

Hydrothermal carbonization of compressed water hyacinth: Effects of operation parameters on energy conversion and characterization of products

Tassapak Wutisirirattanachai ¹⁾, Solomon Addisu Legesse ²⁾, and Shinjiro Sato ^{1)*}

1) Graduate School of Science and Engineering, Soka University, 1-236, Tangi-cho, Hachioji, Tokyo, 192-8577, Japan

2) College of Agriculture and Environmental Sciences, Bahir Dar University, Bahir Dar, Ethiopia

* Corresponding author: ssato@soka.ac.jp

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Abstract A massive infestation of a free-floating aquatic invasive plant, water hyacinth (*Eichhornia crassipes*) has been causing numerous problems in Ethiopia. Water hyacinth (WH) is mainly composed of lignin, crystalline cellulose, and hemicellulose polymer, thus its solid part can be used as a potential alternative energy source through thermochemical treatment. Hydrothermal carbonization (HTC) is a conversion of biomass into solid components of char (biochar) and carbon-rich liquid products (biooil and aqueous phase) by heating biomass in the presence of water in a closed and autogenous environment. The objective of this study was to evaluate the effects of various operating conditions on energy conversion efficiency and characteristics of the final products using water hyacinth through hydrothermal carbonization. Hydrothermal treatment was carried out at three different operating temperatures (210°C, 240°C, and 270°C) for three different retention times (1, 2, and 4 hr) to obtain biochar, biooil, and aqueous phases. The study shows the possibility to convert WH biomass to biochar and biooil through HTC, where the best performance in energy conversion from both products was 64.5% at operating temperature of 240°C and retention time of 4 hr. Operation temperature and retention time significantly affected yield and higher heating value of biochar and biooil. Biochar yield decreased and biooil yield increased with increasing operating temperature and retention time. However, there are still low yield of biooil which potentially contains high gross energy. Therefore, it is necessary to evaluate other variables such as type of feedstock, amount and type of liquid mixed with feedstock, and operating environment to improve energy conversion efficiency through hydrothermal carbonization.

Keywords: biocrude oil; biofuel; biowaste; hydrothermal treatment, van Krevelen diagram

1. INTRODUCTION

A massive infestation of water hyacinth (*Eichhornia crassipes*), a free-floating aquatic invasive weed, has

been causing numerous problems in Ethiopia (Hill et al. 2011). The invasion of water hyacinth affects water transportation systems, hydroelectric operations, hydraulics of canals and rivers, flooding hazard, human health,

fishing, irrigation, navigation, livestock, and aquatic biodiversity (Dersseh et al. 2019). Water hyacinth has invaded the largest lake in the Ethiopian highlands, Lake Tana, and was officially recognized in September 2011 and the infestation area of the lake has reportedly reached up to 50,000 ha (Abera 2018). The rapid and extensive increase has led to urgent necessity of research aimed at solving the problem or finding ways to utilize the plant. One of effective ways of treating water hyacinth is separating the plant into liquid and solid parts by squeezing or compressing, of which each part can be utilized for further treatments (Hudakorn & Sritrakul 2020). Water hyacinth is mainly composed of lignin, crystalline cellulose, and hemicellulose polymer, thus its solid part can be used as a potential alternative energy source (Moki et al. 2020, Zhang et al. 2020).

Thermochemical treatment is one of effective technologies to convert biomass into energy sources because it does not require expensive equipment, concludes treatment quickly, and produces valuable products (Yao & Ma 2019). There are two main thermochemical treatments which are pyrolysis and hydrothermal carbonization. Pyrolysis is a traditional method in thermochemical conversion which is the degradation (decomposition) of biomass by heat in the absence of oxygen (Demirbas & Arin 2002). On the other hand, hydrothermal carbonization (HTC) is the conversion of biomass into solid components of char (biochar) and carbon-rich liquid products (biooil and aqueous phase) by heating biomass in the presence of water in closed and autogenous environment (Sahoo et al. 2019). HTC has unique advantages in terms of biomass conversion compared to pyrolysis: 1) HTC requires relatively low reaction temperatures (e.g., 180°C–350°C); 2) HTC keeps water in liquid state under autogenous pressure prevents conversion of biomass to biogas such as carbon dioxide and nitrous oxide (Li et al. 2013); 3) HTC can be used as the most suitable

pretreatment methods for wet biomass with high organic content (Zhang et al. 2020). There have been many studies on the transformation of water hyacinth through hydrothermal carbonization (Román 2020, Zhang et al. 2020), however have rarely been studied on energy conversion efficiency of water hyacinth into biochar and biooil.

Therefore, the objective of this study was to evaluate the effects of various operation conditions on energy conversion efficiency and characteristics of the final products using compressed water hyacinth through hydrothermal carbonization.

2. MATERIALS AND METHODS

2.1 Materials

Water-born water hyacinth used in this study was collected from Suijo Park in Saitama, Japan. The collected water hyacinth was immediately transported to a laboratory at Soka University and crushed and squeezed to separate to liquid and solid residues. The solid residue was dried at 45°C for 48 hr in a drying oven (WFO-510, Eyela). The dried solid residue was sieved with particle size less than 2 mm (WH), and then stored in a drying oven at 90°C for 24 hr for further experiments. The WH sample used in this study showed 11.8% of moisture content, 39.6% of total carbon, and 16.4 MJ kg⁻¹ of higher heating value (HHV; Table 1).

2.2 Hydrothermal carbonization of water hyacinth

To investigate the effects of operating reaction temperatures and retention time on HTC, three operating reaction temperatures (210°C, 240°C, and 270°C) and three retention times (1, 2, and 4 hr) were performed using an electric furnace (FUW242PA, Advantec). Hereafter, the operating conditions are expressed as operating reaction temperature/retention time such as 210/1 and

Table 1. Main characteristics of solid residue of water hyacinth.

pH	Moisture content (%)	C (%)	H (%)	N (%)	O [†] (%)	Higher heating value (MJ kg ⁻¹)
6.75	11.8	39.6	6.11	1.81	52.5	16.4

[†] Difference by 100% – (C%+H%+N%)

270/4. Approximately 4.0 g of dried WH and 60 mL of distilled water were filled in a 100 mL stainless steel autoclave vessel (TX-202, Huanyu). The vessel was purged with high purity nitrogen gas to avoid oxygen in the vessel. The vessel was completely enclosed with a steel handle. The temperature was raised to the desired temperature at a heating rate of 5°C min⁻¹ and kept for a specific retention time at the reaction temperature (Zhang et al. 2020). After the reaction, the vessel was left to cool down to room temperature. The sample was taken out and recovered from the vessel, and stirred with 80 mL of dichloromethane for 2 hr in glass beaker to separate into solid product, biooil, and aqueous phase (Jaiswal et al. 2021). The solid and liquid phases were separated by vacuum filtration by filter paper (Whatman No.1). The solid residual was rinsed in deionized water for 24 hr, dried in the oven at 95°C, weighed and designated as solid product. The liquid phase was loaded into a separatory funnel, then separated to obtain a water-soluble oxygenated hydrocarbon designated as aqueous phase, and dichloromethane mixed with biooil in the bottom of the funnel. The dichloromethane compound was evaporated at room temperature, weighed, and designated as biooil. Each experiment was performed at least three times to ensure repeatability and reliability.

The yield percentage of each of the final products was calculated as follows:

$$\text{Solid product yield (SY)(wt\%)} = \frac{\text{weight of solid product}}{\text{weight of WH}} \times 100\% \quad (1)$$

$$\text{Biooil yield (OY)(wt\%)} = \frac{\text{weight of biooil}}{\text{weight of WH}} \times 100\% \quad (2)$$

$$\begin{aligned} \text{Aqueous phase and gas yield (AY)(wt\%)} \\ = \frac{\text{weight of (WH - solid product - biooil)}}{\text{weight of WH}} \times 100\% \end{aligned} \quad (3)$$

2.3 Analytical procedures of hydrothermal products of water hyacinth

The dried WH and the final products (solid product and biooil) from each HTC operation were analyzed for elemental composition and higher heating value (HHV). The elemental composition of each sample was determined using a CHNS organic elemental analyzer (2400II, PerkinElmer). The total amount of O was calculated by the difference between 100% and sum of C%, N%, and H%. The HHV of each sample was determined by a bomb calorimeter (6050, Parr) in accordance to is based on ASTM D240-19 (ASTM 2019).

In order to determine energy conversion efficiency of HTC operation, energy densification and energy recovery efficiency of the final products (solid product or biochar and biooil) were calculated by the following formulas:

$$\text{Energy densification (ED)} = \frac{\text{HHV of solid product or biooil}}{\text{HHV of WH}} \quad (4)$$

$$\begin{aligned} \text{Energy recovery efficiency (\%)} \\ = (\text{yield of biochar or biooil (\%)} \times \text{ED of biochar or biooil}) \end{aligned} \quad (5)$$

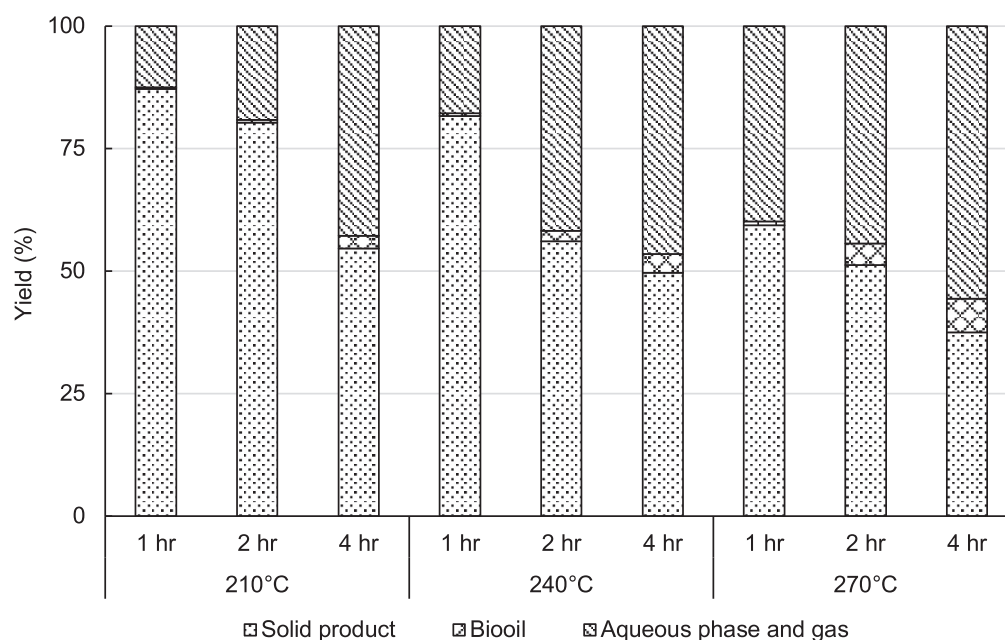


Figure 1. Relative yield percentages of solid product, biooil, and aqueous product and gas from hydrothermal carbonization of water hyacinth.

3. RESULTS

3.1 Yields of hydrothermal carbonization products

The HTC reaction of WH was performed at three different temperatures of 210°C, 240°C, and 270°C for three different retention times of 1, 2, and 4 hr. Relative yields of solid product (SY), biooil (OY), and aqueous phase and gas (AY) showed wide ranges (37.5%–87.2%, 0.35%–6.86%, 12.5%–55.6%, respectively (Fig. 1). The operating condition of 210/1 resulted in the highest SY and was not significantly different from those of 210/2 and 240/1, but these values were significantly higher than those of other operating conditions. The maximum OY and AY values were observed at operating condition of 270/4.

3.2 Elemental analysis of hydrothermal carbonization products

Relative C, H, N, and O contents of solid product ranged between 42.7%–50.0%, 3.56%–5.27%,

1.48%–1.97%, and 44.5%–50.3%, respectively, and those of biooil between 64.8%–71.1%, 9.58%–16.9%, 0.54–2.47%, 11.5%–24.3%, respectively (Table 2). As operating temperature and retention time increased, C and N contents increased and H and O contents decreased in the solid product, while there were no particular trends in elemental contents except for increasing N in biooil with increasing operating temperature and retention time.

3.3 Fuel characteristic of hydrothermal carbonization products

Comparing with WH (16.4 MJ kg⁻¹), HHV values of solid product and biooil overall increased to 16.0–19.7 and 23.1–27.8 MJ kg⁻¹, respectively (Table 2). The maximum HHV of solid product and biooil were observed at operating conditions of 270/4 and 240/4, respectively. HHV values of solid product overall increased with both increasing operating temperature and retention time, while those of biooil overall increased only with increasing retention time (Fig. 2).

Table 2. Elemental analysis and higher heating value for water hyacinth, solid product, and biooil.

Operating conditions [†]	Elemental analysis (%)				Higher heating value (MJ kg ⁻¹)	Energy densification
	C	H	N	O [‡]		
Water hyacinth						
	39.6	6.11	1.81	52.5	16.4	-
Solid product						
210/1	42.7	5.27	1.51	50.5	16.1	0.983
210/2	43.0	5.04	1.54	50.4	16.0	0.974
210/4	44.4	4.16	1.82	49.6	17.9	1.09
240/1	43.2	5.05	1.48	50.3	16.0	0.976
240/2	46.1	4.20	1.85	47.9	17.8	1.09
240/4	48.8	4.33	1.59	45.3	19.2	1.17
270/1	45.5	4.49	1.67	48.3	17.1	1.04
270/2	46.8	4.20	1.70	47.4	18.5	1.13
270/4	50.0	3.56	1.97	44.5	19.7	1.20
Biooil						
210/1	69.9	14.8	0.540	14.7	23.1	1.41
210/2	70.2	16.2	0.780	12.9	25.4	1.55
210/4	67.8	12.1	1.63	18.4	25.9	1.58
240/1	71.1	16.9	0.567	11.5	24.8	1.51
240/2	64.5	9.60	1.60	24.3	26.3	1.60
240/4	67.0	10.8	2.16	20.1	27.8	1.69
270/1	69.1	13.4	1.20	16.3	25.0	1.52
270/2	65.3	9.58	2.04	23.1	23.8	1.45
270/4	69.9	10.8	2.47	16.8	26.2	1.60

[†] Operating conditions refer to operating temperature (210°C, 240°C, and 270°C) and retention time (1, 2, and 4 hr).

[‡] Difference by 100% – (C%+H%+N%)

$$\text{Energy densification (ED)} = \frac{\text{HHV of product}}{\text{HHV of WH}}$$

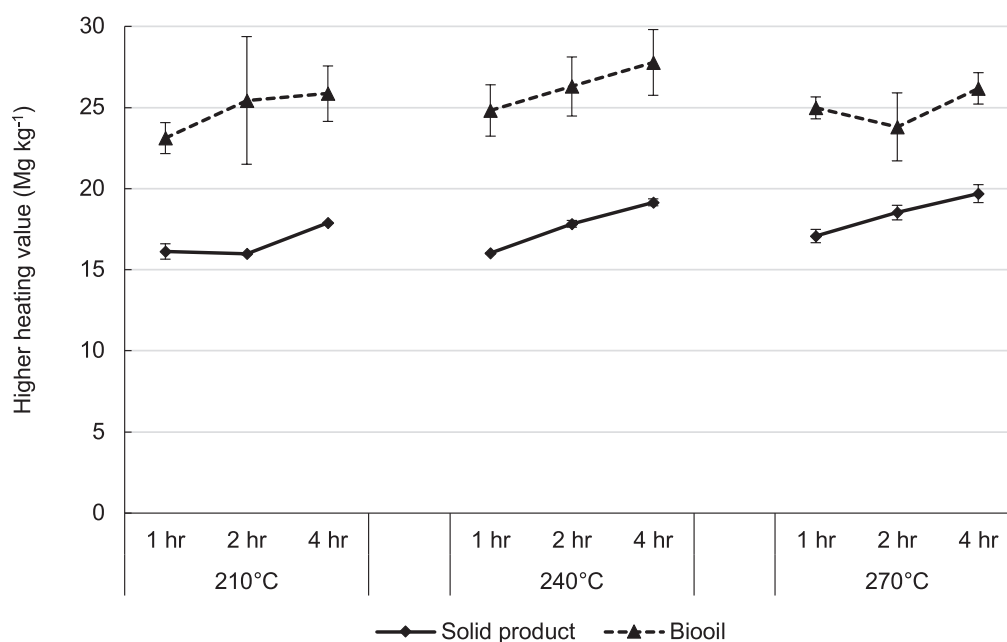


Figure 2. Higher heating values of solid product and biooil from hydrothermal carbonization of water hyacinth.

In addition, when operating temperature and retention time increased, ED of solid product overall increased, however ED of biooil was overall positively affected with only increasing retention time (Table 2). The highest ED values were obtained at operating conditions of 270/4 and 240/4 for solid product and biooil, respectively. Noteworthy was that ED values of solid product at operating conditions of 210/1, 210/2, and 240/1 were below 1.00 (0.983, 0.974, 0.976, respectively (Table 2), which meant HHV of solid product was lower than that of WH feedstock. This may be explained by a possibility of operating temperature and retention time being too low and short to complete HTC reactions to form biochar and biooil (uncarbonized). In fact, visual observation of solid product at these operating conditions (Fig. 3b, 3c, and 3d) revealed that appearance was similar to that of WH (Fig. 3a) and different from that at operation condition of 270/1 (Fig. 3e), which could represent functional biochar produced through HTC. Therefore, in this study, solid product of these 3 operating conditions were excluded as uncarbonized

product, and those at the rest of operation conditions were defined as biochar.

3.4 Energy characteristic of hydrothermal carbonization products

Energy recovery efficiency of biochar (excluding uncarbonized product at operating conditions of 210/1, 210/2, and 240/1) exhibited 45.0%–61.7% and overall showed a decreasing tendency with increasing operating temperature (comparing only 4 hr retention time among 3 different temperatures) and retention time (comparing only between 240°C and 270°C temperatures, respectively (Fig. 4). On the other hand, energy recovery efficiency of biooil exhibited 1.28%–10.9% and overall showed an increasing tendency with increasing operating temperature (comparing only 4 hr retention time among 3 different temperatures) and retention time (comparing only between 240°C and 270°C temperatures, respectively; Fig. 4). The sum of energy recovery efficiency of biochar and biooil resulted in an almost equivalent range of 63.0% and 64.5%, except for

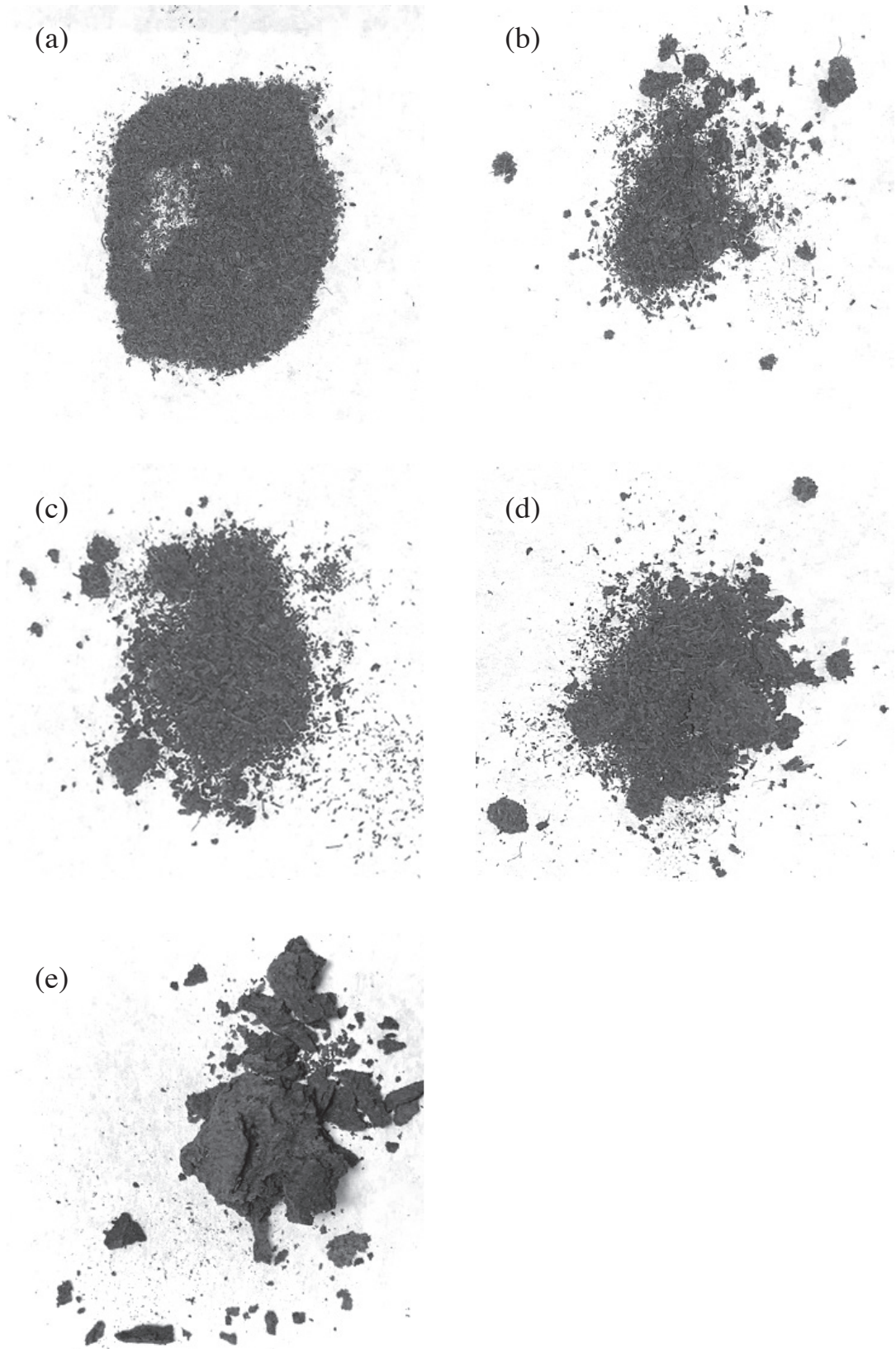


Figure 3. Images of (a) water hyacinth before and solid products after hydrothermal carbonization of water hyacinth with operating conditions (b) 210/1, (c) 210/2, (d) 240/1, and (e) 270/1.

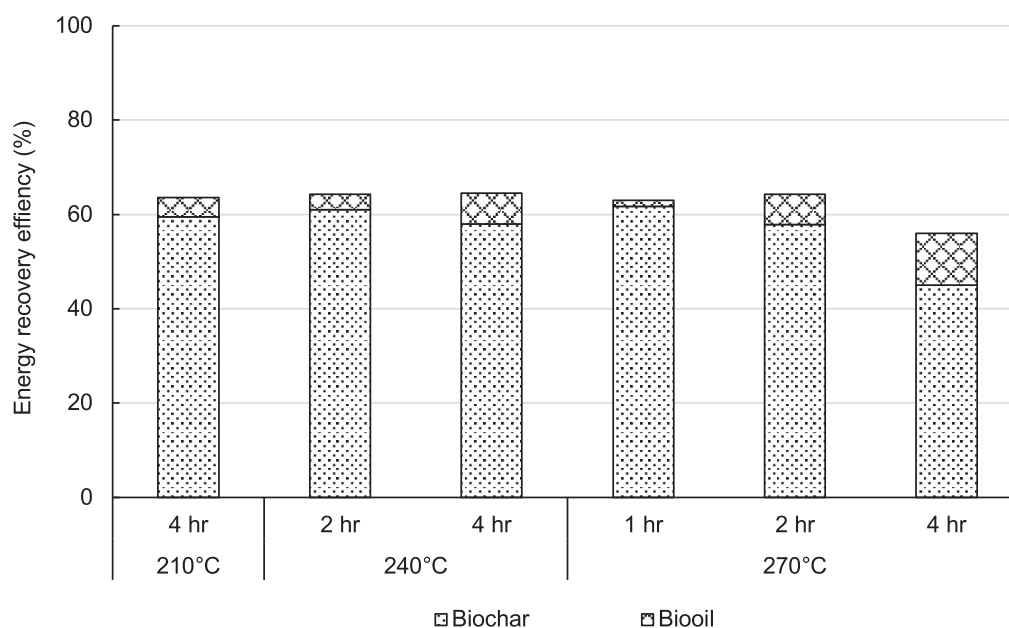


Figure 4. Energy recovery efficiency of biochar and biooil from hydrothermal carbonization of water hyacinth.

that at operating condition of 270/4 showing 56.0%.

4. DISCUSSION

4.1 Effects of operating temperature and retention time on energy conversion

In this study, energy conversion from varying operating conditions showed 56% to 64.5% (Fig. 3). The best energy conversion efficiency was achieved at operation 240/4 comprising of 58.0% from biochar and 6.5% from biooil, most of which came from biochar. The higher the operating temperature and the longer retention time were, the lower the yield of biochar obtained (Fig. 1) which was the main reason for lower energy conversion. Higher temperature and longer reaction time could lead to enhanced migration of C to gas phase as mostly CO₂ meaning reduced energy conversion efficiency as biochar (Singh 2015). Comparing energy conversion of hydrothermal carbonization with other studies, Zhang (2020) showed the highest hydrochar (biochar produced through HTC) energy recovery efficiency from WH of

66.1% at operating condition of 210°C for 0.5 hr and the lowest of 39.8% at 270°C for 1 hr. Román (2012) showed that the highest energy conversion efficiency on hydrochar from walnut shell was 58.9% at operating temperature of 190°C for 20 hr, and the lowest was 49.6% at 230°C for 20 hr. The different results in energy conversion efficiency may indicate that there are other variables than operating temperature and retention time such as type of feedstock, amount and type of liquid mixed with feedstock, and operating environment such as size and withstanding pressure of carbonizing reactor. In this study, the sum of energy conversion of biochar and biooil was comparable with other studies and approximately same for all operating conditions except for 270/4. Therefore, it appears that these operating conditions used in this study were suitable for HTC of water hyacinth. However, depending on demand for different final products, an operation of 270/1 may be suitable for large energy conversion by biochar and an operation of 270/4 can produce high energy conversion by biooil.

4.2 Effects of operating temperature and retention time on hydrothermal reaction

Operating temperature played important roles on the final product yields during HTC process in this study, which was consistent with previous studies that showed temperature was the most critical parameter for the conversion process (Garrote et al. 1999). At all retention times (1, 2, and 4 hr), SY decreased and OY increased with increasing operating temperature (from 210°C to 240°C then 270°C; Fig. 1). Reza (2014) explained that temperature accelerated kinetic energy on HTC reaction at the beginning of the process. Mok & Antal (1992) referred that through decarboxylation and depolymerization reactions happened at the first stage of HTC, hemicellulose and lignin in biomass began to degrade with water at operating temperature from 190°C, and dissolve into water from 220°C. These reactions also led to forming the liquid phase intermediates in which a variety of organic compounds were incorporated (Xiu et al. 2010). The intermediates could undergo a wide range of reactions such as isomerization, dehydration, and condensation, which could separate the intermediates into biochar, biooil, and aqueous phase (Bobleter 1994).

Retention time also showed important effects on the final product yields during HTC reaction. At all operating temperatures (210°C, 240°C, and 270°C), increasing retention time (from 1 to 2 then 4 hr) caused decrease of SY and increase of OY (Fig. 1). Noteworthy was that operating conditions of 210/1, 210/2, and 240/1 probably did not create enough energy to complete HTC reactions to form biochar resulting in high SY (80.3%–87.2%) of uncarbonized products. However, longer retention time even with lower temperatures (210/4, and 240/2, and 240/4) could create enough energy to complete the first HTC reaction of decomposition to form biochar, then as a result SY was reduced (49.7%–56.1%). In case of operating temperature of 270°C, even with

short retention times (1 and 2 hr), it is possible that there was enough energy to complete the decomposition reaction to form biochar. Therefore, SY values at 270/1 and 270/2 (51.3%–59.3%) were comparable with those at operating conditions of 210/4, and 240/2, and 240/4. However, 270/4 caused significant reduction of SY to 37.5% probably because the secondary HTC reaction of repolymerization may have continued longer to partially convert biochar to biooil (Zhang et al. 2020), resulting in high OY (6.86%).

Relationship between O/C and H/C atomic ratios of feedstock (WH) and the final products (solid product and biooil) under different operating conditions can be summarized as van Krevelen diagram (Fig. 5), which can represent different reactions during HTC such as dehydration, decarboxylation, and demethylation. WH used in this study had O/C (0.99) and H/C (1.8) ratios which fell in a category of carbohydrate based on van Krevelen diagram for major biomolecular component areas (Julien et al. 2016). Solid product produced in this study had O/C and H/C ratios ranging 0.67–0.89 and 0.8–1.5, respectively, which decreased when operating temperature and retention time increased (Fig. 5). van Krevelen diagram can suggest that the dominant HTC mechanism was dehydration resulting in C-richer solid product from WH with increasing temperature and retention time, which was represented by a tannin category in van Krevelen diagram (van Krevelen 1982, Kambo et al. 2017). In addition, decreasing O and H contents of solid product with increasing temperature and retention time (Table 2) can also explain the reason for decreasing solid product yield (Fig. 1) through dehydration reaction during HTC (Lu et al. 2013). On the other hand, biooil produced in this study had O/C and H/C ratios ranging 0.12–0.28 and 1.75–2.83, respectively, which partially fell in a category of biocrude oil based on van Krevelen diagram (Cheng et al. 2021). Increased H/C and decreased O/C ratios of

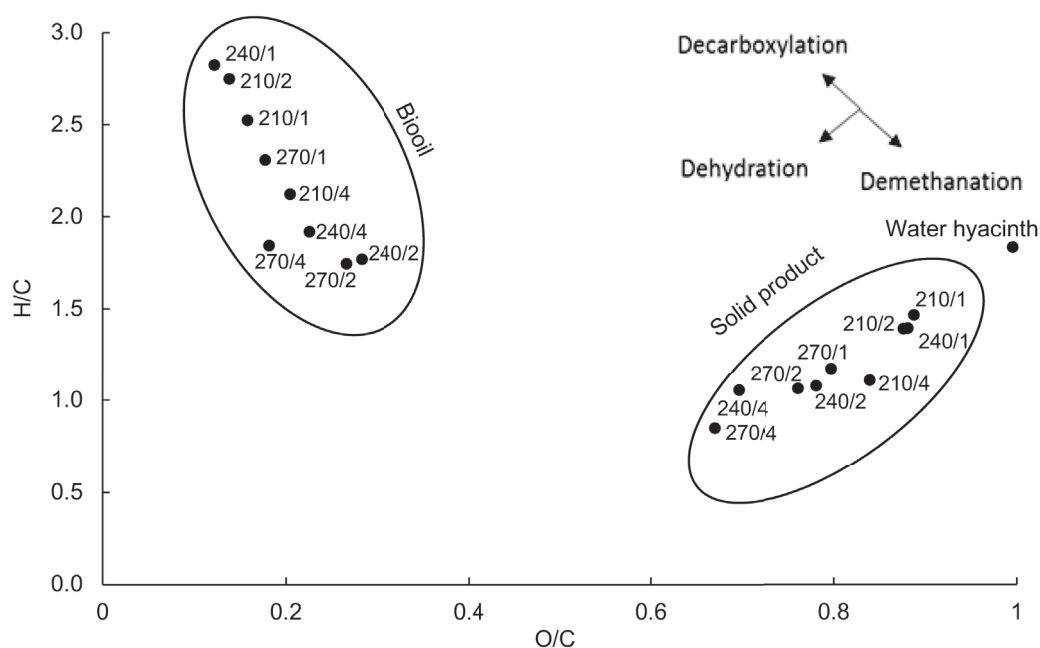


Figure 5. van Krevelen diagram showing the H/C and O/C atomic ratios of water hyacinth, solid product, and biooil from hydrothermal carbonization of water hyacinth.

biooil compared with those of WH could be explained by hydrodeoxygenation which could happen during the initial reaction of HTC (Bi et al. 2014). Among biooil produced at different operating conditions, those at middle temperature and retention time (e.g., 210/4, 240/2, and 240/4) probably went through a next HTC reaction of demethanation which lost more H per mole of C. Then, next reaction could be decarboxylation which lost more O per mole of C resulting in reduced O/C ratio for biooil produced at higher temperature (e.g., 270/2 and 270/4).

5. CONCLUSION

This study investigated the effects of various hydrothermal reactions at different temperatures and retention times on energy conversion efficiency and characteristics of the final products using compressed water hyacinth through hydrothermal carbonization. In this study, it was shown possible to convert water hyacinth biomass to biochar and

biooil through HTC. HTC's best performance in energy conversion from biochar and biooil was 64.5% at operating temperature of 240°C and retention time of 4 hr. Biochar and biooil yields greatly varied depending on operating conditions. Therefore, depending on demand for different final products, operation of 270/1 may be suitable for large energy conversion by biochar and operation of 270/4 can produce high energy conversion by biooil. It was shown that hydrothermal carbonization had a potential to increase value on WH feedstock due to producing C-rich fuel when appropriate operating conditions were applied. However, there are still low yields of biooil which potentially contains high gross energy. Therefore, there are needs to evaluate other variables such as type of feedstock, amount and type of liquid mixed with feedstock and operating environment such as size and withstanding pressure of carbonizing reactor to improve energy conversion efficiency through hydrothermal carbonization.

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