

# A critical assessment of the fire properties of different wood species and bark from small- and bench-scale fire experiments

Juliana Sally Renner<sup>1</sup> · Rhoda Afriyie Mensah<sup>1</sup> · Lin Jiang<sup>1</sup> · Qiang Xu<sup>1</sup>

Received: 31 October 2021 / Accepted: 2 June 2022 / Published online: 1 August 2022 © Akadémiai Kiadó, Budapest, Hungary 2022

#### Abstract

Wood and bark are ubiquitous and obviously the most abundant raw material for various applications including construction. While wood is relatively flammable, the bark is known to have high fire resistance and insulation capabilities due to its high lignin content. This study, therefore, assesses the flammability of different wood species with their bark to ascertain the unique insulation properties. Material characterization methods such as microscale combustion calorimetry method A and B tests, oxygen bomb calorimetry, and thermogravity analysis tests were employed to acquire the fire properties of beech, fir, oak, pine, and spruce with bark and without bark. From the TGA test, oak with bark recorded a significantly lower mass loss rate and had the highest char residue. The MCC experiments showed that beech, oak, and spruce without bark had higher peak heat release rates and heat release capacities compared to the samples with bark. However, the results for fir and pine with bark showed higher heat release properties. In addition, the calorific and heating values were higher at 0% moisture content compared to 8%.

Keywords Wood · Bark · Microscale combustion calorimetry · Thermogravity analysis · Oxygen bomb calorimetry

#### List of symbols

β	Heating rate in MCC test/Ks <sup>-1</sup>
pHRR	Heat release rate/W g <sup>-1</sup>
$\eta_{\rm c}$	Heat release capacity/J $g^{-1} K^{-1}$
THR	Total heat release/KJ g <sup>-1</sup>
pTemp	Temperature at pHRR/°C
MLR	Mass loss rate

# Introduction

Forest products such as timber are readily available and have been used in several applications including construction, energy, etc. Timber consists mainly of wood and bark [1]. Wood has quite a number of desirable properties such as low heat conductivity, small bulk density, relatively high strength, and other good mechanical properties [2]. It is made up of cellulose, hemicellulose, lignin, and some

Lin Jiang jianglin@ustc.edu.cn

Qiang Xu xuqiang@njust.edu.cn

<sup>1</sup> School of Mechanical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China extractives in varying amounts depending on the species [3]. Wood is classified under softwood and hardwood where the hardwood is said to be heavier and denser than softwood but it differs from species to species [4]. Wood is preferred as a construction material because of its economic and environmental benefits as well as its excellent properties. In line with this, wood is used for structural and nonstructural applications such as high-rise buildings, ceilings, beams, decor, and crafts [1]. An equally important material used in building is the bark. It is used for roofing structures because of its ability to withstand harsh weather conditions and protect the wood from insect attacks and diseases [5]. Recently, its usage as composite for panels, boards, etc. is growing tremendously. Some constructions require the use of both wood and bark for instance building trusses, flooring and wall covering, carving, furniture, and arch girders. Additionally, bark has been utilized in products including particleboards and fiberboards, and some small quantities are added in semichemical and kraft pulping processes. Owing to its valuable chemical structure and properties, bark has also been used in adhesives, resins, insulation foams, and other chemical applications because of the amounts of tannins from the bark extractives [6]. However, differences in structure, age, or tissue maturity and chemical constituents of various barks or species, and differences in the properties

of inner and outer bark have hindered an absolute usage of bark. Nevertheless, bark has gained attention more lately in the building and construction industries due to its thermal insulation abilities [7].

Recent development in research has added value to wood with bark. Efforts have been made in the investigation on the use of wood and bark in the construction sector for insulation panels. The bark contains higher lignin content compared to wood which makes it a suitable insulation material. Tsalagkas et al. [8] studied the thermal conductivity of barkbased panels overlaid on the surface of different fiberglass and paper sheet for insulation. It was found that bark-based panels with fiberglass had an improved thermal performance than paper sheet panels. Wood and bark have different chemical reactions in pyrolysis. The bark has a self-heating ability that slows the heat propagation process and therefore serves as an insulator [9].

Despite the aforementioned applications, like polymers, wood and bark are both combustible materials and liable to fire hazards that endanger lives and properties [10]. Although several investigations are ongoing, wood buildings fire outbreaks are constantly growing. US fire department reported an estimate of 1.3 million fires in 2019, causing 3700 deaths and 16,600 injuries and property damage amounting to 14.8 billion dollars [11]. Previous studies have indicated that material flammability properties play a vital role in fires as such it is imperative to study the fire behavior of wood and bark to elucidate the flammability characteristics. Moreover, flammability testing is probably one of the most relevant testing protocols within the construction sector because of its role in assessing the safety implications of fire [12, 13]. Therefore, assessing the fire performance of wood and the bark at the design stage will ensure a safer environment.

Different methodologies have been used to measure the fire performance of materials. Fire experiments are grouped into small, bench, and large scales based on the sample requirements. Examples are thermogravity analysis test (TGA) and microscale combustion calorimetry (MCC). A significant number of researchers have used TGA techniques to study pyrolysis of different wood species. Shama et al. [14] used TGA techniques to measure mass loss of eucalyptus wood to determine the activation energies from iso-conversional methods. The results from the pyrolysis revealed that an increase in the heating rate increased mass loss rate and the decomposition temperature peaks of the wood specimen shifted toward the higher temperature. Again, Roger et al. [15] used TGA, DTA, DSC, and cone calorimetry tests to assess the fire performance of wood. A correlation analysis conducted showed that the results had strong correlations. Although TGA has gained high recognition for studying the thermal decomposition of materials, it only considers thermal analysis and does not take into

account the combustion reactions, and thus, microscale combustion calorimetry test is preferred recently.

Microscale combustion calorimetry (MCC) is convenient to use since it combines both pyrolysis and combustion. There are two standard measurement methods of MCC: method A and method B [16, 17]. In method A, the procedure involves pyrolysis of specimen in an inert gas under a controlled thermal decomposition and complete oxidation of the volatile pyrolysis in a combustor. Considerable studies have utilized this method to measure flammability parameters to evaluate the fire behavior of various materials. However, in method B, the process is typically applied for combustion analysis where combustion parameters including specific combustion rate, combustion temperature, and combustion residue are measured. Following the ASTM D7309-19 [18], controlled thermal oxidation decomposition is performed by decomposing specimen in nitrogen and air mixture before oxidation.

By virtue of the nature of the pyrolysis and combustion process, method A enables char formation while method B allows complete combustion of residue. Subsequently, the total heat of combustion for both gases and char can be obtained from method B. Additionally, in method B, the material can be evaluated by its ability to resist thermal oxidation when subjected to fire [19]. It should, however, be noted that, due to the setup of the MCC apparatus, there were slight differences in the experimental conditions within the combustion chamber. Method A test procedure has less available oxygen for combustion while method B has sufficient oxygen to completely combust all pyrolysis gases since it is exposed to the oxidizing atmosphere. Therefore, it is of great importance to consider both methods to further improve the MCC experimental methodology and to enable a full understanding of material flammability, and check their practicability.

MCC has an advantage over TGA since it uniquely measures the heat release capacity of a material [16–18]. Dahane et al. [19] tested wood bark with bench-scale protocols (horizontal and vertical configuration) to estimate the flammability of the wood sample and its fire spread. They found out that both test methods showed good repeatability and reproducibility. Hostikka and Matala [20] measured the HRR of birch wood using TGA, MCC, and cone calorimetry experiments. MCC was integrated to determine the material's heat of combustion. The birch wood results for MCC showed that the total mass losses are slightly higher than that of TGA and the peak temperature at 20 K min<sup>-1</sup> heating rate is lower than the corresponding values of TGA. In their work, only method A of the MCC test was investigated. Aleksi and Hostikka [21] recently worked on a model for the pyrolysis of two wood samples using small-scale methods, TGA, DSC, and MCC. The results revealed good consistency enabling the determination of heat of pyrolysis and the heat of combustion for each wood constituent. However, only method A was used to conduct their investigation, and this reveals that there are inadequate investigations of method B of MCC pyrolysis which will help ascertain the influence of oxidation in the flammability characteristics of wood.

Oxygen bomb calorimetry is used to measure the calorific value of a material. This gives information on the expected quantity of volatile matter and also the combustion behavior and thermal conversion process [22]. In Ref [23], the author used TGA, MCC, and oxygen bomb calorimetry to test the fire behavior of plants such as pines, junipers, and eucalyptus globulus, and the results correlated well with each method. Therefore, incorporating TGA, MCC, and oxygen bomb calorimetry test will give a comprehensive understanding of the flammability characteristics of wood and the bark. Again, the combination of these methodologies will also serve as a reference tool to predict flammability measurements of bench-scale from small-scale tests [24–26].

In this paper, two pyrolysis modes of MCC (method A and method B), TGA, and oxygen bomb calorimetry methods are used to accurately assess the flammability characteristics from selected timber species samples. This study is useful to understand the complete thermal decomposition as well as the combustion of wood with bark and wood without bark. The results will provide potentially useful flammability measurement information on both pyrolysis and combustibility of wood and bark as building materials. It will also help to obtain more in-depth knowledge on how timber species differ in their fire performance especially pyrolysis through thermal and thermo-oxidative modes.

# **Materials and methods**

## **Materials**

Timber materials were selected from five species: beech, fir, oak, pine, and spruce. These species have been used widely as a choice of building materials for good designs and maintenance in the construction sector over the years. The samples selected were obtained from a Developmental Workshop of the Technical University in Zvolen, Slovakia. The samples were milled to get the wood dust for the experiment. To obtain the required sample fraction size of 0.355–0.5 µm (50% bark mass fraction) for further analysis, the sieve analysis using the analytical sieve machine AS 200 Basic was used. The samples were dried with a Memmert UFB 500 Basic to 0% moisture content for further analysis. To determine the calorific value of the sample, the bomb calorimeter IKAC 5000 control was used, and STN ISO 1928: 2003-07 solid fuels were applied. The labels for the samples and their harvesting conditions are listed in Table 1.

#### **Characterization methods**

#### Thermogravity analysis

TGA is an experiment used to determine the mass loss of a material with respect to temperature and time. Thermogravimetry test instruments can be used to evaluate the thermal characteristics of a timber material under a controlled heating environment [24]. TGA runs were performed on wood dust samples with mass 2.4 to 5 mg under an atmosphere of air and nitrogen in the temperature range from 25 to 800 °C at a flow rate of 60 °C min<sup>-1</sup> using Netzsech STA 7300 channel. The weighed specimen was placed in a sample holder and heated up to 800 °C with a heating rate of

#### Table 1 Sample description

Sample	Label	Locality	Stand nb	Stand age	Altitude	Timber harvesting time
BB	Beech with bark	Hakovo	_		_	_
BW	Beech without bark					
FB	Fir with bark	Kremenny Jarok (Linai)	_		-	Winter
FW	Fir without bark					
OB	Oak with bark	Kremenny Jarok (Linai)	607	110	320 m above sea level	Summer
OW	Oak without bark					
PB	Pine with bark	Kremenny Jarok (Linaj)	607	110	320 m above sea level	Summer
PW	Pine without bark					
SB	Spruce with bark	Hakovo	220	120	800 m above sea level	Summer
SW	Spruce without bark					

10 K s<sup>-1</sup>. Air or nitrogen was supplied into the chamber to simulate a thermo-oxidative decomposition or pyrolysis environment. The nitrogen and air flow rates were Gas1:  $N_2$  100 mL min<sup>-1</sup> and Gas2:  $O_2$  100 mL min<sup>-1</sup>. The samples were labeled as, e.g., BB-Air, BB-Nitrogen, BW-Air, BW-Nitrogen, denoting beech wood with bark in air and beech with bark in nitrogen, beech without bark in air and beech without bark in a nitrogen environment, and so on. The samples were tested in duplicate and triplicates to ensure repeatability of data.

#### Microscale combustion calorimetry

The MCC experiments were conducted according to the standard and guidelines in ASTM D7309-19 [25]. Wood and bark dust samples were tested in MCC-3 equipment from Govmark Limited at Nanjing University of Science and Technology Laboratory in Jiangsu, China. The sample size ranged from 1 to 2 mg with standard error of 0.042 mg and 0.053 mg, respectively. The pyrolysis temperature was between 0 and 600 °C while the combustor temperature was set at 900 °C. Oxygen (O<sub>2</sub>) and nitrogen (N<sub>2</sub>) flow rates were also set at 20 and 80 cm<sup>3</sup> min<sup>-1</sup>. The samples were heated at thirteen selected heating rates ( $\beta$ ) ranging from 0.1 to 5.5 K s<sup>-1</sup> under controlled conditions. All tests conducted followed MCC method A and method B procedure.

Method A procedure The test sample undergoes a controlled thermal decomposition when subjected to controlled heating in an anaerobic environment. Combustion products including water, carbon dioxide, and acids are removed from the oxygen and nitrogen mixture. The gases released by the sample during the operation are swept from the sample chamber by nitrogen, and then excess oxygen is added for complete oxidation in a high-temperature combustion furnace [23, 25]. The volumetric flow rate and the volumetric oxygen concentration of the gas stream exiting the combustion furnace are continuously measured during the test to estimate the heat release rate by means of oxygen consumption [27, 28]. The samples were tested in triplicates, and an average of the measured results was recorded. The samples for method A were labeled as BB-0.1 K-A01, BW-0.1 K-A01 representing the first specimen tested under 0.1 K s<sup>-1</sup> for beech with bark, beech without bark, and so forth. The following were measured and recorded from the test, and thus the heat release rate which is the maximum value of specific heat release rate was recorded during the test. Heat release temperature is the sample temperature at which the specific heat release rate is maximum during a controlled thermal decomposition, and heat release capacity was calculated by the maximum heat release rate during a controlled thermal decomposition divided by the heating rate during the experiment and time to heat release measured, respectively.

Method B procedure The sample is thermally decomposed in a mixture of oxygen and nitrogen under controlled conditions (approximately 20 and 80 cm<sup>3</sup> min<sup>-1</sup> nitrogen and oxygen flow) prior to complete oxidation of the discharge in the combustion chamber. The sample gases evolved during the controlled heating conditions are swept from the sample chamber by the oxidizing purge gas. Excess oxygen was added to the volatile gases entering the combustor to ensure complete combustion. The volumetric flow rate and volumetric oxygen concentration of the gas stream exiting the combustion furnace are constantly measured during the experiment to estimate the heat release rate by means of oxygen consumption. In method B, total heat release (THR), heat release capacity (HRC), and peak heat release rate (pHRR) which is the maximum value recorded for the combustion rate during the test were measured. Combustion temperature (pT) which is the sample temperature at a specific combustion rate was recorded during controlled thermal oxidative decomposition per unit initial sample mass at thirteen different heating rates. The samples for method B were labeled BB-0.1 K-B01, BW-0.1 K-B01 indicating the first sample tested under a heating rate of 0.1 Ks-1 for beech with bark (BB), beech without bark (BW), and so on.

#### Oxygen bomb calorimeter

An oxygen bomb calorimeter with model C 5000 (Adiabatic Calorimeter, IKA) from Germany was used to measure the calorific value of the different timber species. The concept is the most prevalent technique for measuring calorific value both in the laboratory and in an industrial environment [29]. It is a constant volume-type calorimeter that measures the heat of a particular reaction or measures the calorific value of the fuels. The bomb calorimeter is designed as a built-in instrument where it can withstand large pressures produced within the calorimeter due to the reaction or burning of fuels. The electrical energy is used as an ignition source for the burning of testing fuels and the heating filament which consists of tungsten materials. The measured samples were positioned in the crucible and electrically ignited to burn with pressure in a pure oxygen environment. During the combustion, the rate of heat released and the elevated temperatures were measured and recorded.

# **Results and discussion**

## **TGA results**

The results from the TGA experiment were all obtained using a constant heating rate of 10 K s<sup>-1</sup>. The experiment duration depended on the thermal stability of the wood samples. The thermal degradation behavior of the wood samples from TGA results is shown in Table 2. The mass loss rate fraction (%) of the initial sample  $(m/m_0)$  is displayed as a function of temperature. For wood pyrolysis, generally, thermal degradation occurs in the three main components of wood hemicellulose, cellulose, and lignin [30, 31]. Hemicellulose decomposes from the shoulder region with a mass loss rate at a temperature range of 161 °C to 196.3 °C in an oxygen environment while in nitrogen, it occurs at the temperature range of 158.7 °C to 212 °C. Cellulose also decomposes at the peak of the curve with the mass loss rate and the temperature ranges of 306.4 °C to 409 °C (air) and 333.7 °C to 419 °C in a nitrogen environment. This was followed by a rapid decay. Lignin decomposition occurs at a wide range of temperatures at 592.9 °C and 799.6 °C (air), 714.3 °C, and 799 °C (nitrogen); BW recorded the lowest temperature 592.9 °C in this stage. With the thermal decomposition experiment, the initial decomposition occurred at a higher temperature, thus, ca. 212 °C than in thermal oxidative environment, ca. 196.3 °C for wood samples. However, some wood decomposition occurred at slightly lower temperatures in the air than in the nitrogen atmosphere owing to their cellular structures. Wood samples FB, SB, and SW did not form an appreciable char during the decomposition stage in an oxygen environment with FB obtaining the lowest value of 0.2% peak MLR with increased temperature. The mass of the sample decreases due to volatile fraction released and wood material oxidation while PB was observed to record the highest peak MLR of 26.3%. Oak with bark (OB) measured a significant highest value of 19.6% peak MLR in the nitrogen gas test. TGA curves for oxygen and nitrogen are shown in Figs. 1 and 2 which describe the three regions identified for wood sample BB, BW, FB, FW, OB, OW, PB, PW, SB, and SW for the thermal decomposition process. During the first stage for example, with a temperature range of 27.2°C to 177.6 °C (BB-Air) and 36.8 °C to 212 °C (BW-Nitrogen), the initial mass loss rate was due to water evaporation, moisture loss or dehydration, and some light volatile compounds. The second stage is where the major thermal decomposition occurs corresponding to the temperature range from 409 to 451.2 °C, SB-Air with the lowest temperature of 409 °C. FB-Air was observed to record the highest temperature. This is associated with a rapid loss of wood sample mass induced by the intensive release of volatile compounds responsible for the combustion of the samples. The final stage peak temperature recorded is about 800 °C responsible for lignin decomposition with different mass loss rates which shows that wood species have different lignin content as confirmed in the literature [32]. We could see from Figs. 1 and 2 that overall high degradation occurred in air than in nitrogen atmosphere owing to oxidation [33].



Fig. 1 Thermal decomposition curves of wood samples in air with heating rate of 10 K s.<sup>-1</sup>. Different colors mean different wood samples with bark and without bark

Sample	Temperatu	Temperature/°C			Temperatu	MLR %		
	Oxygen				Nitrogen			
	1st stage	2nd stage	3rd stage	Final	1st stage	2nd stage	3rd stage	Final
BB	177.6	306.4	799	1.2	193	333.7	798	15.6
BW	161	310.5	592.9	23	212	338.2	714.3	19.2
FB	185.7	316.3	798	0.2	198	342.2	717	18.5
FW	177.9	308	799	1.2	178.5	332.8	798.7	16.5
OB	178.2	318.7	797	9.8	206.8	324.4	717	19.6
OW	175.6	319.2	799	5.1	175.9	336.2	797	16.0
PB	178.7	434.6	799.6	26.3	180.8	343.1	799	10.3
PW	175.7	328.9	798	1.0	158.7	343	798	14.3
SB	178.5	409	798	0.17	171.7	340.6	798	13.7
SW	196.3	327.3	798	0.9	162.5	348.9	799	12.6

Table 2	Results for O <sub>2</sub> and N <sub>2</sub>
experim	ents for wood samples



**Fig.2** Thermal oxidative decomposition curves of wood samples in nitrogen environment with a heating rate of 10 K s.<sup>-1</sup>. Different colors mean different wood samples with bark and without bark

# **MCC** results

MCC flammability parameters, thus the peak heat release rate (pHRR), heat release capacity (HRC), peak temperature at pHRR (pTemp), total heat release (THR), and char yield or pyrolysis residue ( $Y_p$ ), were used to determine the flammability of the wood and bark in this section. Aside pHRR, the other parameters are calculated from the basic measurements according to method A and method B procedures in the MCC standard (ASTM D7309-19) [23]. HRC explains the thermal stability of a material and shows the average heat release in the combustion stage per the temperature [25, 33].

Observations are discussed and analyzed from the two pyrolysis modes of MCC. Generally, the flammability of a material is characterized by the quantity of heat released when the material is subjected to fire. Therefore, the most relevant characteristic that are being measured from the MCC experiment is heat release rate (HRR) [34], and thus it gives information on HRR as a function of temperature or time as well as the heat of combustion [35]. Specific HRR at 13 heating rates was plotted against their corresponding temperatures for wood samples (BB, BW, FB, FW, OB, OW, PB, PW, SB, and SW) in Fig. 3 for both method A and method B.

It can be seen in all the figures highlighted that the peak of the curves indicating the pHRR and their respective temperatures increases with increasing heating rates. Furthermore, method A showed a single peak while method B showed a complex behavior of shoulder peaks resulting from excess oxygen added for complete oxidation. On the other hand, it was observed that wood samples without bark achieved a higher heat release rate than the wood sample with bark at the same heating rate in method A. Generally, one of the main differences between the chemical composition of wood and bark is the amounts of inorganic ions, where the mineral matter content is significantly higher in the bark than in wood [36]. As a result of the differences in relation to the amount of hemicellulose, cellulose, lignin, and some extractives, wood and bark are expected to behave differently during thermal decomposition.

It is well known that the bark contains a high quantity of extractives consisting of phenolics, waxes, flavonoids, resins, etc., and high lignin amounting to 50% to 70%, while wood is composed of mainly cellulose about 40% to 45%. Thermal degradation usually starts from hemicellulose, cellulose, and lignin. During pyrolysis, cellulose breaks down into smaller sugar units through a degradation reaction. These compounds serve as a protective layer and thus prevent heat transfer and delay temperature rise, restraining the effect of heat and mass diffusion during combustion [34]. It could also be observed that wood and bark flammability vary across species and this could be ascribed to the differences in the cellulose and lignin content. It was also noted that high heating rates, production of volatiles, and lower level of oxygen available for combustion in the combustion chamber affect the pHRR. The drop of the temperature was due to insufficient oxygen to completely combust all pyrolysis gases, therefore adding oxygen facilitated the reaction to complete the combustion of volatiles in the combustion chamber. The results from MCC data for testing 1 mg of wood and bark samples are presented in Table S1 to Table S10 (supplementary).

From Table 3, beech without bark (BW) from method A recorded the highest average peak HRR of 357.05 Wg<sup>-1</sup> and the highest HRC was 148.09 J g<sup>-1</sup> K<sup>-1</sup> at a peak temperature of 395.43 °C, while the lowest pHRR and HRC observed was oak wood with bark (OB) which measured 276.47 Wg<sup>-1</sup> and 119.65 J g<sup>-1</sup> K<sup>-1</sup> at a peak temperature of 380.54 °C. According to the results, these materials, fir with bark and beech without bark (FB and BW), had the highest THR, 11.98 kJ g<sup>-1</sup>, and the beech with bark (BB) sample showed the lowest HRC value of 8.92 J g<sup>-1</sup> K<sup>-1</sup>. Nevertheless, some samples measured insignificant values of heat release rate and heat release capacity at low heating rates (0.1 K s<sup>-1</sup> to 0.5 K s<sup>-1</sup>) and this may be associated with noise signals in the measurements, etc., during the test, and therefore, they were not considered in this study.

Under method B, the highest pHRR recorded was spruce without bark (SW) with an average value of 438.85 Wg<sup>-1</sup>, HRC of 197.48 J g<sup>-1</sup> K<sup>-1</sup>, and THR 15.97 kJ g<sup>-1</sup> at a peak temperature of 383.85 °C. Again, the lowest pHRR was from BW which recorded 273.74 W g<sup>-1</sup> at a peak temperature of 370.64 °C. Comparing both pyrolysis modes, it could be seen that the average pHRR for method B was higher than that of method A as well as the THR values. This may be associated with the differences in their pyrolysis procedures. After the test, the char yields were insignificant for all the



**Fig. 3** Plots of HRR versus temperature with 13 heating rates ranging from 0.1to 5.5 K s.<sup>-1</sup> by MCC methods A and B: **A** BB-MA, beech with bark method A; **b** BB-MB, beech with bark method B; **c** BW-MA; beech without bark method A; **d** BW-MB, beech without bark method B; **g** FB-MA, fir with bark method A; **f** FB-MB, fir with bark method A; **h** FW-MB, fir without bark method A; **i** OB-MA, oak with bark method A; **j** OB-MB, oak with bark method B; **k** OW-MA, oak without bark

method A; **l** OW-MB: oak without bark method B; **m** PB-MA, pine with bark method A; **n** PB-MB, pine with bark method B; **o** PW-MA, pine without bark method A; **p** PW-MB, pine without bark method B; **q** SB-MA, spruce with bark method A; **r** SB-MB, spruce with bark method B; **s** SW-MA, spruce without bark method A; **t** SW-MB, spruce without bark method B. Different colors denote different heating rates



Fig. 3 (continued)

 Table 3
 Calculated average results of MCC test of wood samples for wood with bark and without bark

Sample label	Method A				Method B			
	pHRR/Wg <sup>-1</sup>	HRC/J $g^{-1} K^{-1}$	THR/KJ g <sup>-1</sup>	pTemp/°C	pHRR/Wg <sup>-1</sup>	HRC/J $g^{-1} K^{-1}$	THR/KJ g <sup>-1</sup>	pTemp/°C
BB	321.72	134.82	8.92	396.25	282.29	114.75	11.51	367.00
BW	357.05	148.09	11.98	395.43	273.74	113.89	13.10	370.64
FB	328.87	138.51	11.98	395.43	301.82	124.53	13.26	370.64
FW	325.65	128.66	9.06	391.54	275.90	110.75	12.96	370.23
OB	276.47	119.65	10.16	380.54	333.54	154.44	12.97	367.23
OW	317.79	132.94	9.67	400.44	361.93	163.85	12.05	376.98
PB	327.37	144.55	11.19	409.53	377.64	167.81	14.00	378.27
PW	303.79	125.22	11.28	411.29	367.58	155.45	12.92	384.70
SB	309.89	126.17	9.56	397.33	356.73	147.49	12.47	371.98

wood samples, except for spruce with bark (SB) in method A which recorded 0.18% residual mass.

As shown in Fig. 3a–t, pHRR curves against temperature for SW sample showed the highest pHRR indicated as 820.08 Wg<sup>-1</sup> for method B. Nevertheless, it was also observed that method B pHRR against temperature curves showed significant higher peaks than method A except FW, BW, and BB samples where FW was the lowest with peak HRR of 508.06 Wg<sup>-1</sup> at a peak temperature of 395.93 °C (see Fig. 3b, d, and h).

## Oxygen bomb calorimetry analysis

The calorific values of wood samples were grouped into two: wood with bark and without bark of five different species were determined using the oxygen bomb calorimeter. The calorific value is the most important characteristic of fuel used to quantify the heat released during the complete combustion of a material [29]. The net calorific values were measured by the oxygen bomb calorimeter at 0% and 8% moisture content ( $H_o$  (roh) and  $H_o$  (an) kJ kg<sup>-1</sup>). The heating values which measure the amount of heat released during combustion of fuel under controlled measured temperature were obtained. Moisture contents of 0% and 8% were used to determine the calorific values of the samples to obtain their various energy released during the experiment.

Thus, from Table 6, determination of wood with bark sample was observed under  $H_0$  (roh) at 8% and  $H_0$  (an) at 0%, and the average values of 18,362 kJ kg<sup>-1</sup> and 19,903.3 kJ kg<sup>-1</sup> were observed for FB sample as the highest calorific value or the heating value while BB recorded the lowest value of 16,179 kJ kg<sup>-1</sup> and 17,615 kJ kg<sup>-1</sup> at the same moisture content. Observations for wood without bark were that FW had the highest values  $H_0$ ,  $H_1$  in kJ kg<sup>-1</sup> and BW and the lowest values  $H_0$ ,  $H_1$  in kJ kg<sup>-1</sup> for the same moisture content (see Table 6). Therefore, firwood with bark (FB) and without bark (FW) recorded a significant highest value while beech wood with bark (BB) and without bark (BW) measured the lowest for calorific values or heating values. This may be due to differences in the extent of heat released or energy released during combustion and the differences in their chemical structure. Also, the specie factor has a strong influence on the calorific value variation though the same value of moisture content may be used. It could be observed from the tables that the moisture content affects the heating value or the calorific value. It is a fact that with the decrease in moisture content, the heating value of the fuel is high. Thus, the lower heating value of fuel increases with an increase in hydrogen content. Nevertheless, higher moisture content requires high energy to evaporate the water. Hence, vaporization of water requires energy from the burning process, consequently, a decrease in the heating value of the fuel.

A set of results from Tables 4 and 5 is provided for each sample tested. Table 4 gives a set of detailed calorimetric data for each experiment carried out for wood sample with bark, Table 5 represents wood sample without bark, and Table 6 shows average calorific values or heating values with moisture contents 0% and 8%.

 Table 4
 Sample measurements description for timber species with bark

	BB	FB	OB	PB	SB
Pan mass before	9.8781	9.8315	9.7780	10.4064	10.1165
test (g)	9.9243	9.9422	10.4087	9.7760	10.4097
	9.8540	9.126	9.7765	9.7757	9.8416
					9.8322
Bag mass (g)	0.1202	0.1201	0.1144	0.1022	0.1165
	0.1223	0.1213	0.1240	0.1073	0.1217
	0.1204	0.1204	0.1208	0.1121	0.1122
					0.1140
Sample (g)	0.9241	0.9332	0.8252	0.8107	0.8190
	0.9635	0.9520	0.8237	0.8141	0.8245
	0.9223	0.9294		0.8162	0.8443
					0.8375
H <sub>2</sub> value	6.05	6.1	6.0	6.2	6.1
Moisture content (%)	8%	8%	8%	8%	8%
Heat (J)	17.643	19.895	18.558	19.201	19.476
	17.771	19.813	18.210	19.176	19.179
	17.431	20.002	18.536	19.324	18.877
					19.641
$H_0  ({\rm roh})$ at $8\%$	16.149	18.354	17.073	17.656	17.918
	16.381	18.269	16.754	17.598	17.645
	16.007	18.463	17.053	17.869	17.366
					18.069
$H_0$ (an) at 0%	17.643	19.895	18.558	18.987	19.476
	17.771	19.813	18.210	19.141	19.179
	17.431	20.002	18.536	19.344	18.877
					19.641
$H_{\rm u}$ (roh) at 8%	14.751	16.934	15.674	16.345	16.499
	14.987	16.848	15.354	16.201	16.225
	14.608	17.041	15.654	16.603	15.947
					16.650
$H_{\rm u}$ (an) at 0%	16.335	18.579	17.249	17.622	18.146
	15.071	18.457	16.902	17.891	17.848
	16.122	18.701	17.227	18.226	17.546
					18.310
Pan mass after	9.9021	9.8463	9.7787	9.9982	10.4113
test (g)	10.0023	9.9463	10.4177	9.8592	10.4114
	9.8576	9.9286	9.7783	9.8201	9.8385
					9.8325

BW FW OW PW S Pan mass before 9.9943 9.921 10.4083 10.2565 9 test (g) 9.8062 9.9036 9.7768 9.9630 ç 9.8922 9.8298 9.8724 9 9.8903 Ç Bag mass (g) 0.1206 0.1213 0.1320 0.1134 0 0.1277 0.1205 0.1220 0.1085 ( 0.1193 0.0950 0.1013 0.1114 ( 0.9021 0.9564 0.8171 0.8211 Sample (g) 0 0.8974 0.9142 0.8347 0.8136 0 0.8692 0.9095 0.8236 0.8121 ( ſ 6.1 6.2 H<sub>2</sub> value 6.1 6.1 6 Moisture content (%) 8% 8% 8% 8% 8% Heat (J) 17.621 19.654 19.337 18.962 18.654 17.487 19.574 19.113 18.841 18.655 17.501 19.760 19.156 19.036 19.307 18.321  $H_0$  (roh) at 8% 16.082 18.116 17.790 17.436 17.162 17.584 15.942 18.030 17.524 17.162 15.963 18.221 17.624 17.444 17.763 16.856  $H_0$  (an) at 0% 17.621 19.654 19.337 18.962 18.654 17.487 19.574 19.113 18.841 18.655 17.501 19.760 19.156 19.036 19.307 18.321 16.040 15.722  $H_{\rm u}$  (roh) at 8% 14.661 16.693 16.371 14.521 16.610 16.165 16.231 15.723 14.542 16.801 16.204 16.002 16.323 15.416 18.329 18.007  $H_{\rm u}$  (an) at 0% 16.296 17.643 17.302 16.122 18.209 17.782 17.800 17.302 18.465 17.826 17.403 17.955 16.206 16.969 Pan mass after 10.0124 10.0107 10.4098 10.2131 9.8419 test (g) 9.8823 9.9164 9.7776 9.8426 9.7843 9.9232 9.8352 9.8956 9.8412 9.8957 9.7798

 $\left. \begin{array}{c} H_{\rm u}({\rm roh}) \\ H_{\rm u}({\rm an}) \end{array} \right\}$ 

roh-calorific value or heating value at self-defined sample

moisture, i.e., 8% in this case; an-calorific value or heating

heating value

 Table 5
 Sample measurements description for timber species without bark

 $H_0(\text{roh})$ 

 $H_0(an)$ 

calorific value

value at 0% sample moisture.

**Table 6** Mean calorific value/heating values of samples measured at0% and 8% of timber species with bark and without bark

Sample label	Ave. $H_{\rm o}({\rm roh})$ 8% kJ kg <sup>-1</sup>	Ave. $H_0(an)$ 0% kJ kg <sup>-1</sup>	Ave. $H_{\rm u}({\rm roh})$ 8% kJ kg <sup>-1</sup>	Ave. $H_{\rm u}({\rm an}) 0\%$ kJ kg <sup>-1</sup>
BB	16,179		14,782	15,842.7
BW	15,995.7	17,536.3	14,574.7	16,208
FB	18,362	19,903.3	16,941	18,579
FW	18,122.3	19,662.7	16,701.3	18,334.3
OB	16,960	18,434.7	15,560.7	17,126
OW	17,666	19,202	16,246.7	17,871.7
PB	17,707.7	19,157.3	16,383	17,913
PW	17,468	18,946.3	16,091	17,615.3
SB	17,749.5	19,293.3	16,330.3	17,962.5
SW	17.235.8	18,734.3	15,796	17,382

# Conclusions

Flammability experiments are conducted to determine the fire behavior of materials. In this study, mass loss, heat release properties, and heating values of five wood species with and without barks were obtained. The results from the thermogravimetry analysis of the wood samples in air environments were slightly lower than samples tested in nitrogen gas at the initial decomposition stages. The thermal degradation of the wood species was found to occur in three stages, and thus the hemicellulose decomposes at the temperature range from 161 °C to 196 °C, cellulose (332.8 °C and 348.9 °C), and lignin degradation (717 °C and 799 °C) in air and nitrogen experiments. Beech without bark (BW) had the lowest temperature of 592.9 °C at the final stage of decomposition in oxygen gas while pine with bark (PB) showed the highest temperature in air, 799.6 °C, and nitrogen gas at 799 °C. Spruce without bark (SW) was also observed to have the highest temperature at 196.3 °C in air. The highest mass loss rate thus, 26.3%, was recorded by PB during the thermal decomposition process while fir with bark had the lowest mass loss rate of 0.2% in an air environment. The thermal oxidation process was observed to have a low mass loss rate compared to thermal degradation although BW and PB recorded the highest mass loss rate ranging from 23 to 26.3%. Additionally, oak with bark recorded a significantly lower mass loss rate and had the highest char residue. This reveals that different species decomposes during pyrolysis at a different temperature at the same heating rate. The thermal stability of the lignin content of wood with bark samples was considered greater than that of hemicellulose but lesser than that of cellulose in this case. From the thermogravimetry analysis, it could be seen that high degradation occurred in air than in the nitrogen atmosphere owing to oxidation.

From the MCC experiments, method B showed a significantly higher peak HRR and THR. It should, however, be noted that method B can be utilized to assess the reaction of the wood species to oxidative degradation. Method A recorded lower pHRR, HRC, and THR compared to method B. Higher peaks of HRR in method B were due to the addition of oxygen which combusted the samples completely, or the differences in the pyrolysis procedure. Overall, oak with bark (OB) measured the lowest pHRR, with an average value of 276.47 Wg<sup>-1</sup> at a peak temperature of 380.54 °C. Materials with low HRR have better flammability characteristics. Insufficient char yields were observed for the test samples except spruce with bark (SB) method A. Beech, oak, and spruce without bark had higher peak heat release rates and heat release capacities compared to the samples with bark. However, the results for fir and pine with bark showed higher heat release properties.

Calorific values and heating values were measured by an oxygen bomb calorimetry test. Beech without bark (BW) recorded the lowest calorific value of 15,995.7 kJ kg<sup>-1</sup> at 8% moisture content and the lowest heating value of 14,574.7 kJ kg<sup>-1</sup> at 8% moisture content. Beech with bark (BB) recorded the lowest average calorific value of 17,536.3 kJ kg<sup>-1</sup> at 0% moisture content and the lowest average heating value of 15,842.7 kJ kg<sup>-1</sup> at 0% moisture content. The high calorific value or heating value depicts the high degree of energy released during combustion. The moisture content influences the heating value or the calorific value. Higher moisture content requires high energy to evaporate the water for the burning process, thus a decrease in the heating value of the wood samples.

The lignin content of wood species with bark highly influences the pyrolysis processes slowing down decomposition, even though bark degradation rates are variable across species. Hence, oak with bark had dominantly demonstrated to possess better flammability properties compared to the other wood samples with bark. In general, wood bark is a good insulator and thus delays the burning of wood exhibiting very low flammability.

Supplementary Information The online version contains supplementary material available at https://doi.org/10.1007/s10973-022-11443-z.

Acknowledgements The authors would like to thank National Key Research and Development (R&D) Plan of China under Grant No. 2020YFC1522800, the National Natural Science Foundation of China (NSFC, Grant 52111530091), and the International Science and Technology Cooperation Base at ministerial level.

# References

 Ramage M, Burridge H, Busse-Wicher M, Fereday G, Reynolds T, Shah D, Wu G, et al. The wood from the trees: the use of timber in construction. Renew Sustain Energy Rev. 2017;68(Part 1):333–59. https://doi.org/10.1016/j.rser.2016.09.107.

- Ross RJ. Wood handbook—Wood as an engineering material. General Technical Report FPL-GTR-190. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory. p. 508
- Lachowicz H, Wróblewska H, Wojtan R, Sajdak M. The effect of tree age on the chemical composition of the wood of silver birch (Betula pendula Roth.) in Poland. Wood Sci Technol. 2019;53(5):1135–55. https://doi.org/10.1007/ s00226-019-01121-z.
- Hoadley RB. Understanding wood: a craftsman's guide to wood technology. Newtown: Taunton Press; 2000.
- Kain G, Barbu MC, Hinterreiter S, Richter K, Petutschnigg A. Using bark as a heat insulation material. BioResources. 2013;8(3):3718–31.
- Jablonsky M, Nosalova J, Sladkova A, et al. Valorisation of softwood bark through extraction of utilizable chemicals. A Review. Biotechnol Adv. 2017;35:726–50. https://doi.org/10.1016/j.biote chadv.2017.07.007.
- De Araujo VA, Biazzon JC, Cortez-Barbosa J, Morales EA, Gava M, Garcia JN. Timber housing production systems in Brazil. Bull Transilv Univ Brasov. 2020;13(62):69–80. https://doi.org/10. 31926/but.fwiafe.2020.13.62.1.6.
- Tsalagkas D, Börcsök Z, Pásztory Z. Thermal, physical and mechanical properties of surface overlaid bark-based insulation panels. Eur J Wood Wood Prod. 2019;77(5):721–30. https://doi. org/10.1007/s00107-019-01436-5.
- Gupta M, Yang J, Roy C. Specific heat and thermal conductivity of softwood bark and softwood char particles. Fuel. 2003;82(8):919– 27. https://doi.org/10.1016/S0016-2361(02)00398-8.
- Lowden LA, Hull TR. Flammability behaviour of wood and a review of the methods for its reduction. Fire Sci Rev. 2013;2:4. https://doi.org/10.1186/2193-0414-2-4.
- 11. Ahrens M, Evarts B. Fire loss in the United States during 2019. Quincy, MA: National Fire Protection Association; 2019.
- Kashiwagi T, Du F, Winey KI, Groth KM, Shields JR, Bellayer SP, Kim H, Douglas JF. Flammability properties of polymer nanocomposites with single-walled carbon nanotubes: effects of nanotube dispersion and concentration. Polymer. 2005;46(2):471–81.
- Xu Q, Jin C, Jiang Y. Compare the flammability of two extruded polystyrene foams with micro-scale combustion calorimeter and cone calorimeter tests. J Therm Anal Calorim. 2017;127:2359–66. https://doi.org/10.1007/s10973-016-5754-6.
- Sharma P, Diwan PK. Study of thermal decomposition process and the reaction mechanism of the eucalyptus wood. Wood Sci Technol. 2017;51:1081–94. https://doi.org/10.1007/s00226-017-0924-7.
- Rowell RM, et al. Cell wall chemistry. In: Rowell R, editor., et al., Handbook of wood chemistry and wood composites. 2nd ed. Boca Raton, FL: CRC Press; 2013. p. 33–72.
- Mensah RA, Xu Q, Asante-Okyere S, Jin C, Bentum-Micah G. Correlation analysis of cone calorimetry and microscale combustion calorimetry experiments. J Therm Anal Calorim. 2019;136(2):589–99. https://doi.org/10.1007/s10973-018-7661-5.
- Snegirev AY. Generalized approach to model pyrolysis of flammable materials. Thermochim Acta. 2014;590:242–50. https:// doi.org/10.1016/j.tca.2014.07.009.
- 18. Babrauskas V, Peacock RD. Heat release rate: the single most important variable in fire hazard. Fire Saf J. 1992;18:255–72.
- Dehane B, Madrigal J, Hernando C, Bouhraoua R, Guijarro M. New bench-scale protocols for characterizing bark flammability and fire resistance in trees: application to Algerian cork. J Fire Sci. 2015;33(3):202–17. https://doi.org/10.1177/0734904114568858.
- Hostikka S, Matala A. Pyrolysis model for predicting the heat release rate of birch wood. Combust Sci Technol. 2017;189(8):1373–93. https://doi.org/10.1080/00102202.2017. 1295959.

- Rinta-Paavola A, Hostikka S. A model for the pyrolysis of two Nordic structural timbers. Fire Mater. 2021. https://doi.org/10. 1002/fam.2947.
- 22. ASTM E872-82. (2013). Standard test method for volatile matter in the analysis of particulate wood fuels. *Annual Book of ASTM Standard, American Society for Testing and Materials*
- 23. Robert H, White A, Wayne C, Zipperer BC. Testing and classification of individual plants for fire behaviour: plant selection for the wildland–urban interface. Int J Wildland Fire. 2007;19(2):213–27. https://doi.org/10.1071/WF07128.
- 24. Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry. ASTM D7309–13. 2013.
- Lyon RE, Walters RN. Pyrolysis combustion flow calorimetry. J Anal Appl Pyrolysis. 2004;71(1):27–46.
- Mensah RA, Jiang L, Asante-Okyere S, Qiang X, Jin C. Kinetic Parameter estimation from thermogravimetry and microscale combustion calorimetry. Int J Mech Mechatron Eng. 2020;14(2):66–75.
- Xu Q, Jin C, Mailingova A, et al. Discuss the heat release capacity of polymer derived from microscale combustion calorimeter. J Therm Anal Calorim. 2018;133:649–57. https://doi.org/10.1007/ s10973-017-6866-3.
- Wilkens Flecknoe-Brown K, van Hees P. Sensitivity analysis on the microscale combustion calorimeter for polyurethane foam using a full factorial design methodology. J Fire Sci. 2018;36(6):453–71. https://doi.org/10.1177/0734904118798603.
- 29. Gravalos I, Xyradakis P, Kateris D, Gialamas T, Bartzialis D, Giannoulis K. An experimental determination of gross calorific

value of different agroforestry species and bio-based industry residues. Nat Resour. 2016;7:57–68. https://doi.org/10.4236/nr.2016. 71006.

- Nassar MM, MacKay GDM. Mechanism of thermal decomposition of lignin. Wood Fiber Sci. 1984;16(3):441–53.
- Domansky R, Rendos F. On the pyrolysis of wood and its components. Holz Roh-Werkst. 1962;20:473–6.
- Börcsök Z, Pásztory Z. The role of lignin in wood working processes using elevated temperatures: an abbreviated literature survey. Eur J Wood Wood Prod. 2021;79(3):511–26. https://doi.org/ 10.1007/s00107-020-01637-3.
- Zhuge J, Chen X, KS A, Manica DP. Microscale combustion calorimeter—application and limitation. Fire Mater. 2016;40(8):987– 98. https://doi.org/10.1002/fam.2358.
- Walters RN, Lyon RE. Microscale combustion calorimeter for determining flammability parameters of materials. Evol Technol Compet Edge. 1997;42:1335–44.
- 35. Tran HC, White RH. Burning rate of solid wood measured in a heat release rate calorimeter. Fire Mater. 1992;16(4):197–206.
- 36. Chow S-Z, Pickles KJ. Thermal softening and degradation of wood and bark. Wood fiber Sci. 1971;3(3):166–78.

**Publisher's Note** Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.