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Thermogravimetric study of high temperature treatment of aspen: effect of treatment parameters on weight loss and mechanical properties

D. Kocaefe · B. Chaudhry · S. Poncsak · M. Bouazara · A. Pichette

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Abstract Aspen was heat-treated at high temperature in a laboratory thermogravimetric analyzer. The high temperature wood treatment technology is new in North America and needs to be adapted to the North American species. The objective of this study is to understand how the treatment affects the mechanical properties of aspen such as hardness, modulus of elasticity and modulus of rupture, and, consequently, the quality of the product. The wood samples were heated in an inert gas environment under different operating conditions. The parameters studied are the final treatment temperature, heating rate, holding time, and the gas humidity. The weight loss and the temperature history of wood were monitored during the treatment. After the experiments, the properties of the samples were measured, and the effects of the above parameters on the properties were analyzed. The weight loss increased with increasing temperature, heating rate, holding time, and gas humidity. Increasing temperature caused an increase in MOE and a decrease in MOR. Overall hardness decreased with increasing temperature above 160 °C and it increased with increasing holding time and heating rate whereas it decreased with increasing gas humidity. Holding time and heating rate did not seem to affect MOE. Gas humidity increased MOE. MOR increased with holding time and decreased with heating rate and gas humidity.

D. Kocaefe (✉) · B. Chaudhry · S. Poncsak · M. Bouazara · A. Pichette
Department of Applied Sciences, University of Quebec at Chicoutimi, 555, boul. de l'Université, Chicoutimi, QC, Canada G7H 2B1
e-mail: dkocaefe@uqac.ca

Introduction

Wood is a versatile material with a large field of application; nevertheless, it has some limitations. It does not have a good dimensional stability in humid environment and is vulnerable to mold and other fungi infestation. The classical treatment to overcome these shortcomings is the impregnation of chemical substances into the wood such as creosote and chrome-cooper-arsenic salt. Although much progress has been made in order to minimize the toxic effect of these preservatives, they are still considered environmentally hazardous.

One of the alternatives to the chemical treatment is the high-temperature heat treatment of wood. There are different technologies used for the wood heat treatment in Europe: the Dutch PLATO Process, Retification Process (or NOW, France), Bois Perdure Process (France), OHT-Process (Germany), and the Thermo Wood Process (Finland). In the OHT-Process, the wood is treated in oil. In all the other processes, the wood is treated by slowly increasing its temperature above 200 °C in an inert and humid gas atmosphere. The differences between different technologies are the design of the furnaces, gas composition, and the recipes used [1].

The effect of heat treatment on the mechanical properties is complex. During the first part of heating (drying), wood looses its free moisture which is the water contained in the cell cavity. When the fiber saturation point (FSP) is reached, the cell cavity becomes empty but the cell walls are still fully saturated. After this point, the moisture in the cell wall is removed if drying is continued. Above FSP, the mechanical properties do not change with changing

moisture content [2–4]. When the moisture content is reduced below FSP, the cell wall collapses causing shrinkage in wood. At this stage, wood can re-absorb water if it is left in a humid environment, consequently, it will swell. It is reported in the literature that the mechanical properties improve with increasing moisture content or decreasing temperature as the moisture content falls below the fiber saturation point [5] during drying.

The conventional drying takes place up to 100–115 °C whereas, during heat treatment, wood is heated above 200 °C. The heat treatment affects the structure of wood. As it is well known, wood is composed of three structural polymers, namely cellulose, hemicelluloses, and lignin. Depending on the heating time, temperature, and the concentration of other components present, wood can behave as a viscoelastic material, a viscous fluid or a linear elastic solid [6]. The hemicelluloses and lignin are amorphous whereas the cellulose is crystalline. For amorphous polymers, the temperature at which there is a transition between glassy and rubbery states is called the glass transition temperature. The properties of wood change significantly if it is heated to higher temperature than the glass transition temperature (heat treatment) due to the degradation of the hemicelluloses and part of the cellulose. It is reported in the literature that the change in mechanical properties of wood, especially in strength, at high temperatures is mainly due to hemicelluloses degradation (hydrolysis). It has a lower molecular weight compared to the other wood polymers, therefore, it degrades faster. Also the cellulose crystallization and lignin modification take place [7, 8]. The lignin on the cells wall softens at temperatures [9] above its glass transition temperature, which is around 160 °C. Stamm and collaborators [10] have showed that the heat treatment affected lignin and hemicelluloses and resulted in water-soluble polymer formation. The moisture content, treatment temperature, presence and absence of oxygen and the treatment time are the factors, which influence most the hydrolysis reactions, consequently, the mechanical properties.

The moisture content of wood affects the mechanical properties in a number of ways. First of all, it changes the glass transition temperature of wood [11, 12]. In addition, the evaporation of moisture influences the temperature of wood. During the hemicelluloses hydrolysis, organic acids form. The moisture removal helps to carry away the acids formed during the hydrolysis. Stamm [13] observed that the degradation of wood is reduced if acids stay within the wood and the wood is degraded 10 times faster in the presence of moisture compared to the dry wood [14]. Also, the hygroscopic character of wood is reduced faster under humid conditions [15].

In addition to the effect of moisture, the degradation rate, and, consequently, the loss in weight and in mechanical properties are increased with increasing oxygen concentration. As the oxygen concentration is increased, the mass loss is increased [16]. Longer treatment times and higher temperatures have a negative affect on degradation [14], consequently, on the mechanical properties. Also, the effects of above parameters are different for each species. Due to a large number of parameters affecting the degradation, it is difficult to pinpoint a definite mechanism explaining the changes occurring in mechanical properties during the treatment.

A number of studies investigating the effect of heat treatment on the mechanical properties are reported in the literature. Wood looses its elasticity during the heat treatment [17]. The wood becomes more rigid and fragile, and its density increases. The rupture strength decreases. For example, in the Plato process [1], the rupture modulus decreases by about 18% compared to that of non-treated wood. The hardness depends on the wood species: in leafy trees, it increases as a result of heat treatment; however, in the conifers, it decreases mainly because of the degradation of the wood. Stamm et al. [10] reported that the dimensional stability of wood improves (40% decrease on swelling and shrinking); however, its mechanical strength decreases by about 50% compared to the original wood under the conditions they used. The heating can damage the wood cellular structure. Viitaniemi [18] and Rowell [9] among others studied the effect of gas humidity on the quality of wood and obtained a much better flexural stiffness adjusting the humidity during treatment.

Heat treatment at high temperature improves the bio-durability of wood. The mold and fungi growth requires suitable temperature, nutrients, oxygen, and moisture [19]. The thermal treatment at high temperature reduces the hygroscopic properties of wood [20, 21] due to the degradation of hemicelluloses and prevents the re-absorption of water, consequently, increases the resistance of wood to biological attacks.

Heat treatment improves the dimensional stability of wood. When wood is in a humid environment, the water molecules penetrate between the wood polymers, and hydrogen bonds are formed; therefore, wood swells. It is known that treating wood at temperatures higher than those used in the conventional drying process prevents subsequent swelling and shrinking [10, 14] because the treated wood is less hygroscopic. During the heat treatment, some of the cell walls re-organize, and the number of OH–H bonds decrease

[22]. Also softening lignin can flow and block the pores of the cells, limiting the water entrance into the wood structure during the treatment [9].

At high temperatures, the densification of the wood takes place. The densification is the result of cross-linking between the wood constituents themselves as well as between the wood constituents and the sub-products forming as a result of the degradation process, which starts slowly at the temperatures used for the heat treatment.

As explained above, wood becomes more resistant to biological attacks; however, it may lose some of its elasticity when it is subjected to heat treatment. It is important to optimize the process parameters in order to minimize the depolymerization reactions that are responsible for the mechanical degradation of wood and, at the same time, maintaining the resistance to fungi attacks [21]. There is a competition between two reactions: (a) the degradation, which breaks the chemical bonds, and (b) the reticulation, which creates new bonds, mainly in the lignin which has a significant impact on the mechanical properties. Therefore, it is important to determine how the treatment conditions affect the mechanical properties of wood since this effect is different for different species. This article focuses on this aspect. Wood samples were heat-treated using a thermogravimetric analyzer under different conditions. Then, various properties (hardness, MOE, MOR) of the treated samples were measured in order to study the effect of the treatment conditions on these properties.

Experimental

System

In this study, aspen samples (0.035 m × 0.035 m ×0.2 m) are heat-treated in a thermogravimetric analyzer. A schematic diagram of the experimental system is shown in Fig. 1. The wood samples were placed in a tubular furnace and suspended from an electronic balance. This furnace was equipped with a programmable three-zone temperature controller. The experiments were carried out in an inert gas (nitrogen) atmosphere. The gas was preheated in the lower zone of the furnace and distributed uniformly in the tube. The gas humidity was adjusted by heating water and injecting very fine droplets into the gas. The gas humidity was determined from dry gas and wet bulb gas temperature measurements and a hygrometer. The gas temperature was measured with a thermocouple placed close to the wood sample. The details of the furnace are shown in Fig. 2.

During the experiment, the temperature of the gas was controlled. It was heated at a known linear heating rate up to a predetermined maximum temperature. There was a half an hour plateau where the temperature of the gas was kept constant at 100 °C to facilitate the evaporation. The heat was transferred from the hot gas to the wood. Two different types of heat treatment experiments were carried out. In the first set of experiments, the weight loss of the sample with respect to time was monitored continuously with the computerized data acquisition system. These data

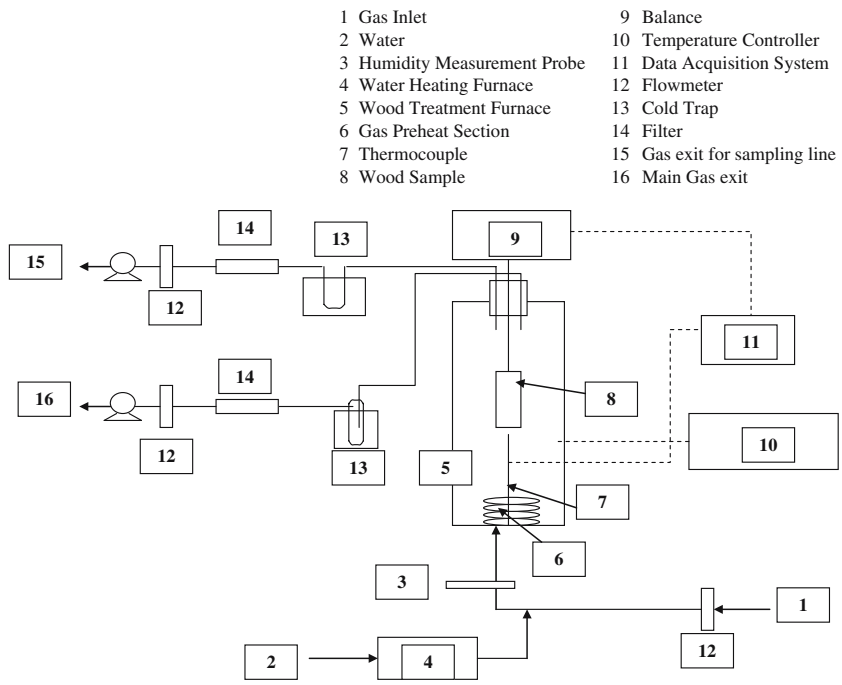

Fig. 1 Schematic diagram of the experimental system

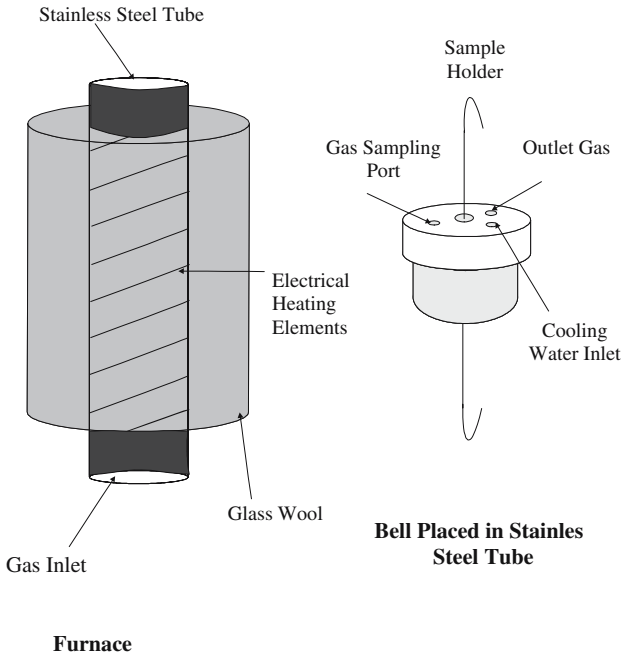

Fig. 2 Detailed view of the heat treatment furnace

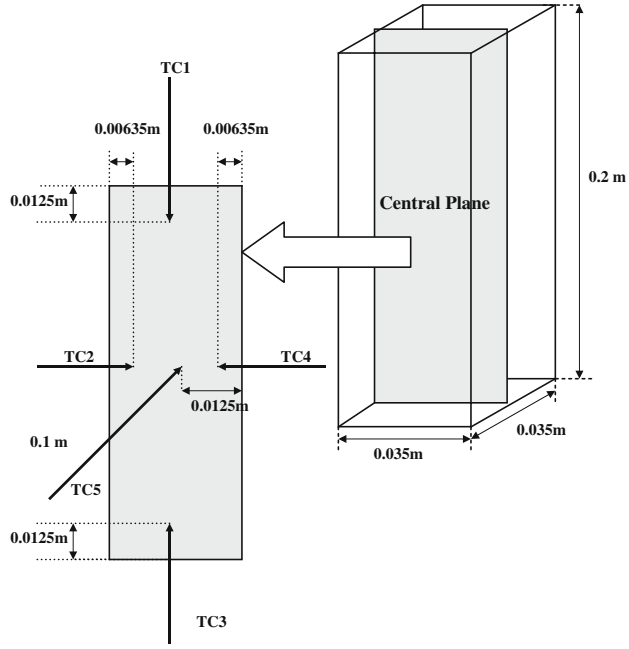

Fig. 3 Position of thermocouples

show how the weight loss is affected by the treatment conditions. In the second set of experiments, five thermocouples were placed in the sample. The positions of the thermocouples are shown in Fig. 3. During this experiment, the evolution of temperatures with time was monitored at these positions.

After the experiments, the sample was cooled down with a water spray. The samples were dried in air. Since this is a comparative study and the treated samples do not re-absorb water, they were not conditioned in a chamber with controlled humidity. Following air drying, the mechanical properties of the samples were measured, and the effect of the heat treatment on these properties was assessed.

Heat treatment parameters

The heat treatment parameters affect the final wood properties significantly. In this study, the effect of heating rate, final treatment temperature, holding time at the final temperature, and the gas humidity on the heat treatment were investigated; and the general trends were identified for aspen. The heat treatment conditions for the base case were:

- Final temperature: 220 °C
- Heating rate: 20 °C/h
- Holding time at maximum treatment temperature: 0 min
- Absolute humidity of gas: 160 g water/m^3 gas.

First, the experiments were carried out at the base conditions described above. During the subsequent experiments, one parameter was changed at a time. The experimental conditions are summarized in Table 1.

Mechanical properties

There are not established norms to measure the mechanical properties of heat-treated wood since it is a new material. However, it can be considered as a ''Modified Material''. Therefore, the norms for the ''Modified Material'' were followed during the measurements.

Table 1 Experimental treatment conditions

Maximal temperature (°C)	Gas heating rate (°C/h)	Holding time (min)	Gas humidity (g water vapor/m^3 dry gas)
210	20	0	160
220[a]	20[a]	0[a]	160[a]
230	20	0	160
220	10	0	160
220	30	0	160
220	20	15	160
220	20	30	160
220	20	45	160
220	20	0	0
220	20	0	80

[a] Base conditions

The tests were carried out with samples without visible cracks and knots. The size of the samples treated was much smaller than the sample size recommended by the ASTM standards. The experimental set-up and the procedure were adapted to small samples. It must be noted that the properties measured here may not be directly compared with the properties measured using the standard size samples. However, as explained below, this is a comparative study and gives a good indication of how the properties are affected by the heat treatment parameters.

Hardness

The hardness tests (see Fig. 4) were carried out following the procedures established by ASTM 143-94 and ASTM 1324-93. In these tests, a 0.5 in (0.0127 m) diameter steel ball was forced into the wood [23] by applying a constant load for a known period; consequently, the ball was embedded into the wood. The diameter of the indentation was measured and the Brinell hardness was found by dividing the load applied in (kg) by the surface area of the indentation in (mm^2).

The tests were carried out on both the tangential and the radial faces of each sample. The tangential face chosen was the one on the interior side of the tree as recommended by the ASTM. On each face, five points were chosen to carry out the tests and the average value for each surface was calculated. Then, the average of the radial and tangential hardness was calculated for each sample, and this value was used to compare different samples, as suggested by the ASTM 1324-93 standard.

Three-point flexural test

The flexural test measures the force required to bend a sample under three-point loading conditions. Flexural strength is the measure of how well a material resists bending, or what the stiffness of the material is. Three-point bending tests were carried out following the ASTM 143-93 standard in a universal machine test Alliance RT100, MTS. The load was applied to the tangential face on the interior side of the sample. The samples had a cross-sectional area of 0.0254 m × 0.0254 m and were 0.2 m long. During the test, a

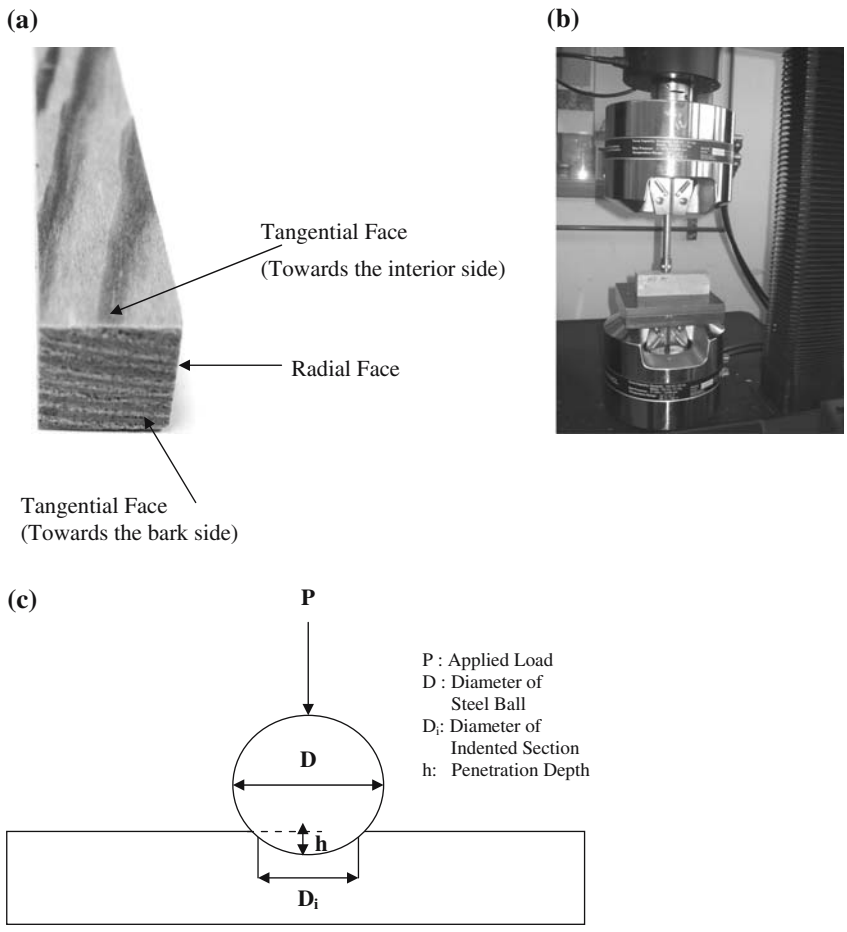

Fig. 4 Hardness test (**a**) Definition of radial, tangential and transversal faces of wood. (**b**) Hardness Measurement Set-Up. (**c**) Parameters measured for Brinell hardness calculation

unidirectional load was applied to the middle of the sample at a speed of 1.3 mm/s in the vertical direction. The sample was supported from two sides as shown in Fig. 5.

From the flexural (bending) tests, different mechanical parameters can be calculated such as the maximum bending moment (Mf_{max}, N/mm), the modulus of rupture (MOR), and the modulus of elasticity (MOE).

The modulus of rupture is the indication of the maximum bending load that a sample can support, which is the measure of the stress required to cause failure. Increasing MOR means that a greater force is required to cause failure. Its unit is N/mm^2.

The modulus of elasticity (MOE, elastic modulus or Young's modulus, N/mm^2) is the measurement of stiffness that determines the deflection caused by a load. Higher MOE means greater stiffness.

Error analysis

The accuracy of the measurements for dimensions, the weight, and the moisture content of the samples were ±0.005 mm, ±0.001 g, 0.1%, respectively. The thermocouple readings were accurate to 3.8%. Three-point flexion and hardness tests in both directions (on radial and tangential faces) were repeated 12 times. Accuracy of the measurement of the ball penetration depth was ±0.01 mm and that of the applied force was ±0.005 N. The standard deviation (SD) and relative error (RE) were calculated for MOE, MOR, and hardness measurements.

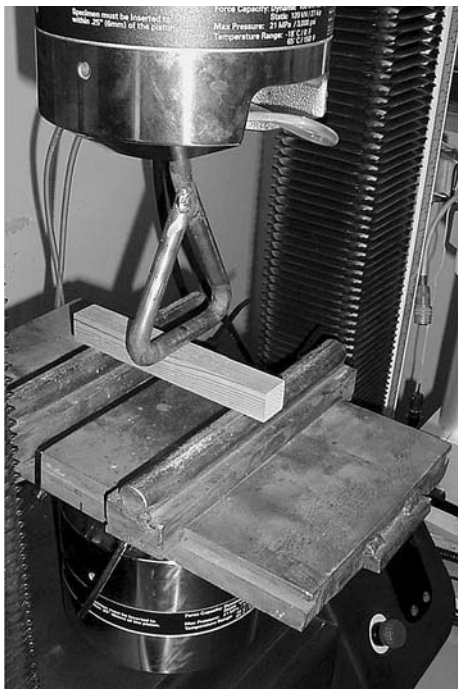

Fig. 5 Three-point flexural test apparatus

Results

The experiments were carried out using different heat treatment conditions. The change of temperature and weight loss with time were measured during these experiments. Then, the mechanical properties of the samples were measured in order to understand the effect of treatment conditions on the wood properties. This study is part of a larger research program on the adaptation of wood heat-treatment process to Quebec species. It is difficult and costly to asses the affect of process operating conditions on the product quality at the industrial scale. This study essentially constitutes the first screening test. The conditions identified here will be tried in pilot and industrial scale experiments.

Temperature and weight loss evolution

Table 2 presents the percent total weight loss measured under different heat treatment conditions. Figure 6 shows how the temperatures (which were measured at different thermocouple positions; see Fig. 3) changed with the normalized time (actual time divided by the treatment time, t/t_{max}). For this experiment, the final treatment temperature (T_f) was 230 °C. The heating rate, the gas humidity, and the holding time were the same as the base case given above. The observed trends were similar in all experiments. As it can be seen from this figure, the temperature readings were almost identical up to 160 °C. The small differences in these measurements might be due to the placement of thermocouples at different positions at which different type of wood structures might be

Table 2 Total weight loss of wood at the end of heat treatment

Parameter studied	Condition	Weight loss (%)
Gas heating rate (°C/h)	10	20.1
	20	22.0
	30	24.5
Maximum temperature (°C)	210	19.6
	220	22.0
	230	22.3
Holding time (min)	0	22.0
	15	20.7
	30	17.2
	45	18.9
Gas humidity (g water/cm^3 gas)	0	15.7
	80	19.4
	160	22.0

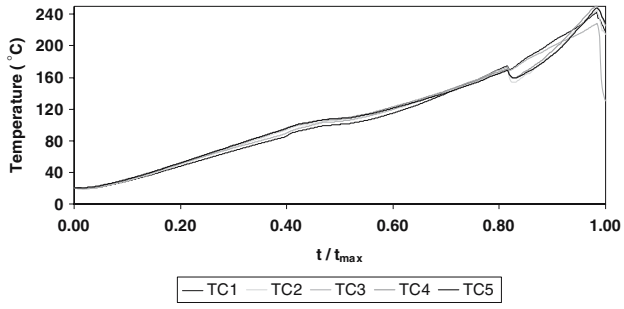

Fig. 6 Wood temperature versus time data for five thermocouples for the base case (Final temperature: 220 °C; Heating rate: 20 °C /h; Holding time at maximum treatment temperature: 0 min; Absolute humidity of gas: 160 g water/m^3 gas)

present (summer wood, spring wood, etc.). It is well known that these different structures have different cell sizes, and their water storage capacities are different. Also, the possibility of slight differences in the calibration of thermocouples and the low thermal conductivity of wood might be other reasons, which can explain the small differences observed among the thermocouple readings. However, it can be said that the temperature of the sample can be considered uniform. The core temperature shown by TC5 in Fig. 6 is very similar to other temperatures measured in the sample (see Fig. 3 for the thermocouple positions). This was the objective since the mechanical tests should be carried out on a sample treated uniformly. After 160 °C, the temperature of the wood increased suddenly shown by the steeper slope of the temperature versus time curve although the inlet gas temperature was increased linearly. This indicates that, at this temperature, exothermic reactions start taking place, and this increases the wood temperature faster than the surrounding gas temperature. At this point, the differences between thermocouple readings were more pronounced. This might be due to the presence of different wood structures at the measurement points. Since they have different cell sizes and water contents, the rate of reaction differs at different positions. Since the temperatures were similar at different positions, for comparing the effect of different treatment parameters on the evolution of wood temperature the temperature readings at TC5 thermocouple position were used.

The effect of heating rate on the wood temperature is presented in Fig. 7a. Three different gas heating rates (10 °C/h, 20 °C/h, 30 °C/h) were studied. As it can be seen from this figure, the wood temperature followed the gas temperature at the lowest heating rate. However, as the heating rate is increased, the slope of temperature curve showed a sudden increase. The slope of this section became steeper with increasing heating rate. The reactions taking place inside the

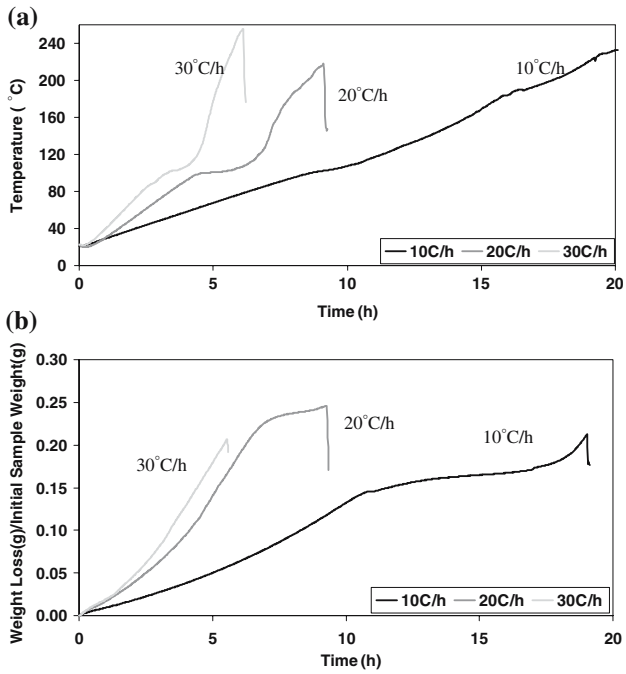

Fig. 7 Effect of heating rate on (**a**) Wood temperature measured at TC5 thermocouple position; (**b**) Normalized weight loss (Final temperature: 220 °C; Holding time at maximum treatment temperature: 0 min; Absolute humidity of gas: 160 g water/m^3 gas)

wood started at earlier times when the gas heating rate was increased as indicated by the shift of the curve to the left. Table 3 shows the wood heating rates (change in wood temperature divided by the time interval) measured during the experiments between 160 °C and 220 °C; and they are significantly higher than the imposed gas heating rates indicating the occurrence of exothermic reactions, most probably due to cross-linking. The normalized weight loss (weight loss divided by the initial weight of the sample) versus time data is shown in Fig. 7b for three gas heating rates. At a given time, the weight loss increased with increasing heating rate.

The effect of holding time at the maximum treatment temperature was also studied. In the base case, the sample was cooled down as soon as the desired

Table 3 Measured wood heating rates between 160 °C and 220 °C

Gas heating rate (°C/h)	Wood heating rate (°C/h)
10	13.28
20	29.34
30	74.23

Final temperature: 220 °C; Holding time at maximum treatment temperature: 0 min; Absolute humidity of gas: 160 g water/m^3 gas

Table 4 Effect of holding time on wood weight loss

Holding time (min)	Weight loss (%)[a]
15	0.83
30	1.79
45	2.12

[a] With respect to the initial sample weight

treatment temperature was reached (zero minute of holding time). In the following experiments, the samples were kept at the maximum treatment temperature (220 °C) for 15 min, 30 min, and 45 min before cooling. Table 4 presents the weight loss observed during these holding periods. As it can be seen, the weight loss observed was very small even for the 45-min period. The effect of the holding time is more significant on the mechanical properties as it will be explained later.

The effect of gas humidity on the wood temperature and weight loss is shown in Fig. 8. For the humidity of 0 g water/m^3 gas (dry) and 80 g water/m^3 gas, the wood temperature evolutions were found similar. When the gas humidity was increased to 160 g water/m^3 gas, the temperature was slightly lower in the initial section compared to those of the lower humidities. This difference increased at the evaporation temperature. The temperature for the experiment carried out with the humidity of 160 g water/m^3 gas stayed constant for a certain period followed by a sudden increase in temperature. With this increase, the temperatures of three experiments became equal towards the end of the treatment. If the weight loss curves are studied, it can be seen that the weight loss increased with increasing moisture content. As the gas moisture content increases, the moisture concentration difference between the gas and wood decreases. Therefore, the mass transfer rate would be expected to decrease with the decreasing gradient. Here, however, the reverse trend was observed. This might be explained as follows: When the concentration gradient is high, the mass transfer is fast at the surface. This causes hardening and shrinking at the surface layers [24] slowing down further water removal. This trend was also reported by other researchers. Viitaniemi [9] found that the presence of high humidity content in gas leads to higher losses.

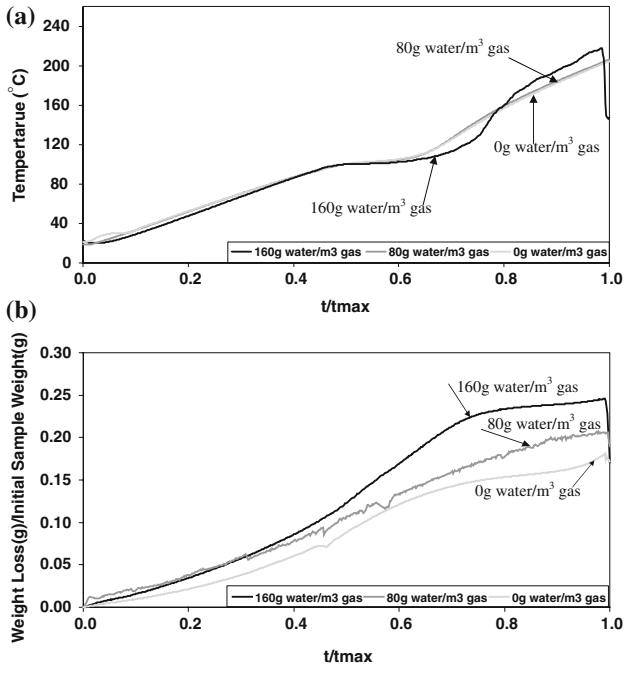

Fig. 8 Effect of gas humidity on (**a**) Wood temperature; (**b**) Normalized weight loss (Final temperature: 220 °C; Heating rate: 20 °C/h; Holding time at maximum treatment temperature: 0 min)

Mechanical properties

The mechanical properties of the aspen samples, heat-treated under different conditions, as well as the untreated ones were measured. The results are presented in the following sections.

Hardness

Figure 9 and Table 5 present the effects of the treatment conditions on the hardness of aspen. As it was explained before, the hardness measurements were carried out both on radial and tangential faces. It was found that the hardness in the radial direction decreased with increasing temperature during treatment (see Fig. 9a). However, in the tangential direction, the hardness values passed through a maximum around 160 °C. At this temperature, the structural changes start as indicated by the temperature data (see Fig. 6). It was also observed that the hardness in the radial direction decreased and the hardness in the tangential direction increased with increasing holding time (see Fig. 9b) and increasing heating rate (see Fig. 9c). The humidity of gas did not seem to affect the radial hardness. The tangential one decreased with increasing gas humidity (see Fig. 9d). The ASTM standards recommend that the average of the tangential and radial hardness values be taken as the hardness of the material. The average hardness for aspen showed a tendency similar to the tangential hardness.

Three-point flexural tests

The maximum bending moment and the modulus of rupture (MOR) showed a slight decrease initially with

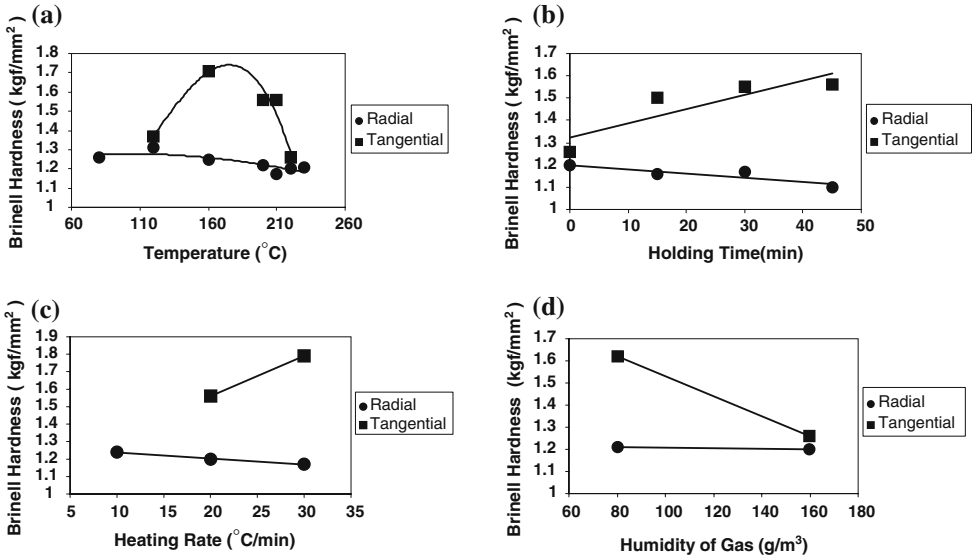

Fig. 9 Effect of treatment parameters on hardness (**a**) Effect of temperature; (**b**) Effect of holding time; (**c**) Effect of heating rate; (**d**) Effect of gas humidity

Table 5 Brinell hardness of treated wood

	Hardness (kgf/mm^2)	
	Radial	Tangential
Maximum temperature (°C)		
80	1.26	–
120	1.31	1.37
160	1.25	1.71
200	1.22	1.56
210	1.17	1.56
220[a]	1.20	1.26
230	1.21	–
Gas heating rate (°C/min)		
10	1.24	–
20[a]	1.20	1.56
30	1.17	1.79
Holding time (min)		
0[a]	1.20	1.26
15	1.16	1.50
30	1.17	1.55
45	1.10	1.56
Gas humidity (g water/m^3 gas)		
0	1.18	-
80	1.21	1.62
160[a]	1.20	1.26

[a] Base condition

temperature. It was observed that they were almost constant between 100 °C and 200 °C. Above 200 °C, MOR shows a clear decrease. The modulus of elasticity (MOE) increased slightly with increasing temperature. The results show that aspen becomes more and more fragile with increasing treatment temperature (see Fig. 10), especially at temperatures higher than 200 °C. The maximum treatment temperature should be chosen carefully.

Figure 11 shows the effect of holding time on the flexural properties of aspen treated at a maximum temperature of 220 °C. The maximum bending moment and MOR decreased slightly whereas MOE did not change significantly with increasing holding time. As it can be seen from this results, increasing the holding time at this temperature did not have a significant affect on the flexural properties. Therefore, the wood temperature for aspen can be kept constant for a certain period of time in order to have a uniform wood temperature within the furnace before stopping the treatment.

The heating rate did not seem to affect MOE whereas the maximum bending moment and MOR slightly increased with increasing heating rate as shown in Fig. 12. This might be due to the fact that when higher heating rates are used, the contact time between the wood and high temperature gas is shorter compared to that of lower heating rates for the same final temperature.

The mechanical tests were carried out for the samples treated at two different gas humidities (0 g water/m^3 and 80 g water/m^3). The results are shown in Fig. 13. It seems that there is slight increase in the maximum bending moment, MOE, and MOR with increasing gas humidity. The similar effect of gas humidity on the reactions taking place during the transformation of wood was also observed by other researchers [18].

Error analysis

The relative error (RD) and the standard deviation (SD) for the maximum bending moment (Mf_{max}), modulus of rupture (MOR), and the modulus of elasticity (MOE)

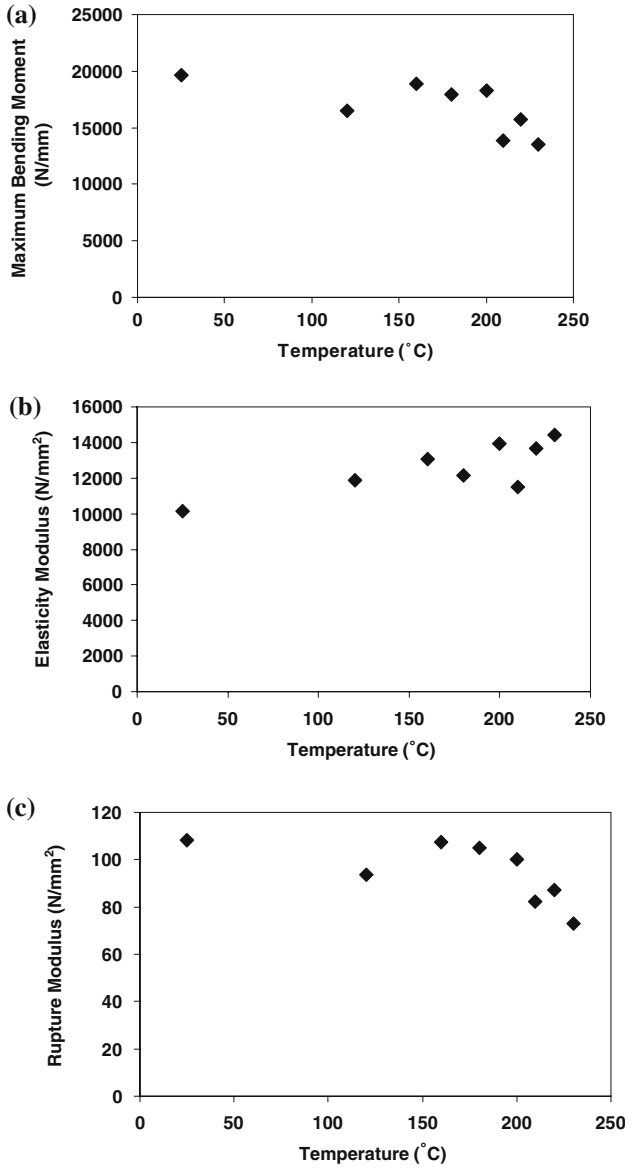

Fig. 10 Effect of treatment temperature on mechanical properties (**a**) Maximum bending moment; (**b**) MOE; (**c**) MOR (Heating rate: 20 °C/h; Holding time at maximum treatment temperature: 0 min; Absolute humidity of gas: 160 g water/m^3 gas)

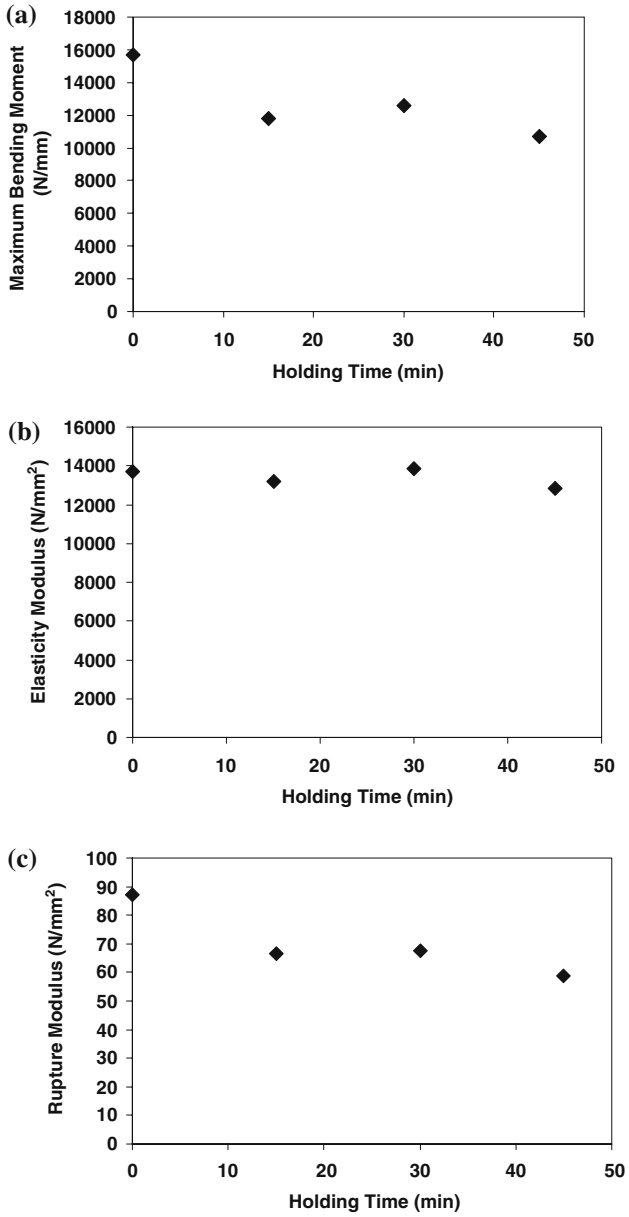

Fig. 11 Effect of holding time on mechanical properties (**a**) Maximum bending moment; (**b**) MOE; (**c**) MOR (Final temperature: 220 °C; Heating rate: 20 °C/h; Absolute humidity of gas: 160 g water/m^3 gas)

measurements are presented in Table 6, and Table 7 presents those of the hardness measurements in the tangential and radial directions. The results show that the precision of measurements are ±5–10% for the Mf_{max} and MOR, 2–5% for MOE, and 1–6% for hardness, both in radial and tangential directions.

Conclusions

Aspen is heat-treated in a laboratory thermogravimetric system. The temperature of wood at different positions within the sample and the overall weight loss of the sample were monitored continuously. Afterwards, the treatment, the hardness, and the flexural properties were measured in order to determine the general trends. Laboratory scale experiments are less costly than the pilot scale or industrial scale trials. However, the trends observed in these experiments should be validated at larger scales.

During the heat treatment of aspen, first the moisture and some volatiles evaporate. Then, the structural changes start to take place around 160 °C. The presence

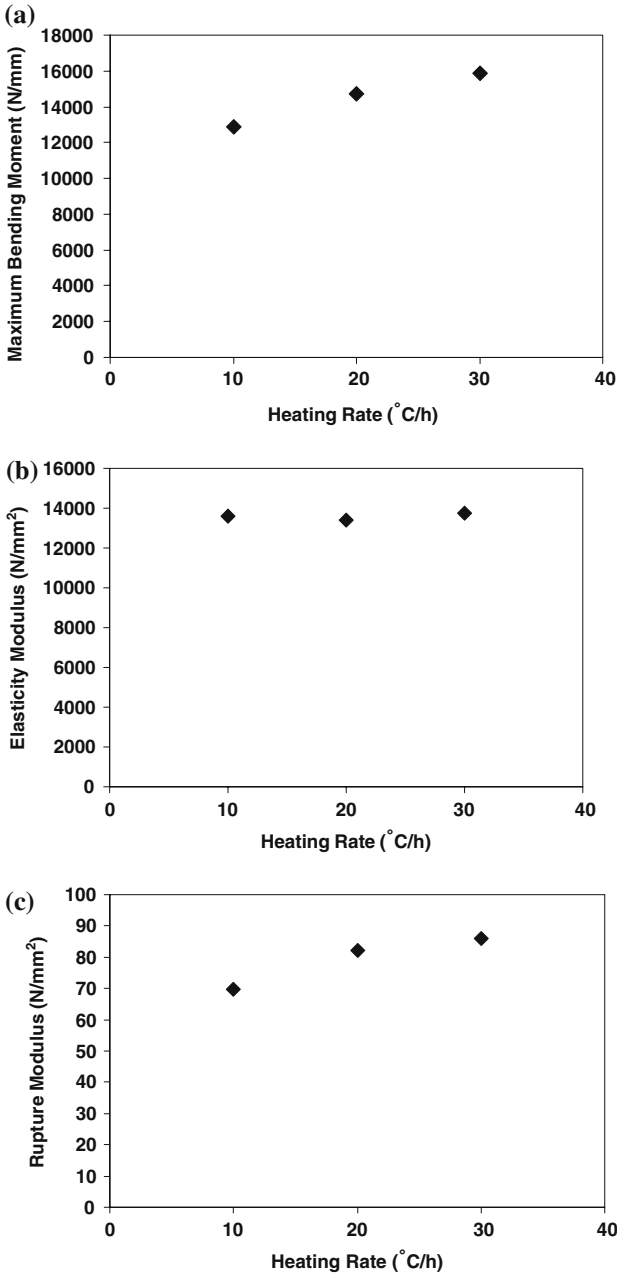

Fig. 12 Effect of heating rate on mechanical properties (**a**) Maximum bending moment; (**b**) MOE; (**c**) MOR (Final temperature: 220 °C; Holding time at maximum treatment temperature: 0 min; Absolute humidity of gas: 160 g water/m^3 gas)

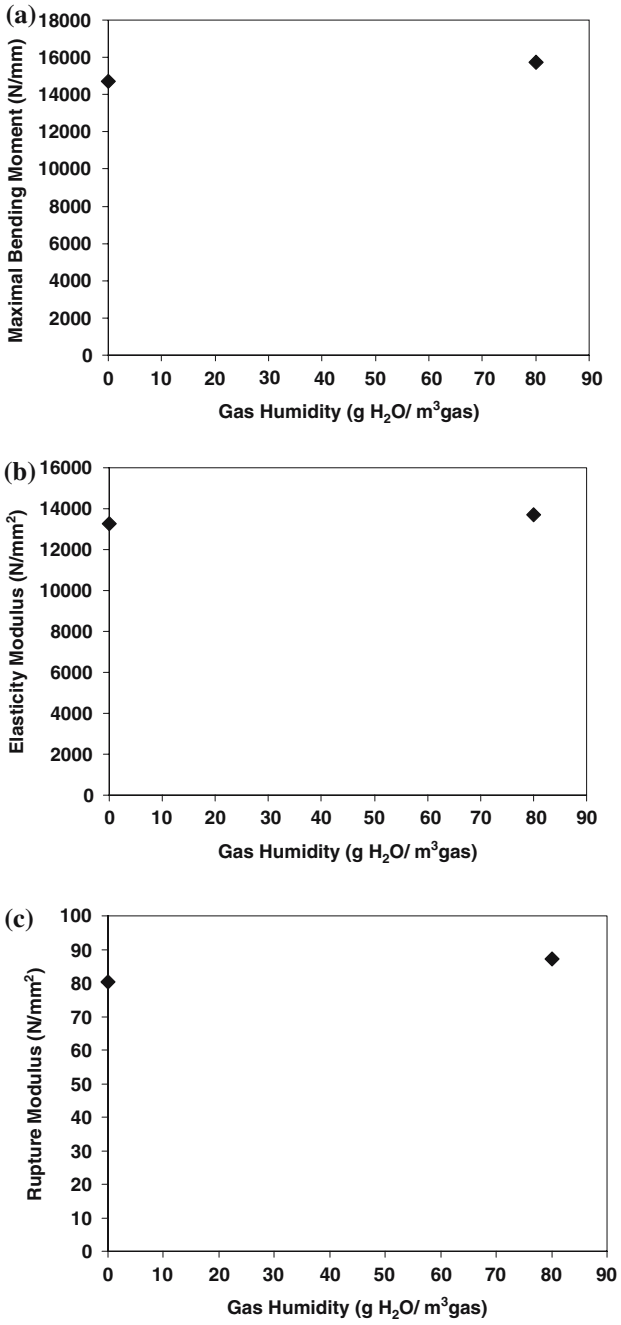

Fig. 13 Effect of gas humidity on mechanical properties (**a**) Maximum bending moment; (**b**) MOE; (**c**) MOR (Final temperature: 220 °C; Heating rate: 20 °C/h; Holding time at maximum treatment temperature: 0 min)

of an exothermic reaction was observed from the temperature versus time curves indicated by a sudden increase of the slope in the vicinity of this temperature. This increase was more pronounced at higher heating rates. The weight loss increased with increasing final temperature, holding time, and gas humidity.

The mechanical properties of treated aspen are affected by the treatment parameters. Increasing heating rate and holding time increased the tangential hardness and slightly decreased the radial hardness of aspen whereas increasing the gas humidity decreased the tangential hardness but did not seem to affect the radial hardness. For this species, MOE was slightly increased by the maximum treatment temperature and the gas humidity; however, it did not seem to be affected by holding time and heating rate. The maximum bending moment and MOR decreased when the

Table 6 Relative error (RD) and standard deviation (SD) of maximum bending moment (Mf_{max}), modulus of rupture (MOR), and modulus of elasticity (MOE) measurements

Experimental parameter changed relative to the base case	Mf_{max}		MOR		MOE	
	RE (±%)	SD (N/mm)	RE (±%)	SD (N/mm)	RE (±%)	SD (N/mm)
Untreated aspen	5.53	2,166.34	4.41	9.57	33.59	6,826.23
Temperature: 120 °C	4.64	2,169.05	2.80	7.41	2.47	828.91
Temperature: 160 °C	4.24	2,780.00	2.69	10.00	2.83	1,281.55
Temperature: 180 °C	5.59	2,121.41	6.59	14.81	4.94	1,249.03
Temperature: 200 °C	4.31	1,576.91	3.83	7.67	4.39	1,224.85
Temperature: 210 °C	8.46	2,889.32	9.09	17.90	4.33	1,572.04
Base case	7.64	3,883.29	8.03	22.94	2.46	1,194.46
Temperature: 230 °C	10.51	2,847.99	10.26	14.98	4.88	1,410.22
Holding time: 15 min	5.87	2,229.91	7.17	15.41	4.17	1,515.52
Holding time: 30 min	4.16	1,480.29	4.35	8.30	1.62	634.92
Holding time: 45 min	9.28	2,812.28	10.21	17.00	3.69	1,343.64
Heating rate: 10 °C/h	6.35	1,637.55	6.43	8.97	1.87	510.05
Heating rate: 30 °C/h	7.24	3,864.23	7.18	20.78	2.83	1,336.32
Gas humidity: 0 g water/m^3 gas	6.99	3,565.41	6.87	19.14	3.05	1,401.43
Gas humidity: 80 g water/m^3 gas	5.62	1,923.69	5.89	11.11	3.12	883.69

Table 7 Relative error (RD) and standard deviation (SD) of radial and tangential hardness measurements

Experimental parameter changed relative to the base case	Radial		Tangential	
	RE (±%)	SD (kgf/mm^2)	RE (±%)	SD (kgf/mm^2)
Untreated aspen	2.53	0.0713	1.32	0.0389
Temperature: 120 °C	1.44	0.0422	0.89	0.0273
Temperature: 160 °C	4.21	0.1173	5.04	0.1920
Temperature: 200 °C	2.67	0.0956	2.50	0.0872
Temperature: 210 °C	1.07	0.0566	4.83	0.1685
Base case	4.55	0.1226	1.39	0.0393
Temperature: 230 °C	2.73	0.0957	3.34	0.1217
Holding time: 15 min	3.51	0.0911	5.40	0.1806
Holding time: 30 min	2.50	0.0658	4.64	0.1608
Holding time: 45 min	0.89	0.0219	3.36	0.1172
Heating rate: 10 °C/h	1.97	0.0548	4.85	0.2105
Heating rate: 30 °C/h	5.80	0.1520	5.97	0.2395
Gas humidity: 0 g water/m^3 gas	2.08	0.0552	1.57	0.0392
Gas humidity: 80 g water/m^3 gas	2.45	0.0664	4.59	0.1659

treatment temperature exceeded 200 °C. These properties also decreased with holding time and increased with heating rate and gas humidity.

Error analysis was carried out, and the relative error and the standard deviation were calculated for the measurements. The results showed that the accuracy of the measurements is acceptable.

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