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Chemical and Combustion Characteristics of Hydrochars Obtained from Various Biomasses by Hydrothermal Carbonization

Tuğrul YUMAK[1*], Ali SINAĞ[2]

Highlights:

- Waste biomasses were subjected to hydrothermal carbonization.
- Hydrochars have higher HHV compared to lignite and raw biomass.
- Hydrochars can be assessed as an effective and environmentally friendly solid fuels.

Keywords:

- Biomass
- Hydrochar
- Hydrothermal Carbonization
- Combustion Properties
- Solid Fuels

ABSTRACT:

In this work, various waste biomasses were subjected to hydrothermal carbonization under mild conditions. The effect of operating temperature, duration time, and biomass-to-water ratio on the chemical and combustion characteristics of the hydrochars were determined. The results were compared to MKP Alpagut lignite to understand the future of hydrochars as an effective and environmentally friendly solid fuel. It was found that the temperature dominantly affects the yield and the chemical characteristics of the hydrochars compared to the duration time and biomass-to-water ratio for real biomasses. Hydrochars obtained from real biomasses showed higher combustion temperatures and slightly higher ignition temperatures. Moreover, the HHV of hydrochar is significantly higher than the MKP lignite and raw biomass. Our results prove that the hydrothermal carbonization process can be assessed as one of the best techniques for the thermochemical conversion of waste biomass into high-value-added valuable solid fuels.

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INTRODUCTION

Hydrothermal carbonization (HTC) is a type of thermochemical conversion processed under mild conditions compared to torrefaction, pyrolysis, or gasification for the treatment/pretreatment of high moisture-content biomass (Heidari et al., 2019). Water-submerged biomass is subjected to thermal treatment at a temperature range of 180–260°C in a closed system usually under an autogenous pressure (2-6 MPa) in HTC (Libra et al., 2011; Hoekman et al., 2012). After decades by the first report of HTC by Bergius in 1913, the process has gained much more attention not only for producing solid fuels or coal-like materials but also for synthesizing important chemicals and recovering liquid and gaseous fuels (Sinag et al., 2003; Kruse et al., 2013; Tekin et al., 2014; Kambo & Dutta, 2015). The exothermic process in HTC lowers the oxygen and hydrogen content via hydrolysis, dehydration, decarboxylation, polymerization, poly-condensation, and aromatization mechanisms (Funke & Ziegler, 2010; Pauline & Joseph, 2020). Besides owning the advantages of environmentally friendly, mild operating conditions, and easy controllable operating parameters, its ability to convert the wet feedstock at relatively high yields without prior dewatering and drying make HTC the most promising technique to produce hydrochar or bio-coal (Sharma et al., 2020). Hydrochar from waste biomass has significant advantages over fossil fuels (coal or lignite) such as being sustainable, carbon neutral and unlimited supply of waste carbon resources (Nizamuddin et al., 2017). Due to the high carbon content, high energy density, low fiber structure, and higher heating value (HHV), hydrochar has a high potential to be used as a solid fuel instead of coal. However, it should be noted that some other parameters such as ignition temperature, combustion temperature, and particle size are quite important from the point of fuel view.

Recent works focused on the effect of experimental parameters on the physicochemical and combustion parameters of hydrochar derived from such waste biomass, animal waste, agricultural wastes, and household wastes. Liang et al. have compared the combustion characteristics of forest waste-derived hydrochar and raw feedstock (Liang et al., 2022). They found that the HHV of the hydrochar obtained at 280°C is 1.46 times that of the raw material (Liang et al., 2022). Also, it is reported that an increase in the temperature led to an increase in the yield of gaseous and liquid products resulting in the decrease in the mass yield of hydrochar (Liang et al., 2022). Assis and Chirwa reported the fuel characteristics and combustion performance of hydrochars obtained from recycling paper mill wastes (Assis & Chirwa, 2023). They concluded that the increasing temperatures promoted dehydration and decarboxylation mechanism, leading to hydrochars with reduced H/C and O/C ratios, reducing the burnout temperature, thereby improving overall fuel properties (Assis & Chirwa, 2023). Rather than the temperature, Brachi et al. focused on the influence of other experimental parameters on the mass yields of solid, liquid, and gaseous products obtained from HTC and found that the water-to-feedstock ratio has a significant effect on the distribution between solid and liquid fractions (Brachi et al., 2017). In terms of the economy of the HTC process, reports indicate that hydrochar can replace conventional coal (Saari et al., 2016; Mazumder et al., 2022). It appears feasible to aim for a unit price of hydrochar that achieves a positive economic balance (breakeven selling price), enabling comparison with conventional coal prices (González-Arias et al., 2022).

In this work, the thermal and chemical properties of hydrochar obtained from waste biomasses via hydrothermal carbonization under mild conditions were investigated. Hazelnut shell and olive oil pomace were selected as real waste biomasses due to their annually huge waste potential as nearly 133000 t and 670000 t, respectively. The effect of the experimental parameters such as operating temperature, biomass-to-water ratio, and duration time, were discussed on the yield, thermal and chemical properties of the obtained hydrochar. In addition, the obtained results were compared to Bursa-Mustafakemalpaşa Alpagut (MKP) lignite. As a result, it is determined that the hydroochar obtained from olive oil pomace and hazelnut shells had a higher upper calorific value and lower sulfur and mineral content than MKP lignite. Thus, it has been revealed that hazelnut shells and olive oil pomace can be used as environmentally friendly solid fuel sources.

MATERIALS AND METHODS

Hazelnut shell (procured from Gürsoy Fındık, Ordu, Türkiye) (HS) and olive oil pomace (supplied from local producers in Muğla, Türkiye) (OP) were used as received without any processing except powdering.

The hydrothermal carbonization experiments were conducted in an SS316 stainless steel pressure reactor with an inner volume of 75 mL under different reaction conditions. The proper amount of biomass was dispersed in 40 mL of distilled water in each experiment and transferred into the reactor. The reactor was heated to the desired temperature and held at different reaction times under stirring. After the process, the hydrochar samples were collected by vacuum filtration, washed with distilled water, and dried in an oven at 80°C overnight. The samples were labeled as BiomassTemperature-Duration Time-Biomass-to-Water Ratio. For example, the label HS200-6-1:20 refers to the hydrochar sample obtained from hazelnut shell at 200°C for a duration of 6 hours and with a biomass-to-water ratio of 1:20.

Temperature (150°C -175°C -200°C), duration time (2-4-6 hours), and biomass-to-water ratio (1:10, 1:20, 1:50) as primary variables (Román et al., 2012) were examined on hydrochar yield. The effect of primary variables on the combustion characteristics were also investigated as well, and compared to MKP lignite.

The thermal characteristics of the hydrochars were investigated by TG-DT analysis (Setaram Labsys) at a temperature range of 25-800°C with a heating rate of 20°C/min under airflow. The real HHV of the hydrochars was obtained by a bomb calorimeter. The elemental composition (C, H, N, S, and O content) was determined by LECO, CHNS-932 elemental analyzer and the results were used to calculate the theoretical HHV by using the formula given somewhere else (Mahinpey et al., 2009).

RESULTS AND DISCUSSION

Effect of Primary Variables on the Hydrochar Yield

The solid product obtained by HTC can be classified into three groups: primary char, secondary char, and carbon nanoparticles (Titirici & Antonietti, 2009). Primary char refers to the solid phase deriving from the solid–solid conversion of biomass, while secondary char is the fraction forming via the condensation and polymerization of the dissolved organics in the liquid phase derived from the primary biomass (Nicolae et al., 2020). Regardless of this classification, the term “solid product” is used since the investigation of the thermal and combustion characteristics of the resultant hydrochars is aimed in this work.

Fig. 1 represents the change in the mass yield of synthesized hydrochars at 6 hours at a biomass-to-water ratio of 1:10 with the change in the temperature. The increase in the opreating temperature led to a slight decrease in the mass yield of water-insoluble biomass-derived hydrochars.

The decrease in the mass yield is less sharp compared to cellulose (X. Lu et al., 2013; L. Wang et al., 2019) at the given temperature range. This is attributed to the presence of lignin, which may stabilize the cellulose, thus preventing the disruption of the crystalline structure at lower temperatures (Falco et al., 2011). In addition, the hemicellulose with the lowest thermal stability present in the real biomass can be responsible for the lower mass yield compared to cellulose at lower temperatures. Since the total hemicellulose and organics content of olive oil pomace (%41.8) is higher than the hazelnut shell (%25.6), lower mass yields of hydrochar resulting from the faster decomposition of hemicellulose for olive oil pomace are as expected in Fig.1. As a result, it is obvious that the temperature has a significant effect on the mass yield of solid product obtained by HTC of biomasses.

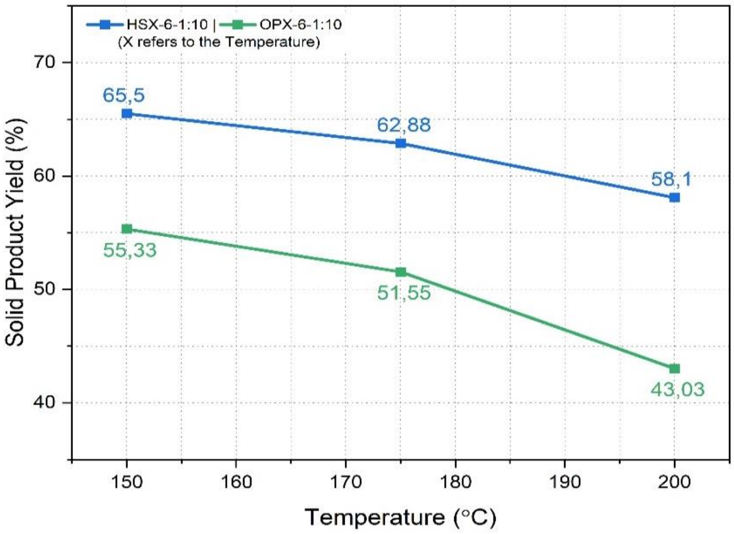

Figure 1. The Effect of Temperature on the Mass Yield of Hydrochars

The effect of duration time on the mass yield of hydrochars at 200°C at a biomass-to-water ratio of 1:10 can be seen Fig. 2. For the olive oil pomace derived hydrochars, the mass yield decreased with the increasing duration time. This may be a result of the increasing degradation rate of the raw biomass due to the increased heat transfer. It is also thought that the parameters selected in this work seems to be insufficient for the formation of secondary char formation. The mass yield change trend for hazelnut shell-derived samples closely mirrors that of glucose (Titirici et al. 2008). For the real biomass derived hydrohars, the data obtained at different temperatures reveal interesting results (Yumak, 2016). For this reason, again, it is seen that the temperature has dominantly affected the solid mass yield. Our results are in good match with the early reported data (X. Lu et al., 2013).

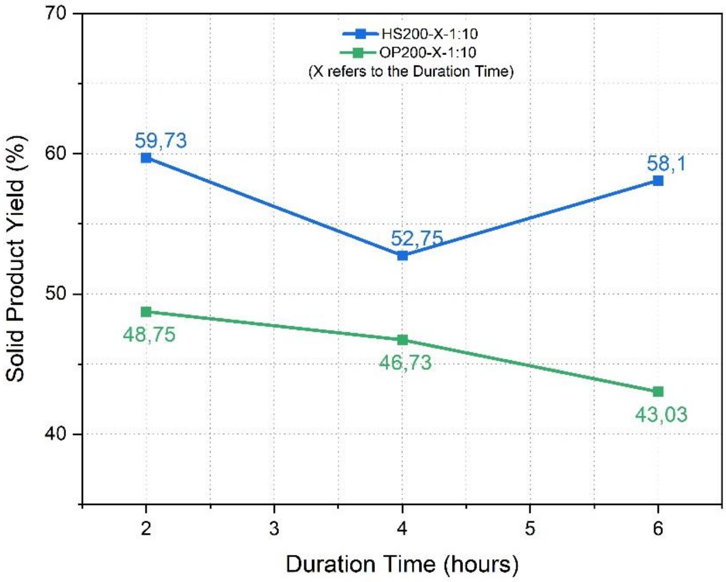

Figure 2. The Effect of Duration Time on the Mass Yield of Hydrochars

The impact of biomass-to-water ratio on the mass yield of hydrochars is depicted in Figure 3. Under the specified parameters, a decrease in mass yield was noted for the HTC of olive oil pomace. Conversely, heterogeneous changes were observed in hazelnut Shell samples. First, there's an increase step, followed by a decrease step, or vice versa trend was observed. (Yumak, 2016). Therefore, it can be said that the temperature, similar to the duration time, dominated the effect of biomass-to-water ratio.

Under the chosen experimental conditions, temperature stands as the sole independent parameter. However, the impact of duration time and biomass-to-water ratio on the resulting mass yield is significantly contingent upon temperature.

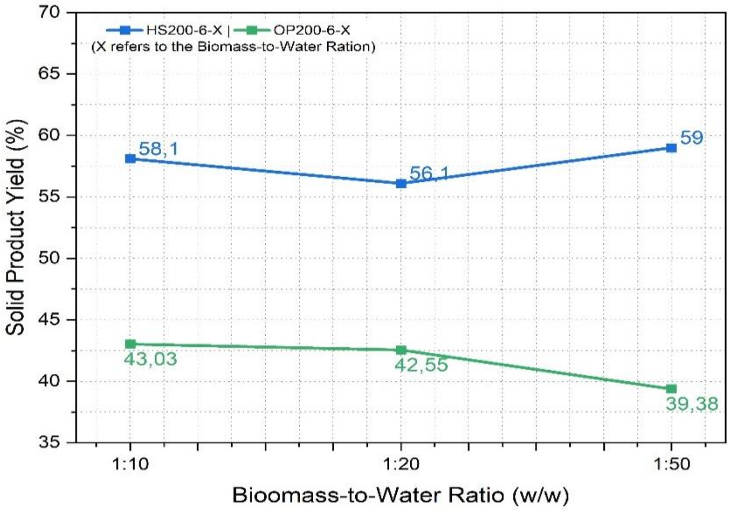

Figure 3. The Effect of Biomass-to-Water Ratio on the Mass Yield of Hydrochars

Elemental composition and HHV of hydrochars

The ultimate analysis, calculated HHV and measured HHV of the raw materials, synthesized hydrochars, and MKP lignite are given in Table 1. HHV which is one of the most important parameters for the comparison of solid fuels were calculated by Eq.1 (Mahinpey et al., 2009) and determined by a bomb calorimeter in kJ/kg.

$$HHV(kJ/kg) = 1.87C^2 - 1.44C - 2802H + 63.8CH + 129N + 20147 \quad (1)$$

It is seen that the carbon and hydrogen contents of hydrochars are higher than both MKP lignite and the relevant raw material, while the oxygen content is less. For this reason, the calculated HHV of hydrochars are higher than MKP lignite and raw materials, as expected. No sulfur was detected in the hydrochars obtained from hazelnut shells and olive oil pomace; however, a trace amount of sulfur was found by XRF analysis (data not shown here) which is more sensitive than elemental analysis. More detailed data can be found elsewhere (Yumak, 2016). The sulfur content of hydrochars obtained from real biomass is considerably lower than MKP lignite. This reveals that biochars are much more environmentally friendly than lignite.

In addition, the increase in the temperature causes an increase in the carbon and hydrogen content and a decrease in the oxygen content resulting in an increase in the calculated HHV. However, it seems that the duration time and biomass/water ratio do not create a significant change in element content. It was determined that increasing the amount of biomass used did not have a significant effect on the carbon and hydrogen content but caused a decrease in the oxygen content.

Moreover, the measured HHV of hydrochars obtained from real biomass was significantly higher than the calculated HHV. This can be explained by the fact that the structure of the real biomass is disrupted with the HTC process, the percentage of carbon content increases through dehydration and decarboxylation steps, and a more homogeneous structure occurs. At the same time, the increase in the amount of fixed carbon with the HTC process indicates that an increase in the measured HHV is expected. As the temperature increases, the measured HHV of hydrochars obtained from hazelnut shells and olive oil pomace increases. The increase in hydrochars obtained from hazelnut shells is higher than those of olive oil pomace that can be attributed to carbon content of biochars. The measured HHV of hydrochars is also considerably higher than MKP lignite. This is attributed to the lower moisture and ash content, higher carbon, and hydrogen of hydrochars compared to MKP lignite.

Table 1. Ultimate Analysis (% content of C, H, N, S, and O), and the Heating Values of the Hydrochars

Sample	**C**	**H**	**N**	**S**	**O^***	**HHV^c**	**HHV^m**
MKP lignite	42.96	3.00	1.13	2.70	11.92	17374.27	15850
Hazelnut Shell	49.96	6.13	1.64	0.06	42.21	20194.64	19220
HS200-4-1:20	54.67	5.56	0.86	0.00	38.91	21788.39	27490
HS175-4-1:20	54.22	5.89	1.01	0.00	38.88	21838.17	nd
HS150-4-1:20	52.29	5.26	1.12	0.00	41.33	20684.13	24120
HS200-6-1:20	55.38	5.19	1.00	0.00	38.43	21831.62	nd
HS200-2-1:20	55.03	5.31	1.05	0.00	38.61	21785.39	24882
HS200-6-1:50	54.69	5.14	0.88	0.00	39.29	21510.64	nd
Olive oil pomace	46.02	4.96	1.78	0.07	47.17	18375.12	19800
OP200-6-1:10	56.29	5.68	1.75	0.00	36.28	22675.44	23961
OP175-6-1:10	54.00	6.14	1.63	0.00	38.23	21983.44	23613
OP150-6-1:10	50.24	6.04	1.65	0.00	42.07	20281.28	nd
OP200-2-1:10	54.80	5.89	1.60	0.00	37.71	22166.96	23582
OP200-6-1:50	51.41	5.77	1.18	0.00	41.64	20596.39	nd

O^*: Oxygen content is calculated from the difference; HHV^c: Calculated HHV from the elemental analysis results; HHV^m: Measured by bomb calorimeter; nd: Not determined

Proximate analysis and combustion characteristics of hydrochars

The proximate analysis, and combustion characteristics of hydrochars are presented in Table 2. The combustion characteristics of the hydrochars are determined by Thermogravimetric analysis (TGA) data as explained somewhere else (C. Wang et al., 2009; J. J. Lu & Chen, 2015).

For the hydrochars obtained from hazelnut shells, the moisture content is higher compared to hazelnut shells themselves. However, as the temperature increases, the moisture content of the resulting hydrochars significantly decreases, reaching a lower value compared to MKP lignite. Similarly, an increase in the temperature leads to an increase in volatile matter content and a decrease in fixed carbon content. Additionally, the temperature increase also results in an increase in ignition temperature. As the amount of biomass used decreases, the moisture content, volatile matter content, and ignition temperature of the resulting hydrochar decrease, while the fixed carbon content and combustion temperature increase. The olive oil pomace derived hydrochars have significantly lower moisture content compared to both MKP lignite and raw olive oil pomace itself. An increase in the temperature did not show a consistent effect on volatile and fixed carbon content, the hydrochars obtained at 175°C exhibit the highest fixed carbon content and combustion temperature. It is observed that an increase in duration time led to an increase in moisture content, and a decrease in fixed carbon content and ignition temperature.

Having slower reaction times between gas and solid phases compared to gas-gas phases, high content of fixed carbon is preferred for achieving a longer-lasting combustion. From the Table 2, it is seen that the HTC process for waste biomasses results in a significant increase in the fixed carbon content. Also, considering the high ash content (~15-20%) of MKP lignite and the considerably low ash content of hydrochars, it is obvious that the hydrochars represent much better solid fuel properties. The ignition temperature, defined as the temperature necessary to provide the activation energy for combustion, is proximate to MKP lignite for hydrochars derived from hazelnut shells, surpassing MKP lignite for hydrochars derived from olive oil pomace. A low ignition temperature is preferred for easier combustion from the perspective of application and combustion process design. A low ignition temperature indicates that solid fuels are more active.

Table 2. Proximate Analysis and Combustion Characteristics of Hydrochars

Sample	Moisture content %	Volatile matter%	Ash + Fixed Carbon %	Ignition Temperature (°C)	Combustion Temperature (°C)
MKP lignite	8.16	24.05	67.79	256	509+
Hazelnut shell	5.06	68.46	26.48	245	514
HS200-2-1:20	7.89	59.47	51.58	257	480
HS200-4-1:20	7.33	32.67	60.00	271	503
HS175-4-1:20	8.35	29.95	62.70	264	628
HS150-4-1:20	8.82	24.45	66.73	255	571
HS200-6-1:20	8.47	31.14	60.39	267	541
HS200-6-1:50	6.31	26.49	67.20	234	550
Olive oil pomace	9.22	45.3	45.48	238	626
OP200-2-1:10	4.64	34.28	61.08	286	381+
OP200-6-1:10	5.66	41.48	52.86	261	554
OP175-6-1:10	5.41	34.09	60.50	290	570
OP150-6-1:10	6.25	41.93	51.82	267	532
OP200-6-1:50	4.14	30.68	65.18	278	368+

CONCLUSION

Hydrothermal carbonization was successfully applied to the waste biomasses. The effect of primary experimental variables such as temperature, duration time, and biomass/water ratio were discussed on the yield, chemical composition, and combustion characteristics of the obtained solid products as fuel. The results were compared to MKP Alpagut lignite to understand the future of hydrochars as an effective and environmentally friendly solid fuels. It is found that the process temperature dominantly affects the yield and the chemical characteristics of the hydrochars compared to the duration time and biomass-to-water ratio. Therefore, it is thought that the reaction steps are determined and controlled by the temperature for real biomasses. HTC process led to an increase in the carbon and hydrogen content resulting in higher HHV compared to MKP lignite. Also, lower ignition temperature and higher combustion temperature of hydrochars were found indicating that the HTC process is one of the best techniques for thermochemical conversion of waste biomass into high value added valuable solid fuels. It is considered that the synthesis of sulfur-free and high-calorific-value hydrochars from waste biomasses through HTC process will be an important application area for meeting future energy needs, especially for urban and industrial purposes. Therefore, future works should be focus on the economic feasibility of the HTC process.

Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author’s Contributions

The authors confirm contribution to the paper as follows: study conception and design: AS, TY; data collection: TY; analysis and interpretation of results: TY, AS; draft manuscript preparation: TY; supervision: AS. All authors reviewed the results and approved the final version of the manuscript.

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