Influence of torrefaction on yields and characteristics of densified solid biofuel

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Abstract. Torrefaction is a mild thermal pretreatment for enhancing the combustion property of biomass to be properly applied to biomass or co-firing power plants. The present research investigated mass yields, fuel characteristics and hydrophobicity of rubber wood pellet torrefied at different temperatures and residence times. Proximate and ultimate analyses, the enhancement factor of higher heating value, energy yield, lignocellulosic components and Fourier transform infrared spectroscopy (FT-IR) of torrefied samples were performed to evaluate the influence of the process parameters and compared to those of the untreated ones. The non-condensable and condensable products were analyzed by GC and GC-MS. The results showed that the torrefaction severity index (TSI) correlated sufficiently with the properties of torrefied biomass and was more sensitive to torrefaction temperature than residence time. The increase in the TSI value resulted in an increase in heating value but a decrease in energy yield. The energy density increased by 14% at 260 °C and reached up to 35% at torrefaction temperature of 300 °C. FT-IR analysis revealed that the contents of hydroxyl and carbonyl groups appeared in torrefied wood pellets were decreased and increased, respectively, due to the torrefaction process. Furthermore, the hydrophobicity of torrefied pellets can be significantly improved as torrefaction temperature increases. The non-condensable gaseous products were mostly composed of CO₂, CO, and traces of CH₄. While the organic compounds found in condensable liquid products were acids, ketones, furans and phenols.

1. Introduction
Wood pellets are regarded as one of alternative energy sources which are already utilized in industrial boilers and power plants in order to cope with the challenge of greenhouse gas emissions [1]. The global wood pellet market is predicted to increase up to 54 million tonnes in 2024. Consumption is high in UK, Denmark, Benelux countries, Japan, and South Korea. Thailand has abundant biomass resources, which is the extraordinary advantages for Thailand to produce and use wood pellets as a kind of green energy. The main raw materials for making wood pellets in Thailand are waste wood from para-rubber, which is abundant in the south of Thailand [2]. However, energy use of wood pellets is often difficult due to several disadvantages such as low calorific value, high oxygen content and hygroscopic behavior.
Previous research reported that energy density of commercial wood pellets varies between 4,060 and 4,777 kcal/kg which is substantially lower that of coals (4,776-7,165 kcal/kg). Wood pellets are sensitive to moisture uptake from the surrounding environment resulting in losing their pellet structure during transport and storage [1]. To overcome these limitations and make wood pellets suitable for replacing or co-firing with coal in heat and power generation applications, a pre-treatment is often necessary [3]. Torrefaction is a thermal pretreatment process which helps alter the physical properties and chemical composition of biomass. Torrefaction is defined as a mild pyrolysis occurring at a temperature range of 200–300 °C in the absence of oxygen. This process enhances the physiochemical properties of the biomass, given that it has a high energy density, lower O/C ratio solid, grindable, hydrophobic, and compactable, making it perform better for energy applications [3]. Chen et al. [4] have recently reported that torrefaction process effectively improved the quality of biomass in terms of higher calorific value, lower oxygen content, lower moisture content and higher hydrophobicity. Shang et al. [5] reported that the calorific value of torrefied product obtained from torrefaction of Scots pine increased from 4,394 kcal/kg to 5,803 kcal/kg. Aziz et al. [6] have reported that torrefaction was affected by many parameters including the chemical composition and operation conditions. Barbanerea et al. [7] reported that a significant amount of hemicellulose decomposed when torrefaction temperature increased from 230 °C to 290 °C. An increase in the torrefaction temperature resulted in a decreasing yield of solid product but increasing yield of volatile products, including liquid and gaseous products. The loss of biomass can reach about 30% of its mass through the conversion into volatile products. At the same time, about 10% of the total energy contained in the raw material reduced after torrefaction. The decrease in mass and energy yields was mainly due to the breakage of a large number of hydroxyl functional groups, obtaining a torrefied biomass with less moisture absorption capacity which became less sensitive to biological degradation and could be stored for long period.

Although torrefaction of biomass have been extensively studied, information of the relationship among torrefaction degree and torrefaction performance still remains unclear. To provide study on the correlation of biomass properties and torrefaction severity, in the current study, rubber wood pellet was adopted as feedstock. The torrefaction was conducted under different temperatures (200, 230, 260 and 300 °C) and residence times (10, 30, 60 and 90 min). A detailed characterization of torrefied products was conducted by proximate and ultimate analyses, lignocellulosic components and FT-IR spectroscopy. The relationships between quantities of torrefied biomass and TSI were determined. The hygroscopicity of raw and torrefied wood pellets was evaluated through moisture uptake tests. Gaseous and liquid products were also analyzed by GC and GC-MS, respectively. Finally, the mass and energy balances at different temperatures were estimated and discussed in detail.

2. Materials and Methods

2.1. Torrefaction experiment

Rubber wood pellets obtained from a wood processing factory in the Eastern region of Thailand with a characteristic length of 30-40 mm and diameter of 7-8 mm were used as raw materials for the torrefaction process. For each run, 50 g of wood pellets were loaded into the reactor, which then placed at the center of a vertical tube furnace. Before carrying out the experiment, the reactor was purged sufficiently with N2 and then heated up to the desired conditions with a heating rate of 10 °C/min. N2 was continuously supplied throughout the torrefaction experiment with a flow rate of 500 mL/min to remove volatile out of the reactor. The volatile gases were immediately passed through a condenser coupled with a recirculating bath to collect the condensable product. The non-condensable faction was sampled using gas sampling bag. After the torrefaction process, the solid products were cooled and weighted to examine the solid yield. The mass yield of solid product was calculated using equation (1). The energy yield of the torrefaction was obtained using equation (2). According to literature, energy density, torrefaction severity index and enhancement factor were calculated by equation (3), (4) and (5), respectively [8].

\[ \text{Mass yield (MY)} = \frac{m_{\text{torr}}}{m_{\text{raw}}} \]  \hspace{1cm} (1)
Energy yield (EY) = mass yield \times \frac{HHV_{torr}}{HHV_{raw}} \quad (2)

Energy density = \frac{Energy yield}{Mass yield} \quad (3)

Torrefaction severity index (TSI) = \frac{100 - MY}{100 - MY_{100^\circ C,90 \text{min}}} \quad (4)

Enhancement factor (ef) = \frac{HHV_{torrified}}{HHV_{raw \text{ biomass}}} \quad (5)

2.2. Sample analysis

The contents of moisture, volatiles, ash and fixed carbon of raw and torrefied samples were determined using a thermogravimetric analyzer, TGA701. A LECO TruSpec CHN elemental analyzer was employed to determine the C, H, N and S of raw and torrefied samples. The O content was obtained from the difference. The heating value of the samples was performed in a bomb calorimeter (Parr 6300) using the standard test method ASTM D5865. Infrared spectra were recorded using the attenuated total reflectance Fourier transform infrared spectroscopy (Prestige-21 Shimadzu FT-IR spectrometer). Hemicellulose, cellulose and lignin of the samples were obtained using the Van Soest method. The biomass was extracted sequentially with neutral detergent (removal of extracts), acid detergent reagent (removal of hemicellulose), and 72% H_2SO_4 (removal of cellulose). The hydrophobic property of the samples was expressed in terms of water uptake. The water uptake was measured by placing the samples in a controlled environment chamber (relative humidity 80% at 30 °C) for 48 h. The organic composition of the condensable product was analyzed by GC-MS (Agilent 7820A/5975C). The composition and content of non-condensable gases were analyzed by GC (Agilent 7890A).

3. Results and Discussion

3.1. Torrefied biomass

Table 1 shows the results of proximate, ultimate, and heating value analyses of untreated and torrefied wood pellets. The moisture content of untreated wood pellets was 6.53%, but because torrefaction is a deep drying process, the moisture content was reduced. This reduction minimizes the microbial activity, hence avoiding biomass degradation and moisture absorption during storage and transportation [3]. An inverse behaviour was observed for ash content. The ash content of wood pellets after torrefaction increased from an initial value of 2.00% to 3.55%, due to loss of organic matter during torrefaction process. Ash content gradually increased with increasing severity of torrefaction. However, the ash content of torrefied wood pellets remained in the range for applications in gasification and combustion systems (<10%) [4]. The volatile content of torrefied wood pellets was lower than that of untreated wood pellets (79.38 wt%) because of the thermal degradation of hemicellulose and depolymerization of cellulose. The volatile formed during thermal degradation of wood pellets comprised non-condensable and condensable products, and their contents were affected by temperature and duration time [9]. As can be seen in Table 1, temperature showed more significant parameter on volatile content rather than residence time. The volatile content decreased from 78.41% to 50.24%, when the torrefaction temperature increased from 200 °C to 300 °C. At the same time the fixed carbon showed a significant increase from 18.61% for untreated wood pellets to 46.20% for torrefied wood pellets at 300 °C. The results were similar to the previous results studied by Manouchehrinejad [1] where the fixed carbon increased, while volatile decreased with an increase in severity of torrefaction. The removal of volatile from wood pellets resulted in the improvement of the fuel ratio (fixed carbon/volatile matter) whose value increased from 0.23 for untreated wood pellets to 0.92 for torrefied wood pellets at 300 °C.

The ultimate analysis data in Table 1 shows a significant effect on carbon and oxygen contents when wood pellets were torrefied. The carbon content of torrefied wood pellets was much higher than that of the untreated wood pellets, while the oxygen content of torrefied wood pellets was reduced after torrefaction. Carbon content increased from 47.78% to 65.75%, when the torrefaction severity increased. This enhancement of carbon in torrefied wood pellets was directly associated with the elimination of oxygen and hydrogen in the structure of biomass, which agreed with the results of other studies for
woody biomass [4, 9]. Due to the change of elemental carbon, oxygen and hydrogen, the higher heating value (HHV) of torrefied wood pellets also increased dramatically when torrefaction severity increased. At temperature of 300 °C, the HHV of torrefied wood pellets increased from the original material by 38%. According to the results from Table 1, the variation in the physical and chemical properties of torrefied products was sensitive to the torrefaction temperature, whereas the influence of residence time was relatively slight.

Typically, the TSI value is used to evaluate torrefaction performance in terms of weight loss at the most severe conditions (300 °C, 90 min). The profile of TSI versus temperature for residence times of 10, 30, 60 and 90 min is plotted in Figure 1(a). All profiles are characterized by linear correlations in that the coefficients of determination (R²) are above 0.88. The TSI value was between 0.2 and 1.0. Meanwhile, it can be seen that the TSI was more sensitive to torrefaction temperature than residence time, implying that torrefaction temperature showed a more significant influence on the solid yield compared to residence time. Figure 1(b) shows the profiles of enhancement factor and energy yield versus TSI. The enhancement factor and energy yield had a linear relationship with TSI that was consistent with the results reported by Silva et al. where the HHV increased with decreasing solid yield [8]. The increase in the TSI value resulted in an increase in enhancement factor but a decrease in energy yield. The coefficients of determination (R²) for the enhancement factor and energy yield were greater than 0.97. Hence, the TSI value can be used as a feasible parameter to indicate torrefaction performance.

Water uptake behaviors can represent as the stability of biomass upon humid conditions. Figure 2 presents the water uptake behaviors of torrefied and non-torrefied wood pellets. It can be seen that the amount of moisture uptake decreased with increasing torrefaction temperature and time. The decrease in water uptake capacity of torrefied wood pellets indicated the increase in the hydrophobicity of wood pellets. This behaviors may be due to the removal of hydroxyl groups from the wood pellets that hindered the formation of hydrogen bonds [3]. Consequently, the torrefied wood pellets were able to resist moisture from being absorbed. Castro et al. [10] also observed that torrefaction increased the wood chips resistance to degradation of moisture and fungi. The mass loss was only 2% after 12 weeks of exposure.

Table 1. Physiochemical characterization of untreated and torrefied wood pellets

| Samples                  | Proximate Analysis (wt%) | Ultimate Analysis (wt%) | HHVb (kcal/kg) |
|--------------------------|--------------------------|-------------------------|----------------|
|                          | MCa                      | VMb                     | FCb            | Ashb | H   | C   | N   | O   |              |
| Untreated wood pellets   |                          |                         |                |      |     |     |     |     |              |
| 200 °C_10 min            | 2.08±0.00                | 78.41±0.11              | 19.60±0.06     | 1.99±0.04 | 5.71±0.15 | 47.78±0.02 | 0.04±0.01 | 44.47±0.15 | 4621±6.09 |
| 200 °C_30 min            | 3.32±0.00                | 77.44±0.38              | 20.35±0.16     | 2.21±0.21 | 5.98±0.15 | 51.27±0.87 | 0.09±0.11 | 40.45±0.69 | 4705±10.0 |
| 200 °C_60 min            | 2.87±0.45                | 77.60±0.13              | 20.45±0.13     | 1.96±0.01 | 5.94±0.08 | 50.29±1.14 | 0.00±0.00 | 41.82±1.23 | 4737±11.5 |
| 200 °C_90 min            | 2.69±0.66                | 77.67±0.08              | 20.37±0.06     | 1.97±0.03 | 5.86±0.13 | 49.5±62.35 | 0.03±0.04 | 42.5±2.47 | 4780±12.1 |
| 230 °C_10 min            | 1.88±0.03                | 76.52±0.52              | 21.38±0.43     | 2.10±0.06 | 5.75±0.45 | 49.20±0.51 | 0.05±0.00 | 42.9±6.1 | 4808±6.41 |
| 230 °C_30 min            | 2.80±1.31                | 75.91±0.38              | 21.90±0.23     | 2.19±0.14 | 5.82±0.05 | 52.08±6.2 | 0.11±0.10 | 39.80±0.82 | 4857±6.40 |
| 230 °C_60 min            | 2.96±0.07                | 75.10±0.58              | 22.76±0.48     | 2.13±0.10 | 5.88±0.06 | 52.66±0.03 | 0.00±0.00 | 39.33±0.07 | 4906±1.70 |
| 230 °C_90 min            | 3.09±0.26                | 74.98±0.16              | 22.88±0.05     | 2.14±0.11 | 5.82±0.07 | 51.73±1.40 | 0.00±0.00 | 40.31±1.59 | 4912±0.24 |
| 260 °C_10 min            | 2.39±0.07                | 72.11±0.38              | 25.52±0.43     | 2.37±0.06 | 5.60±0.02 | 51.98±0.41 | 0.09±0.02 | 39.97±0.47 | 5111±8.60 |
| 260 °C_30 min            | 3.94±0.06                | 71.59±0.07              | 25.86±0.33     | 2.55±0.26 | 5.75±0.08 | 54.7±0.97 | 0.15±0.08 | 36.86±0.80 | 5104±1.50 |
| 260 °C_60 min            | 2.59±0.30                | 71.70±0.90              | 26.05±0.84     | 2.25±0.07 | 5.60±0.08 | 52.99±0.09 | 0.09±0.03 | 39.07±0.12 | 5138±2.60 |
| 260 °C_90 min            | 3.38±0.28                | 71.02±0.17              | 26.63±0.24     | 2.35±0.07 | 5.68±0.05 | 55.4±1.44 | 0.03±0.01 | 36.46±1.56 | 5151±3.50 |
| 300 °C_10 min            | 2.65±0.01                | 59.82±0.05              | 37.20±0.05     | 2.98±0.30 | 5.30±0.14 | 59.46±0.66 | 0.17±0.06 | 32.10±0.89 | 5754±0.35 |
| 300 °C_30 min            | 2.57±0.10                | 50.48±0.93              | 45.66±0.30     | 3.87±0.55 | 4.96±0.15 | 65.75±0.15 | 0.34±0.04 | 26.62±0.59 | 6251±3.02 |
| 300 °C_60 min            | 3.04±0.23                | 51.52±0.70              | 45.03±0.67     | 3.45±0.03 | 5.00±0.19 | 63.71±0.95 | 0.19±0.03 | 27.64±0.11 | 6195±1.19 |
| 300 °C_90 min            | 3.59±0.59                | 50.24±0.23              | 46.20±0.74     | 3.55±0.49 | 4.96±0.47 | 64.01±0.32 | 0.22±0.15 | 27.26±0.15 | 6247±4.04 |

MC a is moisture content, VM is volatile matter, FC is fixed carbon, b Wet basis, b Dry basis

The results of the chemical component analysis of wood pellets after torrefaction at different temperatures with a residence time of 30 min are presented in Table 2. The major components in biomass are hemicellulose, cellulose and lignin. With an increase in the torrefaction temperature, the
hemicellulose content decreased. Hemicellulose decomposes when heated to 200–280 °C with the removal of hydroxyl and carboxyl groups, producing CO₂, CO, H₂O, and oxygen-containing organic compounds [1]. In contrast, cellulose content in biomass increased from 47.37% at 200 °C to 59.49% at 260 °C, and then decreased to 50.45% at 300 °C. These results were assigned to the degradation of cellulose which took place at temperature in the range of 240 – 350 °C [3]. Lignin is the most thermally stable component, and this contributed to an increase in lignin content as torrefaction temperature increased.

The change of structure composition of wood pellets during torrefaction was evaluated by FTIR analysis and the FTIR spectra is presented in Figure 3. The peak at 3370 cm⁻¹ was assigned to the O-H stretching vibration of hydroxyl group. The peaks at 2328 and 2373 cm⁻¹ were mainly due to the stretching vibration of C≡C in the alkyne group, which is found in lignin. The peak at 1620 cm⁻¹ corresponded to C=C stretching vibration of alkene. The peak centered at 1029 cm⁻¹ was assigned to C-O-C stretching vibration, which was found in cellulose and hemicellulose. Due to torrefaction treatment, there were some structural changes in the torrefied wood pellets compared to untreated wood pellets. The intensity of peaks at 3370 and 1029 cm⁻¹ decreased with increasing temperature, indicating that torrefaction promoted the oxygen removal, which would be responsible for the enhancement of hydrophobicity of the torrefied wood pellets. Similar results also were obtained in the torrefaction experiment of biomass conducted by Silva et al. [8].

3.2. Non-condensable gaseous products

The composition of gaseous products formed during torrefaction at different temperatures with a residence time of 30 min is shown in Table 2. The non-condensable gaseous products obtained from torrefaction of wood pellets mainly consisted of CO₂ and CO, together with a trace of CH₄. Manouchehrinejad et al. [1] reported that the release of CO₂ during torrefaction was assigned to the decarboxylation reaction of unstable carboxyl group in hemicellulose, while the formation of CO was mainly due to the secondary reactions of CO₂ and steam with porous char and the decarbonylation reaction. Due to the secondary reaction, the higher amount of CO was formed when increasing torrefaction severity, while CO₂ concentration decreased. A small amount of CH₄ was detected at temperature above 230 °C, due to the increase of energy supplied that sufficient to promote the fragmentation of the complex structures.

![Figure 1](image-url) (a) Relationship between TSI and torrefaction conditions and (b) Profiles and regression lines
Table 2. Component analysis of torrefied wood pellets and composition of gaseous products obtained from torrefaction at different temperatures with a residence time of 30 min.

| Samples          | Component analysis (wt%) | Composition of gaseous products (vol%) |
|------------------|--------------------------|----------------------------------------|
|                  | Hemicellulose | Cellulose | Lignin | CO₂  | CO   | CH₄   |
| 200 °C_30 min    | 7.58±0.01   | 47.37±0.03 | 25.36±0.20 | 62.58±2.10 | 37.41±2.10 | n.d.⁸ |
| 230 °C_30 min    | 9.29±0.01   | 46.80±0.01 | 30.56±0.01 | 59.82±4.74 | 40.17±4.74 | n.d. |
| 260 °C_30 min    | 1.18±0.20   | 59.49±0.03 | 27.09±0.03 | 49.03±0.81 | 50.72±0.83 | 0.19±0.02 |
| 300 °C_30 min    | 4.65±0.10   | 50.45±0.02 | 30.69±0.01 | 53.60±0.04 | 44.26±0.03 | 2.12±0.01 |

* n.d., not determined

Figure 2. The moisture uptake rate of untreated and torrefied wood pellets.

Figure 3. FTIR spectrum of untreated and torrefied wood pellets.

3.3. Condensable liquid products

The condensable liquid product obtained after torrefaction of rubber wood pellet at different temperatures with a residence time of 30 min consisted of aqueous phase and oil phase. The main compounds detected in an oil phase were acids, ketones, furans and phenols, as presented in Figure 4. Acetic acid was mainly formed from dissociation of O-acetyl groups in hemicellulose structure of biomass. For furan groups, furfural was the main compound identified. Furfural compound can be generated by dehydration of five-carbon sugars such as xylose and arabinose in hemicellulose [8]. When torrefaction temperature increased, phenol became the main components in oil product. The formation of phenolic derivatives was mainly due to the depolymerization of hemicellulose and lignin. As many organic components were detected in torrefaction liquid products, it is has a potential to be used as chemicals. Particularly, furan has been considered as one of an important and renewable platform chemicals in the bio-based renaissance. It can be used as the starting materials for new products as well as for the replacement of oil-derived chemicals [11].
Figure 4. Composition of oil products

3.4. Mass and energy balance

Figure 5 shows mass and energy balance for torrefaction conducted between 200 and 300 °C with a residence time of 30 min. The torrefied product was recovered in 51 to 90% mass yield and it retained 69 to 95% of the original energy content. After torrefaction at 260 °C, 1 mass unit (M) and 1 energy unit (E) of wood pellets were converted into 0.78 M and 0.89 E of solid, 0.15 M and 0.10 E of liquid, and 0.07 M and 0.009 E of gas. Approximately 78% mass and 89% energy retained in torrefied biomass, and approximately 22% mass and 11% energy shifted to liquid and gaseous products. The energy density increased by 14% at 260 °C and reached up to 35% at torrefaction temperature of 300 °C; however, only 51% mass retained in torrefied biomass. Although torrefaction under severe conditions produced torrefied product with high HHV, a large amount of energy was loss due to the loss of mass during torrefaction, explaining decrease of energy yield. Hence, the torrefaction temperature of 260 °C was recommended to upgrade wood pellets for the production of solid fuel. As compared to the original biomass, torrefied wood pellets could be a good feedstock for replacing coal in heat and power generations. However, as assuming a price of coal equal to USD 70/tonne at 25 GJ/tonne (USD 2.80/GJ), high-grade solid biofuels cannot easily compete on a price basis even though it might have a low production cost or be located nearby [12]. Using torrefied biomass as a supplementary fuel in coal-fired power plants carries a great deal of promise as a potential bridge technology since it enable higher co-firing rates than conventional pellets and shows a greater potential to achieve negative greenhouse gas emissions. In addition, the use of torrefied biomass in co-firing application would generate an increase in demand for sustainable biomass, adding value to wastes and residues, creating new market opportunities, and supporting rural economies.

4. Conclusion

The quantities of torrefied biomass can be predicted via the torrefaction severity index. A higher torrefaction severity index could upgrade wood pellets with better heating value, but the value of energy yield decreased. The increase in torrefaction severity also improved hydrophobicity of wood pellets. FT-IR analysis showed that torrefaction promoted the oxygen removal, which would be responsible for the enhancement of hydrophobicity of the torrefied wood pellets. The non-condensable gaseous products obtained from torrefaction of wood pellets mainly consisted of CO₂ and CO, together with a trace of CH₄. The analysis of liquid product suggested that it composed of acids, ketones, furans and phenols. The distribution of products and their characteristics were sensitive to the torrefaction temperature, while the effect of residence time was relatively slight. The HHV of torrefied wood pellets was found to increase from the original raw material by about 38%; however only 51% mass retained in torrefied biomass. Accordingly, the torrefaction temperature of 260 °C was recommended to upgrade wood pellets for the production of solid fuel. Findings of the currents studies implies that torrefaction can be promising way to produce biomass with a better fuel property compared to that of untreated biomass.
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