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Research paper



Comparison of solid biofuels produced from olive pomace with two different conversion methods: torrefaction and hydrothermal carbonization

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Abstract

Olive pomace is a by-product of olive oil production process and has the potential to be a solid biofuel after thermal treatment. In this study, olive pomace was treated by two thermal conversion methods: hydrothermal carbonization (HTC) and torrefaction. Experiments were carried out under the temperature values of 250, 275, 300 and 350°C; reaction times of 10, 20 and 30 minutes for torrefaction and temperature values of 180, 200 and 220°C; reaction time of 2, 3 and 4 hours for HTC. Products with the same energy yield value (62 %) obtained the higher heating values of 23.73 and 25.20 MJ/kg for torrefaction (275°C for 20 minutes) and hydrothermal carbonization (220°C for 2 hours), respectively. Hydrothermal carbonization method has the potential to produce chars at lower temperature values and without a drying process; and obtain products with improved higher heating values, energy yields and atomic O/C and H/C ratios compare to torrefaction products.

Keywords: *Torrefaction, hydrothermal carbonization, olive pomace, fixed carbon, biomass*

1. Introduction

The world energy demand is mostly met by fossil fuels. According to International Energy Agency, the world share of energy sources is petroleum 37 %, natural gas 29 %, coal 15 %, renewable energies 10 % and nuclear energy 9 % for 2016. It is indicated in IEA 2016 scenario that fossil fuels' share will continue to be important in 2040. However, market prices for these fuels will rise with the reducing amount of reserves [1]. Renewable and sustainable resources are thought to be significant alternatives in this foreseen energy crisis. Among all the sustainable energies, biomass energy has the broad availability worldwide and this makes it an interesting option [2]. On the other hand, as an energy resource, biomass has its disadvantages such as structural heterogeneity, non-uniform physical properties, low energy density, hygroscopic nature, and low bulk density [3]. These characteristics prevent the use of biomass instead of fossil fuels. Consequently, biomass requires a pretreatment method before the thermochemical conversion. Hydrothermal carbonization (HTC) and torrefaction treatments are the two most useful methods with relatively low temperature requirements within all other thermochemical conversion processes [4].

HTC process occurs at temperature values between 180 – 260 °C and biomass is mixed with subcritical water so that the products are not affected by the high moisture content. Hydrothermal carbonization has the ability to deal with extremely wet biomass resources such as sewage sludge, paper mill and also forestry processing residues and agricultural wastes [5]. It is used to produce a solid fuel called hydrochar which has reduced O/C and H/C ratios, increased calorific value and enhanced hydrophobicity [6].

Torrefaction is a thermochemical pretreatment of biomass in the temperature range of 200 –300 °C, under the inert gas environment for residence time of minutes to couple of hours [7]. Advantages of torrefied biomass are decreased moisture content, enhanced resistance to water damage and microbial growth, and increased friability [8].

Olive oil extraction industry has an important share in economy of Mediterranean countries such as Turkey, Spain, Italy and Greece [9]. It is estimated that the world-wide production of olive oil was about 3 million tons for the harvest period of 2013–2014 [10]. The rapid increase in the amount of wastes generated in this process has caused serious environmental issues due to wastes' high organic matter content and its phytotoxicity [11]. Olive mills not only produce significant quantities of wastewater but solid wastes as well [12]. Olive pomace is found to have proper higher heating values. It is possible to remove its moisture and volatile matter content and obtain an energy-dense solid biofuel.

In this study, it is aimed to use hydrothermal carbonization and torrefaction methods to produce energy-dense solid biofuels from olive pomace and compare them by their elemental, proximate and calorific properties. Hydrothermal carbonization and torrefaction products are called as hydrochar and biochar, respectively. There

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were some studies [13, 14, 15, 16 and 17] carried out with olive pomace but none of them included both hydrothermal carbonization and torrefaction methods and their product comparison. The main objective of this study is to show fuel characteristics of olive pomace and it is potential to generate an alternative biofuel by these two methods.

2. Material and Methods

2.1. Material

Olive pomace was taken from Germencik, Aydın, Turkey. The material had 40 % of moisture content when it was arrived. It was dried in a hot-room (37 °C) until its moisture content was decreased less than 10 % for torrefaction reactions. Particle sizes of olive pomace were over 1 mm.

2.2. Hydrothermal Carbonization Experiments

Hydrothermal carbonization experiments were generated in a 100 ml stainless steel reactor (Amar Equipment Pvt. Ltd.). Biomass and deionized water mixture were loaded in the reactor as 10 % solid/liquid ratio. The reactor was heated with an electric heater to desired temperature (180, 200 and 220 °C) and held for desired reaction time (2, 3 and 4 hours). Inside the reactor, increasing temperature was created a pressure value between 8 to 10 bars. After the reaction, the reactor was cooled to room temperature by the automatic cooler. Remaining products, hydrochars, were dried at 104 °C for 24 hours and kept for further analyses.

2.2. Torrefaction Experiments

Torrefaction experiments were carried out in a pilot scale continuous screw type torrefaction reactor with 5 kg/h feeding capacity in Solar Energy Institute of Ege University, Turkey. The torrefaction system consists four screw units: the first screw is the biomass feeder, the second is the biomass dryer and pre-heater, the third is the torrefaction reactor and the forth is the cooler. First, the dryer (around 120 °C) and torrefaction (250, 275, 300 and 350 °C) reactors were heated to the desired temperatures. Then, the biomass feed screw was started. All experiments were conducted in an inert atmosphere. Torrefaction reaction times were determined to be 10, 20 and 30 minutes for each temperature degree. After they were cooled in the system, biochars were collected. Obtained biochars were kept for further analyses.

2.2. Product Characterization

Each analysis was repeated for three times. Biomass, biochars and hydrochars were analyzed for ash, volatile matter contents by respectively ASTM D 1102-84 and ASTM E 897-82 standards. Fixed carbon content was calculated from ash and volatile matter results. The carbon (C), hydrogen (H) and nitrogen (N) contents were measure with Leco Tru Spec (USA) by ASTM-D5373 standards. Oxygen contents were calculated from the equation. Sulfur (S) contents were also measured by ASTM D-4239 standards. The higher heating values of the biomass and products were calculated with bomb calorimeter (Parr Instrument Co., Model 6300, Moline, IL, USA).

3. Results and Discussion

3.1. Fuel Characteristics of Chars

Proximate analyses results of the products and raw material are given in Table 1. The ash, volatile matter and fixed carbon values of biochars were observed to change dramatically with the increase of temperature between 250 and 350 °C for torrefaction reactions. As the temperature increased, the amount of volatile matter decreased and fixed carbon and ash contents increased.

Table 1: Proximate Analyses Results of Bioproducts

	Parameters	Ash (%)	Volatile Matter (%)	Fixed Carbon (%)
Raw		1.61	94.11	4.28
Torrefaction	**250 °C 10 min**	2.35	91.64	6.01
	275 °C 10 min	3.63	74.74	21.63
	300 °C 10 min	6.83	48.66	44.51
	350 °C 10 min	14.27	34.36	51.37
	250 °C 20 min	3.89	69.50	26.61
	275 °C 20 min	4.42	67.46	28.12
	300 °C 20 min	13.50	44.17	42.33
	350 °C 20 min	14.68	34.39	50.93
	250 °C 30 min	3.14	66.48	30.38
	275 °C 30 min	5.74	56.61	37.65
	300 °C 30 min	19.34	38.98	41.68
	350 °C 30 min	20.46	33.19	46.35
Hyrdrothermal Carbonization	**180 °C 2 h**	0.50	79.75	19.75
	180 °C 3 h	0.64	78.35	21.01
	180 °C 4 h	0.51	77.27	22.22
	200 °C 2 h	0.81	74.88	24.30
	200 °C 3 h	0.37	74.88	24.75
	200 °C 4 h	0.38	74.38	25.24
	220 °C 2 h	1.25	67.90	30.86
	220 °C 3 h	0.84	62.32	36.84
	220 °C 4 h	0.69	61.81	37.50

The HTC process provided lower ash contents. However, a consistent decrease with temperature or reaction time wasn't observed. Similar decreases for the HTC of grape pomace and mischantus samples were also observed in other studies [18, 19]. In this study, ash contents of hydrochars slightly changed (Figure 1).

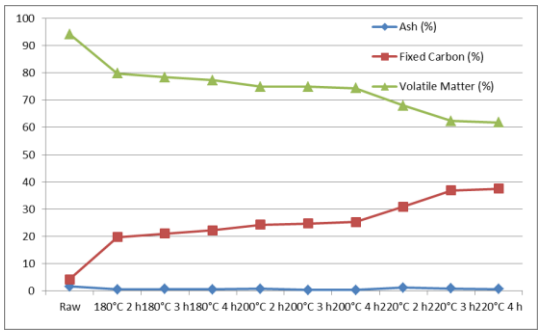


Fig.1: Ash, Fixed Carbon and Volatile Matter Contents of Hydrochar

On the other hand, biochars' ash contents highly increased as in literature for olive pomace and grape pomace [15, 18]. The resolution of inorganic matters in subcritical water is the reason for these different results for ash contents. In hydrothermal carbonization process, subcritical water provides the removal of inorganic materials from the biomass and the ash content of hydrochar decreases [20].

On the contrary, inorganic matter content increased during the torrefaction process (Figure 2). Hydrothermal carbonization products have the advantage of low ash content to prevent fouling and slagging in processes such as combustion, gasification and pyrolysis [20, 21 and 22]. The results of proximate analyses generally showed the characteristic similarities between converted products and coal. In this study, it is observed that volatile matter contents decreased and fixed carbon contents increased for both methods. Volatile matter and fixed carbon contents were seemed to be compatible with other studies [17]. These processes are known to be efficient on reducing the volatile matter content of biomass. The volatile matter and fixed carbon contents of HTC processes (220 °C 3 h and 4 h) and torrefaction process (275 °C, 30 min) were found to be similar. It is known that high volatile matter content of biomass causes low combustion performance and release of environmentally hazardous substances [23]. The difference between volatile matter contents of biomass and coal brings on separated combustion zones [20].

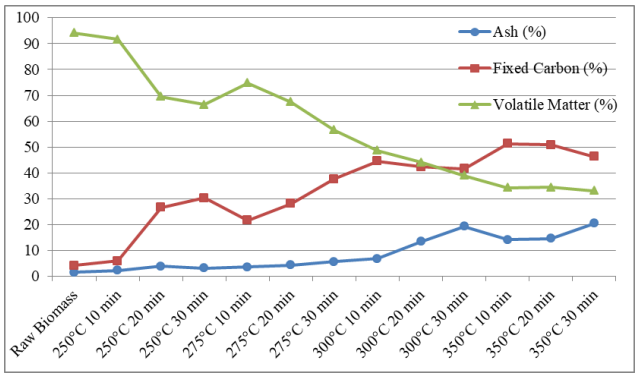


Fig.2: Ash, Fixed Carbon and Volatile Matter Contents of Biochar

Elemental analyses results of hydrochar, biochar and raw biomass are shown in Table 2. The higher carbon and lower oxygen contents of converted products provided enhanced higher heating values. In HTC processes, higher carbon contents were obtained under the elevated temperature conditions. However, the change of reaction time had only a slight effect on carbon contents. On the other hand, for the torrefaction processes, carbon contents rose with the increase of both temperature and reaction time.

The increase on the temperature, the decrease on oxygen content and a slight change on hydrogen content indicates the carbonization for both methods. Biochar of 300 °C, 30 min and hydrochar of 220 °C, 4 h provided approximate values of carbon contents, respectively 63.52 % and 63.01 %. It is clearly seen that the effect of carbonization on hydrochar under the lower temperatures was more significant.

The reaction times of HTC processes didn't have an important effect on carbon content of olive pomace. In literature, it was studied with olive pomace on 180, 200 and 220 °C for 30 minutes and observed carbon contents increased from 50.3 % to 55.3, 58.1 and 60 % [17]. These results didn't differ from the results for this study, so it is possible to recommend lower reaction times for the HTC process of olive pomace.

Table 2: Elemental Analyses Results of Bioproducts

	Parameters	C(%)	H (%)	N (%)	S (%)	O (%)
Raw		49.61	6.40	0.58	0.05	41.76
Torrefaction	**250 °C 10 min**	49.93	6.44	0.70	0.05	40.54
	275 °C 10 min	49.86	6.33	0.68	0.05	39.45
	300 °C 10 min	58.42	4.68	0.91	0.03	29.14
	350 °C 10 min	61.34	5.57	1.10	0.04	17.68
	250 °C 20 min	53.66	6.06	0.86	0.03	35.50
	275 °C 20 min	54.95	6.08	1.11	0.05	33.40
	300 °C 20 min	59.28	4.77	1.26	0.02	21.17
	350 °C 20 min	63.18	5.26	1.15	0.02	15.70
	250 °C 30 min	54.28	5.80	0.95	0.03	35.80
	275 °C 30 min	59.30	5.81	1.21	0.03	27.92
	300 °C 30 min	63.52	5.00	1.04	0.03	11.07
	350 °C 30 min	68.16	4.70	1.26	0.03	5.39
Hyrdrothermal Carbonization	**180 °C 2 h**	54.28	6.69	1.15	0.03	37.35
	180 °C 3 h	53.36	6.49	0.82	0.03	38.67
	180 °C 4 h	54.46	6.67	1.04	0.05	37.27
	200 °C 2 h	56.13	6.69	1.05	0.06	35.25
	200 °C 3 h	56.07	6.51	0.96	0.05	36.04
	200 °C 4 h	57.01	6.55	1.00	0.05	35.01
	220 °C 2 h	59.82	6.54	1.16	0.05	31.18
	220 °C 3 h	62.37	6.39	1.19	0.07	29.14
	220 °C 4 h	63.01	6.42	1.15	0.06	28.67

3.2. Van Krevelen Graphs

Atomic H/C and O/C ratios of biochar and hydrochar were shown on van Krevelen graphs to observe the changes on atomic content of olive pomace (Figure 3 and 4, respectively). On HTC processes, atomic O/C and H/C ratios clearly reduced with the increase on the temperature and a coal-like product was generated.

A linear correlation can be observed between the hydrogen/carbon and oxygen/carbon ratios as in other studies [24, 25]. Torrefaction processes which occured at lower temperatures (250 and 275 °C) had higher H/C and O/C ratios than hydrochar and the results were approximate to raw biomass's ratios.

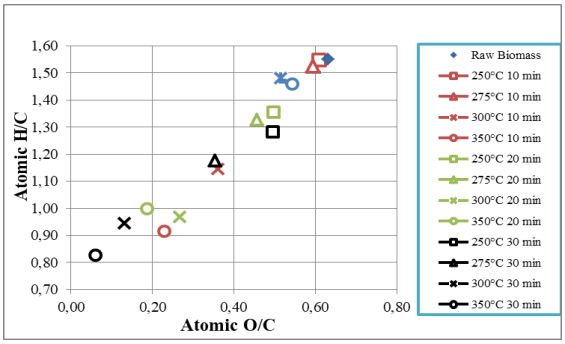


Fig.3: Atomic O/C and H/C ratios of biochar

Hydrochars of 220 °C, 3 h and 4 h had H/C ratios of 1.23 and 1.22; O/C ratios of 0.35 and 0.34. Biochar of 275 °C 30 min also had H/C ratio of 1.17 and O/C 0.35; H/C ratio of 1.09 and O/C ratio of 0.30 for 300 °C 10 min and H/C ratio of 0.97 and O/C ratio of 0.27 for 300 °C 20 min. Lignite has the atomic H/C and O/C ratios between 0.8 - 1.3 and 0.2 - 0.38 which shows that these products are comparable with lignite as a solid fuel. Lower H/C and O/C

ratios indicate the water vapor of biomass had removed and smaller amounts of smoke will be observed during combustion [8].

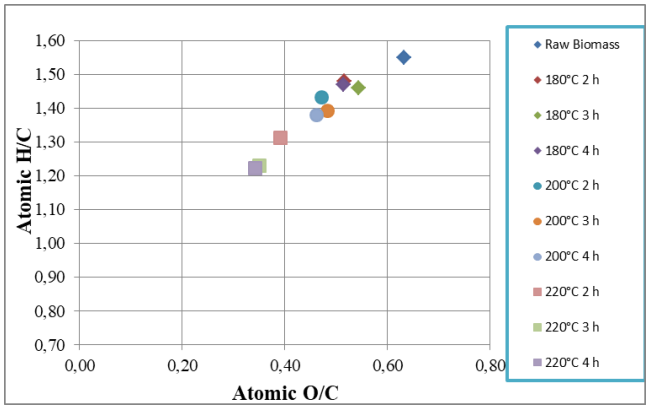


Fig.4: Atomic O/C and H/C ratios of hydrochar

3.3. Higher Heating Value, Energy and Mass Yields

In this study, biochar yields were observed 88.87 % and 40.02 % for temperatures of 250 °C and 300 °C (Table 3). The dramatic decrease of biochar yield with the increasing temperature is due to the decomposition of cellulose at 300 °C [26, 27]. The mass yields of torrefaction reaction at 250 °C and 20 minutes and HTC reaction at 180 °C and 2 h were measured as respectively 68 % and 63 %. The main reactions occur in both processes were dehydration and decarboxylation. Additionally, during the HTC, extractable materials (both organics and inorganics) were separated simultaneously due to their high solubility ratios in subcritical water [28]. It is the reason for lower mass yields of hydrochars.

Energy yields were measured according to equation (1):

$$\text{Energy Yield} = \text{Mass Yield} \times [(\text{Product's HHV})/(\text{Raw Biomass' HHV})] \quad (1)$$

During HTC process, energy densities were increased from 1.09 to 1.32 between 180 – 220 °C (Table 3). On the other hand, energy yields decreased with the increasing temperature values. At 220 °C, the decrease mostly depended on reduced mass yields. It was studied on pine between 215 - 295 °C and energy yields were observed to increase from 70 % to % 77 [29]. On the other hand, in another study on coconut and eucalyptus leaves (temperatures between 200 – 300 °C), energy yields decreased respectively from 76.67 % to 65 % and from 87.34 % to 61.32 % [30]. These opposite results were because of the difference between the biomass resources. Biochars obtained at 250 °C 10 min had the energy yield of 88.87 %. However, it had a lower higher heating value than the hydrochar of 180 °C and 2 h. It is crucial for these processes to provide increased mass yields to obtain higher energy yields. Frankly, HTC process seems to generate higher energy density. Energy contents of hydrochars were between 22.14 and 26.94 MJ/kg.

Table 3: HHV, Mass Yield, Energy Yield and Energy Density Values

	Parameters	HHV (MJ/kg)	Mass Yield (%)	Energy Yield (%)	Energy Density
Raw		20,34			
Torrefaction	**250 °C 10 min**	20,58	87,85	88,87	1,01
	275 °C 10 min	22,68	74,24	82,79	1,12
	300 °C 10 min	23,08	35,29	40,04	1,13
	350 °C 10 min	28,64	29,30	41,25	1,41
	250 °C 20 min	20,78	68,99	70,49	1,02
	275 °C 20 min	23,73	53,42	62,32	1,17
	300 °C 20 min	25,33	34,37	42,80	1,25
	350 °C 20 min	29,41	29,85	43,16	1,45
	250 °C 30 min	20,78	37,73	49,62	1,32
	275 °C 30 min	23,73	34,77	49,11	1,41
	300 °C 30 min	25,33	28,29	40,24	1,42
	350 °C 30 min	29,41	29,24	42,41	1,45
HTC	**180 °C 2 h**	22,14	63,10	68,69	1,09
	180 °C 3 h	22,26	60,04	65,72	1,09
	180 °C 4 h	22,34	59,99	65,90	1,10
	200 °C 2 h	23,29	59,50	68,13	1,15
	200 °C 3 h	23,38	57,81	66,44	1,15
	200 °C 4 h	23,41	57,30	65,95	1,15
	220 °C 2 h	25,20	50,08	62,05	1,24
	220 °C 3 h	26,32	45,06	58,30	1,29
	220 °C 4 h	26,94	44,89	59,46	1,32

In another study with olive pomace, the higher heating values of 22.1, 23.3 and 24.3 MJ/kg from the reactions of respectively 180, 200 and 220 °C for 30 min were observed [17]. These results seemed compatible with this study. Additionally, higher heating values were obtained with higher reaction times (3 and 4 h).

As in literature, torrefaction of olive pomace at higher temperatures provided greater higher heating values. In a study on olive pomace (26.4 MJ/kg of HHV) at 200, 250 and 300 °C for 2 hours HHV results were obtained as respectively 26.5, 28.7 and 29.4 MJ/kg [15]. However, these results were measured as high as this because of the olive pomace used in that study consists 26 % of olive oil.

Guizani et al. (2016) carried out another study with olive pomace which contained similar energy amount as the olive pomace used for this study. HHV of 20.50, 21.9, 22.7, 23.7 and 25.8 MJ/kg were obtained by the torrefaction reactions at respectively 230, 250, 270, 290 and 310 °C for 1 hour [16]. These results showed similarity with the higher heating values obtained in this study.

The main aim of using these conversion methods was to increase the energy yields. However, it is crucial to evaluate the mass yields for economic reasons. Mass yields were reduced with the increasing temperature values. During HTC processes, above the 200 °C, mass yields remained stable around 60 %. At 300 and 350 °C, mass yields of torrefaction processes were measured between 29 – 35 %. These dramatic decreases on mass yields of torrefaction above 250 °C are similar for some other studies. It is

observed that obtaining higher energy contents at lower temperatures makes hydrochar obtained from HTC process a more desirable solid fuel than biochar.

4. Conclusions

Higher heating values of torrefaction at 275°C for 20 minutes and hydrothermal carbonization at 220°C for 2 hours were measured as respectively 23.73 and 25.20 MJ/kg for the same energy yield value of 62 %. It is concluded that HTC process provides an energy-dense solid biofuel from olive pomace under lower temperature values, without a pre-drying process. The ash content of hydrochars was observed to be decreased while the ash content of biochars increased. HTC process has the advantage to generate high quality solid biofuels with lower ash content. The lower ash amount of biofuel provides the higher combustion, gasification and pyrolysis performances.

According to the HTC results, the residence time didn't show a significant effect on proximate and elemental analysis results of hydrochars. Therefore, it is suggested to perform experiments with shorter residence times for further studies. However, in order to make a more accurate comparison with torrefaction, performing higher temperature degrees (around 250 °C) for HTC experiments are recommended. The available commercial hydrothermal carbonization systems are very limited because of some engineering problems. It's also recommended to establish pilot HTC systems for further studies. Also, developing solar energy integrated systems might reduce the costs for both methods.

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