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ORIGINAL ARTICLE

Investigation of fuel properties of hydrochars obtained from pomegranate peel: characterization and combustion kinetic

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Abstract
In this study, hydrochars were obtained from pomegranate peel (PP), which is a waste biomass, by hydrothermal carbonization method at different temperatures (200–240 °C) and through 1 h of residence time. Analysis techniques such as proximate, elemental, FTIR, and SEM were used for the characterization of the samples. According to elemental analysis results, with the increase in temperature, the carbon content of hydrochars and higher heating value increased. FTIR analysis results showed that there are aromatic and oxygen-containing structures (C = C, C = O, C–O) on the surface of hydrochars. According to the SEM analysis results, there are fibrous and slightly porous structures on the surface of the hydrochars. The combustion behavior of the products was investigated by thermogravimetric analysis. Thermogravimetric analysis results showed that the fuel properties of hydrochars improved with the increase in temperature. The Coats-Redfern method was used to estimate the kinetic parameters of the combustion processes. The activation energy values of the hydrochars ranged between 5.95 and 18.66 kJ/mol.

Keywords Hydrothermal carbonization · Thermogravimetric analysis · Pomegranate peel

1 Introduction

The excessive consumption of fossil fuels (oil, coal, and natural gas) in recent years has increased environmental and global problems. Consumption of fossil fuels causes carbon dioxide emissions into the atmosphere. Carbon dioxide emissions both increase the greenhouse gas effect and cause climate change [1, 2]. Preferring renewable energy instead of fossil fuels is an alternative remedy to decrease global problems caused by gas emissions. Biomass is a renewable energy source that is found in large quantities and is used to obtain biofuels [3]. Sustainable green energy has become a focal point in many areas of the world. In order to meet the energy request in the world, many researchers have started to look for solutions to obtain energy using renewable resources [4]. One of the best alternatives to fossil fuels is bioenergy. In recent years, thermochemical conversion techniques have been used to produce biofuels from biomass [5].

Hydrothermal carbonization (HTC), one of the promising thermochemical methods, has been used to obtain a carbon-rich solid called hydrochars. With the hydrothermal carbonization method, hydrochars can be produced without the need for pre-drying and this method is not expensive [1, 6]. With the HTC method, the thermochemical conversion of biomass is carried out by adding water into a closed reactor. Temperature and pressure are in the range of 180–260 °C, 2–10 MPa, respectively. Under suitable HTC conditions, hydrochars can provide thermal conversion performance [7]. Process parameters (temperature, biomass and water ratio, pressure, etc.) are effective in determining the properties of the products. In particular, temperature is a critical factor in defining product properties and has a significant influence on the convert pathways of the raw material [5, 8]. Numerous studies have shown that the fuel properties of various biomasses (agricultural waste, municipal solid waste and food waste, etc.) can be greatly improved by HTC. For example, Ma et al. [1], it has been reported that the calorific values of hydrochars produced by hydrothermal carbonization from pomelo peel at different temperatures (180–240 °C) vary in the range of 18.3–27.7 MJ/kg. While the raw pomelo peel has a low calorific value (13.1 MJ/kg), the high heating

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value of hydrochars show that hydrothermal carbonization is an effective method. As another example, Maqhuzu et al. [9] can be shown in a study they did. They produced hydrochars from municipal solid wastes by hydrothermal carbonization method, which can replace coal and be used as an alternative biofuel. In another literature study, it has been reported that hydrochars obtained from sawdust biomass by hydrothermal carbonization have high heating value compared to raw material [10]. For example, Wilk et al. [11], it has been reported that the high heating values of hydrochars produced by hydrothermal carbonization from the beet pulp at different temperatures (180–220 °C) vary in the range of 19.61–25.36 MJ/kg. Food wastes were subjected to hydrothermal carbonization at 180, 220, and 220 °C for 120 min. The high heating value among the obtained hydrochars was reported as 23.61 MJ/kg [12]. Pomegranate is a biomass consumed as fruit and widely used in medical and industrial areas [13]. Since pomegranate has wide usage areas, demand for this fruit is quite high in Turkey as well as in the world. When the results of the Turkish Ministry of Food and Agriculture are investigated, annual pomegranate production in Turkey overruns 500,000 tons [14]. Compared to other wastes, the peel of the pomegranate fruit, which is produced in large quantities, can be economically evaluated as a renewable energy source.

In this study, hydrochars were produced from low-cost PP at different temperatures (200, 220, and 240 °C) by hydrothermal carbonization method. In the first step, the characterization of PP and hydrochars was made. In the second step, the effect of temperature, which is the most important factor among the process parameters, on the fuel properties of hydrochars was investigated. In the last step, the combustion kinetics of PP and hydrochars were investigated. Hydrochar products obtained as a result of hydrothermal carbonization of PP have been reported in the literature. These hydrochars were mostly used as adsorbent in adsorption studies [14, 15]. The absence of many studies in the literature on the investigation of the fuel properties of hydrochars obtained from PP with HTC has added originality to this study.

2 Materials and methods

2.1 Materials

Raw PP was dried in an oven at 80 °C for 12 h. Dried PP was reduced in size by passing it through a grinder with stainless steel blades. The sieved samples with dimension less than 250 µm were used in the experiments.

2.2 Hydrothermal carbonization

Hydrothermal carbonization was carried out in a 40 mL volume of reactor. The raw material and water were added to the hydrothermal reactor at a mass ratio of 1:10. Hydrothermal carbonization was carried out at a reaction temperature of 200, 220, and 240 °C and a reaction time of 1 h. The hydrochar samples obtained from the PP was labeled as HC-200, HC-220, and HC-240.

2.3 Characterization of samples

The characterization of the hydrochar products was made using analysis methods such as Fourier transform infrared spectroscopy (FT-IR), elemental analysis, and scanning electron microscopy (SEM). Fourier transform infrared spectrometer (Mattson 1000) device was used for FT-IR analysis. FT-IR spectra of PP and hydrochar samples were drawn in the wavenumber range of 650 cm^{-1} and 4000 cm^{-1}. For elemental analysis (%C, H, S, and N) LECO brand elemental analyzer (CHNS-932) was used. The %C, N, H, and S amounts were measured simultaneously, and the oxygen content was calculated from the percentage difference. SEM analysis (Jeol, JSM-7001F) was performed to determine the three-dimensional surface properties of the products. Dimensioning of SEM images of raw pomegranate peel and hydrochar products was taken as 1, 10, and 100 µm. The yield of hydrochar products, HHV, energy densification, energy yield, fuel ratio, and combustion characteristic index (S) were calculated using the following Eqs. (1–6). T_i and T_b are the ignition and burnout temperatures.$(\frac{dW}{dt})_{max}$ and $(\frac{dW}{dt})_{mean}$ represent the maximum and mean values of the derivative termogravimetric (DTG) curve, respectively [16], Lang et al., [8].

$$\mathrm{HHV} = 0.3491\mathrm{C} + 1.1783\mathrm{H} - 0.1034\mathrm{O} - 0.015\mathrm{N} - 0.0221\mathrm{Ash} \quad (1)$$

$$\text{O content } (\%) = 100 - \mathrm{C} - \mathrm{H} - \mathrm{N} - \mathrm{S} - \mathrm{Ash} \quad (2)$$

$$\text{Energy densification} = \frac{\text{HHV of hydrochar}}{\text{HHV of feedstock}} \quad (3)$$

$$\text{Hydrochar yield } (\%) = \frac{\text{Hydrochar weight}}{\text{Feedstock weight}} \times 100 \quad (4)$$

$$\text{Energy yield } (\%) = \text{Hydrochar yield} \times \text{Energy densification} \quad (5)$$

$$\mathrm{S} = \frac{\left(\frac{dW}{dt}\right)_{max} \times \left(\frac{dW}{dt}\right)_{mean}}{T_i^2 \times T_b} \quad (6)$$

2.4 Thermogravimetric analysis and combustion of samples

Combustion behavior of raw pomegranate peel and hydrochars was investigated. In the measurements, Shimadzu brand DTG-60 model simultaneous TG/DTA analyzer was used. Approximately 7 mg of sample was added to the sample holder in the device. The ambient temperature at 25 °C was increased to 900 °C and the heating rate was 20 °C/min. The air flow rate was kept at 50 mL/min during the combustion process. The TG and DTG curves of the products were used to estimate the combustion parameters. These parameters are defined as ignition temperature (T_i), combustion temperature (T_b), maximum peak temperature (T_m), and maximum weight loss ratio (DTG_{max}), respectively.

2.5 Kinetic study of samples

In this study, the kinetic parameter (activation energy) of PP and hydrochar samples during combustion was calculated using the Coats-Redfern method given in the literature. The method has been successfully applied to different types of hydrochar to understand the reaction kinetics of the combustion process. It has been reported in the literature that the combustion process has been successfully applied to different types of hydrochars to understand the reaction kinetics [4, 17, 18]. The change in mass and temperature during the combustion process is defined as:

$$\frac{d\alpha}{dT} = \frac{A}{\beta} e^{\frac{-Ea}{RT}(1-\alpha)^n} \tag{7}$$

where A stands for pre-exponential factor, E represents the activation energy, and n stands for reactor order [4]. Equation (8) used in the Coats-Redfern method and simplified by accepting the first-order reaction rate expression is given below [18].

$$ln\left[\frac{-ln(1-\alpha)}{T^2}\right] = ln\frac{AR}{\beta E_a} - \frac{E_a}{RT}(n=1) \tag{8}$$

The graph of $ln\left[\frac{-ln(1-\alpha)}{T^2}\right]$ against $1/T$ was plotted to determine activation energy. Actıvation energy (E_a) calculated from the slope of lines.

3 Results and discussion

3.1 Physicochemical properties of samples

The proximate and elemental analysis results of raw PP and hydrochars are presented in Table 1. After the hydrothermal carbonization process, four elements (C, H, N, and S) were determined by elemental analysis. According to the literature, nitrogen (N) and sulfur (S) values of biomass vary between 0–8.65% and 0–2.01%, respectively [19]. S and N values in biomass are expected to be low in order not to generate greenhouse gases. As can be seen from Table 1, the nitrogen and sulfur content of the PP is very low. The nitrogen content of the hydrochars increased with increasing temperature. As the temperature increased by HTC, there was a decrease in the hydrogen (H) and oxygen (O) content of hydrochars, while an increase in the carbon (C) content occurred. Similar results were found in Cheng et al.'s study for HTC of coconut waste. Cheng et al. [20] reported that the carbon (C) content of hydrochars increased as the temperature increased.

The higher the carbon content, the higher the higher heating value. [22]. In this study, the carbon content of hydrochars obtained from PP waste increased as the reaction temperature increased. As the carbon content increased, an increase was observed in the higher heating value results (15.64–18.78 MJ/kg) of hydrochars. Proximate analysis (determination of ash and volatile matter) was performed for the characterization of raw pomegranate peel. The volatile matter content of hydrochars increased with increasing temperature. But, the yield of hydrochars decreased as the temperature increased. The reduction in mass yield is due to the release of volatile matter in addition to the sequential hydrolysis and decarboxylation reaction that takes place during hydrochars development [12]. The volatile matter content of biomass reported in the literature varies in the range of 50–94%. The ash content is between 0.1 and 25% [19]. The ash content of raw pomegranate peels is lower than that of other pomegranate peels reported in the literature [13]. The ash content of the hydrochars increased up to a certain temperature and then decreased. In general, the higher heating values of hydrochars obtained from different biomass vary between 18 and 40 MJ/kg in the literature [5, 16]. For example, Cai et al. [23] reported that the hydrochars' higher heating value reached from 26.1 to 27.2 MJ/kg when the temperature of 260 °C was increased. For example, Sayğılı and Sayğılı [14], hydrochars were obtained as a result of hydrothermal carbonization of pomegranate residues at 180, 220, and 240 °C and 6 h reaction time, and their higher heating values were reported as 20.34, 20.40, and 21.27 MJ/kg, respectively. The high heating values of the hydrochars obtained in this study increased as the reaction temperature increased. However, with the increase in temperature, hydrochars and energy yield values decreased. The energy yield of hydrochars is directly proportional to their mass yield [4]. However, when compared with the different hydrochar yields reported in the literature, the hydrochar energy yields in this study were high [16].

Table 1 Characterization of biomasses and the hydrochars

Parameters	This study				[20]				[21]			
	Biomass	Hydrochars			Biomass	Hydrochars			Biomass	Hydrochars		
	PP	200 °C	220 °C	240 °C	Coconut waste	180 °C	200 °C	220 °C	Food waste	180 °C	210 °C	240 °C
Hydrochar yield (%)	-	71.00	53.00	42.00	-	70.43	66.12	59.19	-	53.86	51.41	44.65
Proximate analysis (wt.%)												
Volatile matter	68.22	49.11	54.40	62.07	78.85	78.72	73.73	64.46	82.6	70.2	60.8	56.3
Ash	2.89	2.80	2.44	3.27	0.39	0.02	0.05	0.00	8.3	5.9	6.7	7.5
Fixed carbon[a]	34.67	48.09	43.16	34.66	20.76	21.26	26.22	35.54	9.1	23.9	32.5	36.2
Elemental analysis (wt.%)												
C	43.14	50.19	52.87	55.35	48.28	60.24	64.43	68.83	-	-	-	-
H	5.65	4.59	4.85	4.87	7.17	6.92	7.04	6.66	-	-	-	-
N	0.73	0.93	0.97	1.11	0.50	0.48	0.45	0.46	-	-	-	-
S	-	0.11	0.04	0.03	0.06	0.00	0.00	0.00	-	-	-	-
O[a]	47.59	41.38	38.83	35.37	43.99	32.35	28.09	24.05	-	-	-	-
Fuel properties												
HHV (MJ/kg)	13.66	15.64	17.45	18.78	-	25.83	27.88	29.39	16.13	21.72	26.24	28.86
Energy densification	-	1.15	1.28	1.38	-	-	-	-	-	1.35	1.53	1.71
Energy yield	-	81.65	67.84	57.96	-	87.71	88.85	83.85	-	72.71	78.65	76.35

([a]calculated by difference)

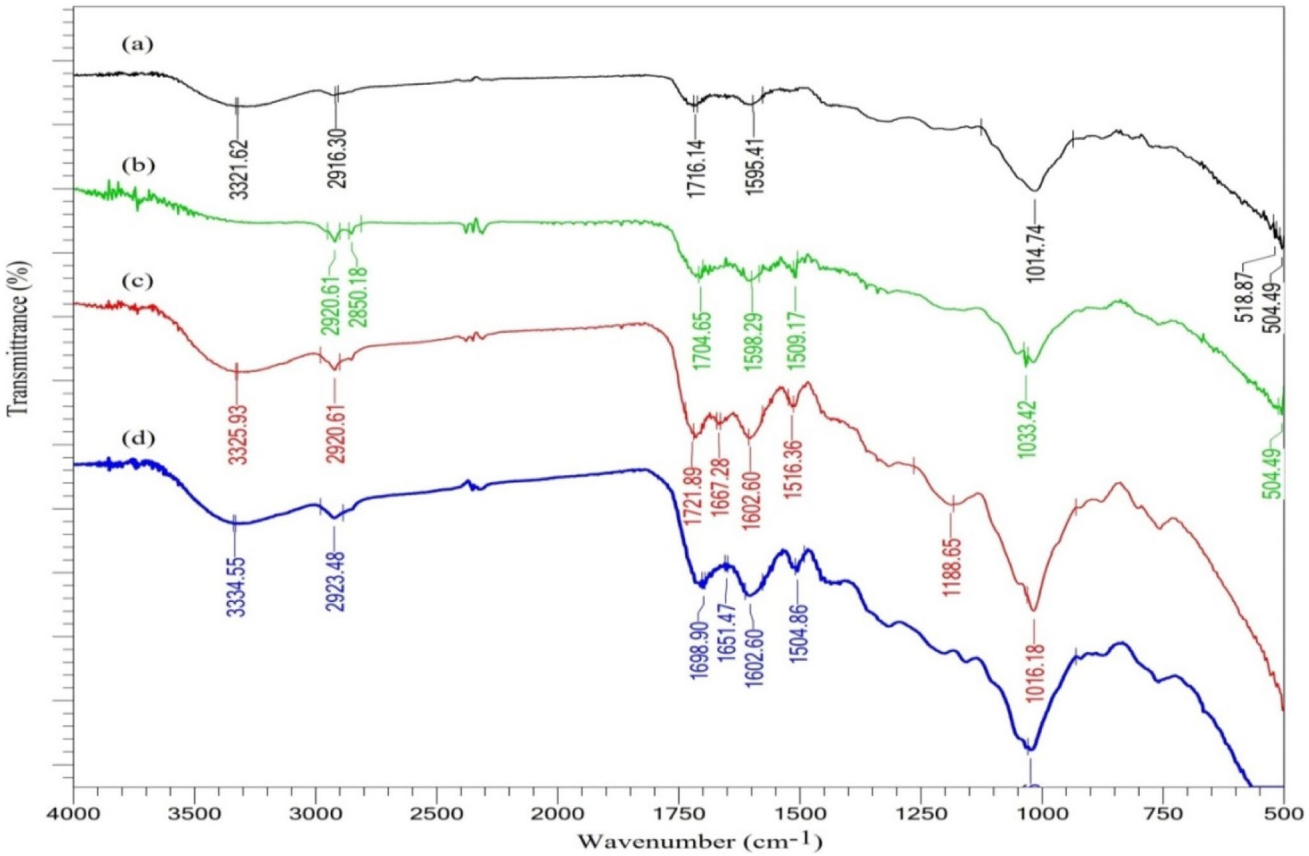

Fig. 1 FTIR spectra of (**a**) PP and (**b**) HC-200 (**c**) HC-220 (**d**) HC-240 products

Functional groups on the surface of raw PP and hydrochars were identified using FT-IR technology. The spectra of the pomegranate peel and hydrochars obtained as a result of the FT-IR analysis are given in Fig. 1. In the FT-IR spectrum of raw pomegranate peel, the broad vibration seen at 3321 cm^{-1} is the O–H vibration peak caused by water. The current peak at 2916 cm^{-1} is the C–H stretching vibrations of aliphatic hydrocarbons. The intense peak of carbonyl (C = O) asymmetric stretching is at 1716 cm^{-1} [19, 24]. This peak is indicative of the presence of a carboxyl, carbonyl, or ester group and confirms the oxygenated functional groups on the hydrochars [14]. The peaks at 1450 cm^{-1} are attributed to the – C = C stretch on the aromatic ring carbons and may be due to the aromaticity of hydrochars [25].

When the FTIR spectrum of the HC-200 product was examined after HTC treatment, a decreasing trend was observed in the intensity of the O–H stretching vibration. At the same time, an increase in the intensity of C–H stretching vibrations was observed around 2900 cm^{-1}. The increase in these peaks of hydrochars is higher compared to raw pomegranate peel. With the increase of the reaction temperature, the carbon content increased and the formation of aromatic coal was supported. The peaks observed around ≈1700 cm^{-1} in the structure of hydrochars were denser and stronger than the pomegranate peel. Peaks around 1400 cm^{-1} were associated with the – C = C stretch in the aromatic structure and its relative density slightly increased with increasing HTC temperature. Peaks at 1000–1050 cm^{-1} in all products are attributable to Si–O–Si tensile vibrations [8, 26].

3.2 SEM analysis

SEM analysis was performed to determine the structural differences in surface morphology and pores of raw pomegranate peel and hydrochar samples. SEM images of the samples are presented in Fig. 2. When the SEM images of the raw PP products are examined, it is seen that they have a fibrous structure. Similar results of SEM images of pomegranate peel have also been reported in the literature [13]. Compared to raw PP products, hydrochars have a slightly porous structure. In particular, we can say that the HC-200 product has a fibrous structure, as well as a more oval and spherical structure, and there are almost no pores in the structure. It is clear that all hydrochars have very few pores and exhibit

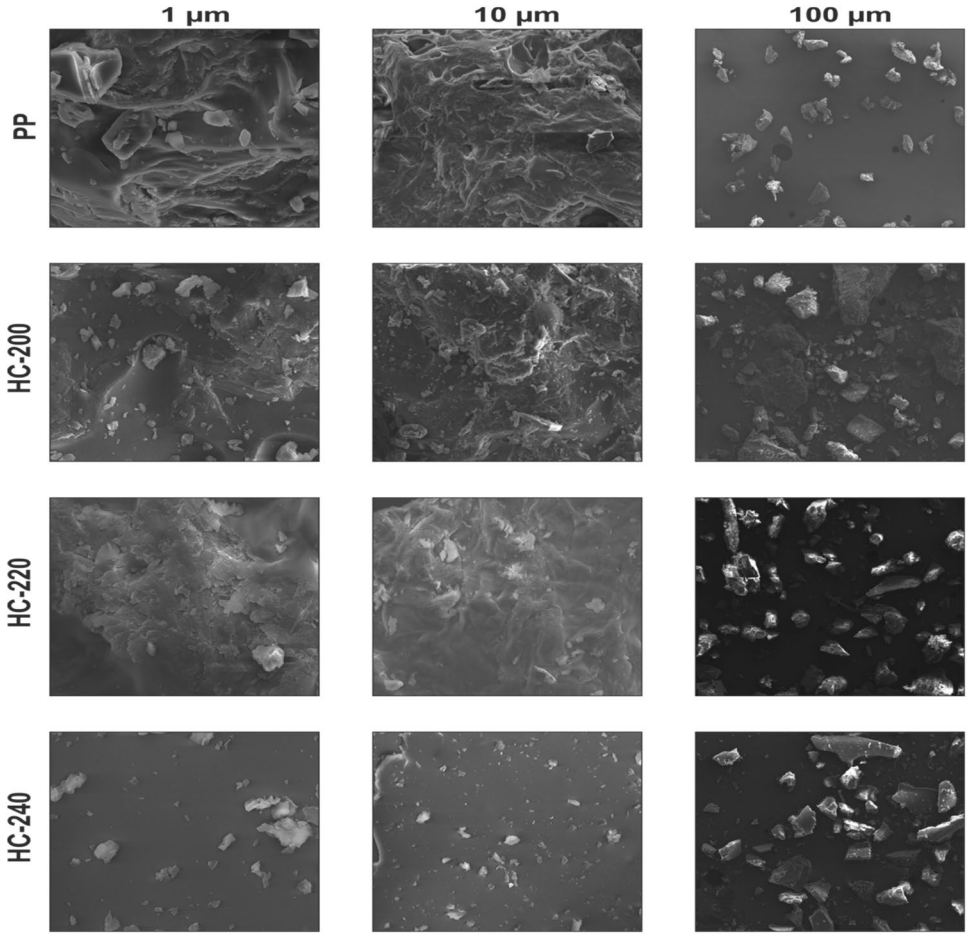

Fig. 2 SEM images of PP and the hydrochars

a similar structure. It has been reported in the literature that hydrochar products obtained by HTC are used as adsorbent in adsorption studies [14].

3.3 Thermogravimetric analysis and combustion characteristics

Thermogravimetric analysis of raw pomegranate peel and hydrochars was carried out by heating them from room temperature to 900 °C in an air atmosphere at a heating rate of 20 °C/min. The thermal curves of the samples (TG and DTG) are shown in Fig. 3. The decomposition of the raw pomegranate peel occurred in two steps. The first decomposition (first stage) in raw pomegranate peel was observed as a mass loss of 10.67% in the temperature range of 146–208 °C. At this stage, moisture and volatile molecular weight substances on the surface are removed. According to the DTG curves, the second major decomposition (second stage) was observed as a sharp peak in the temperature range of 416–538 °C. At this stage, a mass loss of 85.65% was observed. As lignocellulosic biomass, pomegranate peel consists of hemicellulose, cellulose, and lignin. The temperature range for the decomposition of lignin, cellulose, and hemicellulose has been announced in the literature as 160–900, 310–400, and 210–325 °C, respectively. Therefore, cellulose and lignin were separated from the structure in the main decomposition [27]. Raw pomegranate peel first and second stage DTG values were − 5.28 and − 144.03%/min, respectively, and remarkably, it is clear that the DTG values in stage 2 of PP are much larger. While the decomposition of HC-200 and HC-240 samples occurred in two steps, the decomposition of HC-220 sample occurred in three stages. The main decomposition of HC-200, HC-220, and HC-240 samples is in the temperature range of 410–525, 393–573, and 406–595 °C, respectively. As in this study, hydrochars that completed the combustion process in at least three stages have been reported in the literature [26, 28]. When the DTG curves of the hydrochars are examined; the maximum weight loss rate in the second stage belongs to the highest HC-240 sample. The maximum weight loss rate increased with the increase of the hydrothermal reaction temperature.

Combustion parameters of raw PP and hydrochars are presented in Table 2. Ignition and burnout temperatures

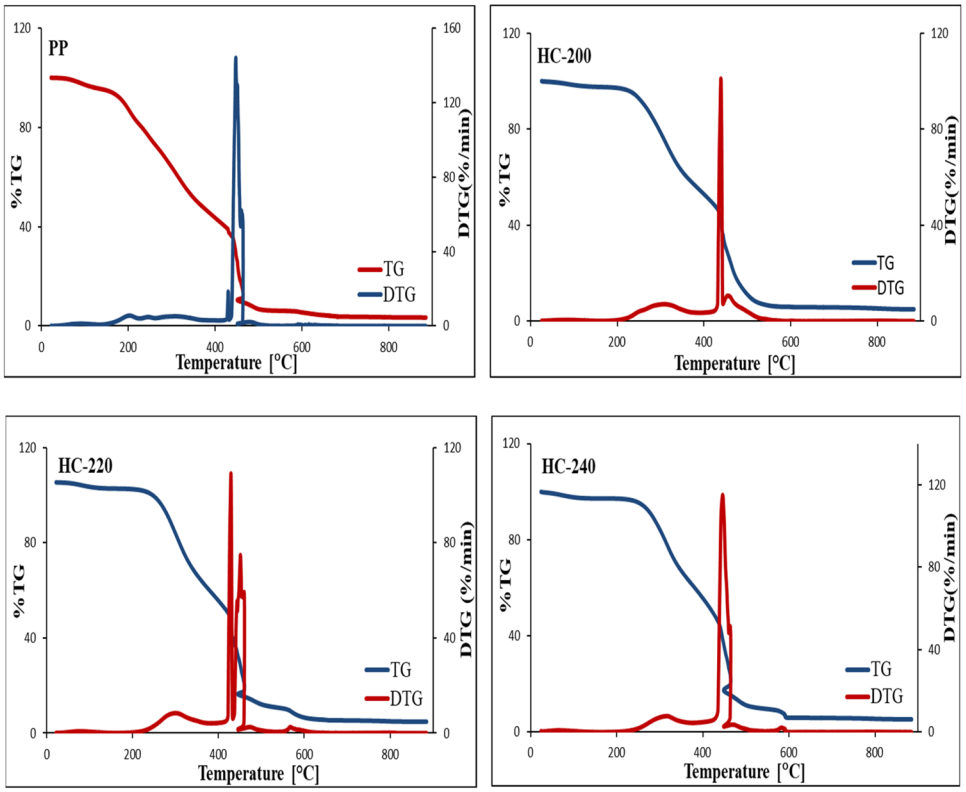

Fig. 3 TG and DTG curves for PP and hydrochars

Table 2 Characteristic combustion parameters of PP and hydrochars

Sample	T_i (°C)	T_m (°C)	T_b (°C)	$-\Delta TG_{max}$ (%/min)	ΔTG_{mean} (%/min)	$S \times 10^{-6}$ ($\%^2/°C^3.min^2$)
PP	146	447	538	144, 03	1.97	25
HC-200	226	438	525	100, 98	1.95	7.3
HC-220	230	429	573	109, 17	2.05	31
HC-240	243	444	595	113, 69	1.94	6.3

are the main parameters used to evaluate fuel selection. There are many parameters used in fuel selection. But ignition and combustion temperatures are one of the most important parameters. For example, when it comes to the storage and safety of fuels, the ignition temperature (T_i) is taken into account [29]. High T_i reduces the spontaneous combustion of hydrochars and makes it a safe solid fuel. In this study, the "intersection method (IM)" was used to determine the ignition and burnout temperature [29]. When Table 2 is examined, it is seen that hydrochars have higher T_i and T_b values when compared to the pomegranate peel. It is obvious that T_i and T_b values of hydrochars gradually increase with increasing temperature. For example, similar to our study, the T_b value of hydrochars produced from recycled paper waste showed a slight increase as the HTC temperature increased [30]. Combustion characteristic index (S) is an important parameter used to evaluate the combustion performance of solid fuel. The higher the S value, the better the combustion performance [11]. The increase in HTC temperature had a significant effect on the S values. Among the hydrochars, the highest S index was determined as HC-220. This value was higher than the S index value of raw PP.

3.4 The combustion kinetics of PP and hydrochars

Combustion kinetics of raw PP and hydrochars were investigated by using mass loss data against temperature obtained as a result of TGA analysis. Determination of kinetic parameters is very important for the efficiency of a facility to be established and operated [31]. In general, the approximation

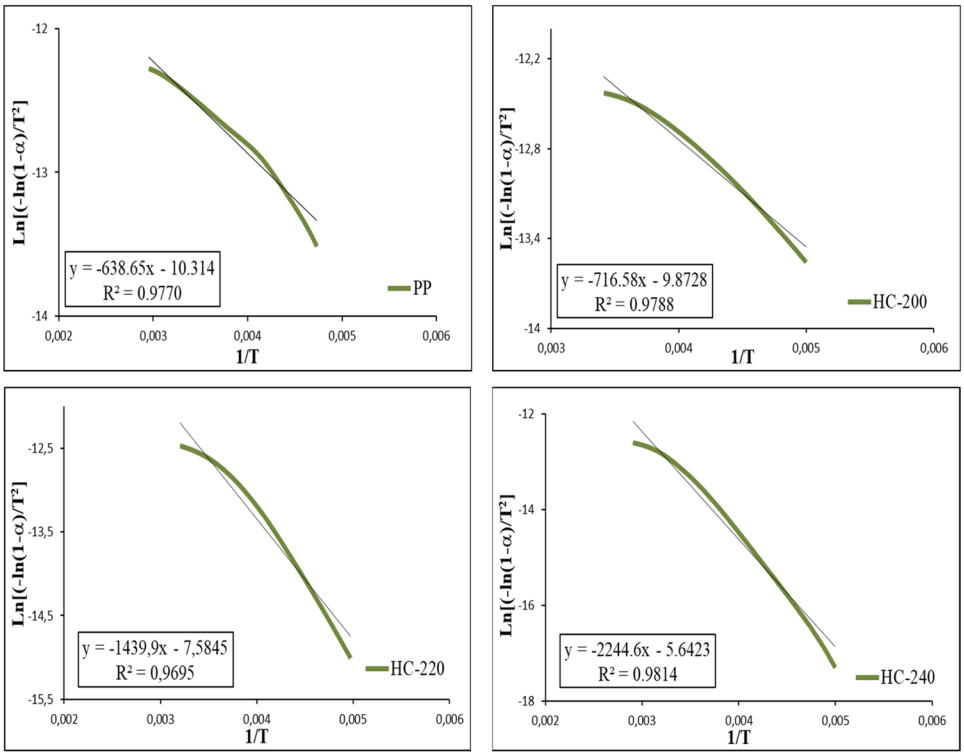

Fig. 4 Plot curves for PP and hydrochars

of the energy barrier and intensity of combustion reaction for the fuel is made with the help of information of activation energy [17]. In this study, the Coats-Redfern method was used to estimate the kinetic parameters of the combustion processes. Activation energy (E_a) is one of the main parameters defining biomass combustion kinetics. Since the activation energy is the minimum energy required to start a reaction [32], the kinetic analysis of the samples was made and the activation energies were calculated. Linear line graphs were drawn using the results of thermogravimetric analysis and the Coats-Redfern equation. Using the pilot curves shown in Fig. 4. Activation energy values of PP, HC-200, HC-220, and HC-240 products were calculated as 5.3, 5.95, 11.97, and 18.66 kJ/mol, respectively. Correlation coefficient values for all samples ranged from 0.9695 to 0.9814. The activation energy value of hydrochars is higher when compared to raw PP. The activation energy of the hydrochar samples increased as the HTC temperature increased. This significant increase in activation energy upon increase in reaction temperature is attributed to the loss of volatile matter during the HTC process [17]. Many researchers have used the Coats-Redfern method to estimate the kinetic parameters for the combustion processes of different biomass and hydrochars [12, 17, 18]. In the literature, there are not enough studies on the combustion kinetics of PP wastes. Within the scope of this study, the activation energy of the hydrochars was calculated by using the linear line graphs obtained using the thermogravimetric analysis data and it was found that it was lower than the other hydrochars reported in the literature [12, 33].

4 Conclusions

Hydrochar products with high carbon content and higher heating value have been obtained from the PP biomass. With increasing temperature, the carbon content and higher heating values of hydrochars increased compared to raw PP. The product with the highest heating value is HC-240. However, the ash value of this product is higher than other hydrochars and its *S* index value is low. The low ash value of hydrochars is an advantage in terms of fuel properties. Ash content of HC-200 and HC-220 products decreased compared to raw PP. The lowest activation energy value among hydrochars is HC-200. The low ash content of the HC-220 value and the high *S* index value indicate that it has biofuel potential.

Author contribution This article is a single author article.

Data availability All data generated or analyzed during this study are included in this published article.

Declarations

Ethics approval Not applicable.

Competing interests The author declares no competing interests.

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