Production of solid biofuel via hydrothermal carbonization of wood shavings: influence of biomass-to-water ratio and carbonization temperature on the fuel characteristics of dried hydrochar

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Abstract. Hydrothermal carbonization (HTC) is recognised as a low temperature and efficient method for the conversion of biomass to solid biofuel. In this study, the effect of process temperature and biomass-to-water ratio (B/W) on the fuel properties of hydrochar produced from wood shavings was investigated. HTC was conducted in an autoclave using reaction temperature of 230 °C and 260 °C for 20 minutes with B/W ratio of 0.11 to 0.43. The produced hydrochars were characterised by the mass yield (MY), higher heating value (HHV), proximate and ultimate properties. The results showed that the properties of the hydrochars improved with increasing process temperature and B/W ratio. The higher heating value (HHV) increased to 26.74 MJ/kg as the severity of the reaction was increased to the process temperature of 260 °C. Also, the atomic H/C and O/C ratios of hydrochars carbonised at 230 °C and 260 °C were closed to the regions of a peat and lignite on the plotted van Krevelen diagram. Hence, the produced hydrochar has a promising potential as a sustainable solid biofuel for energy application.

Keywords: Thermochemical Conversion, Biomass/Water Ratio, Wood Shavings, Hydrothermal Carbonization, Hydrochar

1 Introduction

The use of non-renewable fuel as source of energy has raised much concerns globally because of its bad effect on the immediate environment [1], [2]. These concerns include climate change which is mostly caused by greenhouse gas emission from the utilization of fossil fuel [2]. Hence, the use of renewable energy source such as biomass has become necessary and offers attractive alternative. However, the direct use of raw biomass as solid biofuel is faced with some challenges such as low energy density [3]-[6]. Therefore, it is imperative to convert biomass to a more energy dense solid by pre-treatment. One of the pre-treatment alternatives is the hydrothermal carbonization (HTC) [7], [8].

HTC is a thermochemical conversion technique for the conversion of biomass to a more energy improved solid biofuel [7]. HTC offers some attractive advantages over other thermochemical conversion techniques for biomass such as the use of moderate temperature (180 to 260 °C) [7], [8]. The conversion process uses subcritical water and autogenous pressures of 2 to 10 MPa in an autoclave reactor [7]-[10]. At these process conditions, the subcritical fluid serves as a solvent and a catalyst leading to complex organic reactions such as hydrolysis, decarboxylation, dehydration, aromatization, and re-condensation [11], [12]. These complex reactions lead to the formation of a solid biofuel usually referred to hydrochar.

To produce hydrochar, the raw biomass needs to be mixed with water in ratio before carbonization and usually referred to as biomass-to-water ratio (B/W). The effect of carbonization temperature as a process variable has been extensively studied [7], [8] compared to the B/W ratio. B/W ratio is a very key variable in the HTC processes because it affects the amount of water required [13]. The enormous amount of water requirement in HTC has been reported as one of main challenges for its large-scale commercialization [7], [8]. Increasing B/W implies decreasing the quantity of water needed to produce hydrochar. Because the decomposition of biomass in HTC is governed by water [11], hence the effect of increasing B/W becomes imperative to be studied. To the authors' knowledge, limited studies are available on the influence of B/W ratio on the fuel

properties of hydrochar. Some of the available literature on the effect of B/W ratio in HTC revealed that increasing the ratio of B/W improves the mass yield (MY) and the higher heating value (HHV) of hydrochar [14]-[17]. For example, Volpe and Fiori [17] investigated the effect of process temperature at increasing B/W ratio to 0.25 (equivalent to 80 wt.% water content in wet basis) using olive waste and found that the mass yield and the higher heating value (HHV) of the produced hydrochar improved. In this study, the influence of carbonisation severity and B/W ratio (0.1 – 0.43) on the properties of hydochar produced from wood shavings was investigated.

2 Materials and methods

2.1 Materials

Wood shavings was collected from the wood engineering laboratory of Hokkaido University, Japan, and was oven-dried for 24 hours at 105 $^{\circ}$ C to reduce the moisture content in order to ease size reduction and to accurately determine the B/W ratio.

2.2 Feedstock preparation and carbonisation procedure

Carbonization was done using a 70 mL batch reactor (TVS-N2, Taiatsu Techno, Tokyo, Japan) shown in Fig. 1. It has a pressure and temperature limits of 8 MPa and 300 °C.



Fig. 1. Schematic of the carbonization reactor used in this study.

For the preparation of raw feedstock, 5 to 15 ± 0.01 g of the sample and 35 to 45 ± 0.01 g of distilled water was mixed to arrive at the needed ratio of B/W which was calculated using equation (1).

(1)

Where, is the mass of the feedstock (g) and m_w is the mass of distilled water (g)

The prepared samples based on different B/W is shown in Fig. 2.



Fig. 2. The prepared feedstock at different B/W ratios.

The reactor was turned off at the predetermined time for reaction and was inserted in cold water to aid fast cooling. The reactor's pressure relief tap was turned-on to allow the released gases to escape through a fume cupboard. The carbonised wood shavings was removed from the reactor and the weight taken. Then, the weighed hydrochar was dried for 24 h in an oven at a temperature of 105 °C. The dried hydrochar sample was grounded, sieved, and stored for further properties determination. The experiment was again repeated twice and average values recorded.

2.3 Properties and analytical determination

The MY after carbonization, energy densification ratio (EDR) and the yield in energy (EY) were evaluated through equations (2) to (4) [10]. The heating value was determined by combusting 0.5 g of the sample in a calorimeter (OSK 200, Ogawa Sampling, Saitama, Japan) with oxygen at a pressure of 0.3 MPa [18].

(2) (3)

(4)

Where (g) is the measured mass of the dried hydrochar, (g) is mass of the dried wood shavings, and (MJ/kg) and (MJ/kg) are the heating values of the carbonised solid fuel and wood shavings.

The percentage of the ash in the wood shavings and the dried hydrochar were determined by burning 1 g at 600 °C for 3 h in a muffle furnace (ADVANTEC, FUL220FA, Japan). ASTM standard procedure (E872) was used to determine the volatile matter (VM), by heating the samples at 950 °C for 7 minutes in a furnace. The fixed carbon (FC [%] = 100 - VM [%] – ash [%]) was calculated by difference while the fuel ratio (FR) was calculated by taking the ratio of FC to VM (FC/VM).

Elemental analyser (Exeter Analytical, North Chelmsford, MA, USA) was used to determine the percentage contents of carbon, hydrogen and nitrogen (C, H and N) while the percentage of oxygen was evaluated by difference (O [%] = 100 - C [%] – H [%] – N [%] – ash [%]).

3 Results and discussion

3.1 The mass yield of the hydrochars

Fig. 3 shows the MY of hydrochar produced at the two carbonization temperatures and B/W ratios while Fig. 4 shows the dried sample of the produced hydrochar at 260 $^{\circ}$ C. MY in a thermochemical conversion process shows severity of the reaction, hence, higher carbonization temperature increases biomass degradation. The degradation is mostly accompanied by the elimination of radical groups such as hydroxyl, carbonyl and carboxyl, thereby producing H₂O, CO and CO₂ [19], [20] hence, the MY decreased with increasing carbonization

temperature. The MY also increased as the ratio of B/W was increased. The reason for the observed increase could be due to the physical deposition of degraded substances which were dissolved in the process water. These degraded substances are likely to be higher at higher B/W ratio. This may be have been caused by the formation of other substances in the process liquid as a result of polymerization and the precipitation of some insoluble solids on the hydrochar of which the chances are higher at higher ratios of B/W, hence, increasing the MY [7], [13], [17], [21], [22].



Fig. 3. The mass yield of the produced hydrochar.



Fig. 4. Samples of the dried dried hydrochar at the different B/W ratios.

3.2 The HHV, EDR and EY of the produced hydrochars

The HHV of the produced solid biofuel increased as the process temperature was increased (Fig. 5). The carbonization reactions such as hydrolysis, decarboxylation, dehydration, aromatization and re-condensation increase at higher temperatures, this contributes to the increasing HHV of the produced hydrochars [11], [16], [23]. The observed HHV showed a slight increase at higher B/W ratio, this finding is in line with the observations from other studies [17], [25]. Also, given the fact that polymerization of broken down compounds on the surface of carbonized solid increases at higher B/W ratios. These broken down substances in the liquid phase have been reported as one of the reasons for the improving HHV of hydrochar. One of such result was reported by Volpe and Fiori [17] the study revealed that a higher B/W ratio during the carbonization of waste from olive increased secondary char formation and resulted in a higher percentage of the carbon content, of hydrochar. Furthermore, the reuse of the process water which contains dissolved organic acids from HTC, improves hydrochar fuel properties through catalysis of decarboxylation and dehydration reactions [26]. Therefore, the increased HHV could be due to the increased acid concentration in the liquid phase since it is likely that the acidity would be higher at higher ratio of B/W where the content of water is less. The EDR and EY are shown in Table 1. The EDR increased by 42 % at 260 °C but remained relatively unchanged despite the increasing the B/W ratio. The EY improved by increasing the B/W ratio for both the HTC carbonisation

severities of 230 °C and 260 °C. The increased EY could be due to the increasing MY (because of increasing the B/W ratio).



Fig. 5. The HHV of the hydrochars.

3.3 The proximate and ultimate characteristics of the produced hydrochars

The proximate and ultimate compositions of the wood shavings and the carbonized wood shavings are given in Table 1. The result showed that, the VM reduced while the FC increased with increase in the carbonization temperature but slightly improved with increase in the ratio B/W. The observed decrease in the VM could be due to its conversion to liquid or gaseous products during the carbonization process [27], [28] increasing the FC. The FR is used for ranking the quality of a fuel, [29] thus slightly increased by the increased process temperature and the ratio B/W. Indeed, the FR of the hydrochar at 260 °C for the different B/W ratios was more than 0.6 documented for lignite [30], [31]. The ash content remained relatively unchanged by the influence of the carbonization temperature and B/W ratio and could be due to the low ash of the feedstock.

The analysis of the ultimate characteristics indicated that the percentage of the carbon increased by the increase in the process temperature but was not significantly affected by the increase in the B/W ratio. The percentage of the carbon content improved from 59.2 ± 1.2 to 68.6 ± 0.3 at 260 °C-B/W ratio of 0.43. The percentage content of the hydrogen decreased at the end of the HTC pretreatment. The percentage content of hydrogen further reduced to 4.6 ± 0.04 due to the increased severity of the carbonization at 260 °C. The oxygen content decreased to 26.4 ± 1.0 at 260 °C-B/W ratio of 0.43 because of the increased severity of the process condition. It is established that, oxygen content in biomass is reduced by decarbonylation, decarboxylation and dehydration reactions through HTC, and the extent of the reduction in oxygen content is more noticeable at higher conversion temperatures, hence, led to the increased percentage content of carbon in the carbonized wood shavings [12], [32], [33].

Table 1. The proximate, utilitate and energy characteristics of the carbonized solid fuer										
Parameters	Raw	230°C-	230°C-	230°C-B/W	260°C-B/W	260°C-	260°C-			
		B/W 0.11	B/W 0.25	0.43	0.11	B/W 0.25	B/W 0.43			
Proximate										
VM (%)	85.8±0.3	70.9±0.2	66.5±0.2	66.3±0.1	53.1±0.4	53.1±0.05	52.9±0.01			

Table 1. The proximate, ultimate and energy characteristics of the carbonized solid fuel

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FC* (%)	14.8±0.3	28.8±2.0	33.3±2.3	33.6±3.0	46.4±0.9	46.8±0.3	47.0±0.30
Ash (%)	0.08±0.1	0.26±0.2	0.19±0.1	0.11.5±0.1	0.53±0.4	0.12±0.04	0.08±0.01
FR	0.29±0.01	0.41±0.01	0.50±0.03	0.51±0.01	0.87±0.01	0.88±0.12	0.89±0.01
Ultimate							
C (%)	49.7±0.1	59.2±1.2	60.7±1.1	59.5±2.4	68.4±0.2	68.5±0.2	68.60.3
H (%)	5.9±0.00	5.1±0.02	5.0±0.02	5.1±0.03	4.6±0.04	4.6±0.04	4.7±0.01
N (%)	0.3±0.1	0.3±0.0	0.3±0.00	0.3±0.00	0.3±0.00	0.3±0.00	0.30±0.0
O* (%)	44.0±0.1	35.4±1.0	33.9±0.9	35.1±2.1	26.8±0.2	26.6±0.3	26.4±1.0
Energy							
EDR	-	1.22±0.02	1.26±0.01	1.23±0.04	1.41±0.01	1.41±0.01	1.42±0.01
EY (%)	-	76±0.4	82.4±0.1	85.4±0.5	80.8±0.6	84.9±0.3	87.9±0.20

*Calculated by difference

3.4 The plotted van krevelen representation for the produced hydrochars.

The van Krevelen diagram gives an idea on the kind of fuel and the changes in biomass properties after conversion. A fuel with lesser ratio of atomic O/C - H/C is more desired because it indicates the tendency that less smoke will be produced accompanied with less vapour and less loss of energy when combusted [19], [34]. The obtained data for the ultimate characteristics was transformed to the van Krevelen plot for the produced hydrochars as shown in Fig. 6. The Fig. 6 indicated that the extent of transformation of the produced solid fuel to bio-coal increased as the carbonization severity at which they were produced increased. This is indicated by the shift from the right-hand top corner to the left-hand bottom corner of the diagram. The lowered O/C and H/C atomic ratios indicated that a good fuel was formed. The produced solid biofuel were closed to the region of the preduced solid biofuel.



Fig. 6. Van Krevelin Plot.

4 Conclusion

This study investigated the influence of carbonization temperature and the ratio of B/W in the HTC of wood shavings. This study revealed that carbonised wood shavings of improved fuel characteristics can be obtained at higher B/W ratio with no inherent negative effect on hydrochar properties. The proximate and ultimate characteristics of the hydrochar slightly improved and remained stable in some instance by increasing B/W ratio. The produced hydrochar fell within the region of peat and lignite which indicated that it can be used for co-combustion with coal.

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