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Research Article

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The Effect of Slow Pyrolysis Temperature on Selected Properties of Biochar Derived from Grape Pomace

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Abstract In this research, it was studied on the conversion of the grape pomace, which is released as a result of the processing of grapes, into biochar as an alternative by using the slow pyrolysis method. For this purpose, the changes in the biochar samples properties obtained via slow pyrolysis process applied at different temperatures were investigated. Pyrolysis experiments were carried out at 400, 500 and 600 °C temperature conditions. Biochar yields and quality characteristics of samples were investigated. In order to prepare the grape pomace for the pyrolysis process, the drying and grinding processes were carried out firstly. For this aim; proximate analyzes, elemental analysis, heat values, bulk density, and SEM analysis were made. Results showed that increase of pyrolysis temperature decreased the biochar yield. The highest biochar yield was obtained for 400 °C as 63,8% while minimum yield was obtained for 600 °C as 31,0%. There was a general decrease in the moisture values and ash content of the biochar samples with increasing of pyrolysis temperature. Heating value, fixed carbon content and volatile matter content of the biochar samples changed between 29.64-26.82 MJ/kg, 33.80-58.80% and 8.43-30.16%, respectively. Contents of C, H, N were determined between 79.74-80.11%, 2.58-2.68%, and 1.57-1.97%, respectively. Oxygen value was determined as 0% for all biochar samples. The increase in the pyrolysis temperature caused an increase in the density values. The densities of biochar samples varied between 347-381 kg/m3. The SEM results indicate that an increase in temperature leads to an increase in SEM porosity.

Keywords Grape pomace, slow pyrolysis, biochar, heat value

1. Introduction

Among the new and renewable energy sources, biomass is an environmentally friendly energy source that has a wide range of applications, especially for developing countries. The use of plant residues, which are one of the most important biomass sources, to generate energy holds a significant place in the studies related to biomass. The utilization of waste and residues resulting from agricultural production and processing is particularly important. One of the most important agricultural products in our country and in the world is grape. Grape pomace, which is the only solid waste generated during production and consists of the fleshy part, skin, seeds, and stems of grapes, has a complex composition. This waste accounts for approximately 17-20% of the total grape mass [1]. A significant portion of this grape pomace accumulates in processing points in large quantities and, due to its inability to be effectively utilized, causes significant environmental pollution issues when discarded. Due to its characteristics, grape pomace is known to have high potential in terms of being used as an energy source, activated carbon, fuel, and chemical production, clean energy generation, proper disposal of waste, and low-cost raw material production [2].

Pyrolysis is a preferred thermochemical process in the conversion of difficult-to-utilize biomass sources into more valuable solid, liquid, and gas primary products. This process is considered one of the best methods for converting biomass into various fuels [3]. The solid product obtained from the pyrolysis process (char, biochar) contains elemental carbon along with hydrogen and various inorganic species. The solid product can be utilized in various industrial sectors. For example, it can be used for the production of activated carbon, as solid fuel in different forms for boilers, for obtaining hydrogen-rich gas mixture through thermal cracking, etc. [4].

When the literature on the pyrolysis of biomass is examined; it is seen that there are many researches on the slow pyrolysis of wastes generated after the processing of agricultural products. It is understood that slow pyrolysis applications of wastes resulting from the processing of oily plants into oil are generally investigated in research on food processing plant wastes. Pressed sunflower (Helianthus annus L.) pulp [5], rapeseed pulp [6], industrial olive waste [7], safflower seed pulp [8], rapeseed oil pulp [9] are some of these wastes.

Although studies have been carried out in recent years on the evaluation of grape pomace by pyrolysis method, their number is quite low. In these studies, different pyrolysis methods were applied to the grape pomace. Xu et al. (2009) [10] conducted research on flash pyrolysis of grape wastes in a temperature range of 300-600 °C using a reactor with a bubble fluidized bed reactor and determined the heating value of the gas obtained. Optimum pyrolysis temperature were determined by maximizing the liquid yield, the energy from the bio-oil, and the net energy. Pala et al. (2014) [11] worked on the charring of grape pomace using hydrothermal torrefaction method (in nitrogen environment and at 250 and 300 °C) and as a result, they determined that the solid product yield varied between 47% and 78%, and the energy density was found as 1.42-1.15 compared to the original product. Volpe et al. (2016) [12] studied the carbon reactivity in the thermal decomposition of grape pomace in the temperature range of 150-650 °C. The solid product obtained as a result of pyrolysis was subjected to Thermo Gravimetric Analysis (TGA) in order to determine the carbon reactivity.

The main objective of this study is to determine the effect of pyrolysis temperature on the yield and quality characteristics of biochar obtained from grape pomace through slow pyrolysis. For this purpose, in addition to the characteristics of the raw material, namely grape pomace, the thermal values, proximate analyses, elemental contents, chemical compositions, and other quality characteristics of the biochar samples obtained through pyrolysis processes were comprehensively determined, analyzed, and compared.

2. Materials and Methods

Preparation of grape pomace

The grape pomace used in the pyrolysis process is the processing residue after extracting grape juice from the Hamburg Muscat of Hamburg black grape variety and is obtained from Tekirdag Viticulture Research Institute. Grape pomace consists of skin, stem, and seed components and its initial moisture content is approximately 59%. To prepare the pomace for pyrolysis, it was dried in a hot air drying oven (Eksis brand) at a temperature of 60 °C and an air velocity of 1 m/s until it reached a moisture content of 6.72%. In order to achieve homogeneity and ensure a uniform temperature distribution, the pomace was reduced to an average particle size of 0.75±0.25 mm.

Slow pyrolysis system

The slow pyrolysis system, shown in Figure 1 with its schematic representation and components, was used for the slow pyrolysis process of grape pomace. In order to control the pyrolysis conditions, temperature variations and mass changes during the experiments were displayed and the data was stored. For this purpose, a PLC control unit and an operator panel were added to the pyrolysis reactor. A thermocouple was installed on the pyrolysis reactor to measure temperature, and load cells were placed underneath.

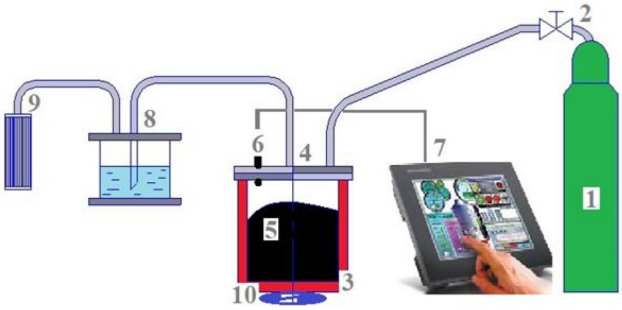

Figure 1: The components of the slow pyrolysis system (fixed-bed): (1) nitrogen cylinder, (2) valve and flowmeter, (3) heating resistor, (4) reactor, (5) biomass or biochar, (6) thermocouple, (7) control unit (8) condensation vessel, (9) carbon filter, (10) weight measurement system (for biochar).

Pyrolysis experiments and determining product yields

Pyrolysis experiments were conducted at three different pyrolysis temperatures: 400, 500, and 600 °C. The heating rate was set at an average of 26 °C/min. The residence time in the reactor was determined as 30 minutes based on preliminary experiments, where the gas release duration was observed. Throughout the experiments, temperature measurements from thermocouples were monitored and recorded using the digital indicators on the control panel to ensure control over the reactor temperature. At the end of the pyrolysis process, the remaining solid product in the reactor was weighed and proportioned to the amount of pomace fed to the reactor to determine the biochar yield [13].

Analyses of pomace and biochar

Moisture content, volatile matters, ash content, and fixed carbon values were determinded within the scope of the proximate analysis.The moisture content (MC) (%w.b.) of grape pomace and biochar samples was determined following the EN 14774-2 standard, utilizing Equation 1, which considers the sample weight before drying (m_i) (g) and after drying (m_f) (g).

$$MC(\%) = \frac{m_i - m_f}{m_i} * 100 \qquad (1)$$

To determine the ash content, fixed carbon, and volatile matter of the samples, a Nuve brand MF110 Model muffle furnace was utilized. The ash content was determined in accordance with the EN14775 standard by proportioning the obtained ash amount to the initial weight of the product. For the determination of volatile matter and fixed carbon percentages in both the pomace and biochar samples, the samples were dried for 24 hours at 105 °C. Prepared samples weighing 0.8-1 g were placed in alumina crucible and subjected to a muffle furnace at 600±50 °C for 6 minutes, followed by 950±20 °C for another 6 minutes. Subsequently, the samples were weighed, and the volatile matter amount was calculated based on the difference between the initial and final sample weights. The fixed carbon percentage (FC%) was calculated using Equation 2, which depends on the ash (A%) and volatile matter (VM%) percentages [14].

$$FC\% = 100 - (A\% + VM\%) \qquad (2)$$

The elemental analyses, including carbon, hydrogen, nitrogen, and sulfur contents, were conducted using a elemental analyzer (Elementar Brand, Vario Micro Cube Model). The percentage of oxygen (O%) was calculated based on the percentages of carbon (C%), hydrogen (H%), nitrogen (N%), sulfur (S%), and ash (A%) using Equation 3.

$$O\% = (100 - C\% - H\% - N\% - S\% - K\%) \qquad (3)$$

The higher heating values (HHV) of both pomace and biochar samples were determined using the IKA C100 calorimeter in accordance with the EN 14918 standard.

For microscopic examination of the surface properties of the solid products resulting from the pyrolysis experiments and capturing SEM images, a FEI brand QUANTA FEG 250 model Scanning Electron Microscope (SEM) was utilized.

Bulk density values of both pomace and biochar samples were determined following the PN-EN 15103 (2009) [15] standard. A calibrated container with a constant volume was filled with the material and weighed. The density value was calculated by dividing the mass of the samples by the volume of the container.

Statistical analysis

One-Way ANOVA (analysis of variance) was conducted to assess the properties of biochar obtained at three different pyrolysis temperatures (400, 500, and 600 °C). The Duncan test was used to examine the significance level (P) of 0.05 for the differences. The PASW Statistics 18 statistical package program was used for this analysis.

3. Results and Discussion

Grape pomace properties

The moisture, dry matter, and water activity percentages of wet grape pomace obtained after grape processing were measured with 3 repetitions and determined as 59%, 41%, and 0.95%, respectively. After the pomace was dried, the moisture, dry matter content, and water activity values were found to be 6.72%, 93.28%, and 0.25%, respectively. Proximate analyses of raw materials are crucial chemical properties of pomace that significantly impact fuel properties in the pyrolysis process. Table 1 presents the average values of the results, the heat values and elemental contents of the pomace, in comparison with the values determined for biochar samples obtained under different prysolysis temperatures.

Biochar yields

The average values of biochar obtained from experiments conducted on grape pomace pyrolysis are given in Figure 2. As seen in this figure, the highest biochar yield is obtained at a pyrolysis temperature of 400°C, with a yield of 63.8%, while at 500°C, this value decreased to an average of 35.9%. At 600°C pyrolysis temperature, the yield decreased to 31%. Ibn Ferjani et al. (2019) [16] investigated the effects of process temperature on the properties of biochar in slow pyrolysis applied to grape pomace. Similar to our results, experiments conducted at pyrolysis temperatures between 300 and 700°C showed that increasing the temperature reduces the biochar yield, reaching around 33% at approximately 500°C, and the biochar yield remains relatively unchanged at temperatures above 500°C. In our research, the results obtained are consistent with these findings. Khiari and Jeguirim (2018) [17] also obtained a 40% biochar yield from the slow pyrolysis process conducted on grape pomace using thermogravimetric method, and they emphasized that this yield is quite high for agricultural by-products. This high biochar yield can be attributed to the high lignin content present in grape pomace.

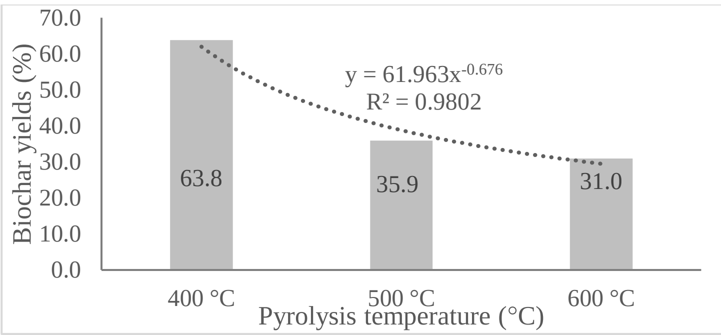

Figure 2: Changing of biochar yield versus pyrolysis condition

Pomace and biochar samples properties

Results of proximate, elemental and density analyses of the obtained for raw pomace and biochar samples were given in Table 1. The difference in moisture content between biochar samples obtained under different slow pyrolysis temperatures was found to be statistically significant (P<0.05). The moisture content of the biochar

samples decreased with increasing pyrolysis temperature. While the moisture content in biochar samples obtained at 400°C was determined to be 3.23%, it decreased to 2.59% with increasing the temperature to 600°C.
In all biochar samples, the ash content increased compared to the raw material's ash content (3.64%) after the pyrolysis process. Also it has been determined that the ash content increased with the increase in pyrolysis temperature in biochar samples (Table 1). In combustion systems, it is desirable for the ash content to be as low as possible. Similar to this research results, Angin (2013) [18] reported an increase in ash content of biochar with an increase in pyrolysis temperature. When examining the ash content of biochar samples obtained from grape pomace, it is evident that the ash content is considerably higher compared to other biomass-derived biochar samples, such as cotton stalks, orange peels, and palm waste [19].
Consistent with the literature data [18, 20], our research shows that the volatile matter content in all biochar samples obtained has significantly decreased compared to the volatile matter content of the raw material (73.41%) after the pyrolysis process and an increase in pyrolysis temperature resulted in a decrease in the volatile matter content of the biochar. The minimum volatile matter content was found to be 8.43% in sample that obtained by pyrolysis at the 600 °C, while the maximum volatile matter content of 30.6% was found in the in sample obtained by pyrolysis at the 400 °C (Table 1). The volatile matter content obtained from biochar samples derived from grape pomace does not exceed the values provided for fossil-derived coal types. However, it is believed that in biochars produced at 600°C through the pyrolysis process, this value may be too low to sustain combustion.
When examining the fixed carbon contents of the pomace and biochar samples (Table 1), it is evident that the pyrolysis process leads to an increase in fixed carbon content in every case. Many researchers have observed a similar trend in their studies [21, 22, 23]. The lowest and highest fixed carbon contents were found to be 33.80% and 58.80%, respectively, in the biochar samples obtained at 400 and 600°C. These characteristics of biochar samples contribute to their potential use as a fuel similar to coal.
When examining the results of the calorific values given in Table 1, it has been determined that the calorific values of the biochars obtained increase with the increase in pyrolysis temperature. Similarly, many researchers have found that the calorific values of biochar samples obtained at higher pyrolysis temperatures also increase [18, 19]. The calorific value of the biochar obtained at 400°C pyrolysis temperature was measured at 29.64 MJ/kg, while the calorific value of the biochar obtained at 600°C pyrolysis temperature was measured at 29.82 MJ/kg. On the other hand, the difference in calorific values between the biochar samples obtained at 400 and 500°C temperatures was found to be statistically insignificant ($P>0.05$).
Elemental analysis results of biochar samples obtained from slow pyrolysis of grape pomace under different temperature conditions are given in Table 1. These results showed that the pyrolysis process increased the carbon content. While the carbon value of the raw grape pomace was 48.30%, this value was 80.11% in the biochar samples obtained after pyrolysis at 400 °C. With the increase in temperature from 400 °C to 500 °C, the amount of carbon increased to 80.44%. On the other hand, with the increase in temperature to 600 °C, there was a slight decrease in the carbon percentage (79.74%). Pyrolysis process decreased the oxygen content. The oxygen content of the raw grape pomace was 41.70%, while the oxygen content was 0% in the biochar samples obtained after the pyrolysis process. When the hydrogen contents of the samples are examined, it is seen that the hydrogen content decreases with the pyrolysis process. While the hydrogen content of the raw grape pomace was determined as 7.04%, the hydrogen content of the biochar samples varied between 2.58-2.68%. The increasing of pyrolysis temperature also caused a decrease in the amount of hydrogen. During the combustion process, nitrogen undergoes transformation into N_2 and NO_X gases, which can have harmful effects on the environment. Only a small fraction of nitrogen converts into ash during this process. Similarly, sulfur content transforms into SO_2 form, which can either condense on the heat exchanger surfaces or contribute to the formation of ash. Therefore, low sulfur and nitrogen content is desired in fuels. The nitrogen content of the samples is between 1.57-1,92% and it is higher than the nitrogen content of the raw material (1.2%). The sulfur content was measured as zero, which is highly desirable for these products.

Table 1: Comparison of the results determined for dry grape pomace and biochar samples

Sample	Moisture content (%w.b.)	Ash content (%)	Fixed carbon (%)	Volatile matter (%)	Higher heating value (MJ/kg)	C (%)	H (%)	O (%)	N (%)	Bulk density (kg/m^3)
Raw pomace	6.74^a	3.64^a	22.95^a	73.41^a	20.76^a	48.30^a	7.04^a	41.70^a	1.20^a	463^a
S-400	3.23^b	36.05^b	33.80^b	30.16^b	29.64^b	80.11^b	2.68^b	0^b	1.92^b	347^b
S-500	3.17^b	35.38^c	42.34^c	22.28^c	29.66^b	80.44^c	2.63^c	0^b	1.57^c	354^c
S-600	2.59^c	32.77^d	58.80^d	8.43^d	29.82^c	79.74^d	2.58^d	0b	1.97^d	381^d

* The same letters in each sample are not significantly different (P<0.05)

Table 1 shows the average bulk densities of the pomace and the biochar samples obtained under different pyrolysis temperatures. The differences between the bulk densities were found to be statistically significant (P<0.05). It was determined that there was a decrease in the bulk density of all biochar samples compared to the pomace density. The increase in the pyrolysis temperature also caused an increase in the density values. While the density of pomace was 463 kg/m^3, the densities of biochar samples varied between 347-381 kg/m^3. It has been reported that high-density materials have a higher combustion efficiency due to their higher mass in the same volume [23].

SEM analysis results

When comparing the SEM images, it can be observed that in all samples subjected to the pyrolysis process, porosity begins to form, and there is a general increase in the number of pores with the increase in temperature. The formation of pores can be attributed to the gradual removal or evaporation of organic and volatile compounds from the biomass, which is dependent on the pyrolysis temperature. Based on the SEM images, it is evident that the particle sizes and shapes of the biochar are influenced by the pyrolysis temperature. Ertaş (2010) [24], in a research characterizing products obtained from the slow pyrolysis of some biomass residues, stated that an increase in temperature generally results in an increase in the size and proportion of voids, and the cell walls of solid products become very thin and fragile due to the effect of temperature. Overall, the increased in temperature has increased carbon density while eliminating oxygen through dehydration and evaporation. In our samples, the SEM results also indicate that an increase in temperature leads to an increase in SEM porosity.

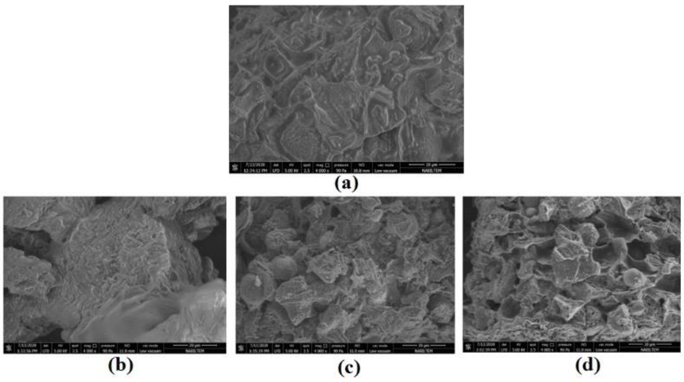

Figure 3: SEM images of raw pomace and biochar samples

4. Conclusion

Proximate analyses regarding grape pomace indicate that this waste has a moisture content of 6.72%, an ash content of 3.64%, a volatile matter percentage of 73.41%, and a fixed carbon percentage of 22.95%. Especially low moisture and ash content values demonstrate that grape pomace is suitable as a biomass source. However, when considering the volatile matter content of the biochar obtained from the pyrolysis of grape pomace at 600°C, it is evident that this value is insufficient to support combustion. Therefore, if pyrolysis products are intended to be used as fuel in biochar combustion systems, it is recommended to conduct pyrolysis at 400°C and 500°C.

Comparing the calorific values of the biochar obtained after the process with solid fuels (e.g., standard lignite with a value of 23.00 MJ/kg), it is advisable to use the biochar as a solid fuel in industrial applications.

The necessity for the carbon content of the biochar obtained from pyrolysis to be higher than 50% of the dry mass is met by all biochar samples obtained in this research. As oxygen reduces the calorific value of fuels and causes corrosion, biochar samples obtained at all temperatures are preferable since they have zero oxygen content.

Considering that high density materials tend to have higher combustion efficiency, pyrolyzed biochar at high temperatures may be used as a fuel.

In conclusion, based on the evaluations of biochar yields from pyrolysis processes and the quality of the products, it is recommended to use slow pyrolysis process for grape pomace. Considering the results obtained from this research, further studies are suggested to enhance the effectiveness of the pyrolysis method used in this work. These studies can focus on pre-treatments applicable to the feedstock before pyrolysis, the use of different pyrolysis techniques, and other related subjects.

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