

Effects of Torrefaction on Carbonization Characteristics of Solid Olive Mill Residue

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Torrefaction is an important biomass pretreatment method that impacts fuel characteristics of biomass, specifically during the torrefaction process. Besides improving the fuel characteristics of biomass, torrefaction also contributes to increased quality of liquid and gaseous energy carriers obtained from fast pyrolysis and gasification. In this study, the effect of torrefaction on the solid energy carrier biochar, produced by carbonization, was studied by using solid olive mill residue (SOMR) as raw biomass. The carbonization characteristics of SOMR and torrefied SOMR (tSOMR) were compared by using ultimate and proximate analysis results. The higher heating value (HHV) and energy yields of biochars produced from SOMR and tSOMR were compared. The results showed that torrefaction contributed to the reduction of energy given to the biomass during the carbonization process by decreasing the holding time.

Keywords: Biochar; Biomass; Carbonization; Pretreatment; Solid olive mill residue; Torrefaction

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INTRODUCTION

Torrefaction is a thermochemical process that can be used to improve the fuel characteristics of biomass fuels. The process occurs in an inert atmosphere between 200 and 300 °C. Torrefaction gained importance in the last decade because of its demonstrated impacts on biomass. This process overcomes important drawbacks associated with raw biomass. Torrefaction experiments with different biomass samples have shown that it produces solid hydrophobic fuel with reduced moisture content (Felfli *et al.* 2005; Sadaka and Negi 2009), increased energy density (Prins *et al.* 2006a; Yan *et al.* 2009; Rousset *et al.* 2011), and increased higher heating value (HHV) (Bridgeman *et al.* 2008; Couhert *et al.* 2009; Deng *et al.* 2009; Yan *et al.* 2009).

The main product of torrefaction is solid fuel, which can be directly combusted for energy generation. Torrefied biomass can also be used as fuel for other thermochemical biomass treatment processes, such as pyrolysis and gasification. The effect of torrefaction on these processes has been investigated widely. Neupane *et al.* (2015) tested the effect of torrefaction on the fast pyrolysis characteristics of biomass, showing that the chemical composition of bio-oils produced from fast pyrolysis was improved by lowering its oxygen content and enhancing the aromatic yield (Neupane *et al.* 2015). Zheng *et al.* (2013) showed that a torrefaction as a thermal pretreatment can improve the quality of bio-oil obtained from fast pyrolysis of corn cobs. Sarkar *et al.* (2014) studied the effect of torrefaction on the gasification of switchgrass; their results showed that torrefaction remarkably improved the gas yields, syngas lower heating value, and gasifier efficiencies

(Sarkar *et al.* 2014). Daniyanto *et al.* (2015) showed that pretreatment torrefaction improves the quality of Indonesian sugarcane bagasse as a feedstock of gasification. Despite the proven merits of torrefaction on gasification and fast pyrolysis, the effect of this process on carbonization has not yet been studied.

Torrefaction and carbonization are both thermochemical treatments applied to biomass. The processes are very similar; both occur in the absence of oxygen with small heating rates. The main difference between carbonization and torrefaction is the process temperature. Carbonization occurs above 300 °C (Sadaka *et al.* 2014), and torrefaction occurs between 200 and 300 °C. The main product of both processes is the solid energy carrier, which may be regarded as biochar. However, biochar produced from carbonization and torrefaction have different fuel properties. The biochar produced by carbonization contains higher carbon and less oxygen, and it has a greater higher heating value (HHV) and lower volatile matter content. All of these values are indicators of a more qualified solid fuel.

In this study, the effect of torrefaction on the carbonization characteristics of biomass was investigated by using solid olive mill residue (SOMR). SOMR is lignocellulosic agricultural waste obtained from olive oil extraction. The effect of torrefaction on the carbonization characteristics of SOMR was tested with both raw SOMR and torrefied SOMR (tSOMR). The biochars produced from SOMR and tSOMR were characterized by ultimate and proximate analysis to clarify the effect of torrefaction on the carbonization characteristics.

EXPERIMENTAL

SOMR was supplied by the local Aydın Olive Mill Company (3-phase), Akçay, Cyprus. SOMR was dried for 8 h prior to experiments.

Figure 1 shows the schematic diagram of the torrefaction/carbonization equipment. The equipment consisted of an electric heater and a glass tube. The glass tube, with radius of 0.02 m and height of 0.29 m, was placed into the electric heater (Velp Scientifica, Milan, Italy), which had a depth of 0.065 m. The electric heater was adjusted to heat the samples to the desired carbonization temperatures (CT) with a heating rate of 15 °C/min. A K-type thermocouple was inserted into the glass tube for measuring the torrefaction temperature inside the tube. Each carbonization experiment was conducted with 5 g of dry SOMR. The torrefaction/carbonization processes were carried out under a nitrogen flow of 20 mL/min. The oxygen inside the glass tube was taken out by flowing nitrogen at 50 mL/min for 10 min.

Carbonization experiments were conducted in two different sets to clarify the effect of torrefaction on the carbonization characteristics of SOMR. The first set included direct carbonization experiments, which were conducted with dry SOMR at CTs of 350 °C, 400 °C, and 450 °C, with holding times of 20 min. The holding time refers to the time that the SOMR was held at the associated CT and does not include heating time. The second set of carbonization experiments was conducted with torrefied SOMR (tSOMR). Dry SOMR was first torrefied at 240 °C for 10 min, and then the process temperature was raised to the CT. During this process, the CT was kept at 350 °C, 400 °C, and 450 °C, with a holding time of 10 min to produce the biochar from tSOMR.

TGA analysis of SOMR was also conducted with a Mettler Toledo TGA/DSC 1 Star System (Switzerland) under inert atmosphere (nitrogen), for which the specimens were

at a rate of 20 °C/ min. Biochars obtained from the direct carbonization of SOMR at 350 °C, 400 °C, and 450 °C are represented by 350, 400, and 450, respectively, in the figures and tables. Similarly, biochars produced from tSOMR at 350 °C, 400 °C, and 450 °C are represented by 240+350, 240+400, and 240+450, respectively, in the figures and tables. Carbonization experiments with SOMR and tSOMR were repeated twice at each CT, and results are expressed as average. It must be pointed out that the results do not deviate more than 3% from each average.

The dry-basis elemental composition of the produced biochars was obtained by ultimate analysis with a Costech ECS 4010 series element analyzer (Italy). The proximate analysis was carried out in a muffle furnace, which gave the volatile matter (VM), fixed carbon (FC), and ash content composition in wt% of the sample dry basis.

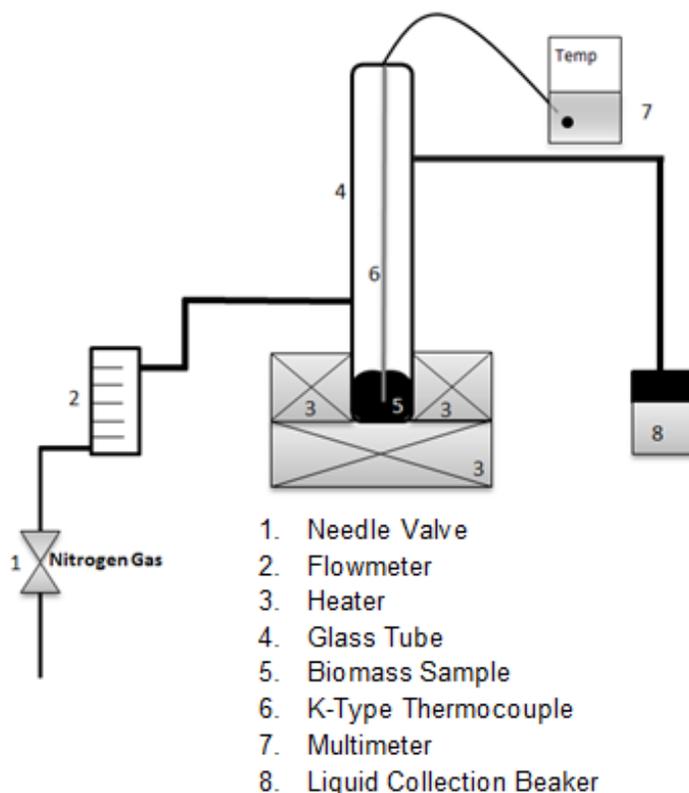


Fig. 1. Torrefaction/carbonization equipment

The mass yield, energy yield, and HHV (MJ/kg) of the produced charcoals were calculated according to Eqs. 1, 2, and 3 (Demirbaş *et al.* 1997):

$$\text{Mass Yield} = \frac{\text{Mass of solid product}}{\text{Mass of raw olive pomace}} \times 100 \quad (1)$$

$$\text{Energy Yield} = \text{Mass Yield} \times \frac{(\text{HHV})_{\text{Torrefied SOMR}}}{(\text{HHV})_{\text{Raw SOMR}}} \times 100 \quad (2)$$

$$\text{HHV (MJ/kg)} = 0.335\text{C (wt\%)} + 1.42\text{H (wt\%)} - 0.154\text{O (wt\%)} - 0.145\text{N (wt\%)} \quad (3)$$

where C is carbon, O is oxygen, H is hydrogen, and N is nitrogen, as measured in wt%.

RESULTS

Mass Yield

The dry-basis biochar yield of each carbonization process is given in Fig. 2. Mass yield measurements showed that the mass yield decreased with increased CT. The mass yield decreased to 33.18 wt% from 38 wt% when the CT had risen from 350 °C to 450 °C. However, the biochar yield of tSOMR was higher than that of directly carbonized SOMR. The biochar yield of tSOMR was 44%, and the biochar yield of SOMR was 38% at a CT of 350 °C.

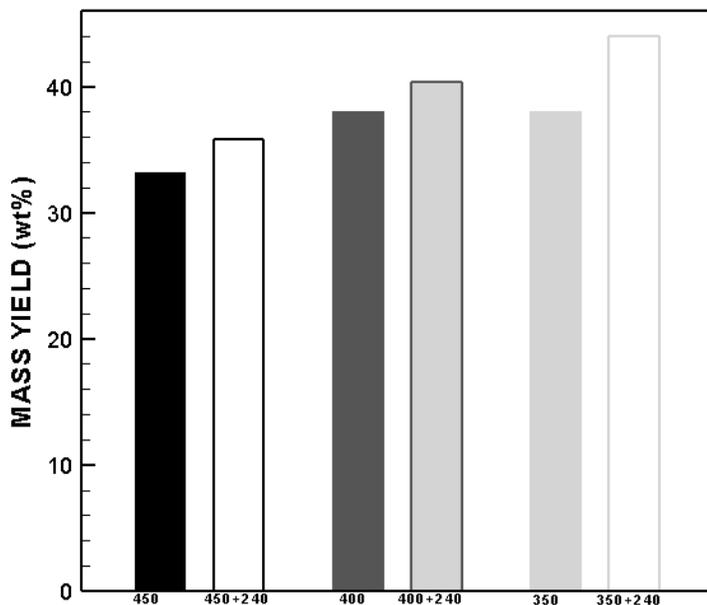


Fig. 2. Mass yield of biochar produced from SOMR and tSOMR

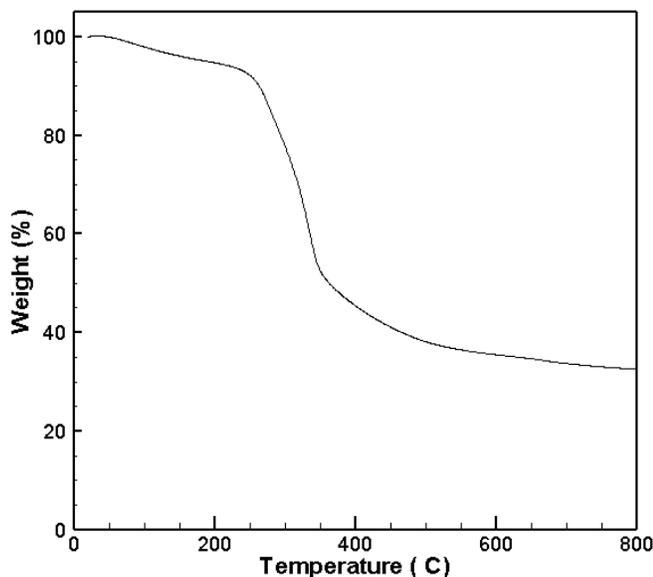


Fig. 3. TGA analysis of SOMR

Similarly, at a CT of 400 °C, the biochar yield of tSOMR was 40.40%, where the biochar yield of SOMR was 36%. Similar behavior was also obtained at 450 °C, and mass yields of 33.18% and 35.8% were obtained from carbonization of SOMR and tSOMR, respectively.

The mass yield reduction during carbonization was attributed to the thermal effect that resulted in the loss of moisture and was followed by the depolymerization of the cell wall constituents, *i.e.*, hemicellulose, cellulose, and lignin (Yang *et al.* 2006). Balat *et al.* (2009) reported that biomass depolymerization typically resulted in the release of carbon monoxide, carbon dioxide, and water and in the formation of aromatic carbon structures. The reduced mass yield during the torrefaction process also is a result of bound moisture removal and thermal degradation to form volatile products such as H₂O, CO, CO₂, H₂, acetic acid, and other organics (Prins *et al.* 2006b).

The pyrolytic behavior of hemicellulose, cellulose, and lignin were investigated by Yang *et al.* (2007). The degradation of hemicellulose and cellulose occurred between 220 °C and 315 °C and 315 °C and 400 °C, respectively (Yang *et al.* 2007). However, the same report also revealed that lignin was the most difficult constituent to decompose; its decomposition occurred slowly between 400 °C and 900 °C (Yang *et al.* 2007).

SOMR has non-homogenous structure and its composition varies according to the olive species, culture conditions, origin of the olives, and extraction process (Dermechea *et al.* 2013). Vlyssides *et al.* (1998) stated that cellulose, hemicellulose, and lignin content of SOMR varies within ranges of 17.37 to 24.14%wt, 7.92 to 11.00%wt, and 0.21 to 14.18%wt, respectively.

The thermal degradation behavior of SOMR was evaluated with TGA analysis, and results are shown in Fig. 3. The mass of SOMR was observed to change continuously up to 200 °C. Furthermore, mass reduction became more remarkable between 270 °C and 485 °C, where severe devolatilization occurs.

In this study, the reduction in mass yield at CTs of 350 °C and 400 °C was mainly caused by cellulose and hemicellulose degradation. In addition, at a CT of 450 °C, lignin decomposition contributed to a reduction in mass yield (Poudel and Oh 2014).

During the carbonization of tSOMR, removal of light volatiles and decomposition of xylan and hemicellulose contributed to mass loss during the torrefaction stage. Increasing the CT to 350 °C, 400 °C, and 450 °C resulted in more effective decomposition of cellulose and hemicellulose.

Carbonization experiments with different biomass materials have shown that increased holding time results in lower mass yield during carbonization (Sadaka *et al.* 2014). The decomposition of cellulose and lignin are two constituents that are significantly affected by holding time. Rantuch and Chrebet (2014) showed that rising holding time from 10 min to 20 min significantly changes the mass yield of cellulose carbonization. Also Brebu and Vasile (2010) stated that lignin decomposes slowly at temperatures above 400 °C. In this study, SOMR was carbonized for 20 min and tSOMR was carbonized for 10 min at a CT of 350 °C, 400 °C, and 450 °C. In this study, higher holding time during the carbonization of SOMR at each associated CTs resulted in more effective decomposition of cellulose and lignin, and it resulted in a lower mass yield compared to tSOMR.

Ultimate Analysis Results

Ultimate analysis results of carbonized SOMR and tSOMR are given in Table 1. Carbonization of SOMR yielded biochar with reduced hydrogen and oxygen contents

compared with raw SOMR. Carbonization also yielded biochar with higher carbon content. Elemental composition of carbonized SOMR showed a more pronounced reduction in hydrogen and oxygen content. The carbon content increased with an increased CT. Both torrefaction and carbonization resulted in reduced hydrogen and oxygen content, which mainly corresponded with destroyed hydroxyl groups (–OH) in the biomass (Bergman *et al.* 2005; Phanphanich and Mani 2011; Giudicianni *et al.* 2013).

Ultimate analysis showed that tSOMR biochars had less hydrogen content than raw SOMR biochars. The carbon content measurements showed that, although there was no significant difference, tSOMR biochars had higher carbon content compared to SOMR biochars at CT of 350 °C and 400 °C. However, the carbon content of tSOMR biochars at CT 450 °C contained less carbon where the difference was around 1.29% (not regarded as statistically significant).

The oxygen contents of SOMR and tSOMR biochars were compared. At a CT of 350 °C, tSOMR yielded biochar with 1.02 wt% higher oxygen content than SOMR. The opposite trend was obtained at a CT of 400 °C and 450 °C. Results showed that tSOMR biochar contained 0.02% and 0.73% less oxygen relative to SOMR biochar, respectively.

Ultimate analysis results demonstrated that both direct carbonization of SOMR and tSOMR remarkably changed the elemental composition of raw SOMR. Furthermore, carbonization of both SOMR and tSOMR yielded changes in the H/C and O/C ratios of raw SOMR. The H/C and O/C ratios of SOMR and tSOMR biochars are demonstrated in Figs. 4 and 5, respectively.

Experimental results showed that tSOMR biochars had a lower H/C ratio. The H/C ratio of tSOMR biochars was 0.82, whereas the H/C ratio of SOMR biochars was 0.89 at a CT of 450 °C. At a CT of 400 °C, SOMR biochar had an H/C ratio of 1.12, whereas the H/C ratio of tSOMR biochar was 1.10. A similar effect was observed on the H/C ratios of biochars at the CT of 350 °C. The H/C ratio of biochar from tSOMR was 1.13, whereas the H/C ratio of directly carbonized SOMR was 1.17 at the CT of 350 °C.

Table 1. Ultimate and Proximate Analysis Results

Sample	C (wt%)*	H (wt%)*	N (wt%)*	O (wt%)*	Ash (wt%)*	VM (wt%)*	FC (wt%)*
450	65.54	4.86	1.61	15.98	12.01	46.66	41.33
450+240	64.25	4.41	1.39	15.25	14.70	47.00	38.30
400	63.07	5.90	1.73	17.80	11.50	47.12	41.38
400+240	63.39	5.85	1.80	17.78	11.23	48.90	39.87
350	62.04	6.08	2.02	18.64	11.22	54.00	34.78
350+240	63.01	6.02	1.80	19.67	9.50	60.00	30.50
Raw SOMR	47.62	6.50	1.82	39.82	4.20	88.84	6.96

*dry basis, C: Carbon, H: Hydrogen, N: Nitrogen, O: Oxygen, VM: Volatile Matter, FC: Fixed Carbon

Carbonization experiments also revealed that tSOMR biochars had a higher O/C ratio at CT of 350 °C and 400 °C. At a CT of 350 °C, the O/C ratio of tSOMR biochar was 0.23, and the O/C ratio of SOMR biochar was 0.21. At a CT of 400 °C, SOMR biochars

had an O/C ratio of 0.20, whereas the O/C ratio of tSOMR biochar was 0.21. However, the O/C ratio of both tSOMR and SOMR biochars were the same (0.18).

The reduction in the H/C ratio implied higher structural stability in biochars compared with uncharred materials, due to increased aromatic structures developed by charring (Kloss *et al.* 2012; Schimmelpfennig and Glaser 2012). In addition, the decreased O/C ratio indicated a higher-degree of carbonization due to the elimination of hydrophilic functional groups (Keiluweit *et al.* 2010; Schimmerpfennig and Glaser 2012).

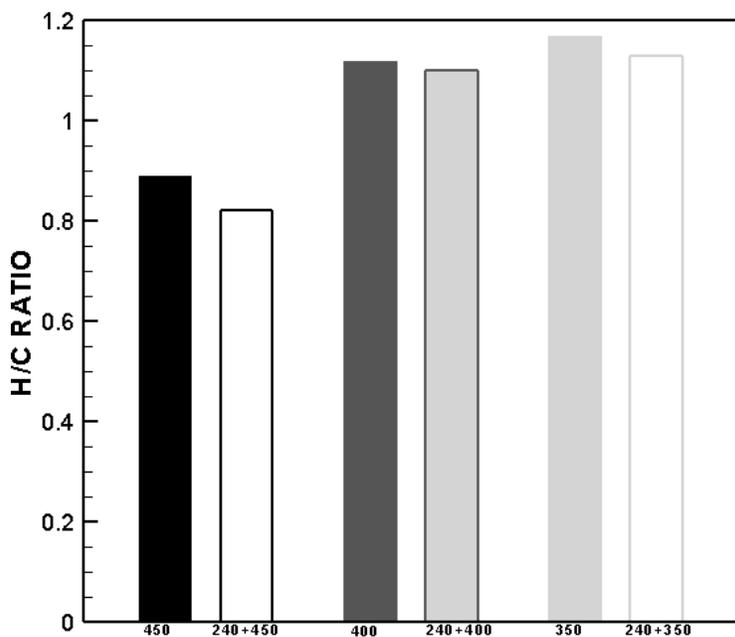


Fig. 4. H/C ratio of biochar produced from SOMR and tSOMR

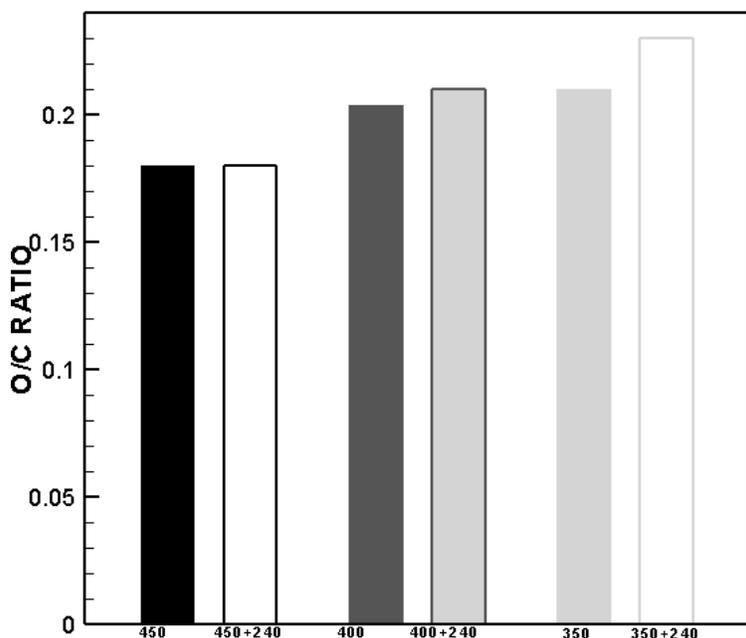


Fig. 5. O/C ratio of biochar produced from SOMR and tSOMR

Proximate Analysis Results

The proximate analysis results of biochars produced from raw SOMR and tSOMR are shown in Table 1. The ash and fixed carbon content of biochars increased with an increased CT. However, the volatile matter content of biochars showed a different trend.

tSOMR yielded biochar with less ash at CT of 350 °C and 400 °C. However, the biochar produced from carbonization of tSOMR at 450 °C contained more ash compared to SOMR. High ash content inhibits the combustion of fuel because oxygen may not easily penetrate through the ash (Syamsiro *et al.* 2012). It is estimated that higher ash content of tSOMR carbonized at 450 °C is a result of non-homogenous structure of SOMR. The ash content measurements showed that torrefaction of SOMR before carbonization ameliorated combustion problems at lower CT. Proximate analysis results also demonstrated that tSOMR yielded biochar with increased VM. Complete combustion of VM leads to dark smoke, heat loss, and pollution hazards (Patel and Gami 2012).

The FC content of tSOMR, which is the amount of the carbon present in the biochar sample (Pandey and Dhakal 2013), followed a trend similar to ash content. tSOMR carbonization resulted in biochar with less FC content than SOMR at both CTs.

Energy Yield and Higher Heating Value

The potential use of biochar as a fuel is defined by the HHV value (Hmid *et al.* 2014). The HHV of all produced biochars in a dry basis are given in Fig. 6. Carbonization experiments done by Volpe *et al.* (2014) showed that at carbonization temperatures exceeding 400 °C, the gross calorific value of biochar produced from olive pomace decreased. Consistent with the study of Volpe *et al.* (2014), the HHV of biochar increased when the CT risen to 400 °C from 350 °C. Nevertheless, biochar with the lower HHV of 26.16 MJ/kg was obtained at the CT of 450 °C. At the CT of 350 °C, the HHV of tSOMR biochar was 26.30 MJ/kg, and the HHV of SOMR biochar was 26.54 MJ/kg. However, at the CT of 400 °C, the HHV of tSOMR biochar was 26.65 MJ/kg, and the SOMR biochar was 26.61 MJ/kg at 400 °C. Also, biochar with lower HHV was obtained from tSOMR compared to SOMR at 450°C.

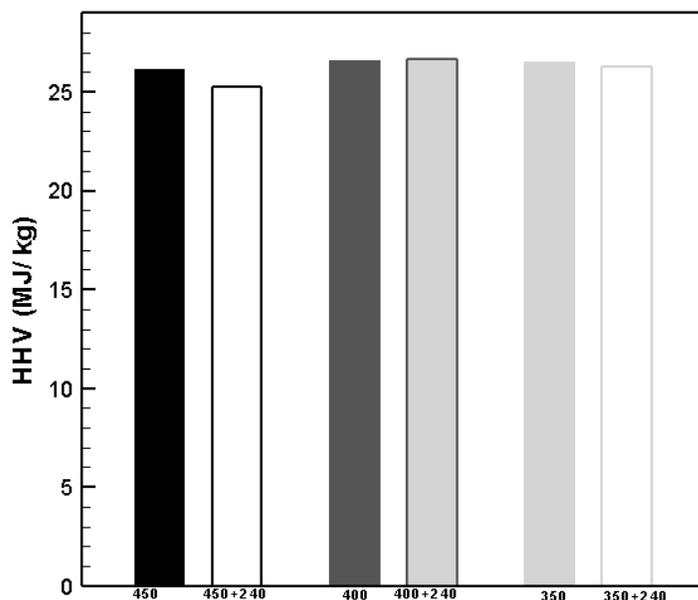


Fig. 6. HHV of biochars produced from SOMR and tSOMR

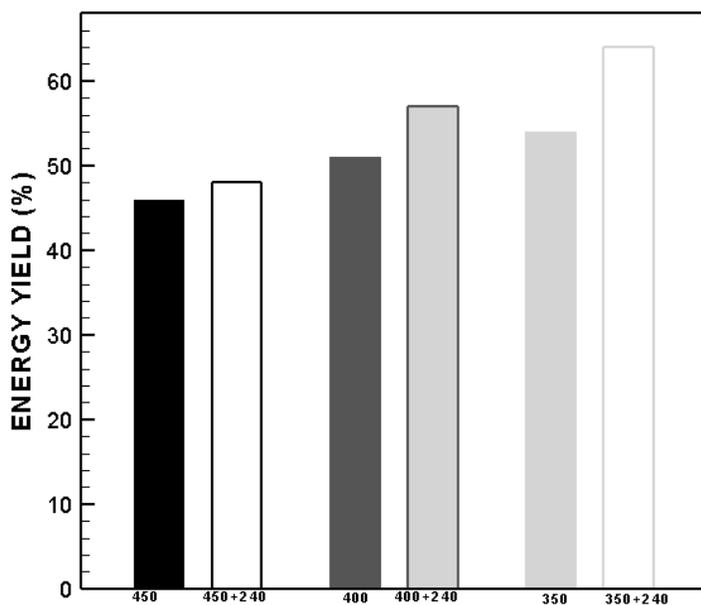


Fig. 7. Energy yield of biochars produced from SOMR and tSOMR

Besides HHV, the energy yield was calculated on a dry basis (Fig. 7). Energy yield is the main indicator of the amount of energy retained after the thermal process (Wannapeera *et al.* 2011). The energy yield of biochars decreased with increased CT, from 54% to 46% when the CT was raised to 450 °C from 350 °C. tSOMR biochars had a higher energy yield. At a CT of 350 °C, the energy yield of tSOMR biochar was 64% and the SOMR biochar was 54%, whereas at 400 °C, the energy yield of tSOMR and SOMR were 57% and 51%, respectively. Similar behavior in energy yield also were observed at a CT of 450 °C. Higher energy yield from carbonization of tSOMR is a result of higher mass yield from carbonization of tSOMR compared to SOMR.

Effect of Torrefaction on the Carbonization Characteristics of SOMR

The effect of torrefaction on the carbonization of SOMR was investigated at three carbonization temperatures. Elemental analysis showed that torrefaction of SOMR before carbonization yielded a solid fuel with higher carbon at lower CT and less hydrogen content at all the studied CT values. The effect of torrefaction on the elemental composition of SOMR was more remarkable at lower CT.

The H/C and O/C ratios are two important parameters associated with biochars produced by carbonization. Ultimate analysis results revealed that torrefaction contributed to reduced H/C. Also, the O/C ratios of tSOMR biochars produced were very close to the O/C ratios of SOMR biochars. Generally, lignocellulose with low H/C and O/C ratios is a good fuel because it has low energy loss and emits little smoke or water vapor during combustion (Liu *et al.* 2013).

Proximate analysis of produced biochars demonstrated that tSOMR biochars contained less ash when they had been prepared at lower carbonization temperatures. Lower ash content provided more efficient combustion for tSOMR biochar compared with SOMR biochar. tSOMR yielded biochar with higher volatile matter content, where the difference did not exceed 6% with SOMR biochar.

Beyond the ultimate and proximate analysis results, torrefaction had considerable effect on the energy yield of produced biochars. Energy yield calculations showed that more energy was retained in biochar if the SOMR was torrefied before carbonization.

In this study, the holding time used for carbonization of tSOMR at both CTs was half of the holding time used for the carbonization of SOMR. Sadaka *et al.* (2014) and Shaaban *et al.* (2014) have shown that, holding time significantly changes the elemental composition of biomass. However, the produced biochars from SOMR and tSOMR had very similar ultimate and proximate analysis characteristics. Thus, torrefaction significantly contributes to improve fuel characteristics of biomass during carbonization. Nevertheless, torrefaction, can be used to reduce the holding time during carbonization, which then requires less energy input to the carbonization system.

CONCLUSIONS

1. The effect of torrefaction on carbonization was investigated by using SOMR at three different carbonization temperatures.
2. The carbonization of torrefied SOMR yielded biochar with higher carbon content than did directly carbonized SOMR at lower CT. Also, at lower carbonization temperature, tSOMR yielded biochar with a higher HHV. The HHV and energy yield calculations revealed that more energy was retained in biochar produced from tSOMR.
3. tSOMR yielded biochar with lower FC content, higher VM content at all CT₂, and also lower ash content at 350 °C and 450 °C.
4. At lower carbonization temperature of 350 °C, the effect of torrefaction was much more significant. Biochar produced from tSOMR can be regarded as having higher quality, as it had higher carbon, HHV, and energy yield, and less ash content at a low CT of 350 °C.
5. Torrefaction contributed to reduced holding time during the carbonization of SOMR.

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