

Gaseous Decomposition Products from Wood Degradation *via* Thermogravimetric and Fourier Transform Infrared Analysis during Thermal Modification of Beech and Pine Woods

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Wood thermal modification is an environmentally friendly method used to improve wood characteristics, and its degradation mechanism directly influences its application in construction, building, and home decoration. Combined thermogravimetry and Fourier transform infrared (TG–FTIR) studies were conducted to evaluate the hardwood and softwood on-site at 160 °C and 220 °C. The results indicated that the mass loss rates (MLRs) for both hardwood and softwood samples decreased over time and ultimately became constant. The total mass loss increased with an increase in temperature. The total mass loss was about 3% at 160 °C, and about 4.7% to 6.5% at 220 °C. Decomposition also occurred much more easily in the hardwood than in the softwood. More species of gas products were emitted from hardwood than softwood, and the number of species of gas products increased with an increase in temperature and processing time. The FTIR spectra of softwood were similar, whereas those of hardwood were remarkably different at different temperatures. Water and alcohol were generated from both the hardwood and softwood, whereas contents of these gaseous products were visible in hardwood decomposed at 220 °C during the thermal treatment. Ketones, ethers, acids, and aromatics were found in both hardwood and softwood, whereas CO₂ was only found in the hardwood that underwent thermal treatment.

Keywords: FTIR; Thermostability; Decomposition; Mass loss; Gas product; Generation mechanism

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INTRODUCTION

Wood, a renewable and natural material, has been widely used in construction, building, and wood processing (Wang *et al.* 2018) because of its high strength-to-weight ratio, high processability, environmental friendliness, and degradation (Popescu and Popescu 2013; Li *et al.* 2017). However, as a type of porous medium (Keey *et al.* 2012), wood can shrink and swell when it desorbs and absorbs moisture because of hygroexpansion and anisotropy (Skaar 1988; He and Yi 2016). Thus, these characteristics markedly shorten the service life and value of wood products and limit its broad application.

Wood thermal modification is an environmentally friendly method and has been widely used to improve dimensional stability (Rowell *et al.* 2009; Srinivas and Pandey 2012; Meints *et al.* 2018), decrease hygroscopicity, and improve durability against weathering, among other characteristics in wood (Kamdem *et al.* 2002; Kocafee *et al.*

2007; Herrera *et al.* 2015; Li *et al.* 2017; Croitoru *et al.* 2018; Shen *et al.* 2018; Wood *et al.* 2018). All these improvements have a close relationship to wood chemical and physical modification, which change as a result of thermal degradation. Research has been conducted to analyze the physical characteristics and chemical components of wood products after thermal modification (Priadi and Hiziroglu 2013; Aydemir *et al.* 2015; Croitoru *et al.* 2018; Herrera *et al.* 2018; Tomak *et al.* 2018), or to evaluate wood thermal degradation characteristics of wood pyrolysis (Bach *et al.* 2017; Haberle *et al.* 2017; Lin *et al.* 2018; Yang *et al.* 2018). However, few studies have focused on the volatile substance emission during wood thermal modification, which has great impact on wood properties. Moreover, recent studies on pyrolytic degradation of wood mainly have investigated high temperatures (mostly above 300 °C). These findings fail to guide wood thermal modification because the highest temperature for wood thermal modification is approximately 240 °C, and it is typically lower than 200 °C (International Thermowood Association 2003).

Wood degradation during thermal modification largely influences the physical properties of wood, including its density, strength, hardness, moisture content, biological durability, weather resistance, and dimensional stability, among others (International Thermowood Association 2003). TG-FTIR was used to analyze wood thermal modification on-site at 160 °C and 220 °C. The modification process was demonstrated through analyzing the process of wood weight loss, gaseous release, and its generation mechanism. The results can be used in the design of an improved wood thermal modification schedule to save time and energy consumption, as well as to predict wood characteristics and improve wood product quality.

EXPERIMENTAL

Materials

Beech (*Fagus sylvatica*) (hardwood), and pine (*Pinus elliottii*) (softwood), which are two of the most widely known commercial tree species in the wood industry, were used as samples. These wood species were obtained from YuXiang Wood Products Co., Ltd., GuangXi, China. The samples were ground into powder. Then they were dried in a chamber for 24 h at 103 °C to the oven-dried mass.

Methods

Thermogravimetric analysis of the samples was carried out under an N₂ atmosphere using a computerized thermobalance (STA449F3, Netzsch, Bavaria, Germany). The N₂ flow continually flushed the apparatus at a fixed rate of 100 mL/min. To prevent the possible effects on heat and mass transfer within the inner wood during heat treatment, a 10 mg sample that could pass through a 300-mesh sieve was placed in a crucible, and the temperatures were raised to the target temperatures (160 °C and 220 °C) in 1 min for each study. The process of volatile emission during the heat treatment process was examined using the thermogravimetric analyzer combined with Fourier transform infrared spectroscopy (FTIR). The N₂ atmosphere flow, which continually flushed the apparatus at a fixed rate of 100 mL/min, swept gaseous and condensable products into the gas cell of the FTIR spectrometer (Tensor 27; Bruker Optics, Bruker, Germany). The IR scanning range was set from 500 to 4000 cm⁻¹. All experiments were carried out in triplicate, and the average values represent the results.

RESULTS AND DISCUSSION

Wood Thermostability

To demonstrate the thermal degradation properties of wood samples, the average values of thermogravimetry (TG) and derivative thermogravimetry (DTG) curves for beech and pine samples treated at different temperatures are presented in Figs. 1 and 2. The figure show that the mass loss rates (MLRs) for the beech and pine samples decreased over time and ultimately became constant under different conditions. The total mass loss increased with an increase in temperature. Figure 1 shows that for the beech and pine samples treated at 160 °C, the total mass loss was approximately 3% following the thermal treatment. The total mass loss for beech was nearly constant at 5.3 min and was approximately 2.6% before 5.3 min. The total mass loss for pine was nearly constant at 5.8 min and was approximately 2.5% before 5.8 min.

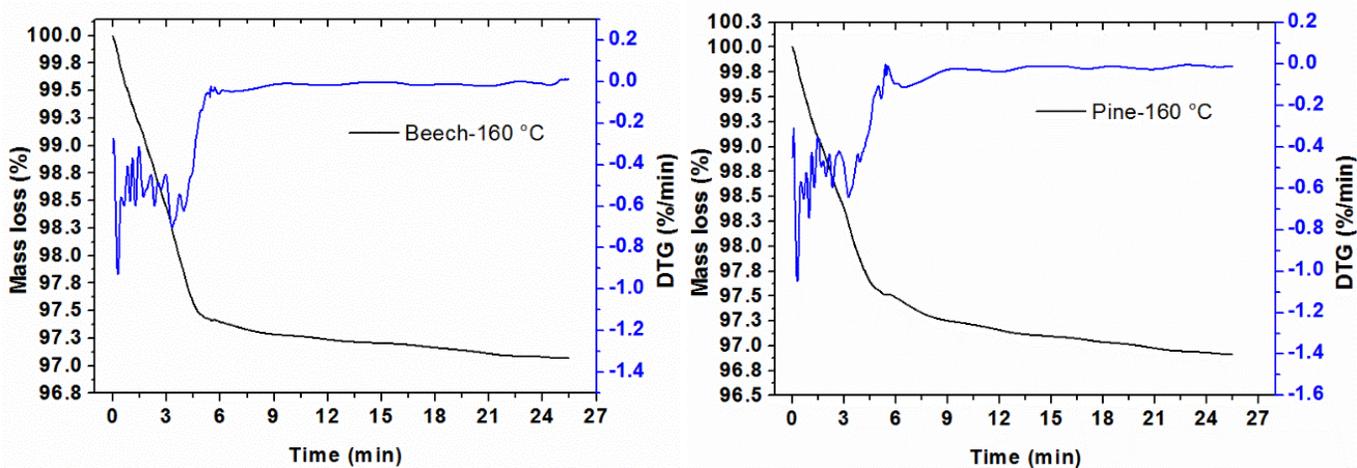


Fig. 1. TG-DTG curves of samples treated at 160 °C

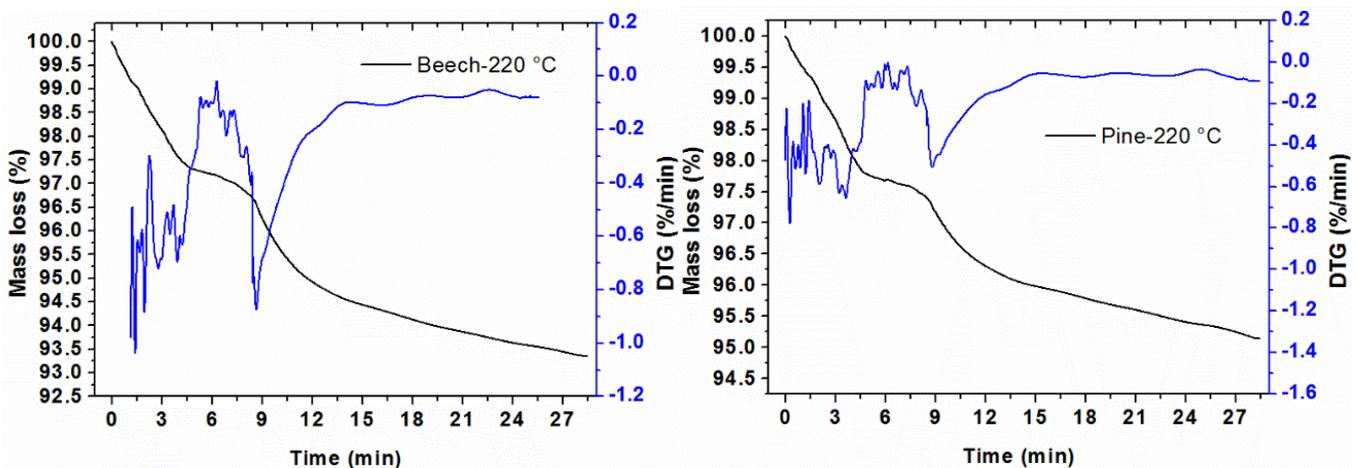


Fig. 2. TG-DTG curves of samples treated at 220 °C

Figure 2 shows that for the beech and pine samples treated at 220 °C, the total mass loss was approximately 6.5% and 4.7%, respectively, under the thermal treatment. The total mass loss for beech was nearly constant at 13.8 min and was approximately 5.5%

before 13.8 min; for pine, it was nearly constant at 14.6 min and was approximately 4.0% before 14.6 min. Moreover, peak decomposition was observed before the mass loss became constant, and the peak increased with an increase in temperature. Most of the mass loss at the initial stage of the thermal treatment could be attributed to water retention in wood tissue (Keey *et al.* 2012). Even when the wood samples were dried, the evaporation of water or the low-molecular weight compounds and some extractives could lead to mass loss (Chen *et al.* 2011; He *et al.* 2018). The total mass loss for beech was larger than the loss measured for pine. The reason might have been that among the wood components, the most sensitive to thermal treatment were the hemicelluloses (Popescu *et al.* 2018), which comprised a larger percentage in the hardwood species (beech) than in the softwood species (pine). In addition, degradation occurs more easily in hardwood than in softwood (International Thermowood Association 2003).

Characteristics of Gas Products Released and Decomposition Mechanisms

Wood mainly consists of cellulose, hemicellulose, lignin, as well as some other extracts (Johansson *et al.* 1999). Cellulose constitutes approximately 40% to 50% of wood, forms the framework of wood cell walls, and is the most resistant component in the inner wood. The interlaced cellulosic microfibrils are present within a matrix composed of hemicellulose and lignin (Bauer *et al.* 1973). Hemicellulose constitutes approximately 25% to 35% of wood and is the least stable and the most sensitive to thermal treatment among wood's components (Popescu *et al.* 2018). Hemicellulose is composed primarily of xylan and mannan (Blasi and Lanzetta 1997; Shen *et al.* 2010). Lignin comprises 20% to 30% of wood. The structure of lignin changes when the temperature exceeds 120 °C (Popescu *et al.* 2018). Extractives comprise less than 5% of wood (International Thermowood Association 2003). The degradation of wood components remarkably influences the physical properties of wood. Thus, the characteristics of gas products released from wood were analyzed to explain the degradation process of wood.

An FTIR spectrometer was used to analyze the characteristics of volatiles' emissions during thermal degradation. The concentrations of gaseous compounds present in the cell can be evaluated based on the Beer-Lambert law, which states that the concentration is proportional to absorbance of illumination when the optical system is kept constant. Different compounds can be separately evaluated at their wavenumbers of maximum light absorbance (He *et al.* 2018). The gas products released at different times and the corresponding temperatures are shown in Fig. 3. More gas species were released from beech than from pine, which might be due to the fact that the hemicellulose is the least stable major component in wood, and because more hemicelluloses are found in hardwood species than in softwood species (International Thermowood Association 2003; Shen *et al.* 2010). Moreover, the gas species released by beech increased with an increase in temperature; the variation in gas species was also greater in beech than in pine. This phenomenon could have been the reason that wood becomes unstable and some components are degraded with increasing temperature (International Thermowood Association 2003). In addition, the number of gas products increased with time, which could have been the reason why more energy was absorbed by the samples over time (Yang *et al.* 2018). Some extracts, or low-molecule weight components, were released, and some acids generated by hemicellulose degradation increased the degradation of other components (Tjeerdsma *et al.* 1998).

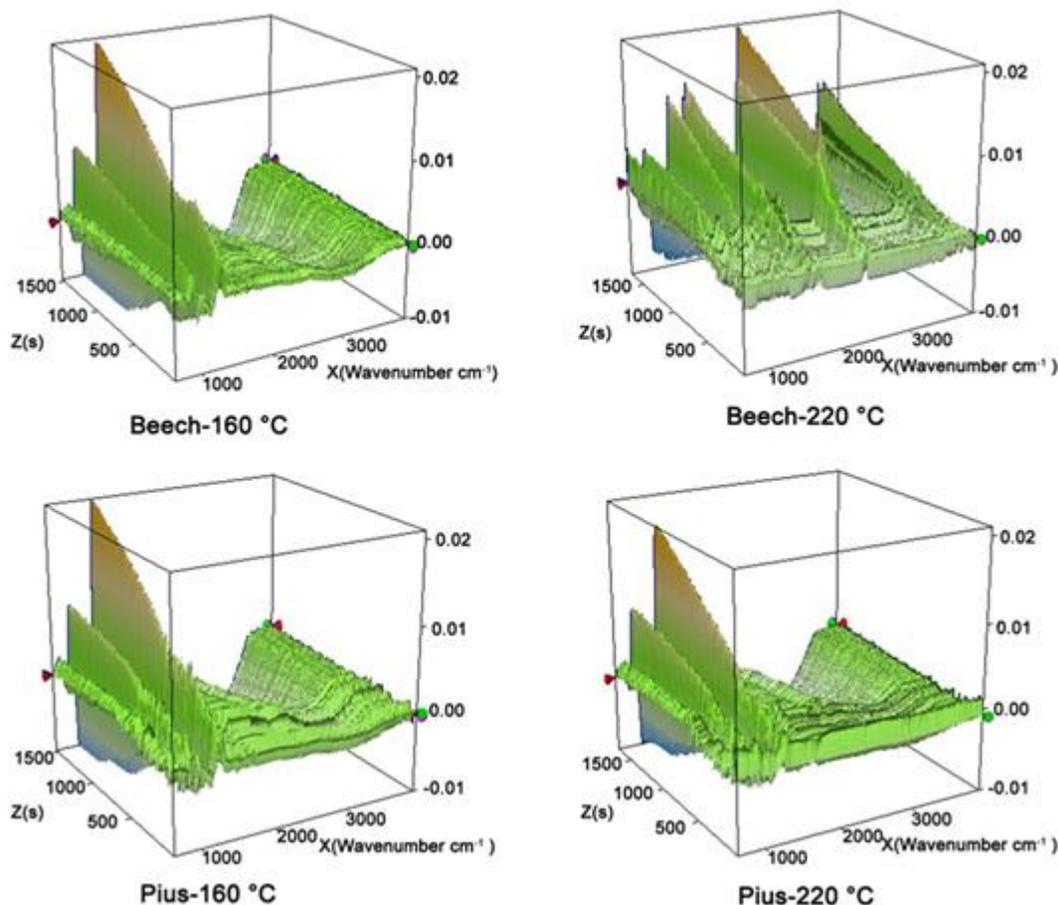


Fig. 3. Gas products released at different conditions

To elucidate the types of gas components emitted from wood under different treatment conditions, as well as the degradation mechanism of the wood samples, the FTIR spectra of the gas phase at the initial stage (1 min), middle (6.0 min), and final stages (25 min) of the thermal degradation are shown in Figs. 4 and 5.

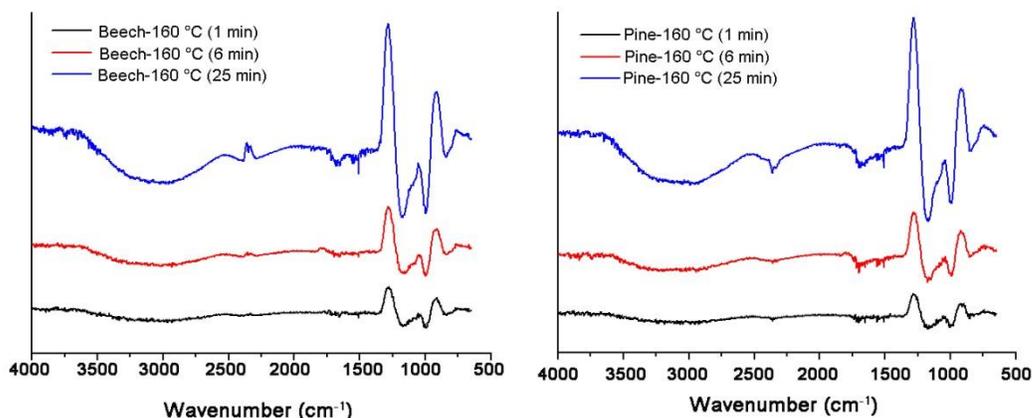


Fig. 4. FTIR spectra for samples treated at 160 °C

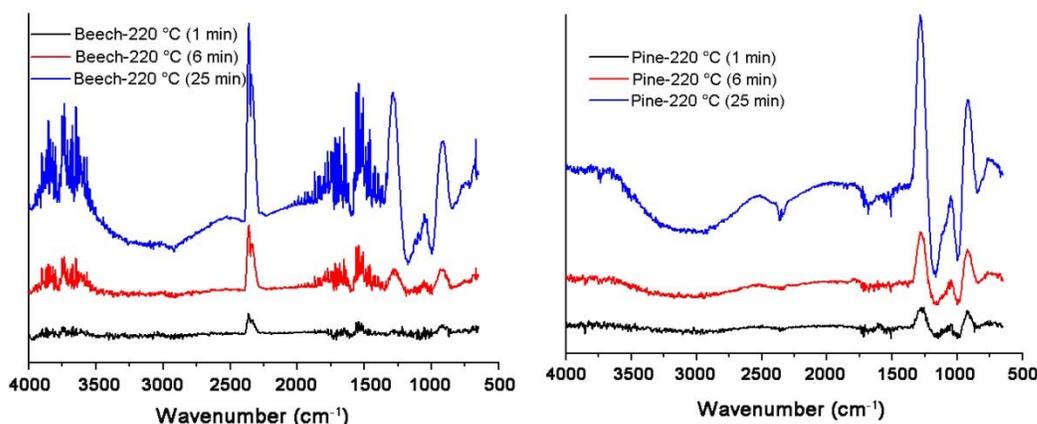


Fig. 5. FTIR spectra for samples treated at 220 °C

Figures 4 and 5 indicate that the FTIR spectra of the pine samples treated at 160 °C and 220 °C were similar, whereas the FTIR spectra of the beech samples treated at 160 °C and 220 °C were remarkably different. The intensity of the gas components in all of the samples increased as time increased. Many small peaks occurred in the ranges 1800 cm^{-1} to 1300 cm^{-1} and 4000 cm^{-1} to 3500 cm^{-1} , which corresponded to -OH stretching in the pine samples under all conditions, in beech samples at 160 °C, and at the initial stage of 220 °C. Moreover, visible absorbance peaks around these wavenumbers were found in the beech samples treated at 220 °C at 6 min and 25 min. Peaks around 1800 cm^{-1} to 1300 cm^{-1} and 4000 cm^{-1} to 3500 cm^{-1} represent the evaporation of free water and bound water, or methanol in the material (Liu *et al.* 2008; Werner *et al.* 2014). The water components are generated by the cracking of aliphatic hydroxyl groups in the lateral chains (Liu *et al.* 2008), cross-linking reactions that occur within and between glucose molecules, and the dehydroxylation in hemicellulose and cellulose (Yang *et al.* 2018). Thus, water or other hydroxy components were generated during the entire process (Yao *et al.* 2017). The decrease in hydroxyl group content of the remaining wood can explain the improvement in the wood's dimensional stability and the decreasing hygroscopicity caused by wood thermal modification. Moreover, the absorbance intensity increased with increases in temperature and time for the beech samples, which indicated strong progression of a dehydration reaction; this could have been a prelude to char formation (Shen and Gu 2009; Zhang *et al.* 2015). The formation of alcohols should be

assigned to the O-methyl group linked to the C4 position of the glucuronic acid unit (Ponder and Richards 1991) or cleavage of the methyl group in lignin (Popescu *et al.* 2018).

In addition, absorbance peaks around 1280 cm^{-1} , 1050 cm^{-1} , and 900 cm^{-1} , corresponding to C-O and C-C stretching, were observed in the beech and pine samples at 160 °C and 220 °C. These absorbance peaks represented the evolution of ketones, ethers, acids, and aromatics (Liu *et al.* 2008; Shen *et al.* 2010; Gao *et al.* 2013; Zhang *et al.* 2016). These gas components were generated by the decomposition of levoglucosan, hydroxyacetone, or 5-(hydroxymethyl)-furfural, as well as other components in wood (Piskorz *et al.* 1989; Shen and Gu 2009; Yang *et al.* 2018).

Visible absorbance peaks around 2360 cm^{-1} , corresponding to C=O stretching, were observed in the beech samples treated at 220 °C, and at the end stages of treatment at 160 °C; meanwhile, no such peaks were observed in the pine samples under all conditions. This wavenumber represents the evolution of CO₂ (Yang *et al.* 2018), which is primarily generated by the decarboxylation reaction and breakage of carbonyl groups, and the decomposition of dehydrocellulose in cellulose at low temperatures (Yang *et al.* 2018), or the decarboxylation of -COOH groups on glucuronic acid units (Patwardhan *et al.* 2011). It is also generated by cracking and reforming of the C=O and -COOH groups (Yang *et al.* 2007), or the decarboxylation of O-acetyl groups linked to xylan (Shen *et al.* 2010), or the cleavage of the acetyl groups and carbonyl groups from the xylan chain of hemicellulose (Yang *et al.* 2007). The decomposition of hemicellulose, cellulose, or lignin due to the results of a decrease in water uptake, volumetric swelling values (Croitoru *et al.* 2018), and the decrease of wood hygroscopicity (Li *et al.* 2017) increase the opportunity of protection against fungal colonization and biodeterioration (Wang *et al.* 2018).

Gas Products at Different Times

To compare the variation in gas products between beech and pine under different conditions, the characteristics of gas product emissions over time were investigated; these are presented in Figs. 6 and 7. The quantities of most gaseous products increased over time, and this result was consistent with existing research (Popescu *et al.* 2018). The peak of gaseous product emissions could not be reached because the samples absorbed much more energy over time. Meanwhile, the peak decomposition temperature for cellulose, hemicellulose, and lignin could not be reached under the thermal treatment employed (Zhao *et al.* 2017). Some new components generated at the initial stage of the thermal treatment, such as acetic acid, could increase the rate of decomposition of hemicellulose, cellulose, and lignin over time (Tjeerdsma *et al.* 1998). The most gaseous products of the beech and pine samples treated at 160 °C were generated at 1280 cm^{-1} , representing ketones, ethers, acids, and aromatics. The least gaseous products were generated around 3750 cm^{-1} , representing water or alcohol. The gaseous products obtained from the pine samples treated at 160 °C and 220 °C were similar. However, most gaseous products obtained from the beech samples treated at 220 °C were generated around 2360 cm^{-1} , representing CO₂.

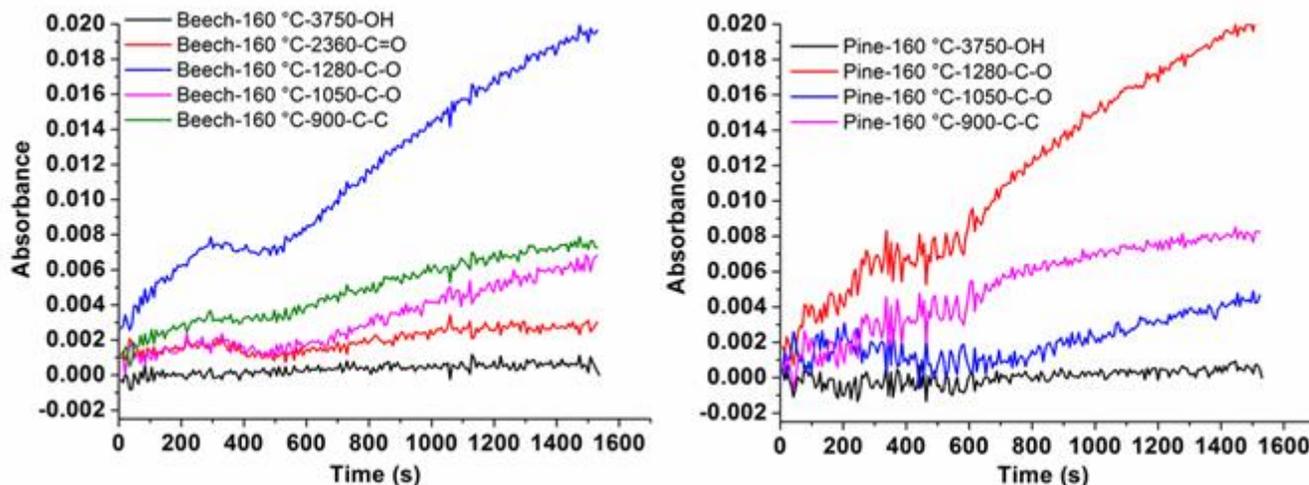


Fig. 6. FTIR spectrum of the gaseous products emissions at 160 °C

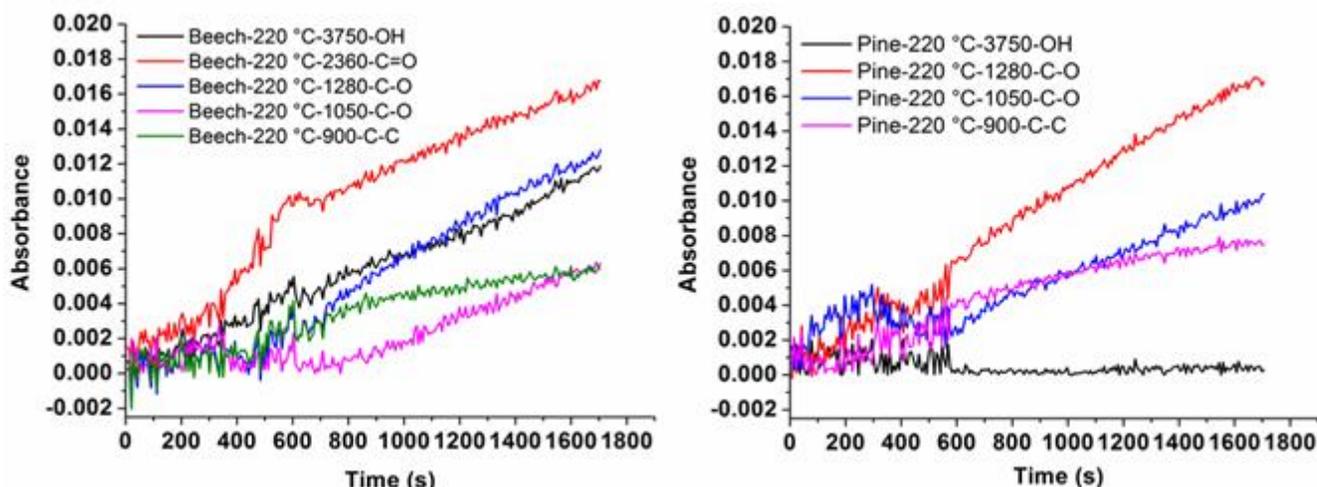


Fig. 7. FTIR spectrum of the gaseous products emissions at 220 °C

The lowest yield in gaseous products obtained from the beech samples was generated at 1050 cm^{-1} , representing ketones, ethers, acids, and aromatics. Moreover, most wood components of beech samples decomposed at 220 °C.

CONCLUSIONS

1. The mass loss increased with increases in temperatures and time. For the samples treated at 160 °C and 220 °C, the total mass losses were approximately 3% and 4.7% to 6.5%, respectively. More species of gas products were emitted from the beech (hardwood) than from pine (softwood), and these species increased with increases in temperature and time.
2. The FTIR spectra of the pine samples heated at different temperatures were similar, whereas those of the beech samples were remarkable different under thermal treatment at 160 °C and 220 °C. A large number of small peaks occurred at 1800 cm^{-1} to 1300 cm^{-1} and 4000 cm^{-1} to 3500 cm^{-1} , which corresponded to -OH stretching in

the pine samples under all the conditions, and in the beech samples at 160 °C, and at the initial stage of thermal treatment at 220 °C. Visible absorbance peaks around 1280 cm⁻¹, 1050 cm⁻¹, and 900 cm⁻¹, corresponded to C-O and C-C stretching, and were observed in both the beech and pine samples under all conditions. Visible absorbance peaks around 2360 cm⁻¹ that corresponded to C=O stretching were observed in the beech samples treated at 220 °C, and at the final stage of 160 °C. Meanwhile, no such peaks were observed in pine under all conditions.

3. Acid, water, or methanol, ketones, ethers, carbon dioxide, and aromatics were the main gaseous products during thermal modification. Most of the gaseous products of the beech and pine samples at 160 °C were generated at around 1280 cm⁻¹; the gaseous products of the pine samples were similar at 160 °C and 220 °C. These results can be used to guide the design of an improved wood thermal modification schedule.

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