



Methane Gas Production through Co-digestion of Olive Mill Wastewater and Sewage Sludge

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Abstract

Treating wastewater by co-digestion processes reduces the amount of organic material in the wastewater and produces biogas. This work investigates the Co-digestion of Olive Mill Wastewater (OMWW) and domestic sludge to produce methane gas. The research involves studying the effect of mixing OMWW with domestic sludge at different mixing ratios on methane production rate. Different ratios of OMWW-sludge were prepared (100:0, 70:30, 50:50, 30:70, 10:90) with two replicate of each ratio. A total solid content of 5 g was fixed in each ratio, and the amount of each feed was calculated accordingly. The co-digestion process was monitored for 27 days under at a temperature of $37C^{\circ}$. The final total organic matter (TOM) was subjected to characterization, namely: total solid, total volatile solid, total nitrogen, total phosphor, and trace element concentration.

The results indicated that the productivity of methane gas increased with increasing the percentage of OMWW in the mixed feed. In addition, the amount of total nitrogen and total phosphor in the final TOM are within acceptable limits to be used as soil amendment. The concentrations of trace elements are below the maximum allowable limits. This finding supports that there is no negative side effect of the process on the environment.

Keywords: olive mill; wastewater; co-digestion; sludge; waste management.

I. Introduction

Olive is one of the major agricultural crops in the Mediterranean regions. 98% of the total surface area of olive tree culture and total productive trees are provided from the Mediterranean area [6] of which about 75% is produced in the European Union (EU) [8].

Olive is the major agricultural crop in Jordan. Over the last 15 years the demand for olive oil has increased, causing an increase in olive cultivation by 5% [4]. 127600 hectares are the total area of cultivated olives in the Kingdom which form 72 percent of the area that cultivated with fruit trees and 34 percent of all the cultivated area in Jordan [18]. There are about 130 olive mills in Jordan for the extraction of oil from olive and produce around 200,000 m³ of olive mill wastewater

OMWW annually [4]. There is a large amount of olive mill waste producing during oil extraction. Olive mill wastewater OMWW produced as a liquid by-product of the oil production, where OMWW presents a main environmental problem when it disposed without a good control or treatment [15].

Waste water that generates from the olive mill is produced as a result of:

a. Washing olive.

b. An olive`s water.

C. Water for washing the facilities.

d. Water used in extraction operation [11], [13].

In the traditional discontinuous processes, after the olives are washed and crushed, they kneaded with hot water, the paste that resulting after that pressed to pull the oil. The waste that generates from this process contain liquid waste that composed of olive juice, water was added and contains residual oil lastly, the vertical centrifugation is used to separate olive oil from the water .This method uses a little amount of water, but generate a big amount of water pollution as wastewater. There are a many countries use this method until now [5].

1.1. OMWW characteristics

Large amount of effluent is produced as a liquid form and a solid residue when mechanical procedures are taken place to extraction the olive oil in olive mills, the method that used in extracting oil and a system that applied have a large effect on the nature of the effluents from the olive mill [8].

Olive oil wastewater (OMWW) can be defined as a liquid by- product that generate from the olive mills when olive oil extract by pressure method or centrifugation methods [4].

A dangerous environmental problems are appearing when OMWW are discharged into the environment without any treatment processes or controls were applied because OMWW contain a high amount of chemical species that resist to degradation such as a phenolic compound and high content of organic chemical oxygen demand(COD) [19].In general, OMWW contains:

- a. water with 83 percent,
- b. organic compounds with 15 percent and,

c. inorganic compounds with 2 percent [1].

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Additionally it is characterized by high concentration of: a. Cations and anions,

- b. Chemical oxygen demand (COD),
- c. Biological oxygen demand (BOD),
- d. Polyphenolic compounds,
- e. Fat and,
- f. Nutrient [14].

Typically, OMWW can be described as a violet dark brown to black color, the load of its organic compound is higher than the organic load of domestic wastewater by 100-150 times and it is the highest percent among all components, it has a specific oil odor [1], its pH between 4 and 5, high electrical conductivity, high concentration of total suspends solid TSS and total dissolved solid TDS, traces of sugar, it has inorganic compound and the predominant component of inorganic is Potassium K (~4 g/L) [19], high solid content and finally high content of polyphenols. Some properties of OMWW are good (fertilizers)

The lower pH is caused by the presence of phenolic compound, the colors related to the color of olives that processed, the storage condition, maturing of olives, a technology that applied in extraction operation, and age of OMWW. The phytotoxicity effects depend on the concentration of phenols, suspended solid, and the presence of dissolved solids [11].

1.1.1. Main organic compounds

Organic materials include different components Fig. 1:





The chemical oxygen demand COD, biological oxygen demand BOD as a very high concentration reach in the maximum value to 220,000 and 100,000 mg 1^{-1} respectively, and it include also a high concentration of Fat, Oil, and Grease (FOG) [19]. Some of these compounds have a high toxicity and have a high stabilization cost [1], [19].

1.1.2. Inorganic compounds

There are many different species of inorganic matter that exist in OMWW includes cations; Potassium K^+ , Magnesium Mg^{2+} , Calcium Ca²⁺, Sodium Na⁺, Iron Fe²⁺, Zinc Zn²⁺, Manganese Mn^{2+} , Copper Cu²⁺ and anions such as Chlorine Cl⁻, Hydrogen phosphate H₂PO₄⁻, Fluorine F⁻, Sulfate SO4²⁻, Nitric oxide NO⁻. The largest concentration of cations is Potassium K^+ then followed by Magnesium Mg^{2+} while the predominant concentration of anions is Chlorine Cl⁻ and followed by Hydrogen phosphate $H_2PO_4^-$ which in this form decreasing pH and contribute to increasing acidity [6].

1.1.3. Microbial content of OMWW



Fig. 2: Forms of microorganisms in OMWW.

The clean water is consumed, whereas large amount of wastewater is generated in all stages of oil olive extraction with different. that depend on many parameters such as; harvesting time, olive variety, the olive ripening, use of pesticides and fertilizers, cultivation soil, and climatic conditions [6], [20].

1.2. Environmental impacts

OMWW affects soil and crops in the long run because it contains phenolic compounds, lipids, and salinity and should not directly used in cultivation areas. OMWW has concentrations of ionic species (K^{+1} , Na^{+1} , and HCO^{3-}) that cause a salinity of the soil if the OMWW applied in irrigation field without treatment processes [2] the method used in olive oil extraction is one of the parameters that the environmental impact of OMWW depended on them [13].

The inappropriate discharge of OMWW in domestic wastewater ponds causes the disruption of biological activities because of the presence of a large load of toxic organic compounds. The aerobic digestion in open system causes a strong odor and problems for surface and ground water[20], also anaerobic digestion causes a strong unpleasant odor and potential threat to surface and ground water [19] in addition a high amounts of solid waste are generated. The serious environmental problems due to the OMWW organic load and composition which caused by their resistance to degradation [8]. So the intense concern of environment of olive oil production must be considered [20].

OMWW is frequently dumped, untreated, either in soil or into water sources causing:

- a. Phytotoxicity,
- b. Proliferation of insects,
- c. Increasing salinity,
- d. Reducing the permeability of the soil,





- e. Decreasing the degree of aeration[15],
- f. Coloring of natural waters,
- g. Threat to the aquatic life,
- h. Causing surface and ground water pollution,
- i. Changing plant growth and,
- j. Pungent odors [18].

Therefore, precautionary measure must be done prior the discharge of OMWW into the environment (soil and water) and the disposal of OMWW directly without treating is unallowable [5].

1.3. Treatment processes

Treatment of OMWW could be initiated by sedimentation, pretreatment with lime before disposal or can be disposed-to artificial open ponds [20]. Some Mediterranean countries specified the rate of spreading of the OMWW for the purposes of enhancing the cultivation processes such as Italians. They allow a spread up to 80 m³ha⁻¹ annually as a natural fertilizer and no harmful effects for crops and the environment is indicted [14].

1.3.1. Detoxification processes

There are a several processes that can be used in the OMWW treatment this includes: physical method, physic-chemical methods (flocculation, coagulation, filtration, open evaporating ponds and incineration), thermal method, and biological treatment methods that cause the reduction of organic load from OMWW, digestion with other effluents or combined between them [4], [18] where all these processes are detoxification processes.

Physical treatment

Physical processes applied a mechanical means to separate the different phases (solids, liquids, and gases) from OMWW. However, physical processes unable to achieve acceptable limits of toxicity and organic load alone when it used in OMWW treatment. Physical processes include: dilution, evaporation and sedimentation, filtration and centrifugation, and dissolved-air flotation [16], [20].

Physio-chemical treatment

Many techniques used among physio-chemical treatment for OMWW to eliminate organic matter from the liquid phase [13] by adding chemical materials. These techniques are: Neutralization technique, flocculation technique, precipitation, adsorption, Chemical oxidation, and Ion exchange [20].

These processes, however, have a many disadvantages such as; sludge-disposal problems, low efficiency, and high cost; thus tend to use other integrated treatment methods to achieving a complete treatment [13].

Thermal treatment

OMWW can be concentrated by using thermal treatment which involves reducing the water content and total volume by these three options: physio-thermal processes, irreversible thermo-chemical, combined physical and biological [5], [7].

Biological treatment

Biological treatment is the most suitable process and least expensive wastewater methods [13]. The biodegradable chemical species that exist in OMWW can be degraded through this method by using microorganisms that breaking down these species with an environmentally consideration [20].Biological treatment can be achieved by using aerobic, anaerobic or combined between them[13].

The detoxification processes run by fungi is more effective than that run by bacteria that applied to degrade the phenolic compound. The removal rate of fungi for COD as 40 - 88 %, for phenols as 60 - 100 %, and for colouration as 45 - 80 %[8].

Aerobic processes applied as a pretreatment for reducing COD, TSS and phenolic compound which enhancing anaerobic treatment. Aerobic condition includes the availability of oxygen and nutrient where the aerobic microorganism thrives in these conditions [20].

Several researchers [6] studied the biological treatment under aerobic conditions using three different cultures (*Azotobacter chroococcum*, *Geotrichum candidum*, *Aspergillus terreus*) to examine the effect of aerobic pretreatment on the anaerobic digestion of OMWW. It was found that the rate of anaerobic degradation was increased by about 2.5–4.5 times more than anaerobic treatment without aerobic pretreatment. However, Aerobic treatment is not recommended due to:

- a. It is an expensive treatment.
- b. Needs dilution.
- c. High residence time is necessary.
- d. Needs pH adjustment
- e. Acclimatization of microorganisms.

Solving these problems and producing biogas anaerobically is an acceptable solution [11].

Anaerobic treatments include the degradation of organic matter and produce a biogas such as methane via a microorganism in condition of absence of oxygen [20].

There are many parameters that affect anaerobic digestion, which explained as follows:

- 1. Solids Retention Time (SRT): typical range of 15 25 days.
- 2. Temperature: Two kinds of temperature applied in anaerobic processes: 1) mesophilic with a design temperature (30 40 °C), and thermophilic with a design temperature (50 60 °C) [8].
- 3. pH: The optimal range of pH between 6.0 and 8.0 where the ideal range is between 6.5 and 7.5.
- 4. Alkalinity: The best value of alkalinity is between 2500 and 3500 mgl^{-1} [17].





- 5. Mixing: To achieve the homogeneity in the digestion processes the mixing is required and it is applied by: external mechanical mixers, internal mechanical mixers, recalculating the materials exist in digester by pump, or gas recalculating [17]. And the digester themselves can be designed to enhance the mixing by designing it as an egg-shape.
- 6. Volatile Fatty Acids (VFAs): The favorable value of VFAs is less than 1000 mg l⁻¹.

Anaerobic treatment degrade a moderate and high strength waste water that has a high amount of biodegradable organic matter and it is the basic biological processes for OMWW treatment. Anaerobic treatment is widely used especially for the production of biogases which have a big capability to recover energy, saving energy, and reduce operational cost [6].

Digestion with Other Effluents

To enhance the efficiency for reduction of feed COD and total phenols with no needs for addition of nutrients (nitrogen and phosphorous) is achieved when OMWW are mixed and digested with other effluents that rich in nutrients which known as co-digestion processes [13]. Anaerobic co-digestion refers to addition of waste other than municipal sewage sludge such as OMWW to conventional anaerobic digesters [17].

Reference [9] has reported the anaerobic co-digestion when OMWW mixed with rabbit and pigeon wastes, the system was operated with different ratios between pigeon waste and rabbit waste with OMWW(pigeon waste or rabbit waste: OMWW; (100:0, 80:20, 60:40, 40:60)). It was found that the gas production was decreased when the percent of OMWW increased that due to the presence of lignocellulos materials with low digestibility, low concentration of nitrogen that is required to microorganisms activity and the presence of polyphenols compound where it is difficult to degraded by microorganisms.

Combined treatment

A complete removing of OMWW pollutant can't be achieved effectively and ecologically satisfactory by using a single treatment process, where there are needs to apply pretreatment processes to achieve effective management of OMWW. The combination of detoxification and utilization of OMW for the production of valuable by-products are needed [6], [20].

Experimental studies show that there are processes can be applied as pretreatment processes to enhance anaerobic treatment these includes: ultrafiltration which response to remove lipids and polyphenols, also can be use centrifugation in separated phase which include a sedimentation in smaller volume [12].

When using ozonation and aerobic treatment with each other combined or applied ozonation before aerobic treatment the COD reduction of 82.5% can be achieved, which greater than the percentage of COD reduction when ozonation and aerobic processes are applied alone [20].

1.3.2. Advanced oxidation processes

When wastewater has toxic and persistent pollutants, there is an attractive alternative that can be used to treat these pollutants these alternatives known as advanced oxidation processes (AOPs) which involve: ozonation, fenton's process, electrochemical oxidation, wet air oxidation [10].

II. Materials and method

2.1. OMWW and sludge sampling and characterizations

Olive mill waste water samples were collected from Modern Al-Rabbah station in Al-Karak governorate. The samples were characterized to determine their total solid, volatile solids, biological oxygen demand (BOD), electrical conductivity, phenols concentrations, total nitrogen, total phosphor, potassium (P), sodium (K), and others parameters.

Total solids defined as solids residue that remains after the sample was dried at 105 °C. This test conducted in the laboratory where sample was placed into a crucible and weighed (W₁), after the sample was dried in oven at 105 °C for 24 hours then weighed (W₂):

$$Water \ content = \frac{W1 - W2}{W1} * 100 \tag{1}$$

$$Total \ solid = 100 - water \ conent$$
 (2)

The volatile solids defined as solids residue after the sample was ignited at 550 $^{\circ}$ C in the furnace for 1 hour and then it was left to cool and then weighted (W₃):

$$Volatile \ solid = \frac{W2 - W3}{W2} * 100 \tag{3}$$

Biological oxygen demand (BOD) for OMWW was determined according to standard BOD₅ procedure.

Sludge samples collected from Merwed waste water treatment plant (WWTP) in Al-Karak governorate, sludge was taken after the secondary treatment stage. Total solid and volatile solid were conducted for the sludge. From the same waste water treatment plant inoculum bacteria was also collected from anaerobic tank.

2.2. Experiment design and procedure

The co-digestion experiments were carried out according to standard jar experimental procedure. Fig. 3 shows a schematic representation of the experimental apparatus. The apparatus is basically divided into parts, namely, fermentation reactor and gas collection flask. Dark glass bottles (4 L) are used as an avenue to accomplish the co-digestion of feed. For each feed,





two bottles with the same conditions are set up to ensure the reproducibility of results. Feed prepared with different ratios OMWW: sludge (100:0, 70:30, 50:50, 30:70, 10:90) where the total solid percentage of all samples were fixed at 50g. The bottles are immersed in a water bath to maintain the temperature of co-digestion at 37 °C. The temperature is held constant by using a temperature controller. The bottles are closed with rubber stoppers with allowance for tubing to carry the biogas for purification. The biogas is first purified from CO_2 and H_2S by simultaneous reaction and absorption in a bath containing caustic soda solution (1 M). The un-dissolved CH_4 gas released from the caustic soda solution and collected in a 1-L inverted graduated cylinder for volumetric quantification. The biogas from each bottle will be collected separately in an inverted graduated cylinder.

The experimental apparatuses are constructed and located in a closed and sunlight-free place with good ventilation to ensure safety. The experiments are started by introducing the feed in each bottle for co-digestion. To each bottle, 50 ml of inoculums is added to initiate the anaerobic digestion process. The generated gas is daily measured by recording the accumulated volume collected in the inverted graduated cylinder for four weeks.



Fig. 3: Anaerobic co-digestion system.

2.3. Samples analysis

After a period of four weeks the system was stopped and the samples were dried to determine these properties: total nitrogen (TN), total phosphor (TP), and heavy metals analyses using standard analysis procedures.

III. Results and discussion

3.1. Characteristics of OMWW and sludge:

Many properties such as; pH, electrical conductivity (EC), biological oxygen demand (BOD), concentrations of some heavy metals, and other properties for OMWW presented in the table 1.

Table1: Properties of OMWW.

Properties	This study	Ref. [4] values
pН	4.85 ± 0.15	4.91 ± 0.45
EC	$8.68\pm0.11(dS m^{-1})$	$7.64 \pm 0.46 (dS m^{-1})$
Phenols	1247 ± 58.7 (ppm)	2269±435.56(ppm)
Total nitrogen(TN)	$372 \pm 25.3 \%$	544 ± 322 %
Calcium (Ca)	116.6±23.12(ppm)	294 ± 125 (ppm)
Magnesium (Mg)	152.7±33.17(ppm)	227 ± 83.86(ppm)
Chloride (Cl)	510 ±34.31(ppm)	504±204.7(ppm)
Total phosphor	146.3±35.2(ppm)	245 ± 56.93 (ppm)
Potassium (K)	968 ± 47.6(ppm)	294 ±125.37(ppm)
Sodium (Na)	45.2 ± 6.3 (ppm)	59.7 ± 5.05 (ppm)
Cadmium (Cd)	nd <0.009 (ppm)	nd <0.001 (ppm)
BOD	450 (ppm)	36329 (ppm)

As it can be seen in Table 1, there are differences in the values of measurements when compared with those reported in literature [4]. It should be pointed out that samples of OMWW for both researches were taken from the same station This difference might attributed to sample collection time of OMWW, harvesting time of the olives, degree of ripening, climatic conditions and use of different pesticides and fertilizers.

3.2. Gas productivity



Fig. 4: Gas productivity for each ratio [volume (ml) of gas produced by 1 g of total solid].

Figure 4 shows Gas productivity for each ratio. The results show the volume (ml) of gas produced by 1 g of total solid present in the sample. The largest amount of OMWW has the largest productivity of gas, which means increasing gas productivity by increasing the amount of OMWW through the samples. The best ratio for gas productivity is 100:0 (OMWW: Sludge).In sample A, 1 g of total solid produced 155 ml of biogas.







Fig. 5: gas productivity for samples A.

Figure 5 shows the accumulated gas production for sample A. Both replicas of sample A exhibited typical gas production profile. However, the lag phase was different for the two replicas. Sample A1 started producing gas on the 12th day, while sample A2 started producing gas on the 6th day. Bacteria in sample A1 needed more time to adapt for the co-digestion process. The difference in the lag phase for the two replicas might be attributed to differences in concentration of inoculum used in each replica. In addition, the temperature distribution in each bottle might be not the same due to improper heating. In the log phase, gas production was high for both replicas during the first period of the log phase then became fair at later stages.



For **B** samples when ratio 70:30 (OMWW: sludge) lag phase of bacteria in **B**₁ sample is longer than that in **B**₂, where gas production started in **B**₁ on the 8th day while for **B**₂ on the 6th day (Fig. 6).



Fig. 7: Gas productivity for samples C.

For C samples when ratio 50:50 (OMWW: sludge), C_1 started to produce gas on the 10^{th} day while for C_2 started on the 9^{th} day (Fig. 7).



Fig. 8: Gas productivity for samples D.

In samples **D** when ratio 30:70 (OMWW: sludge), **D**₁ had shorter lag phase than **D**₂, beginning to produce gas on 6^{th} day while **D**₂ started on the 16^{th} (Fig. 8).



Fig. 9: Gas productivity for samples E.





For samples **E** when ratio 10:90 (OMWW: sludge), \mathbf{E}_1 started to produce gas on the 8th day while \mathbf{E}_2 started on the 6th day (Fig. 9).

Gas productivity for all samples was stopped on the 27th day where bacteria entered their death phase and all organic matter was exhausted.

For all ratios studied, the gas productivity profile for each replica has the same trend. However, values of gas productivity for samples with same conditions are different; it is maybe there is a problem in heat distribution within the water bath and the pump that used not enough. Therefore, a better heating distribution system may be required.

3.3. Heavy metals, Total nitrogen, and Total phosphor

Heavy metals, total nitrogen, and total phosphor were analyzed to assess environmental effect if this treated water can be disposed into the water stream, valleys, and water bodies or whether to be used for irrigation purposes. There is a maximum allowable concentration for each metal. Similarly, there is maximum allowable concentration for total nitrogen and total phosphor in reclaimed water or to be used for irrigation purposes.



Fig. 10: Concentrations of copper (ppm) for all ratio.

As shown in Fig. 10 the concentration of Cu in treated wastewater is more than the allowable limit as per the Jordanian standard for reclaimed water reuse for irrigation (0.2 ppm), and less than the allowable limit as per Jordanian standard for reclaimed water disposed into stream, valley or water bodies (1.5 ppm). Therefore, the treated water can be disposed into environment without any adverse effects (except sample D) but, cannot be used for irrigation purposes.



Fig. 11: Concentrations of Iron (ppm) for all ratio.

As shown in Fig. 11, the concentration of Fe in treated wastewater is less than the allowable limit present in Jordanian standard (5 ppm) for reclaimed water reuse for irrigation and disposal into stream (except sample C).



Fig. 12: Concentrations of Lead (ppm) for all ratios.

As shown in Fig12, the concentration of Pb in treated wastewater is less than the allowable limit present in Jordanian standard for reclaimed water reuse for irrigation and disposal into stream, except sample E; it exceed the allowable limit for reclaimed water for disposal into stream (0.2 ppm).



Fig. 13: Average concentration for Zinc (ppm).





As shown in Fig. 13, the concentration of Zn in treated wastewater is more than the allowable limit present in Jordanian standard for reclaimed water reuse for irrigation (0.2 ppm), and less than the allowable limit present in Jordanian standard for reclaimed water disposed into stream, valley or water bodies (5 ppm). Therefore, this reclaimed water can be disposed into environment without any adverse effects but, cannot be used for irrigation purposes.



Fig. 14: Total nitrogen concentrations (ppm) for all ratios.

For irrigation purposes if reclaimed wastewater used for picking flowers, productive trees, exterior road surfaces and green spaces, and also reclaimed wastewater that will be disposed into the water stream, valleys, and water bodies, the concentration of Total Nitrogen **TN** must be lower than 70 ppm. Also, the maximum allowable concentration of **TN** in reclaimed wastewater that will be used for irrigation purposes is 100 ppm. As shown in Fig. 14, the concentrations of **TN** in samples are lower than all maximum allowable concentrations except sample **D** which exceeded these limits.



Fig. 15: Total Phosphor concentrations (ppm) for all ratios.

The maximum allowable concentrations of **TP** in reclaimed wastewater that will be disposed into water stream, water bodies, and valleys is15 ppm, while reclaimed wastewater that will be used for irrigation purposes is 30 ppm. As shown in Fig. 15, all **TP** concentrations are lower than these limits as shown.

IV. Conclusion

Anaerobic co-digestion of olive mill wastewater OMWW and sewage sludge provides a good approach in wastewater management for wastewater treatment as well as giving a new and untraditional energy source for Jordan and may possibly reduce the amount of energy imported.

When different ratio of OMMW: sewage sludge mixed and treated, the highest gas productivity is achieved when the percentage of OMWW is 100%. The results confirmed that 1 g of total solid present in OMWW may produce 155 ml of biogas.

Treatment in this way provides a good alternative for waste management of wastewater instead of disposal in rivers, water bodies and valleys, causing many environmental problems affecting ground, surface water and other elements of the environment that ultimately affect human health.

The concentration of some of the heavy metals exceeded the allowable limit present in Jordanian standard for reclaimed water reuse for irrigation or disposal into stream and further treatment is recommended.

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