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# Production of Biogas from Olive Pomace

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**Abstract:** The present work treats anaerobic digestion as an alternative way for olive pomace treatment for energy and fertilizer production. Physico-chemical tests were conducted to identify the olive pomace biochemical methane potential (BMP). Effects of critical parameters, including, C/N ratio, dry matter and total volatile matter of the samples were carried out in order to optimize biogas composition. The first tests performed using 4g of pomace oil, with a mass ratio of inoculum: feedstock of 1:1, show a stable biogas production after 48hrs that can attain up to 200 ml/day. The total volume of biogas produced was 500 ml containing 51% wt methane.

Keywords: anaerobic digestion, biogas, olive pomace.

## 1. Introduction:

Recently, an increasing interest for biogas energy production has been seen globally<sup>1)</sup>. A major contribution to this was noticed in North African region, especially in Tunisia, which is related to the valorization of residues from the olive industry as an ecological and economical source. Indeed, it would allow the protection of the environment and the control of energy which represents one of the pillars of sustainable development and concerns a major part to the world.

The depletion of fossil energy resources and the awareness of the impact of greenhouse gas emissions on the environment<sup>2)</sup> have been the two main reasons for looking for alternative sources of energy.

Produced gas will be used for various energetic purposes, such as heat, combined heat and power or as a vehicle fuel, which can generate benefits on environmental and economic plans<sup>3)</sup>.

Population growth generates different types of wastes all over the world. Organic wastes decomposition causes large quantities of greenhouse gas emissions. Olive is a staple food in the Mediterranean countries. It widely grows in many countries of Africa, Europe and Asia.

International Olive Council estimated world olive production in 2018 to be around 6 000 000 tons<sup>4)</sup>. Out of this, olive oil production represents 20%, while 33% are generated in form of olive pomace<sup>5)</sup>.

The pomace is the solid residue from pressing that is formed of pulp and olive stones. This waste contains on average of 28.5% water, 41.5% hull, 21.5% pulp and 8.5% oil. The olive pomace is composed of a

lignocellulosic matrix (cellulose, hemicelluloses and lignin), phenolic compounds, uronic acids, and oily residues<sup>5), 6)</sup>.

The main difficulties to treat this solid waste are related to their high phenolic compounds content which makes them toxic for the environment. For example, Tunisia is among the top four olive oil producing countries. Its production represents a yearly average of 2 00 000 tons of olive pomace waste<sup>7)</sup>. These wastes are not used as Fertilizers because of their high acidity and they are not used for animals feed because of their toxicity and to the high content of glycoside compounds<sup>8)</sup>. That is why farmers find a serious problem to deal with this waste<sup>9)</sup>, which is linked to the formation of nauseous odors and contaminated air, a source of diseases for humans and toxic for the surrounding ecosystems<sup>10)</sup>.

On the other hand, a significant percentage of oil remains in the pomace<sup>11)</sup>. These oils can be recovered by chemical extraction using a solvent<sup>12)</sup> where highly acid oil is produced. The remaining pomace is called exhausted pomace. Because of its high acidity, olive pomace oil is often valorized in non-alimentary uses such as soap production.

Tunisia attaches great importance to olive oil industry and is more concerned by environmental issues related to it. As the sustainability is one of the priorities in its economic and social development plans<sup>9)</sup>, interest is growing in sustainable management of these wastes.

Anaerobic digestion of olive pomace waste could be a sustainable solution that makes it possible to generate income while dealing with wastes from the olive industry.

Anaerobic digestion is a complex biological process of decomposing biomass by bacteria that is activated in the absence of oxygen. At the end of the process, biogas and fertilizer are formed. A great deal of scientific research on similar raw materials shows the effectiveness of these treatments.

In this study, anaerobic digestion of raw and exhausted OP is studied in order to evaluate their stoichiometric, theoretical and biochemical methane production potentials<sup>13)</sup>.

## 2. Materials and methods

### 2.1 Feedstock sampling and pretreatment

Raw and exhausted olive pomace samples were furnished by ABOU EL WALID industry, where the raw olive pomace is treated with hexane in order to recover two products, a solid: the exhausted olive pomace and a liquid: olive pomace oil.

Raw and exhausted olive pomace (ROP and EOP, respectively) were grated using a mixer and then dried at 105 °C and stored at room temperature in sealed containers.

### 2.2 Samples characterization

A fine powder is obtained by milling dry OP using a Retsch MM vibratory mill B117130 ball mill.

Thermogravimetric analysis (TGA) was performed using the Setsys Evolution device in order to determine its composition. To do so, a first plateau was set at 105°C to determine humidity content. Then three consecutive plateaus at 250°C, 300°C, 500 °C to quantify hemicellulose, cellulose and lignin respectively.

Elementary analysis or estimation in (CHNO/S) is done using a Flash 1112 Series analyzer.

The protein content was calculated by multiplying by 6.25 the total nitrogen obtained from the CHNS-O analysis extract<sup>14)</sup>.

Total solids (TS), volatile solids (VS) and ash content of the samples were also according to AFNOR standard<sup>15)</sup>.

### 2.3 Determination of total lipids

Total lipid extraction was performed using solvents to extract oil from the samples using C. Gerhardt's SOXTHERM® rapid method. The total lipid content was determined using Equation 2 from the initial weight of the sample and the weight of the cylindrical glass vessel before and after extraction.

$$\% \text{ Lipid content} = \frac{W2-W1}{W3} * 100 \quad (1)$$

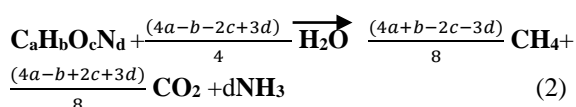
Where W1 = Weight of the container with pumice stone before extraction;

W2 = Weight of the container containing the lipid extract and the pumice;

W3 = Weight of the sample.

### 2.4 Estimation of the stoichiometric methane (B0) yield of the samples

The empirical formula ( $C_aH_bO_cN_dS_x$ ) was determined on the basis of the elemental composition (C, H, N, S, O) of the samples<sup>16)</sup>. The stoichiometric methane (B0) yield of the samples was determined according to Equations 2 and 3 established by Paul W et al.<sup>17)</sup>. The results are not precise, give maximum potential for methane and will often be much too optimistic because it does not take into account the effects of inhibitors, non-degradable materials, nor the energy demand of microbes.



In this Equation, under the assumption that the organic matter is stoichiometrically converted to methane, carbon dioxide and ammonia.

So the specific methane yield expressed in milliliters of  $CH_4$  per gram of volatile solids (VS) can therefore be calculated as follows:

$$B_0 = \frac{1}{8} * \left( \frac{4a+b-2c-3d}{12a+b+16c+14d} \right) * V_m \quad (3)$$

$V_m$  is the molar volume of methane under normal pressure and temperature.

### 2.5 Theoretical methanogenic potential's evaluation

Using the composition of the substrate and the degree of biodegradability, the yield of biogas / methane production is improved<sup>18)</sup>.

Depending on the concentration (in% TS) of carbohydrates, proteins and lipids in the substrate, the maximum theoretical yield (BT) of the samples is determined using equation 4

$$B_T = \frac{1}{100} (A \times Cl + B \times Cp + C \times Cc) \quad (4)$$

A= the specific methane yields of lipids = 1.014

B= the specific methane yields of proteins = 0.496

C= the specific methane yields of carbohydrates = 0.415

Cl= concentrations of the lipids

Cp= concentrations (based on % TS) of the proteins

And Cc= concentrations (based on % TS) of the carbohydrates

## 2.6 Evaluation of biochemical potential of methane (BMP)

The realization of the biochemical potential of methane was according to the protocol developed by Angelidaki et al.<sup>19)</sup> with minor modifications. The Table1 shows the BMP flasks composition

Table 1. The composition of BMP flasks

Parameters	ROP	EOP
Volatiles solid Substrate (g)	1.71	1.71
Volatile solid Inoculum (g)	1.71	1.71
Water (ml)	4	2
C/N ratio	55	49
pH	7.1±0.1	6.9±0.5

The fermentation of olive pomace was under anaerobic conditions at a constant temperature of 37 ° C. Control of the volume of gas is done by graduated bottles via the Mariott method<sup>10)</sup>.

## 3. Results and discussions

In the biomethane production process, the composition of the raw material which plays an important role for the determining of the methanogenic potential as illustrated in the equation B<sub>T</sub>. The biomass characterization is shown in Table 2.

Table2: Biomass characterization

Samples' Parameter*	ROP	EOP
Dry Matter (%)	4.6	4.2
Volatile Solids (%)	97.00±0,01	90.00±0,00
Lipids (%)	6.3	2
Proteins (%)	5.13	6
Hemicellulose (%)	19.8	18.9
Cellulose (%)	22.77	22.12
Lignin (%)	17.8	18.6

\*All data are given as a percentage of dry matter (DM).

### 3.1 The effect of the C/N ratio on methane production

The C/N ratio calculated in the ROP and EOP samples makes it possible to conclude that this raw material can not be used as the sole substrate for anaerobic digestion. C/N values are not in the optimal range, ie 20-30<sup>20)</sup>. For both ROP and EOP samples, the C/N ratio is high, 55.25 and 49.33 respectively, indicating that the nitrogen deficiency of olive pomace could lead to a decrease in the efficiency of methane production due to deactivation of methanogens and possible risks of failure of the whole process<sup>21)</sup>. However, attention must be paid to the nitrogen deficiency of olive pomace, and this nitrogen deficiency must be compensated for by a substrate with high nitrogen content, like cattle manure waste, or spent coffee ground are good biological sources of nitrogen<sup>22),23)</sup>.

### 3.2 Stoichiometric potential: Establishment of the empirical formula

The elemental composition of the different samples made it possible to establish their empirical formula according to the method defined by Murphy et al. <sup>24)</sup>. Table 3 shows the elemental composition of the different samples.

Table 3: Elemental composition and Atomic constituents of the samples

Sample	Chemical element	Elemental composition (% DM)	Mass contribution of each element(g/L)	Atomic mass	Ratio of mass contribution by the atomic mass	Number of moles/ mole OP
ROP	Carbon	45.31± 0.06	453	12	37.75	66
	Hydrogen	5.54± 0.06	55	1	55	96
	Nitrogen	0.82± 0.06	8	14	0.57	1
	Oxygen	41.51± 0.06	415	16	25.93	45
EOP	Carbon	47.36± 0.06	473	12	39.41	62
	Hydrogen	6.04± 0.06	60	1	60	94
	Nitrogen	0.96± 0.06	9	14	0.64	1
	Oxygen	45.52± 0.06	455	16	28.43	44

The following empirical formulas emerge:  $C_{66}H_{96}O_{45}N$  and  $C_{62}H_{94}O_{44}N$  respectively for raw olive pomace (ROP) and exhausted olive pomace (EOP). By integrating respectively, the number of atoms and the concentration of organic compounds of each sample in the formulas

established in equations 3 and 4, the values  $B_0$  and  $B_T$  obtained are shown in the Table 4.

Table 4: Results of maximum stoichiometric  $B_0$  and theoretical  $B_T$  yields of samples.

Samples	Empirical formula	$B_0$ (ml $CH_4$ g <sup>-1</sup> VS)	$B_T$ (ml $CH_4$ g <sup>-1</sup> VS)	BMP(ml $CH_4$ g <sup>-1</sup> VS)
ROP	$C_{66}H_{96}O_{45}N$	686	247	95
EOP	$C_{62}H_{94}O_{44}N$	672	199	54

Conforming to the results in the table 3, the theoretic methane potential ( $B_T$ ) is 2.5 and 4 times higher than BMP of ROP and EOP respectively. This effect can be attributed to the accessibility of carbohydrates to methanogenic microorganisms. Hence the need for mechanical pre-treatment and chemical pre-treatment, in order to improve this accessibility <sup>25)</sup>.

### 3.3 Result of the stoichiometric and theoretical methanogenic potential

According to results, we observed that the samples had significant differences in stoichiometric and theoretical methane yields. Those differences are mainly related to lipids content of ROP that enhance its biomethane potential.

The main reason of these results could be summarized in the specific methane percentages extracted from Angelidaki et al. <sup>26)</sup> to calculate the theoretical potential  $B_T$  are linked to lipids, proteins and carbohydrates determined by empirical formulas of the substrates used. It is crucial to mention that the sample's organic matter content can change as a function of time, climate or region and this factors can significantly affect the theoretical potential.

## 4. Conclusion

The olive pomace is a perfect substrate for micro-organisms, it is necessary to adjust some parameters such as the pH and the C/N ratio by providing a source of nitrogen in order to favor the methanogenic potential.

The comparison between the stoichiometric yield, and the theoretical yield, gives that, the BMP is 0.4 and 2.5 times less than theoretic methane potential of ROP and

EOP respectively. This effect can be attributed to the accessibility of carbohydrates to methanogenic microorganisms.

However, these different studies show that the pretreatment of olive pomace allows increasing the methane yield during anaerobic digestion. Hence the need for mechanical pre-treatment and chemical pre-treatment, in order to enhance the energetic outcome of anaerobic digestion of olive pomace.

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### Nomenclature

<i>ROP</i>	raw olive pomace
<i>EOP</i>	exhausted olive pomace
<i>BT</i>	the maximum theoretical yield
<i>A</i>	the specific methane yields of lipids
<i>B</i>	the specific methane yields of proteins
<i>C</i>	the specific methane yields of carbohydrates
<i>Cl</i>	concentrations of the lipids
<i>Cp</i>	concentrations (based on % TS) of the proteins
<i>Cc</i>	concentrations (based on % TS) of the carbohydrates
<i>B0</i>	the maximum methane stoichiometric
<i>Vm</i>	the molar volume of methane

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