

EFFECT OF HEATING RATE ON THE THERMAL DEGRADATION BEHAVIOUR OF NORWAY SPRUCE WOOD EVALUATED BY SIMULTANEOUS THERMAL ANALYSIS

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ABSTRACT

The effect of heating rate on the thermal degradation behaviour of Norway spruce (*Picea abies*) wood was investigated under an inert nitrogen atmosphere using simultaneous thermal analysis. Wood samples were heated at rates of 10, 25 and 50 K·min⁻¹ over a nominal temperature range of 25–500 °C, with three independent measurements performed at each heating rate. Thermogravimetric (TG), derivative thermogravimetric (DTG) and differential scanning calorimetry (DSC) data were evaluated to characterise the degradation process. Increasing the heating rate shifted the characteristic mass-loss temperatures towards higher values. Between 10 and 50 K·min⁻¹, the temperatures corresponding to 10, 20 and 50 % mass loss increased by 10.93, 12.47 and 15.07 K, respectively. The principal DTG features and the associated DSC responses also shifted towards higher temperatures. Residual mass at the end of the recorded temperature programme increased from 21.83 to 27.21 %, although differences in the actual final recorded temperatures limit direct comparison at an identical terminal temperature. The findings demonstrate that heating rate substantially influences the apparent thermal degradation behaviour of Norway spruce wood. These results highlight the importance of accounting for the applied heating programme when interpreting and comparing non-isothermal thermal analysis data.

Keywords: Norway spruce wood; heating rate; thermal degradation; simultaneous thermal analysis; nitrogen atmosphere.

INTRODUCTION

Wood is widely used as a structural material and renewable energy resource, making its thermal degradation behaviour relevant to fire safety, thermal modification and thermochemical conversion. Under an inert atmosphere, wood undergoes a complex, multistage decomposition process involving its principal structural components: hemicelluloses, cellulose and lignin. These components differ in thermal stability and contribute differently to volatile release and the formation of solid residues. Hemicelluloses generally begin to decompose at lower temperatures, cellulose exhibits a more pronounced mass-loss peak over a relatively narrow temperature interval, and lignin degrades gradually across a broader temperature range. However, the decomposition of these components overlaps in whole wood, complicating the identification of individual degradation stages (Órfão *et al.*, 1999; Yang *et al.*, 2007; Di Blasi, 2008; Poletto *et al.*, 2012).

The heating rate is a key experimental parameter influencing the thermal degradation behaviour observed during non-isothermal analysis. Increasing the heating rate generally shifts the thermogravimetric (TG) and derivative thermogravimetric (DTG) curves towards higher temperatures, affecting the apparent onset of decomposition, the position and intensity of mass-loss-rate maxima, and the temperature intervals associated with individual degradation stages. These shifts may arise from the combined effects of temperature-dependent reaction rates, thermal lag and the limited time available for decomposition as the temperature increases. Heating rate may also influence the residual mass measured at a fixed final temperature because different heating programmes provide different residence times within the principal degradation region. Consequently, changes in characteristic degradation temperatures or residual mass cannot necessarily be interpreted as changes in the intrinsic thermal stability or composition of the material (Branca and Di Blasi, 2003; Branca *et al.*, 2005; Mehrabian *et al.*, 2012; Jaroenkhasemmesuk and Tippayawong, 2016).

Previous investigations have demonstrated heating-rate-dependent thermal degradation behaviour in various lignocellulosic materials, although their results must be interpreted in relation to the materials and experimental conditions employed. Studies of whole wood and woody biomass, including poplar wood, pinewood and sawdust, have reported changes in characteristic degradation temperatures, DTG peak positions and devolatilisation behaviour with increasing heating rate (Branca *et al.*, 2005; Słopiecka *et al.*, 2012; Mishra *et al.*, 2015; Jaroenkhasemmesuk and Tippayawong, 2016). Research on isolated hemicellulose, cellulose and lignin has provided further insight into the thermal behaviour of individual wood components, although the degradation characteristics of these isolated fractions cannot be directly extrapolated to whole wood because of overlapping reactions and interactions between components (Órfão *et al.*, 1999; Yang *et al.*, 2007). Moreover, differences in sample preparation, particle size, sample mass, heating rate, experimental atmosphere and final temperature can influence the resulting thermal characteristics. These factors must therefore be considered when comparing degradation parameters reported in different studies (Skreiberg *et al.*, 2011; Mehrabian *et al.*, 2012; Silva *et al.*, 2023).

Norway spruce (*Picea abies* [L.] H. Karst.) has been investigated in several experimental and modelling studies addressing wood pyrolysis and thermal decomposition. Samuelsson *et al.* (2015) developed a model-free rate expression describing the non-isothermal and isothermal pyrolysis of Norway spruce, while Tapasvi *et al.* (2013) and Barta-Rajnai *et al.* (2017) examined the decomposition kinetics of wood and related materials, including the effects of torrefaction. Other investigations have considered spruce-derived kraft fractions and the pyrolysis of wood chips, highlighting differences associated with material preparation and experimental scale (Samuelsson *et al.*, 2016; Samuelsson *et al.*, 2017). More recently, Terrei *et al.* (2022) and Flity *et al.* (2023) investigated the thermal degradation of spruce wood under inert conditions using experimental data and multiscale modelling approaches. Simultaneous thermal analysis (STA) has also been applied to Norway spruce, including the characterisation of fungal depolymerisation, demonstrating the possibility of recording mass changes and associated thermal effects in a single experiment (Amiandamhen *et al.*, 2024).

These studies provide valuable information on spruce pyrolysis, degradation kinetics and material-scale thermal behaviour. However, their differing research objectives and experimental conditions limit direct comparisons of heating-rate-dependent degradation characteristics. In particular, studies focusing on kinetic modelling, thermally modified materials or multiscale simulations do not necessarily provide a directly comparable assessment of TG, DTG and differential scanning calorimetry (DSC) responses of untreated Norway spruce under identical experimental conditions. A combined evaluation of these

signals is relevant because TG and DTG characterise the extent and rate of mass loss, while DSC provides complementary information on the thermal effects accompanying decomposition. Their simultaneous assessment can therefore help distinguish changes in the apparent position and intensity of degradation events from differences in the extent of decomposition at a specified final temperature.

The aim of this study was to evaluate the effect of heating rate on the thermal degradation behaviour of Norway spruce wood using simultaneous thermal analysis under an inert nitrogen atmosphere. Measurements were performed at heating rates of 10, 25 and 50 K·min⁻¹ over a temperature range of 25–500 °C. The investigation focused on identifying the principal degradation stages, quantifying changes in characteristic mass-loss temperatures (T_{10} , T_{20} and T_{50}), and determining the temperatures and maximum rates of mass loss from the DTG curves. The associated DSC responses and residual mass at 500 °C were also evaluated. By comparing these characteristics under consistent experimental conditions, the study aims to establish how the selected heating rate affects the interpretation and comparability of thermal degradation data for Norway spruce wood.

MATERIALS AND METHODS

Material

Bark-free Norway spruce (*Picea abies* (L.) H. Karst.) wood obtained from the University Forest Enterprise of the Technical University in Zvolen, Slovakia, was used as the experimental material. The wood was mechanically milled using a PULVERISETTE 15 cutting mill (FRITSCH GmbH, Idar-Oberstein, Germany) and subsequently sieved using an AS 200 basic sieve shaker (RETSCH GmbH, Haan, Germany). A particle-size fraction of 100–250 µm was selected for the thermal analysis.

Before measurement, the prepared wood powder was dried at 105 °C for 24 h to constant mass, cooled in a desiccator and stored in a sealed desiccator until analysis to minimise moisture uptake. The initial sample mass ranged from 8.2 to 9.5 mg.

Simultaneous thermal analysis

The thermal degradation behaviour of Norway spruce wood was investigated by simultaneous thermogravimetry and differential scanning calorimetry (TG/DSC) using a NETZSCH STA 509 Jupiter® Classic instrument (NETZSCH-Gerätebau GmbH, Selb, Germany). The samples were placed in open Al₂O₃ crucibles with a volume of 85 µL and distributed as a thin, uniform layer to minimise temperature gradients within the sample. The reference crucible was left empty.

The experiments were performed under non-isothermal conditions from 25 to 500 °C at constant heating rates of 10, 25 and 50 K·min⁻¹. Three independent measurements were performed at each heating rate.

High-purity nitrogen (N₂, 99.999 %) was used as the inert experimental atmosphere. Nitrogen was supplied to the sample compartment as the purge gas at a flow rate of 50 mL·min⁻¹. A separate nitrogen protective-gas flow of 20 mL·min⁻¹ was used exclusively to protect the balance system and did not form part of the experimental atmosphere surrounding the sample.

Thermogravimetric (TG) and differential scanning calorimetry (DSC) signals were recorded simultaneously. TG data were normalised to the initial sample mass and expressed as relative mass (%), while the DSC signal was normalised to the sample mass and expressed in mW·mg⁻¹. The DSC signal was evaluated according to the instrumental sign convention

used during acquisition, with negative heat-flow values representing the exothermic direction ($\text{EXO} = -1$).

Data processing and evaluated parameters

TG, DTG and DSC data were processed using NETZSCH Proteus® Analysis software, version 9.4.4 (NETZSCH-Gerätebau GmbH, Selb, Germany).

The characteristic temperatures T_{10} , T_{20} and T_{50} were determined from the TG curves and defined as the temperatures corresponding to mass losses of 10, 20 and 50 % of the initial sample mass, respectively. The residual mass at the end of the applied temperature programme was also determined.

Derivative thermogravimetric (DTG) curves were generated from the TG data by numerical differentiation of mass with respect to temperature without additional smoothing. The DTG signal was expressed in $\% \cdot \text{K}^{-1}$. The temperature of the principal maximum mass-loss rate (T_{max}) was identified from the minimum of the DTG curve.

The DSC curves were evaluated to compare changes in the thermal response accompanying degradation at the three heating rates. The evaluation focused on the displacement and shape of characteristic thermal features rather than on kinetic or enthalpy analysis.

Statistical evaluation

The TG, DTG and DSC records were evaluated separately for each experimental run. Quantitative parameters were calculated from three independent measurements at each heating rate and expressed as mean values $\pm$ standard deviation (SD).

The effect of heating rate was evaluated descriptively by comparing characteristic mass-loss temperatures, DTG profiles, DSC responses and residual mass. No inferential statistical tests were applied; consequently, the term *significant* is not used to denote statistical significance.

RESULTS AND DISCUSSION

Thermal degradation stages of Norway spruce wood

The thermal degradation behaviour of Norway spruce wood was affected systematically by the imposed heating rate. The TG and DTG curves obtained under nitrogen at 10, 25 and 50 $\text{K} \cdot \text{min}^{-1}$ are shown in Fig. 1. The characteristic TG parameters derived from the individual measurements are summarised in Tab. 1.

The overall TG profiles were characteristic of the multistage degradation of lignocellulosic material. A minor initial mass decrease was followed by the principal degradation interval and subsequently by a slower decrease in mass towards the end of the temperature programme. This behaviour reflects the overlapping degradation of the principal structural components of wood. Hemicelluloses contribute predominantly to the lower-temperature part of the degradation region, cellulose dominates the rapid mass-loss interval, whereas lignin decomposes over a considerably broader temperature range (Yang *et al.*, 2007; Branca *et al.*, 2005). Because these reactions overlap in whole wood, the experimentally observed TG and DTG responses cannot be assigned exclusively to individual chemical components.

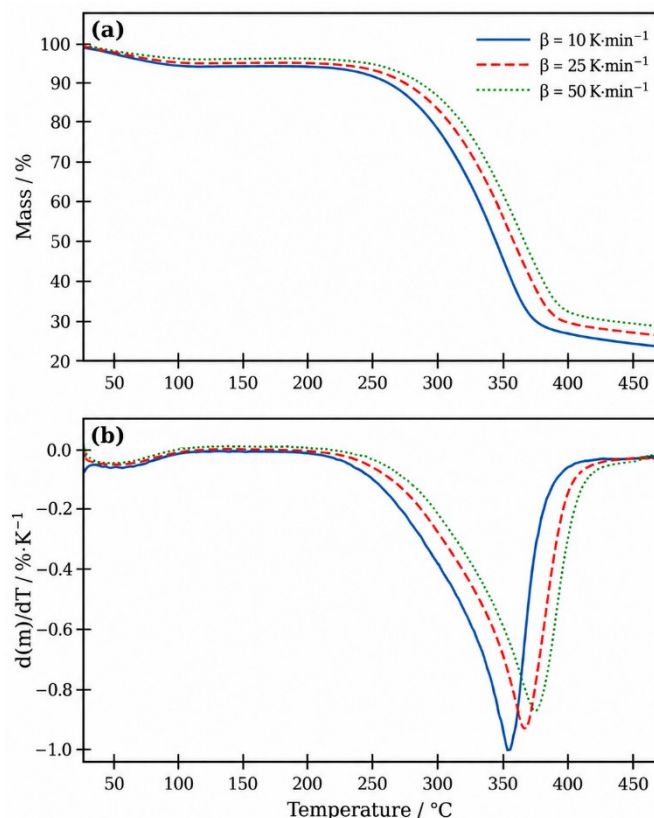


Fig. 1 TG (a) and DTG (b) curves of Norway spruce wood under a nitrogen atmosphere at heating rates of 10, 25 and 50 K·min⁻¹. Curves represent mean values of three independent measurements.

Increasing the heating rate shifted the TG profiles towards higher temperatures. This trend was observed for all three characteristic mass-loss temperatures. T_{10} increased from 275.96 ± 1.75 °C at 10 K·min⁻¹ to 283.77 ± 6.55 °C at 25 K·min⁻¹ and 286.89 ± 1.88 °C at 50 K·min⁻¹. The corresponding increase between 10 and 50 K·min⁻¹ was 10.93 K.

A similar behaviour was observed for T_{20} . The mean value increased from 306.16 ± 1.07 °C at 10 K·min⁻¹ to 316.90 ± 3.80 °C at 25 K·min⁻¹ and 318.63 ± 0.20 °C at 50 K·min⁻¹. The total displacement between the lowest and highest heating rates was 12.47 K.

The largest displacement among the three TG-derived characteristic temperatures was observed for T_{50} . Its value increased from 351.48 ± 0.89 °C at 10 K·min⁻¹ to 364.78 ± 2.71 °C at 25 K·min⁻¹ and 366.55 ± 1.83 °C at 50 K·min⁻¹, corresponding to a difference of 15.07 K.

Tab. 1 Characteristic TG parameters of Norway spruce wood determined under a nitrogen atmosphere at different heating rates. Values are expressed as mean $\pm$ standard deviation (n = 3).

Parameter	10 K·min ⁻¹	25 K·min ⁻¹	50 K·min ⁻¹
T_{10} / °C	275.96 ± 1.75	283.77 ± 6.55	286.89 ± 1.88
T_{20} / °C	306.16 ± 1.07	316.90 ± 3.80	318.63 ± 0.20
T_{50} / °C	351.48 ± 0.89	364.78 ± 2.71	366.55 ± 1.83
Residual mass / %	21.83 ± 1.86	24.71 ± 4.14	27.21 ± 0.1

The progressive displacement of T_{10} , T_{20} and T_{50} demonstrates that characteristic temperatures obtained by dynamic thermal analysis are dependent on the imposed heating programme and should not be regarded as invariant material constants. At a higher heating rate, less time is available for degradation to proceed at any particular temperature. The sample therefore reaches a higher furnace temperature before an equivalent degree of mass loss is attained. Thermal lag within the sample and between the sample and the programmed furnace temperature may additionally contribute to this displacement. Similar heating-rate-dependent shifts have been reported for wood and other lignocellulosic materials (Branca *et al.*, 2005; Słopiecka *et al.*, 2012; Jaroenkhaseemmesuk and Tippayawong, 2016).

The DTG curves provide complementary information on the rate and overlap of the degradation processes (Fig. 1b). At all heating rates, a dominant principal mass-loss event was observed together with a less pronounced lower-temperature contribution. The main DTG feature shifted towards higher temperatures as the heating rate increased. At the same time, the lower-temperature feature became less clearly separated from the principal degradation event at the higher heating rates.

This behaviour is consistent with increasing overlap of degradation processes when the temperature programme is accelerated. In whole Norway spruce wood, the principal DTG maximum should therefore be interpreted as the combined response of the lignocellulosic matrix rather than as the decomposition of cellulose alone. Studies of isolated cellulose, hemicellulose and lignin demonstrate markedly different degradation ranges, but their responses overlap substantially when the components are present together in the wood matrix (Yang *et al.*, 2007).

The heating-rate effect was also reflected in the amount of solid material remaining at the end of the temperature programme. The mean residual mass increased from 21.83 ± 1.86 % at $10 \text{ K} \cdot \text{min}^{-1}$ to 24.71 ± 4.14 % at $25 \text{ K} \cdot \text{min}^{-1}$ and 27.21 ± 0.11 % at $50 \text{ K} \cdot \text{min}^{-1}$ (Tab. 1). The difference between 10 and $50 \text{ K} \cdot \text{min}^{-1}$ was therefore 5.38 %.

The increase in residual mass indicates that a larger fraction of the initial sample remained in the solid phase when the sample was subjected to the faster heating programme. A plausible explanation is the reduced time available for primary and secondary decomposition reactions before the upper temperature of the programme was reached. The result should not, however, be interpreted as direct evidence of an intrinsic increase in char yield. The chemical composition, degree of carbonisation and microstructure of the residual solid were not determined in the present study.

The variability of the TG-derived parameters also differed among the heating rates. The largest scatter was generally observed at $25 \text{ K} \cdot \text{min}^{-1}$, particularly for T_{10} and residual mass, whereas the measurements at $50 \text{ K} \cdot \text{min}^{-1}$ showed comparatively low variability for T_{20} and residual mass. Because only three independent measurements were available for each condition, these differences are described as experimental variability and are not interpreted as statistically significant effects.

The simultaneously recorded DSC curves are shown in Fig. 2. Unlike TG, which directly quantifies changes in sample mass, DSC records the thermal response accompanying the overlapping degradation processes. The DSC profiles changed both in position and magnitude with increasing heating rate.

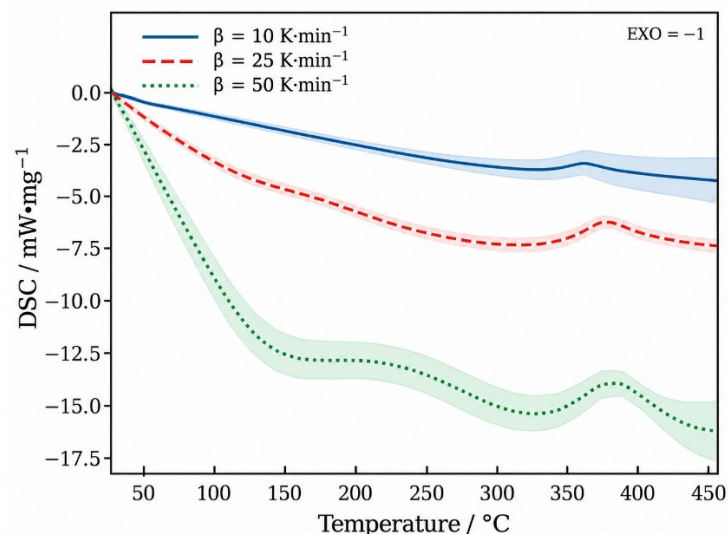


Fig. 2 DSC curves of Norway spruce wood under a nitrogen atmosphere at heating rates of 10, 25 and 50 K·min⁻¹. The curves represent mean values of three independent measurements for each heating rate. Negative heat-flow values indicate the exothermic direction (EXO = -1).

The principal DSC features shifted towards higher temperatures as the heating rate increased, consistent with the displacement observed in the TG and DTG records. This correspondence indicates that the heating programme affected not only the temperature at which a given extent of mass loss was reached but also the temperature range over which the associated thermal processes occurred.

The magnitude of the DSC signal also increased with heating rate. However, direct comparison of absolute DSC amplitudes obtained at different heating rates requires caution. The recorded heat-flow signal depends on the rate at which the sample is driven through the degradation interval and therefore does not, by itself, represent the total heat involved in the process. Since integrated enthalpy values were not evaluated in the present study, differences in DSC amplitude are interpreted only as differences in the recorded heat-flow response.

Within the main degradation interval, the DSC curves contain contributions from several simultaneously occurring processes. Cellulose depolymerisation, degradation of hemicelluloses, gradual lignin decomposition and secondary transformations of the developing carbonaceous residue may contribute to the measured signal. TG/DTG/DSC analysis alone does not permit these individual chemical processes to be identified unequivocally. Consequently, the interpretation of the DSC features is restricted to their experimentally observed heating-rate dependence.

Taken together, the TG, DTG and DSC results demonstrate that heating rate has a substantial influence on the apparent thermal response of Norway spruce wood under otherwise comparable experimental conditions. Increasing the heating rate displaced characteristic TG temperatures and the principal degradation features towards higher temperatures and resulted in a larger residual solid fraction at the end of the temperature programme.

These observations have an important methodological implication. Characteristic temperatures obtained from non-isothermal thermal analysis should always be interpreted together with the heating programme used to obtain them. Direct comparison of T_{10} , T_{20} , T_{50} , DTG features or residual mass between different studies may otherwise lead to misleading conclusions regarding differences in thermal stability.

The present study deliberately focuses on the experimental heating-rate dependence of untreated Norway spruce wood under one inert atmosphere. It does not attempt to derive

kinetic parameters or to extrapolate milligram-scale measurements directly to the behaviour of structural timber during fire exposure. Larger wood specimens are additionally affected by internal temperature gradients, moisture transport, specimen geometry and coupled heat and mass transfer. The results should therefore primarily be regarded as controlled material-scale data for comparing STA measurements carried out at different heating rates.

CONCLUSIONS

This study evaluated the effect of heating rate on the thermal degradation behaviour of Norway spruce wood under an inert nitrogen atmosphere using simultaneous thermal analysis. Increasing the heating rate from 10 to 50 K·min⁻¹ shifted the characteristic mass-loss temperatures towards higher values. The increases in T_{10} , T_{20} and T_{50} were 10.93, 12.47 and 15.07 K, respectively. The largest displacement was therefore observed at 50 % mass loss, demonstrating that the influence of heating rate became more pronounced during the principal degradation interval.

The TG and DTG profiles revealed heating-rate-dependent changes in the position and shape of the principal degradation event, while the DSC curves showed corresponding changes in the thermal response. The residual mass measured at the end of the recorded temperature programme increased from 21.83 % at 10 K·min⁻¹ to 27.21 % at 50 K·min⁻¹. However, differences in the final recorded temperatures limit direct comparison of these values at an identical terminal temperature.

The findings demonstrate that heating rate substantially influences the thermal characteristics obtained from non-isothermal analysis of Norway spruce wood. Characteristic degradation temperatures, DTG profiles and residual mass should therefore be interpreted in relation to the applied heating programme when comparing experimental results.

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