

# PRODUCTION OF TORREFIED BIOMASS PELLETS FROM WOODY AND AGRICULTURAL RESIDUES

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## Abstract

Two different combined process sequences of biomass pretreatment between pelletization after torrefaction (PAT) and pelletization before torrefaction (PBT) were comparatively investigated to produce torrefied biomass pellets (TBP) from woody biomasses, e.g. *Leucaena* (LC) and rubberwood (RW), and agricultural residues, e.g. rice straw (RS) and sugarcane leaves (SCL). In this study, each sample was thermally treated at 260-300°C for 5 min during torrefaction process. It was found that both woody biomasses and agricultural residues had mass yield lower than 63 wt%, while the bulk density of TBPs were improved higher than 400 kg/m<sup>3</sup>. For equilibrium moisture content (EMC) analysis, TBPs via PBT method had lower EMC than raw pellet after being kept at 30°C for 12 days. For the thermochemical properties, the TBPs had higher FC, %C, and HHVs than raw pellets in all biomass and increased with torrefaction temperature. When comparing the TBPs between PAT and PBT torrefied pellets, the HHVs of PBT torrefied pellets at 300°C were achieved highest at 27 MJ/kg (dry-ash-free, daf, basis) for SCL sample, which was considered as higher than the standard value at ≥21 MJ/kg of thermally treated biomass pellets (ISO/TS 17225-8:2016) and also in the range at 25.7-28.2 MJ/kg of coal. In addition, the combustion performance index ( $S_n$ ) of PAT and PBT torrefied pellets was lower than raw pellets, showing a similar property as coal and lignite. Briefly, this study suggests using PBT pretreatment process to produce high quality solid fuel, particularly for agricultural residues such as SCL for a potential substitute of currently used coal.

**Keywords:** Biomass; fuel properties; pelletization; torrefaction; torrefied pellets

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## Introduction

Utilization of dry biomass for heat or power generation in industries via thermo-chemical conversions can reduce energy cost and improve environmental issue of green house gas emission by replacing currently used fossil fuels. However, direct usage of biomass still has several disadvantages of high moisture content, high oxygen content, low bulk density, low calorific value, and low energy density, leading to lower fuel efficiency. To overcome these problems, physical and/or thermal pretreatment, such as pelletization and torrefaction, of biomass is necessary. Pelletization is one of densification processes by compacting biomass material to form a densified solid fuel under heat and pressure. This pelletization method can only increase bulk and apparent density of biomass, but calorific value is still the same low with high oxygen content (Oberberger and Thek, 2010; Said *et al.*, 2015; Valdés *et al.*, 2018; Gilvari *et al.*, 2019; Ríos-Badrán *et al.*, 2020; Mian *et al.*, 2020). Torrefaction, therefore, is another thermal treatment to improve these properties. Torrefaction, so called as mild pyrolysis, is normally carried out at low temperature range of 200-350°C under inert atmosphere. It can enhance fuel properties of biomass such as an increase in heating value and energy density by decreasing O/C ratio in biomass structure, while decreasing volumetric density (Wannapeera and Worasuwanarak, 2012; Strandberg *et al.*, 2015; Inthapat, 2016; Sankaran *et al.*, 2018; Chih *et al.*, 2019; García Nieto *et al.*, 2019). Thus, the combination of pelletization and torrefaction has been proposed to increase density and calorific value, respectively, for easy handling and storage, low transportation cost, including high performance and efficiency of subsequent utilization of high-quality solid fuels closed to lignite coals, mostly used in various industries.

Torrefied biomass pellets (TBP) can be produced by two different processes, (i) pelletization after torrefaction (PAT) and (ii) pelletization before torrefaction (PBT). For PAT process, raw biomass is torrefied first, then torrefied biomass is fed into palletization process. The production of PAT torrefied pellets may have a difficulty in the palletization of torrefied biomass due to its loss of the binder nature of hemicellulose in biomass structure during torrefaction process. So, this production method may require the binder such as corn starch to glue between particle during pelletizing (Stelte *et al.*, 2011; Peng *et al.*, 2013a; 2013b; Sarkar *et al.*, 2014; Brachi *et al.*, 2018; Manouchehrinejad and Mani, 2018).

In case of PBT process, biomass is compacted as biomass pellets prior to be torrefied under thermal inert condition. Pelletization in the PBT process with or without binding agents had been investigated by several researchers. It was found that the binder helped to improve densification characteristics and reduced compression energy required (Hu *et al.*, 2015; Brachi *et al.*, 2018; Manouchehrinejad and Mani, 2018; Brachi *et al.*, 2019). Both PAT and PBT processes can be simply applied to produce the high-quality solid fuels. The comparison between PAT and PBT processes, therefore, is very important to select the most suitable process of TBP production.

Recently, Ghiasi *et al.* (2014) studied on the energy consumption, mass and energy balance, and quality properties of PAT and PBT torrefied biomass pellets using Douglas-fir from woody biomass as a raw material. They used torrefaction temperature at 260°C for 15 min to produce the TBP. In PAT process, during pelletization, the sample was mixed with water and wheat flour binder to produce the TBP. But the PBT torrefied pellets were compacted by using only water to mix with the sample. The PAT process required energy consumption only 2.31 MJ, which was lower than PBT process at 2.49 MJ. Torrefied biomass used in PAT process had high friction in die holes, it caused high torque to drive the pellet mill compared with raw biomass using in PBT process. But they use a binder for the PAT process that can increase the interparticle bonding to enhance the binding affinity of the torrefied material. So, it caused specific electrical energy consumption such as drying and pelletization processes (Ghiasi *et al.*, 2014). The bulk density of PAT torrefied pellet (1,207 kg/m<sup>3</sup>) was also higher than that of PBT torrefied pellet (1,031 kg/m<sup>3</sup>). In contrast, the carbon content and higher heating value (HHV) of PBT torrefied pellet (51 wt% and 22.0 MJ/kg) were slightly higher than those of PAT torrefied pellet (49.5 wt% and 20.8 MJ/kg). Soponpongpiat *et al.* (2019) had focused on the study of properties and pyrolysis characteristics of torrefied biomass pellets using cassava rhizome from agricultural residue as a raw biomass. They used three different torrefaction temperature of 230-280°C for 30 min and used only water as a binder in pelletization process. In this study, the bulk density of PBT torrefied pellet (486-583 kg/m<sup>3</sup>) was lower than those of PAT torrefied pellet (740-777 kg/m<sup>3</sup>), which were in a good agreement with Ghiasi *et al.* (2014). Moreover, the HHVs and maximum moisture adsorption of

PBT torrefied pellets (18.63-23.83 MJ/kg and 3.73-4.64%) were clearly higher than those of PAT torrefied pellet (17.56-22.3 MJ/kg and 1.95-2.82%). For Yoshida *et al.* (2021), they studied the effect of torrefaction at 215-300°C for 60 min in each condition of TBP, on the water resistance both indoor and outdoor test. The TBPs were pelletized using only water with woody biomasses from cedar and oak as their raw biomass.

So, the comparison between PAT and PBT processes using different biomass types from woody and agricultural biomasses are still lack of information. Moreover, combustion investigation of PAT and PBT torrefied pellets is necessary for subsequent applications. The aims of this study were to evaluate the influence of both torrefaction and pelletization on the physical properties of TBP using four biomass types from woody biomasses and agricultural residues. Then, fuel properties of TBP prepared from two different process sequences such as fixed carbon (FC), the percentage of carbon content (%C) and higher heating value (HHV), including mass yield, bulk density and equilibrium moisture content (EMC), were deeply investigated and compared. In this study the standard values of thermally treated biomass pellets from ISO/TS 17225-8:2016 was used to compare major fuel properties of the prepared TBPs via both PAT and PBT process; 1) bulk density ( $\geq 650 \text{ kg/m}^3$ ), 2) ash content ( $\leq 3\%$ ), and 3) net calorific value ( $\geq 21 \text{ MJ/kg}$ ). Finally, the subsequent combustion behavior of PAT and PBT torrefied pellets were also compared.

## Experimental

### Raw Materials

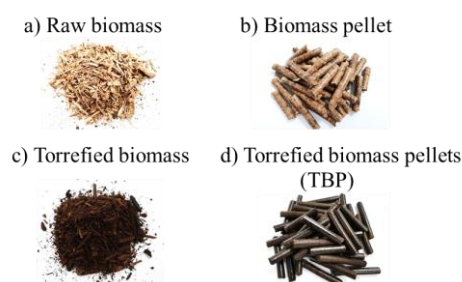
Leucaena (LC) and rubberwood (RW) of woody biomass, and rice straw (RS), and sugarcane leaves (SCL) of agricultural residue, were prepared in a size range of 0.5-1.0 cm (No. 18 and 35 mesh) as shown in Figure 1(a) prior to perform in a combined pelletization and torrefaction process. The products can be seen in Figures 1(b)-(d) as biomass pellet, torrefied biomass, and torrefied biomass pellets (TBP), respectively.

### Pelletization Process

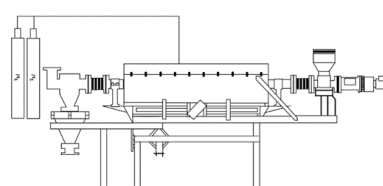
Figures 1(b) and 1(d) showed raw and torrefied biomass pellets (TBP), respectively, with a diameter of 6 mm and a length of 50 mm in size, prepared by the flat die pellet mill (Farmtech, PM-100M). Before pelletization, raw and torrefied biomass (Figures 1(a) and 1(c)) were mixed with 10 wt% water and 2 wt% corn starch as binders.

### Torrefaction Process

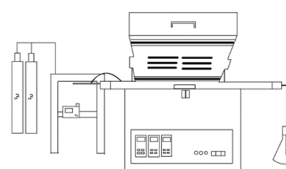
The torrefaction process was carried out at a heating rate of 10°C/min to three different temperatures of 260, 280, and 300°C for the holding time of 5 min under an inert atmosphere using nitrogen ( $\text{N}_2$ ) gas. The production of torrefied biomass prior to be pelletized for PAT samples was performed using a continuous vibrating furnace as shown in Figure 2(a). The samples approximately 12 kg were fed into this continuous vibrating reactor with a mass flow rate of 10 kg/h. However, raw biomass pellets were torrefied in a fixed-bed tube furnace, as shown Figure 2(b), to produce PBT samples under the same torrefaction conditions used for PAT sample preparation. Figure 3 shows a research scope in this study by comparing two different process sequences of biomass pretreatment



**Figure 1.** Raw biomass (woody and agricultural residue sample) and product samples used and prepared in this work a) raw biomass, b) biomass pellets, c) torrefied biomass, and d) torrefied biomass pellets (TBP)



a) A continuous vibrating furnace



b) A fixed-bed tube furnace

**Figure 2.** Furnaces used for torrefaction process in this study a) a continuous vibrating furnace and b) a fixed-bed tube furnace

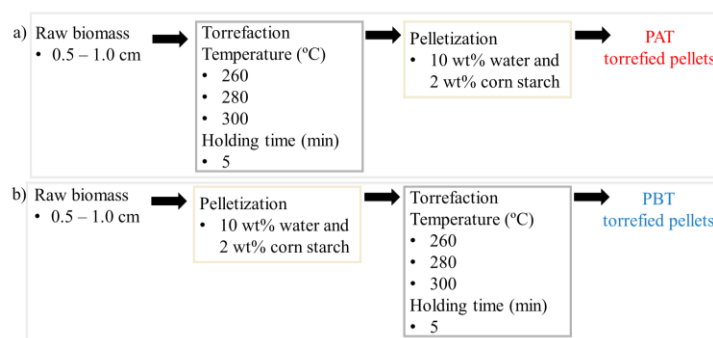


Figure 3. Two different process sequences of (a) PAT and (b) PBT for biomass pretreatment

method (a) pelletization after torrefaction (PAT) and (b) pelletization before torrefaction (PBT).

#### Determination of Mass Yield and Bulk Density of Raw and Torrefied Pellets

Mass yield of TBP samples obtained from PAT and PBT processes were measured by weighing the samples before and after torrefaction process. The bulk density of raw pellets and TBP samples was determined using the random packing method based on a 50-mL beaker container for mass in weight per unit volume.

#### Equilibrium Moisture Content Analysis

Equilibrium moisture content (EMC) of raw biomass pellets and TBP samples was analyzed by the different relative humidity at 30°C in the range of 32% to 96% through four different salt solution types of 31.6%  $\text{MgCl}_2$ , 48.4%  $\text{Mg}(\text{NO}_3)_2$ , 82.3%  $\text{KCl}$ , and 96.4%  $\text{K}_2\text{SO}_4$ . Before analysis, the sample was dried at 105°C overnight, then kept and weighed each day at 2 to 12 days. The EMCs of raw biomass pellets and TBP samples were then calculated using Equation (1), where  $W_e$  and  $W_d$  refer to equilibrium weight and dry weight of the sample by Acharjee *et al.* (2011) and Wattananoi (2011).

$$\text{EMC} = ((W_e - W_d) / W_d) \times 100 \quad (1)$$

#### Proximate Analysis

The percentage weight (wt%) of volatile matter (VM) and ash were measured using a thermogravimetric analyzer (TGA-50, Shimadzu) with the unit on a dry basis (db) for both raw pellets and TBP samples. At the same time, fixed carbon (FC) content was determined by difference. After moisture removal at 110°C, the reduced size of the pellet products, approximately 5–20 mg, was heated up to 900°C at a heating rate of 10°C/min under  $\text{N}_2$  atmosphere. After that the atmosphere was changed

from  $\text{N}_2$  to air zero and held for 10 min for proximate analysis to obtain the ash value in the last step.

#### Ultimate Analysis

The weight percentages of carbon (%C), hydrogen (%H), and nitrogen (%N) contents of raw pellets and TBPs (dry samples) were measured by an CHNO analyzer (J-SCIENCE LAB Co., Ltd, JM10 Micro Corder, Japan) on with the unit of dry-ash-free (daf). Whereas the oxygen content (%O) was determined by difference. Moreover, higher heating value (HHV) were calculated from its elemental composition by using Equations (2) and (3). The Equation (2) is an ordinary least squares regression (OLS) and Equation (3) is a partial least squares regression (PLS) model. The results from both equations were averaged (Equation 4) to calculate the HHVs on a dry ash free basis (daf) (Bridgeman *et al.*, 2008). Then the HHVs (daf) on the unit of kJ/kg was converted into MJ/kg. The elemental composition was also used to calculate the H/C and O/C ratio.

$$\text{HHV (OLS)} = 1.87C^2 - 144C - 2802H + 63.8CH + 129N + 20147 \quad (2)$$

$$\text{HHV (PLS)} = 5.22C^2 - 319C - 1647H + 38.6CH + 133N + 21028 \quad (3)$$

$$\text{HHV (d.a.f., kJ/kg)} = \frac{\text{HHV (OLS)} + \text{HHV (PLS)}}{2} \quad (4)$$

#### Combustion Behavior Analysis

The raw biomass pellets and TBP samples were investigated and observed using thermogravimetric analyzer, TGA (TGA-50, Shimadzu) under air atmosphere to study combustion behavior. The sample was cut into a smaller size before heated up from 110 to

**Table 1. Property of raw and torrefied biomass pellet for all biomass**

| Leucaena (LC)          |       |         |       |     |      |       |     |      |       |     |      |
|------------------------|-------|---------|-------|-----|------|-------|-----|------|-------|-----|------|
|                        | Raw   |         | 260   |     |      | 280   |     |      | 300   |     |      |
|                        | Chips | Pellets | Chips | PAT | PBT  | Chips | PAT | PBT  | Chips | PAT | PBT  |
| Yield (wt%)            | -     |         | 84.0  |     | 82.5 | 77.9  |     | 74.3 | NA    |     |      |
| Bulk density (kg/m³)   | 260   | 650     | 200   | 560 | 536  | 109   | 540 | 508  | NA    |     |      |
| Rubberwood (RW)        |       |         |       |     |      |       |     |      |       |     |      |
|                        | Raw   |         | 260   |     |      | 280   |     |      | 300   |     |      |
|                        | Chips | Pellets | Chips | PAT | PBT  | Chips | PAT | PBT  | Chips | PAT | PBT  |
| Yield (wt%)            | -     |         | 87.6  |     | 87.4 | 84.4  |     | 75.2 | 79.7  |     | 63.5 |
| Bulk density (kg/m³)   | 250   | 630     | 200   | 588 | 570  | 185   | 581 | 565  | 165   | 565 | 540  |
| Rice straw (RS)        |       |         |       |     |      |       |     |      |       |     |      |
|                        | Raw   |         | 260   |     |      | 280   |     |      | 300   |     |      |
|                        | Chips | Pellets | Chips | PAT | PBT  | Chips | PAT | PBT  | Chips | PAT | PBT  |
| Yield (wt%)            | -     |         | 87.3  |     | 89.9 | 68.4  |     | 68.8 | 51.1  |     | 51.9 |
| Bulk density (kg/m³)   | 84.9  | 627     | 90.0  | 601 | 591  | 78.5  | 526 | 481  | 81.0  | 551 | 394  |
| Sugarcane leaves (SCL) |       |         |       |     |      |       |     |      |       |     |      |
|                        | Raw   |         | 260   |     |      | 280   |     |      | 300   |     |      |
|                        | Chips | Pellets | Chips | PAT | PBT  | Chips | PAT | PBT  | Chips | PAT | PBT  |
| Yield (wt%)            | -     |         | 67.5  |     | 73.3 | 54.2  |     | 61.4 | 50.6  |     | 49.7 |
| Bulk density (kg/m³)   | 82.9  | 537     | 92.5  | 563 | 517  | 77.3  | 586 | 470  | 77.3  | 622 | 417  |

600°C with a heating rate of 10°C/min under air condition. The resulting data was used to plot thermogravimetric (TG) and derivative thermogravimetric (DTG) curves to estimate the combustion behaviors and parameters including ignition temperatures ( $T_i$ ), burnout temperature ( $T_b$ ), the average rate of the weight loss  $(dw/dt)_{mean}$  and the maximum rate of weight loss  $(dw/dt)_{max}$ . Finally, these parameters were used to evaluate combustion performance index ( $S_n$ ) by using Equation (5) (Nie *et al.*, 2001).

$$S_n = \frac{(dw/dt)_{max}(dw/dt)_{mean}}{T_i^2 T_b} \quad (5)$$

## Results and Discussions

### Mass yield and Bulk Density

Mass yield and bulk density of TBP samples for each pretreatment condition are shown in Table 1. The mass yield of TBP samples prepared by both PAT and PBT methods significantly decreased with increasing torrefaction temperature from 260°C to 300°C. After torrefaction at 300°C, the torrefied mass yields were approximately 63-80 wt% for woody biomass (RW samples), which were higher than those obtained from agricultural residues (RS and SCL) decreased to 49-52 wt%. The results showed that TBP derived from agricultural residues (RS and SCL) exhibited relatively higher thermal decomposition during torrefaction leading to a severe decrease in mass

yield due to their higher thermal decomposition of hemicellulose with higher ash content at about 10 wt%, which may act as a catalyst. The data of ash content was reported and discussed later in proximate analyses. However, there was an experimental limitation for the LC sample torrefied at 300°C for only 5 min that could not be pelletized in the flat die due to its too dry condition. So, the data of LC sample torrefied at 300°C was not available in this study and designated as NA in Tables 1-3. Consequently, the torrefaction time under other conditions was also limited at this short period of time for only 5 min to avoid the difficulty during pelletization from too dry torrefied samples. Moreover, the torrefied mass yields of woody biomass (LC and RW) obtained from PAT method were higher than those of PBT method for all torrefaction temperatures, while they showed rather similar mass yields at about 50 wt% for agricultural residues (RS and SCL) once being torrefied at higher temperature of 300°C for both PAT and PBT methods.

Table 1 also shows the bulk density of raw biomass pellets and TBPs derived from LC, RW, RS, and SCL. The results expressed a significant increase in the bulk density of raw biomass pellets up to 530-650 kg/m<sup>3</sup> compared to those of raw biomass chip, which were clearly lower at 250-260 kg/m<sup>3</sup> for woody biomass and even much lower at 80-90 kg/m<sup>3</sup> for agricultural residues. However, the bulk density of TBPs had slightly decreased after the torrefaction process. The bulk density of TBPs mostly showed a slight decrease from 560-590

**Table 2. Proximate analysis, ultimate analysis, and HHVs of raw and torrefied biomass pellets for all biomass**

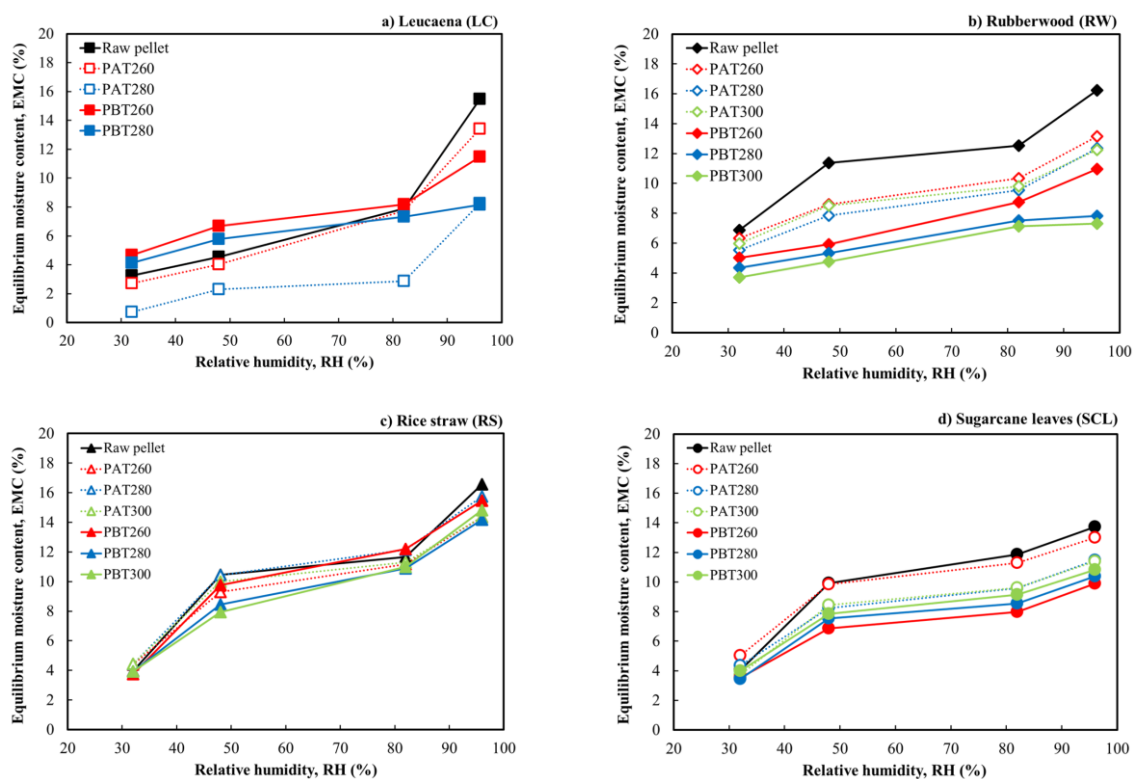
|                                           | <b>Leucaena (LC)</b>          |                         |            |            |                         |            |            |
|-------------------------------------------|-------------------------------|-------------------------|------------|------------|-------------------------|------------|------------|
|                                           | <b>Raw pellet</b>             | <b>PAT</b>              |            |            | <b>PBT</b>              |            |            |
|                                           |                               | <b>Temperature (°C)</b> |            |            | <b>Temperature (°C)</b> |            |            |
|                                           |                               | <b>260</b>              | <b>280</b> | <b>300</b> | <b>260</b>              | <b>280</b> | <b>300</b> |
| Proximate analysis (wt%, dry basis, d.b.) |                               |                         |            |            |                         |            |            |
| - Volatile matter (VM)                    | 81.4                          | 73.4                    | 71.2       | NA         | 73.7                    | 70.6       | NA         |
| - Fixed carbon (FC)                       | 16.9                          | 24.3                    | 26.1       | NA         | 24.2                    | 27.1       | NA         |
| - Ash                                     | 1.7                           | 2.4                     | 2.7        | NA         | 2.1                     | 2.2        | NA         |
| Ultimate analysis (wt%, dry-ash-free)     |                               |                         |            |            |                         |            |            |
| - C                                       | 46.7                          | 50.8                    | 53.4       | NA         | 52.1                    | 57.0       | NA         |
| - H                                       | 6.3                           | 6.0                     | 5.9        | NA         | 5.9                     | 5.9        | NA         |
| - O (different)                           | 46.6                          | 42.7                    | 40.1       | NA         | 41.5                    | 36.5       | NA         |
| Higher heating value, HHV (MJ/kg, daf.)   | 18.61                         | 20.30                   | 21.42      | NA         | 20.83                   | 23.08      | NA         |
|                                           | <b>Rubberwood (RW)</b>        |                         |            |            |                         |            |            |
|                                           | <b>Raw pellet</b>             | <b>PAT</b>              |            |            | <b>PBT</b>              |            |            |
|                                           |                               | <b>Temperature (°C)</b> |            |            | <b>Temperature (°C)</b> |            |            |
|                                           |                               | <b>260</b>              | <b>280</b> | <b>300</b> | <b>260</b>              | <b>280</b> | <b>300</b> |
| Proximate analysis (wt%, dry basis, d.b.) |                               |                         |            |            |                         |            |            |
| - Volatile matter (VM)                    | 85.6                          | 83.5                    | 79.0       | 77.4       | 80.9                    | 78.7       | 71.0       |
| - Fixed carbon (FC)                       | 11.6                          | 14.6                    | 18.6       | 19.9       | 16.7                    | 19.0       | 26.8       |
| - Ash                                     | 2.8                           | 1.8                     | 2.3        | 2.7        | 2.5                     | 2.4        | 2.1        |
| Ultimate analysis (wt%, dry-ash-free)     |                               |                         |            |            |                         |            |            |
| - C                                       | 48.0                          | 49.1                    | 49.2       | 51.1       | 50.7                    | 54.0       | 57.8       |
| - H                                       | 6.5                           | 6.6                     | 6.5        | 6.5        | 6.2                     | 6.1        | 5.8        |
| - O (different)                           | 45.1                          | 44.0                    | 44.0       | 42.1       | 42.8                    | 39.5       | 36.1       |
| Higher heating value, HHV (MJ/kg, daf.)   | 19.21                         | 19.72                   | 19.73      | 20.60      | 20.30                   | 21.77      | 23.36      |
|                                           | <b>Rice straw (RS)</b>        |                         |            |            |                         |            |            |
|                                           | <b>Raw pellet</b>             | <b>PAT</b>              |            |            | <b>PBT</b>              |            |            |
|                                           |                               | <b>Temperature (°C)</b> |            |            | <b>Temperature (°C)</b> |            |            |
|                                           |                               | <b>260</b>              | <b>280</b> | <b>300</b> | <b>260</b>              | <b>280</b> | <b>300</b> |
| Proximate analysis (wt%, dry basis, d.b.) |                               |                         |            |            |                         |            |            |
| - Volatile matter (VM)                    | 71.8                          | 68.4                    | 59.6       | 45.9       | 70.7                    | 60.8       | 39.8       |
| - Fixed carbon (FC)                       | 15.6                          | 17.9                    | 24.3       | 32.6       | 16.5                    | 22.1       | 35.4       |
| - Ash                                     | 12.6                          | 13.7                    | 16.1       | 21.5       | 12.8                    | 17.1       | 24.8       |
| Ultimate analysis (wt%, dry-ash-free)     |                               |                         |            |            |                         |            |            |
| - C                                       | 43.0                          | 44.3                    | 52.3       | 57.4       | 46.9                    | 52.7       | 61.9       |
| - H                                       | 6.2                           | 6.0                     | 5.8        | 4.9        | 6.0                     | 5.9        | 5.1        |
| - O (different)                           | 49.8                          | 48.9                    | 40.8       | 36.4       | 46.2                    | 40.3       | 31.3       |
| Higher heating value, HHV (MJ/kg, daf.)   | 17.18                         | 17.67                   | 20.95      | 22.65      | 18.71                   | 21.17      | 24.88      |
|                                           | <b>Sugarcane leaves (SCL)</b> |                         |            |            |                         |            |            |
|                                           | <b>Raw pellet</b>             | <b>PAT</b>              |            |            | <b>PBT</b>              |            |            |
|                                           |                               | <b>Temperature (°C)</b> |            |            | <b>Temperature (°C)</b> |            |            |
|                                           |                               | <b>260</b>              | <b>280</b> | <b>300</b> | <b>260</b>              | <b>280</b> | <b>300</b> |
| Proximate analysis (wt%, dry basis, d.b.) |                               |                         |            |            |                         |            |            |
| - Volatile matter (VM)                    | 74.4                          | 62.1                    | 52.9       | 49.5       | 59.4                    | 54.0       | 39.3       |
| - Fixed carbon (FC)                       | 15.1                          | 25.3                    | 31.5       | 33.2       | 23.5                    | 27.2       | 36.2       |
| - Ash                                     | 10.5                          | 12.6                    | 15.6       | 17.3       | 17.1                    | 18.8       | 24.5       |
| Ultimate analysis (wt%, dry-ash-free)     |                               |                         |            |            |                         |            |            |
| - C                                       | 45.8                          | 53.6                    | 58.5       | 59.6       | 53.8                    | 56.4       | 65.9       |
| - H                                       | 6.6                           | 6.0                     | 5.7        | 5.5        | 6.0                     | 5.7        | 5.3        |
| - O (different)                           | 46.8                          | 39.7                    | 34.8       | 33.8       | 39.3                    | 37.0       | 27.7       |
| Higher heating value, HHV (MJ/kg, daf.)   | 18.32                         | 21.59                   | 23.69      | 24.08      | 21.69                   | 22.71      | 26.96      |

kg/m<sup>3</sup> to 500-540 kg/m<sup>3</sup> when increasing torrefaction temperature from 260 to 300°C for woody biomasses. A decreasing degree in bulk density was significantly much higher from 510-590 kg/m<sup>3</sup> to 390-420 kg/m<sup>3</sup> for PBT samples derived from agricultural residue torrefied at 260 to 300°C. However, the PAT torrefied pellets of SCL showed an opposite trend of a slight increase in bulk density

after increasing torrefaction temperature. The bulk density of TBPs was decreased from raw pellets due to the removal of volatile matter, leading to more voids in biomass structure and less contacts between the particles in the pellets (Wang *et al.*, 2020). Some researchers explained the reason of decreasing pellet density due to the loss of chemically bonded water and low melting point compounds, which act as

**Table 3. Combustion characteristic parameters for raw and TBP at 300°C of all biomass**

|                                 | Leucaena (LC)   |         |         | Rubberwood (RW)        |         |         |
|---------------------------------|-----------------|---------|---------|------------------------|---------|---------|
|                                 | Raw pellet      | PAT_300 | PBT_300 | Raw pellet             | PAT_300 | PBT_300 |
| $T_i$ (°C)                      | 206.9           | NA      | NA      | 202.1                  | 207.3   | 237.2   |
| $T_b$ (°C)                      | 461.7           | NA      | NA      | 407.2                  | 463.9   | 475.8   |
| $(dW/dt)_{\text{mean}}$ (%/min) | 2.05            | NA      | NA      | 2.52                   | 2.05    | 2.05    |
| $(dW/dt)_{\text{max}}$ (%/min)  | 12.38           | NA      | NA      | 12.14                  | 11.10   | 10.31   |
| $S_n$ ( $\times 10^{-7}$ )      | 12.83           | NA      | NA      | 15.93                  | 11.39   | 7.88    |
|                                 | Rice straw (RS) |         |         | Sugarcane leaves (SCL) |         |         |
|                                 | Raw pellet      | PAT_300 | PBT_300 | Raw pellet             | PAT_300 | PBT_300 |
| $T_i$ (°C)                      | 201.8           | 205.7   | 220.3   | 194.7                  | 203.3   | 226.5   |
| $T_b$ (°C)                      | 473.4           | 474.3   | 468.6   | 479.4                  | 484.8   | 484.8   |
| $(dW/dt)_{\text{mean}}$ (%/min) | 2.05            | 1.99    | 2.05    | 2.05                   | 2.05    | 2.04    |
| $(dW/dt)_{\text{max}}$ (%/min)  | 11.74           | 6.96    | 13.89   | 10.53                  | 7.16    | 8.94    |
| $S_n$ ( $\times 10^{-7}$ )      | 12.47           | 6.91    | 12.51   | 11.87                  | 7.34    | 7.33    |

**Figure 4. Moisture adsorption test by comparing %RH and EMC of raw, PAT, and PBT pellets in each condition of a) LC, b) RW, c) RS, and d) SCL**

a binding agent (Li *et al.*, 2012). The decreasing of bulk density for TBPs might be because the hydrogen bonding in the inter-particle was weakened after torrefaction, leading to more void and space among the particle within the pellet.

When comparing between TBPs prepared via PAT and PBT methods, the bulk density of PBT torrefied pellets was relatively lower than that of PAT torrefied pellets obtained at the same torrefaction temperature, following the decreasing trend of torrefied mass yield. This could be explained that the torrefied material had increased

fragility and greater resistance to motion before pelletization. So, PAT torrefied pellet had lower void inside their structure, leading to relatively higher bulk density (Ghiasi *et al.*, 2014). Most of the TBP obtained from both PAT and PBT method in this study showed lower bulk density than the standard value of thermally treated biomass pellets at  $\geq 650 \text{ kg/m}^3$  from ISO/TS 17225-8:2016 with reasons explained above. However, this decreased bulk density might be possibly solved by using the higher compaction pressure or higher die temperature during pelletization to increase the

pellet density, which led to increasing bulk density (Rudolfsson *et al.*, 2017). The higher compaction pressure or higher die temperature may cause a reduction in the size of voids and also help to decrease the water adsorption in TBP, which was explained later in an analysis of equilibrium moisture content.

### Equilibrium Moisture Content

The moisture adsorption of raw biomass pellet and TBPs of each biomass type can be determined by using an analysis of equilibrium moisture content (EMC) at different relative humidity (%RH) with time (for 12 days) as shown in Figure 4. In all samples, the EMC gradually increased with increasing the %RH. The moisture uptakes of the TBPs were lower than those of raw biomass pellets under all %RH conditions because the ratio of carbon increased, while the ratio of hydrogen and oxygen decreased to form water via dehydration during the torrefaction as explained later in ultimate analyses, leading to preventing water to get access inside biomass structure (Ghiasi *et al.*, 2014). Moreover, the volatile matter was removed during the torrefaction process to produce TBPs. This reason was also supported by Wang *et al.* (2020) that it caused a hydrophilic H-O bond transformed to others. So, it can confirm the higher hydrophobicity property of the TBPs. Moreover, when comparing the EMC obtained at higher %RH between PAT and PBT torrefied pellets, the PBT torrefied pellets had mostly lower EMC than PAT torrefied pellets, clearly seen in RW and SCL samples as shown in Figures 4(b) and 4(d). This result was similar to the results reported by Yoshida *et al.* (2021). Kaliyan and Morey (2010), Serrano *et al.* (2011), Li *et al.* (2012), and Rudolfsson *et al.* (2017) also reported that the lower moisture adsorption was achieved for the torrefied pellets due to the decomposition of hydroxyl groups and lignin coating on particles. This led to the formation of larger voids, a decrease in bulk density, and then lower moisture adsorption due to the severe decomposition of cell wall in the torrefied particles.

### Proximate Analyses

The proximate analyses of raw biomass pellets and TBPs for each biomass type using TGA, are shown in Table 2. It was found that the fixed carbon (FC) of TBPs increased, while the volatile matter (VM) decreased with increasing torrefaction temperature for all biomass. When comparing PAT and PBT torrefied pellets from woody biomass and agricultural residue, the PBT torrefied pellets for all biomass types had higher FC than the PAT torrefied pellets. This could be explained by the reason that the torrefaction of raw biomass pellets could release

more volatile matter due to its better heat transfer in pellet structure. Moreover, the proximate analysis revealed that raw agricultural residues had the ash content over 10 wt%, while the ash content in raw woody biomass was much lower at only 2-3 wt%. This higher amount of ash content might have a catalytic effect during the torrefaction of agricultural residues. The ash content in TBP from woody biomasses could be still kept at lower than 3% after torrefaction following the standard value of ISO/TS 17225-8:2016 (ash  $\leq 3\%$ ). Whereas, acid pretreatment of agricultural residues before pelletization and torrefaction might be necessary to decrease the ash content in TBP below the standard value. The FC of TBPs for all biomass in this study were highest when torrefied at 300°C. The TBPs at 300°C of RW were increased up to 19.9-26.8 wt%, which was similar to 26.6 wt% obtained from the LC pellet torrefied at 300°C by Setkit *et al.* (2021). In contrast, the FC of TBPs from RS and SCL dramatically increased over 32 wt%, much higher than those of RW. When comparing between PAT and PBT torrefied pellets for all biomass, it was found that the PBT torrefied pellets had higher FC than the PAT torrefied pellets at the same torrefaction temperature. The FC of TBPs had been much improved from that of raw biomass toward the FC of selected coal at 55.7-57.7 wt% as a targeted solid fuel (Ro *et al.*, 2019). An increase in FC of the TBPs definitely led to a reduction of the VM in TBPs for all biomass type, especially lower than 40 wt% for RS and SCL after torrefied at 300°C. The higher VM caused a lower FC and heating value in samples accordingly, which may affect the subsequent vapor combustion (Sukarta *et al.*, 2018).

### Ultimate Analyses

The ultimate analyses of raw biomass pellets and TBPs for each biomass type was also shown in Table 2. The %C of TBPs was much improved with torrefaction temperature. Moreover, the TBPs from agricultural residues of RS and SCL showed much higher %C than woody biomasses of LC and RW at the same torrefaction temperature. The TBPs at 300°C via PAT method for RW, RS, and SCL could be leveled up to 51.1, 57.4, and 59.6 wt%, respectively. Moreover, the PBT torrefied pellets had higher %C than the PAT torrefied pellets in all samples, following an increasing trend of FC as previously discussed. The %C could be further enhanced up to 61.9-65.9 wt% for the PBT 300°C torrefied pellet samples of RS and SCL, respectively. In terms of FC and %C, it could be briefly concluded that PBT torrefied pellet seems to be more suitably used as a solid fuel for both woody biomass and agricultural residue compared to fuel properties of PAT torrefied pellets. This result was

similar with Ghiassi *et al.* (2014). They also reported that the PBT torrefied pellet had better fuel properties than PAT torrefied pellet for Douglas-fir as a raw biomass.

An increase in %C in TBPs led to a decrease in %H and %O, which caused a reduction of the H/C and O/C ratio as plotted in the Van Krevelen diagram shown in Figure 5. The results of raw and TBPs prepared at the temperature range of 260-300°C was also compared with Thai lignite coal (Sonobe *et al.*, 2008). The value of H/C and O/C ratio of TBPs were occurred on the dehydration reaction line. Moreover, the value of H/C and O/C ratio prepared from agricultural residue (RS and SCL) were clearly lower than those prepared from woody biomass for TBPs at 300°C. When comparing TBPs at 300°C in all biomass types, PBT torrefied pellets had also lower H/C and O/C ratio than PAT torrefied pellets. The TBPs in all biomass at 280°C had decreased the value of H/C and O/C ratio lower to 1.72 and 0.87, respectively. And the H/C and O/C ratio had severely decreased to 1.57 and 0.67 after being torrefied at 300°C. Furthermore, the H/C and O/C ratio had the lowest value for the TBPs produced at 300°C for RW, RS, and SCL samples with the value of H/C at 1.20 and 0.47 for O/C. The ratios were very closed to the values of Thai lignite coal which had the H/C and O/C of 1.01 and 0.43, respectively (Sonobe *et al.*, 2008). This indicated that the fuel properties of TBPs at 300°C had similar or even better than Thai lignite coal, especially PBT torrefied pellets obtained from RS and SCL samples.

#### Determination of Higher Heating Value

The higher heating value (HHV) was calculated using the elemental composition with dry-ash-free (daf) basis from CHNO analyzer as shown in Equations (2)-(4). The HHV of TBPs increased significantly when increasing torrefaction temperature as shown in Table 2. The TBPs of LC at 260°C was increased from 18.6 MJ/kg in raw biomass to 20.3-21.4 MJ/kg in case of PAT torrefied pellets at 260-280°C and further increased up to 20.8-23.1 MJ/kg for PBT torrefied pellets. For the TBPs of RW also showed the similar results, which their HHVs were increased up to 20.6 and 23.4 MJ/kg for PAT and PBT pellets torrefied at 300°C, respectively. Moreover, HHVs of the TBPs derived from agricultural residue samples were much more increased than those obtained from woody biomass when increasing torrefaction temperature. TBPs of RS were enhanced from 17.2 MJ/kg in raw sample to 17.7-22.7 MJ/kg and 18.7-24.9 MJ/kg for PAT and PBT torrefied pellets, respectively. Furthermore, TBPs of SCL showed the highest HHVs among the samples in our study, which could

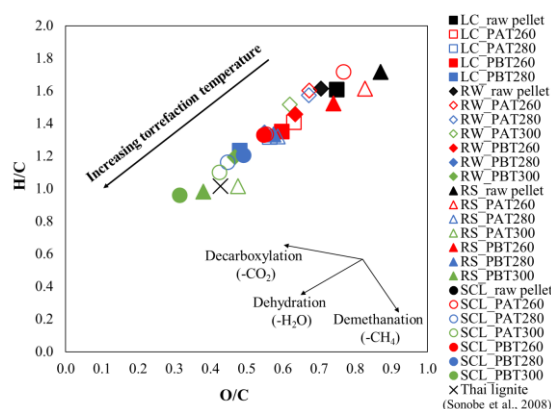
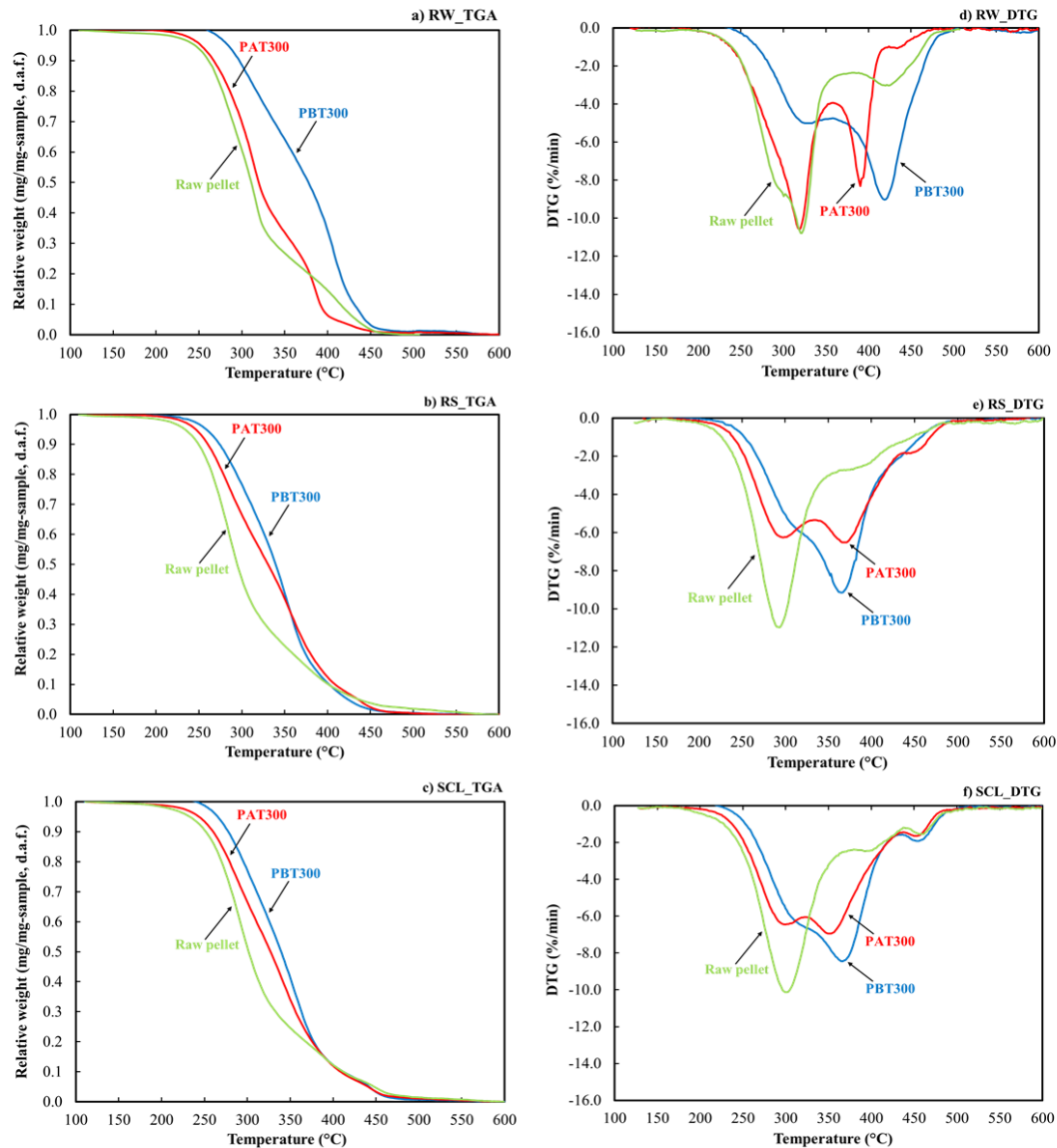


Figure 5. Van Krevelen diagram of raw pellets and TBPs torrefied at 260-300°C of LC, RW, RS, and SCL, compared with Thai lignite

be improved up to 24.1 and 27.0 MJ/kg in cases of PAT and PBT pellets torrefied at 300°C. The HHVs of these TBPs could be leveled up as similar or even better than targeted coal of 24 MJ/kg (GREET Life Cycle Model, 2012). According to above discussed fuel properties of the TBPs, it could be briefly concluded that the SCL-derived TBP prepared via PBT method at 300°C was most suitable for use as solid fuels in subsequent combustion or pyrolysis applications. HHVs of the TBPs in this study had highest around 27 MJ/kg (dry-ash-free, daf, basis). This value was significantly higher than the standard value of  $\geq 21$  MJ/kg (ISO/TS 17225-8:2016). Moreover, the HHVs were also in the range of coal at 25.7 MJ/kg for lignite and 28.2 MJ/kg for bituminous coal reported by Rokni and Levendis (2016).

#### Combustion Characteristic Analysis

Figure 6 shows the thermogravimetric (TG) (Figures 6(a-c)) and derivative thermogravimetric (DTG) (Figures 6(d-f)) curves of combustion characteristics for raw biomass pellets and TBPs at 300°C of RW, RS, and SCL samples prepared via PAT and PBT methods. When comparing the TGA curves as shown in Figures 6(a-c)) for all biomass, it showed the different thermal decomposition characteristics between woody biomass (RW) and agricultural residue (RS and SCL). Woody biomass (RW) showed rather similar combustion behaviors of raw biomass pellet and PAT300 torrefied pellet, while PBT300 torrefied pellet had higher combustion temperature range. However, PBT300 torrefied pellets of RS and SCL samples expressed nearly the same combustion characteristics at slightly higher decomposition temperature compared to PAT300 torrefied pellets and raw biomass pellet, respectively. The DTG



**Figure 6.** TGA (a), (c), and (e) and DTG (b), (d), and (f) combustion curves of raw and TBP at 300°C for RW, RS, and SCL, respectively

curves of these combustion characteristics showed two main peaks as shown in Figures 6(d-f). The first peak was mostly in the range from 190 to 350°C, attributed to VM combustion zone. Moreover, for PAT and PBT torrefied pellets, the peaks were shifted to higher thermal decomposition temperature range, while raw biomass pellet showed faster thermal decomposition rate in all biomass. The second peak was in the range from 330 to 485°C, attributed to char or FC combustion zone. In case of RW sample, the VM combustion zone was obviously observed for raw biomass pellet and PAT torrefied pellet with relatively higher intensity of the first peak. In contrast, for RS and SCL

samples, the first peak was observed for only raw biomass pellet, while the second peak intensity of char combustion was clearly increased for PAT300 and PBT300 torrefied pellets. For the char combustion zone, the PBT torrefied pellets of all biomass indicated steeper and clearer second peak due to their lower VM with higher %C and FC content. These results had a good agreement with the FC and %C data as shown in Table 2. Moreover, the data from DTG curves were used to determine combustion characteristic parameters as expressed in Table 3. They consist of the ignition temperatures ( $T_i$ ), burnout temperature ( $T_b$ ), average rate of weight loss  $((dW/dt)_{\text{mean}})$ , maximum rate

of weight loss ( $(dW/dt)_{\max}$ ), and combustion performance index ( $S_n$ ). Raw biomass pellet had relatively lower initial ignition temperature or  $T_i$  at 194.7-206.9°C than all TBPs (203.3-237.2°C) because raw biomass pellet had higher VM with higher H/C and O/C ratio, leading to an easier and quicker ignition. However, the  $T_b$ ,  $dW/dt_{\text{mean}}$ , and  $dW/dt_{\max}$  were still unclear to explain as a trend in this study due to the effect of torrefaction and the complexity of torrefied biomass structure. The lower the  $T_b$  in TBPs, the less the time to burn and loss unburnt carbon. These parameters were used to calculate the combustion performance index, abbreviated as  $S_n$ , using Equation (5). The  $S_n$  values of raw biomass pellets were mostly highest in all samples (approximately  $11 \times 10^{-7}$  to  $16 \times 10^{-7}$ ). The  $S_n$  of TBPs decreased to about  $7.3 \times 10^{-7}$  to  $7.8 \times 10^{-7}$  for PBT300 samples of RW and SCL, while the PAT300 torrefied pellet showed slightly higher  $S_n$  for RW sample at  $11.4 \times 10^{-7}$ . In contrast, the TBP samples of RS showed the opposite trend of the  $S_n$  for PAT and PBT torrefied pellets due to its lower burnout temperature of the PBT300 sample. Finally, the combustion performance index values determined in our study were rather in a good agreement with other torrefied biomasses and coals reported in previous literatures, which were lower than those of raw biomass. For example, Setkit *et al.* (2021) obtained Leucaena torrefied pellets at 300°C with the  $S_n$  value at  $18.6 \times 10^{-7}$ , which was slightly lower than that of raw pellet at  $19.3 \times 10^{-7}$ . Furthermore, Sezer *et al.* (2021) also determined  $S_n$  of Imbat coal under air combustion with a heating rate of 10°C/min as  $0.29 \times 10^{-7}$ , much lower than that of raw almond shell, as  $1.37 \times 10^{-7}$ .

## Conclusions

Torrefied biomass pellets (TBP) were produced at various torrefaction temperature of 260-300°C for 5 min using two different sequence processes of pelletization after torrefaction (PAT) and pelletization before torrefaction (PBT). Woody biomasses (Leucaena (LC), rubberwood (RW)) and agricultural residues (rice straw (RS), and sugarcane leaves (SCL)) were used as raw materials in this study. It was found that lower mass yield and bulk density could be obtained for PBT torrefied pellets in all biomass, but they had lower moisture adsorption than raw biomass pellet and PAT samples. Moreover, the PBT torrefied pellets showed significantly improved fuel properties with higher amounts of fixed carbon (FC), %C, and higher heating value (HHV). Particularly for the PBT300 of SCL, the HHV could be enhanced up to 27.0 MJ/kg from 18.3 MJ/kg in raw biomass, which

was even better than those of some bituminous coals and lignite used as commercial solid fuels reported in literatures. These improved properties led to a reduction in the H/C and O/C ratio, affecting their lower combustion performance index. This was also in an agreement with the combustion property of coal and lignite, which also had lower combustion performance index than raw biomass. According to ISO/TS 17225-8:2016, all TBPs obtained by PBT method at 300°C in this study showed higher HHVs (dry-ash-free basis) than the standard value of  $\geq 21$  MJ/kg. In terms of ash content, woody biomass showed its low content below 3%, while the agricultural biomass requires acid pretreatment to further reduce the ash content in the final TBPs. Moreover, bulk density of all TBPs should be improved by increasing compaction pressure or die temperature during pelletization to meet the standard value of  $\geq 650$  kg/m<sup>3</sup>. Consequently, it can be summarized that the TBPs prepared via PBT methods using woody biomass or agricultural residues as a raw material could be considered as one of potential candidates to produce high quality solid fuels to substitute usage of coal or lignite in the future.

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