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Hydrothermal Treatment of Herb Residue for Solid Fuel Production

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Abstract. Hydrothermal processing is appraised as one of advanced technologies for wet solid waste handling. In this study, herb residue was subjected to hydrothermal treatment. Calorific value, yield, and also proximate analysis of obtained hydro-char were investigated. A cylindrical reactor with an internal volume of 2.5 Litres made of stainless steel and a low-tech component was used in the experiment. The reactor was equipped with a stirrer to ensure heat transfer took place through the entire parts of the solid-water mixture. Solid products were dried by a microwave oven before analysis. The results show that the final temperature, holding time, and solid-water ratio have various effects on the hydro-char yield, calorific value, and proximate analysis of the hydrothermal products. The hydro-char yield decreased with the increase in final temperature and holding time. Meanwhile, the highest hydro-char yield was obtained at the solid-water ratio of ¼. The hydro-char calorific value increased with the increase in final temperature, holding time, and solid-water ratio. The rise in final temperature, holding time, and solid-water ratio resulted in a lower moisture content and volatile matter but higher fixed carbon. Meanwhile, the ash content increased with the solid-water ratio.

Keywords: herb residue, hydrothermal, production, solid fuel, treatment

INTRODUCTION

Hydrothermal as one of thermochemical conversion technology, has been widely applied to process various kinds of wastes, such as hospital solid waste, biomass, pyrolytic plastic waste residue (Jain, Balasubramanian, and Srinivasan 2016; Garrote, Domínguez, and Parajó 1999; Ruksathamcharoen et al. 2019; Leng et al. 2020; Alam et al. 2019), *Ganoderma lucidum* (Machmudah et al. 2019), and palm oil mill effluent (Lee, Chin, and Cheng 2019). The hydrothermal processing of wet biomass comprises hydrolysis, condensation, decarboxylation, and dehydration (Sevilla and Fuertes 2009). It is considered as a promising technology for handling municipal solid waste, such as a short time process for large volume, odorless, and no need for biomass moisture removal. Besides, pathogen elimination and sterile and hygienic products can be obtained (Silvia Román et al. 2018). On the other hand, the complexity of the hydrothermal process and very feedstock-dependent outputs could

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lead to the implementation of laboratory and pilot plant scale only (Wiedner et al. 2013). Therefore, this could be a challenge to develop this technology.

Wang et al. (C. Wang et al. 2020) developed a hydrothermal treatment of wood sawdust to produce wood vinegar and briquette fuels. Response surface methodology was employed to explore the interaction of hydrothermal temperature and the ratio of wood sawdust to deionized water on the yield of wood vinegar. The findings revealed that during the process, the hydrothermal temperature was the most important factor. Hydrothermal carbonization of grape marc has been investigated (Basso et al. 2016). The hydro-char yield decreased with the residence time and processing temperature. Another work on hydrothermal carbonization on the sunflower stem and walnut shell was studied in various circumstances (S. Román et al. 2012). The process increased their heating values up to 1.75 and 1.5 fold compared to natural biomass. Temperature and the biomass-to-water ratio were found to have a major impact on the process. Meanwhile, the hydrothermal treatment on pig manure and rice straw has been investigated (Liu et al. 2017). For each feedstock, higher temperatures brought out lower hydro-char yields and higher hydro-char ash contents. Those findings indicate that processing time and temperature are important factors in controlling the properties of hydro-char. Simiar results were also reported by Lu and coworkers on cellulose hydrothermal treatment (Lu et al. 2013).

However, the literature on hydrothermal treatment of the herb residue is still very limited. "Herb residue" is solid waste originating from the medicine mill industry that uses natural plants as raw materials. Its volume is predicted to increase in the following years, as indicated by medicinal plants' production, supply, and demand (Statistics 2018). Moreover, around 1.5 million tons of herb residue has been produced annually in China (P. Wang, Yu, and Zhan 2012). Domestically, based on the Indonesian Association of Herbal and Traditional Medicine Entrepreneurs, there have been around 342 herbal medicine factories in Indonesia (Gabungan Pengusaha Jamu dan Obat Tradisional Indonesia 2018). Indeed, this pharmaceutical industry development would inevitably increase the volume of herb residue. Consequently, this considerable residue should be appropriately handled to deter environmental threats. Therefore, in this present work, the effect of final temperature, holding time, and the solid-water ratio in hydrothermal treatment on the calorific value, solid yield, and proximate analysis of obtained hydro-char were investigated.

MATERIALS AND METHODS

The raw material was collected from a herbal medicine factory located in Sukoharjo City, Central Java Province, Indonesia. A cylindrical reactor with an internal volume of 2.5 Litres made of stainless steel was used in the experiment. The reactor was equipped with a stirrer to ensure heat transfer took place through the entire parts of the solid-water mixture. For every run, the herb residue of 300 g was placed in the reactor and mixed with water of 900 g, 1200 g, and 1500 g. Then, the reactor was shut and heated up until a specific set temperature of 180 °C, 200 °C, and 220 °C, then held for 30, 60, and 90 minutes. The decomposition temperature of cellulose and hemicellulose at 150 °C and 180 °C, respectively, (Yang et al. 2007) were basis for selecting the processing temperatures.

Once the holding time was achieved, the operation was stopped and cooled. After the reactor reached room temperature, the discharge valve was opened to release gaseous products. A cloth filter was used to separate the solid (hydro-char) and liquid products. After that, an electric oven was operated for 2-3 hours to dry the hydro-char before analysis. The experimental equipment is illustrated in Fig. 1.

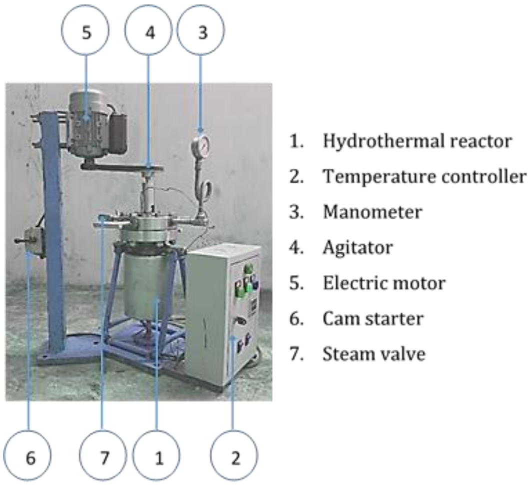

Fig. 1: Hydrothermal equipment

This system used a batch process to allow a simple control and provide a low level of complexity. An electric heater was employed for the heating process. By varying the hydrothermal carbonization process final temperature, holding time, and solid-water ratio, the measured response variables were hydro-char calorific value, hydro-char yield, and proximate analysis of the hydro-char. The hydro-char yield was calculated by the following formula (Ahmad et al. 2010):

$$\text{Hydro} - \text{char yield} = \frac{\text{weight of the dried solid product}}{\text{weight of the dried feedstock}} \text{ x } 100\ \% \quad (1)$$

RESULTS AND DISCUSSION

Raw material and products characterization

Hydrothermal treatment with variations of temperature, time, and the solid-water ratio was applied to dry herb residue samples. Proximate analyses for raw material and the products were carried out according to ASTM D5142-90. A bomb calorimeter Gallenkamp Autobomb CBA-305-010M was used to determine the calorific values of all samples. Table 1 presents the values of proximate analysis and calorific value of the raw material and the hydro-chars.

Table 1. Proximate analysis and calorific values or the raw material and hydro-chars

	Moisture content (%)	Volatile matter (%)	Fixed Carbon (%)	Ash (%)	Calorific value cal/gram
Raw	10.057	62.519	21.394	6.031	4160.379
HT 1	4.335	63.594	25.626	6.446	4598.882
HT 2	4.189	58.252	31.220	6.340	5145.301
HT 3	3.293	54.364	35.825	6.519	5442.778
HT 4	4.109	56.072	33.155	6.665	5191.558
HT 5	3.708	55.187	34.064	7.043	5841.435
HT 6	3.045	56.607	34.155	6.239	5662.191
HT 7	2.434	54.622	36.533	6.362	5690.007

Note: HT 1: 1/5, 180 °C, 30 min; HT 2: 1/5, 200 °C, 30 min; HT 3: 1/5, 220 °C, 30 min; HT 4: 1/4, 200 °C, 30 min; HT 5: 1/3, 200 °C, 30 min; HT 6: 1/5, 200 °C, 60 min; HT 7: 1/5, 200 °C, 90 min

Time required to reach the final temperature

The time spent to achieve the final temperature relied on the set final temperature and the solid-water ratio. Fig. 2 shows that higher final temperature consumed a longer time (180 °C, 47 min; 200°C, 51 min; and 220 °C, 65 min). In this study, for the same final temperature, the solid-water ratio of 1/5, 1/4, and 1/3 spent time of around 51 min, 58 min, and 60 min, respectively. It means that the higher the ratio of solid water, the longer the time needed.

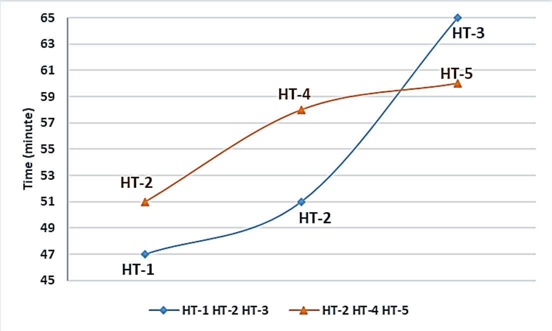

Fig. 2: Time consumed to attain the final temperature

Effect of final temperature on the hydro-char yield

Fig. 3 illustrates the hydro-char yield gained due to the final temperature variation. The increased final temperature was followed by the increased reactor average pressure (180 °C, 11.00 kg/cm^2; 200 °C, 19.17 kg/cm^2; 220 °C, 36.67 kg/cm^2). The higher the final temperature, the lower the hydro-char yield. The hydro-char yields were 48.50%, 47.33%, and 46.23%, respectively.

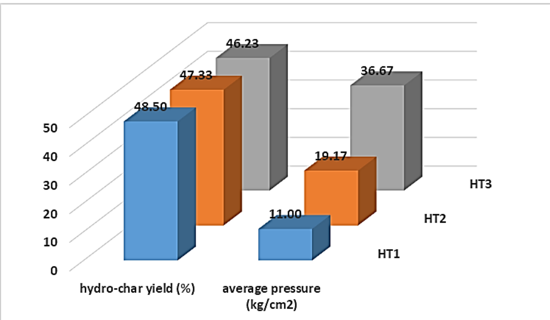

Fig. 3: Effect of final temperature on the hydro-char yield

Effect of holding time on the hydro-char yield

The hydro-char yield was affected by the holding time, as shown in Fig. 4. Increasing the holding time also increased the reactor average pressure (30 min,19.17 kg/cm^2; 60 min, 25.67 kg/cm^2; 90 min, 30.56 kg/cm^2). The longer holding time brought out a lower hydro-char yield. The hydro-char yields were 47.33 %, 46.10 %, and 45.87 %, respectively.

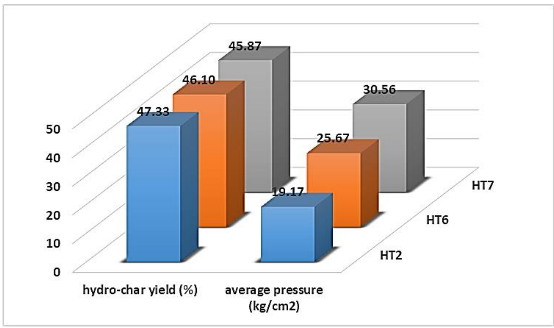

Fig. 4: Effect of holding time on the hydro-char yield

Effect of solid-water ratio on the hydro-char yield

The hydro-char yield was affected by the solid-water ratio, as depicted in Fig. 5. The increased solid-water ratio from 1/5, 1/4, dan 1/3 resulted in the average reactor pressure of 19.17, 17.17, and 23.00 kg/cm^2. The hydro-char yields were 47.33 %, 49.10 %, and 48.10 %, respectively. It was generally confirmed that the solid-water ratio of ¼ gave the highest value of hydro-char yield.

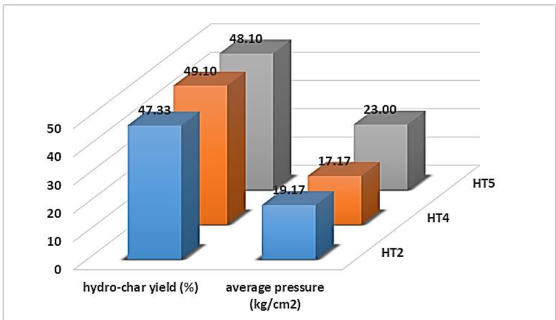

Fig 5: Effect of solid-water ratio on the hydro-char yield

Effect of final temperature on the calorific value

Fig. 6 shows the product appearance of the hydrothermal treatment. The hydro-char produced from a final temperature of 220 °C has the darkest brown colour, whereas the hydro-char obtained from final temperatures of 200 °C is darker than 180 °C.

The calorific value of herb residue subjected to the hydrothermal treatment increased with the final temperature (4598.882 cal/gram, 180 °C; 5145.301 cal/gram, 200 °C; and 5442.778 cal/gram, 220 °C), as shown in Fig. 7. These calorific values are higher than the feedstock (4160.379 cal/gra

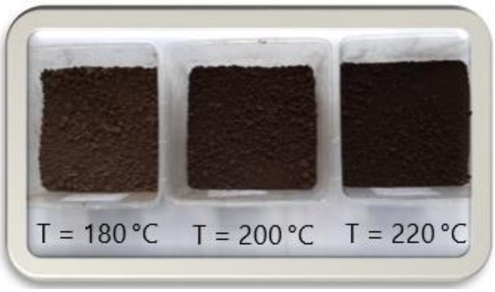

Fig. 6: Product appearance due to different final temperatures

This trend was similar to the hydrothermal carbonization of sea lettuce (Shrestha, Acharya, and Farooque 2021), woody biomass and coal mixture (Nonaka, Hirajima, and Sasaki 2011), and olive waste (Surup et al. 2020). This result confirmed the previous study on the influence of temperature on the energy density of co-hydrothermal carbonization of the coal-biomass blend (Saba, Saha, and Reza 2017).

Effect of holding time on the calorific value

Fig. 8 shows that longer holding time produced a higher hydro-char calorific value. The calorific values of the corresponding holding times are 5145.301 cal/gram for 30 min, 5662.191 cal/gram for 60 min, and 5690.007 cal/gram for 90 min. The trend observed for the calorific value was similar to the previous study on hydrothermal carbonization of sea lettuce, where the increasing residence time increased the calorific value (Shrestha, Acharya, and Farooque 2021).

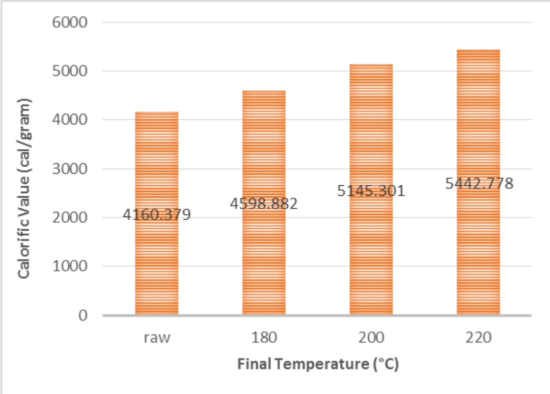

Fig. 7: Effect of final temperature on the calorific value

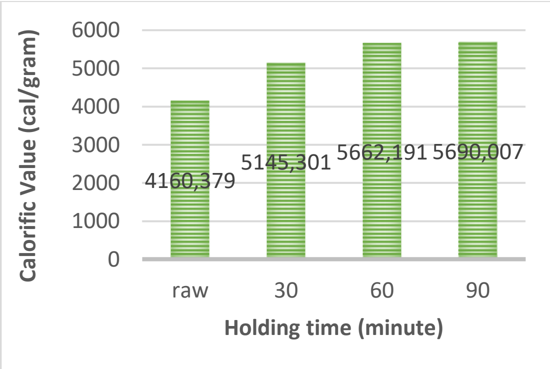

Fig. 8: Effect of holding time on the calorific value

Effect of solid-water ratio on the calorific value

Fig. 9 shows that the calorific value of hydro-char produced from the hydrothermal treatment at the same temperature of 200 °C increased with the increasing solid-water ratio. The hdyro-char calorific values with the corresponding solid water ratios were

5841.435 cal/gram at 1/3, 5191.558 cal/gram at 1/4, and 5145.301 cal/gram at 1/5 of solid-water ratio.

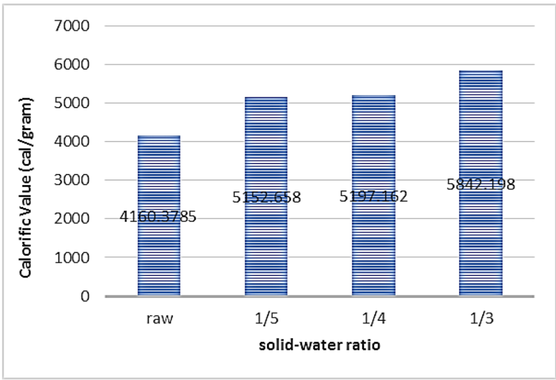

Fig. 9: Effect of solid-water ratio on the calorific value

Effect of final temperature on the proximate analysis

Fig. 10 shows the reduction of moisture content with increasing final temperature. The feedstock's moisture content of 10.057 % reduced to 4.335 % for 180 °C, 4.189 % for 200 °C, and 3.293 % for 220 °C of final temperature.

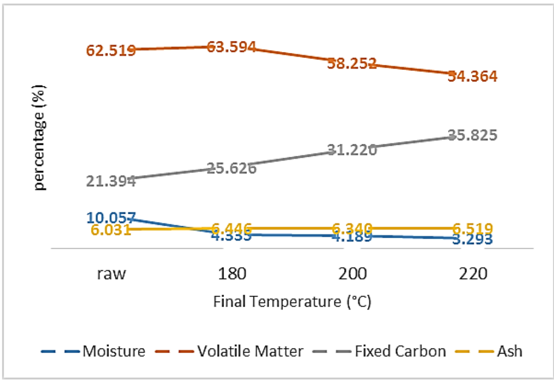

Fig. 10: Effect of final temperature on the proximate analysis

This trend was also experienced by the volatile matter content, which tended to decrease from 63.594% at 180°C to 58.252% at 200°C, 54.364% at 220°C. Meanwhile, a rise in fixed carbon was observed from the raw material with 21.394 % to 25.626% at 180°C, 31.220% at 200°C, and 35.825% at 220°C. This finding agrees with the work by Merzari and coworkers (Merzari et al. 2018) that studied the hydrothermal treatment on the municipal solid waste and agave pulp. The reduction of volatile matter and the increased fixed carbon were due to dehydration and decarboxylation reactions that took place during hydrothermal carbonisation (Funke and Ziegler 2010). Previous studies found that the calorific value was strongly affected by increased carbon content (Thipkhunthod et al. 2005; Sheng and Azevedo 2005) and the ratio of fixed carbon to volatile matter (Park, Lee, and Kim 2018).

There was an insignificant change in the ash content with the increasing final temperature. The ash content of 6.031% of the raw material changed to be 6.446% at 180°C, 6.340% at 200°C, and 6.519% at 220°C.

Effect of holding time on the proximate analysis

The reduction of moisture content with increasing holding time can be seen in Fig. 11. The feedstock's moisture content of 10.057% reduced to 4.189% at 30 min, 3.045% at 60 min, and 2.434% at 60 min of holding time.

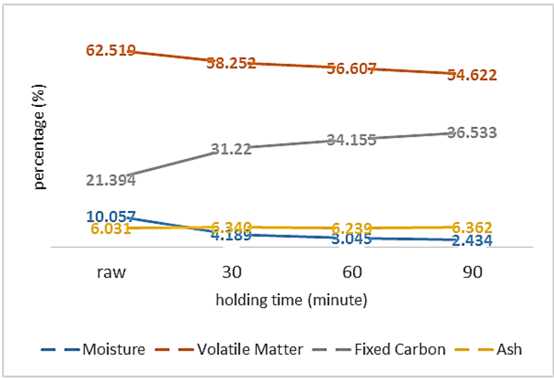

Fig. 11: Effect of holding time on the proximate analysis

This trend was also undergone by the volatile matter content, which tended to decrease from the raw material of 62.519% to be 58.252% at 30 min, 56.607% at 60 min, and 54.622% at 90 min. Meanwhile, there was a rise in fixed carbon from the feedstock from 21.394 % to 31.220% at 30 min, 34.155% at 60 min, and 36.533% at 90 min of holding time. This result agrees with the previous work conducted on hydrothermal treatment of refined sugar and grape seeds (Fiori et al. 2014).

However, there was an insignificant change in the ash content during the increasing holding time. The ash content of 6.031% of the feedstock slightly changed to 6.340% at 30 min, 6.239% at 60 min, and 6.362% at 90 min of holding time.

Effect of solid-water ratio on the proximate analysis

As shown by Fig. 12, there was a reduction of moisture content with an increasing solid-water ratio. The raw material's moisture content of 10.057 % reduced to 4.189% at 1/5, 4.109% at 1/4, and 3.708% at 1/3 of solid-to-water ratio.

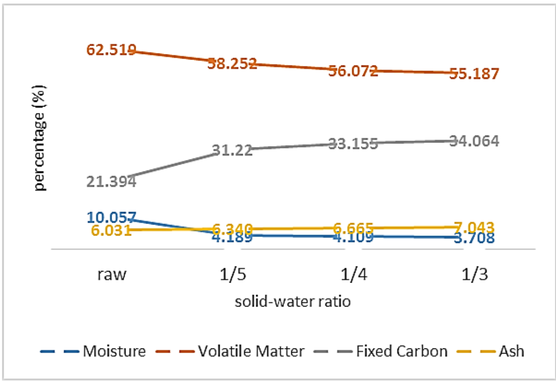

Fig. 12: Effect of solid-water ratio on the proximate analysis

This trend was also experienced by the volatile matter content, which tended to decrease from the feedstock (62,519%) to 58.252% at 1/5, 56.072% at 1/4, and 55.187% at 1/3 of solid-water ratio.

Meanwhile, there was an increase in fixed carbon from the feedstock (21.394%) to 31.22% at 1/5, 33.155% at 1/4, and 34.064% at 1/3 of solid-water ratio.

There was also a rise in the ash content during the increasing solid-water ratio. The ash content of 6.031% in the feedstock increased to 6.340% at 1/5, 6.665% at 1/4, and 7.043% at 1/3 of solid-water ratio.

Solid fuel quality was highly affected by the moisture content. The lower moisture content resulted in a better quality of fuel: thermal performance and gas emission (Huangfu et al. 2014; He, Lau, and Sokhansanj 2019). Besides, the dewatering performance of hydro-char can be improved by a hydrothermal reaction (Park, Lee, and Kim 2018). The capillary forces between the cells or flocks of material particles hold interstitial water, while bound water is normally contained within the particles. The bound water became free water when the flocks were splitted up, and it can be easily segregated (Meng et al. 2012). By hydrothermal treatment, the material's physical structure was broken down, so that the bound water converted to free water within the material.

CONCLUSIONS

Based on the results and analysis, we concluded that the holding time, final temperature, and solid-water ratio of the hydrothermal treatment significantly affected the yield, calorific value, and proximate analysis of the hydro-char::

a. The hydro-char yield decreased with the increase in final temperature and holding time. Meanwhile, the highest hydro-char yield was obtained at the solid-water ratio of ¼.
b. The hydro-char calorific value increased with the final temperature, holding time, and solid-water ratio.
c. The moisture content and volatile matter decreased but the fixed carbon increased with the rise in final temperature, holding time, and solid-water ratio. Meanwhile, the ash content was clearly increased in the rise of the solid-water ratio.

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