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Effect of pyrolysis temperature and heating rate on biochar obtained from pyrolysis of safflower seed press cake

Dilek Angın *

Department of Food Engineering, Faculty of Engineering, University of Sakarya, Esentepe Campus 54187, Sakarya, Turkey

HIGHLIGHTS

- ▶ High quality biochar can be produced from safflower seed press cake by pyrolysis.
- ▶ Temperature has a stronger effect on structure of biochars than heating rate.
- ▶ HHV, pH, fixed carbon, ash and carbon contents of biochar increased at 600 °C.
- ▶ SPC biochar can be used as a chemical feedstock for industrial purpose.

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ABSTRACT

Biochar is carbon-rich product generated from biomass through pyrolysis. In this study, the effects of pyrolysis temperature and heating rate on the yield and physicochemical and morphological properties of biochars obtained from safflower seed press cake were investigated. The results showed that the biochar yield and quality depend principally on the applied temperature where pyrolysis at 600 °C leaves a biochar with higher fixed carbon content (80.70%) and percentage carbon (73.75%), and higher heating value (30.27 MJ kg^{-1}) in comparison with the original feedstock (SPC) and low volatile matter content (9.80%). The biochars had low surface areas (1.89–4.23 m^2/g) and contained predominantly aromatic compounds. The biochar could be used for the production of activated carbon, in fuel applications, and water purification processes.

1. Introduction

Biomass can be converted into fuel and chemical feedstock by biological (fermentation and anaerobic digestion) or thermochemical (gasification, liquefaction) routes. Among these conversion processes, pyrolysis allows a high energy recovery and produces fewer pollutants than other options, e.g. incineration. Moreover, this process generates a wide spectrum of products (solid, liquid and gas) with numerous applications, thus making pyrolysis treatment self-sufficient in terms of energy usage (Fernández and Menéndez, 2011).

Typically, pyrolysis is divided into three modes: fast, intermediate, and slow. The mode and the conditions of pyrolysis can influence the relative proportions of the three products (gas, liquid and solid) obtained. Slow pyrolysis uses a low heating rate and a long vapor residence time, and it is used to maximize solid product yields (Kwapinski et al., 2010).

The solid product from pyrolysis contains char, ash and unchanged biomass material and it is known as char or biochar. Biochar is carbon-rich and can be used as a fuel in the form of briquettes or a char-oil water slurry (Li and Zhang, 2005). The biochar can be used in the preparation of active carbon when its pore structure and surface area are appropriate (Demirbaş et al., 2006). Biochar has been attracting growing interest due to its potential in carbon sequestration and in improving soil health (Hossain et al., 2011).

The properties of biochar are different from those of activated carbon. Generally, biochar is a not fully carbonized product because pyrolysis is often carried out under low temperatures (<500 °C) (Zheng et al., 2010).

Biochar can be made from various waste biomass sources under different processing conditions. Conditions such as the heating rate, temperature and biomass particle size play roles in the structural evolution of biochar during rearrangement of the solid phase (Yao et al., 2011; Li and Zhang, 2005; Asadullah et al., 2011). Also, the quality of biochar for industrial and domestic utilization is often determined by its physicochemical characteristics such as volatile matter, fixed carbon, ash content, % carbon content, and higher heating value (HHV). Moreover, the surface properties of biochar affect the reactivity and combustion behavior of the biochar (Titiladunayo et al., 2012).

* Tel.: +90 264 2955927; fax: +90 264 2955927.
E-mail address: angin@sakarya.edu.tr

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Safflower (*Charthamus tinctorius* L.) is a plant with one of the highest oil contents. While safflower oil can be used as edible and industrial purposes, safflower seed cake is only used as animal feed (Stanford et al., 2001). Pyrolysis of safflower seed press cake (SPC) has already been studied (Şensöz and Angın, 2008a,b), but, the effect of process conditions on the properties of biochar have not yet been investigated. Therefore, the purpose of the present study was to investigate the effects of the pyrolysis temperatures and heating rates on the yield, pore structure, and physicochemical properties of biochar produced from SPC. Ultimate and proximate analyses were carried out and calorific values and chemical compositions of the biochars were investigated. The BET surface area, total and micropore volumes were also identified.

2. Experimental

2.1. Materials and sample preparation

Safflower seeds were supplied from Eskisehir Anatolia Agricultural Research Institute (ATAEM). Safflower seed press cake (SPC) was obtained from a mixture of Dincer and Yenice varieties by the cold-press extraction method (Şensöz et al., 2001). Prior to use, the safflower seed press cake (SPC) was air-dried and screened to give an fraction average particle size of 1.8 mm. SPC samples were stored in glass jars at room temperature under dry conditions.

2.2. Pyrolysis experiments

Pyrolysis experiments were performed with 20 g of biomass samples (SPC) in a previously described system (Şensöz and Angın, 2008a) at 400, 450, 500, 550 and 600 °C and at heating rates of 10, 30 and 50 °C min^{-1}. After each experiment the biochar yield was obtained from the final weight of the char. The biochar yields were expressed on a dry ash-free (daf) basis and the average yields from at least three experiments are presented. The experimental error was less than <± 0.5 wt%.

2.3. Characterization of the biochars

Proximate analyses were determined according to the ASTM 3174 and ASTM 3175. Fixed carbon content was determined by difference. Ultimate analyses were performed on a CARLO ERBA model EA 1108 Elemental Analyzer (Carlo Erba Instruments). Oxygen was determined by difference. Surface areas and pore volumes of the biochars were determined as by the application of the Brunauer–Emmett–Teller (BET) and t-plot analysis software available with instrument (Micromeritics GeminiV). Surface morphologies were visualized by scanning electron microscopy (SEM). Chemical functional groups were determined by Fourier transform infrared spectra (FTIR) using SHIMADZU IR Prestige 21. pH values were measured by adding biochar to de-ionized water in a mass ratio of 1:20. The solution was then hand shaken and allowed to stand for 5 min before measuring the pH with a pH meter Hanna Instruments pH 211 (Inyang et al., 2010). Higher heating values (HHV) were determined by GALLENKAMP Auto Adiabatic Bomb Calorimeter according to the ASTM D240. SEM images were performed using JEOL-JSM-6060LV Scanning Electron Microscope.

3. Results and discussion

3.1. Biochar yields

The effect of pyrolysis temperature and heating rate on the biochar yield is shown in Fig. 1. An increase of pyrolysis temperature and heating rate led to a decrease in the yields of biochar. The decrease in the biochar yields with increasing temperature could either be due to greater primary decomposition or through secondary decomposition of char residues. The high yield of biochar at low temperatures indicates that the material has been only partially pyrolysed (Katyal et al., 2003). The effect of pyrolysis heating rate was more important at low temperatures, while at high temperatures, pyrolysis heating rates showed a similar trend with respect to biochar yields.

At a 400 °C, the yields of biochar changed from 34.18% to 29.70% (4.48% change) as the heating rate increased from 10 to 50 °C min^{-1}; however, at 600 °C, the biochar yield showed only 1.76% change for the some increase in heating rate.

3.2. Proximate analysis

The results of the proximate analysis, pH and higher heating value (HHV) of biochars produced at different pyrolysis temperatures and heating rates are given in Table 1. The volatile matter content of the biochars decreased from 25.20% to 11.60% at a heating rate of 10 °C min^{-1} and decreased from 21.40% to 10.80% for a heating rate of 30 °C min^{-1}. It decreases from 19.80% to 9.80% at a heating rate of 50 °C min^{-1} as the pyrolysis temperature was raised from 400 to 600 °C.

The ash content is a measure of the non-volatile matter and non-combustible component of the biochar. The ash content of the biochars increased from 7.50% to 9.20% for 10 °C min^{-1} runs, it increases from 8.40% to 9.30% for 30 °C min^{-1} runs, and increased from 8.50% to 9.50% for 50 °C min^{-1} runs as the final temperature was raised from 400 to 600 °C. Thus, the ash contents of the biochars were higher than those of cotton stalk, orange peels, and palm waste biochars (Chen et al., 2012; Chen and Chen, 2009; Lua and Guo, 1998).

As the pyrolysis temperature increased from 400 to 600 °C, the fixed carbon content of the biochars increased and fixed carbon contents as high as 79.20–80.70% were achieved for biochar pyrolysis at 600 °C in this study. In similar studies with increasing temperature, fixed carbon content for rapeseed cake, cotton stalk, and palm stones was increased from 57.08% to 73.05%, 52.44% to 84.19% and 31.0% to 85.10%, respectively (Ucar and Ozkan, 2008; Chen et al., 2012; Guo and Lua, 1998).

3.3. Higher heating value

Higher heating value indicates the biochars potential to be used as fuel. The higher heating value of the biochars increased from 28.15 to 30.06 MJ kg^{-1} for the heating rate of 10 °C min^{-1}, it in-

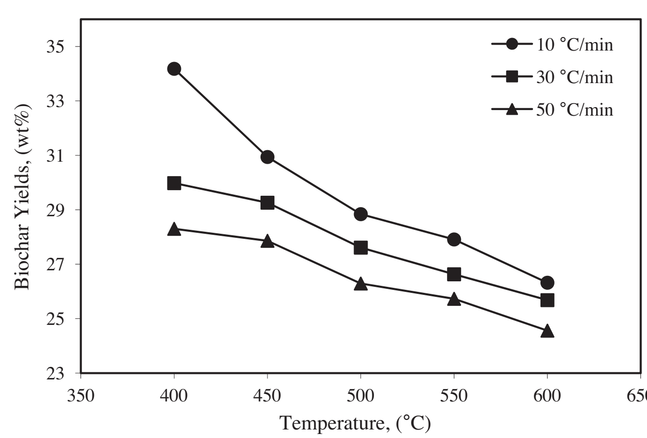

Fig. 1. Effect of pyrolysis temperature and heating rate on safflower seed cake biochar yields.

Table 1
Characteristics of safflower seed cake biochars produced at different pyrolysis temperatures and heating rates.

	Pyrolysis temperature (°C)				
	400	450	500	550	600
Heating rate of 10 °C min^{-1}					
Volatile matter (%)	25.20	20.00	16.50	13.90	11.60
Ash (%)	7.50	8.20	8.50	8.90	9.20
Fixed carbon (%)	67.30	71.80	75.00	77.20	79.20
pH	8.18	9.13	9.44	9.67	9.89
HHV (MJ kg^{-1})	28.15	28.86	29.39	29.71	30.06
Heating rate of 30 °C min^{-1}					
Volatile matter (%)	21.40	18.70	15.20	12.30	10.80
Ash (%)	8.40	8.50	8.60	9.10	9.30
Fixed carbon (%)	70.20	72.80	76.20	78.60	79.90
pH	7.59	8.71	9.52	9.70	10.15
HHV (MJ kg^{-1})	28.51	28.98	29.59	29.97	30.17
Heating rate of 50 °C min^{-1}					
Volatile matter (%)	19.80	17.40	14.30	11.40	9.80
Ash (%)	8.50	8.60	8.70	9.10	9.50
Fixed carbon (%)	71.70	74.00	77.00	79.50	80.70
pH	8.07	8.46	9.30	9.56	9.77
HHV (MJ kg^{-1})	28.77	29.20	29.73	30.12	30.27

creased from 28.51 to 30.17 MJ kg^{-1} for the heating rate of 30 °C min^{-1} and increased from 28.77 to 30.27 MJ kg^{-1} for the heating rate of 50 °C min^{-1} as the pyrolysis temperature was raised from 400 to 600 °C. The higher heating values of the biochars increased with an increase in the pyrolysis temperature, but are nearly the same at different heating rates (Table 1).

The higher heating values of the biochars were similar in comparison with that of other biochars such as those derived from beech trunkbark, cotton stalk, *Cynara carduncului* L. (Demirbaş, 2004; Chen et al., 2012; Encinar et al., 2000).

The HHVs of the biochars were comparable with those of solid fuels ranging from lignite to anthracite (Raveendran and Ganesh, 1996), suggesting the biochars have potential usage as solid fuels.

3.4. PH values

The pH values of the biochars obtained under different pyrolysis temperature and heating rate are given in Table 1. Pyrolysis heating rate did not have a significant effect on pH values, but the pH values of the biochars increased with increasing pyrolysis temperatures. The pH of the biochars were between 7.59 and 10.15, which is similar to values reported for biochar produced from sugar beet tailings, sewage sludge, sugarcane bagasse at high temperature (Yao et al., 2011; Jindarom et al., 2007; Inyang et al., 2010).

3.5. Ultimate analysis

The results of the elemental analysis of biochars obtained at various pyrolysis temperatures and heating rates are shown in Table 2. The heating rate did not have a significant effect on the elemental composition of the biochars. With an increase in the pyrolysis temperature from 400 to 600 °C, the carbon content of the biochars increased from 68.22 to 73.75 wt.%; however, the hydrogen content decreased from 4.07 to 2.31 wt.%, and oxygen content decreased from 24.44 to 20.10 wt.% for all heating rates.

As the temperature rose, H/C and O/C atomic ratios gradually decreased, implying that the biochars became increasingly more aromatic and carbonaceous (Fu et al., 2011). The O/C ratios of the biochars were the lowest at 600 °C, while the O/C ratios of biochars the highest at 400 °C.

In general, high nitrogen content of biochar can provide nutrients to soil and improve crop productivity (Sanna et al., 2011).

Table 2
Elemental analyses of safflower seed cake biochars produced at different pyrolysis temperatures and heating rates.

	Pyrolysis temperature (°C)				
	400	450	500	550	600
Heating rate of 10 °C min^{-1}					
C (wt.%)	68.76	70.43	71.37	72.96	73.72
H (wt.%)	4.07	3.49	2.96	2.67	2.34
N (wt.%)	3.77	3.69	3.91	3.74	3.84
O* (wt.%)	23.49	22.39	21.76	20.63	20.10
H/C (wt.%)	0.71	0.59	0.50	0.44	0.38
O/C (wt.%)	0.26	0.24	0.23	0.21	0.20
Heating rate of 30 °C min^{-1}					
C (wt.%)	68.22	68.65	70.01	71.76	73.75
H (wt.%)	3.43	3.15	2.88	2.68	2.31
N (wt.%)	3.91	4.04	3.83	4.21	3.53
O* (wt.%)	24.44	24.16	23.28	21.35	20.41
H/C (wt.%)	0.60	0.55	0.49	0.45	0.38
O/C (wt.%)	0.27	0.26	0.25	0.22	0.21
Heating rate of 50 °C min^{-1}					
C (wt.%)	68.54	70.25	71.79	72.13	73.16
H (wt.%)	3.67	3.18	2.87	2.81	2.63
N (wt.%)	3.63	3.59	3.33	3.43	3.60
O* (wt.%)	24.16	22.98	22.01	21.63	20.61
H/C (wt.%)	0.64	0.54	0.48	0.47	0.43
O/C (wt.%)	0.26	0.25	0.23	0.22	0.21

Therefore, nitrogen analysis was performed. Nitrogen contents changed in the range of 3.74–3.91%; 3.53–4.21% and 3.33–3.63% for the heating rates of 10, 30, 50 °C min^{-1}, respectively, at the different pyrolysis temperatures.

The elemental content of the SPC biochars were similar to those of biochar produced from other biomass samples such as orange peels, wheat grains, pinewood sawdust, maize stalk, rice and cotton straw (Chen and Chen, 2009; Sanna et al., 2011; Amutio et al., 2012; Fu et al., 2011).

Since the sulfur content of SPC was below the detection limit, the bio-char could be used in fuel applications and activated carbon production.

3.6. FTIR analysis

The FTIR analyses indicated that the functional groups of biochars obtained at different pyrolysis heating rates were very similar and that aromatic and aliphatic groups are predominant (Fig. 2).

The peaks at about 3600 and 3700 cm^{-1} corresponded to vibrations of OH groups and were still detected in the sample treated at 600 °C. The bands at about 2900 and 3000 cm^{-1} corresponded to aliphatic CH_3 asymmetric and symmetric stretching vibration, respectively. The bands in raw materials are clearly seen in Fig. 2. However, the bands disappeared for the samples produced 400 °C and above. The peaks between 1900 and 2300 cm^{-1} indicate carboxyl and carbonly groups. The absorbance peaks between 1415 and 1800 cm^{-1} represent C=C stretching vibrations that are indicative of alkanes and aromatics (Inyang et al., 2010). The peaks between 1050 and 1350 cm^{-1} occurred due to the presence of primary, secondary and tertiary alcohols, phenols, ethers and esters showing C–O stretching and O–H deformation vibrations. The peaks between 630 and 850 cm^{-1} correspond to aromatic C–H stretching vibrations that indicate the presence of adjacent aromatic hydrogens in biochars. These peaks were visible in biochars, but not in raw material (SPC). Generally, the effect of pyrolysis temperature and heating rate on the biochar FTIR profiles are not very pronounced. The increasing upward drift in the baseline of the spectrum from low to high wavenumbers at the high pyrolysis temperature was probably due to the increase in the aromatics content (Hossain et al., 2011).

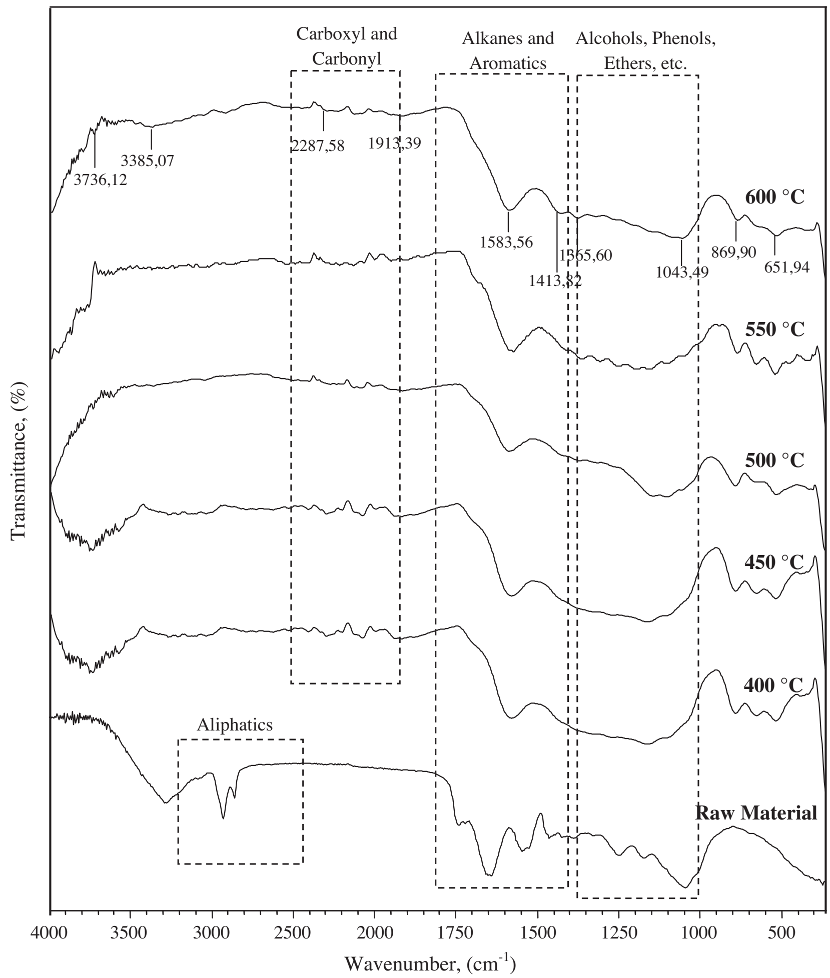

Fig. 2. FTIR spectra of raw safflower seed cake and its biochars obtained at different pyrolysis temperatures and heating rate of 50 °C min^{-1}.

3.7. BET surface areas and pore volumes

The effect of pyrolysis temperature and the heating rate on the BET surface areas and pore volumes (total and micro) of the biochars are shown in Table 3. When the pyrolysis temperature was increased from 400 to 500 °C, the V_{micro}, V_{total} and S_{BET} values increased significantly and reached maxima (4.23 m^2/g, 0.0067 cm^3/g and 0.0080 cm^3/g for the BET surface area, micropore and total pore volumes, respectively). However, at 550 and 600 °C, the trend was reversed. As the pyrolysis temperature rose up 500 °C, the number of micropores significantly increased with the removal of volatile matter, giving rise to an increase in the pore volume and surface area. Above 500 °C, structural ordering, pore widening and/or the coalescence of neighboring pores seem to predominate, leading to a decrease in the pore volume and surface area. Besides, as a result of softening, melting, fusing and carbonization, pores in the biochars might be partially blocked (Fu et al., 2011).

With increasing heating rates from 10 to 50 °C min^{-1}, the BET surface area and pore volume values decreased. This phenomenon can be explained by the release of volatiles at higher heating rates. At lower heating rates, pyrolysis products have sufficient time to diffuse from the particles. With increasing heating rate, the time for the volatiles to be discharged is significantly shortened, leading to an accumulation of volatiles between and within particles. This may increase the chance of carbon deposits blocking pore entrances. As a consequence of this situation, the BET surface area and pore volume values of the biochars decreased as has been observed in other biomass pyrolysis studies (Guo and Lua, 1998; Mui et al., 2010).

3.8. Scanning electron microscopy

Scanning electron microscopy (SEM) was applied to characterize the shape and the size of the biochar particle, as well as their

Table 3
Effect of pyrolysis temperature and heating rate on surface areas and pore volumes of safflower seed cake biochars.

Pyrolysis temperature (°C)	Pyrolysis heating rate								
	10 °C/dk			30 °C/dk			50 °C/dk		
	S_{BET} (m^2/g)	V_{micro} (cm^3/g)	V_{Total} (cm^3/g)	S_{BET} (m^2/g)	V_{micro} (cm^3/g)	V_{Total} (cm^3/g)	S_{BET} (m^2/g)	V_{micro} (cm^3/g)	V_{Total} (cm^3/g)
400	2.67	0.0042	0.0050	2.26	0.0036	0.0043	1.89	0.0029	0.0035
450	3.33	0.0053	0.0063	2.92	0.0046	0.0055	2.71	0.0043	0.0052
500	4.23	0.0067	0.0080	3.98	0.0063	0.0075	3.64	0.0057	0.0069
550	3.78	0.0060	0.0071	3.26	0.0051	0.0061	2.83	0.0045	0.0054
600	3.41	0.0054	0.0064	2.85	0.0049	0.0053	2.47	0.0039	0.0047

porous surface structure. Supplemental Fig. S1 shows SEM photographs of the biochars produced at pyrolysis temperatures of 400, 500 and 600 °C and heating rates of 10, 30 and 50 °C min^{-1}.

The SEM photograph of biochars obtained at 400 and 500 °C many orderly pores over the surface, forming a system of developed pore structures. Due to these well-developed pores, the biochars possessed a high BET surface area and adsorptive capacity. At 600 °C, the biochars had lower BET surface areas due to the shrinkage of chars at post-softening and swelling temperatures, resulting in narrowing or closing pores. The pyrolysis heating rate had no noticeable effect biochar morphology.

4. Conclusion

Pyrolysis studies indicated that it can be achieved a more valuable and functional product (biochar) from safflower seed press cake (SPC) which is only used as animal feed currently. The SPC biochars can be effectively used as a raw material for the preparation of activated carbon. Biochars obtained at high pyrolysis temperatures (600 °C) are suitable for direct use in fuel applications due to their high fixed carbon content, higher heating value, and low volatile matter content. Biochars can also be evaluated as a chemical feedstock for industrial purpose.

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