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Improvement of Fuel Properties of Corn Stalk by Torrefaction

Mısır Sapının Yakıt Özelliklerinin Torrefaksiyon ile İyileştirilmesi

Nazlı Çaycı

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ABSTRACT

Agricultural residues are one of the important biomass sources. Its high moisture content, low calorific value, large volume and low bulk density make it difficult to use biomass directly as fuel. In this study, it was aimed to improve the solid fuel properties of agricultural wastes, which have a significant potential and a certain shape in our country, by torrefied them in their current shapes. In the presented study, corn stalk, which is a problem for the producer to evaluate and dispose of at the end of the season, was used as agricultural waste. Corn stalk is a waste biomass that is very difficult to transport and store due to its low density. For this purpose, 2 cm long corn stalks were torrefied for different residence times at three process temperatures, 220°C, 260°C and 300°C, in a pyrex glass reactor placed in a circular-section vertical refractory chamber containing resistance wires. Torrefaction process was carried out in two different environments: stagnant air and inert gas (nitrogen).

It has been determined that a coal-like appearance occurs as the processing temperature increases in the samples subjected to torrefaction. In order to obtain a solid product from corn stalk with properties similar to coal, it was determined that a short residence time at 300°C in an inert environment and a long residence time at low temperatures (260-220°C) in a stagnant air environment were suitable. As a result of the torrefaction process performed at different temperatures, O/C and H/C ratios were found to be in the range of 0.45-0.67 and 0.85-1.58, respectively. These values indicate that a peat-like solid fuel with a high lignin content is obtained as a result of torrefaction of corn stalk in both environments. In addition, it was determined that the higher heating value of the solid products obtained as a result of the torrefaction process was higher than the raw sample.

Keywords: Biomass, agricultural waste, torrefaction, solid fuel.

ÖZET

Tarımsal artıklar önemli biyokütle kaynaklarından biridir. Yüksek nem içeriği, düşük kalorifik değeri, büyük hacmi ve düşük kütle yoğunluğu biyokütlenin doğrudan yakıt olarak kullanılmasını zorlaştırmaktadır. Bu çalışmada, ülkemizde önemli bir potansiyele ve belli bir şekle sahip olan tarımsal atıkların, mecut şekilleri ile torrefaksiyona tabi tutularak katı yakıt özelliklerinin iyileştirilmesi amaçlandı. Sunulan çalışmada üreticinin sezon sonunda değerlendirip bertaraf etmesi sorun olan mısır sapı tarımsal atık olarak kullanılmıştır. Mısır sapı, düşük yoğunluğu nedeniyle taşınması ve depolanması oldukça zor olan atık bir biyokütledir. Bu amaçla, 2 cm uzunluğundaki mısır sapları, direnç telleri içeren dairesel kesitli dikey bir refrakter odaya yerleştirilen bir pireks cam reaktöründe, 220°C, 260°C ve 300°C olmak üzere üç işlem sıcaklığında farklı kalış süreleri için torrefaksiyona tabi tutuldu. Torrefaksiyon işlemi durgun hava ve inert gaz (azot) olmak üzere iki farklı ortamda gerçekleştirilmiştir.Torrefaksiyona tabi tutulan numunelerde işlem sıcaklığı arttıkça kömür benzeri bir görünümün oluştuğu tespit edilmiştir. Mısır sapından kömüre benzer özelliklere sahip katı bir ürün elde etmek için inert ortamda 300°C'de kısa kalma süresinin ve durgun hava ortamında ise düşük sıcaklıklarda (260-220°C) uzun kalma süresinin gerekli olduğu belirlenmiştir. Farklı sıcaklıklarda gerçekleştirilen torrefaksiyon işlemi sonucunda O/C ve H/C oranları sırasıyla 0,45-0,67 ve 0,85-1,58 aralığında bulunmuştur. Bu değerler mısır sapının her iki ortamda da torefaksiyonu sonucu turba benzeri, lignin içeriği yüksek bir katı yakıta dönüştüğünü göstermektedir. Ayrıca kavurma işlemi sonucunda elde edilen katı ürünlerin üst ısıl değerinin ham numuneye göre daha yüksek olduğu tespit edilmiştir.

Anahtar Kelimeler: Biyokütle, tarımsal atık, torrefaksiyon, katı yakıt.

1. INTRODUCTION

78% of the world's energy resources are fossil fuels, 19% are renewable energy, and 3% are nuclear energy resources. Since fossil fuel resources are rapidly depleting and causing irreparable damage to natural life and the environment while depleting, studies on utilizing renewable energy resources have gained greater importance in recent years (Kapluhan, 2014). Solar energy, wind energy, hydraulic energy, geothermal energy and biomass energy are the main renewable energy sources. Biomass energy has an important share among the energy sources that can meet the increasing energy demand in a sustainable way without harming the environment. Biomass is currently the world's fourth largest energy source and the most important replacement for fossil fuels. Various biomass resources exist in nature, and global biomass production is estimated to be approximately 100 billion tons per year (Ubando et al., 2020).

Biomass energy has found a wide application area all over the world with its development-oriented features that enable environmentally friendly sustainable energy production and environmental management (Kapluhan, 2014). Biomass resources include many wastes such as agricultural, municipal and domestic, forestry and animal wastes. As a result of converting these wastes into energy, an important energy source and savings will be provided (Şahin, 2019). Agricultural residues are one of the important biomass sources. Because they are abundant and their use in fuel production does not affect food production.

Its high moisture content, low calorific value, large volume and low bulk density make it difficult to use biomass as fuel without pre-treatment. Many biomass recovery (conversion) methods are applied to reduce these problems. Torrefaction process applied to improve solid fuel properties is one of these methods (Chen & Kuo, 2011). Torrefaction is low-temperature pyrolysis and is generally defined as a deoxygenation process applied to biomass under inert conditions in the temperature range of 200-300 °C (Chen et.al., 2011). Many positive results are obtained in the torrefaction process applied to biomass. These are increasing the calorific value and energy density, decreasing O/C, H/C ratios and moisture content due to the increase in carbon content, having a hydrophobic structure, increasing grindability and giving biomass more uniform properties (Chen et.al., 2015a).

Turkey is a developing country with rich agricultural potential. In terms of agricultural production, field crops come first with a rate of 55.18%. In addition, vegetable and fruit production comes with 43.29% and tea and spice plants with 1.53%. A significant amount of agricultural waste is generated and attracts attention as a source of biomass. The purpose of secondary biofuel production is; ıt is the conversion of unusable parts of plants such as stems, cobs and branches, which have no food value, unusable herbaceous plants and aquatic plants into solid fuel with better properties (Yıldız, 2015).

In the presented study, corn stalk, which is an agricultural waste, was used. It is a waste that is problematic for the producer to evaluate and dispose of at the end of the season. Corn stalk, which remains in the field after the corn produced as grain is harvested and cannot be used as animal feed or fertilizer, is a waste biomass that is very difficult to transport and store due to its low density. In addition, the electrification generated during grinding due to its structure greatly limits the use of this biomass. In the presented study, it is aimed to improve the solid fuel properties of these wastes, which have a significant potential and have a certain shape in our country, by torrefied them. In addition, in order to ensure that the applied process is more economical, the results obtained by torrefaction in the volatile matter (stagnant air) environment formed as a result of heating the biomass were compared with the inert environment. Process conditions for the production of a quality solid fuel from agricultural waste were compared and their effects on product yield were investigated. In order to determine the change in the fuel properties of the solid product, brief and elemental analyzes were carried out and the higher heating values were determined and it was determined how much of the energy of the biomass was preserved at the end of the torrefaction process.

2. MATERIAL AND METHOD

The corn stalk used in the study was obtained from Muratçık village in Elazığ. Corn stalks were dried in the laboratory for at least two weeks before use. The dried corn sample was cut with vine shears to a length of approximately 2 cm, as shown in Figure 1, and was made ready for the torrefaction process. Some of the dried samples were ground and their proximaate and elemental analyzes were carried out and the higher heating value was determined.

Proximate analysis, where volatile matter and ash ratios were determined, was performed in the muffle furnace according to ASTM D 1782-84 standards (Conag et. al., 2018). The moisture of the biomass was determined on the Mettler LJ16 moisture analyzer. To determine the properties of raw materials and torrefied samples, elemental analyzes were carried out with the LECO (CHNS-932) Elemental Analyzer at İnönü University Scientific Research Center. In addition, JULIUS PETERS BERLIN adiabatic calorimeter was used to determine the higher heating value (HHV) of raw and torrefactioned samples.

Torrefaction experiments were carried out in the system shown in Figure 2. Experimental study, It was made in a vertical refractory chamber designed by Prof. Dr. Dursun PEHLİVAN, containing resistance wires, with a circular section of 45 mm inner and 115 mm outer diameter, and 105 mm height. Torrefaction process was carried out in a pyrex glass reactor with a diameter of 3.5 cm and a length of 15 cm placed in the oven. The chamber was heated by a voltage transformer and the temperature was measured with a thermo couple (NiCr) in contact with the pyrex reactor. The inert environment in the reactor was provided with N_2. In addition, a liquid product collection container placed in an ice bath was added to the experimental set to collect the liquid product formed during torrefaction.

Corn stalks, which were dried until the moisture content was 5-6%, were weighed approximately 4 g and placed in the reactor. The reactor was then placed in the chamber and its lid was closed. In the experiments carried out in an inert environment, nitrogen was passed at a flow rate of 100 ml/min for 5 minutes to remove oxygen in the reactor before heating the system and the chamber was heated to operating temperature with a voltage transformer. In both environments, the system was heated to operating temperature for a fixed preheating time of 10 minutes.

Figure 1. Corn stalks

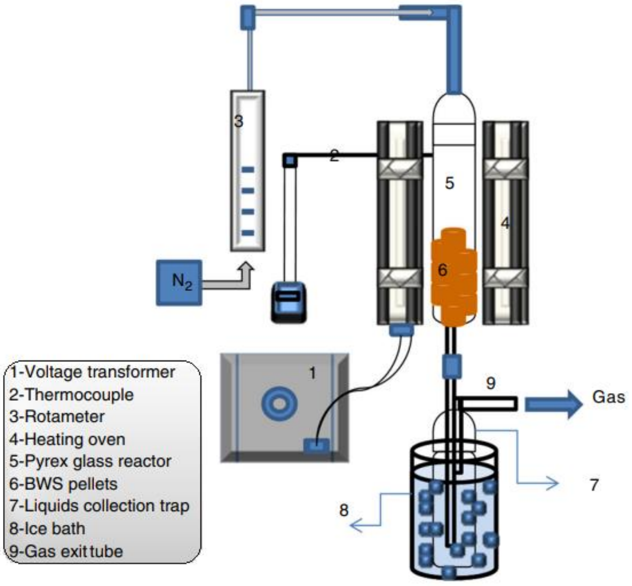

Figure 2. Torrefaction experimental system

After the furnace reached the operating temperature, the torrefaction process was carried out in nitrogen atmosphere and stagnant air environments. At the end of the study period, heating of the system was stopped and the reactor was cooled. The solid products (biochar) removed from the reactor and completely cooled were weighed and the solid product yield was calculated according to Equation (1). The liquid+gas product yield was calculated from the difference according to Equation (2). Here, m_{bc} and m_{raw} denote the weights of biochar and raw biomass, respectively.

$$\text{Solid product yield (\%)} = \frac{m_{bc}}{m_{raw}} \times 100 \qquad (1)$$

$$\text{Liquid+Gas yield (\%)} = 100 - \text{Solid product yield (\%)} \qquad (2)$$

To investigate the effect of temperature on product yield and the properties of the solid product, torrefaction experiments were carried out at 220, 260 and 300°C torrefaction temperatures and for processing times of 0, 5, 10 and 20, and in nitrogen and stagnant air environments. 0 min here indicates the preheating conditions applied for the system to reach the desired process temperature.

3. RESULTS AND DISCUSSIONS

3.1. Effect of Process Conditions on Product Yield

The color change in corn stalk depending on torrefaction temperature and time is given in Figure 3. Similar behavior is observed in the torrefaction process performed in two different environments. It was determined that a coal-like appearance was formed in the samples with increasing torrefaction temperature and time. However, as the process temperature and time increased in a stagnant air environment, it was determined that a weaker solid product was obtained, and when the temperature and time were increased further, it burned.

Table 1 shows the solid and liquid+gas product yields obtained as a result of torrefaction of corn stalks. It was determined that solid product yield decreased and liquid+gas yield increased with increasing process temperature in both environments. This is due to the fact that the compounds forming the lignocellulic structure have different decomposition temperatures.

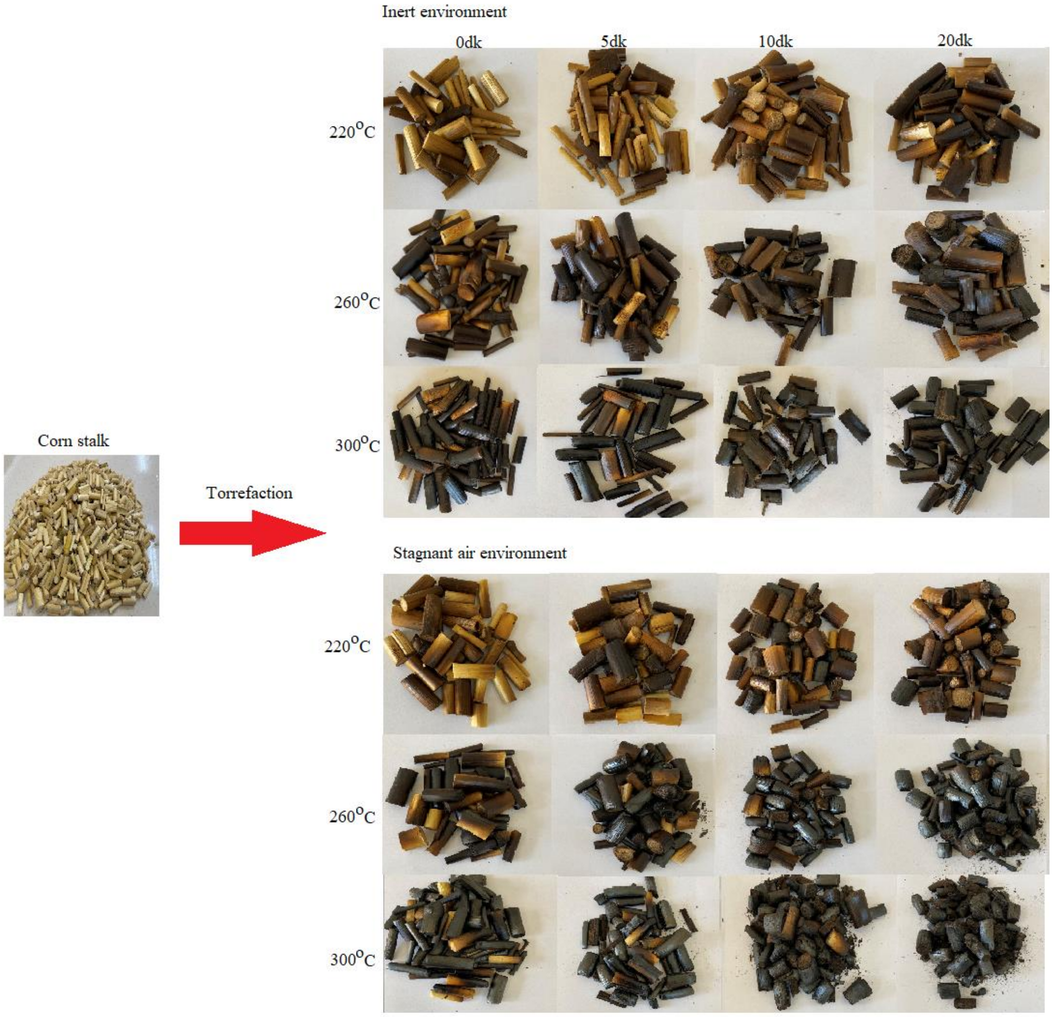

Figure 3. Raw ve torrefied corn stalks

Table 1. Product yields obtained by torrefaction of corn stalks at different temperatures (processing time 20 min).

Sample	**Temperature (°C)**	**Solid (%)**	**Liquid + gas (%)**
Torrefied in inert environment	220	77.27	22.73
	260	60.75	39.25
	300	46.55	53.45
Torrefied in stagnant air environment	220	70.51	26.49
	260	50.97	49.03
	300	36.99	63.01

In the temperature range of 220-260°C, where low and mild torrefaction occurs, moisture and small molecular weight compounds formed as a result of the breakdown of hemicellulose are separated from the biomass. Under severe torrefaction conditions at 300°C, the decrease in solid product yield increases as cellulose begins to decompose. Since the degradation of lignin in biomass occurs in awider temperature range, a lignin-rich solid product is formed after torrefaction (Akkuş, 2018).

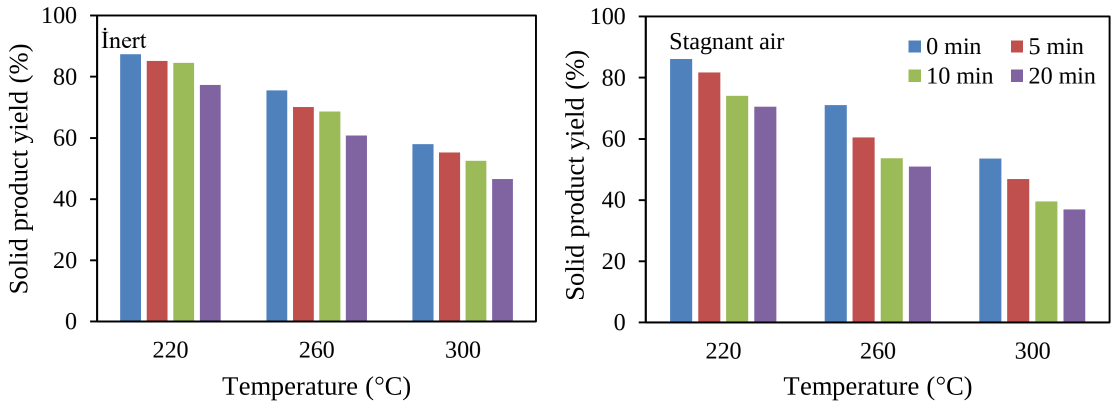

Figure 4. Variation of solid product yield obtained as a result of torrefaction of corn stalk in inert and stagnant air environments with temperature and processing time**.**

One of the parameters that determine torrefaction performance is the processing time. For this purpose, torrefaction was carried out in two environments at different processing times for each torrefaction temperature. As seen in Figure 4, while the processing time in an inert environment is effective at high torrefaction temperatures, it is also effective at low temperatures in a stagnant air environment (Duranay&Pehlivan, 2021).

Figure 5 shows the effect of processing time on product yields at 260°C in an inert and stagnant air environment. It is observed that the solid product yield decreases with increasing processing time in both environments. It has been determined that the cooling caused by inert gas in short residence times and the oxidation reactions occurring in long residence times in the stagnant air environment affect the solid product yield.

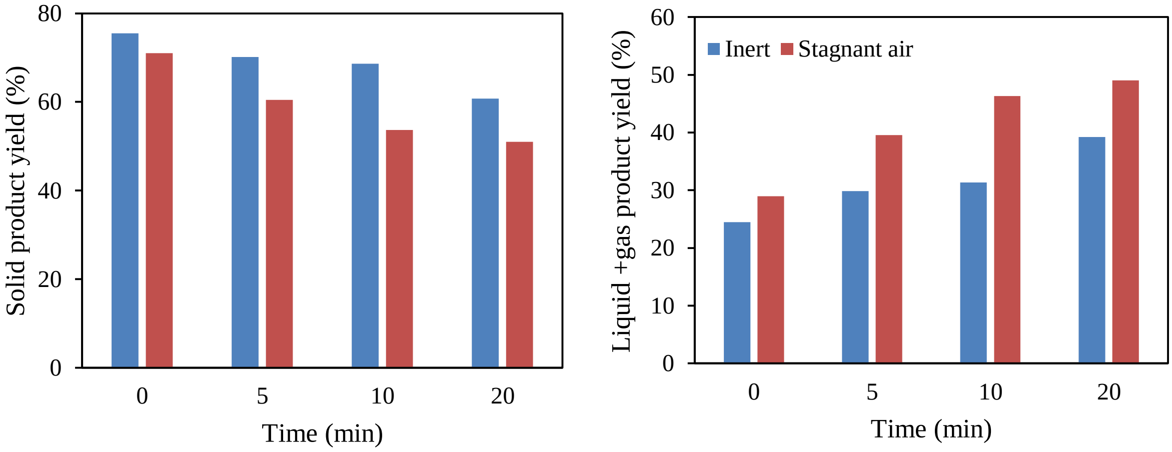

Figure 5. Comparison of product yields obtained as a result of torrefaction of corn stalk in an inert and stagnant air environment at 260°C.

3.2.Characterization of Solid Product

3.2.1.Proximate analysis of solid product

Proximate analysis of biomass includes measurement of moisture content, ash, volatile matter and fixed carbon. The fixed carbon content of biomass is generally low and ranges from 9 wt% to 25 wt%, volatile matter ranging from 63 to 88 wt% (Sarker et.al., 2021).

Table 2 gives brief analysis values of the solid products obtained as a result of torrefaction of corn stalk at different temperatures for 20 minutes. It is seen that the original sample has a high volatile matter ratio and a low fixed carbon and ash ratio. Additionally, the sample contains 5.8% moisture. It was determined that while the volatile matter ratio decreased with increasing process temperature, the ash and fixed carbon ratio increased. Torrefaction process causes light volatile substances to be separated from biomass by dehydration, depolymerization and disintegration reactions, and moisture by dehydration. Therefore, volatile matter and moisture contents decrease while fixed carbon increases. It was determined that when the torrefaction temperature was increased up to 300°C in an inert environment, the volatile matter rate decreased by 45%, while the ash rate increased by 2.5 times and the fixed carbon rate increased by 4 times. In a stagnant air environment, it was determined that when the torrefaction temperature was increased up to 300°C, the volatile matter rate decreased by 55%, while the ash rate increased by 2.8 times and the fixed carbon rate increased by 4.7 times.

Table 2. Proximate analysis data of raw and torrefied corn stalks.

Sample	Temperature (°C)	Volatile matter (%)	Fixed carbon (%)	Ash (%)
Raw		85.18	10.62	4.20
Torrefied in inert environment	220 260 300	78.06 64.56 46.76	15.19 25.43 42.35	6.75 10.01 10.89
Torrefied in stagnant air environment	220 260 300	62.41 54.49 38.57	30.38 38.48 49.78	7.21 7.05 11.65

3.2.2. Elemental analysis of solid product

The main elements found in biomass are carbon, hydrogen, oxygen, nitrogen and sulfur. Table 3 shows the changes in the elemental composition of corn stalks torrefied in an inert and stagnant air environment. It can be seen that while the carbon content increases with increasing process temperature in both environments, the hydrogen and oxygen contents decrease. With the increase in carbon content, the decrease in oxygen and hydrogen levels in torrefied samples is due to the release of volatile substances (including water vapor) and stable gases (e.g. CO, CO_2, CH_4 and H_2) during the process (Kanwal et.al 2019). In addition, dehydration and decarboxylation reactions, which cause the release of volatiles, cause the breakdown of carbohydrates at higher refractory temperatures (Yue et.al., 2017). In addition to these reactions, oxidation reactions occurring at high temperatures and residence times in stagnant air also affect the composition of the solid product (Akkuş, 2018). It has been determined that there is carbon loss due to oxidation reactions that occur in torrefaction processes carried out for long periods at mild temperatures (260°C) and at high temperatures (300°C) in a stagnant air environment. The effect of the torrefaction process on sulfur and nitrogen content was found to be negligible.

Generally, the oxygen/carbon and hydrogen/carbon ratios in raw biomass are in the range of 0.4-0.8 and 1.2-2.0, respectively. After undergoing torrefaction, hydrogen and oxygen are separated from the biomass in the form of moisture and light volatiles, while carbon is retained. This causes carbonization of biomass. With the torrefaction process of corn stalk carried out at different temperatures, it was observed that the O/C and H/C ratios decreased to the range of 0.45-0.67 and 1.01-1.58 for the inert environment, and to the range of 0.53-0.66 and 0.85-1.37 for the stagnant air environment, respectively (Figure 6). The significant change in H/C and O/C ratios as a result of the torefraction process carried out in an inert and stagnant air environment at 300°C is due to the large disintegration of cellulose in this temperature region (Chen et. al., 2015b) When these ratios are placed on the Van Krevelen diagram as in Figure 6, a linear relationship is obtained between these ratios with very good regression coefficients ($R^2 = 0.98$ for the inert environment and $R^2 = 0.97$ for the stagnant air environment). It is known that these rates decrease as the severity of torrefaction increases (Van der Stelt et.al., 2011; Lestander et.al., 2014) The slope of the regression line is 2.67 for the inert environment and 3.42 for the stagnant air environment, indicating that the effect of torrefraction on the atomic H/C ratio is approximately 2.67 times greater in the inert environment and 3.42 times greater in the stagnant air environment than the atomic O/C ratio. In this case, it can be concluded that the lignin ratio increases with increasing torrefaction temperature. As a result of the torrefaction process of corn stalk in both environments, a peat-like solid fuel with a high lignin content was obtained.

Table 3. Elemental analysis data of raw and torrefied corn stalks.

Sample	Temperature (°C)	C %	H %	N %	O % *	S %	Atomic H/C	Atomic O/C
Raw		43.34	7.41	0.51	48.68	0.07	2.01	0.84
Torrefied in inert environment	220	49.16	6.45	0.80	43.57	0.02	1.58	0.67
	260	51.56	5.71	0.68	41.95	0.10	1.33	0.61
	300	59.11	4.97	0.79	35.10	0.02	1.01	0.45
Torrefied in stagnant air environment	220	49.30	5.63	0.72	43.25	0.11	1.37	0.66
	260	58.37	4.09	1.06	36.41	0.08	0.84	0.47
	300	55.66	3.95	0.96	39.38	0.04	0.85	0.53

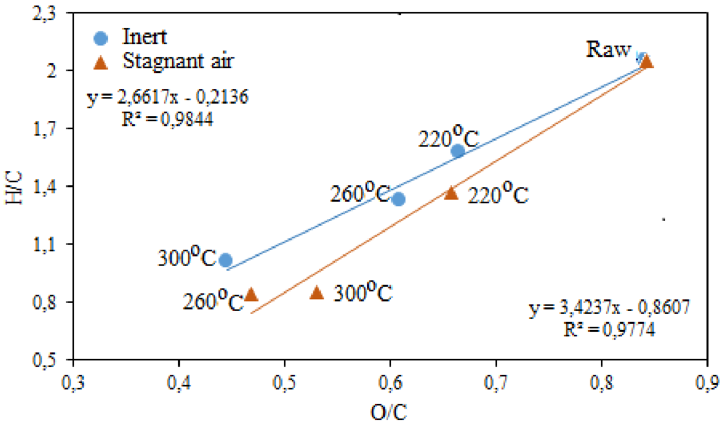

Figure 6. Changes in atomic H/C and O/C ratios of raw and torrefied corn stalks.

3.2.3. Higher heating value (HHV) and energy efficiency of the solid product

Table 4 shows the change in the higher heating value and energy efficiency of raw and torrefied corn stalks with the torrefaction temperature. It has been determined that with increasing temperature, the higher heating value and energy efficiency increase in the inert environment and decrease in the stagnant air environment. It is seen that the higher heating values of biochar obtained in inert environment are higher than those obtained in stagnant air environment. In torrefaction performed at 300°C for 20 minutes, it was determined that when the carbon content increased by 36% in the inert environment, HHV increased by 82%, and when the carbon content increased by 28% in the stagnant air environment, HHV increased by 13%. This difference is due to oxidation reactions occurring in the oxidizing environment. Energy efficiency, which depends on solid yield and calorific value, is an indicator of the amount of energy lost during torrefaction (Chew&Doshi, 2011). Energy efficiency was calculated according to Equations (3) and (4).

$$\text{HHV enhancement factor} = \text{HHV}_{tor}/\text{HHV}_{raw} \quad (3)$$

$$\text{Energy yield \%} = \text{Solid yield \%} \times \text{HHV enhancement factor} \quad (4)$$

Here, HHV_{tor} and HHV_{raw} respectively show higher heating values of torrefied and raw corn stalk. The higher heating value of coal is generally in the range of 25-35 MJ/kg. It is stated in the literature that as the temperature of the torrefaction process increases, the calorific value of biomass approaches that of coal (Chen et al., 2015b). It was determined that 84.49% of the energy of the corn stalk, which was torrefied at 300°C for 20 minutes in an inert environment, was preserved. Since its higher heating value is close to coal, it can be said that it would be appropriate to torrefaction corn stalk under these process conditions.

Table 4. The effect of processing temperature on the HHV and energy efficiency of raw and torrefied corn stalks (processing time 20 min).

Sample	Temperature (°C)	Solid yield (%)	HHV (MJ/kg)	HHV Enhancement factor	Energy yield (%)
Raw			21.33		
Torrefied in inert environment	220	77.27	21.53	1.009	77.97
	260	60.75	28.67	1.344	81.65
	300	46.55	38.72	1.815	84.49
Torrefied in stagnant air environment	220	70.51	24.67	1.156	81.51
	260	50.97	30.85	1.446	73.70
	300	36.99	24.10	1.130	41.80

As the torrefaction intensity (temperature) increases, the O/C ratio decreases and as a result, the higher heating value increases (Chen & Jhou, 2020). This result is clearly seen in Table 4 in the torrefaction process applied to corn stalks in an inert environment. On the other hand, it has been determined that torrefaction at high temperatures (300°C) is not suitable for a stagnant air environment and that there is carbon loss due to oxidation reactions and that the calorific value decreases as a result of this. It has been concluded that if severe torrefaction is to be applied to corn stalk, it is appropriate to do it in an inert environment, and if the process is done in stagnant air to be economical, it is appropriate to do it under mild torrefaction conditions (260 °C and 20 min).

4. CONCLUSIONS

The results obtained from this study, in which the effect of processing conditions on the yield and properties of the solid product resulting from torrefaction applied to corn stalk in order to convert agricultural wastes into usable solid fuels, are given below.

Effect of process conditions on product yield

It was determined that the sample turned into a coal-like solid product with increasing torrefaction temperature and time in inert and stagnant air environment. It was determined that increasing torrefaction intensity and duration in a stagnant air environment was more effective on solid product yield.

In order to obtain a solid product with properties similar to coal from corn stalk, it has been determined that a short residence time under severe torrefaction conditions in inert environment and a long residence time under mild torrefaction conditions in stagnant air environment are suitable.

Solid product characterization

When the short analysis data were compared, it was determined that the compositions of the solid product obtained from corn stalk in inert and stagnant air environment were different.

It has been determined that the carbon content increases while the oxygen and hydrogen content decreases with increasing process temperature in an inert and stagnant air environment. It has been determined that there is carbon loss due to oxidation reactions occurring in the stagnant air environment.

In the Van Krevelen diagram, it was determined that the properties of corn stalk tend to resemble coal with increasing torrefaction intensity (temperature), and the H/C-O/C ratios approach peat with increasing torrefaction intensity.

It has been determined that the higher heating values of biochar obtained in inert environment are higher than those obtained in stagnant air environment.

In the light of the data obtained in the study, it was determined that corn stalk can be converted into higher quality solid fuels similar to coal by torrefaction process.

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