

## Biological treatment of Olive Mills Wastewater (OMW) and biogas production

Ghonwa Khaddour <sup>\*(1)</sup>, and Muhammad Manhal Al-Zoubi <sup>(2)</sup>

(1). Administration of technical affairs, General commission for scientific agricultural research, Latakia center, Syria.

(2) . Administration of natural resources research, General commission for scientific agricultural research, Damascus center, Syria.

(\*Corresponding author: Ghonwa Khaddour, E-Mail: [ghonwakhaddour@hotmail.com](mailto:ghonwakhaddour@hotmail.com)).

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### Abstract:

This research was conducted at the agricultural scientific research center in Latakia/ General Commission for Scientific Agricultural Research (GCSAR) in 2017/2018, to study the possibility of Olive Mill Wastewater (OMW) treatment, through the application of aerobic and anaerobic treatment techniques, in order to reduce the total phenolic content, and investigate biogas production, when anaerobic treatment is performed. The aerobic treatment included a reference (OMW without dilution and with no additives) for comparison, and three groups of experiments, in which OMW was adjusted with water by 45, 20 and 0%, and an additive was used with a 5% concentration of the mixture. Four additives were used in this work: molasses, bakery yeast, Azotobacter (AZ) and Phosphate Solubilizing Bacteria (PSB). Three replicates per addition were adopted. The results of the aerobic treatment showed a reduction in the total phenolic content, in all the experiments compared to the reference, and reached its maximum of 85% when PSB was added and with no dilution. The addition of PSB to OMW, with no dilution, was adopted for the application of anaerobic treatment. It was performed using a laboratory designed digester, and under a controlled temperature of 35 °C. During the anaerobic treatment, it was found that on the day 21 of the experiment, biogas production has started. At the end of the treatment, in the day 47 of the experiment, it was possible to reduce the total phenolic content by 73%, and produce 3 m<sup>3</sup>/day of biogas under regular air pressure.

**Key words:** Olive Mills Wastewater (OMW), aerobic and anaerobic treatment, Phosphate Solubilizing Bacteria (PSB), biogas.

**Introduction:**

Olive Mills Wastewater (OMW) is the liquid waste product of olive oil industry. This waste is characterized by its oily texture, dark color (brown to black), bitter taste and unpleasant smell, in addition to its acidic nature ( $\text{pH} \leq 6$ ) and its high salinity. It contains water (88- 94%); a high proportion of organic matter (5.5- 10.5%); a good amount of metal elements (0.5- 1.5%), such as nitrogen, phosphorus and potassium, and high level of solid particles. It is mainly composed of sugars, fatty acids, complex alcohols, and volatile organic compounds. Most importantly, the presence of phenols, plus the high oxygen demand (chemical and biological), gives this waste a toxic effect against living organisms [16, 23, 24, 32, 34, 39].

In terms of pollution effect, 1 m<sup>3</sup> of OMW is equivalent to 100- 200 m<sup>3</sup> of domestic sewage. Its uncontrolled disposal in water reservoirs leads to severe problems for the whole ecosystem and especially for the natural water bodies (ground water reservoirs, surface aquatic reservoirs, seashores, and sea). OMW dispersion on the ground affects soil porosity, and the cation exchange capacity of the soil, leading to change of environmental conditions for soil microorganisms and consequently to changes in the fertility of the soil. The direct application of OMW on plants inhibits the germination of different seeds and early plant growth of different vegetable species and may cause leaf and fruit abscission as well. Different types of crops show different reactions to OMW spreading and some of them may tolerate a certain amount of OMW during early growing stages, [1, 2, 3, 4, 10, 12, 13, 14, 15, 17, 25, 26, 27, 33, 34, 35, 37].

The Syrian olive oil industry consists of 983 working olive mills (according to a statistical study performed aside to this work), mostly concentrated in the north (56% in Aleppo and Idlib), the Syrian coast (about 42% in Latakia and Tartous) and the southern region (2% in Daraa and Sweida). These mills produce around one million cubic meters of OMW annually. Given the negative influences of this waste, mainly for the environment, it was necessary to find an appropriate solution against the random discharge in the sewage, surface and underground water resources, and on the soils [6, 7, 38, 39].

Different studies have discussed the possibility of OMW treatment to reduce its toxicity following different treatment techniques, including physical, chemical and biological ones [5, 8, 9, 11, 18, 19, 20, 24, 32, 34, 39]. However, these techniques were met with difficulties associated with high construction and investment costs, varying effectiveness in removing waste toxicity, in addition to the short duration of the mills operation (3-4 months).

The biological technique is one of the most effective treatment methods compared to others [32, 34, 39]. It includes two different treatment methods: aerobic and anaerobic.

Aerobic treatment method employs the use of microorganisms to break down biodegradable chemical species present in OMW. This method is considered environmentally friendly and enough effective, and it is usually used on low concentration streams; it relies on microorganisms that thrive under aerobic conditions (availability of oxygen and nutrients), [28, 29]. The toxicity of the phenolic fraction of the OMW can be biologically reduced by the action of these microorganisms.

Anaerobic treatment method is considered as a cost-effective alternative, if compared to aerobic treatment, especially for high organic industrial wastewater. Anaerobic process is mainly based on converting organic compounds to carbon dioxide and methane. This method of treatment has been reported as one of the most promising technologies for the disposal of OMW. Apart from the renewable energy generation in the form of biogas, anaerobic digestion presents some other appealing advantages since it allows small amounts of sludge generation, low nutrient requirements, reduction of greenhouse gases emissions and production of a liquid fertilizer, [22, 36].

Recently, interest in biogas production has increased due to higher fuel costs and improved energy sources. It can be produced by applying anaerobic treatment on organic waste (OMW in this study), [21, 30, 31, 40, 41]. Methane gas is one of the most important components of biogas by up to 50-70%. It is characterized by a blue flame when being burned and that its thermal energy is higher than the thermal energy of gas currently used in our homes by 2-3 times.

In this research, the possibility of Olive Mill Wastewater (OMW) treatment, through the application of aerobic and anaerobic treatment techniques is discussed, in order to reduce the total phenolic content, and investigate biogas production, when anaerobic treatment is performed.

### Goals and importance of the study

In light of successive climatic changes and their negative impacts on natural resources (water and soil), it is necessary to protect these resources and ensure their sustainability through the application of the treatment and recycling of contaminated wastewater coming from various sectors.

The goal of this research is to study the possibility of OMW treatment as a mean to mitigate the environmental impact of the negative random disposal of OMW, and also make use of OMW in the production of a clean, sustainable and inexpensive energy source, considering the high prices of present used fuel and the global tendency to renewable clean energy.

### Material and methods

#### Material

In this study, OMW from a classical pressing mill in Latakia city was used. Samples were analyzed in General Commission for Scientific Agricultural Research (GCSAR) laboratories. Table (1) shows some characteristics of used OMW. The table shows that the total phenolic content is around 1.1 g/l before treatment.

As shown in Table (1), OMW is an acidic liquid. It has a considerable content of potassium (K), nitrogen (N), phosphorus (P), which can directly impact soil and water resources characteristics and crop production. The electrical conductivity (EC) of OMW is moderate. The density ( $\rho$ ) of OMW is slightly higher than water density.

**Table (1): Main characteristics of OMW used in this study.**

Organic matter	K <sub>2</sub> O	P	Total N	pH	EC	$\rho$	Total phenols
	%			-	dS/m	g/cm <sup>3</sup>	g/l
5	0.71	0.13	0.16	5.66	6.26	1.06	1.1
<b>Walkley &amp; Black's method</b>	Flame photo-metric method	Murphy method	Berthelot reaction	pH meter	EC meter	Calculated Mass/volume	Folin-C method

Four additives were used for the treatment of OMW:

- Molasses, used as a nutrient for the bacteria already existing in OMW, and for the bacteria added in later steps.
- Bakery yeast, which is available in the market.
- Azotobacter (AB), which has been isolated from a natural local soil, capable of solubilizing inorganic nitrogen from insoluble compounds in OMW.
- Phosphate solubilizing bacteria (PSB) which has been also isolated from a natural local soil, capable of solubilizing inorganic phosphorus from insoluble compounds in OMW.

### Methods

The experimental work was carried out at the Agricultural Scientific Research Center in Latakia-GCSAR in 2017/2018.

### Aerobic Treatment of Olive Mills Wastewater

The procedure of aerobic treatment applied in this work included four groups of tests, and can be presented as follows:

- (75%) OMW+ (20%) Water+ (5%) Molasses.
- (50%) OMW+ (45%) Water+ (5%) Molasses.
- (95%) OMW+ (5%) Molasses.
- (100%) OMW (Reference).
- (70%) OMW+ (20%) Water+ (5%) Molasses+ (5%) PSB.
- (45%) OMW+ (45%) Water+ (5%) Molasses+ (5%) PSB.
- (90%) OMW+ (5%) Molasses+ (5%) PSB.
- (95%) OMW+ (5%) PSB.
- (70%) OMW+ (20%) Water+ (5%) Molasses+ (5%) AZ.
- (45%) OMW+ (45%) Water+ (5%) Molasses+ (5%) AZ.
- (90%) OMW+ (5%) Molasses+ (5%) AZ.
- (95%) OMW+ (5%) AZ.
- (70%) OMW+ (20%) Water+ (5%) Molasses+ (5%) Yeast.
- (45%) OMW+ (45%) Water+ (5%) Molasses+ (5%) Yeast.
- (90%) OMW+ (5%) Molasses+ (5%) Yeast.
- (95%) OMW+ (5%) Yeast.

In the aerobic treatment, a mixture of 100 ml of OMW, water, and addition according to the test being performed, was prepared, see Fig(1). The mixture was left exposed to air, through a cotton layer above the container, for 15 days. During the experiment, sample weight was daily monitored so that water was to be added if evaporation was noticed. The experiment was repeated 3 times and lasted for around 2 months. At the end of the experiment, the samples were collected to determine their total phenolic content, following the procedure of the Folin-C method. Figure (1) shows some of the OMW samples, during the aerobic treatment.



Figure (1):.Some of OMW samples, during the aerobic treatment.

### Anaerobic treatment of Olive Mills Wastewater

For the anaerobic treatment, the experiment was performed using a laboratory designed digester, see Fig (2).The digester consists of a barrel (A) of a volume 200 liters, which will be filled with around 150 liters of OMW mixture (if any additive). The barrel (B) is for OMW storing, if the test was needed to be repeated. At the top of the barrel (A), there are two openings: the first (C) is for filling the barrel with OMW, and the second (D) is a gas outlet with a pressure gauge (of a precision of 0.3 kPa) to measure the biogas pressure. The gas outlet is connected to a tire (E) to contain the resulting biogas. The gas outlet is also connected on the other side, using a metal connector T, to a household stove (F) to test biogas combustion. OMW temperature is adjusted within the barrel (A)

using a water heater (G) installed at the bottom of barrel. Figure (2) shows the laboratory designed digester used in this work. Note the empty gas tire (E) at the beginning of the test.

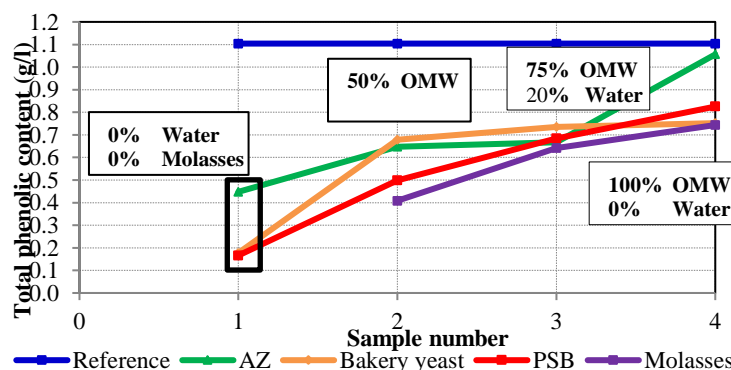


**Figure (2):** Laboratory designed digester used in this study. (A) Barrel to be filled with OMW mixture, (B) Barrel for OMW storing, (C) an opening for filling the barrel with OMW, (D) a gas outlet, (E) a tire to contain the resulting biogas, (F) a household stove to test biogas combustion, (G) a water heater.

The results of the aerobic treatment mixture, that achieve the highest reduction of total phenolic content was adopted for the anaerobic treatment. The barrel (A) of the laboratory digester was filled with the mixture, then was closed. The temperature was fixed at 35°C during the whole experiment using the water heater, and the pressure was monitored daily to determine the start point of biogas production. The experiment was ended when the tire was completely filled with gas. At the end of the experiment, a sample of the treated OMW was collected to determine the total phenolic content.

### Results and discussion

The total phenolic content results within OMW reference and aerobic treatment samples are shown in Figure (3) for the four groups of tests. The results shows that the total phenolic content in all aerobically treated samples was lower than in OMW reference samples (1.1 g/l).



**Figure (3):** The total phenolic content within OMW reference and aerobic treatment.

It is shown that the lowest total phenolic content (highest reduction 85%) is obtained when PSB was added and with no dilution (i.e. 95% OMW+ 0% Water+ 5% Phosphate Solubilizing Bacteria (PSB) solution), note points in the black box.

The addition of PSB to OMW, with no dilution, was considered for the anaerobic treatment experiment as it represents the treatment of the highest reduction in the total phenolic content.

During the anaerobic treatment experiment, it was found that on the day 21 of the experiment, biogas production has started, whereas the pressure gauge indicated an increase in the pressure within the barrel (A). The delay in biogas production can be attributed, on one hand, to the acidic nature of OMW and the accumulation of fatty acids, and to the slow growth of biogas-producing bacteria on the other hand. The biogas production rate reached 3 m<sup>3</sup>/day under regular air pressure at the day 47 of the experiment. This value was calculated following the Boyle Gas law:  $P_1V_1 = P_2V_2$ . Where:  $P_1$ : is gas pressure,  $P_2$ : is atmospheric pressure (equal to 100 kPa),  $V_1$ : is gas volume within the barrel and tire under  $P_1$  pressure, and  $V_2$ : is gas volume within the barrel and tire under  $P_2$  atmospheric pressure.

The analysis of OMW sample after the anaerobic treatment showed a reduction in the total phenolic content by 73% (0.3 g/l compared to the initial value of 1.1 g/l).

Figure (4) shows the laboratory designed digester in the day 47 of the experiment. The change in pressure can be observed on the pressure gauge in Figure (4-A) and the biogas tire filled in Figure (4-B) compared to the tire state in Figure (2) which presents the configuration at the beginning of the experiment.



**Figure (4): the laboratory designed digester in the day 47 of the anaerobic treatment experiment. A: Pressure gauge showing an increase in biogas pressure, B: The biogas tire filled.**

The results introduced in this work coincide with the one presented in other studies [5, 18, 22, 23, 27, 28, 29, 30, 32, 38, 39]. Basically, the biological treatment of OMW allows reducing its total phenolic content. However, depending on several conditions (type of additive, temperature and the percentage of organic matter within OMW+ others) can affect directly the amount of reduction of total phenolic content and biogas production.

### Conclusion and recommendation

In this research the possibility of Olive Mill Wastewater (OMW) treatment, through the application of aerobic and anaerobic treatment techniques is discussed, in order to reduce the total phenolic content, and investigate biogas production, when anaerobic treatment is performed.

OMW from a classical pressing mill of a total phenolic content equal to 1.1 g/l was used. Four additives were used for the aerobic treatment of OMW: molasses, bakery yeast, Azotobacter (AZ) and Phosphate Solubilizing Bacteria (PSB). The results of the aerobic treatment of OMW showed that it is possible to reduce the total phenolic content by around 85%, when PSB was added and with no dilution.

The addition of PSB to OMW, with no dilution, was adopted for the anaerobic treatment. A laboratory designed digester, that allows temperature controlling and pressure monitoring was used.

The digester was filled with the mixture of OMW and 5% PSB solution and the temperature was set to 35°C during the whole test. The experiment was ended when the gas tire of the digester was completely filled with gas. The results showed that on the day 21 of the experiment, biogas production has started, and reached 3 m<sup>3</sup>/day under regular air pressure at the day 47 of the experiment. The analysis of OMW sample after the anaerobic treatment showed a reduction in the total phenolic content by 73% (0.3 g/l compared to the initial value of 1.1 g/l).

Accordingly, It is found that the application of biological treatment (aerobic and anaerobic) for OMW using PSB helps reducing its total phenolic content and thus its toxicity. It allows also the production of a clean, sustainable and inexpensive energy source. Therefore, it is recommended to perform OMW biological treatment before disposal process, to protect the natural resources and maintain their sustainability.

Serious concern arises for the subject of the direct use of OMW in agriculture due to the results of local and international studies which vary between the positive and negative, especially in the long term, which takes into account the cumulative effect of phenols, present in OMW, on soil fertility and productivity. Therefore, it is necessary to conduct several studies focusing on the cumulative effect for using OMW in agricultural for a period of time not less than 10-15 years.

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## المعالجة البيولوجية للمياه الناتجة عن عصر الزيتون (الجفت) وإنتاج الغاز الحيوي

غنوة خضور<sup>(1)</sup> \* ومحمد منهل الزعبي<sup>(2)</sup>

(1). إدارة الشؤون الفنية، الهيئة العامة للبحوث العلمية الزراعية، مركز اللاذقية، سورية.

(2). إدارة بحوث الموارد الطبيعية، الهيئة العامة للبحوث العلمية الزراعية، مركز دمشق، سورية.

(\*المراسلة: د.غنوة خضور، الإيميل: [ghonwakhaddour@hotmail.com](mailto:ghonwakhaddour@hotmail.com)).

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### الملخص:

نفذ هذا البحث في مركز البحوث العلمية الزراعية في اللاذقية/ الهيئة العامة للبحوث العلمية الزراعية عام 2018/2017، لدراسة إمكانية معالجة ماء الجفت، من خلال تطبيق تقنيات المعالجة الهوائية واللاهوائية، وذلك لتخفيض تركيز المركبات الفينولية الكلية، وإنتاج الغاز الحيوي منه عند تخميره لاهوائياً. تضمنت المعالجة الهوائية أربعة معاملات: الشاهد للمقارنة (ماء الجفت دون تمديد بالماء ودون إضافات) وثلاث مستويات أخرى من ماء الجفت ممددة بالماء العادي بنسب (45، 20، 0%) مع اعتماد إضافة بنسبة 5% للمزيج. شملت الدراسة أربع إضافات: المولاس، خميرة الخبز، البكتريا المحللة للأزوت (AZ) والبكتريا المحللة للفوسفات (PSB). اعتمد تصميم التجربة على ثلاثة مكررات للمعاملة الواحدة. أظهرت نتائج التخمير الهوائي إمكانية تخفيض تركيز المركبات الفينولية الكلية، في جميع المعاملات مقارنة بالشاهد، ووصلت نسبة التخفيض إلى أعلى قيمها 85%، وذلك عند إضافة PSB فقط إلى ماء الجفت دون الحاجة لتمديده بالماء. باعتماد إضافة PSB فقط إلى ماء الجفت عند تخميره لاهوائياً، وذلك باستخدام مخمر مخبري مصمم ومنفذ لهذا الغرض وبتوفير حرارة 35°C خلال فترة التخمير، لوحظ في اليوم 21 من التجربة بدء انطلاق الغاز الحيوي، كما أمكن في اليوم 47 من التخمير تخفيض تركيز الفينولات الكلية بنسبة 73%، وإنتاج الغاز الحيوي بمعدل 3 م<sup>3</sup>/اليوم بالضغط الجوي النظامي.

الكلمات المفتاحية: ماء الجفت، المعالجة الهوائية واللاهوائية، البكتريا المحللة للفوسفات (PSB)، الغاز الحيوي.