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Identification of differences and comparison of fuel characteristics of torrefied agro-byproducts under oxidative conditions

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ABSTRACT

Torrefaction is a pretreatment method for upgrading biomass into solid fuels. This study aimed to investigate the properties of agro-byproducts pretreated under different oxidative conditions at temperatures of 210–290 °C for 1 h to determine optimal operating conditions for upgrading biomass. The mass yields of lignocellulosic and herbaceous biomass were 90.27–42.20% and 92.00–45.50% and 85.71–27.23% and 88.09–41.58% under oxidative and reductive conditions, respectively. The calorific value of lignocellulosic and herbaceous biomass under oxidative conditions increased by approximately 0.14–9.60% and 3.98–20.02%, respectively. Energy yield of lignocellulosic and herbaceous biomass showed 63.78–96.93% and 90.77–44.39% showed 88.09–41.58% and 92.38–27.23% under oxygen-rich and deficit conditions. A decrease in oxygen and an increase in carbon dioxide and carbon monoxide were confirmed through gas measurements. Torrefaction evaluations were conducted using energy–mass co-benefit index (EMCI). Decreases of ΔEMCI were observed under certain conditions. Both oxidative and reductive conditions can be employed for pepper stems, wood pellets, and pruned apple branches. Based on standards, the optimal temperatures for pepper stems, wood pellets, and pruned apple branches in oxidative conditions were 250, 270, and 250 °C, respectively.

1. Introduction

Owing to global concerns about environmental pollution, regulations on using existing fossil fuels have become strict. As a result, there has been a growing interest in biomass, which is a carbon-neutral fuel that can be produced over a larger geographical area when compared to fossil fuels. This renewable energy source has been attracting more attention lately. Agricultural by-products are advantageous owing to their continuous production. In 2021, domestic wood pellet production amounted to 658,336 tons, while imported wood pellets accounted for 3,178,560 tons, making up 82.84% of the total pellet consumption in the country [1]. Devices for collecting agricultural by-products have been developed to use this biomass [2]. Anaerobic digestion has been used to produce biogas, and pelletizing [3,4]. Thermochemical pretreatments [3–6] have been tested to produce solid fuels. However, compared with solid fossil fuels, many agricultural byproducts are typically not utilized due to their hydrophilic nature, low energy content, and challenges

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associated with storage [7–13]. An alternative process called torrefaction has been proposed to address these shortcomings. Torrefaction is the process of heat-treating biomass at temperatures ranging from 200 to 300 °C in an oxygen-deficient environment. The procedure can attain the following benefits: a relatively higher calorific value; hydrophobic properties of the product; more extended storage; and ease of transport [3,14–17]. However, maintaining oxygen-deficit conditions and mass loss during the process have been identified as shortcomings. Surface torrefaction has been proposed to address these problems [18]. Surface torrefaction is a fast thermochemical conversion process at high temperatures. It improves biomass characteristics by reducing mass loss and increasing its calorific value without requiring oxygen. In addition, torrefaction using microwave has been suggested. Yek et al. [19] reported that microwave heating for co-torrefaction of biomass pellets can be an economical approach to produce desirable/ pellet fuel from waste and biomass materials. Chen et al. [20] reported that depending on the type of biomass being used, the optimal torrefaction process may vary. Ligneous biomass, for instance, can be effectively torrefied in an oxidative environment with lower oxygen concentrations, while fibrous biomass may benefit more from a non-oxidative torrefaction approach. Wang et al. [21]examined the use of a carrier gas, containing 3–6% O_2, in the oxidative torrefaction of sawdust. This was carried out in both a TG and fluidized bed reactor, and the resulting torrefied sawdust and pellets were compared to traditional torrefaction without any O_2, as well as the dry raw material. Li et al. [22] reported that when the concentration of O_2 and temperature used in torrefaction are increased, it leads to a higher yield of char and a longer combustion time. The yield of SPC char can be predicted by a linear regression equation ($R^2 = 0.85$), which takes into account the decrease in mass yield resulting from the oxidative torrefaction process.

For solid, liquid, and gaseous products Chen et al. [23] investigated the impact of temperature (220–300 °C) and O2 concentration (0–15 vol%) on solid, liquid, and gas products resulting from torrefaction of rice husks. It was observed that CO_2 was the primary gas produced, with its volume fraction increasing further during the process. They reported that the yield of gaseous and liquid products increased with the oxygen concentration and temperature. Wang et al. [24] compared the oxidative torrefaction of sawdust with a carrier gas containing 3–6% O_2 with the traditional torrefaction of sawdust and pellets. In addition, Chen et al. reported characterization of non-oxidative and oxidative torrefaction using scanning electron microscopy (SEM) observations of four types of biomass, i.e., oil palm fiber, coconut fiber, eucalyptus, and Japanese cedar (*Cryptomeria japonica*) [25]. Basu et al. reported that the effect of oxygen concentration on the heating value of a torrefied biomass product is marginal [23]. Otherwise, Sun et al. reported that torrefaction temperatures and oxygen concentrations remarkably influenced the pyrolysis of bamboo [26]. Wang et al. [27] reported moderate and severe removal of hemicellulose can be achieved through torrefaction under air and nitrogen, respectively.

This study aimed to evaluate the potential of upgrading biomass under air conditions. The herbaceous and woody agricultural byproducts were torrefied in air-oxidative and oxygen-deficit conditions. The applicability of non-oxidative and air-oxidative torrefaction is presented through a comparative analysis of experimental results.

2. Materials and methodology

Five types of biomass were selected for this study (Fig. 1): pepper stems (PP), wood pellets (WP), rice husk (RC), kenaf (KN), and pruned apple branches (AP). PP was collected from a local farm in Paju-si, Gyeonggi-do, Korea. WP (hannamo pellet) was collected from the Yeoju Forestry Association, Korea. RC was obtained from Samjeong Nongsan, located in Jangseong-gun, Jeollanam-do, Korea. KN was collected from Chuncheon-si, Gangwon-do, Korea. AP was collected from Hongcheon-gun, Gangwon-do, Korea. PP and AP were pelletized using a pelletizer (SP-75, Geumgang ENG, Korea). Table 1 presents data for each type of biomass.

2.1. Torrefaction process

The torrefaction process was conducted using a stainless-steel can with a diameter of 75 mm and a height of 55 mm. An electrical furnace, specifically the N 7/H/B410 model from Nabertherm GmbH in Lilienthal, Germany, was utilized for this purpose, as depicted in Fig. 2. Due to their volumetric differences, 103 ± 2 g of PP, WP, and AP pellets, 22 ± 1 g of RC, and 15 ± 1 g of KN were added to each can. The experimental duration was set at 1 h, and the process temperature ranged from 210 to 290 °C, increasing in increments of 20 °C. The experiments under air-oxidative conditions were conducted without a lid on the cans. In contrast, the experiments under oxygen-deficit conditions were conducted with a lid. There was a possibility of an explosion due to pressure when opened for emitting gas, and hence pressuring and sealing were not applied. To differentiate between samples based on process temperature and oxidative

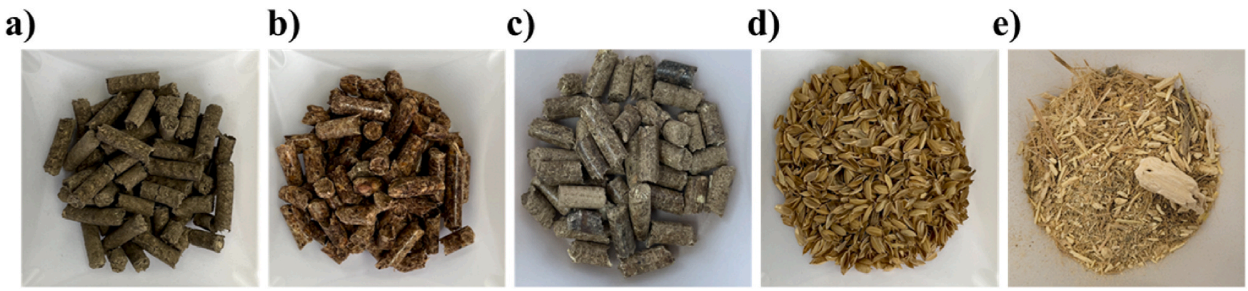

Fig. 1. Untreated biomass sample; a) pepper stem b) wood pellet c) pruned apple branch d) rice husk e) kenaf.

Table 1
Elemental composition, proximate analysis, and higher heating value of each untreated biomass sample.

	Elemental composition [wt%]					Proximate analysis [wt%]				Higher heating value [kcal/kg]
	C	H	N	O	S	MC	VM	FC	Ash	
Pepper stem	43.28	6.05	1.86	42.34	N.D.	11.2	65.78	16.56	6.47	4460
Wood pellet	47.55	6.25	0.06	45.71	N.D.	8.82	75.62	15.14	0.43	4810
Rice husk	40.20	5.71	0.87	40.64	N.D.	11.00	62.08	14.3	12.59	3730
Kenaf	43.81	6.36	1.56	48.27	N.D.	6.54	77.37	12.05	4.04	4290
Apple pruning branch	44.85	6.10	1.12	47.93	N.D.	5.40	73.4	17.3	3.90	4480

*N.D. Not detected**C: Carbon, H: Hydrogen, N: Nitrogen, O: Oxygen, S: Sulfur, MC: Moisture content, VM: Volatile matter, FC: Fixed carbon, Ash: Ash content.

Fig. 2. Electrical furnace and stainless-steel cans used.

conditions, 'O' was added after the abbreviation of the samples processed under oxygen-rich conditions, and only the abbreviation was used for samples processed under oxygen-deficit conditions., and the temperature was added after the abbreviation of the sample; for instance, PPO270 refers to PP torrefied under the oxygen-rich condition at 270 °C. A portable gas analyzer (Optima 7, MRU, Humble, Texas, USA) was used to analyze the gases, such as O2, CO2, and CO, emitted during torrefaction.

Following the torrefaction process, the samples were cooled for 30 min while covered with a lid to prevent any radical reactions with oxygen. Afterward, the resulting mass reduction was measured. The mass yield was calculated using Equation (1) based on the mass reduction.

$$MY\ [\%] = \frac{M_{torr}}{M_{raw}} \times 100 \tag{1}$$

where MY is the mass yield [%], M_{torr} is the mass of biomass after the torrefaction process [g], and M_{raw} is the mass of biomass before the torrefaction process [g]. The statistical software SAS 9.4 (IBM, Armonk, New York, USA) was used to perform Duncan's multiple range test to analyze the differences between the process conditions.

2.2. Fuel characteristic evaluation

2.2.1. Elemental analysis and van krevelen diagram

A FlashEA 1112 elemental analyzer (Thermo Fisher Scientific, Waltham, MA, USA) was used to determine the elemental composition of C, H, N, and S. Oxygen content was estimated using Equation (2) [28]. A van Krevelen diagram was plotted based on the elemental analysis.

$$O = 100 - (C + H + N + S) \tag{2}$$

2.2.2. Calorific value and energy yield

A calorimeter (CAL3K, DDS calorimeters, Gauteng South Africa) was used to estimate the calorific value. To investigate the change in calorific value based on the process temperature and to calculate the parameters related to mass loss, the calorific value was estimated three times. The energy yield was calculated as per Equation (3). It is desirable to obtain a high energy yield from a solid fuel at a low solid volume to increase processing efficiency and facilitate transport. Therefore, an energy–mass co-benefit index (EMCI) represented the difference between the energy and solid yields to quantify the enhancement in the energy content of the remaining mass after torrefaction [29]. The EMCI (%) is the difference between the energy and mass yields (Equation (4)). To analyze the changes in EMCI according to temperature variations, ΔEMCI was calculated using Equation (5).

$$EY\ [\%] = \frac{HHV_{torrefied}}{HHV_{raw}} \times MY \tag{3}$$

$$EMCI\ [\%] = EY - MY = MY\left(\frac{HHV_{torr}}{HHV_{raw}} - 1\right) \tag{4}$$

$$\Delta EMCI\ [\%_p] = EMCI_{T_h} - EMCI_{T_L} \tag{5}$$

where EY is the energy yield [%], $HHV_{torrefied}$ is the higher heating value of torrefied biomass [kcal/kg], HHV_{raw} is the higher heating value of untreated biomass [kcal/kg], EMCI is the energy–mass co-benefit index [%], ΔEMCI denotes the difference in energy–mass co-benefit [%p], $EMCI_{TH}$ denotes energy–mass co-benefit index of higher temperature, and EMCI TL denotes the energy–mass co-benefit index of lower temperature. The Duncan's multiple range test was used to analyze the differences between process conditions of energy yield.

3. Results & discussion

3.1. Impact on mass yield

Fig. 3 represents the mass yield of each type of biomass. The mass yield ranged from 90.38% to 27.23% under oxidative conditions and from 92.40% to 41.58% under reducing conditions. Under oxidative conditions, PP and AP showed rapid mass loss at 250 ℃ and no

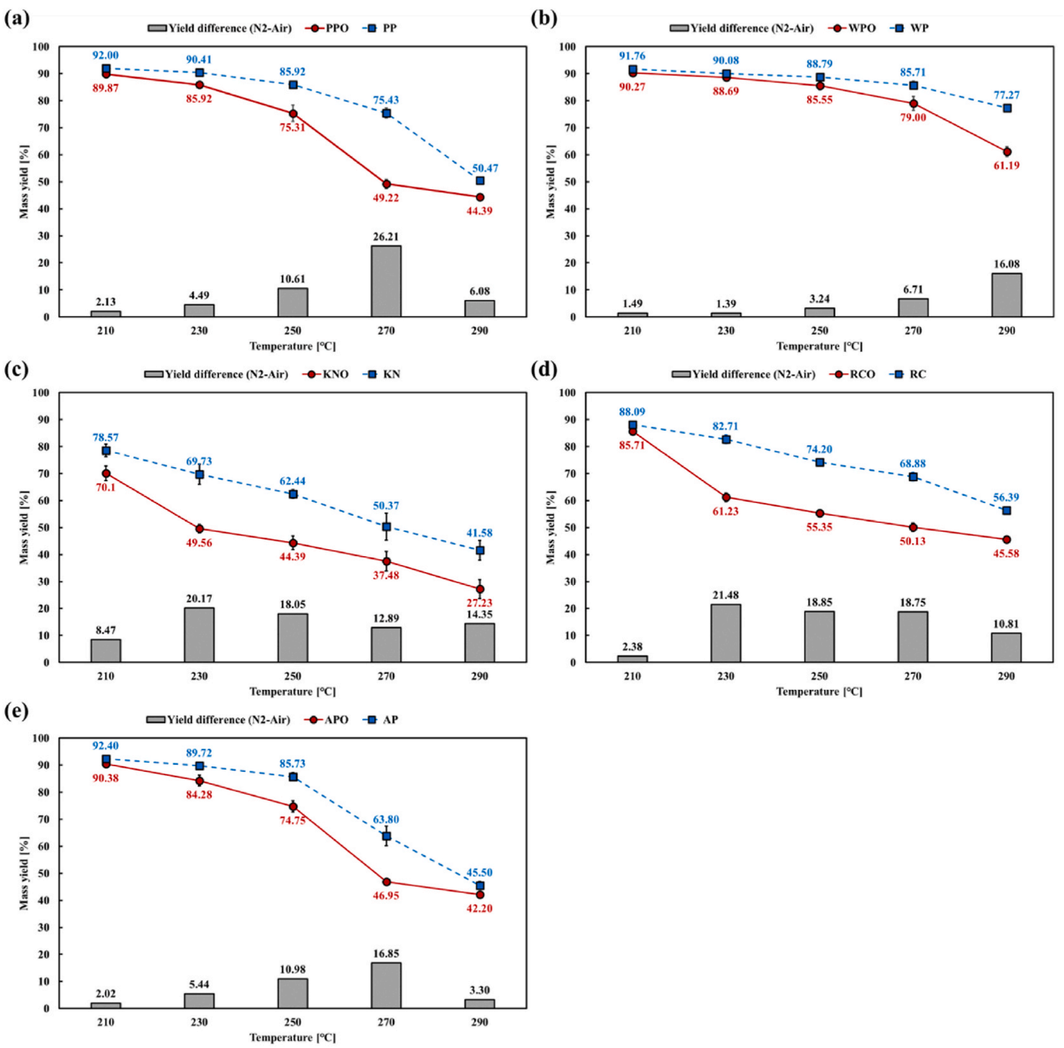

Fig. 3. Mass yield of each biomass.

significant difference at 290 ℃ (Fig. 3 a and e). However, under reducing conditions, a gradual mass loss was observed from 250 to 290 ℃. The rapid mass loss observed in WP at 290 ℃ (Fig. 3 b) under oxidative conditions was higher than that in other types of biomass because of its high thermal resistance. Herbaceous biomass showed rapid mass loss at 230 ℃ owing to its low lignin content (Fig. 3c and d). At the same temperature conditions, lignocellulosic biomass, i.e., PP, WP, and AP, showed higher mass loss under oxidative conditions than under oxygen-deficit conditions. The mass yield under oxidative conditions was similar to that under oxygen-deficit conditions at a 20 ℃ higher temperature. However, the mass loss tendencies of herbaceous and lignocellulosic biomass were different. For example, no significant difference was observed between PP210, PP230, and PPO230. However, rapid mass loss was observed at temperatures above 230 ℃ under oxidative conditions. In addition, the mass yields of RCO250 and RC290 were similar, and the KNO270, KN230, and KN250 were similar. These results suggest that the herbaceous biomass (KNO and RCO) has a higher thermal degradability than lignocellulosic biomass.

3.2. Impacts on fuel characteristics

According to the results of the elemental analysis (Fig. 4), all biomass, except for RCO, showed a gradual increase in the carbon concentration ratio and a decrease in the oxygen concentration ratio with increasing temperature (Fig. 4a, b, d, and e). Changes in concentration ratio occurred because of devolatilization due to the release of oxygen and hydrogen. Significant increases in the carbon ratio were observed in PPO270, APO270, WPO290, and KNO230, which were similar to the decrease in rapid mass yield. The carbon concentration ratio in RCO gradually increased to 250 °C, with an approximately 2% decrease in carbon at 270 °C because of less decrease in mass (Fig. 4c).

The van Krevelen diagram in Fig. 5 demonstrates the improvement in fuel characteristics of all biomass. A more significant improvement in fuel characteristics was observed under oxidative conditions (Fig. 5a, b, e). Most torrefied biomass showed linear

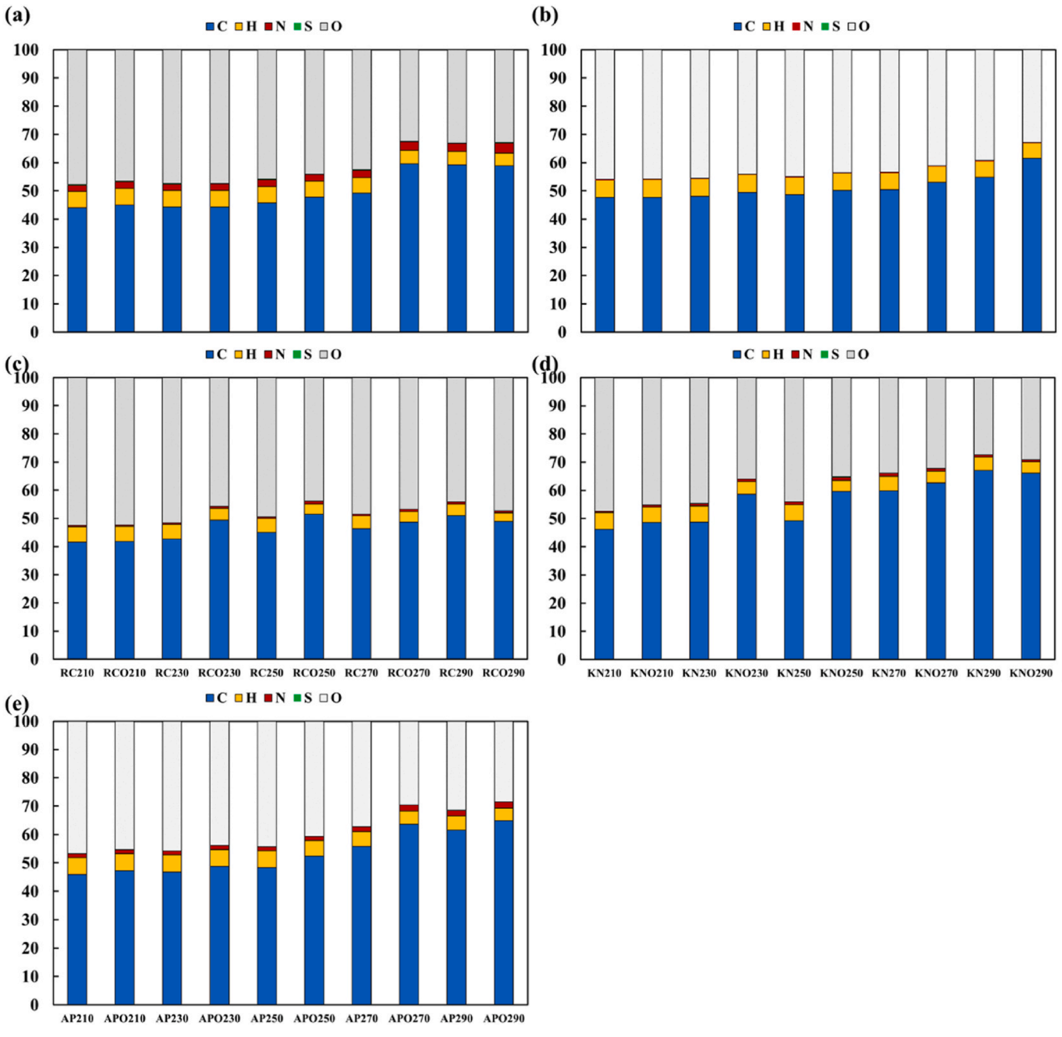

Fig. 4. Average result of elemental composition of each torrefied biomass (3 repetitions).

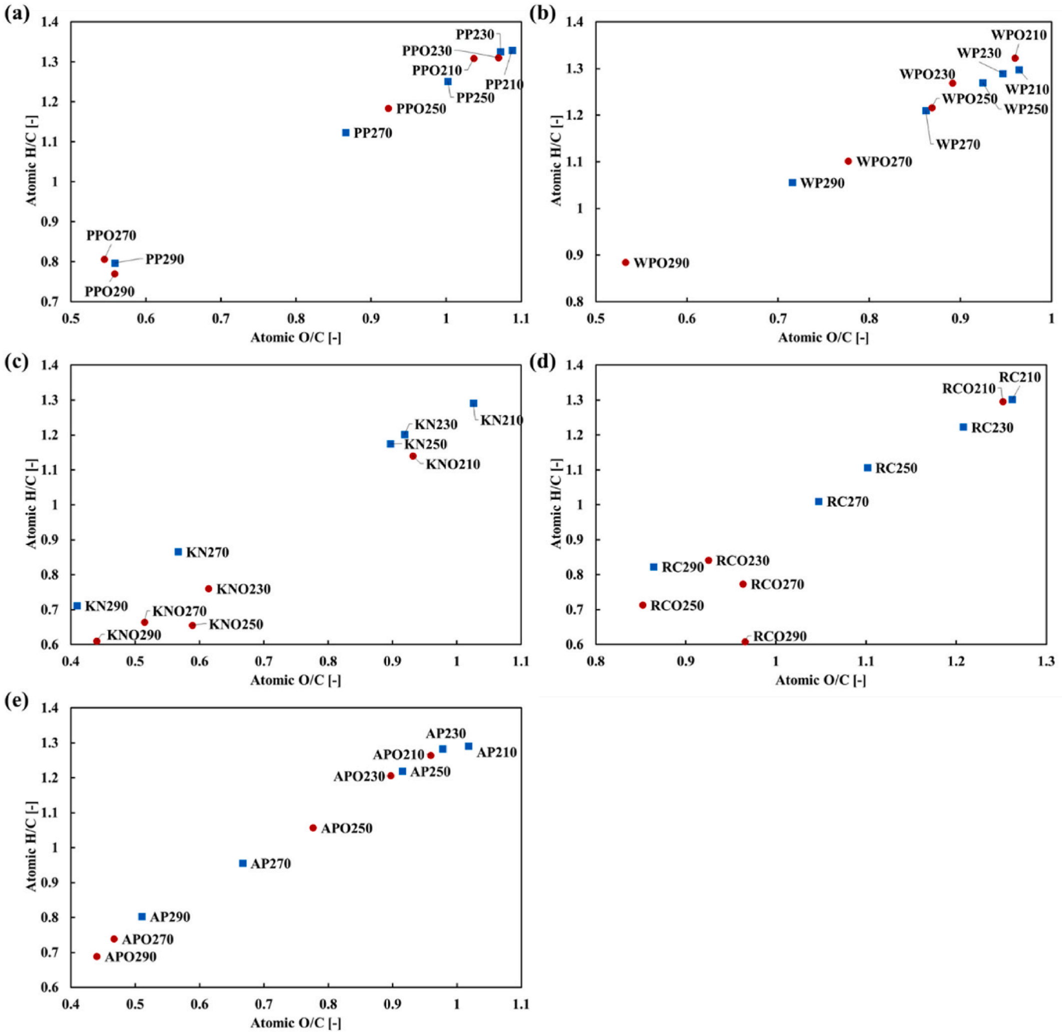

Fig. 5. Van Krevelen diagram of each torrefied biomass (average of 3 repetitions).

improvement in fuel characteristics, whereas KNO and RCO showed nonlinear improvement (Fig. 5c and d). In KNO, the improvement was nonlinear owing to the rapid decrease in hydrogen concentration at 250 °C. In RCO, the decrease was caused by a decrease in carbon and hydrogen concentrations at temperatures above 250 °C. Unlike other biomass, it was determined that there was no rapid change in mass.

3.3. Impacts on calorific value and energy yield

Fig. 6 summarizes the changes in the calorific value and energy yield about process conditions. Calorific value and energy yield were represented by bar and line graph, respectively. The calorific value was higher at high temperatures and in the presence of oxygen. Based on Duncan's multiple range test, the calorific value of biomass under oxidative conditions was similar to that under oxygen-deficit conditions at a 20 °C higher temperature, similar to the change in mass yield. The calorific value of PPO was 4570–6060 kcal/kg, and PP was 4500–5800 kcal/kg (Fig. 6a). Lignocellulosic biomass showed a 0.14–19.60% higher calorific value under oxygen-rich conditions than oxygen-deficit conditions (Fig. 6b and e). Herbaceous biomass led to a 3.98–20.02% higher calorific value under oxygen-rich conditions than oxygen-deficit conditions (Fig. 6c and d). This indicates that this increase in calorific value was caused by mass loss during the torrefaction process.

The energy yield was more than 90% for PP250, AP250, PPO230, and APO230 (Fig. 4). However, a rapid decrease in energy yield was observed in PPO270 and APO250. Under the same temperature, oxygen-rich torrefaction showed less energy yield than that in the oxygen-deficit condition, except for RC under 210 °C because of a higher decrease in mass compared to an increase in heating value. The energy yield of PPO270, AP270, PP290, AP290, PPO290, and APO290 was below 65%. The energy yield of WP was 90% for WP270 and WPO250 but decreased to approximately 85% for WPO270 and WP290. The energy yield of WPO290 was 73%.

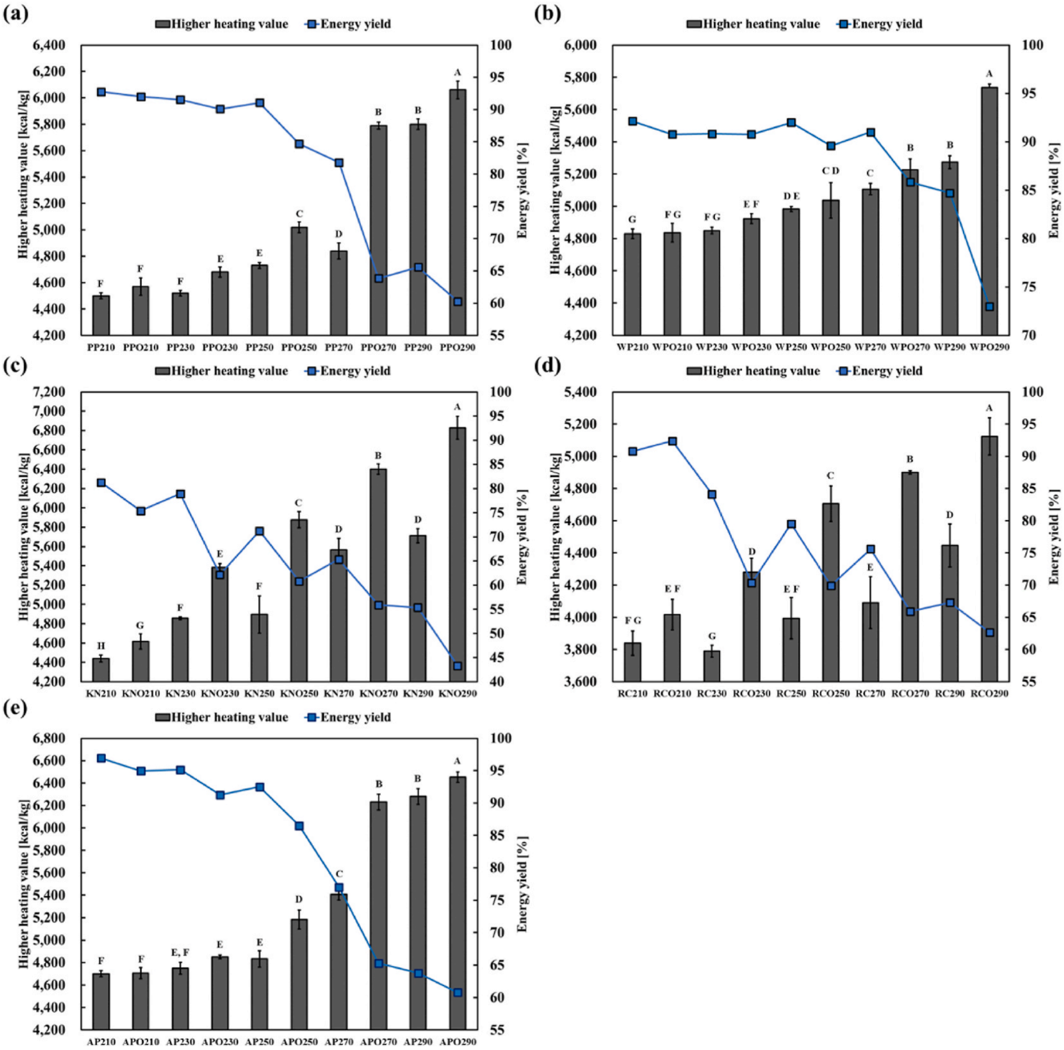

Fig. 6. Calorific value and energy yield of each biomass (average of 3 repetitions). * The same letter indicates no significant difference.

Herbaceous biomass led to a significant decrease in energy yield from 230 °C. It was observed that all types of biomass showed lower energy yields under oxygen-rich conditions than those under oxygen-deficit conditions at the same temperature. However, the energy yield of RCO210 was 92.39%, which was 1.61% higher than RC210. KN210 and KNO210 showed energy yields of 81.28% and 75.40%, respectively, which were lower than other types of biomass. KNO270 and KN290 showed a similar energy yield of approximately 55.5%. The energy yield decreased sharply under the process conditions, marked by a rapid decrease in the calorific value. It was determined that this was related to the decrease in the mass yield. The effect of oxygen was more critical in herbaceous biomass than in lignocellulosic biomass. Considering the rapid decrease in energy yield, suitable oxygen-lean and air-oxidative torrefaction process conditions for RC and KN were under 250 °C and 230 °C, respectively. For AP and PP, each oxygen-lean and air-oxidative torrefaction process under 270 °C and 250 °C were selected as optimal conditions, respectively. For WP, under 290 °C with the oxygen-lean condition and under 270 °C with the air-oxidative condition were selected as optimal processing conditions.

3.4. Impacts on gases emitted during torrefaction

The reactor atmosphere was evaluated at the highest temperature, i.e., 290 °C, to analyze the changes in gases emitted during torrefaction. The results are summarized in Fig. 7. Under oxygen-deficit conditions, the O_2 concentration ranged between 18% and 20% during the experimental period. However, the O_2 concentration tended to decrease to a minimum of 10%. It was determined that the **decomposition by oxidation** of each biomass was accelerated by reacting with O_2, resulting in an increase in CO and CO_2. In the case of lignocellulosic biomass, concentrations of CO_2 and CO increased after ~30 min under oxidative conditions, implying that the thermal decomposition temperature of hemicellulose, cellulose, and lignin was attained after 30 min (Fig. 7a, d, and e). However, in herbaceous biomass, a rapid increase in CO_2 and CO concentrations was observed owing to the lower lignin content and bulk density

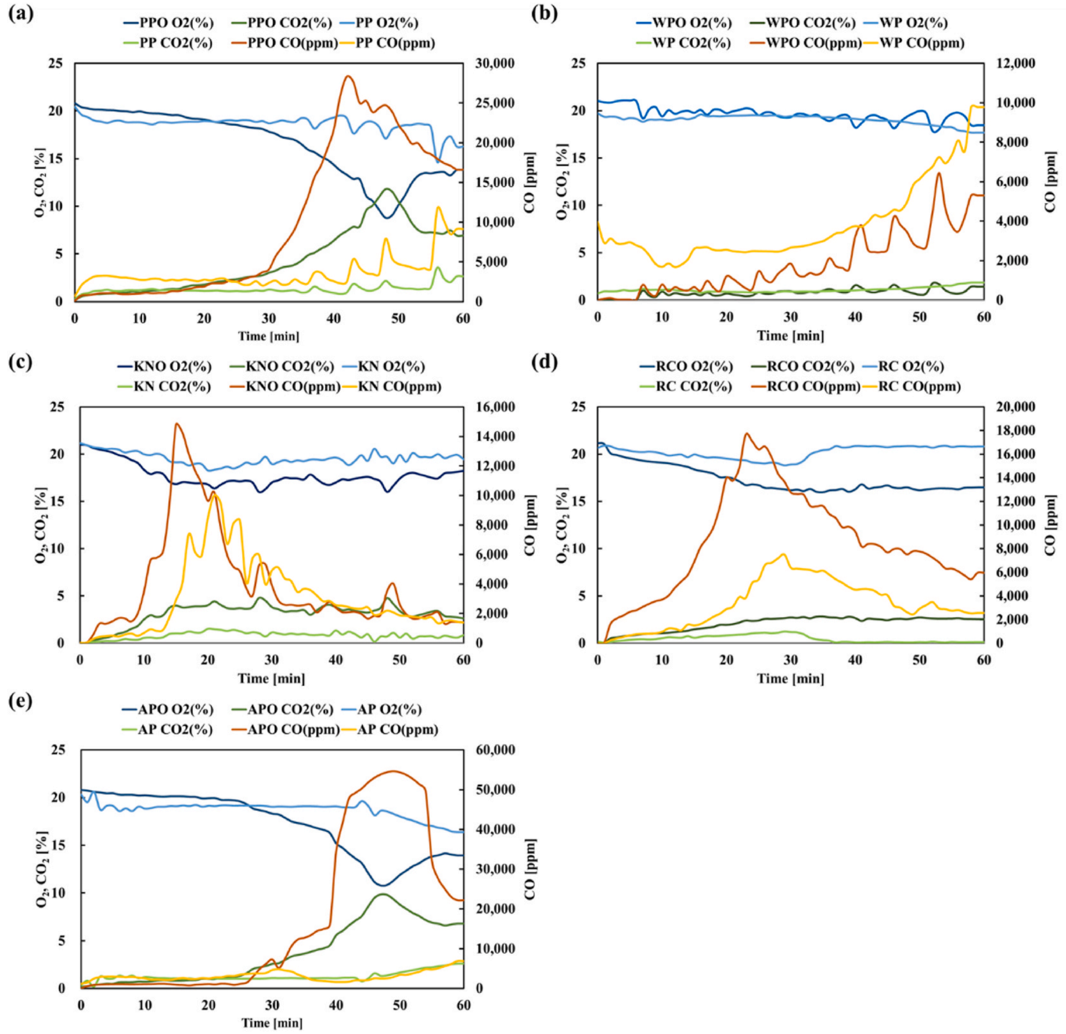

Fig. 7. Changes in gases emitted during torrefaction of each biomass.

than that of lignocellulosic biomass (Fig. 7b and c). WPO showed a maximum CO concentration of 7441 ppm, whereas WP showed a CO concentration of 9917 ppm, which is approximately 133% of that of WPO. At 290 °C, in the case of WPO, CO was rapidly converted to CO_2 owing to sufficient oxygen under oxidative conditions. However, in the case of WP, CO did not sufficiently change to CO_2 owing to reducing conditions.

3.5. *Impacts on energy–mass co-benefit index*

Fig. 8 presents the profiles of EMCI and ΔEMCI. The maximum values of EMCI for most of the biomass, except for KN and KNO, were observed at 290 °C, implying that this was the optimal temperature for torrefaction (Fig. 8a, b, c, and e). The maximum values of EMCI for KN and KNO were observed at 270 °C (Fig. 8d) Although the energy yield tends to decrease due to mass decrease, ΔEMCI can confirm the increase and decrease according to the torrefaction process, and it is feasible to suggest the optimal condition according to the temperature based on the difference in ΔEMCI than by considering the increase in energy yield. Therefore, it was determined that the conditions that led to the highest ΔEMCI were likely optimal for that biomass. Based on this, optimal conditions for each biomass were determined as follows: PP290, PPO250, WP250, WPO290, AP270, APO270, RC250, RCO210, KN230, and KNO230. Hence, it can be suggested that the torrefaction process of agro-byproducts can be carried out using flue gas as its O2 concentration ranges from 0 to 21%.

4. Conclusions

This study focused on comparing the effects of oxygen and temperature conditions on the torrefaction of agro-byproducts. Lignocellulosic biomass showed mass yields of 90.27–42.20% and 92.00–45.50% under oxygen-rich and oxygen-deficit conditions,

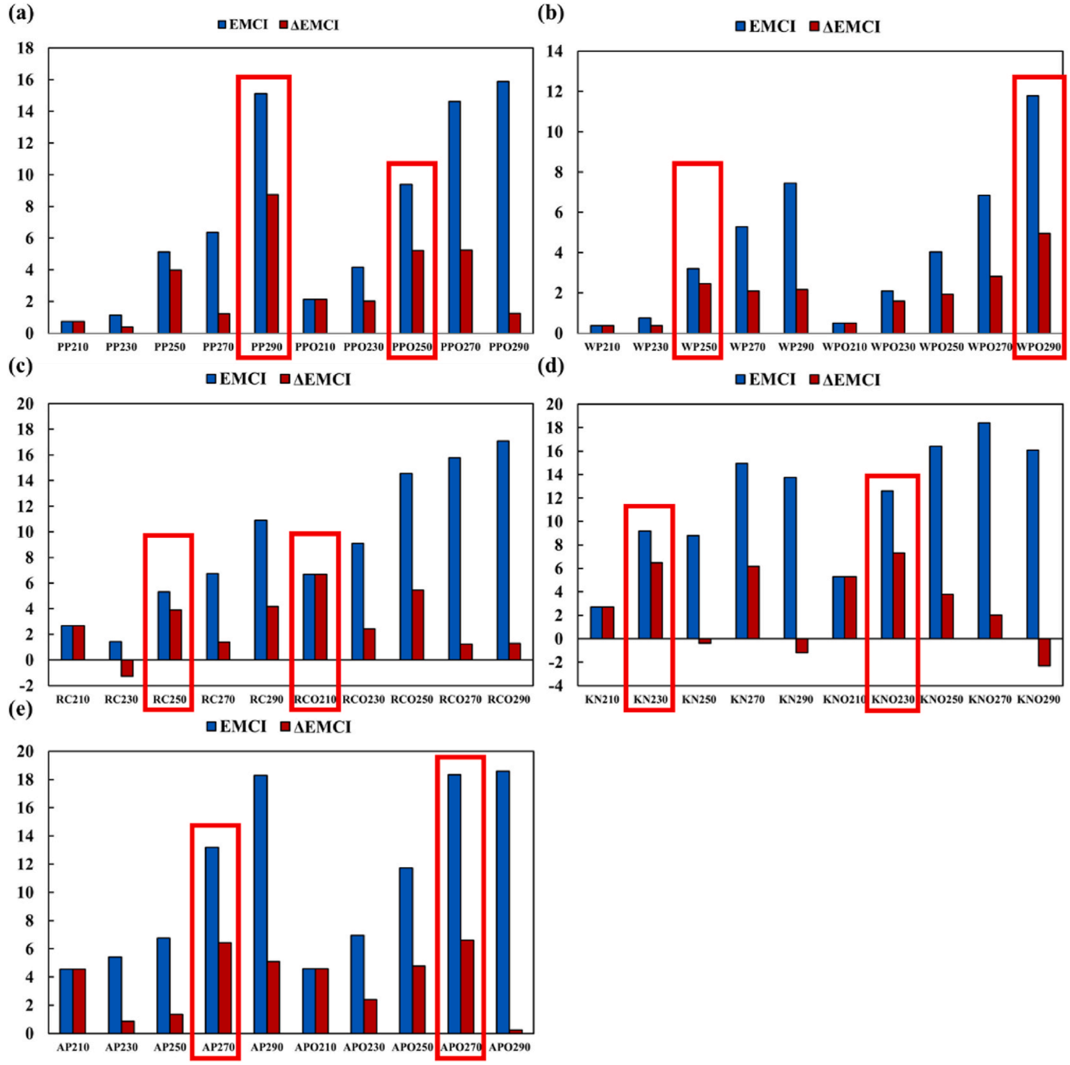

Fig. 8. Energy–mass co-benefit indexes (EMCI) and ΔEMCI of each biomass (Red boxes for optimal condition). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

respectively. Herbaceous biomass showed mass yields of 85.71–27.23% and 88.09–41.58% under oxygen-rich and oxygen-deficit conditions, respectively. Under oxygen-rich conditions, the calorific values of lignocellulosic and herbaceous biomass were 0.14–19.60% and 3.98–20.02% higher, respectively, than those under oxygen-deficit conditions. This was because of the mass loss caused by reactions with oxygen. The energy yield of lignocellulosic biomass under oxygen-rich and deficit conditions were 63.78–96.93% and 90.77–44.39%, respectively. Herbaceous biomass showed 88.09–41.58% and 92.38–27.23% of energy yield under oxygen-rich and deficit conditions. In addition, performance evaluations of torrefaction were conducted using EMCI and ΔEMCI. Decreases of ΔEMCI were observed under certain conditions as follows: RC230, KN250, KN290, and KNO290. Based on these results, torrefaction under 290 °C for herbaceous biomass is not recommended. Overall, the optimal conditions were determined to produce solid fuel with high energy density and low mass loss as follows: PP270, PPO250, WP250, WPO270, AP270, APO250, RC250, RCO210, KN230, and KNO230. In summary, this study recommends the use of oxidative torrefaction due to its advantages of not requiring the maintenance of reduction conditions and being able to operate at relatively low process temperatures. However, it should be improved for the high mass loss compared with oxygen-lean condition. Also, this study was conducted on a lab-scale using limited agro-byproducts. Therefore, in a future study, it is necessary to evaluate various agro-byproducts at pilot-scale using empirical techniques. Furthermore, techno-economic analysis should also be performed.

Author contribution statement

Sun yong Park: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Seok Jun Kim: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Kwang Cheol Oh: Conceived and designed the experiments.

Lahoon Cho: Performed the experiments.

Young Kwang Jeon: Analyzed and interpreted the data.

DaeHyun Kim: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

No data was used for the research described in the article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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