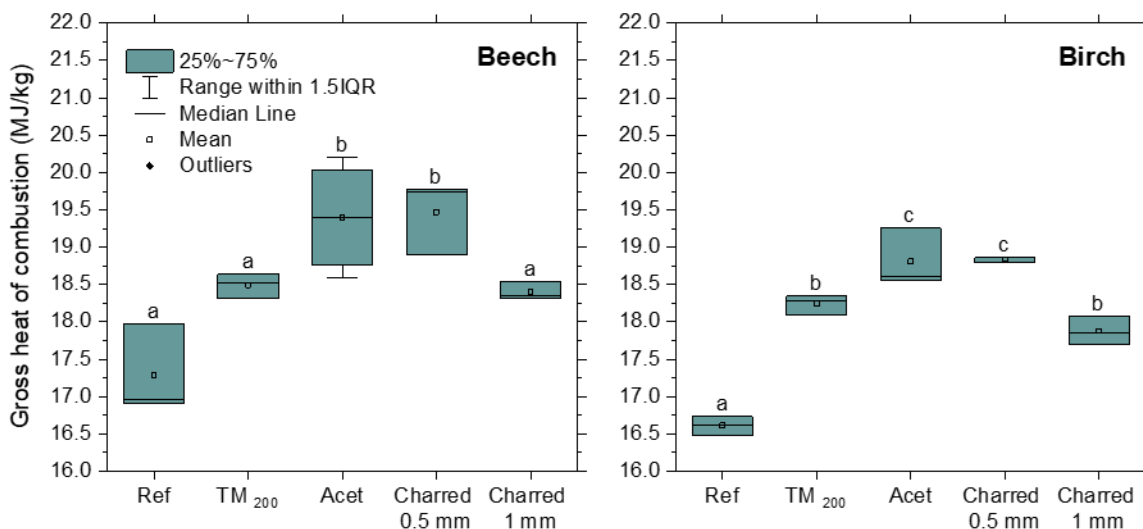


Fig. 3. Gross heat of combustion ($n=5$) recorded for beech (*left*) and birch (*right*) wood specimens

The thermogravimetric (TG) and derivative thermogravimetric (DTG) curves obtained for beech and birch revealed systematic effects of wood species and modification type on thermal degradation behaviour (Fig. 4). Despite these differences, all samples followed the characteristic multi-stage thermal decomposition pattern of lignocellulosic materials. In all specimens, an initial minor mass loss (ML) was observed up to approximately 120 to 150 °C. This stage was associated with the release of physically bound moisture and low-molecular volatile compounds and was reflected by very low DTG intensities. The magnitude of this low-temperature ML differed slightly among the studied groups, with acetylated wood showing a noticeably reduced ML compared with reference samples. This behaviour is consistent with its substantially lower EMC (Table 1), indicating reduced moisture uptake and, consequently, limited moisture desorption during the initial heating stage.

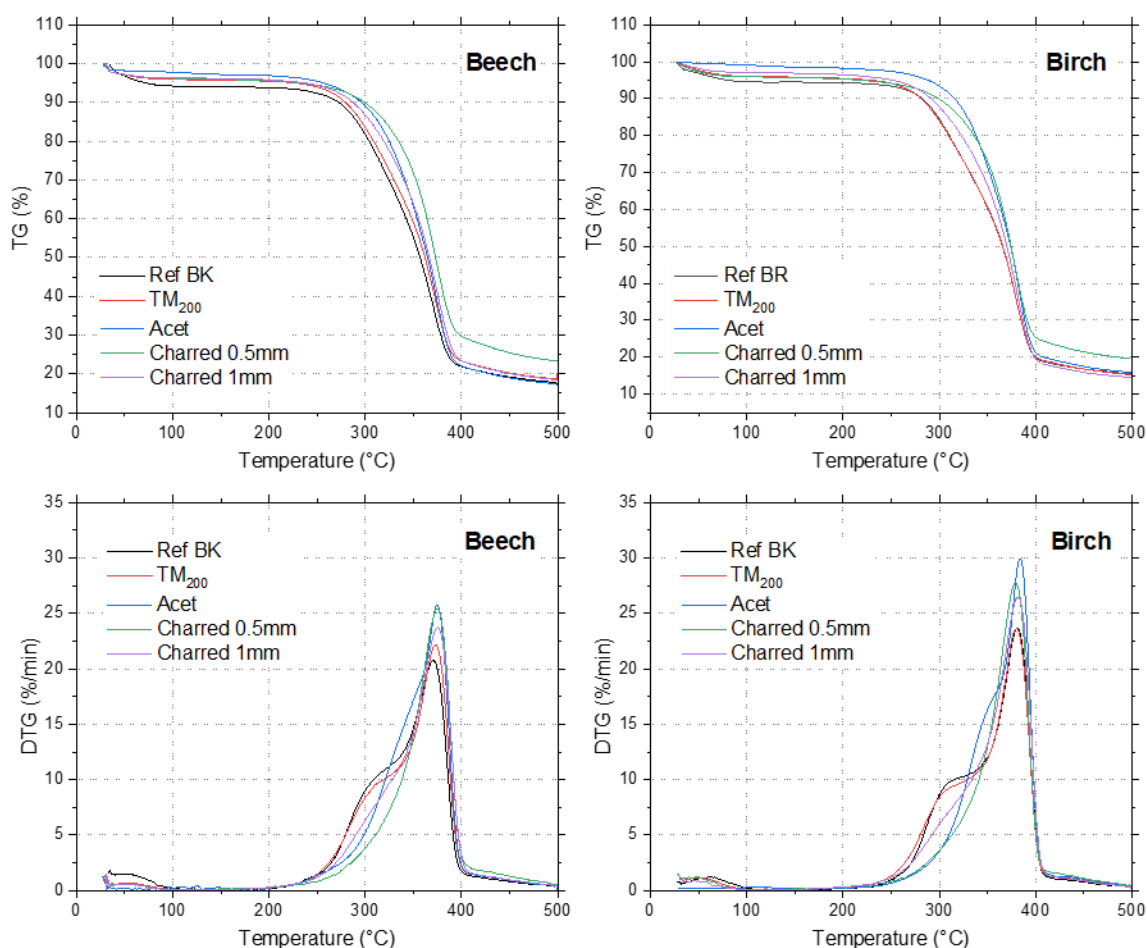


Fig. 4. Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of reference, thermally modified, acetylated, and surface-charred beech and birch wood (n=5)

The main ML occurred between approximately at 200 °C and 380–400 °C and dominated the TG curves of all reference and modified samples. Within this region, a shoulder or secondary DTG feature was observed at lower temperatures, around 250 to 350 °C, followed by a pronounced DTG maximum at approximately 350 to 400 °C. The lower-temperature shoulder is commonly attributed to hemicellulose degradation, whereas the main DTG peak corresponds primarily to cellulose depolymerization (Grønli *et al.* 2002;

Yang *et al.* 2007). This shoulder seems to be more pronounced in birch than in beech, which is consistent with the higher xylan content of birch and its tendency toward earlier and broader hemicellulose decomposition (Shen *et al.* 2010). The evolution of the solid residue during the heating process is illustrated in Figs. 5 and 6 for both beech and birch. The photographic documentation confirms the progressive formation of carbonaceous material and the gradual structural collapse of the samples with increasing temperature. These images provide visual support for the TG/DTG results and illustrate the physical changes associated with the thermal degradation process.

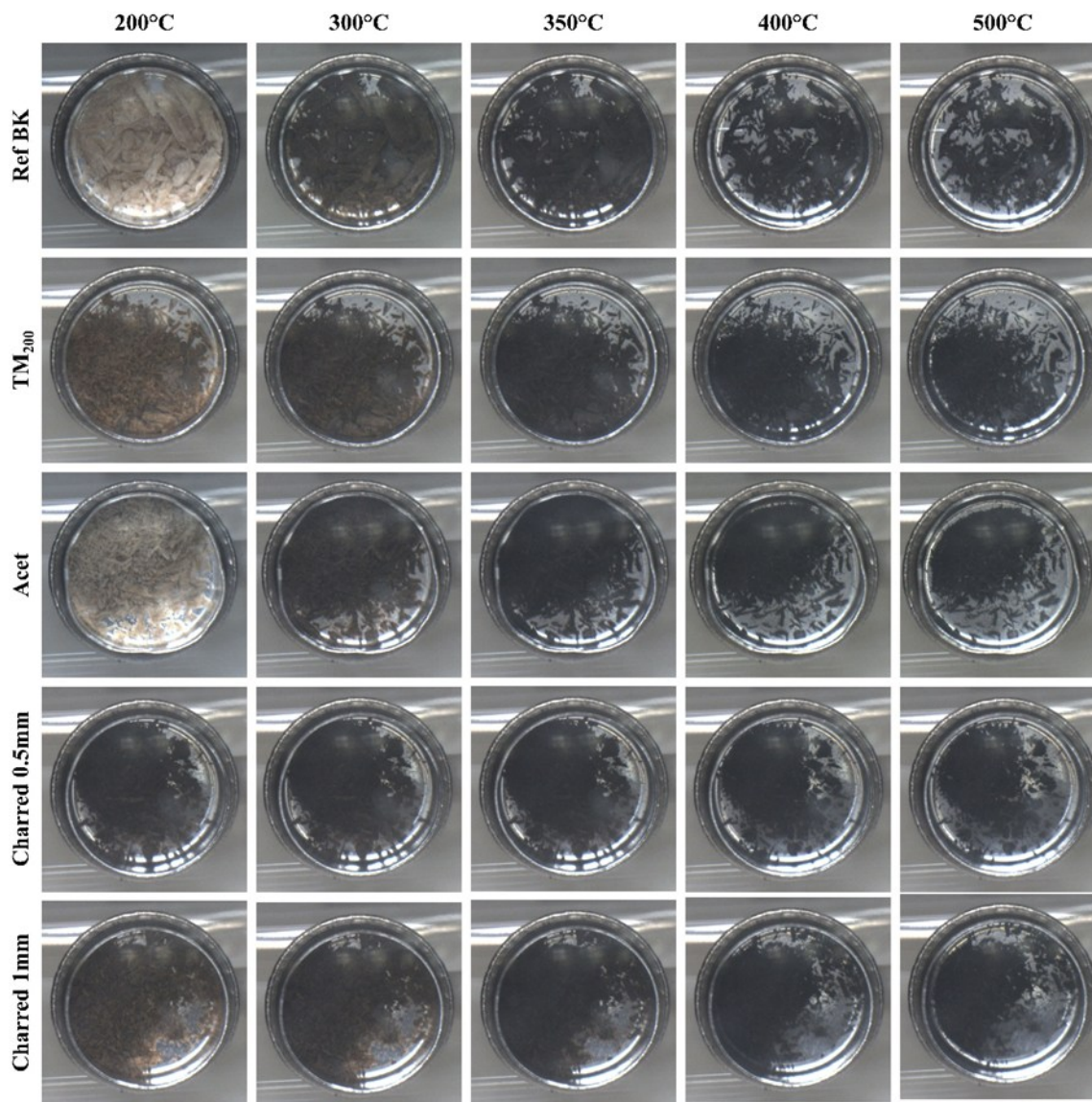


Fig. 5. Visual appearances of solid residues of reference, thermally modified, acetylated, and surface-charred beech wood samples at selected temperatures during TGA

Thermally modified samples exhibited TG and DTG profiles that were broadly similar to those of the respective reference wood, with only small shifts in the onset and shape of the main degradation region. These changes indicate partial degradation of thermally labile hemicelluloses during the modification process, while the cellulose-

dominated decomposition peak were largely unchanged in position. Such behaviour is consistent with thermal modification at moderate temperatures (~180 to 220 °C), where hemicelluloses undergo deacetylation and depolymerisation, whereas cellulose retains its structural integrity and decomposes within a similar temperature range as in untreated wood (Grønli *et al.* 2002; Yang *et al.* 2007; Esteves and Pereira 2009). In this context, TGA has been shown to be a sensitive tool for capturing these modification-induced changes in wood composition and for assessing the degree of thermal modification, particularly through ML in the hemicellulose-dominated temperature range (Korošec *et al.* 2017).

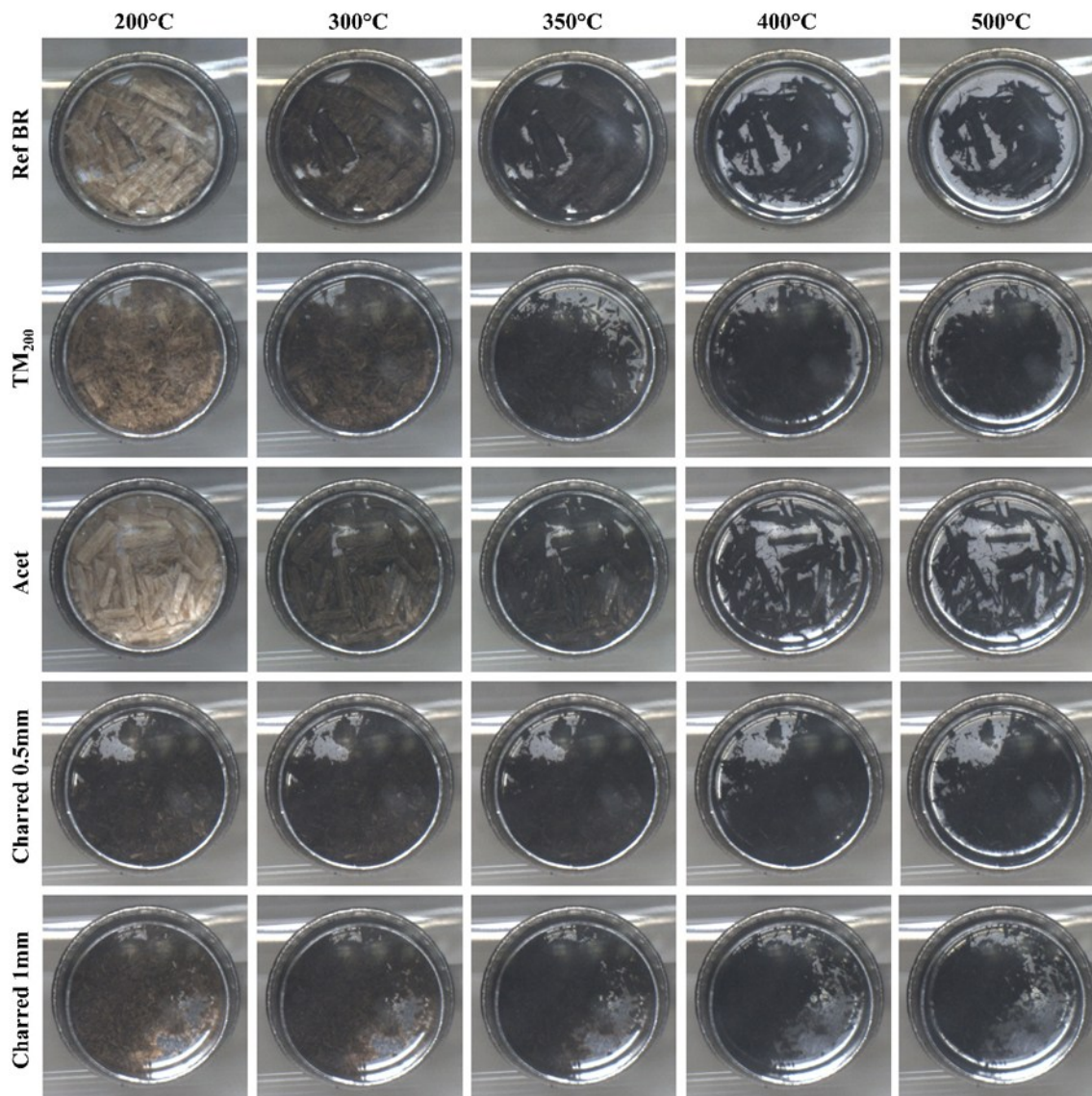


Fig. 6. Visual appearance of solid residues of reference, thermally modified, acetylated, and surface-charred birch wood samples at selected temperatures during TGA

Acetylated wood exhibited a more pronounced modification of the thermal degradation profile compared with reference and thermally modified samples. In both beech and birch, the onset of rapid mass loss was shifted towards slightly higher

temperatures, indicating delayed initiation of thermal decomposition. However, the temperature of the main DTG maximum remained essentially unchanged and occurred at approximately the same temperature as in the reference and thermally modified wood (± 380 °C). The primary difference was in the shape of the DTG curves: the peak intensity was reduced and the characteristic shoulder around ~ 300 °C, visible in reference and TM samples, was strongly diminished or absent in acetylated wood. This behaviour suggests a reduced contribution of early-degrading fractions, which are typically associated with hemicelluloses, and a more gradual overall degradation process.

These effects are consistent with the chemical substitution of hydroxyl groups by acetyl groups, which reduces hygroscopicity, limits the accessibility of reactive sites, and alters the thermal response of polysaccharide components without fundamentally changing the cellulose-dominated decomposition stage (Hill 2006; Rowell 2013). Notably, acetylated wood exhibits a higher DTG peak intensity than the reference and thermally modified groups, indicating a more concentrated ML event during the main decomposition stage despite a similar peak temperature. The stronger response observed for acetylated birch can plausibly be linked to its slightly higher degree of acetylation (Table 1), which would further reduce the contribution of early-degrading hemicellulose-related reactions and shift a larger fraction of the devolatilization into the main cellulose-dominated peak (Hill 2006; Yang *et al.* 2007; Rowell 2013).

The most pronounced differences were observed for the charred samples. Both charred layers show strongly suppressed ML throughout the temperature range up to approximately 350 to 400 °C, reflecting the extensive thermal degradation induced during the surface charring process. The characteristic hemicellulose-related shoulder and the main cellulose DTG peak were largely absent, particularly in the material taken from the outermost 0.5 mm of the surface, where the DTG curves approached a nearly flat profile. This behaviour indicates that the majority of thermally labile polysaccharides in this surface layer had already been decomposed during direct exposure to the heating source. Sample taken from beneath the surface layer showed only minor differences compared with the outermost charred layer, which can be likely attributed to its slightly greater distance from the heating source and the consequently lower degree of thermal degradation during charring process.

CONCLUSIONS

1. The applied wood modification techniques differed markedly in modification intensity and resulting fire and thermal behaviors. Thermal modification resulted in moderate mass loss (ML) and a reduction in equilibrium moisture content (EMC). Acetylation achieved the highest degree of chemical modification with the strongest reduction in hygroscopicity, whereas one-sided surface charring caused only negligible changes in bulk moisture characteristics, confirming that the modification was confined to a thin surface layer.
2. Despite these differences in modification intensity, none of the treatments altered the Euroclass-related reaction-to-fire classification under the EN ISO 11925-2 (2020) small-flame test; however, clear differences in early-stage fire behavior were observed. Acetylated wood exhibited increased flame spread due to reduced moisture buffering and the release of additional combustible volatiles, whereas surface-charred specimens

showed a pronounced reduction in flame propagation, particularly for beech, due to the presence of a continuous, thermally insulating char layer.

3. All modified materials exhibited increased gross heat of combustion compared with reference wood, indicating higher energy content associated with carbon enrichment or chemical modification. However, higher energy density did not translate into improved fire resistance, as total combustion energy reflects potential heat release rather than the kinetics of ignition and flame spread.
4. Thermogravimetric analysis showed that thermal modification retained comparable degradation pathways to reference wood, whereas acetylated wood exhibited altered devolatilization behavior and charred layers displayed strongly suppressed mass loss due to prior thermal degradation.
5. Overall, the results confirm surface charring as the most effective modification for reducing flame spread and highlight the necessity of combining flame-based, calorimetric, and thermal analysis methods for a reliable assessment of fire behavior in modified wood.

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