Technical Possibilities of Biogas Production from Olive and Date Waste in Jordan

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Using renewable energy sources provides a promising solution to minimize the overuse of conventional energy sources as well as to reduce pollution. Biogas technology is one solution that offers the conversion of waste streams to a renewable source of higher value. Anaerobic digestion of organic waste from industrial processes produces energy in the form of biogas, which has an advantage of preventing odor release, and has minimal pathogens. In this study, two different sources of bio-waste were investigated for their biogas potential, namely palm date waste and olive pomace. All of the samples produced biogas; however, the amount produced was only 20% to 40% of what conventional substrates typically produced. Producing biogas that uses only olive biomass offers a solution to the waste disposal problem, but it is not efficient for biogas production. For optimal heat utilization and maximizing biogas production, mixing of different feedstock was identified as a valid solution. Hence, a model of mixing other sources of bio-waste, such as chicken manure, can activate sludge and is proposed to boost the biogas production.

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INTRODUCTION

The Hashemite Kingdom of Jordan is located in the centre of the Middle East with a total area of 88,778 km^2 (DOS 2015a). Due to recent instability and revolutions in the Arab region, Jordan hosts many refugees and as a result the population has rapidly increased. The 2015 census assessed a population increase of more than 9.5 million, while it was only about 7 million in 2011. Approximately 30% of the population is not Jordanian and are refugees, immigrants, or employees mainly from neighbouring countries (DOS 2015c). This unplanned rise in the population puts a tremendous pressure on the social and energy demands, which require novel solutions from the Jordanian government. Using renewable energy sources is a promising solution to cope with the increasing energy demands.

Because Jordan is short on fuels, it has had to depend on importing energy resources regardless of their type, which was approximately 97% of the total primary energy needed in 2015 (MEMR 2015). The Jordanian government strategy during the years 2015 to 2018

is to reduce the dependence on imported fossil fuels and other types of energy resources. This goal will be achieved by increasing the share of renewable energies from 2% in 2015 to 8% in the total energy mix by the year 2018 (MEMR 2015). Next to solar radiation and wind, biogas is considered to be one of the most promising renewable energy sources for Jordan that is not affected by time, weather conditions, or the location (Budzianowski 2016; Sárvári Horváth *et al.* 2016).

Municipal solid waste contains approximately 50% of organic-origin waste (Sweepnet 2014), and until now this waste has hardly been used as a resource in Jordan. According to a report in 2014, 48% of the total waste was landfilled, 45% was openly disposed, and the remaining 7% was recycled (Sweepnet 2014). In contrast, the amount of agricultural waste was more than 4 million tons per year. Landfills can lead to ecological problems due to leakage and gas emissions. Aljaradin and Persson (2012) proposed that anaerobic digestion was an environment-friendly solution to treat organic waste in Jordan.

The production of biogas through anaerobic digestion (AD) is considered as an environmentally friendly process utilizing organic waste produced as a by-product of the agricultural and farming industries (Appels *et al.* 2011). Moreover, it can help tackle the problem of the organic waste fraction of municipalities' waste and food industries' waste (Yadvika *et al.* 2004; Lorenz *et al.* 2013) . The main product is a renewable energy source called biogas, and the by-product is a digestate that can be processed further to produce bio-fertilizers (Amon *et al.* 2007; Lukehurst *et al.* 2010). In theory, all kinds of bio-waste can be used as a feedstock for the AD plants. However, the loading process should be carefully planned to allow the chain of bio-degradation by the anaerobic bacteria to yield the biogas at the end of the process (Rajagopal *et al.* 2013; Yuan and Zhu 2016). The degradation process is the result of the collaboration between different groups of bacteria in a way that each group forms the substrate of another group. Efficient processes can be achieved if the rate of each degradation process is balanced (Yong *et al.* 2015).

Due to the economical, environmental, and social benefits of bioenergy, biogas industries have been established across many European Union countries including Germany, Denmark, Austria, Sweden, Netherlands, France, Spain, Italy, United Kingdom, and Belgium. Other countries are still in the developing stage or evaluating these technologies (Sophie 2008; Budzianowski 2016; Sárvári Horváth *et al.* 2016). Approximately 80% of Jordan is considered arid (Hadadin *et al.* 2010). One of the major challenges Jordan faces is the water supply of its population. Therefore, it is important that the production of biogas is solely based on agricultural and organic waste. The use of specific plants, like Jatropha (*Jatropha integerrima*), solely planted for the production of energy cannot be considered as a viable approach.

Recently, several studies have considered the potential of using biogas production in Jordan. Previous reports discussed the use of organic waste and manure (Abu-Ashour *et al.* 2010; Momani and Shawaqfah 2013; Al-Hamamre *et al.* 2014). In addition, biogas production from olive mill was reported as being a promising method to reduce the impact of olive oil industry effluent on the environment. Tekin and Dagic (2000) studied the biogas production from olive pomace and used a 1 L anaerobic digester. The concentration of olive pomace was mixed with water to achieve 5, 10, and 15 wt%. The biogas yield was 0.028 to 0.09 $l\,g^{-1}$ TS added. Alagöz *et al.* (2015) investigated the effect of microwave and ultrasonic pretreatment on anaerobic digestion of olive pomace and wastewater sludge; 180 mL of $CH_4\,g^{-1}$ VS was reported for olive pomace without treatment, while a 24% to 52% increase of biomethane production was reported after treatment (Aylin Alagöz *et al.* 2015). Gunay and Karadag reviewed different approaches for anaerobic digestion of olive mill

effluent. Different technologies and various pretreatment methods were compared for their effect on the digestion process. A co-digestion process with a pretreatment of the olive effluent was recommended to improve the biomethane production (Gunay and Karadag 2015)

In this work, two important agricultural products, olive and date waste, were used for the evaluation of biogas production. To boost the biogas production of the used substrates, several mixing criteria were proposed.

Olives and Dates in Jordan's Agriculture

Olive trees are one of the most planted trees in Jordan and cover approximately 36% of the cultivated area (Bassam 2015). In the last five years, Jordan produced approximately 1.4% of the global production of table olives. A large percentage of this product is used for olive oil (IOC 2016). Olive oil production reached approximately 0.9% of olive oil's production worldwide (IOC 2016). As a by-product of this process, a large amount of olive pomace is generated. Currently, there is no clear process to eliminate this amount of waste, besides ambient drying and incineration for household heating. However, this solution is limited due to the health and environmental problems associated with the burning process, and the low heating values of the pomace. One promising solution of this problem is the anaerobic digestion of the pomace to produce biogas and a quality digestate as a bio-fertilizer (Tekin and Dalgıç 2000).

Another possible waste that can be utilized is from date and palm trees. In 2015, approximately 27,000 acres were planted with 477,800 palm trees. The annual date production in 2015 amounted to approximately 20,140 tons of dates (DOS 2015b). Thus, it is possible to utilize the waste produced from this growing sector, and it can be used as a part of other agricultural wastes. In this work, the characteristics of dates and olive waste, including their leaves, are presented and the possibilities to produce biogas from these substrates are discussed.

EXPERIMENTAL

To determine the biogas potential of dates and olive waste, the total solids, volatile solids, and calorific values were determined using standard procedures. The total solids (TS) and the volatile solids (VS) values were important factors to be determined prior to a biogas potential test to fix the ratio of substrate to inoculum. Generally, the value should be less than 0.5 Otherwise an unbalanced chain reaction may occur and result in the elimination of the methanogenesis step (VDI 4630 2006; Yuan and Zhu 2016). The biogas production under standard conditions was equally measured using an Eudiometer system, and the methane content was measured.

Materials

The inoculum for biogas potential tests was taken as thickened-digested sludge from the municipal wastewater treatment plant in Hamburg, Germany and incubated at 35 °C for 5 d before the start of the experiment. The incubation step was necessary to reduce the gas production in the sludge. Sodium chloride (NaCl), citric acid, and crystalline cellulose were acquired from Sigma Aldrich (Saint Louis, USA). All chemicals were used without further purification.

Sample selection

Representative olive oil industry pomace and pressed olive samples were collected from three different olive mills located in the north and the west of Jordan. The olive waste samples from Aldajani (Ajloun) and Hofa (Al-Wasateyeh) olive mills were air-dried in the sunshine, while the sample from the Alsa'adi mill olive pomace was freshly pressed and not yet dried. The green olive leaves were brought from Al-Wasateyeh, Jordan. Representative samples of palm leaves (green and dry) were collected from Al- Karameh, Jordan Valley.

Methods

Total solids

According to the German standard DIN 38414 (1985), for each sample, three dried crucibles were heated to 105 °C ± 2 °C for 1 h and left to cool down in a desiccator to the ambient temperature. Next, their weight was recorded. Then, the crucibles were filled with a certain amount of the samples and placed in an oven at 105 °C for 24 h. Finally, the crucibles were placed in the desiccator and left to cool down to the ambient temperature and their weight was recorded.

Volatile solids

According to the German standard DIN 38409 (1987), the dried sample from the TS measurement was ground to a particle size of < 0.25 mm. The particles were then filled in ceramic crucibles, weighed, and placed in a furnace. The heating program consisted of 20 min at 200 °C, 30 min at 300 °C, and finally 5 h at 550 °C with a heating rate of 10 °C/min. Then, the heated sample was placed in a desiccator and left to cool to the ambient temperature. Finally, the weight of the remaining ash was recorded.

Calorific value

The calorific values of the biomass were measured according to the German standard DIN 51900 (2005), using an IKA C 5000 pump calorimeter (IKA®-Werke, Staufen, Germany).

Anaerobic digestion and biogas formation potential

Anaerobic digestion tests were conducted in triplicate, following the recommendations of the Association of German Engineers Standards (VDI 4630 2006).

To estimate the biogas potential production from the samples, the Eudiometer system was used. It was composed of 500-mL reactors, each one connected to a Eudiometer tube, which had been previously filled with a barrier solution that consisted of 200 g of NaCl/L and 5 g/L of citric acid. The biogas produced in each reactor was released in the tube so that the volume could be measured daily *via* solution displacement. The biogas volume was calculated under ambient conditions of the climate room at 310 K and 1 bar. The samples of produced biogas were analyzed using a GC-TCD model HP 6890 (Hewlett-Packard, USA), which allowed for the detection of methane and carbon dioxide. A reference set was investigated for their biogas production. The results were discarded when the results were not comparable to the standard value.

Theoretical biogas potential

The theoretical biogas calculation was based on the feedstock availability and was performed according to several mathematical models to predict the biogas potential.

Hashimoto *et al.* (1981) and Tarvin and Buswell (1934) developed rigorous calculation methods of biogas production using various organic feedstock (Tarvin and Buswell 1934; Hashimoto *et al.* 1981). The two methods were equally reviewed and verified in comparison with other calculation methods (Nielfa *et al.* 2015).

The equation for methane potential was first introduced by Tarvin and Buswell (1934). The model is very effective and used in several studies (Nielfa *et al.* 2015). Equation 1 represents the theoretical production rate for any substrate:

$$BMP_{thCOD} = \frac{n_{CH_4}RT}{pVSS_{added}}$$

(1)

The total number of methane moles can be calculated using Eq. 2, once the chemical oxygen demand (COD) was obtained,

$$n_{CH_4} = \frac{COD}{64(g/mol)}$$

(2)

where (BMP_{thCOD}), *R, T, pVSS,* and *nCH₄* represent the gas constant, ambient temperature (K), the volatile solids (g), and methane moles (mole), respectively. Both Eqs. 1 and 2 are described by Nielfa *et al.* (2015).

RESULTS AND DISCUSSION

Basic Characterization

Table *1* shows the results of the tests for all 6 samples. As expected, the fresh leaves had lower total solids and organic dry matter than the dried leaves. The values of both types of leaves were comparable. The highest values were expected from the olive pomace. The olive pomace was made of residues from olive oil production, *i.e*. the fruit and kernel after the olives were pressed. The value of the Alsa'adi's olive pomace was much lower than that of Aldajani's and Hofa's olive pomace due to the higher level of water received during the raining season and lower than average temperature compared with other locations of sampling.

Table 1. Characterization of the Investigated Substrates for their Dry Matter, Volatile Matter, and Calorific Value

Sample	Total Solids (TS)	Organic Dry Matter (VS)	Calorific Value
	kg kg$_{FM}^{-1}$	kg kg$_{FM}^{-1}$	kJ kg$_{FM}^{-1}$
Olive Leaves	0.48	0.42	8917
Green Palm Leaves	0.46	0.44	8706
Dry Palm Leaves	0.77	0.69	14109
Aldajani Olive Mill Olive Pomace	0.90	0.86	19170
Alsa'adi Olive Mill Olive Pomace	0.57	0.55	11190
Hofa Olive Mill Olive Pomace	0.90	0.88	19710

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The calorific values of the 6 samples had similar proportions. The fresh leaves had the lowest values, while the olive pomace had the highest value. The same behavior in Alsa'adi olive pomace was observed with a much lower calorific value than the other two olive pomaces and dry palm leaves.

Biogas Formation Potential Test

After the basic characterization of the total solids, volatile solids, and the calorific values for the samples, the gas formation potential test (GB21) was performed to investigate the sample potential for the production of biogas. As shown in Table 2, the biogas potential (BGP) of the samples fell in the range of 92 $Nl \cdot Kg_{FM}^{-1}$ to 147 $Nl \cdot Kg_{FM}^{-1}$. The lowest value was observed in the Alsa'adi Olive Mill olive pomace, while the highest was observed in Hofa Olive Mill olive pomace. Moreover, the average BGP from the green palm leaves and the dry palm leaves were higher than olive leaves or olive pomace. However, the TS, VS, and calorific values for the pomaces of Aldajani Olive Mill and Hofa Olive Mill were similar; the biogas and methane potentials showed a noticeable difference. Considerably lower values of Alsa'adi Olive Mill olive pomace were observed.

Table 2. Characterization of the Investigated Substrates for their Biogas Formation Potential and Methane Potential

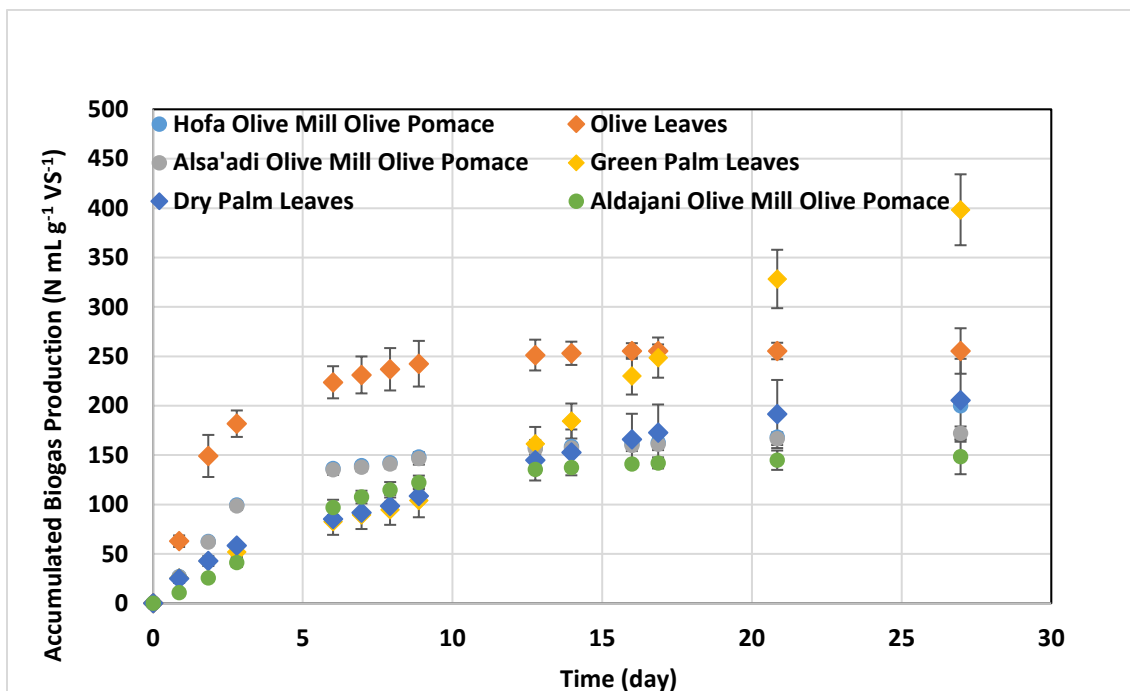| Sample	Biogas Formation Potential	Methane Potential
	NL kg_{FM}^{-1}	$L_{Methane}$ kg_{FM}^{-1}
Olive Leaves	107	71.29
Green Palm Leaves	142	94.75
Dry Palm Leaves	132	90.46
Aldajani Olive Mill Olive Pomace	125	90.18
Alsa'adi Olive Mill Olive Pomace	92	64.97
Hofa Olive Mill Olive Pomace	147	103.81

Fig. 1. Accumulative biogas production of the substrates using GB21 climate room method

Figure 1 shows the cumulative biogas production of the tested samples over 27 d. Most samples reached a plateau after 15 d. The only sample that deviated from this behavior was the green palm leaves. Furthermore, the olive leaf samples produced the majority of the biogas within 7 d of the start of the experiment. In addition, the samples from Alsa'adi Olive Mill and Hofa Olive Mill showed similar behavior in this test, which was comparable to that of the Aldajani Olive Mill olive pomace. In contrast, the behavior of the dry and green palm leaves were different.

Usually, biogas plants are design to use energy crops such as sugar beet, fodder beet, corn, and serials such as triticale and wheat. The biogas production from such crops range between 420 to 800 N ml·g^{-1}VS^{-1} (Carlini et al. 2015; Esposito et al. 2012; Macias-Corral et al. 2008; Weiland 2003, 2010). Comparing the results presented in this article to those available in the literature, shows that the yield of biogas smaller. However, the biogas technology can offer an environmental solution to the olive oil industries. Further, co-digestion with other organic substrate can boost the biogas yield.

Table 3 shows the results of the gas chromatographic (GC) analysis for the produced biogas for the tested samples. The main compositions of the analyzed biogas were methane, carbon dioxide, and some traces of oxygen. The methane percentages in all of the biogases produced ranged from approximately 66.6% to 72.4%. The lowest value was recorded for the olive leaves, while the largest was for the Aldajani olive pomace. Moreover, the percentages of carbon dioxide in all produced biogas ranged between 25.8% and 32.5%. The lowest value observed was for the sample from Aldajani olive pomace, and the largest was for the green palm leaves. Finally, the largest bio-methane production was for the Hofa olive pomace and the smallest was for the Alsa'adi olive pomace (Table 1).

Table 3. Biogas Compositional Analysis of Different Substrate Used

Sample	CH_4 (%)	O_2 (%)	CO_2 (%)
Olive Leaves	66.44 ± 3.21	1.32 ± 0.04	32.24 ± 1.54
Green Palm Leaves	66.65 ± 3.30	0.85 ± 0.07	32.50 ± 1.62
Dry Palm Leaves	68.64 ± 3.45	1.07 ± 0.05	30.29 ± 1.52
Aldajani Olive Pomace	72.37 ± 3.25	1.80 ± 0.07	25.83 ± 1.41
Alsa'adi Olive Pomace	70.85 ± 3.55	1.35 ± 0.03	27.80 ± 1.27
Hofa Olive Pomace	70.67 ± 3.41	1.81 ± 0.1	27.52 ± 1.05

Simulation of Feedstock Mixing Options

The measured biogas yield in this study was relatively low (Table 2) and it tackles the problem of waste management and disposal. The low biogas yield resulted from low phosphorus levels in the substrates, which is a basic element for the methanogenic optimal growth (Marques 2001; Macias-Corral *et al.* 2008; Fantozzi and Buratti 2009) However, the yield can be improved by mixing the cellulosic materials with other substrates (Macias-Corral *et al.* 2008). Feedstock mixing would provide additional nutrients, supplements, phosphorus, other minerals, and a balance of the feedstock with the C:N ratio to maximize the biogas production (Fantozzi and Buratti 2009). When the ratio of C:N increased, it resulted in a rapid consumption of nitrogen before the carbon was metabolized. As an illustrative example, Wu *et al.* (2010) found that C:N ratio of 20:1 was favorable for co-digestion of oat straw and wheat straw. In contrast, if the ratio of N:C was high it led to the accumulation of high concentrations of ammonium, which is toxic to the environment and hostile to the methanogens bacteria. The current C:N ratio of 50:1 explained that the low biogas yield was most likely attributable to the quick depletion of N (Wu *et al.* 2010). Fantozzi and Buratti (2009) found that mixing olive husk and inoculum (rumen fluid) increase the methane yield by ten fold in comparison to when live husk was used alone. This is an interesting finding and supports the assumption that olive paste requires additional feedstock mixing to maximize the biogas yield (Fantozzi and Buratti 2009). Mixing feedstock includes the local waste available in abundance, but unveils a management burden. The potential mixing feedstock includes animal manure, agriculture waste, and waste from slaughtering houses. Previous studies showed that various feedstock mixing improve significantly the biogas yield (Marques 2001; Macias-Corral *et al.* 2008; Fantozzi and Buratti 2009; Wu *et al.* 2010). The mixing feedstock would provide essential elements and a balance of carbon, phosphorus, and nitrogen (C:P:N). Mathematical modeling and process simulation offers practical tools for biogas prediction. One of the earliest models developed was the Hashimoto equation and was used efficiently to predict the biomethane from various feedstock (Hashimoto *et al.* 1981). However, the chemical oxygen demand (COD) content was utilized to predict the methane potentials in Eq. 1. Macias-Corral (2008) showed that the co-digestion of municipal solid waste and cow manure provides synergistic effect which tackles the imbalance of the nutrients and boosts the methane yield.

A spreadsheet that utilized the mathematical modeling was developed to simulate the addition of wastewater sludge (WS: 5% dry matter) and chicken manure (CM) at

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various maxing ratios with olive waste. The WS and CM was chosen because it is available abundantly in Jordan. It shows that mixing animal manure and olive waste would boost the biogas yield at a higher rate. Replacing the animal manure with household waste would not boost the yield due to its low nitrogen content. The mixing of feedstock was found to be an attractive process option to enhance the yield production of biogas. The mixing modification subsequently would improve the process economy remarkably and provide an economical tool for waste management combined with power generation from a biogas facility.

The following process was used to estimate the baseline for a successful production theme. The process will be based on processing 10,000 tons of olive past (OP) annually with several configurations. Because the olive waste provided low biogas yield due to an unbalanced ratio of C:N, several mixing scenarios were used with other waste to balance the C:N ratio and provide an additional source of phosphorus. Marques (2001) showed that the organic matter which, in association to its high C/N ratio and low pH, enhance the biological degradation processes. Table 4 shows several mixing scenarios with potential biogas production with the main feed kept constant at 10,000 tons. The biogas potential production from mixed feedstock was based on theoretical calculations using Eqs. 1 and 2, and an online model was developed by BiogasWorld. The olive waste was mixed with wastewater sludge (WS: 5% dry matter), chicken manure (CM), and at various mixing ratios. The WS and CM were selected due to their abundance in Jordan. These wastes are generally hard to manage and dispose. The first scenario (S1) presented the base case where only olive past (OP) was used in the proposed biogas plant. The second scenario (S2), had a ratio of 10:5:1 of OP:WS:CM, respectively. The third scenario (S3), had a ratio of 10:10:1 for OP:WS:CM, respectively. The fourth scenario (S4), mixed OP and CM and had a 1:1 ratio. A similar finding was obtained by Marques 2001 by finding that addition of piggery effluent enhanced to higher degree the anaerobic digestion of olive mill (Marques 2001). The aim of biogas usage is for combined heat and power (CHP) to the local farm community. The simulation was based on the solid content before digestion at 30% TS where the solid content should be adjusted for the digester type. Mixing the feedstock boosted the biogas production by several fold. This was due to the balanced ratio of C:N by other feedstock added to OP. A considerable amount of GHG reduction was achieved in the overall process. However, the mixing was tuned and optimized to maximize the overall biogas yield. The best obtained scenario, when the OP was mixed equally with CM, provided over 1.4 million m^3 of biogas, which is equivalent to about 28 million kWh of electricity. Altering the process configuration, for example biogas production in multi-stage reactor might offer another possibility of improving the overall yield of the process (Krishnan *et al.* 2016).

Table 4. Simulation Results of Several Feedstock Mixing Scenarios for Optimal Biogas Production

	Process Scenarios			
	S1	S2	S3	S4
Dry Matter EL (kWh)	254,040	2,584,200	2,715,600	2,847,000
(m^3/year)	850,000	1,310,050	1,380,238	1,442,813
Mixing Ratio	1:0:0	10:5:1	10:10:1	1:1
Feedstock	OP	OP:WS:CM	OP:WS:CM	OP:CM
Methane Potential GHG Reduction (tons CO$_2$ eq. yr^{-1})	55	557	587	614

CONCLUSIONS

1. Olive pomace and date palm leaves were successfully investigated using a GB21 test for evaluating the potential of biogas formation.

2. The biogas composition was mainly methane and carbon dioxide.

3. The maximum methane production was obtained for the Hofa Olive mill's olive pomace with a value of 103 NL·Kg$_{FM}^{-1}$.

4. Although the value was smaller than energy crops or animal manure, the process should still be considered for environmental reasons. Moreover, possible valorization of the process can be achieved by processing the digestate to a bio fertilizer.

5. Olive pomace could be utilized for biogas production if it was mixed with other organic waste to supply crucial nutrients for methanogenesis.

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