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Effect of Torrefaction on Energy Properties of Millet Stalk

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A R T I C L E I N F O	A B S T R A C T
	The paper presents tests connected to the torrefaction of agro-biomass residues as a case of biomass valorisation. The aim of the work is to compare the changes in energy and chemical properties of millet stalk (*Panicum miliaceum* L.) before and after the torrefaction process. The torrefaction of the millet stalk was done by using a scale reactor in two temperatures, 275°C and 300°C, in an N_2 atmosphere. The millet stalk torrefied at 300°C has more promising parameters, i.e., higher heating value HHV 24,57 MJ/kg, the content of carbon 64,90% and energy density 1,42 compering to biochar produced at 275°C - 22,57 MJ/kg, 60,90% and 1,31 respectively. The results showed that torrefaction improves the parameters of the millet stalk for higher-quality biofuel, which can be used for heat generation.

Torefaksiyonun Akdarı Saplarının Enerji Özellikleri Üzerine Etkisi

M A K A L E B İ L G İ S İ	Ö Z
	Makale, tarımsal biyokütle kalıntılarının parçalanmasına bağlı test sonuçlarının değerlendirilmesini anlatmaktadır. Çalışmanın amacı, akdarı sapının (*Panicum miliaceum* L.) torrefaksiyon (kömürleştirme) işleminden öncesi ve sonrasındaki, enerji ve kimyasal özelliklerindeki değişiklikleri karşılaştırmaktır. Akdarı saplarının kömürleştirme işlemi, 275°C de ve 300°C de olmak üzere iki farklı reaktör sıcaklığında ve ortama kontrollü N_2 uygulanmak sureti ile gerçekleştirilmiştir. Çalışma sonucunda, akdarı saplarından, 300°C de torifiye (kömürleştirilen) edilerek hazırlanan numunelerin 275°C de elde edilen biyoyakıt örneklerine göre daha ümit verici parametrelere sahip olduğu ve üst ısıtma değerinin (HHV) 24,57 MJ / kg, karbon içeriğinin % 64,90 ve enerji yoğunluğunun 1,42 olarak gözlendiği, 275°C de elde edilen biyo yakıtın, yine sırasıyla üst ısıtma değeri (HHV) 22,57 MJ / kg, karbon içeriği % 60,90 ve enerji yoğunluğu % 1,31 olarak tespit edilmiştir. Bu sonuçlar, akdarı sapının biyoyakıt parametre özellikleri açısından, ısı üretimi için torrefaksiyon (kömürleştirme) işleminin, materyalin kullanılabilirliğinin, daha yüksek kaliteli biyoyakıt elde edilmesinde iyileştirici bir etmen olduğunu göstermiştir.

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Introduction

Biomass residues from the agro-industry are one of the most attractive sources of bioenergy. It seems to be an important source to meet the growing energy needs for electricity and heating.

It is estimated that the agricultural sector generates around 30% of total waste, with the majority of organic waste (FAO, 2021).

According to the European Biomass Association (Bioenergy Europe, 2021), it can be stated that 146,000 kt/year of dry residues could be sustainably collected in Europe, which is a source of potential energy production of 55 Mt.

Turkey has great potential for agriculture due to 23.07 million ha of arable land. It is estimated that biomass could theoretically cover almost a third of Turkey's annual electricity consumption. It is assumed that the total amount of solid agricultural waste is about 50-65 million tonnes (Yilmaz et al., 2018).

Biomass residues from agro-industry, for example, wheat straw, corn stalks, sugarcane bagasse, and fruit and vegetable peels, can be used as a source of energy through various forms of bioenergy production such as anaerobic digestion, combustion, and gasification (Wzorek et al., 2021). It can be done under one condition that the production of energy from biomass residues should proceed without harm to food production.

Agricultural residues can potentially provide energy, but their effect on energy efficiency conversion in thermochemical and biological processes depends on their chemical and energy. To improve this effect, methods of biomass pretreatment are used to improve its energy properties i.e., drying, compacting, and recently also torrefaction.

Torrefaction is a process of heating soild biomass in an inert atmosphere 200–300ºC to improve biomass properties (Barkov et al., 2019). This process results in a reduction of moisture content, an increase in energy density, and a decrease in the ash content of the material. During torrefaction, biomass undergoes thermal decomposition of hemicellulose, cellulose, and some parts of lignin. This decomposition process has been extensively investigated by many researchers (Bridgeman et al., 2008; Chen et al., 2014; Simonic et al., 2020).

Torrefaction has been widely applied to wood biomass over the past few years. However, there are several research papers that have been published about the torrefaction of agricultural waste. For example, Chen et al. 2014a woked about the thermal behavior of oil palm fiber and eucalyptus during this process, Chen et al. (2014) focused on torrefaction of cotton and corn stalk, Szufa et al. (2020) ine oat and maize straws and Zhang et al. (2018) about torrefaction of rice husk evaluating the influence of the torrefaction conditions on its properties and structures.

Torrefied biomass can also be used for a purpose other than energy application. For example, the conversion of agricultural residues to biochar for soil improvement is a highly discussed subject currently. According to Osman et al. (2022) biochar from agricultural waste can improve the properties of soil in terms of water-holding capacity, aeration, nutrient fixation, and well as permeability and swelling. It is also stated that torrefied biomass reduces drought by increasing soil water content and reducing soil erosion (Oni et al., 2019).

However, it is worth noting that the torrefaction process still needs further research to optimize the process and make it more economically viable. In addition, for each type of biomass residues, optimal process parameters should be selected to obtain the most favourable product properties.

Due to the fact that there are not many studies focused on the torrefaction of millet stalk, it was decided to subject them to torrefaction studies.

Millet is a major type of grain crop that is grown in many parts of the world, particularly in Asia and Africa. Some of the top producers of millet are India, China and Nigeria. According to FAO (FAO, 2021), global millet production was estimated at 30.5 million metric tons, and 5.7 million tonnes of millet was produced in Turkey in 2020 (TURKSTAT, 2021).

Millet stalks makeup about 8% of the residues from millet production and can be a raw material to produce biochar.

The aim of the study is to is to evaluate the effect of the torrefaction process, especially the temperature of the process, on the energy and chemical parameters of millet stalk (*Panicum miliaceum* L.). The results obtained from this study can provide valuable information on the potential for millet stalk as a biofuel feedstock.

Materials and methods

Material

Millet stalk was collected after harvesting and cut into 3–5 cm segments. To investigate the impact of irrigation level on energy parameters of raw millet stalk, two samples were used in the research: sample R-I belongs to no irrigation (0%) level treatment, and sample R-II belongs to 125% irrigation level treatment, the properties of the millet stalk were analysed before and after the torrefaction process. The field study of this research was performed in Aydın Adnan Menderes University, Faculty of Agriculture, Biosystem Engineering experiment was done in 2021.

Methods
Energy properties
Raw and torrefied samples were subjected to proximate and ultimate analyses according to the following procedures.
Moisture content
The content of moisture was measured at 105ºC according to the EN ISO 18134 standard.

The moisture (M) of the materials was calculated using the formula (1):

$$M (\%) = (m_1 - m_2/m_1)\, 100 \qquad (1)$$

where: m_1 is the weight of the moist sample and m_2 is the weight of the dry sample.

After drying, samples were milled and sieved to obtain 1 mm particles.

Ash

The ash content (A) was tested according to EN ISO 18122 standard. The measurement was performed in a muffle at 550ºC where samples stayed for 3 hours.

Ash content was calculated using equation (2):

$$A\ (\%) = (m_a/m_t)\ 100 \tag{2}$$

where: m_a is the mass of the sample after heating, and m_t is the mass before heating.

Voltaire matter

Measurement of voltaire matter (VM) was carried out according to EN ISO 18123 standard. Samples in closed vessels were heated to a temperature of 900ºC for 5 minutes.

The volatile matter content was calculated using the following formula:

$$VM\ (\%) = (m_v/m_t)\ 100 \tag{3}$$

where: m_v is the sample weight after heating and m_t is the sample weight before heating.

Fixed carbon

Fixed carbon (FC) content is obtained by difference, applying the formula:

$$FC = 100 - VM - A \tag{4}$$

Elementary analysis

C (carbon), H (hydrogen), and (N) nitrogen content was carried out using Elementary Vario Macro Cube analyser according to EN ISO 16948 standard. The EN ISO 16994 was applied to determinate the sulphur (S) content. The concentration of oxygen (O) was established from mass balance by the difference.

The higher heating value

The higher heating value (HHV) was estimated by the IKA Calorimeter C 200 using EN 14918:2010 and ISO 1928 standards.

Torrefaction procedure

The raw material samples were milled and sieved to obtain a size of 250 μm. The sample of 10 g +/- 0.1 g was placed in the torrefaction reactor and purged with nitrogen (N_2) for 10 min at a flow rate of 50 dm^3/min. Initiation of the reactor heating system with the gas flow rate reduction to 10 l/min. The tests were performed for 45 minutes at temperatures 275 and 300°C.

The samples that were analysed were named T-1(275) and T-2(300), according to the temperature at which the process was performed.

Three repetitions were performed for each torrefaction temperature. Before and after the torrefaction test, the sample was weighed to determine mass loss.
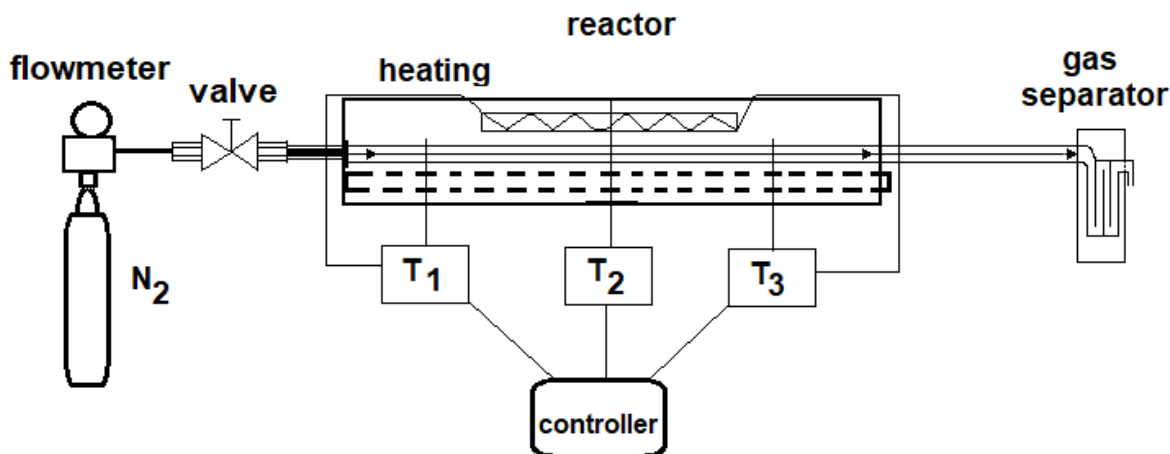

Figure 1. Scheme pf torrefaction reactor.

Results and discussion

The parameters of the raw and the torrefied millet stalk are shown in Table 1 and Table 2.

Analyzing the properties of raw materials (R-1 and R-2), it can be stated that no impact of irrigation method on the fuel properties of millet stalk was observed. The results of proximate and ultimate analyses as well as higher heating value (HHV) are very similar (Table 1). Therefore, only sample R-1 was subjected to torrefaction. Furthermore, a significant difference can already be seen between the properties of raw and torrefied materials. The content of volatile matters has been reduced, which has resulted in an increase in ash (A) and also fixed carbon (FC). An observed increase in ash content of torrefied millet stalk was mainly due to mass loss during the process becouse of mass concertation.

Additionally, an increase in HHV has also been observed. It can be noticed that the higher heating value (HHV) of the torrefied materials increases with the increase of the process temperature. For example, the sample torrefied at 300ºC (T2-300) has about 8.14% higher heating value than the sample torrefied at 275ºC (T1-275). A similar effect was obtained by Poudel et al. (2014) for the torrefaction of corn stalks, as well as by Chen et al. (2014) for the cotton stalks.

Torrefaction allows to obtain a biofuel with higher carbon and lower oxygen content comparing to the raw biomass (Table 2). The phenomena of decreasing O and H can be explained with the removal of volatile components, which contain these atoms during the process.

Table 1. Fuel properties of raw and torrefied millet stalk

Samples	Proximate analysis, wt% d.m.				HHV MJ/kg
	M	A	VM	FC	
R-1	7.12	4.64	77.33	18.03	17.15
R-2	7.02	4.62	77.74	17.64	17.30
T-1 (275)	2.98	11.14	51.57	37.29	22.72
T-2 (300)	2.80	11.69	42.12	46.19	24.57

Table 2. Elementary analysis of raw and torrefied millet stalk

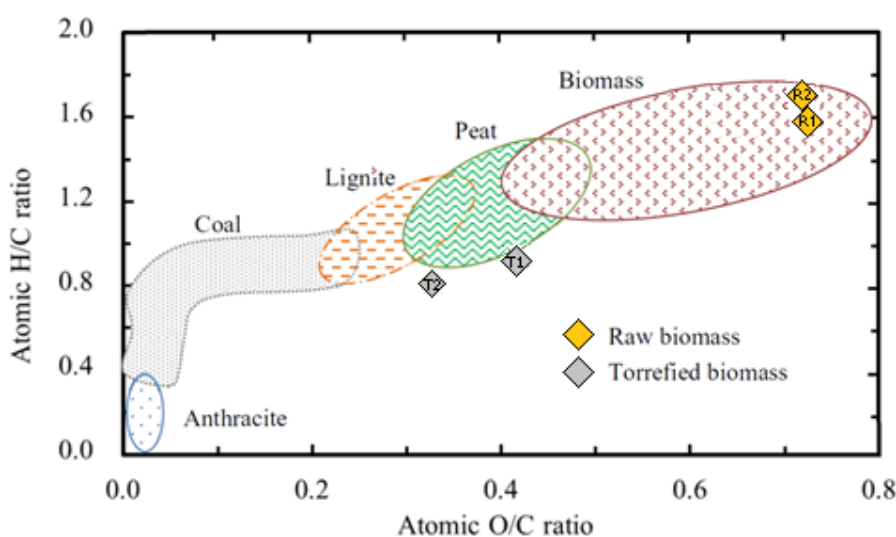| Samples	C	H	N	S	O	H/C	O/C
	% d.m.						
R-1	46.60	6.18	0.96	0.09	45.69	1.58	0.73
R-2	46.95	6.53	1.09	0.08	45.35	1.65	0.72
T-1 (275)	60.09	4.61	1.24	0.18	33.78	0.91	0.42
T-2 (300)	64.90	4.37	1.26	0.15	29.27	0.80	0.33

d.m. - dry mass

Figure 2. Millet stalks before and after torrefaction based on the plot Szufa et al. (2020).

The carbon (C) in the fuel is a key parameter affecting the amount of heat released in combustion process. The C content increased from 46.60 to 60.09% for the sample torrefied at 275°C and for the sample torrefied at 300°C to 64.90%.

The hydrogen-to-carbon (H/C) and oxygen-to-carbon (O/C) ratios are defned as the reactivity indexes, and it is known that higher H/C and O/C ratios result from higher reactive characteristics of fuel. For raw biomass, H/C and O/C are in the ranges of 1.58–1.65 and 0.73–0.72 respectively (Chen et al., 2015; Wzorek at al., 2007).

For the raw millet stalk, i.e., R-1 and R-2 samples, the H/C ratio was 1.58 and 1.64, respectively, while the O/C ratio was 0.73 and 0.72, which are typical values for lignocellulose biomass. On the other hand, for the torrefied material, it was observed that at a higher torrefaction temperature, the H/C and O/C ratios decreased, and it can be concluded that for the T-1 sample, the thermodynamic losses will be lower, making the combustion process more efficient.

The elemental composition is consistent with the expected changes in the biomass composition after torrefaction obtained by other authors. For example, Sabil et al. (2013) observed that torrefaction at 300°C reduced the oxygen in the palm skin and shell from 47–50% to 38–43%, and the C/O ratio and HHV increased from 0.9–1.0 and 18–19 MJ/kg to 1.3–1.4 and 22–23 MJ/kg, respectively. Phanphanich and Mani (2011) obtained an increase in HHV from 18.5 MJ/kg to 21.58 MJ/kg at 275°C and to 25.4 MJ/kg at the temperature of 300°C for pine wood chips.

The changes in the elemental composition of raw and torrefied biomass are also presented in the Van Krevelen diagram (Figure 2). Obtained data is compared with other types of fuels in terms of H/C ratio versus O/C ratio.

The chemical compositions of millet stalk after torrification (sample T-1 and T-2) are very similar to that of lignite and peat coal.

The process of torrefaction and the quality of pretreated biofuels are mainly characterized by parameters: mass yield (Y_{mass}), energy yield (Y_{energy}) and energy density (E_d) defined as (Grigiante and Antolini, 2015):

$$\text{Mass yield } (Y_{mass}) = \frac{\text{mass after torrefaction}}{\text{mass of raw matrial}} \cdot 100\% \tag{5}$$

$$\text{Energy yield } (Y_{energy}) = Y_{mass} \frac{\text{HHV}_{torrfied\ material}}{\text{HHV}_{raw\ material}} \tag{6}$$

$$Energy\ density\ (E_d) = \frac{HHV_{torrefied}}{HHV_{raw\ material}} \tag{7}$$

Table 3. Effect of torrefaction temperature on mass and energy yield of torrefied millet stalk

Samples	Torrefaction Temperature °C	Y_{mass} %	Y_{energy} %	E_{d-}
Millet stalk				
T1 (275)	275	56.58	74.30	1.31
T2 (300)	300	40.29	57.22	1.42
Agricultural waste (Barskov et al., 2019)	200-350	7.0-98.4	5.0-101.5	nd
Food waste (Barskov et al., 2019)	200-325	20-100	30-100	nd

nd – not data

Figure 3. Milet stalk before and after torrefaction

Table 3 presents the mass yield (Y_{mass}), energy yield (Y_{energy}) and energy density (E_d) of torrefied biomass compared to agricultural and food waste.

It was observed that the mass yield decreased with increasing temperatures. In the tests, the mass yield for the torrefaction sample was obtained in T1 at 56.58% and T2 at 40,29%. Similar results were obtained by other authors, e.g. Felfri et al. (2005), for wood briquettes, got in their study 65% of the mass yield at 250°C, and 54% at 270°C, Deng et al. (2009) for the stalk of rape at 250°C - 65% and at 270°C - 54%, and in Bridgeman's et al. (2008) research the mass yield at 250°C was 82% and at 290°C 55% for wheat straw. Pimchuai et al. (2010) claimed that the reduced mass is related to the loss of volatiles products (such as H_2O, CO, CO_2 and other organics) and moisture without compromising much of its energy.

Energy yield characterizes the ratio of energy retained after the torrefaction to the initial energy of biofuel (Phanphanich and Mani, 2011). Intensifying the energy density of biomass is the main purpose of this process. The energy yield in the torrefied materials is simillar to the energy yield for the agricultural residues (Barscove et al., 2019). Figure 3 shows millet stalk samples before and after torrefaction.

Conclusions

In the aspect of the circular economy, agriculture, such as other industry sectors, should embrace efficient and appropriate waste management. That's why the conversion of agro-biomass residues into useful products seems to be a sustainable strategy. Therefore, torrefaction is a pretreatment method of biomass waste valorisation, which allows it to be processed into products that can be used in other industrial sectors, such as the energy sector.

The paper presents study on the impact of torrefaction on the energy properties of millet stalk.

The results showed that torrefaction improves the parameters of the millet stalk for higher-quality biofuel. Comparing the energy and chemical properties of biochar's obtained at 275 and 300°C, it was concluded that millet stalk torrefied at 300°C had more favourable parameters, i.e. higher heating value HHV, the content of carbon and energy density. Therefore, biochar can be used for heat generation in local farms.

The properties of biochar made from the millet stalks show that it has the potential for different applications, such as soil improvement or the adsorption of organic pollutants. However, in the case of the bioresource economy, bioproducts currently have a few applications, and so further research should be carried out to increase these applications.

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Conflict of Interest

The author declares that no known competing financial interests or personal relationships could have appeared to influence the work reported in this paper.

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