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Research Article

Evaluation of Grain Maize Harvest Residues as Fodder After Partial Torrefaction Under Microwave

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Abstract: With a project supported by Siirt University (Siirt, Türkiye), we determined the conversion potential of residues of field cropped grain maize into animal feed via torrefaction. Torrefaction application was performed in a microwave device for four different periods of time (0, 4, 8, and 12 minutes) in three replications at three different watts (300, 600, and 900 watts). The dry matter digestion was determined for the ground starter and final materials by the Daisy Incubator Technique (ANKOM). Changes in dry matter, protein, and ADF values were measured by standard feed quality analyses. The effects of applications on dry matter ratio, protein, and in vitro digestibility were found to be statistically significant, whereas the effect on ADF ratio was insignificant. Low-term torrefaction applications significantly increased the dry matter ratio, whereas high power and long application periods significantly increased the protein ratio. In vitro, digestibility was decreased due to the increase in the power application and duration of torrefaction. A material with high dry matter and high protein content but low in vitro digestibility was obtained as a result of the torrefaction applications tested in the study under microwave conditions with grain maize harvest residues. Although the applications tested have decreased the value of maize harvest residues as cattle feed, the obtained material with high dry matter and protein content with this torrefaction method has the potential to be used as a nutrient medium in different living environments (e.g. bacterial or fungal) which may be subjected to further investigations.

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1. Introduction

The amount of grain maize (*Zea mays* L.) harvested in Türkiye in 2021 was 6.5 million tons. For the world, this value was 1.15 billion tons for the same year (FAO STAT, 2021). The harvest index (HI) is the ratio of a harvested crop to the total dry plant weight. The HI for maize is between 0.25–0.58 (Yang and Zhang, 2010). When the harvest index is roughly 0.4, it means that six tons of maize harvest residue are produced for four tons of harvested maize grains. When this calculation was applied to given 2019 values, produced maize residues were equal to 6/4*6=9 million tons for Türkiye and 1.73 billion tons for the world.

Cellulose in maize plants, as the most important component of the plant skeleton, is a polysaccharide formed by connecting D-glucose building blocks in long chains (Klemm et al., 2005).

Cellulose is a renewable polymer source that has been used in a wide range of products and materials for about 150 years (Habibi et al., 2010). Biomass consisting of cellulose, hemicellulose, and lignocellulose contains a large amount of fermentable sugar (Anonymous, 2022). Hemicellulose is a polymer containing mainly the five-carbon sugar C5H10O5 (xylose) (NIST, 2011a). Cellulose is a polymer containing the six-carbon sugar C6H12O6 (glucose) (NIST, 2011b). Due to these properties, it is possible to make a significant contribution to food production by making waste biomass digestible without extra farm field occupation for crop cultivation. Many factors such as lignin content, the crystalline structure of cellulose, and particle size limit the digestibility of hemicellulose and cellulose in cellulosic biomass (Hendriks and Zeeman, 2009).

The use of ligninolytic fungi and their enzymes is a potential alternative to provide a practical and environmentally friendly approach to increasing the nutritional value of straw residues. Today, however, the cost of exogenous enzymes is too high to be preferred by small farms. In addition, the relative scarcity of available data on applications using fungi and their enzymes to improve the digestibility of stem residues limits its applicability (Sarnklong et al., 2010).

As an alternative to enzyme application, torrefaction provides thermal conversion of biomass between the temperature range of 200-300°C (Stelt et al., 2011). Hemicellulose, cellulose, and lignin, which are the main components of biomass, are substrates in the torrefaction treatment. Hemicellulose, cellulose, lignin, xylan, and dextran are components that affect biomass torrefaction (Chen and Kuo, 2011). This treatment improves the properties of biomass through thermochemical processing techniques (Bridgeman et al., 2008). Torrefaction, which is among the potential applications for the production of processed pellets for fuel extraction, has attracted great interest from academics and industry in recent years for the production of high-quality syngas and the replacement of coal in thermal power plants and industrial metallurgical plants (Chen et al., 2015). In the application of torrefaction (Deng et al., 2009), which is a basic pretreatment technology that also reduces the problems of high bulk volume, high moisture content, and poor grindability of agricultural biomass, thermal treatment in the relatively low temperature range decomposes the sensitive hemicellulose part of the material (Prins et al., 2006). During torification, 70% of the initial mass is retained (this amount contains 90% of its initial energy content as a solid product), and the 30% lost mass turns into condensable and non-condensable products. Roasting biomass improves physical properties such as grindability while changing the shape, size, and distribution of particles, pelletability of the material, and intermediate and final content (Tumuluru et al., 2011).

In a different study, it was determined that mild torrection (240°C) significantly destroyed hemicellulose in biomass, but cellulose and lignin were slightly affected, while severe torrection (275°C) significantly affected cellulose (Chen and Kuo, 2011). Deng et al., (2009) reported that temperatures of 200°C, 250°C, and 300°C affect the biomass and the type of raw material which affects the conversion rate due to the difference in volatile content in the raw biomass. Yang et al., (2007) found that at torrefaction, the hemicellulose ratio decreased at 220–315°C, and the cellulose ratio decreased at 315–400°C. They also reported that hemicellulose produced a higher CO_2, cellulose produced a higher CO, and lignin produced higher H_2 and CH_4.

In this research, the potential of converting crop harvest residues from grain-grown maize into highly digestible feed by microwave torrefaction was investigated.

2. Material and Methods

In the experiment, the harvest residues of the maize plants from a farmer's field of main season grain maize crop in the province of Siirt in Türkiye in 2021 year were used as material. Harvest residue materials were collected from farmer fields following harvest, filled in straw sacks, and kept in the warehouse under appropriate storage conditions. Laboratory measurements of the experiment were carried out in the 2022 year. In the study, the torrefaction application was applied in a microwave, during four different periods of time (0 (control group), 4, 8, and 12 minutes), at three different watts (300, 600, and 900 Watt), with three replications. Torrefaction was applied to the maize residues in heat resistant glass containers (Figure 1).

a b c

Figure 1. The microwave device used in torrefaction; b) two pyrex containers of different sizes, resistant to very high temperatures and with a lid, used as a torification vessel; c) the intertwined state of two pyrex containers used as a torrection container. The interspace between the outer and inner container is the breathing volume for the inner container, where a piece of cotton is burned in this intermediate volume at each sample preparation stage before torrection, aiming to let CO_2 and CO but not O_2 exchange during torrefaction.

The microwave device used in the trials was a Beko brand, MD-1505 Model, 230 V, Class I device working at 50 Hz, 1, 200 W, and 2.450 MHz operating frequency. The working range was in the range of 90-900 watts, working time was in the range of 0-60 minutes.

The amount of maize residue to fill the inner chamber of the torrection container was chopped to a size of 2-3 cm with the help of scissors and pliers. The amounts of dry leaves, stems, hollow cobs, and tassels were balanced to be similar for different applications (Figure 2). Two different pyrex cooking pots, which are resistant to high temperatures and can fit inside each other, were used for torrefaction. To consume the oxygen of the inter chamber space between these two vessels to build a partial air-breathing reaction vessel set-, burning cotton was left in between two glass pots, and the outer vessel lid was closed immediately before being placed into the microwave just before the beginning of the torification of each sample. Burning cotton was left in the mid-compartment to ensure oxygen consumption in the "intermediate layer", which was the breathing section of the inner container. After the torrection process, the materials were ground with a laboratory-type spiral blade shredder for 0.5 minutes (Figure 4). In order not to mix the material, the empty turning time, the grinding time of the maize residues, and the cleaning time of the grinder for the new sample were each 0.5-minute intervals. Some samples were reserved for DM, protein, and ADF analyses, the remainder was allocated for in vitro digestibility analyses.

The actual dry matter digestion level was determined by the Daisy Incubator Technique (ANKOM) after the materials were ground for a short period. Changes in dry matter, protein, and ADF values were measured by standard feed analyses. Feed quality analyses such as dry matter (DM), ADF, and protein content (CP) were performed in the "Eastern Mediterranean Agricultural Research Institute Quality Laboratory" (Adana, Türkiye).

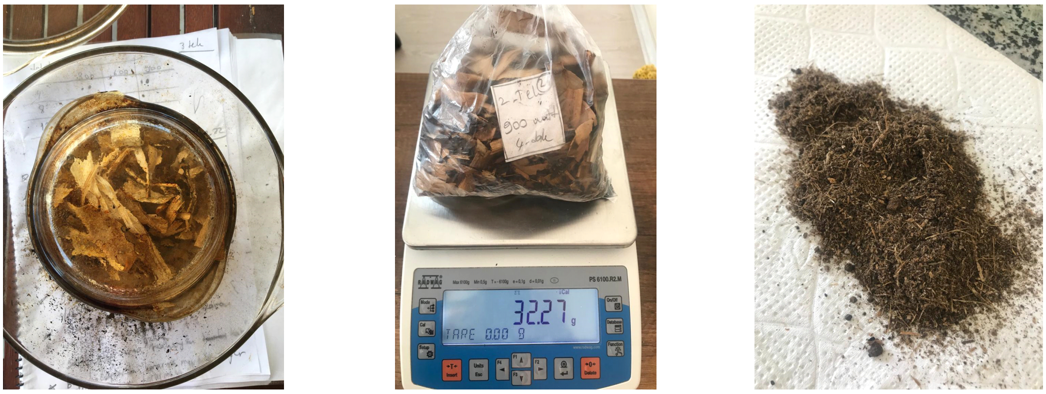
a b c d

Figure 2. Stem part, b) empty cob, c) leaf part, and d) husk (cob cover) of maize harvest residues.

a **b** **c**

Figure 3. Close-up view of the cellulosic stem tissue of the stalk section of maize harvest residues, b) the mixture of harvest residues after the shredding process, and c) sample sample prepared and labeled for applications.

The method of AOAC, (1990) was used for dry matter and nitrogen determination. Goering and Van Soest, (1970) method was used for acid-detergent fiber (ADF) determination. In vitro digestibility analyses were carried out in the laboratory of Uludag University Faculty of Agriculture, Department of Animal Science, Feeds and Animal Nutrition (Bursa, Türkiye). For the in vitro study, rumen content from three healthy rams or cattle that completed their rumen development was obtained to use in Ankom DaisyII Incubator D220. The rumen fluids used in the study were taken from a slaughterhouse, immediately after the animals were slaughtered, accompanied by a carbon dioxide tube. After the sample was taken, it was filtered with the help of two-layer sterile cheesecloth, and two handfuls of rumen solids were added to it and transported to the laboratory without losing time in thermoses at 39°C. In the experiment, nutrient analyses of feeds and residues were determined according to the methods specified in AOAC (1998), and their true dry matter digestibility (TDMd) was applied in the Ankom Daisy Incubator (Ankom, 2002) using the strainer bag technique (Van Soest et al., 1991).



a **b** **c**

Figure 4. A torrefacted sample in a high temperature resistant pyrex glass container set; b) weighing the samples after application; c) one of the ground samples.

The jmppro-13 package program was used for statistical analysis according to the randomized parcels experimental design. According to the results of the F test, the differences between the groups were determined by the LSD (Least Significant Difference) multiple comparison test. In addition, to determine the relationship between applications and features, principal component analysis was performed according to the Scatter plot model in the statistical package program "Genstat 12th" (Copyright 2011, VSN International Ltd).

3. Results and Discussion

The averages of the investigated properties of the material obtained as a result of torrection in the study and the resulting groups are given in Table 1 below.

Table 1. The averages of the examined characteristics and the resulting groups

Applications	DM (%)	Protein (%)	ADF (%)	TDMd*
300 Watt 0 min	88.94 d	4.64 de	44.19	56.85 a
300 Watt 4 min	90.51 c	4.10 de	47.23	52.90 b
300 Watt 8 min	91.30 ab	2.78 e	43.64	50.51 c
300 Watt 12 min	90.90 bc	5.83 de	45.67	47.65 d
600 Watt 0 min	89.61 d	3.42 e	50.40	56.90 a
600 Watt 4 min	91.06 abc	4.38 de	47.67	47.75 d
600 Watt 8 min	90.38 c	11.66 c	43.97	43.90 f
600 Watt 12 min	91.66 a	12.05 bc	46.18	41.32 g
900 Watt 0 min	89.30 d	5.28 de	46.88	56.36 a
900 Watt 4 min	91.28 ab	9.61 cd	45.30	45.62 e
900 Watt 8 min	90.68 bc	17.61 ab	46.08	37.51 h
900 Watt 12 min	90.66 bc	19.54 a	47.31	29.15 ı
Avr.	90.52	8.40	46.21	47.20
LSD	0.35**	2.79**	2.10 öd	0.52**

Note: **: P≤0.01, DM: dry matter, TDMd: True dry matter digestion, ADF: acid detergent fiber. The difference between the means denoted by the same letters is not statistically significant.

Torrection applications were significantly effective on dry matter ratios (Table 2). The lowest DM values (between 88.94-89.61%) were in the control group, whereas the highest DM values (between 91.06-91.66%) were obtained from “300 Watt 8 min”, “600 Watt 4 min”, “600 Watt 12 min” and “900 Watt 4 min” applications. While an application with 300 and 900 watts each produced the highest DM value, two applications at 600 watts of power resulted in the highest DM values.

Torrefaction applications were significantly effective on the amount of protein. The lowest protein values (between 2.78-5.28%) were obtained from all periods at 300 Watt power (Table 2). Higher protein values were recorded at 900 Watt 8 min and 12 min applications, at a rate of 17.61% and 19.54%, respectively. The combination of high power and duration seriously degraded the chemical structure and resulted in significant material structure change through gaseous mass loss. Less period torrefaction applications significantly increased the "dry matter ratio", while high-power and long-term torrefaction increased the "protein ratio", which was associated with the conversion of polysaccharides to volatile form and the remaining protein skeleton. ADF values varied between 43.64-50.40%.

Torrections were significantly effective on in vitro true digestibility (TDMd). The lowest TDMd value (29.15%) was obtained with the most aggressive application (900 watts 12 min). The control group, in which no torrefaction was applied, produced the highest values (56.36-56.90%) in terms of TDMd. The tested torrefaction applications reduced the cattle feed value of maize harvest residues. The feed value decreased almost linearly with increasing duration of torrefaction. In this sense, the greatest amount of decrease was obtained by 900-watt applications.

3.1. Evaluation of the relationship between applications and features according to the scatter plot model

Figures 5, 6, and 7 show the effects of the applications on the examined features by vectors (a), sectors, and mega-periphery (b, c) via the scatter plot model.

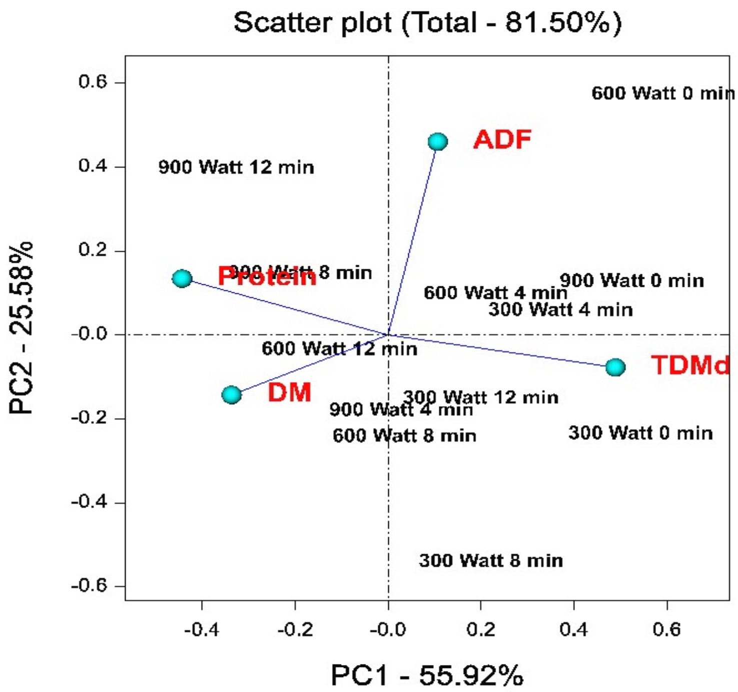


Figure 5. Representation of the effects of the applications on the examined properties with vectors (a) with the scatter plot model.

When evaluating Figure 5, it is visible that the application and features include variation at PC1:55.92, PC2:25.58, PC1+PC2: 81.50. In the scatter plot model, the narrowing angle between the vectors represents the features indicating a high positive correlation, an increase in the angle value indicates decreasing correlation, and the angle value of 90^{o} indicates that there is no correlation (Mohammadi & Amri, 2011).

When Figure 6 is examined, it can be seen that the properties are concentrated in 3 different environments (E1; %TDMd and ADF, E2; DM, E3; Protein). While the "900 Watt 0 minute" application in the first group was the ideal environment for ADF, and %TDMd properties, the "600 Watt 8 minutes" application in the second group was ideal for DM. And the "900 Watt 8 minutes" application in the third group was the most ideal environment for the protein ratio. In addition, it was observed that "300 Watt 8 min, 900 Watt 12 min and 600 Watt 0 min" applications do not stand out in terms of any of the properties examined.

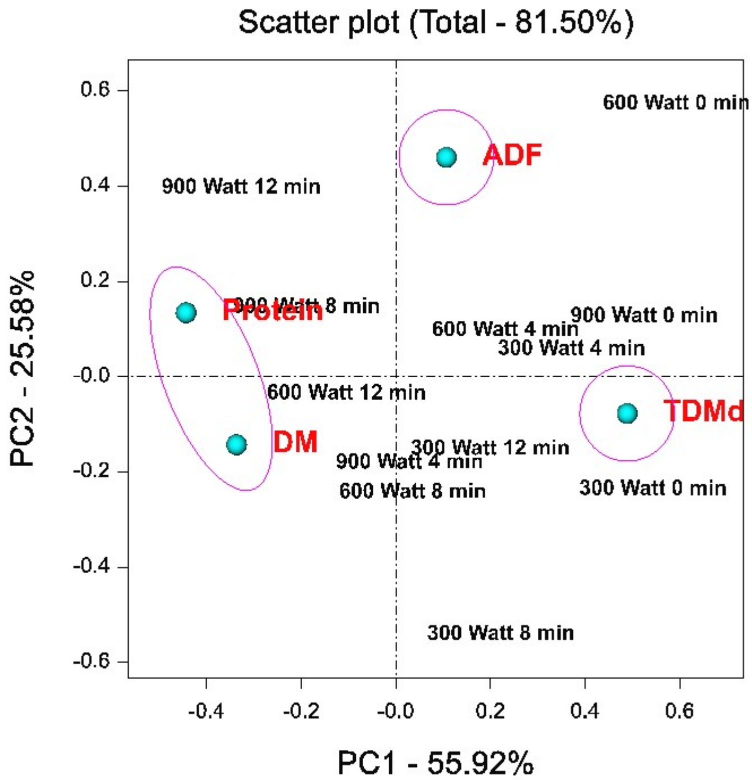


Figure 6. Representation of the effects of the applications on the analyzed features and the resulting averages by sectors (b) via the scatter plot model.

When we look at the relationship between applications and features through polygons and sectors (Figure 7), six different sectors were formed. The distribution of applications and features in different sectors shows that no application stands out in terms of the feature in question, or that no feature stands out in terms of applications. The fact that the application and the feature are in the same sector reveals that there was a positive relationship between the application and the features (Chinipardaz et al., 2016). The applications located on the diagonals of the polygon have the best performance in terms of the features found in the relevant sector (Yan & Tinker, 2006). In Figure 7, it can be seen that 300 Watt 8 min, 900 Watt 12 min, and 600 Watt 0 min applications show the best performance in these features.

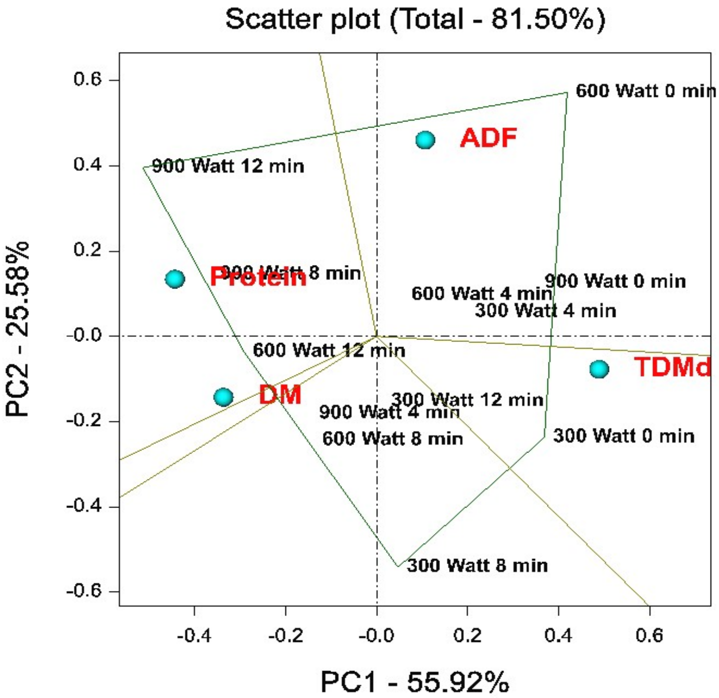


Figure 7. Representation of the effects of the applications on the examined features within the mega environment (c) via the scatter plot model.

3.2. Evaluation of the relationship between the scatter plot matrix and the examined features

The matrix showing the effect of the applications on the examined features through the Scatter Plot Matrix is given in Figure 8 below.

When the scatter plot matrix is examined, it was determined that the relationship between DM and %TDMd was 5% negative and significant ($r2= -0.4159$). If there exists an uneven distribution on the regression line in the graph that expresses the relationship between the two features examined, it can be concluded that the relationship between these features is weak (Eren & Demirel, 2020). However, if the distribution is regular and agglomerated on the regression curve, it can be said that these features are strongly related to each other. In our study, it was determined that the correlation coefficient between %TDMd and protein was 5% negative and significant ($r2= -0.8051$).

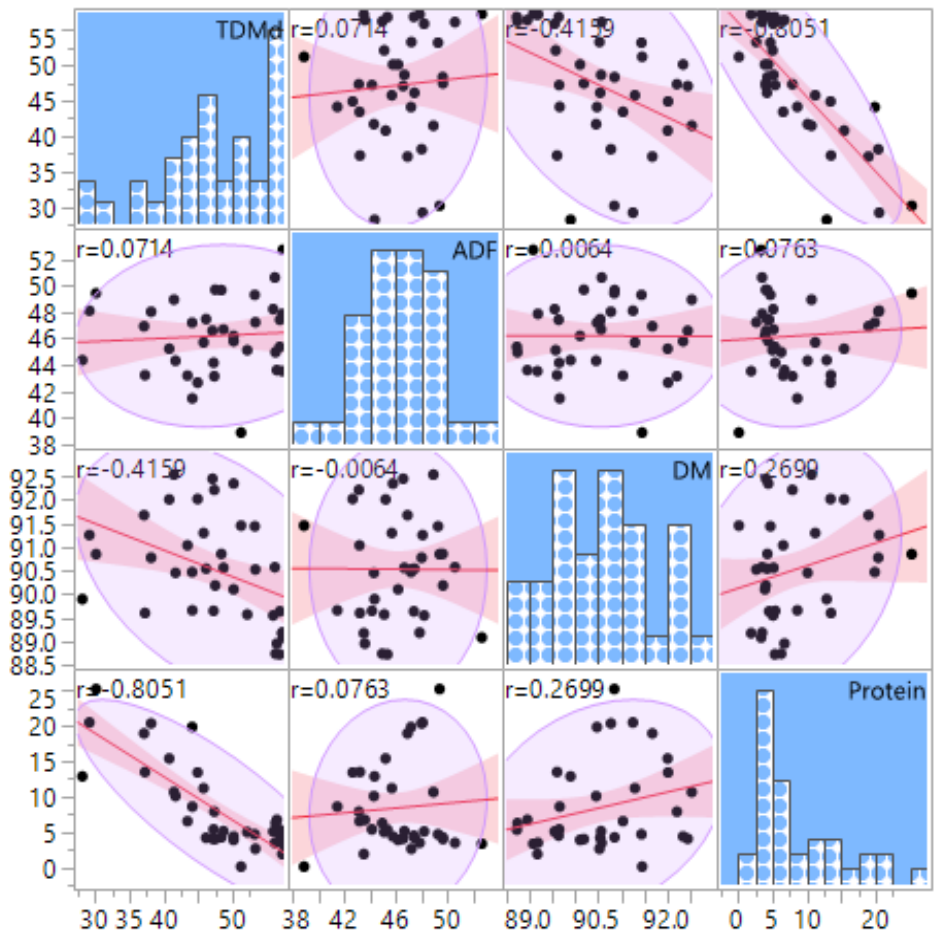


Figure 8. Representation of the effects of the applications on the examined features via scatter plot matrix.

Conclusion

In the study, it was determined that the torrefaction applications were effective on dry matter ratio, protein, and in vitro digestibility, but were not effective on the ADF ratio. Low-term torrefaction applications significantly increased the dry matter ratio, while high-power and long-term torrefaction increased the protein ratio. In vitro, digestibility decreased as the severity and duration of torrefaction increased. As a result, a material with high dry matter and high protein content but low in vitro digestibility was obtained. Although the tested applications reduced the value of grain maize harvest residues as cattle feed, the obtained material with high dry matter and protein content may be a suitable structure for future studies as a substrate for other biological or chemical cultivation aims. Since significant amounts of volatile and flammable gas were discharged during the applications, the potential to produce both energy raw materials and a nutrient medium may contribute to the expansion of the use of these materials.

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