

Chemical Composition and Methane Production Potential of Agricultural Residues: Olive Pomace, Cottonseed Meal and Red Pepper Processing Waste

Tarımsal Artıkların Kimyasal Kompozisyonları ve Metan Üretim Potansiyeli: Zeytin Prınası, Pamuk Tohumu Küspesi ve Kırmızı Biber İşleme Atıkları


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Abstract

Biogas is a renewable energy source produced through the anaerobic digestion of organic materials such as agricultural residues, manure, sewage, and food waste. This process involves the breakdown of these materials by microorganisms in the absence of oxygen, resulting in the production of a mixture of gases, primarily methane (CH₄) and carbon dioxide (CO₂), along with trace amounts of other gases like hydrogen sulfide (H₂S) and ammonia (NH₃). Biogas production from agricultural residues like olive pomace (OLV), cottonseed meal (CTM), and red pepper processing (RPP) waste holds promise for sustainable energy generation and waste management. This study investigates the chemical composition and methane production potential of these residues, emphasizing their protein, fat content, and Acid Detergent Fiber (ADF)/Neutral Detergent Fiber (NDF) ratios. Chemical analyses revealed significant variations among the materials, with cotton waste exhibiting the highest dry matter, organic matter, protein, and fat content, while pepper waste showed the highest ash content, and olive waste had the highest fiber (ADF and NDF) content. Methane production ranged from 0.34 to 0.45 m³ kg⁻¹ of organic dry matter (ODM), with cotton displaying the highest methane yield. Biogas production ranged from 0.61 to 0.78 m³ kg⁻¹ ODM, with cotton again yielding the highest biogas production. Methane content in biogas varied between 54.64% and 57.72%, with cotton also showing the highest methane content. At the end of the study, the dry matter (DM) and organic dry matter (ODM), ash, protein, fat, ADF, and NDF ratios of the materials were determined to be 85.51%-94.09%, 87.91%-92.92%, 7.08%-12.09%, 7.49%-15.93%, 3.76%-8.01%, 52.16%-71.07%, and 34.49%-55.58%, respectively. The materials showed chemical differences. Research highlights include the significant bioenergy potential of olive waste and cottonseed meals, alongside the environmental benefits of utilizing olive pomace for biogas production. Experimental findings reveal varying methane and biogas yields across materials, influenced by their nutrient compositions. The study underscores the viability of integrating these agricultural residues into biogas production systems, contributing to renewable energy initiatives and sustainable agricultural waste management practices.

Keys Words: Biogas, Methane, Agricultural organic wastes

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Öz

Biyogaz, tarımsal artıklar, gübre, kanalizasyon ve gıda atıkları gibi organik materyallerin anaerobik olarak çürütülmesi yoluyla üretilen yenilenebilir bir enerji kaynağıdır. Bu işlem, oksijen yokluğunda bu malzemelerin mikroorganizmalar tarafından parçalanmasını içerir ve bunun sonucunda başta metan (CH₄) ve karbon dioksit (CO₂) olmak üzere bir gaz karışımının yanı sıra eser miktarda hidrojen sülfür (H₂S) ve amonyak (NH₃) gibi diğer gazların üretilmesi sağlanır. Zeytin posası (OLV), pamuk tohumu küspesi (CTM) ve kırmızı biber işleme (RPP) atıkları gibi tarımsal atıklardan biyogaz üretimi, sürdürülebilir enerji üretimi ve atık yönetimi için umut vaat etmektedir. Bu çalışma, bu atıkların kimyasal bileşimini ve metan üretim potansiyelini, protein, yağ içeriği ve Asit Deterjan Sellülozu (ADF)/(Nötral Deterjan Sellülozu) NDF oranlarına vurgu yaparak araştırmaktadır. Kimyasal analizler, organik materyaller arasında önemli farklılıklar olduğunu ortaya koymuş; pamuk atığı en yüksek kuru madde, organik madde, protein ve yağ içeriğini gösterirken, biber atığı en yüksek kül içeriğini, zeytin atığı ise en yüksek lif (ADF ve NDF) içeriğini göstermiştir. Metan üretimi, organik kuru madde (ODM) başına 0.34 ile 0.45 m³ kg⁻¹ arasında değişmiş ve en yüksek metan verimi pamukta gözlemlenmiştir. Biyogaz üretimi, ODM başına 0.61 ile 0.78 m³ kg⁻¹ arasında değişmiş ve en yüksek biyogaz üretimi yine pamuk atıklarında ortaya çıkmıştır. Biyogazdaki metan içeriği %54.64 ile %57.72 arasında değişmiş ve pamuk en yüksek metan içeriğini göstermiştir. Çalışma sonucunda materyallerin kuru madde (KM) ve organik kuru madde (OKM), kül, protein, yağ, ADF (Asit Detergent Fibre) ve NDF (Neutral Detergent Fibre) oranları yapılan analizler sonucunda sırasıyla; %85.51-%94.09, %87.91-%92.92, %7.08-%12.09, %7.49-%15.93, %3.76-%8.01, %52.16-%71.07 ve %34.49-%55.58 olarak belirlenmiştir. Materyaller arasında kimyasal farklılıklar meydana gelmiştir. Araştırma, zeytin atığı ve pamuk tohumu küspesinin önemli biyoyakıt potansiyelini ve zeytin posasının biyogaz üretimi için kullanılmasının çevresel faydalarını vurgulamaktadır. Deneysel bulgular, besin bileşimleri farklı organik atıkların metan ve biyogaz verimlerini ortaya koymaktadır. Çalışma, bu tarımsal atıkların biyogaz üretim sistemlerine entegre edilmesinin yenilenebilir enerji girişimlerine ve sürdürülebilir tarımsal atık yönetimi uygulamalarına katkıda bulunma olasılığını vurgulamaktadır.

Anahtar Kelimeler: Biyogaz, Metan, Tarımsal organik atıklar

1. Introduction

Biogas production from agricultural by-products such as olive pomace (OLV), cottonseed meal (CTM), and red pepper processing (RPP) waste represents a promising strategy for both sustainable energy generation and effective waste management. These by-products, typically considered agricultural waste, can be repurposed through anaerobic digestion to produce valuable bioenergy, reducing environmental pollution and offering a renewable energy source.

Research has shown that anaerobic digestion of olive mill solid waste can result in substantial bioenergy production, significantly mitigating the environmental impacts commonly associated with olive oil production. For instance, Uddin et al. (2021) demonstrated that this process not only diverts waste from landfills but also creates an opportunity to harness energy in a sustainable manner. Moreover, Messineo et al. (2020) have investigated the anaerobic digestibility of both raw and pretreated olive pulp, underscoring the potential for hydrogen and biogas production under various conditions. This highlights the adaptability of olive by-products as feedstock for bioenergy generation under different treatment processes, further enhancing the feasibility of their use on a commercial scale.

Similarly, cottonseed hulls have been identified as an excellent substrate for biogas production. When combined with cow dung in anaerobic digestion processes, the biogas yield is notably enhanced (Venkateshkumar et al., 2021). This synergistic effect between different organic materials improves the efficiency and economic viability of the biogas production process. Investigations into other agricultural by-products, such as citrus pulp and olive pomace, have also demonstrated the feasibility of these materials for biogas production (Valenti et al., 2017). These findings support the notion that agricultural residues, which are often available in large quantities, can be effectively used as feedstocks for bioenergy production, promoting a circular economy in agricultural practices.

The potential for co-digestion processes, which involve the simultaneous digestion of multiple organic wastes, has been explored to enhance biomethane production further. Al-Addous et al. (2017) recommended co-digestion of olive and date waste as a means to optimize biomethane yields. This approach not only improves the overall efficiency of biogas production but also allows for the processing of a wider range of waste materials, thereby reducing waste disposal issues. In addition, other studies have explored the use of diverse feedstocks, such as coffee pulp, chicken feathers, and sugar beet pulp, in biogas production. These studies showcase the versatility and adaptability of anaerobic digestion processes to a variety of organic wastes, expanding the potential feedstock base for biogas plants (Sumardiono et al., 2021; Ziemiński and Kowalska-Wentel, 2016; Sumardiono et al., 2016).

Biogas production from olive mill waste offers significant environmental benefits. For example, Alonso-Fariñas et al. (2020) highlighted the potential for heat and electricity cogeneration through anaerobic digestion, followed by the composting of the digestate. This not only contributes to renewable energy production but also results in valuable by-products, such as compost, that can be used to enrich soil, thereby closing the loop in agricultural waste management. Moreover, the integration of biogas production into circular economy concepts, such as converting cassava pulp and wastewater into biogas, illustrates the potential for sustainable energy generation across various industries (Lerdlattaporn et al., 2020). This approach emphasizes the need for innovative solutions that can transform waste into resources, aligning with global sustainability goals.

Overall, research consistently indicates that biogas production from various agricultural by-products is a viable and sustainable approach. It can significantly contribute to renewable energy generation, reduce agricultural waste, and protect the environment by lowering greenhouse gas emissions and minimizing reliance on fossil fuels (Aybek et al., 2015; Develi et al., 2021). By converting waste materials into valuable energy resources, biogas production supports a more sustainable and resilient energy future.

In Turkey, cotton production is substantial, resulting in a significant amount of cottonseed pomace, also known as cottonseed meal or cake. The country produces approximately 800.000 to 900.000 tons of cotton fiber annually, leading to a considerable quantity of cottonseed by-products. Based on the proportion of cottonseeds to the overall cotton output and the processing methods employed, pomace production is estimated to be in the range of several hundred thousand tons per year (Semerci and Çelik, 2018; Tokel et al., 2022). This large volume of cottonseed by-

products presents an opportunity for biogas production, turning what would otherwise be agricultural waste into a valuable energy source.

Similarly, the olive oil industry in Turkey generates a significant amount of waste. Out of the total 1.700,000 tons of olives grown in the country, 1.300.000 tons of oily olives were processed, resulting in the production of around 175.000 tons of olive oil. This processing results in the generation of approximately 1.774,800 tons of waste annually, which includes 515.000 tons of pomace and 1.259.800 tons of wastewater (Kaya, 2024). From a biogas generation perspective, this represents a substantial quantity of raw material that can be harnessed for energy production, turning a potential environmental liability into a renewable energy asset.

The annual biomass residues produced from greenhouse crops, such as tomatoes, peppers, and eggplants, further contribute to the potential feedstock for biogas production. These residues amount to approximately 1.690.000 tons on a wet basis and 253.000 tons on a dry basis, with pepper crop residue alone accounting for 205.000 tons (wet) and 35.000 tons (dry) (Bilgin et al., 2015). Given the high production potential of these wastes, they can be effectively used as additives in biogas production, enhancing the overall biogas yield.

In this study, the chemical composition, methane production, and biogas yields of cottonseed meal, olive pomace, and pepper processing residues were determined. The findings underscore the viability of using these agricultural by-products as valuable resources for biogas production, supporting sustainable energy initiatives and contributing to the circular economy by transforming waste into renewable energy sources.

2. Materials and Methods

2.1. Procurement of olive pomace (OLV), cottonseed meal (CTM), and red pepper processing (RPP) waste.

The materials used were cottonseed meal, red pepper processing waste, and olive pomace from the olive oil processing plant (*Figure 1*). The materials were first sun-dried naturally and then ground to a size of 1 mm using an industrial grinder. A Kern precision balance was used to weigh the raw materials and the materials obtained in the experiments. A Microtest ash furnace was used to determine the ash and organic matter content. A Nüve oven was used to determine moisture content, a Velp UDK 139 protein meter to determine protein content, an Ankom XT10 fat meter to determine fat content, and an ANKOM NDF/ADF fiber analyzer to determine NDF and ADF content. The inoculum, with a solids content of approximately 2 %, was obtained from the mesophilic environment of the Kahramanmaraş Water and Sewerage Administration (KASKİ) Central Wastewater Treatment Plant and from biogas reactors with a pH of 6.8.



Figure 1. OLV (a), CTM (b), and RPP(c) waste

The Hohenheim Batch Test (HBT) method uses 100 mL glass syringes (*Figure 2*). The patented HBT syringe is shown in *Figure 2*. The syringe consists of the following components: (1) lubricant and seal, (2) 1 mL compartment, (3) gas chamber, (4) gas analysis port, (5) clamp, (6) glass syringe, (7) fermentation materials, (8) plunger, and (9) thin tube. A sealed tube and clamp are attached to the tip of the syringe to prevent gas leakage.

HBT syringes are placed in a hot water bath (*Figure 3*), which has 128 chambers and a heater to heat the water inside.

To determine the methane content in the biogas, an infrared-spectrometric methane-sensor "Advanced Gasmittler" D-AGM Plus 1010 device with a precision of 20 mL was used.

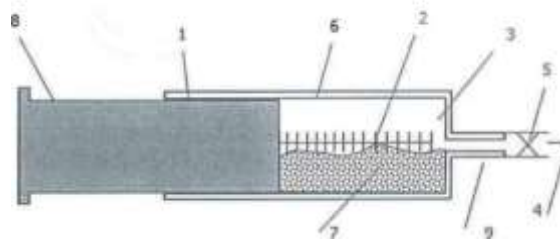


Figure 2. HBT Syringe



Figure 3. Water Bath

2.2. Method

Olive pomace (OLP), cottonseed meal (CTM) and red pepper processing waste (RPP) were dried and ground to pass through a 1 mm sieve. The dry matter, crude ash, organic matter, crude protein (by Kjeldahl method) and crude fat (by Soxhlet extractor method) contents of the prepared materials were determined according to AOAC, (1990). ADF and NDF contents were determined according to Van Soest et al. (1991). For the determination of the methane potential, all samples were weighed to 0.2 g on dry basis and filled into 100 mL test syringes in triplicate (VDI 4630, 2006). The net biogas (Net_{biogas}) was calculated by subtracting the inoculum ($T_{inoculum}$) from the total gas (T_{gas}).

$$Netbiogas = T_{gas} - T_{inoculum} \quad (Eq. 1)$$

2.3. Evaluation of Data

The chemical properties of the materials prepared according to the standard requirements were determined in the laboratory of the Department of Biosystems Engineering of Kahramanmaraş Sütçü İmam University, Faculty of Agriculture. These characteristics included crude protein (CP (%)), crude fat (CF (%)), % dry matter (DM), % organic dry matter (ODM), ADF (%), NDF (%), biogas and methane production, and methane content in biogas. Measurements, performed in triplicate, included mean and standard deviation values, statistical differences and analysis of variance (SPSS one-way ANOVA). Data were interpreted in tables and figures.

3. Results and Discussion

At the end of the study, the dry matter (DM) and organic dry matter (ODM), ash, protein, fat, ADF, and NDF ratios of the materials were determined to be 85.51%-94.09%, 87.91%-92.92%, 7.08%-12.09%, 7.49%-15.93%, 3.76%-8.01%, 54.47%-71.07%, and 35.24%-55.58%, respectively. The materials differed chemically. The highest DM (94.09%), ODM (92.92%), protein (15.93%), and fat (8.01%) were found in cotton waste, while the highest ash (12.09%) was found in pepper waste, and the highest NDF (71.07%) and ADF (55.58%) were found in olive waste. The lowest DM (85.51%), ODM (87.91%), were found in RPP, the lowest ash (7.08%), CP and the lowest protein (7.49%) and fat (3.76%) in OLV waste and the lowest NDF (54.47%) and ADF (35.24%) in CTM waste (Table 1).

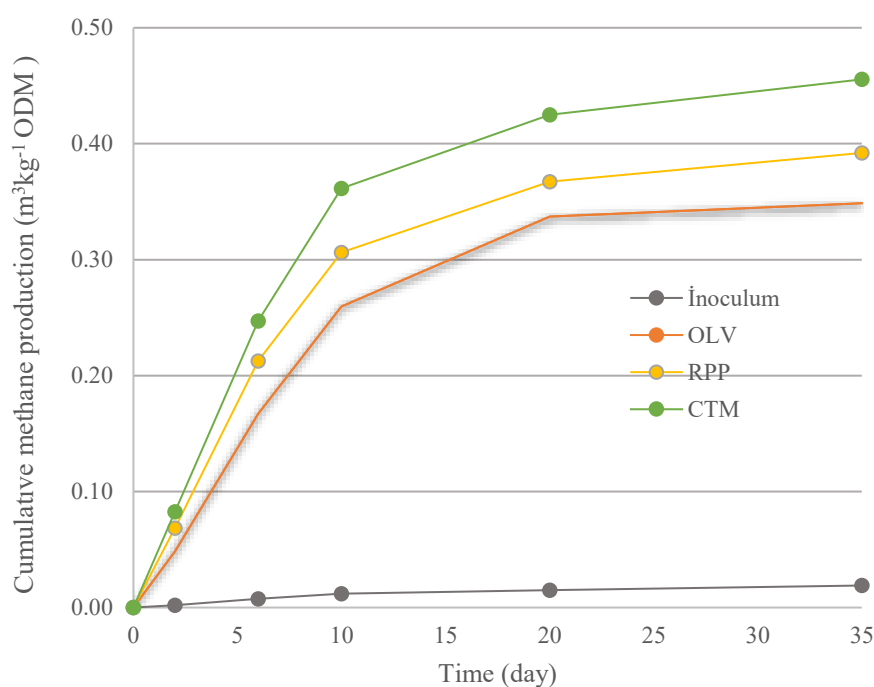
Table 1. Chemical Properties of the Materials

M	DM	ODM	Ash	CP	Fat	NDF	ADF
OLV	87.06±0.57b	92.05±0.15b	7.95±0.16b	7.49±0.35c	3.76±0.04c	71.07±0.64a	55.58±0.43a
RPP	85.51±0.62c	87.91±0.33c	12.09±0.34a	8.61±0.23b	6.74±0.16b	55.76±0.30c	36.49±0.47b
CTM	94.09±0.65a	92.92±0.08a	7.08±0.09c	15.93±0.17a	8.01±0.16a	54.47±0.30b	35.24±0.47c

$P \leq 0.05$; a,b,c,d, differences between the averages shown with different letters in the same column are important

3.1. Examination of the Average Methane Production of All Samples

The time-dependent methane production graph of all materials studied is presented in a figure, while the average cumulative specific methane, biogas values, and methane ratios in the biogas of the materials are presented in a table. In addition, the changes in the average cumulative specific methane and biogas production of the materials are shown in a *Table 2*.

**Figure 4. Time-Dependent Methane Production Graph**

As a result of the measurements, the cumulative methane production of the inoculum (starter) from anaerobic (oxygen-free) decomposition was approximately $0.0245 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$. The average cumulative methane production values of all materials were between $0.34\text{--}0.45 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$. The cumulative methane production was $0.42 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$ for cotton, $0.39 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$ for pepper and $0.34 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$ for olive. The average cumulative biogas production values of all materials ranged between $0.61\text{--}0.78 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$, with the highest cumulative biogas production occurring in cotton ($0.78 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$) and the lowest in olive ($0.61 \text{ m}^3 \text{ kg}^{-1} \text{ ODM}$) (*Figure 4*, *Table 2*). The methane ratios in the biogas varied between $54.64\%\text{--}57.72\%$, with the maximum methane ratio in cotton and the minimum in pepper (*Table 2*, *Figure 5*). Statistical comparison showed that the cumulative specific methane, biogas values and methane ratios in biogas of all materials were not significantly different ($P \leq 0.05$) (*Table 2*).

In the study, the average cumulative specific methane and biogas yields were highest for cotton, pepper, and olive residues, respectively. Chemical analyses indicated that higher protein and fat content, along with lower ADF and NDF ratios, led to higher cumulative methane and biogas production (*Table 1*, *Table 2*). NDF (cellulose+lignin+hemicellulose) and ADF (cellulose+lignin) consist primarily of cell wall components such as cellulose, lignin, and insoluble proteins. An increase in ADF and NDF ratios indicates a higher fibrous structure, which reduces digestibility and potentially reduces gas production. Amino acids from protein hydrolysis are fermented via reactions such as the Strickland reaction without the formation of hydrogen, while long-chain fatty acids (LCFA) from lipid hydrolysis can be converted to acetate and hydrogen at very low hydrogen partial

pressures, which are consumed only by methanogenic or sulfate-reducing bacteria (Dong et al., 2009; Hallenbeck, 2009). Proteins provide nutrients for biological degradation processes and thus create a buffering capacity during anaerobic digestion. However, high concentrations of ammonium nitrogen (NH_4^+) from proteins and LCFAs from lipids can create inhibitory conditions (Ariunbaatar et al., 2015; Chen et al., 2014) and affect hydrogen production during fermentation. Nitrogen is essential for the growth and activity of anaerobic microorganisms. Proteins and amino acids in feedstocks can serve as nitrogen sources to support microbial activity and enhance biogas production. Oils have a high energy content and can produce more biogas per unit weight compared to carbohydrates and proteins because they are composed of long-chain fatty acids, which produce significant amounts of methane when degraded (Chen et al., 2014).

Table 2. Average Cumulative Specific Methane, Biogas Values, and Methane Ratios in Biogas of the Materials

Materials	Methane				Biogas				Metan rate (%)
	Measurements			Avr. \pm Std error	Measurements			Avr. \pm Std error	
	1	2	3	(m^3kg^{-1})	1	2	3	(m^3kg^{-1})	
OLV	0.33	0.34	0.35	$0.34\pm 0.009\text{c}$	0.59	0.61	0.62	$0.61\pm 0.020\text{c}$	56.91a
RPP	0.40	0.37	0.38	$0.39\pm 0.019\text{b}$	0.74	0.69	0.71	$0.71\pm 0.009\text{b}$	54.64b
CTM	0.46	0.45	0.45	$0.45\pm 0.004\text{a}$	0.80	0.78	0.77	$0.78\pm 0.013\text{a}$	57.72a

$P\leq 0.05$; Differences indicated by different letters (a, b, c, d) in the same column for cumulative specific methane, biogas production, and methane ratios in biogas averages are significant.

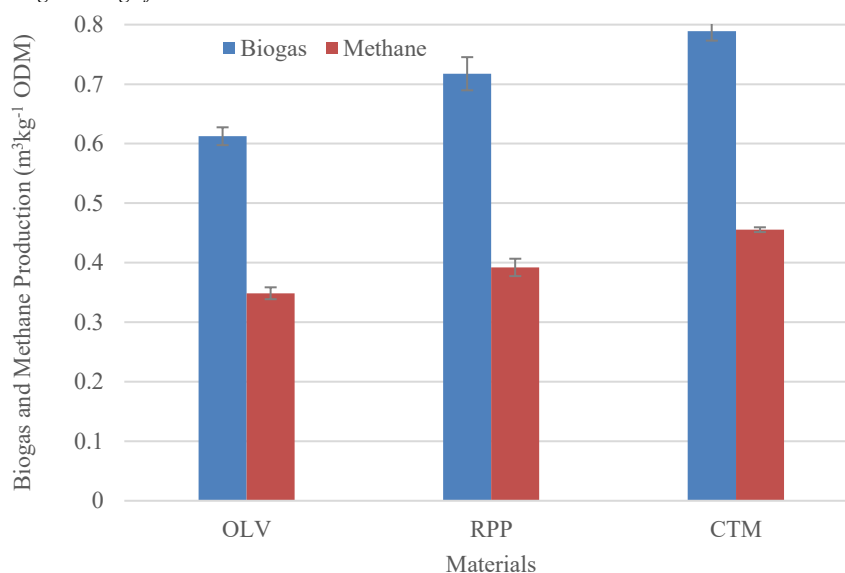


Figure 5. Changes in Average Cumulative Specific Methane and Biogas Production of Materials

In this study, an increase in protein and fat content correlated with increased methane and biogas production. Juanga et al. (2021) reported maximum methane production of 0.32 and 0.37 m^3kg^{-1} from processed and unprocessed cotton residues, Li et al. (2011) the daily average gas production achieved 508.57 mL/d, Borja et al. (2003) found methane production from olive pomace of 0.25 m^3kg^{-1} , Zupancic et al. (2024) in their study of pepper processing waste found biogas production of 0.68 m^3kg^{-1} and methane production of 0.4 m^3kg^{-1} . In this study, biogas production was found to average 0.71 m^3kg^{-1} and methane production was found to average 0.39 m^3kg^{-1} , Tufaner (2020), in its study of olive pomace, found a methane content of 62.6%. Zhang et al. (2018), obtained around 60% methane content in biogas. If we compare it with this study, the methane content was found to be slightly high. It can be said that the reason for this is due to the oil and persistent biodegradable content of olive pomace, which varies according to the variety.

Compared to the literature, methane and biogas production from olive and cotton residues were higher, while methane and biogas production from processed pepper residues were approximately similar. Variations in methane

and biogas production may be due to product diversity and associated differences in chemical and elemental composition.

4. Conclusions and Recommendations

The study determined dry matter (DM) and organic dry matter (ODM) content, ash, protein, fat, ADF, and NDF ratios, average cumulative specific methane and biogas yields, and methane content in biogas for CTM, RPP, and OLV.

- There were chemical differences among the materials.
- Average cumulative methane production ranged from 0.34 to 0.45 m³kg⁻¹ODM, cumulative biogas production ranged from 0.61 to 0.78 m³kg⁻¹, and methane content in biogas ranged from 54.64% to 57.72%.
- Statistical comparisons revealed no significant differences (P≤0.05) in cumulative specific methane and biogas yields or methane content in biogas among the materials studied.
- Chemical analyses showed that higher protein and fat content and lower ADF and NDF ratios correlated with increased cumulative methane and biogas production.
- Cotton, pepper, and olive waste have the potential to multiply as additives in biogas plants. Utilization of these components could be an important resource for sustainable energy production.

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Ethical Statement

There is no need to obtain permission from the ethics committee for this study.

Conflicts of Interest

The authors declare that there are no conflicts of interest with respect to the research, authorship, and/or publication of this article.

Authorship Contribution Statement

Concept: Üçok, S., Yang, X.; Design: Üçok, S., Yang, X; Data Collection or Processing: Üçok, S., Yang, X; Statistical Analyses: Üçok, S.; Literature Search: Üçok, S., Yang, X; Writing, Review and Editing: Üçok, S., Yang, X.

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