

## Physical and Chemical Characteristics of the Bamboo Culm and Wood Carbonized at Low Temperature

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Physical and chemical characteristics of bamboo culm heat-treated at low temperature (200 °C to 320 °C) were analyzed to understand their carbonization mechanisms. The physical features and chemical compositions of four Indonesian and one Korean bamboo species were examined, and they were compared with red pine and cork oak after treatment. Weight loss, pH, and calorific value increased with increasing temperature. Weight loss and calorific value of the bamboo species increased from 200 to 240 °C, while those of the wood species increased from 240 to 280 °C. The pH of the bamboo and wood changed from acidic to neutral at 280 and 320 °C, respectively. The crystalline cellulose of the samples in the X-ray diffraction analysis changed to amorphous at various temperatures for each material. In Fourier transform infrared spectroscopy, all species showed a considerable change in hemicellulose peaks at 280 °C and a substantial change in cellulose peaks at 320 °C. In conclusion, the carbonized bamboo culm showed a considerable difference, at 200 °C to 320 °C, in the physical and chemical characteristics.

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### INTRODUCTION

Bamboo is primarily an alternative eco-friendly green material for wood with various advantages, such as rapid growth, short rotation age, and high tensile strength (Febrianto *et al.* 2015). Moreover, the proportions of chemical components in bamboo is different from that of wood. According to Azeez and Orege (2018), bamboo has a chemical composition of 74% cellulose, 13% hemicellulose, and 10% lignin, with a higher content of minor components compared to wood. By contrast, softwood is composed of about 40% to 44% cellulose, 15% to 35% hemicellulose, and 18% to 25% lignin. Hardwood is composed of about 40% to 44% cellulose, 20% to 32% hemicellulose, and 25% to 35% lignin (Haygreen and Bowyer 1982). Overall, bamboo has higher cellulose content and lower lignin and hemicellulose than wood.

Although the chemical compositions of bamboo and wood are different, the pyrolysis process is similar because they both contain organic components such as hemicellulose, cellulose, and lignin (Krzyszowska *et al.* 2008). In bamboo, hemicellulose is decomposed in the temperature range of 250 to 320 °C, cellulose at 320 to 380 °C, and lignin in the temperature range from ambient to 900 °C (Mahanim *et al.* 2011). In the pyrolysis process of wood, the main components decompose at various temperatures, such as hemicellulose at 180 to 300 °C, cellulose at 240 to 400 °C, and lignin within the temperature range of 280 to 550 °C (Kollmann and Fengel 1965). The rate of mass loss was in the order of cellulose, hemicellulose, and then lignin. The higher the decomposition rate, the lower is the residual content (Myung *et al.* 2004).

Many studies on the anatomical and physical properties of wood carbonized at high temperatures in the range of 200 to 1200 °C have been conducted (Kim and Hanna 2006; Kwon and Kim 2006; Jo *et al.* 2007; Kwon *et al.* 2009, 2012, 2013; Qi *et al.* 2016), with a view to understand the carbonization mechanisms. In addition, several studies have been performed to clarify the mechanism of carbonization from wood to charcoal at low temperatures in the range of 130 to 400 °C (Kwon and Kim 2007; Ates *et al.* 2009; Park *et al.* 2012; Kwon *et al.* 2014; Wilk *et al.* 2016; Lin *et al.* 2018; Poudel *et al.* 2018; Kim *et al.* 2018).

Many studies have been conducted on the properties of carbonized bamboo at various carbonization temperatures, such as the physical and mechanical properties (Hirose *et al.* 2002), elemental and chemical structural analyses (Zuo *et al.* 2003), ammonia adsorption (Asada *et al.* 2006), charcoal yield, calorific values and proximate analysis (Saikia *et al.* 2007), activated carbon properties (Mehanim *et al.* 2011), pH value, Brunauer-Emmett-Teller (BET) surface area, iodine value, pore volume (Huang *et al.* 2014), physicochemical, and fuel properties (Park *et al.* 2020).

However, few studies have been conducted on the transition characteristics of bamboo to charcoal. Rousset *et al.* (2011) investigated the effects of torrefaction on the combustible properties of *Bambusa vulgaris* at 220, 250, and 280 °C. They reported that the gross calorific value of torrefied *B. vulgaris* culm increased with increasing temperature, while the energy yield decreased with increasing temperature. The study revealed that torrefaction at 280 °C changed the chemical composition of bamboo the most. Jiang *et al.* (2013) reported that mass loss, inorganic ash, gross calorific value, combustion rate, and heat release rate of carbonized bamboo pellets increased with increasing carbonization temperature from 180 to 220 °C.

To establish basic data for further utilization of eco-friendly biomass resources such as bamboo, the pyrolysis mechanism from bamboo to charcoal needs to be elucidated. Additionally, to date, there is no comparative study on the carbonization mechanism between bamboo culm and wood carbonized at low temperature. Therefore, in this study, the physical characteristics and chemical structure of bamboo culms carbonized at 200, 240, 280, and 320 °C were examined using four Indonesian and one domestic bamboo species and compared with Korean red pine (*Pinus densiflora*) and cork oak (*Quercus variabilis*) wood species to understand the differences between the carbonization process of bamboo culm and wood.

## EXPERIMENTAL

### Materials

Three bamboo culms from four Indonesian bamboo species aged 3 years old, such as giant bamboo (*Dendrocalamus giganteus*), betung bamboo (*Dendrocalamus asper*), kuning bamboo (*Bambusa vulgaris*), and tali bamboo (*Gigantochloa apus*), were obtained from the bamboo arboretum at the Institut Pertanian Bogor (West Java, Indonesia, 6°20'21" S, 106°33'58" E). Three bamboo culms of a 3-year-old moso bamboo (*Phyllostachys pubescens*) were harvested in Damyang, Korea. (Damyang, 35°18' N, 126°54' E). Fifty-year-old Korean red pine (*Pinus densiflora*) and 35-year-old cork oak (*Quercus variabilis*) trees were harvested from the research forest of Kangwon National University (Korea, Chuncheon, 37°77'N, 127°81'E), to compare the properties of bamboo with wood. The diameter at breast height of the wood species was 47.3 cm for Korean red pine and 17.2 cm for cork oak.

The bamboo culm samples were divided into top, middle, and bottom parts. The bottom parts were used as materials in this study. The fundamental information, density, and air-dried moisture content of the bamboo culms at the bottom are shown in Tables 1 and 2.

**Table 1.** Information of Bottom Parts of the Bamboo Culms

	Giant Bamboo	Betung Bamboo	Kuning Bamboo	Tali Bamboo	Moso Bamboo
Diameter (mm)	146.6±1.5	131.3±6.5	79.9±3.5	86.3±1.1	83.1±2.1
Thickness (mm)	17.2±0.5	23.7±1.2	14.1±1.2	11.6±0.4	9.7±4.2
Height (m)	25-35	20-30	10-20	8-22	10-20

**Table 2.** Density and Air-dried Moisture Content of the Samples

Species	Oven-dried Density (g/cm <sup>3</sup> )	Air-dried Moisture Content (%)
Giant bamboo	0.61±0.04	8.85±0.38
Betung bamboo	0.67±0.03	7.97±1.07
Kuning bamboo	0.61±0.04	8.07±1.41
Tali bamboo	0.67±0.04	8.71±0.78
Moso bamboo	0.63±0.05	9.10±2.20
Korean red pine	0.51±0.06	9.23±2.74
Cork oak	0.77±0.03	10.52±0.39

### Carbonization Method

Samples of bamboo culm and wood with dimensions of 20 mm (L) × 10 mm (R) × 10 mm (T) were carbonized in an electric furnace (Supertherm, HT 16/16, Germany) at 200, 240, 280, and 320 °C. The samples were heated from 40 °C to the target temperature at a heating rate of 6 °C/min. After reaching the target temperature, the samples were kept at that temperature for 10 min and then left in the furnace until cooled and then stored in a room at 20±3 °C and with relative humidity (RH) at 50±5%.

### Measurement of Weight Loss

Six samples from each species and temperature were used to measure weight loss. The weight loss of the samples was calculated using Eq. 1 (Kwon *et al.* 2009),

$$\text{Weight loss (\%)} = \frac{W_1 - W_2}{W_1} \times 100 \quad (1)$$

where  $W_1$  is the weight of the original sample (g), and  $W_2$  is the weight of the carbonized sample (g).

### Measurement of Calorific Value

The calorific values were measured with a calorimeter (Oxygen Bomb Calorimeter 6400, Parr, USA) using 60 mesh carbonized powder (0.5 g). Before measuring the calorific value, the equipment was corrected using benzoic acid (6.321 cal/g) (Lee *et al.* 2019). The measurements were performed three times for each sample.

### Measurement of pH Value

Carbonized powder samples (1 g) were mixed with 100 mL of DI water and boiled for approximately 10 min. After cooling, the pH value was determined using a pH meter (PB-10, Germany) (Lee *et al.* 2019). The measurements were performed three times for each sample.

### Measurement of Crystalline Properties

The crystalline properties of carbonized bamboo and wood samples were measured with an X-ray diffractometer (D-Max 2100V, Rigaku, Japan,  $Cu\alpha$ , 40 kV, 30 mA) using 60 mesh sample powder. The measurement was carried out three times for each sample. The relative crystallinity was calculated using the Segal method (Segal *et al.* 1959), as shown in Eq. 2,

$$CR (\%) = \frac{(I_{200} - I_{am})}{I_{200}} \times 100 \quad (2)$$

where  $CR$  is crystallinity (%),  $I_{200}$  is the crystalline intensity, and  $I_{am}$  is the amorphous intensity.

The crystallite width was determined using the Scherrer equation (Scherrer 1918), as shown in Eq. 3,

$$L(hkl) = \frac{K \cdot \lambda}{\beta \cdot \cos\theta} \quad (3)$$

where  $L$  is the crystallite width (nm),  $K$  is the Scherrer constant (0.9),  $\lambda$  is the X-ray wavelength ( $\lambda = 0.1542$  nm),  $\beta$  is the half-width (radian), and  $\theta$  is the Bragg angle.

### Fourier Transform Infrared Spectra Analysis

The chemical composition of bamboo and wood was analyzed using 60 mesh sample powders for each temperature (control, 240 °C, 280 °C, and 320 °C). In addition, the chemical composition of all samples was examined using the ATR method in the range of 400 to 4000  $\text{cm}^{-1}$  with an FT-IR spectrometer (Nicolet Summit, USA) installed at the Department of Forest Biomaterials Engineering at Kangwon National University. The FT-IR spectra of each sample were analyzed with Microsoft excel 2016 (Microsoft Corp., Santa Rosa, USA).

## Statistical Analysis

The significant differences in weight loss, calorific values, pH values, and crystalline properties at various temperatures were statistically examined by analysis of variance (ANOVA) and post-hoc Duncan's multiple range tests. Statistical analyses were performed using the SPSS software (SPSS ver. 24, IBM Corp., Armonk, NY, USA).

## RESULTS AND DISCUSSION

### Visual Observation of Color Change

Figure 1 shows the macroscopic structure of the samples heat-treated at 200 to 320 °C. Both bamboo and wood specimens showed a clear color change at all temperatures. The color of the sample was yellowish-brown at 200 °C and it became almost black at 240 °C. Finally, all the carbonized samples showed heavy black color at 280 and 320 °C. Shangguan *et al.* (2016) reported that in *Neosinocalamus affinis* bamboo at 3 to 4 years old, the original light color of the samples changed to dark brown at 200 and 230 °C. Li *et al.* (2015) mentioned that the color of *Phyllostachys sulphurea* bamboo torrefied at 280, 300, 320 and 340 °C changed from light brown to dark brown and heavy black as the increase of torrefaction temperature affected structural modification of lignin. Ates *et al.* (2009) examined Calabrian pine (*Pinus brutia*) wood at 130, 180, and 230 °C, and reported that the color of the wood darkened due to increasing lignin content as the heat treatment temperature was increased.



**Fig. 1.** Appearances of the carbonized samples at different temperatures. Black arrow indicates crack in Korean red pine and split in cork oak

Another characteristic of the carbonized samples was the occurrence of a split in the samples. The split was hardly observed in the bamboo samples. Severe splits were often found on the transverse surface of cork oak wood. The Korean red pine carbonized at 320 °C showed cracks on the tangential surface. It could be caused by the difference in

anatomical characteristic between bamboo and wood. Kwon *et al.* (2009) reported that in *Quercus variabilis* wood carbonized at 250 to 500 °C, splits and cracks occurred frequently along the broad ray as the carbonization temperature was increased. Kwon *et al.* (2014) found that the volume of three hardwoods and two softwoods carbonized at 300 to 350 °C decreased as the carbonization temperature was increased and the split was not observed in softwood. According to Byrne and Nagle (1997), typically wood charcoal is broken and cracked due to the shrinkage stresses occurred at the surface of the material decomposing faster than the interior.

### Weight Loss

Table 3 shows the weight loss of the samples at different carbonization temperatures. The weight loss of all samples increased with increasing temperature. Bamboo samples showed an approximately 40% increase in weight loss from 200 to 240 °C, while Korean red pine and cork oak wood showed a relatively small increase of approximately 20%. The weight loss of all the samples increased by approximately 50% to 60% at 280 and 320 °C. There were significant temperature differences for the change in weight loss between bamboo and wood species between 200 and 240 °C. According to Shangguan *et al.* (2016), the weight of heat-treated *Neosinocalamus affinis* bamboo decreased significantly at 170 to 230 °C. Meng *et al.* (2016) described that the weight loss of 4 years old *Phyllostachys pubescens* culm heat-treated at 180 to 200 °C increased with increasing temperature. Kwon *et al.* (2009) and Kim and Hanna (2006) also reported that the weight loss of *Quercus variabilis* wood increased as the temperature was increased during the carbonization process. The change in weight loss due to the increase in carbonization temperature can be explained by the change in chemical composition. According to Meng *et al.* (2016), as temperature increases from ambient temperature to 200 °C,  $\alpha$ -Cellulose and holocellulose in bamboo decreased from 41.5 to 26.6% and from 67.1 to 53.5%, respectively, and lignin increased from 22.1 to 31.7%. During thermal degradation process, hemicellulose decomposed at the temperature between 200 and 300 °C, followed by cellulose above 240 °C and lignin above 280 °C (Beall and Eickner 1970).

**Table 3.** Weight Loss of the Samples at Different Temperatures

Species	Weight Loss (%)			
	200 °C	240 °C	280 °C	320 °C
Giant Bamboo	14.2±1.9 <sup>a</sup>	48.8±9.9 <sup>b</sup>	52.5±5.2 <sup>bc</sup>	56.1±1.9 <sup>c</sup>
Betung Bamboo	6.2±1.7 <sup>a</sup>	42.4±6.8 <sup>b</sup>	58.9±2.4 <sup>c</sup>	59.1±1.4 <sup>c</sup>
Kuning Bamboo	6.6±1.2 <sup>a</sup>	39.0±4.7 <sup>b</sup>	54.8±1.0 <sup>c</sup>	59.1±0.5 <sup>d</sup>
Tali Bamboo	4.2±0.9 <sup>a</sup>	37.9±4.1 <sup>b</sup>	57.6±1.3 <sup>c</sup>	59.9±0.7 <sup>d</sup>
Moso Bamboo	8.6±2.6 <sup>a</sup>	42.5±3.1 <sup>b</sup>	55.1±0.8 <sup>c</sup>	55.8±0.8 <sup>c</sup>
Korean Red Pine	6.3±1.0 <sup>a</sup>	20.3±6.4 <sup>b</sup>	62.7±0.8 <sup>c</sup>	68.6±1.1 <sup>d</sup>
Cork Oak	1.9±0.6 <sup>a</sup>	23.9±1.8 <sup>b</sup>	49.4±4.7 <sup>c</sup>	59.1±2.3 <sup>d</sup>

Note: The same superscript lowercase letters beside the mean values denotes insignificant outcomes at the 5% significance level for comparisons among temperature using Duncan's multiple range tests.

### Calorific Value

Table 4 shows the calorific values of the samples carbonized at various temperatures. The control samples showed almost identical calorific values of

approximately 17 to 18 MJ/kg. Engler *et al.* (2012) and Chen *et al.* (2014) have reported similar effects that the gross calorific value of moso bamboo was 19.44 MJ/kg and 16.85 MJ/kg, respectively. Liu *et al.* (2013) reported that the calorific value of *Quercus variabilis* ranged from approximately 19.24 to 19.46 MJ/kg at different tree heights.

**Table 4.** Calorific Values of the Samples at Different Temperatures

Species	Calorific Value (MJ/kg)				
	Control	200 °C	240 °C	280 °C	320 °C
Giant Bamboo	16.8±82 <sup>a</sup>	18.1±27 <sup>b</sup> (7.8%)	21.9±06 <sup>c</sup> (30.4%)	23.2±31 <sup>d</sup> (38.1%)	24.6±24 <sup>e</sup> (46.4%)
Betung Bamboo	17.8±18 <sup>a</sup>	18.2±23 <sup>a</sup> (2.2%)	22.4±35 <sup>b</sup> (25.8%)	26.5±53 <sup>c</sup> (48.9%)	26.7±17 <sup>c</sup> (50.0%)
Kuning Bamboo	17.4±36 <sup>a</sup>	18.3±45 <sup>b</sup> (5.2%)	22.8±40 <sup>c</sup> (31.0%)	25.9±32 <sup>d</sup> (48.85%)	27.1±34 <sup>e</sup> (55.8%)
Tali Bamboo	17.8±24 <sup>a</sup>	19.2±41 <sup>b</sup> (7.9%)	22.0±80 <sup>c</sup> (23.6%)	27.8±24 <sup>d</sup> (56.2%)	28.3±72 <sup>e</sup> (60.0%)
Moso Bamboo	17.9±51 <sup>a</sup>	19.3±59 <sup>b</sup> (7.8%)	23.6±92 <sup>c</sup> (31.8%)	26.6±15 <sup>d</sup> (48.6%)	27.5±24 <sup>e</sup> (53.6%)
Korean Red Pine	18.3±67 <sup>a</sup>	18.4±69 <sup>a</sup> (0.5%)	19.5±22 <sup>b</sup> (6.6%)	24.1±27 <sup>c</sup> (31.7%)	26.0±28 <sup>d</sup> (42.1%)
Cork Oak	18.3±56 <sup>a</sup>	18.8±55 <sup>a</sup> (2.7%)	20.5±12 <sup>b</sup> (12.0%)	25.0±21 <sup>c</sup> (36.6%)	27.0±18 <sup>d</sup> (47.5%)

Note: Numbers in parentheses indicate the increasing rate of the caloric value. The listing of the same superscript lowercase letters beside the mean values denotes insignificant outcomes at the 5% significance level for comparisons among temperature using Duncan's multiple range tests.

For all the specimens, the calorific value increased as the carbonization temperature was increased. The calorific value of bamboo significantly increased at temperatures ranging from 200 to 240 °C. In contrast, the calorific value of Korean red pine and cork oak increased in the temperature range of 240 to 280 °C.

There have been several studies on the increase in the calorific value of woody biomass with increasing treatment temperature. Chen *et al.* (2017) found that the heating value of *Phyllostachys makinoi* bamboo torrefied from 206 to 300 °C increased from 18.53 to 28.3 MJ/kg. Rousset *et al.* (2011) reported that the average gross calorific value of Kuning bamboo carbonized at 220, 250, and 280 °C increased with increasing temperature as 19.8, 21.5, and 24.0 MJ/kg, respectively. Sridhar *et al.* (2007) reported that the calorific value of *Bambusa* species torrefied at 180 to 350 °C showed a significant increase by 20% at 250 °C, and the calorific value increased with increasing temperature. The authors mentioned that the increase in calorific value could be caused by a decrease of oxygen and an increase of carbon. Rodrigues and Rousset (2009) reported that the calorific value of *Eucalyptus grandis* wood torrefied at 220 to 280 °C greatly increased at 280 °C. Poudel *et al.* (2018) reported that the total calorific value of wood waste biomass torrefied at 200 to 400 °C increased as the temperature increased. They also mentioned that the increase in torrefaction temperature leads to a decrease the amount of oxygen and hydrogen, which increases the relative amount of carbon content. The increase in relative amount of carbon content provides a good CHO index correlated to an increase in calorific values. Wilk *et al.* (2016) also reported that the calorific values of woody biomass containing pine, oak, and birch torrefied at low temperatures of 230, 260, and 290 °C increased with increasing

temperature. Qi *et al.* (2016) reported that the calorific values of the branch wood of *Paulownia tomentosa* and *Pinus densiflora* carbonized at 400, 600, and 800 °C increased as the carbonization temperature was increased. The authors described that the calorific value increased as the number of C-C bonds with high energy increased.

### pH Value

Table 5 shows the pH values of the samples at different temperatures. The pH of control bamboo sample was slightly higher than that of wood, such as 5.05 to 5.33 in bamboos and 4.53 to 4.90 in woody samples. In all the specimens, the pH steadily increased as the carbonization temperature was increased. The transition temperature from acidic to neutral was slightly different for bamboo and wood species. The pH of the bamboo and wood changed to neutral at 280 °C and 320 °C, respectively.

**Table 5.** pH Value of the Samples at Various Temperatures

Species	pH				
	Control	200 °C	240 °C	280 °C	320 °C
Giant Bamboo	5.20±0.02 <sup>a</sup>	5.82±0.02 <sup>b</sup>	6.80±0.02 <sup>c</sup>	6.82±0.02 <sup>c</sup>	7.10±0.02 <sup>d</sup>
Betung Bamboo	5.30±0.02 <sup>a</sup>	5.93±0.02 <sup>b</sup>	6.76±0.02 <sup>c</sup>	7.10±0.02 <sup>d</sup>	7.59±0.02 <sup>e</sup>
Kuning Bamboo	5.05±0.01 <sup>a</sup>	5.45±0.02 <sup>b</sup>	6.69±0.03 <sup>c</sup>	6.76±0.03 <sup>d</sup>	6.82±0.02 <sup>e</sup>
Tali Bamboo	5.33±0.02 <sup>a</sup>	6.01±0.05 <sup>b</sup>	6.65±0.03 <sup>c</sup>	7.20±0.01 <sup>d</sup>	7.36±0.01 <sup>e</sup>
Moso Bamboo	5.22±0.02 <sup>a</sup>	5.79±0.02 <sup>b</sup>	6.70±0.02 <sup>c</sup>	7.34±0.01 <sup>d</sup>	7.52±0.02 <sup>e</sup>
Korean Red Pine	4.53±0.01 <sup>a</sup>	5.02±0.01 <sup>b</sup>	5.71±0.02 <sup>c</sup>	6.11±0.03 <sup>d</sup>	7.04±0.02 <sup>e</sup>
Cork Oak	4.90±0.02 <sup>a</sup>	5.78±0.02 <sup>b</sup>	6.39±0.03 <sup>c</sup>	6.85±0.02 <sup>d</sup>	6.90±0.03 <sup>e</sup>

Note: The listing of the same superscript lowercase letters beside the mean values denotes insignificant outcomes at the 5% significance level for comparisons among temperature using Duncan's multiple range tests.

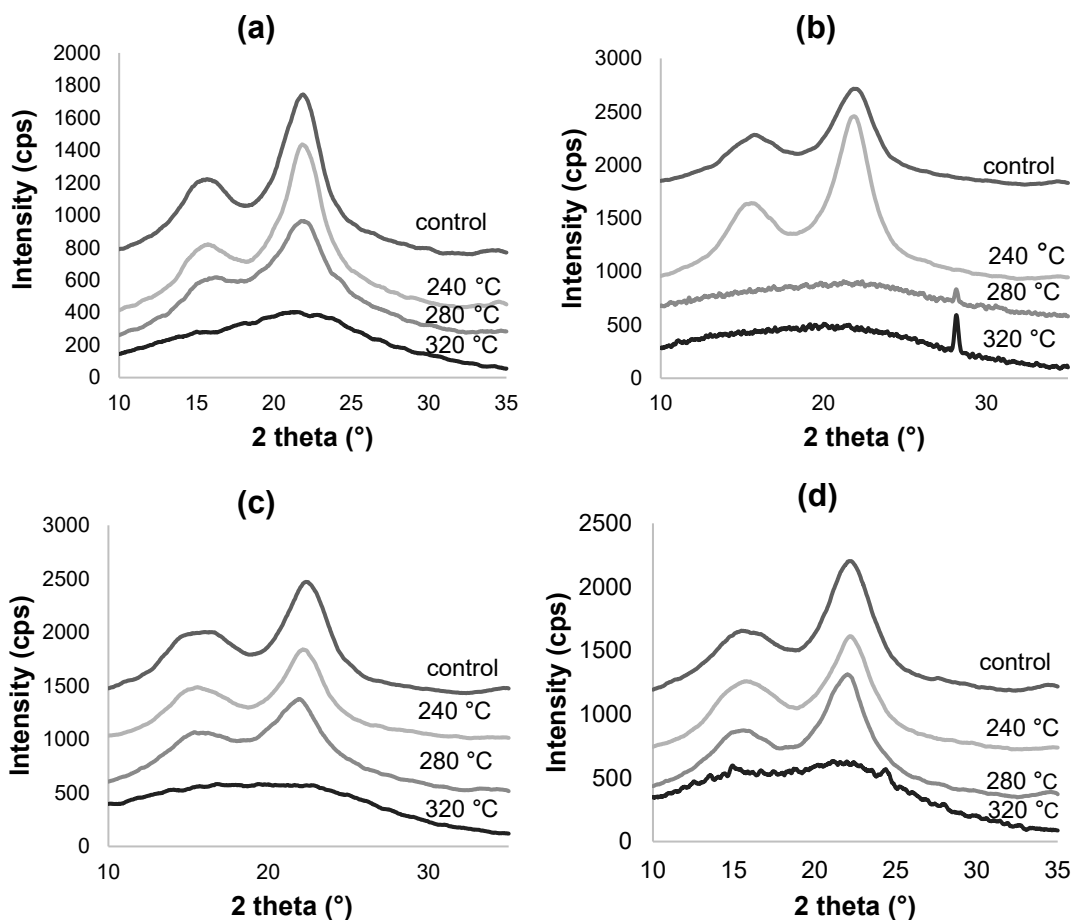
Several studies have been conducted on the transition temperature of woody materials from acidic to neutral, showing some variances between researchers. According to Jo *et al.* (2007), the pH value of *Abies sibirica* carbonized at 400 °C to 800 °C increased with increasing temperature, showing a change from acidic to alkaline at 500 °C. Zhang *et al.* (2017) found that the pH value of moso bamboo carbonized at 200 to 800 °C was 5.46 at 200 °C and 7.12 at 300 °C, which could change to neutral at 300 °C. The authors reported that the increase in pH with increasing carbonization temperature could be due to the increase in ash content, which is rich in alkali and alkaline earth metals (*e.g.*, K, Ca, Na, Mg). Jo *et al.* (2009) reported that the pH values of *Quercus mongolica* wood and *Larix leptolepis* bark carbonized at 300, 600, and 900 °C were low-acidic or neutral at 300 °C and alkaline above 600 °C. According to the authors, as the carbonization temperature increased, the ratio of C-C bonding increased, while that of the C-O-H and C-O-R bonding significantly decreased. Kwon *et al.* (2012) reported that the pH value of the cork oak was



neutral at 400 °C, and the pH value increased as the carbonization temperature was increased. Kwon *et al.* (2013) found that the pH value of cork oak carbonized at 200 to 740 °C increased with increasing carbonization temperature and showed low-acidity until 340 °C. It changed to neutral at temperatures above 540 °C.

### Crystalline Properties

Figure 2 shows the X-ray diffractograms of the carbonized samples. The cellulosic crystalline substances of giant bamboo, tali bamboo, and Korean red pine were present until 280 °C, and they changed to amorphous materials at 320 °C. Betung bamboo, kuning bamboo, and moso bamboo showed amorphous diffraction patterns at 280 °C. The cellulosic crystalline substance of cork oak remained at 320 °C, although none of the other species was present. The results of this study on cork oak are in line with those of Kwon and Kim (2006, 2007). These authors have found that in *Quercus variabilis* wood, the transition of cellulosic crystalline substance to an amorphous form occurred at around 340 °C. Kwon and Kim (2007) noted that *Quercus variabilis* wood carbonized at 310 °C to 350 °C showed cellulosic crystalline peaks up to 340 °C in X-ray analysis. The cellulosic crystalline nature changed to amorphous at 350 °C.



**Fig. 2.** X-ray diffractograms of the carbonized samples. (a) giant and tali bamboo, (b) moso, betung, and kuning bamboo, (c) Korean red pine, and (d) cork oak

Table 6 shows the relative crystallinities of all the samples at each carbonization temperature. There were some differences in each sample according to the carbonization temperature. The relative crystallinities of all the samples were almost constant from the control to 240 °C. On the other hand, the crystallinity of giant bamboo, tali bamboo, Korean red pine, and cork oak showed a significant decrease at 280 °C, and that of tali bamboo showed the highest decrease among the species. The relative crystallinity of cork oak at 320 °C was 15.17%, while the other species showed no cellulosic crystalline substances at 320 °C. Regarding the relative crystallinity with heat treatment, Kim *et al.* (2001) reported that the relative intensity of crystal reflections and amorphous background of *Populus maximowiczii* heat-treated from 100 to 360 °C gradually decreased with increasing temperature until 320 °C. Above 320 °C, the intensity decreased rapidly and became zero at 360 °C. Jiang *et al.* (2012) investigated the crystallinity index of heat-treated moso bamboo particles at 160 to 450 °C, gradually increased from 160 to 265 °C, and it rapidly decreased from 315 to 450 °C. These authors mentioned that, at 160 to 265 °C, the crystalline region of cellulose was not degraded but absorbed water. Small molecule hemicelluloses and the amorphous region of cellulose gradually degraded. Furthermore, the cellulosic crystalline region started to degrade at 315 °C and was destroyed at 450 °C. In addition, Zheng *et al.* (2017) reported that the relative crystallinity of torrefied Yunnan pine at 210 to 300 °C was constant from control to 230 °C and showed a noticeable decrease after 250 °C.

**Table 6.** Relative Crystallinity of the Samples at Different Temperatures

Species	Relative Crystallinity (%)			
	Control	240 °C	280 °C	320 °C
Giant Bamboo	71.17±2.28 <sup>b</sup>	68.94±0.09 <sup>b</sup>	50.95±0.83 <sup>a</sup>	-
Betung Bamboo	70.95±0.83 <sup>a</sup>	70.59±0.47 <sup>a</sup>	-	-
Kuning Bamboo	67.34±1.47 <sup>a</sup>	69.02±1.54 <sup>a</sup>	-	-
Tali Bamboo	69.00±1.87 <sup>b</sup>	68.92±0.64 <sup>b</sup>	35.92±0.56 <sup>a</sup>	-
Moso Bamboo	67.36±2.08 <sup>a</sup>	68.42±0.42 <sup>a</sup>	-	-
Korean Red Pine	65.19±2.89 <sup>b</sup>	69.20±1.19 <sup>b</sup>	42.56±0.65 <sup>a</sup>	-
Cork Oak	68.37±2.30 <sup>c</sup>	68.96±1.53 <sup>c</sup>	56.37±0.67 <sup>b</sup>	15.17±2.68 <sup>a</sup>

Note: The same superscript lowercase letters beside the mean values denotes insignificant outcomes at the 5% significance level for comparisons among temperature using Duncan's multiple range tests.

Table 7 shows the crystallite widths of all samples at each carbonization temperature. The crystallite width increased slightly from the control to 240 °C, except for giant bamboo. In addition, the crystallite widths of giant bamboo, tali bamboo, Korean red pine, and cork oak decreased at 280 °C. The crystallite width of cork oak at 320 °C was 0.71 nm, while the cellulosic crystalline substances of other species were absent at 320 °C. According to Chang *et al.* (2012), the crystallite size of torrefied spruce wood at 260 to 300 °C increased at 280 °C and remained constant until 300 °C. Furthermore, the authors

described that the increase in crystalline size was mainly due to small and imperfect cellulose micro-crystallite decomposition, and large and perfect cellulose micro-crystallites remained in the torrefied biomass.

**Table 7.** Crystallite Width of the Samples at Different Temperature

Species	Crystallite Width (nm)			
	Control	240 °C	280 °C	320 °C
Giant Bamboo	2.32±0.06 <sup>b</sup>	2.36±0.03 <sup>b</sup>	1.95±0.14 <sup>a</sup>	-
Betung Bamboo	2.32±0.04 <sup>a</sup>	2.51±0.05 <sup>b</sup>	-	-
Kuning Bamboo	2.28±0.02 <sup>a</sup>	2.39±0.05 <sup>b</sup>	-	-
Tali Bamboo	2.30±0.04 <sup>a</sup>	2.64±0.03 <sup>b</sup>	2.24±0.25 <sup>a</sup>	-
Moso Bamboo	2.20±0.01 <sup>a</sup>	2.33±0.03 <sup>b</sup>	-	-
Korean Red Pine	2.07±0.05 <sup>a</sup>	2.29±0.08 <sup>b</sup>	2.04±0.04 <sup>a</sup>	-
Cork Oak	2.11±0.11 <sup>b</sup>	2.31±0.06 <sup>c</sup>	2.14±0.03 <sup>b</sup>	0.71±0.05 <sup>a</sup>

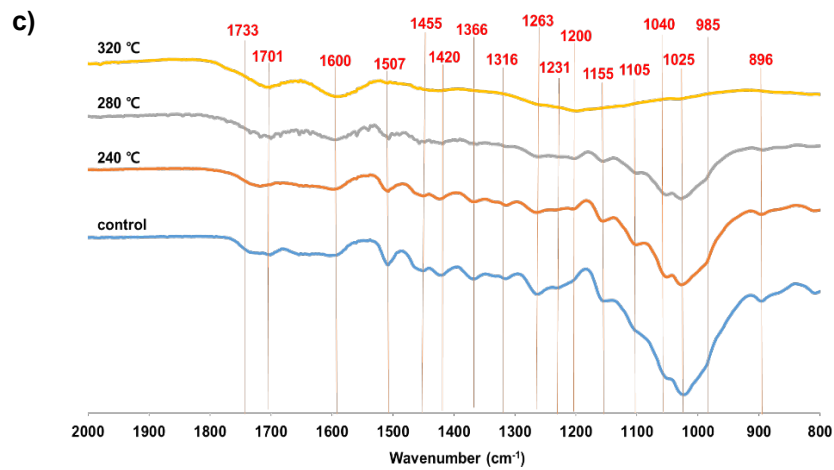
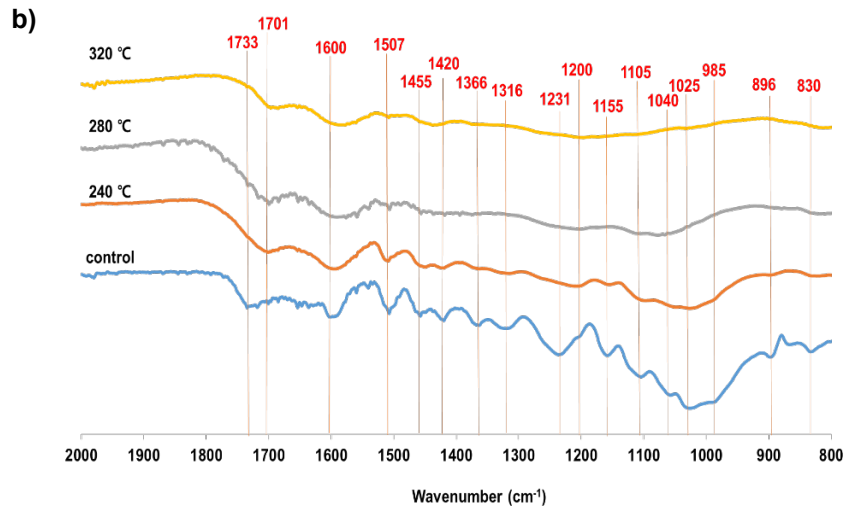
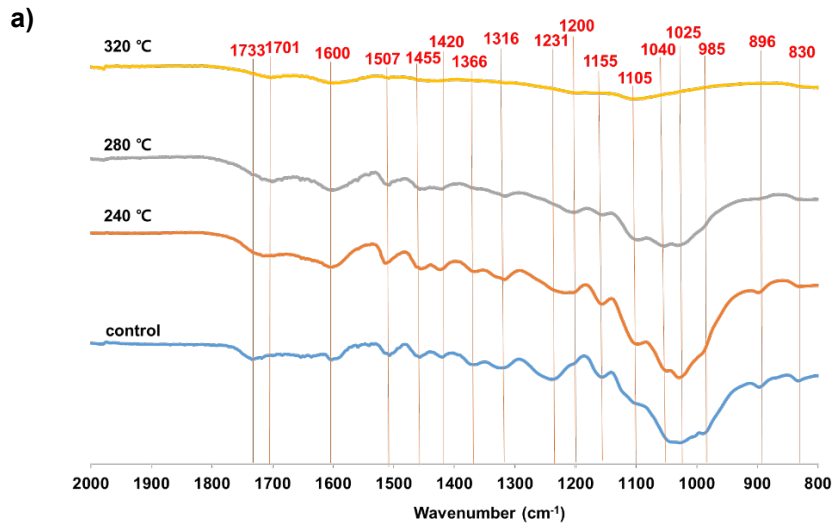
Note: The same superscript lowercase letters beside the mean values denotes insignificant outcomes at the 5% significance level for comparisons among temperature using Duncan's multiple range tests.

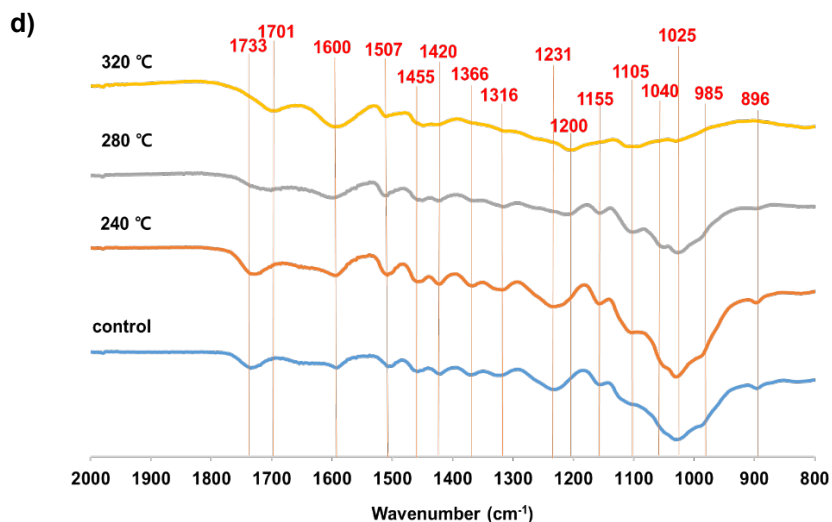
Jiang *et al.* (2012) reported that the crystallite width of heat-treated moso bamboo particles at 160 to 450 °C gradually increased from 160 to 265 °C and rapidly decreased from 315 to 450 °C. The authors explained that the increase in crystallite size of torrefied samples below 300 °C was due to the decomposition of amorphous hemicelluloses and amorphous regions of cellulose, and the rapid decrease in crystallite width after 300 °C was because of decomposition of the crystalline region. Hill *et al.* (2013) showed that the average crystallite lateral dimension of torrefied *Pinus radiata* wood at 220 to 300 °C increased from untreated wood to 250 °C and decreased at 275 °C. Zheng *et al.* (2017) reported that the crystallite size of torrefied Yunnan pine at 210 to 300 °C was gradually reduced from 230 to 300 °C.

### Chemical Structures

Figure 3 presents the FT-IR spectra of the samples according to the carbonization temperature, and the corresponding peak assignments are summarized in Table 8. There were differences in the carbohydrate peaks among the carbonized samples. The untreated bamboos and cork oak showed a peak at 1733 cm<sup>-1</sup> representing the –C=O stretching vibration in esters (unconjugated), ketones, aldehydes, and acids, while the peak was observed at 1701 cm<sup>-1</sup> for untreated Korean red pine. The peak at 1701 cm<sup>-1</sup> was also detected in all the carbonized samples up to 320 °C.

The peak at 1366 cm<sup>-1</sup> was assigned to –C–H deformation in cellulose and hemicelluloses, appeared in all untreated samples. However, the peaks at 1366 cm<sup>-1</sup> for bamboo and wood disappeared at 280 and 320 °C, respectively. The peak at 1316 cm<sup>-1</sup> was related to –CH<sub>2</sub> vibrations in crystalline cellulose, observed in all samples. In giant bamboo, tali bamboo, and Korean red pine, the peak at 1316 cm<sup>-1</sup> disappeared at 320 °C, except for cork oak. The peak at 1316 cm<sup>-1</sup> from betung bamboo, kuning bamboo, and moso bamboo was absent at 280 and 320 °C.





**Fig. 3.** FT-IR spectra of the carbonized samples at different temperatures: (a) giant and tali bamboos; (b) moso, betung, and kuning bamboos; (c) Korean red pine; and (d) cork oak

**Table 8.** Peak Assignment in the FT-IR Spectra for Functional Groups in Samples

Component	Wavenumber (cm <sup>-1</sup> )	Peak Assignment
Lignin	1600 1507 1455 1420	Aromatic skeletal vibrations of lignin (Pandey 1999; Schwanninger <i>et al.</i> 2004)
	1263 1231	–C–O stretching in lignin (Schwanninger <i>et al.</i> 2004; McLean <i>et al.</i> 2014)
	830	–CH out-of-plane bending in guaiacyl units (Faix 1991)
Carbohydrates	1733 1701	–C=O stretchings (unconjugated) in esters, ketones, aldehydes, and acids (Schwanninger <i>et al.</i> 2004, Esteves <i>et al.</i> 2013; Lin <i>et al.</i> 2018)
	1366	–C–H deformations in cellulose and hemicelluloses (Popescu <i>et al.</i> 2007)
	1316	–CH <sub>2</sub> vibration in crystalline cellulose (Evans <i>et al.</i> 1992)
	1200	–OH in-plane bending in cellulose (Popescu <i>et al.</i> 2007)
	1155	–C–O–C asymmetric stretchings in cellulose and hemicellulose (Faix and Böttcher 1992; Popescu <i>et al.</i> 2007)
	1105	–C–O–C stretchings in cellulose and hemicellulose (Traoré <i>et al.</i> 2018)
	1040 1025	–C–O stretching of primary alcohols in cellulose (Popescu <i>et al.</i> 2007)
	985	–C–O valence vibration (Evans <i>et al.</i> 1992)
	896	–C–H deformation in cellulose (Schwanninger <i>et al.</i> 2004)

The peak intensity at  $1200\text{ cm}^{-1}$  due to  $\text{-OH}$  in-plane bending in cellulose gradually decreased with increasing temperature and disappeared at  $320\text{ }^{\circ}\text{C}$  for all bamboos. The peak at  $1200\text{ cm}^{-1}$  was observed in Korean red pine and cork oak at  $320\text{ }^{\circ}\text{C}$ . In giant bamboo, tali bamboo, Korean red pine, and cork oak samples, the peak at  $1155\text{ cm}^{-1}$  related to  $\text{-C-O-C}$  asymmetric stretching in cellulose and hemicellulose was observed at  $280\text{ }^{\circ}\text{C}$ . The peak was absent at  $280\text{ }^{\circ}\text{C}$  for the betung bamboo, kuning bamboo, and moso bamboo. The peak at  $1105\text{ cm}^{-1}$  representing  $\text{-C-O-C}$  stretching in cellulose and hemicellulose appeared in all species until  $280\text{ }^{\circ}\text{C}$ , while the peak still appeared in the carbonized giant bamboo, tali bamboo, and cork oak at  $320\text{ }^{\circ}\text{C}$ .

The peaks at  $1040$  and  $1025\text{ cm}^{-1}$  representing  $\text{-C-O}$  stretching in cellulose appeared in the untreated betung bamboo, kuning bamboo, moso bamboo, and Korean red pine, while giant bamboo, tali bamboo, and cork oak only showed a peak at  $1025\text{ cm}^{-1}$ . In the carbonized giant bamboo and tali bamboo, the peak at  $1040\text{ cm}^{-1}$  was observed until  $280\text{ }^{\circ}\text{C}$  and it disappeared at  $320\text{ }^{\circ}\text{C}$ . Moreover, the peak at  $1025\text{ cm}^{-1}$  gradually decreased with increasing temperature and was absent at  $320\text{ }^{\circ}\text{C}$ . Furthermore, in betung bamboo, kuning bamboo, and moso bamboo, the peak at  $1040\text{ cm}^{-1}$  gradually decreased and disappeared at  $320\text{ }^{\circ}\text{C}$ . In addition, the peak at  $1025\text{ cm}^{-1}$  was absent at  $280$  and  $320\text{ }^{\circ}\text{C}$ . Both peaks at  $1040$  and  $1025\text{ cm}^{-1}$  in Korean red pine samples gradually decreased until at  $280\text{ }^{\circ}\text{C}$  and disappeared at  $320\text{ }^{\circ}\text{C}$ . In cork oak, the peak intensity at  $1040\text{ cm}^{-1}$  disappeared at  $320\text{ }^{\circ}\text{C}$ , while the peak at  $1025\text{ cm}^{-1}$  was observed at  $320\text{ }^{\circ}\text{C}$ .

Giant bamboo, tali bamboo, Korean red pine, and cork oak showed a peak at  $985\text{ cm}^{-1}$  representing  $\text{-C-O}$  valence vibration and the peak at  $896\text{ cm}^{-1}$  related to  $\text{-C-H}$  deformation in cellulose was observed at  $280\text{ }^{\circ}\text{C}$ , and the peaks disappeared at  $320\text{ }^{\circ}\text{C}$ . Peaks from betung bamboo, kuning bamboo, and moso bamboo were all detected until  $240\text{ }^{\circ}\text{C}$ , and then they disappeared.

All species showed FTIR peaks at  $1600$ ,  $1507$ ,  $1455$ , and  $1420\text{ cm}^{-1}$ , representing the aromatic skeletal vibrations of lignin at all temperatures. The intensity of the peaks from giant bamboo and tali bamboo at  $320\text{ }^{\circ}\text{C}$  was weaker when compared to that of the other species. The peak at  $1231\text{ cm}^{-1}$ , which describes the  $\text{-C-O}$  stretching in lignin, appeared for all untreated samples, while the peak at  $1263\text{ cm}^{-1}$  was only for the Korean red pine. The peak at  $1231\text{ cm}^{-1}$ , in all species, disappeared at  $280\text{ }^{\circ}\text{C}$ , while the peak at  $1263\text{ cm}^{-1}$  disappeared at  $320\text{ }^{\circ}\text{C}$ . The peak at  $830\text{ cm}^{-1}$  due to  $\text{-CH}$  out-of-plane bending in guaiacyl units only appeared in the bamboo samples and was observed until  $280\text{ }^{\circ}\text{C}$ .

The FT-IR spectra of the carbonized samples were in line with the X-ray diffraction analysis. The carbohydrate compounds of giant bamboo, tali bamboo, Korean red pine, and cork oak showed a visible change in chemical structure at  $280\text{ }^{\circ}\text{C}$ , and it greatly changed at  $320\text{ }^{\circ}\text{C}$ , showing the destruction of the crystalline cellulose structure. On the other hand, moso bamboo, betung bamboo, and kuning bamboo revealed a considerable change in the cellulose crystalline structure at  $240\text{ }^{\circ}\text{C}$  that changed at  $280\text{ }^{\circ}\text{C}$ . In addition, the lignin content of all samples showed a minor change from  $240$  to  $320\text{ }^{\circ}\text{C}$ . Rousset *et al.* (2011) reported that torrefaction at  $280\text{ }^{\circ}\text{C}$  typically changed the chemical compositions of bamboo. These authors mentioned that cellulose and hemicelluloses are degraded between  $250$  and  $280\text{ }^{\circ}\text{C}$ , while lignin is degraded at higher temperatures ( $>300\text{ }^{\circ}\text{C}$ ). Shangguan *et al.* (2016) reported that the FT-IR intensity of cellulose, hemicellulose, and phenolic acid bands significantly decreased after  $170\text{ }^{\circ}\text{C}$ , showing increased degradation of the cellulose and hemicellulose contents. The authors also mentioned that the intensity of the lignin band was constant between  $20$  and  $230\text{ }^{\circ}\text{C}$ , indicating no degradation of the lignin content.

## CONCLUSIONS

1. Macroscopically, bamboo and wood samples showed significant color change at 200 °C and became charcoal from 240 °C.
2. The splitting in carbonized samples was hardly observed in bamboo, while severe splits and cracks were often found on the transverse surface of the cork oak and radial surface of Korean red pine, respectively.
3. Bamboo showed a rapid increase in weight loss and calorific value at lower carbonization temperature than wood. The acidic properties of bamboo and wood changed into neutral at 280 and 320 °C, respectively.
4. There were considerable differences in the transition temperature from crystalline to amorphous cellulose between the species.
5. The Fourier transform infrared (FT-IR) spectra showed noticeable differences in the chemical structures of cellulose, hemicellulose and lignin between the temperatures and species. The results of the FT-IR spectra are in line with the X-ray analysis.

In summary, the differences in carbonization characteristics within bamboo species and between bamboo and wood carbonized at low temperature were revealed. The results in this study can be used for further understanding on the carbonization process of woody materials.

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