Influence of Torrefaction on the Heating Values and Energy Densities of Hardwood and Softwood

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Lumber manufacturing facilities generate large amounts of wood residues as a by-product of processing. In this study, the torrefaction characteristics of the *Cryptomeria japonica* and *Acacia confusa* residues were evaluated and compared to microcrystalline cellulose. The torrefied products also were analyzed. The results showed that the higher heating value (HHV) of the *C. japonica* and *A. confusa* residues increased to 5,993 and 5,576 kcal/kg after 20 min of torrefaction at 310 °C, which was higher than the microcrystalline cellulose (4,340 kcal/kg). The energy densification of the torrefied biomass could rise to 1.17 to 1.20 times higher than the raw biomass. The condensable gaseous product was an organic acid liquid. The liquid product consisted of some alkanes, alcohols, esters, and amides. The exothermic temperature would be reduced after torrefaction. Additionally, the thermal treatment of the biomass destroyed the cellulose crystals and reduced the cellulose crystallinity index. In order to reduce the usage of fossil fuels, the torrefied solid biomass could be mixed with coal in power plants directly.

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Keywords: Torrefaction; Higher heating value (HHV); Energy density; Co-firing; Cellulose crystallinity index

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INTRODUCTION

In Taiwan, energy is primarily supplied by coal, petroleum, and natural gas (91.74%). Biomass and wastes account for approximately 1.14% of the overall energy supply and 58.46% of the energy supplied by renewables in 2019 (Bureau of Energy 2021). The proportion of utilization of biomass and waste is quite low relative to the total energy supply, so there is great potential to develop biomass energy in the future. Biomass and wastes play an important role in total renewable energy supply, just as petroleum and crude oil do in the total energy supply (Ibitoye *et al.* 2021). As a result, biomass is a suitable candidate to lead in the development of renewable energy sources (Tan *et al.* 2019). The utilization of biomass can reduce the emission of carbon dioxide (CO_2) because decayed biomass contains CO_2 (Stougie *et al.* 2017). When biomass is utilized to produce energy, the emission of CO_2 is effectively neutral. In addition, biomass is considered to be the renewable energy source with the highest potential to contribute to the energy needs of modern society for both the developed and developing economies world-wide (Cambero and Sowlati 2014).

Softwood is a type of wood from the gymnosperm trees, such as conifers, while hardwood is a type of wood from dicot angiosperm trees. Softwood and hardwood can be distinguished by their microstructures. The most important characteristic of softwood is the lack of vessels for water supply. The softwood structure is usually relatively simple and consistent, with water transport taking place only through the trachea and the pit openings between them. Softwood is usually used for building structural parts and woodworking products such as molds, furniture, and factory doors and windows. Softwood is the main raw material for certain paper products such as linerboard, while hardwood is usually used to make exposed products such as furniture, wooden floors, or cutlery, as well as pulp for printing papers. According to the 4th national forest resource investigation reports in 2014, Taiwan's forest stock volume of softwood and hardwood were approximately 26.4% and 57.5%, respectively (Total forest stock volume: 502,000,000 m^3) (Forestry Bureau 2020). This study evaluated the differences in *Cryptomeria japonica* (softwood) and *Acacia confusa* (hardwood) residues after they were subjected to torrefaction.

Torrefaction is known as mild pyrolysis, low temperature cracking, baking, roasting, or high temperature drying. It is a thermochemical technology, the operating temperature of which is typically between 200 and 300 °C in a hypoxic environment (Eseyin *et al.* 2015). Additionally, torrefaction could improve the physical properties, chemical composition, and energy and storage properties of biomass (Tumuluru *et al.* 2021). During torrefaction, moisture and some volatile organic compounds volatilize from the biomass (Tumuluru *et al.* 2021). While the solid product is the principal product of the torrefaction process, liquid and gas through the device may provide some additional heat for torrefaction. The torrefaction is a mild pyrolysis process, but it is conducted at a lower operating temperature compared to conventional thermal cracking (Chen *et al.* 2011), with a heating rate that is generally less than 50 °C/min (Deng *et al.* 2009).

General biomass is primarily composed of hemicellulose, cellulose, and lignin. The combinations of these components have different response mechanisms in the roasting process to improve the biomass energy density. The effect of temperature changes on the hemicellulose, cellulose, and lignin have been evaluated by Bergman *et al.* (2005). The temperature is the key parameter that affects the product properties compared to the residence time (Mohan *et al.* 2006). Additionally, the liquid product analyses from the torrefied biomass, including lauan and bamboo, have been conducted in previous studies (Chen *et al.* 2015). Gas phase products including carbon monoxide (CO), CO_2, methane (CH_4), C_xH_y, toluene, and benzene could be found from the torrefied biomass.

In this study, the torrefaction characteristics of *C. japonica* and *A. confusa* residues were evaluated and compared with a microcrystalline cellulose product. After the biomass torrefaction reaction, the solid product, liquid, and gas phases were analyzed. The co-firing potential of biomass with coal also was evaluated (requiring a heating value of above 5,000 kcal/kg).

EXPERIMENTAL

Raw Materials

The *C. japonica* and *A. confusa* residues that were used in this study were collected from the Wood Utilization and Product Exhibition Center located in Nantou county, Taiwan. The *C. japonica* and *A. confusa* residues were chipped, ground, and passed through a 40 mesh sieve before they were used in the batch experiments. The microcrystalline cellulose (Avicel) was purchased from Sigma-Aldrich (St. Louis, MO, USA). All the other reagents used in this study were of analytical grade. The basic characteristics of the wood residues and the microcrystalline cellulose were analyzed *via* elemental analysis (EA) with an Elementar Vario EL III (Ronkonkoma, NY, USA) device. The moisture content (TAPPI T 258 om-06 (2006)), the solvent extractives content (TAPPI T 204 cm-07 (2007)), the holocellulose content (TAPPI T 249 cm75 (1975)), the Klason lignin content (CNS 14907 (2005)), the pentosan content (TAPPI T 223 cm10 (2010)), and the ash content (TAPPI T 211 om-85 (1985)) were all measured. The proximate analysis was conducted according to the ASTM standard D3172-13 (2013). The thermogravimetric analysis (TGA) was conducted with a PYRIS Diamond unit (PerkinElmer, Waltham, MA, USA) device, and the calorific value analysis was conducted according to the ASTM standard D2015 (2000). The heating value of a biomass fuel is measured using a bomb calorimeter which measures the enthalpy change between reactants and products. All the heating values in this study are given as higher heating value (HHV) of oven dry biomass. A gas chromatography mass spectrophotometer (GC-MS, Thermo Scientific Gas Chromatograph, Thermo Fisher, USA), was used for identifying the organic component of the liquid product from torrefaction. The gas phase of the biomass residue after torrefaction were analyzed with a gas chromatography flame ionization detector (GC-FID) (Hewlett Packard Inc., Wilmington, DE, USA) and a CO detector (Tempest 100; Telegan Gas Monitoring Ltd., Abingdon, Oxfordshire, UK).

Experimental Process

In this study, the raw materials were first analyzed to obtain their basic composition properties, including wood chemical composition analysis, proximate analysis, elemental analysis, and calorific value analysis. The samples were then analyzed with an oxygen bomb calorimeter and a thermal gravimetric analyzer for their energy content and thermal behavior. These tools were used to determine the final torrefaction factor: the torrefied temperature (T) and the residence time (t). A tubular furnace was used in this study for the torrefaction reaction. The N_2 carrier gas was first purged into the reactor for 10 min at a flow rate of 100 mL/min. Then, 5 g of biomass residues (*C. japonica* or *A. confusa*) was placed in the tube at 290, 310, and 330 °C for various reaction times of 10, 20, 30, 40, 50, and 60 min with the N_2 carrier gas, respectively. Additionally, 5 g of microcrystalline cellulose also was torrefied in the tube at 290, 310, and 330 °C for various reaction times of 15, 20, and 25 min with the N_2 carrier gas.

After undergoing the torrefaction process, the product was analyzed at various phases. The solid phase was analyzed by the method outlined above, while the liquid and gas phases were analyzed by gas chromatography for recognition. Both NO_x and sulfur dioxide (SO_2) were detected instantly using the gas analysis detector (Tempest 100; ICS Schneider Messtechnik GmbH, Hohen Neuendorf, Germany). All the emissions were collected in the gas bag through the torrefaction process, so the average content of the NO_x and SO_2 were able to be detected.

RESULTS AND DISCUSSION

Characteristics of the Wood Residues and Microcrystalline Cellulose

Table 1 shows the characteristics of the wood residues and the microcrystalline cellulose. The holocellulose, α-cellulose, and extractive levels of the *A. confusa* were higher than those of the *C. japonica*. The lignin content of the *C. japonica* was higher than that of the *A. confusa*. Additonally, the combustible contents of the *A. confusa* were also higher than the *C. japonica*. However, the order of HHV and carbon contents of the *C. japonica* was higher than the *A. confusa*, which was higher than the microcrystalline cellulose. Higher holocellulose or α-cellulose contents signify a lower HHV, while a higher lignin content signifies a higher HHV.

Table 1. Characteristics of the Wood Residues and the Microcrystalline Cellulose

Characteristics		*C. japonica*	*A. confusa*	Microcrystalline cellulose
Chemical Composition (%)	Holocellulose	64.57	72.64	100
	α-cellulose	43.80	47.58	100
	Lignin	30.66	20.63	N.D.
	Extractives	7.80	9.03	N.D.
	Ash	0.57	0.21	N.D.
Proximate Analysis (%)	Moisture	11.93	9.73	4.61
	Volatile Matter	73.39	74.18	87.95
	Ash	0.50	0.19	N.D.
	Fixed Carbon	14.18	15.90	7.65
	Combustibles	87.57	90.08	95.40
HHV (kcal/kg)		5119.97	4654.18	4173.60
Elemental Analysis (%)	C	50.14	47.18	42.66
	H	6.68	6.64	6.53
	O	43.18	45.55	50.81
	N	N.D.	N.D.	N.D.
	S	N.D.	N.D.	N.D.
	H/C	13.32	14.07	15.31
	O/C	86.12	96.55	119.10

Energy Density of the Wood Residues in Different Torrefaction Conditions

The mass yield, energy yield, and energy density values of the *C. japonica* and *A. confusa* with different torrefaction conditions are shown in Tables 2 and 3. Higher torrefaction temperature and time conditions resulted in a lower mass yield and a lower energy yield for the biomass. Theoretically, the 70% (by weight) of the residue solid product and 10% (by HHV) of the energy loss from the biomass torrefaction reaction was an ideal torrefaction reaction. The energy density of the torrefaction biomass would rise to 1.29 (Bergman *et al.* 2005). Table 4 shows the HHV of the *C. japonica* and *A. confusa* in different torrefaction conditions. Higher torrefaction temperature and times created a more rapid increase in the HHV of the biomass. The HHV of the *C. japonica* at the same torrefaction temperature and time was higher compared to the *A. confusa* and microcrystalline cellulose. The ideal torrefaction temperature and time at 70% of the residue solid for the *C. japonica* was 290 °C and 20 min, respectively, to obtain a 73.37%

mass yield, an 80.03% energy yield, and a 1.09 energy density value. At a torrefaction temperature and time of 290 °C and 30 min, respectively, the *A. confusa* had a mass yield of 70.08%, an energy yield of 80.26%, and an energy density of 1.15. At a torrefaction temperature and time of 310 °C and 25 min, respectively, the microcrystalline cellulose had a mass yield of 73.11%, an energy yield of 82.18%, and an energy density of 1.12.

Table 2. Mass Yield, Energy Yield, and Energy Density of *C. japonica* and *A. confusa* in Different Torrefaction Conditions

Biomass	Temperature (°C)	Time (min)	Mass Yield (%)	Energy Yield (%)	Energy Density
Microcrystalline cellulose	290	15	98.81	99.07	1.00
		20	98.13	99.40	1.01
		25	95.90	98.27	1.02
	310	15	97.85	99.32	1.02
		20	89.80	93.39	1.04
		25	73.11	82.18	1.12
	330	15	88.84	91.29	1.03
		20	55.69	68.66	1.23
		25	26.79	37.22	1.39
C. japonica	290	10	95.23	95.85	1.01
		20	73.37	80.03	1.09
		30	65.46	74.08	1.13
		40	61.70	71.68	1.16
		50	59.04	69.23	1.17
		60	55.97	67.75	1.21
	310	10	84.65	87.05	1.03
		20	60.50	70.82	1.17
		30	51.63	64.42	1.25
		40	47.35	59.28	1.27
		50	47.29	59.91	1.26
		60	46.73	58.89	1.26
	330	10	62.04	72.14	1.16
		20	46.18	58.77	1.27
		30	43.66	56.32	1.29
		40	42.83	55.74	1.30
		50	42.75	55.49	1.30
		60	41.30	53.69	1.30

* Mass yield (%) = (Weight of biomass after torrefaction/ weight of raw biomass) × 100
Energy yield (%) = Mass yield × (Higher heating value of biomass after torrefaction/ Higher heating value of raw biomass) × 100
Energy density = Energy yield/Mass yield

Table 3. Mass Yield, Energy Yield, and Energy Density of *C. japonica* and *A. confusa* in Different Torrefaction Conditions (Continued)

Biomass	Temperature (°C)	Time (min)	Mass Yield (%)	Energy Yield (%)	Energy Density
A. confusa	290	10	82.19	89.13	1.08
		20	73.22	81.05	1.11
		30	70.08	80.26	1.15
		40	63.4	76.18	1.20
		50	61.17	74.13	1.21
		60	58.67	73.01	1.24
	310	10	67.21	75.05	1.12
		20	56.14	67.26	1.20
		30	46.89	63.37	1.35
		40	45.83	62.11	1.36
		50	44.53	61.28	1.38
		60	41.01	57.13	1.39
	330	10	68.27	78.06	1.14
		20	43.52	58.71	1.35
		30	40.70	56.73	1.39
		40	40.43	56.46	1.40
		50	40.15	56.28	1.40
		60	39.92	56.15	1.41

Table 4. Higher Heating Values (HHV) (kcal/kg) of the *C. japonica* and *A. confusa* in Different Torrefaction Conditions

Biomass	Time (min)	Temperature (°C)		
		290	310	330
Microcrystalline cellulose	15	4184.94	4236.31	4288.81
	20	4227.45	4340.57	5146.11
	25	4276.82	4691.76	5798.27
C. japonica	10	5153.62	5265.33	5953.27
	20	5584.51	5992.92	6516.04
	30	5793.89	6387.95	6605.17
	40	5947.78	6410.16	6663.84
	50	6003.77	6486.78	6645.46
	60	6197.23	6452.62	6656.28
A. confusa	10	5047.17	5197.09	5321.60
	20	5151.89	5576.06	6278.65
	30	5330.26	6289.94	6487.26
	40	5592.36	6307.46	6499.51
	50	5640.25	6404.85	6523.97
	60	5791.75	6483.62	6546.40

Solid Product of the Torrefied Biomass

Table 4 shows the HHV of the torrefied wood residues and the microcrystalline cellulose in different torrefaction conditions. The combustible content of the *A. confusa* was also higher than the *C. japonica*. However, the order of the HHV and the carbon contents was as follows: *C. japonica* > *A. confusa* > microcrystalline cellulose. The biomass with higher holocellulose or α-cellulose contents had a lower HHV. The biomass with higher lignin contents had a higher HHV. Additionally, the greater heating values (> 5,000 kcal/kg) for the torrefied biomass could be a partial replacement for coal for power generation.

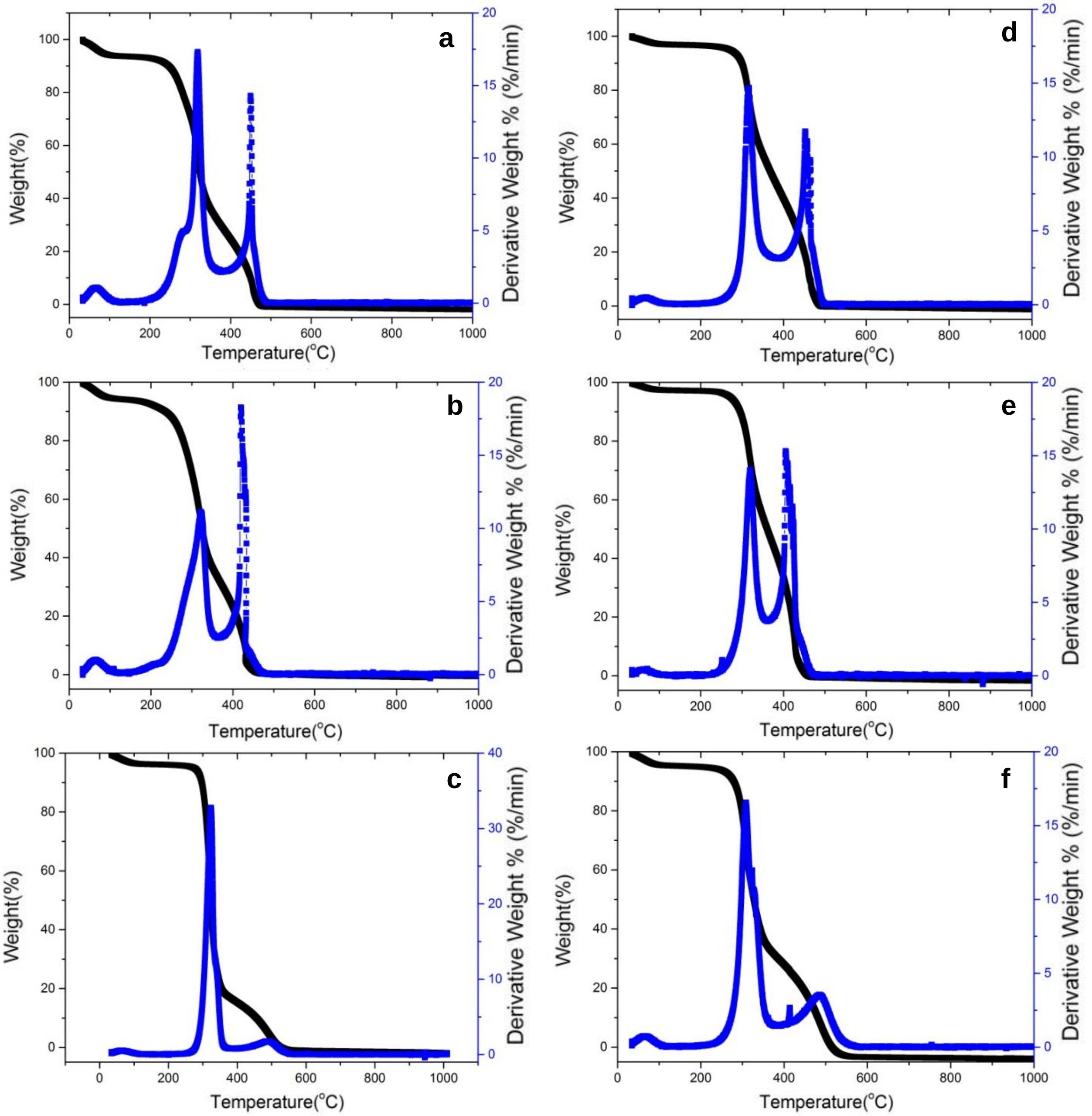

Fig. 1. TGA/DTA curves of the raw biomass (a) *A. confusa*, (b) *C. japonica*, and (c) microcrystalline cellulose, and the torrefied biomass (d) *A. confusa*, (e) *C. japonica*, and (f) microcrystalline cellulose. Torrefaction condition: 310 °C and 20 min.

The results of TGA and differential thermal analysis (DTA) of the raw biomass and the torrefied biomass are shown in Fig. 1. The amount of moisture that was removed from the raw *A. confusa*, *C. japonica*, and microcrystalline cellulose at 100 °C was 5.94%, 5.38%, and 3.49%. Corresponding values for the torrefied specimens were 2.87%, 2.54%, and 4.48%, respectively. The temperature of the overall exothermic reaction was conducted at approximately 318.5 °C and 453.30 °C for the raw *A. confusa*, 419.93 °C and 322.10 °C for the raw *C. japonica*, and 323.73 °C for the raw microcrystalline cellulose. The temperature of the overall exothermic reaction was conducted at approximately 316.76 °C and 452.66 °C for the torrefied *A. confusa,* 405.32 °C and 319.01 °C for the torrefied *C. japonica*, and 308.40 °C for the torrefied microcrystalline cellulose. The residue contents of raw biomass or torrefied biomass were less than 0.5% after reacting at 800 °C. The energy densification of the torrefied biomass could rise to 1.17 to 1.20 times higher than the raw biomass. The torrefaction process is an advantageous method to upgrade biomass to a high energy density solid biofuel. Thus, the exothermic temperature would be reduced after torrefaction.

Figure 2 shows the X-ray diffraction (XRD) patterns of the crystalline structures of the raw biomass and the torrefied biomass samples. The XRD data were collected using CuKα radiation. The peak height method of XRD is the most widely used to determine the cellulose crystallinity (Thygesen *et al.* 2005). The cellulose crystallinity index values of the raw *A. confusa*, *C. japonica*, and microcrystalline cellulose were 53.57%, 48.70%, and 82.75%. The corresponding values for the torrefied specimens were 47.12%, 37.20%, and 41.13%, respectively. The thermal treatment of the biomass would destroy the cellulose crystals and reduce the cellulose crystallinity index. With the increase of torrefaction temperature, the -OH and C=O contents decreased, the C=C contents increased, and the crystallinity index decreased due to degradation and recrystallization (Zheng *et al.* 2017). Cellulose crystallization is directly related to the formation of the intermolecular hydrogen bonds (Himmel *et al.* 2007). Additionally, the crystalline structure also affected the pyrolysis products. The higher crystallinity samples will have the more levoglucosan yield (Wang *et al.* 2014; Mukarakate *et al.* 2016).

Liquid Product and Gas Emission of the Torrefied Biomass

The liquid products from the pyrolysis of the torrefied wood were analyzed by gas chromatography-mass spectrometry (GC-MS). The probable organic compounds of the liquid products from the torrefied biomass are shown in Fig. 3. Furfural ($C_5H_4O_2$, RT:10.5 min), citronellal ($C_{10}H_{18}O$, RT:22.9 min), guaiacol ($C_7H_8O_2$, RT:18.2 min), and glycolaldehyde ($C_2H_4O_2$, RT:4.8 min) were the major organic compounds from the torrefaction of the *A. confusa*, $C_7H_8O_2$ and $C_{10}H_{18}O$ for the torrefied *C. japonica*, and 2-butanol ($C_4H_{10}O$, RT:2.6 min), $C_5H_4O_2$, $C_7H_8O_2$, and methyl 9-decenoate ($C_{11}H_{20}O_2$, RT:23.0 min) for the torrefied microcrystalline cellulose.

The gas concentrations of CO_2, CO, NOx, and SO_2 were continually detected at one minute increments from the 310 °C and 25 min torrefication process (as shown in Fig. 4). The CO_2 concentrations stabilized at approximately 10 min with a CO_2 measurement of 4.7 vol % for the *A. confusa*, 4.9 vol % for the *C. japonica*, and 5.8 vol % for the microcrystalline cellulose. The maximum value of the CO appeared at approximately 13 min with a CO measurement of 8227 ppmv for the *A. confusa*, 8232 ppmv for the *C. japonica* at 16 min, and 3168 ppmv for the microcrystalline cellulose at 25 min. Additionally, the NO_x, and SO_2 concentrations from the biomass torrefication process were lower than the detection limit. Thus, the emission of the NO_x, and SO_2 could be neglected.

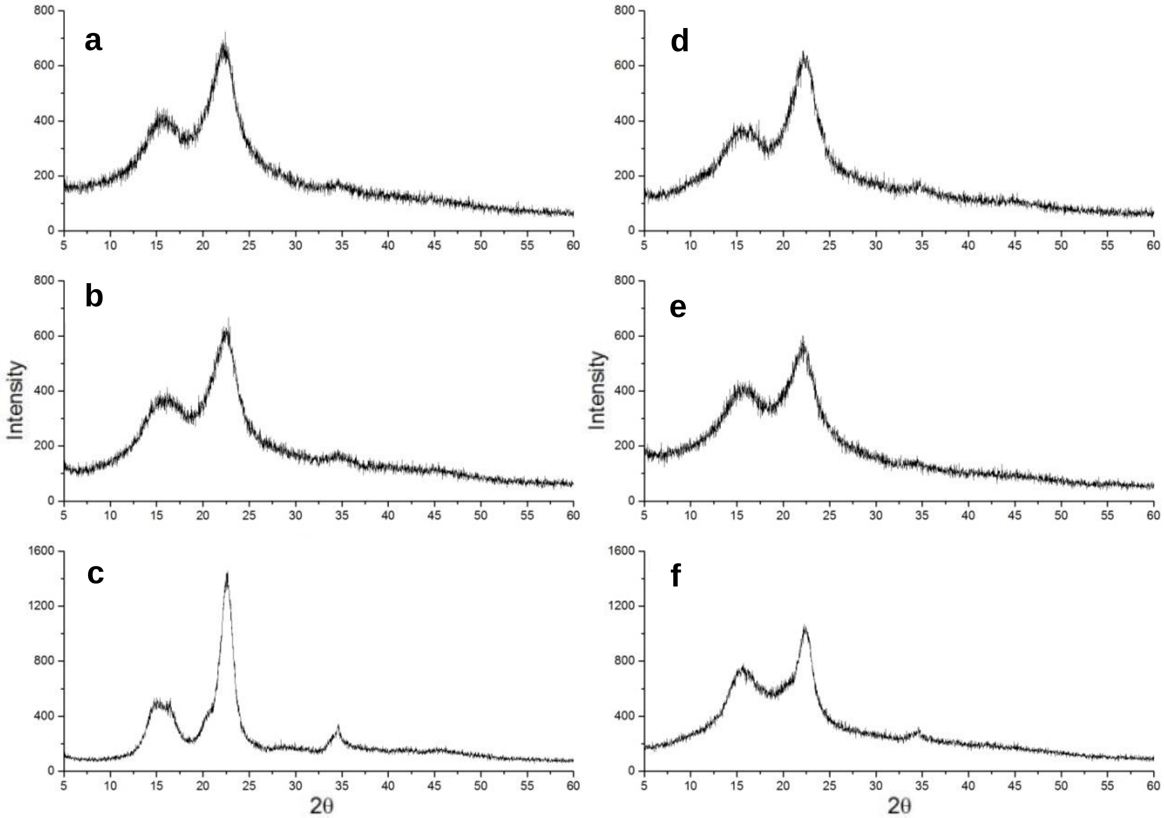

Fig. 2. XRD patterns of the crystalline structures for the raw biomass (a) *A. confusa*, (b) *C. japonica*, and (c) microcrystalline cellulose, and the torrefied biomass (d) *A. confusa*, (e) *C. japonica*, and (f) microcrystalline cellulose.

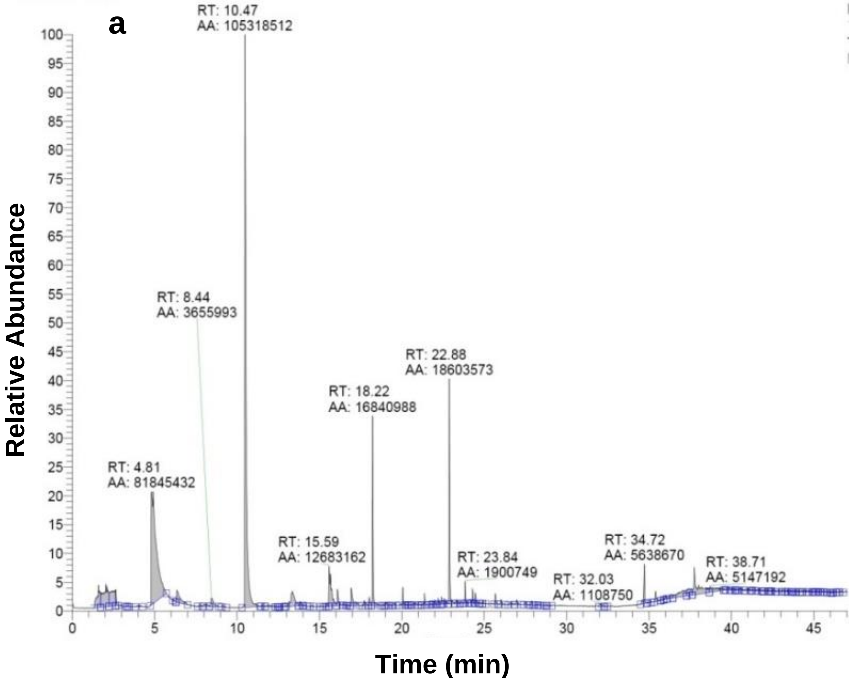

Fig. 3(a). GC-MS chromatogram showing the pattern of the liquid products from the torrefied (a) *A. confusa*, (b) *C. japonica*, and (c) microcrystalline cellulose

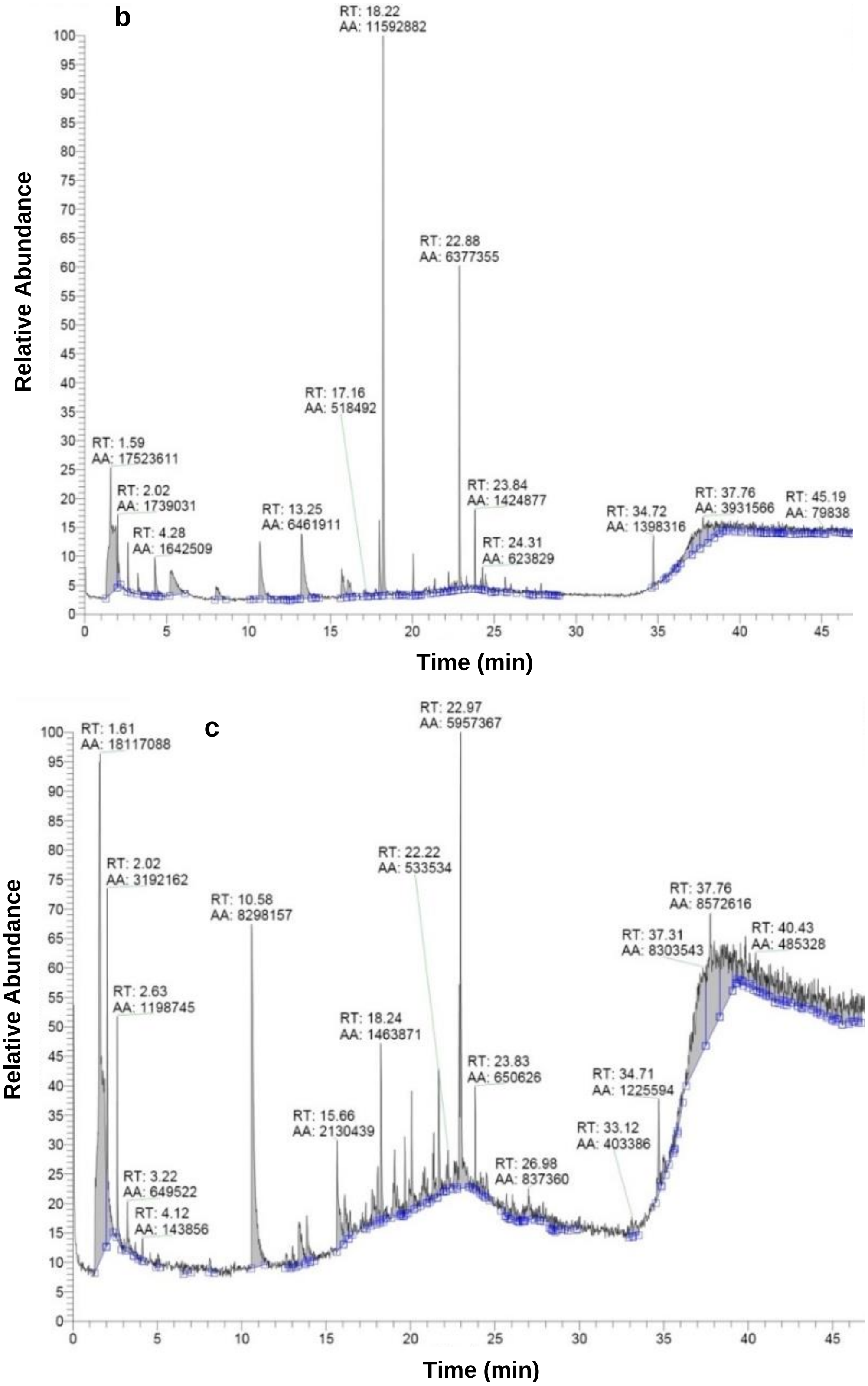

Fig. 3(b & c). GC-MS chromatogram showing the pattern of the liquid products from the torrefied (a) *A. confusa*, (b) *C. japonica*, and (c) microcrystalline cellulose

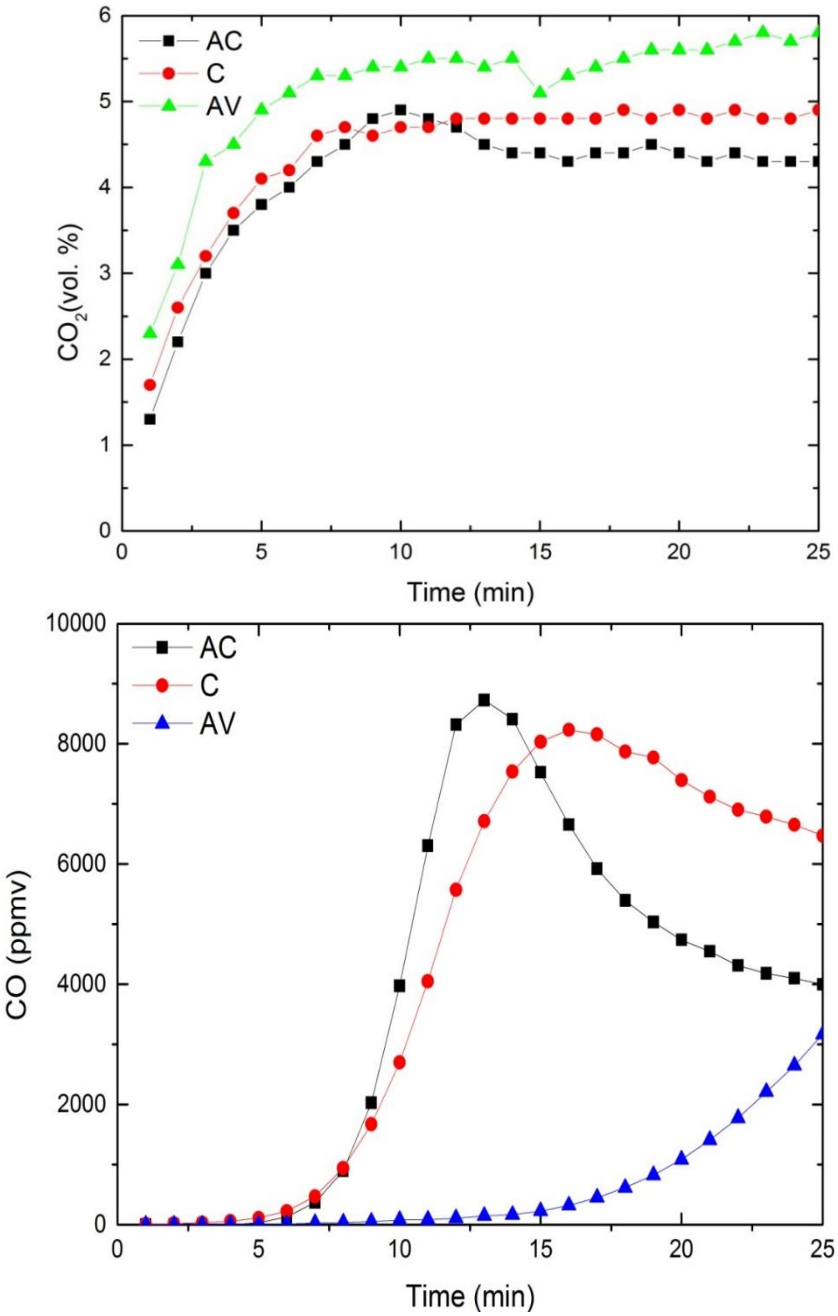

Fig. 4. Gas emissions of the CO_2 and CO from the torrefaction of the *A. confusa*, *C. japonica*, and microcrystalline cellulose at 310 °C and 25 min

CONCLUSIONS

1. The HHV of the *C. japonica* and *A. confusa* residues increased to 5,993 and 5,576 kcal/kg after 20 min of torrefaction at 310 °C, which was higher than the microcrystalline cellulose (4,340 kcal/kg). The greater heating values (> 5,000 kcal/kg) for the torrefied biomass could serve as a partial replacement for coal in power generation.

2. The energy densification of the torrefied biomass could rise to 1.17 to 1.20 times higher than the raw biomass.
3. The thermal treatment of the biomass can destroy the cellulose crystals and reduce the cellulose crystallinity index. The exothermic temperature would be reduced after torrefaction. Additionally, the emission of NO_x, and SO_2 could be neglected.

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