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Biogas production by an anaerobic co-digestion process from olive mill waste: effect of ultrasonic pre-treatment

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ABSTRACT

In the current study, the biochemical methane potential test was carried out to estimate the biogas production from Olive mill waste by using an anaerobic digestion process in the mesophilic phase (T = 37°C). The goal of this study is to promote the hydrolysis step so that a maximum volume of biogas and methane are obtained through performing an ultrasonic treatment of olive mill waste which is rich in organic matter (99.95%). The ultrasonic pretreatment was carried out along the following exposure times: 2, 7, 12 and 17 min which were investigated for the optimization. An increase in kinetic of biodegradability of the substrate and an improvement in methane yield were observed. The obtained results showed that the ultrasound pretreatment has a positive effect on the hydrolysis phase and the volume of produced methane, which results in an increase in soluble chemical oxygen demand levels from 73% to 78%, and a significant increase in methane production from 42 to 88.49 mL/g TVS.

Keywords: Anaerobic digestion; Olive mill waste; Pre-treatment; Ultrasonic

1. Introduction

Olive mill wastewater (OMW) is considered as a major ecological problem. Large quantities of olive mill wastewater are produced in the Mediterranean countries, causing a considerable harm to the environment [1,2]. Every year 1.4–1.8 million tons of olive oil is produced in the Mediterranean countries resulting in 30 million m^3 waste [3]. Moreover, it is highly polluting with a biological oxygen demand (BOD) of 100 g/L and a chemical oxygen demand (COD) of 200 g/L [4,5], which requires biological treatment.

Anaerobic digestion is a biological process that occurs naturally. In this process, anaerobic bacteria break down organic substances [6] leading to the production of energy products such as biogas [7]. This later can be used in the production of vehicles' fuel, electricity and heat, etc. [8–10]. Only in anaerobic digestion, hydrolysis is the limiting step in the production biogas [11,12]. One of the possibilities than can improve the performance of both hydrolysis and anaerobic digestion, is the use of technical pretreatment. The use of several pre-treatment methods is required including mechanical [13], chemical and thermal methods [14], in order to modify the physical and chemical properties of the effluents that are disrupting the microbes and cells to release the soluble organic matter. And this will make it easily degradable in anaerobic digestion [15]. Physical or mechanical pre-treatment includes: high-pressure homogenizer, mechanical thermal pretreatment, [16,17] and ultrasound pretreatment. According to some researchers, the application of physical pretreatment can increase

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the rate of hydrolysis and anaerobic biodegradability of substrates [18]. It can also increase the daily production of biogas and methane. It also reduces volatile products significantly during anaerobic digestion of activated sludge. A 15 min sonication pretreatment increased soluble COD concentration from 50 mg/L up to 2,500 [19,20].

Some authors observed that the ultrasound pretreatment increased the fraction of soluble COD alongside an increasing presence of BOD. Moreover, a rise in volatile acids release was noted. In addition to an increase in biogas production to more than 40% at low specific energy inputs, and an improvement in biogas of 41% tested in secondary sludge [21,22]. Working with secondary sludge, batch biodegradability tests show that by applying 30 kWh/m^3 of sludge, it is possible to increase biogas production by 42% [23].

Other authors have compared between the effects of ultrasonic pretreatment, ozonation pretreatment and thermal pretreatment done by [24] showed that the best results were obtained with ultrasound, with an energy of 6,250 or 9,350 kJ/kg TS and heat treatment at 170°C or 190°C, in terms of anaerobic batch biodegradability [24,25]. Compared thermal hydrolysis (170°C for 30 min), and ultrasonic pretreatment (30 kJ/kg TS) in thermophilic anaerobic digestion. It was observed that thermal treatment has led to the greatest increase in biogas production.

The anaerobic digestion is a very important process. Besides converting biodegradables wastes into renewable energy, it has an environmental aspect which is environmental protection. Therefore it has a double importance: energy production and environmental protection. Pretreatments applied to the substrate and the biomasses are generally considered to increase the volume and the composition of the produced biogas. Sure that it has an increase in energy consumed on one side related to applied pretreatment, but on the other hand it has an increase in produced energy and environmental protection. Note that according to the Food and Agriculture Organization (FAO) of the United Nations, one-third of all food produced for human consumption is wasted (approximately 1.3 billion tons of food waste per year). The AD of food and other organic waste increases the availability of nitrogen and phosphorus, which can be particularly beneficial in organic farming and can limit the use of inorganic fertilizers [26]. AD can also reduce odors [27] and potential pathogens [28].

This research aims at studying the effect of ultrasonic pretreatment on biodegradability, methane potential and volatile solid removal efficiency during anaerobic digestion in mesophilic conditions (T = 37°C).

2. Materials and methods

2.1. Origin of the substrate and inoculum

OMW (olive mill waste) used as substrate in this study were collected from an industrial olive oil production plant, located at Mila in to the east of Algeria. The sampling was done in three different points of the production process.

The sludge waste used as inoculum was obtained from the wastewater treatment plant (WWTP) of Oued El-Athmania, located in Mila (Algeria) which mainly deals with domestic effluents. These wastes were stored at 4°C to avoid possible biological and physicochemical reactions.

The physiochemical characterization of raw substrate and inoculums used in this study was carried out through the determination and measurement of different parameters such as: Total oxygen demand (COD), soluble chemical oxygen demand (COD_S), pH, total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), and total alkalinity. All this parameters were determined according to recommendation Standard Methods for Water and wastewater examination [29]. The obtained results were presented in Table 1.

2.2. Substrate pretreatment and preparation

In order to increase the solubilization of the substrate and therefore the volume of produced biogas, an ultrasonic pretreatment technique was applied. It is carried out using ultrasonic homogenizer (Elmasonic S100) operating at a frequency of 37 kHz). This technique was applied using 400 mL of substrate (OMW) for four different exposure times: t = 2, 7, 12 and 17 min. All determined and measured parameters for the characterization of pretreated substrate such as: COD, COD_S, pH, TS, VS, TSS, VSS and total alkalinity are presented in Table 2. All this parameters were determined according to recommendation Standard Methods for Water and wastewater examination [30].

2.3. Biochemical methane potential test (BMP test)

The Biochemical Methane Potential test (BMP tests) is carried out using a glass bottles with a rubber septum as

Table 1
Average chemical composition of olive mill solid waste and activated sludge (inoculum)

Parameters	Inoculum	OMW
pH	6.23	5.43
TS (g/L)	26.34	125.86
VS (g/L)	15.08	124.52
TSS (g/L)	30.86	77.05
VSS (g/L)	15.44	75.73
Total alkalinity (mg $CaCO_3$/L)	3,500	300
COD_T (gO_2/L)	23.71	118.01
COD_S (gO_2/L)	15.00	35.00

Table 2
Average concentrations of nutriments and traces elements

Nutriments and traces elements	Concentrations (g/L)
H_2PO_4	0.27
K_2HPO_4	1.12
NH_4Cl	0.53
$MgCl_2·6H_2O$	0.100
$CaCl_2·2H_2O$	0.075
$FeCl_3·6H_2O$	0.020

reactors. A volume of 34 mL of mixture (sludge + substrate with an Inoculums/Substrate ratio of 1:1) is introduced into each reactor. In order to avoid nutrient and trace element deficiency during the incubation period, 10 mL of a solution containing varying concentrations of these nutrients was added to each reactor (the concentrations are shown in Table 2). To maintain a pH around neutrality in all the reactors (equal to 7), a basic NaOH solution was used. The headspace in the reactors was flushed for few minutes in order to obtain anaerobic conditions. The reactors volume is adjusted with distilled water in order to have the same test volumes. The experiment includes the substrate (OMW) as positive tests and the blank tests (Inoculums). All the reactors are carried out in triplicates. In the end all reactors were sealed with a rubber stopper then drilled aluminum caps and placed in incubator operating in mesophilic phase at 37°C.

2.4. Biogas measurement

In incubation period, the daily production of biogas from each reactor was measured by the water displacement method by a column of acidified water (pH = 2) [7]. Throughout the experiment period, the bottles were shaken and moved around in the incubator once a day. The aim was to determine methane production from fresh substrate and therefore the methane produced from the controls was subtracted from the methane produced in the reactors containing samples.

Substrate methane production = displaced liquid by sample – displaced liquid by blank.The water displacement method allows us to determine the total biogas volume so to determine CH_4 and CO_2 content in each BMP test we used a KOH solution [7]. The content of CH_4 in biogas was determined as follows [31]. A known volume of the headspace gas (V1) produced in a serum bottle from Experiment 1 was syringed out and injected into another serum bottle which contained 20 g/L of KOH solution. This serum bottle was shaken manually for 3–4 min so that all the CO_2 and H_2S were absorbed in the concentrated KOH solution. The volume of the remaining gas (V2) which was 99.9% CH_4 in the serum bottle was determined by means of a syringe. The ratio of V2/V1 provided the content of CH_4 in the headspace gas. The results present in this work shows the accumulated methane production during incubation, the gas measurement stops when no production could be observed [7].

3. Results and discussion

3.1. Effect of ultrasonic pretreatment on the solubilization of the matter

According to Table 3, the results of physicochemical analyses after the ultrasound treatment, at different exposure times, performed on olive mill waste (OMW) show a very high percentage of VS in all tests. It exceeds 97%.

The TSS/VSS ratio decreases according to the pretreatment time, which is shown in Table 3. This can be explained by the fact that the material quantity, contained in the supernatant, has considerably increased. Thus, the untreated OMW is composed of 61.22% of particulate matter whereas that of the OMW treated diminishes in all pretreatments tested and reaching 45.53% for test C_3 (t = 12 min).

According to Table 3, the effect of ultrasound treatment on the soluble COD, varying from 35 to 90 g/L, where the biggest value corresponds to the C_4 test (t = 17 min) and exposure time increase leads to increasing the transfer from particulate phase into the soluble phase. This increase gets clearer by calculating the COD_S/COD_T ratio. It shows that the majority of the total fraction of the COD is converted into soluble COD varying between 30% and 78% the highest ratio corresponds to the test C_3 (t = 12 min). Solubilization occurs with a high percentage during the ultrasound exposure time t = 12 min and t = 17 min. This allows us to predict that the best biogas production will occur with the above exposure times.

3.2. Variations in the cumulative volume of biogas

The specific biogas production is an important parameter for the expression of the capacity of the waste to produce biogas; it is expressed in terms of volume of produced biogas relatively to the mass of substrate in terms

Table 3
Effect of ultrasonic treatment on the physicochemical parameters

Parameters	Ultrasonic treatment time				
	t = 0	C_1 (t = 2 min)	C_2 (t = 7 min)	C_3 (t = 12 min)	C_4 (t = 17 min)
pH	5.43	5.40	5.40	5.42	5.41
TS (g/L)	125.85	123.41	130.51	128.79	122.83
VS (g/L)	124.52	120.69	127.79	126.15	120.19
VS (%)	98.94	97.78	97.91	97.94	97.84
TSS (g/L)	77.05	71.82	74.25	58.64	69.20
VSS (g/L)	75.73	70.26	72.57	56.91	67.30
TSS/VSS	61.22	57.61	56.7	45.53	56.33
COD_S (gO_2/L)	35.00	50.00	60.00	85.00	90.00
COD_T (gO_2/L)	118.01	121.49	104.02	108.67	123.22
Ratio of COD_S/COD_T (%)	30	41	58	78	73

of the total volatile solids mL/g TVS [7]. Fig. 1 shows the cumulative volumes of specific biogas produced for the ultrasonically pretreated samples for different exposure times: 2, 7, 12 and 17 min after 49 d of incubation time. In the beginning of the incubation, biogas production was low (15 mL/g TVS) and it gradually increased after 10 d. This phase is the lag phase where microorganisms have started to adapt to the system. Moreover in this phase (hydrolysis and acidogenesis) the microorganisms try to converting carbohydrates, proteins and lipids to sugar, amino acid and fatty acid with the production of methane carbon dioxide and other volatile fatty acid (VFA).

The second phase from the 10th day and up to the 35th day is called the exponential phase. In this period, the production of biogas is starting to be affected by the pretreated substrates. Moreover, a maximum of cumulative specific biogas yield was attained 179.82, 176.43, 148.07, 123.87, 96.17 mL/g TVS were successively reached for exposure time t = 12, 7, 2, 17 min and CN (untreated) treatment tests. This means that the cumulative specific production of the biogas in test (t = 12 and 7 min), are almost two times the production of biogas of the non-treated substrate.

Starting from the 35th day, biogas production becomes zero because of the depletion of the substrate. This phase is called leveling phase.

Finally, the results justified a much higher biogas production by increasing the contact time of pretreatment and the best cumulative biogas production were obtained for exposure time 7 and 12 min.

Moreover, from Fig. 1 it can be understood that the cumulative biogas produced from samples pre-treated with the ultrasonic frequency were significantly higher than the non-treated sample. Reducing particle size by the ultrasonic frequency is likely the largest contributor to enhancing methane production that was observed in the current study. Results are consistent with prior studies [7,32] that indicated that gas production rates from anaerobic digestion of ultrasonically pretreated sludge were higher than those for untreated sludge.

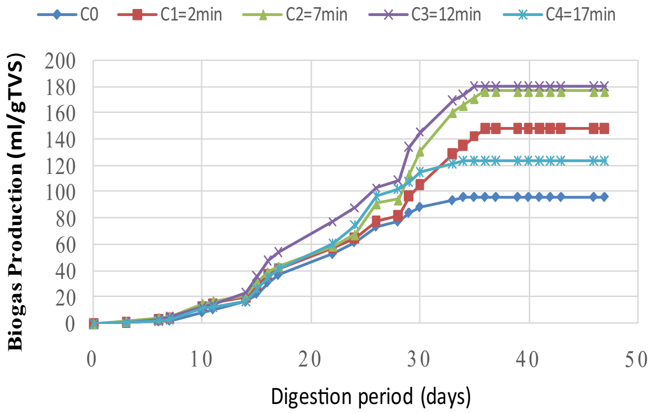

Fig. 1. Biogas production from olive mill waste pre-treated by ultrasound.

3.3. Variations in the cumulative volume of CH_4 and CO_2

Fig. 2a shows the cumulative specific bio-methane production from untreated sample and pre-treated with the ultrasonic frequency for different exposure times: 2, 7, 12 and 17 min. This figure clearly shows that the maximum methane production was observed in test C_2 (t = 7 min and C_3 (t = 12 min) with methane production equal to 88.49 mL/g TVS. Throughout the exponential phase, the percentage of methane in the biogas produced was determined. Fig. 3 shows that the percentage average of methane produced in the pre-treatments tests varies between 50.80% and 53.40%. Some studies [33] reported in the

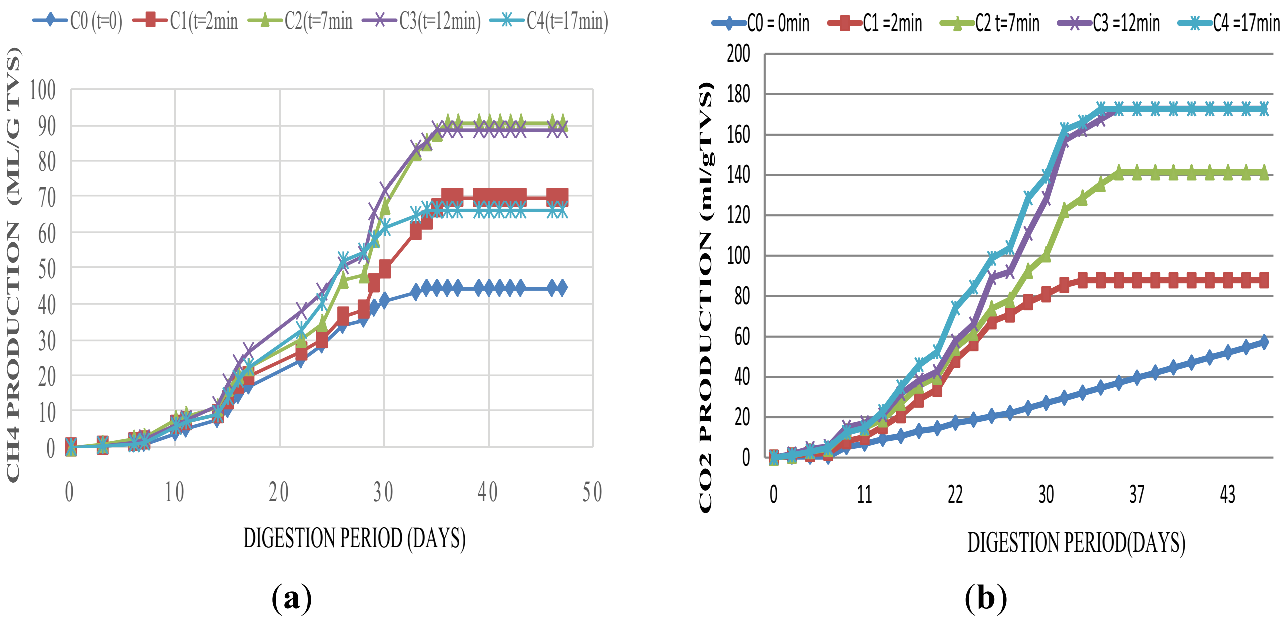

Fig. 2. (a) Cumulative volume of CH_4 and (b) cumulative volume of CO_2.

literature and concerning the solubilization of matter for example confirmed that the sonication solubilizes 25% of the thickened activated sludge and a CH_4 production of 30% reduction of the OM 54%.

3.4. Improvement in methane production for each pre-treatment

In terms of improving the ratio of biogas volumes produced by pre-treated or untreated olive mill waste:

$$\text{Improvement} = \frac{\text{Volume of biogas produced by the pretreated olive mill waste}}{\text{Volume of biogas produced by olive mill waste not pretreated}} \quad (1)$$

Methane production is 1.4–2.3 times higher than that of untreated samples for a period of time equal to 5 d. From the 5th day, a slight improvement appears from 1.5 up to 1.8 times for the C_3 test (t = 12 min), Fig. 4.

3.5. Characteristics of the liquid phase after incubation

The values obtained in the liquid phase after incubation is presented in Table 4. These parameters will be further discussed in detail.

3.5.1. TS and VS removal efficiency

The yields of the total solids including total volatile solids in various pretreatment tests. The output obtained from total solids go from 18.99% to 45% which confirms the degradation of the organic matter. The percentage of volatile matter varied from 48.49% to 58.93% with a better yield in test t = 12 min (Table 4).

3.5.2. Soluble COD and total COD removal yield

According to the values presented in Table 4, a considerable reduction in total and soluble COD is noted compared to those obtained before digestion in non-pre-treated substrate. Removal yield of 71.43% and 77.27% in total COD vs. 40.0% without pretreatment. And it is around 41 and up to 85% for soluble COD vs. 54% without pretreatment.

4. Conclusions

According to the obtained results, it is noticed that the mechanical ultrasound pre-treatment promotes the hydrolysis step and therefore an increase in the soluble COD.

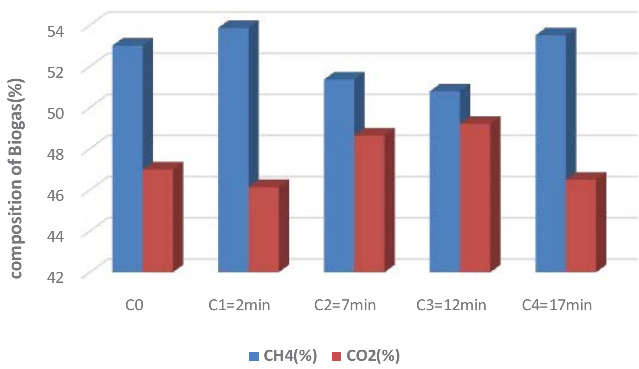

Fig. 3. Methane composition.

Table 4
Characterization of the liquid phase of reactors after incubation

	C_0 (t = 0 min)	C_1 (t = 2 min)	C_2 (t = 7 min)	C_3 (t = 12 min)	C_4 (t = 17 min)
Parameters ultrasonic treatment, before incubation					
pH	7.06	7.00	7.04	7.00	7.00
TS	8.62	8.55	8.74	8.70	8.54
VS	6.34	6.23	6.42	6.38	6.22
COD_T	937.5	984.38	890.38	843.75	965.63
COD_S	30.39	31.33	31.60	32.27	32.40
Parameters ultrasonic treatment, after incubation					
pH	7.70	8.01	7.40	8.00	8.10
TS	5.17	5.58	7.08	4.79	5.52
VS	2.47	2.99	3.30	2.62	3.00
COD_T	562.5	281.25	243.38	206.25	215.63
COD_S	14.06	9.38	18.75	9.38	5.00
Removal efficiency of VS and DOC					
TS %	40%	35%	19%	45%	35%
VS %	61%	52%	49%	59%	52%
COD_T %	40%	71%	73%	76%	78%
COD_S %	54%	70%	41%	71%	85%

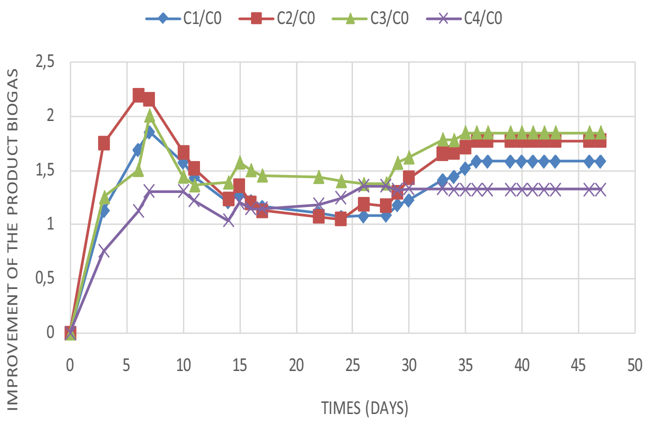

Fig. 4. Improvement of the product biogas.

This leads to a high solubilization rate (73% for t = 12 min, and 78% for t = 7 min). This solubility promoted biogas yield and methane in the mesophilic phase.

After ultrasound pretreatment method, a higher maximum of cumulative specific biogas production were 179.82 and 176.43 mL/g TVS for the exposure time of 12 and 7 min successively. This means that the cumulative specific production of the biogas in test (t = 12 and 7 min), were almost two times the production of biogas of the non-treated substrate. Moreover the produced methane was 1.4–2.3 times greater than that of untreated samples, for the first five days. From the 5th day, an improvement appears from 1.5 and up to 1.8 times for the C_3 test (t = 12 min).

Finally, the amenability of olive mill waste for anaerobic digestion was improved by the application of ultrasound pre-treatment method and it can recommended as pretreatment for others biodegradable substrate.

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