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Anaerobic co-digestion of Olive mill wastewater using animal manure and biogas production

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ABSTRACT

Vegetation waters or wastewaters, resulting from the extraction of olive oil constitute the major problem of the olive culture, because of their polluting power (rich in organic and mineral material) and their acidic pH. Methanization is a biological process for the degradation of organic material, using a microbial flora in the absence of oxygen. This digestion or anaerobic fermentation is carried out in closed enclosures called digesters, within which, the various reactions are optimized and controlled in order to produce the biogas. The aim of this study is to valorize these wastewaters in the presence of poultry and cattle wastes, by anaerobic fermentation, in order to produce a biogas, especially to reduce their toxic effect. The present work is used to monitor on the quantitative and qualitative aspects the process of biogas production by co-digestion of organic wastes (olive wastewaters) and to determine different physicochemical parameters of the fermentation medium (pH, MS, MM, MO, CE, COT, NTK, C/N). The results obtained have clearly showed that the yield of biogas production is greater using wastes of poultry and cattle. The pH decreases during fermentation at 35ºC and increases during fermentation at 52ºC. The most important quantity of biogas produced was about of 4499/3981 mL, obtained during a fermentation during 40 days, in the presence poultry and cattle wastes of at 35ºC / 52 ° C.

Key words: Methanization, biogas, co-digestion, wastewaters, anaerobic fermentation.

1. INTRODUCTION

Olive industry is an important economic activity, concentrated mainly in the Mediterranean countries with 95% of world olive production, of which, 1% produced in Algeria in 2001 [1]. Like all food industries, the extraction process requires huge quantities of water; consequently this industry generates great quantities of liquid effluents (wastewaters). Olive wastewaters are liquid discharges, very rich in organic material (phenolic compounds, lipids), often widespread in nature, in uncontrolled ways on agricultural soils or sometimes temporarily stored in vats, exposing then the water-soil-plant system to inevitable pollution. Various purification treatments are currently applied: biological, physical and chemical. Costly and still insufficient, these treatments consist in reducing their harmful effect on the environment [2, 3].

Anaerobic digestion is now increasingly used to treat organic liquid effluents, in particular, for wastewater containing high loads of biodegradable organic material. Due to the high organic loading of olive wastewaters (50 g DBO L-1), anaerobic digestion is appropriate for this type of effluents [4].

Biomethanization allows the transformation of organic matter into various chemical substances, under anaerobic conditions, under the action of bacteria present in the environment. This process allows the production of biogas. Nevertheless, the action of bacteria is greatly slowed by the presence of polyphenols, which are toxic substances contained in this type of water [5].
The anaerobic digestion of organic material allows to obtain a methane-rich fuel gas which can then be used for heating, lighting and cooking. [15].
Our tudy consists in evaluating the production of biogas by co-digestion of organic waste (olive margins) on a laboratory scale. For this, the evolution of several parameters was followed: the pH, the VFA and the volume of biogas produced, for different proportions of substrate.

2. MATERIAL AND METHODS

2.1. DIGESTERS AND SUBSTRATES TESTED

The present work involves the assessment of anaerobic digestion of olive wastewaters, following a batch system fermentation process for 40 days, in a digester installed in the laboratory of the Center of Professional Agricultural Training for Cattle farming (CFPAEB) in Sidi Thabet, Tunisia.
The start-up of this process has been increasingly difficult to be managed; it firstly requires the culture of methanogenic bacteria. To overcome this problem, two inoculums have been used, the first is bovine dung, rich in methanogenic bacteria, the second is fresh poultry dung.
More precisely, the study of experimental digesters focuses mainly on the effect of the variation of some

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physicochemical parameters in the anaerobic digestion on the production of methane.

2.1.1. TECHNICAL MONITORING OF GAS PRODUCTION

2.1.1.1. QUANTITATIVE MONITORING OF EXPERIMENTAL BIOGAS PRODUCED

The device consists of three digesters with a capacity of 500 ml each:

The digester I: contains 350 ml of olive wastewaters.

-The digester II: contains 170 ml of olive wastewaters + 40 ml of poultry dung + 10 g of straw

-The digester III: contains 190 ml of olive wastewaters + 40 ml of cattle dung

The 03 digesters with repetition were operated with a batch system and placed at a temperature of 35 ° C. and 52 ° C. with stirring for a period of 40 days. (Table 2).

TABLE **1:** Properties of sample (olive wastewater) and inoculums (cattle and poultry dung).

	ST(MS) %	**pH**	**MM%**	**MO %**	**COT %**
Olive WasteWater	12	5,4	8,85	91,15	50,63
Cattle dung	13,5	6,85	10	90	50
Poultry dung	20	7	47	53	29,4

The volume is measured, according to the method of displaced liquid, 'Chaslerie, 2002, [6], (Fig. 1).

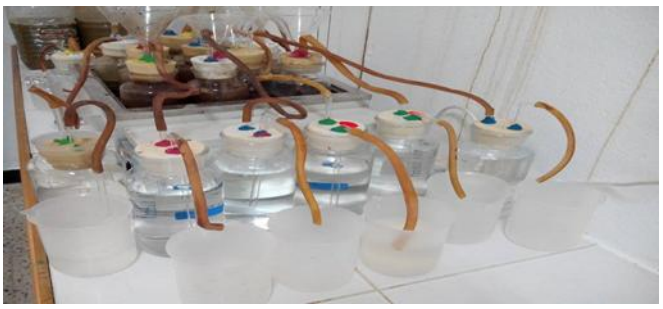

Fig. 1: Experimental set-up of the six digesters linked to the gasometers.

TABLE 2: Contents of the digesters.

	Olive wastewater			Inoculum (cattle dung)			Inoculum(poultry dung)			Volume Of water (ml)	Temperature °C		Totale volume (ml)
Digesters	Volume (ml)	pH	MS (%)	Volume (ml)	pH	MS (%)	Volume (ml)	PH	MS (%)				
Digester I	350	5.4	12	-	-	-	-	-	-	-	35	52	350
Digester II	170		-	-		-	400	-	20	145	35	52	355
Digester III	190	-	-	400	-	13,5	-		-	122,5	35	52	352,5

2.1.1.2. QUALITATIVE MONITORING OF EXPERIMENTAL BIOGAS PRODUCED:

The device consists of three digesters with a capacity of 05 L each, containing olive wastewaters in the absence and presence of the inoculum, connected with the inner tube of a car wheel. The digester volumes have been chosen according to the amount of the dry matter.

Table 3 presents the contents of the digesters considered. (Fig. 2).

In this study, we have valued biogas as gas for cooking and lighting.

TABLE 3: Contents of the digesters considered.

	Olive wastewater			Inoculum (cattle dung)			Inoculum(poultry dung)			Volume Of water (ml)	Temperature °C	Totale volume (ml)
Digesters	Volume (ml)	pH	MS	Volume (ml)	PH	MS	Volume (ml)	pH	MS			
Digester I	3500	5.4	12	-	-	-	-	-	-	-	35 °C	3500
Digester II	1700	-	-	-		-	400	-	20	1450	35°C	3550
Digester III	1900	-	-	400	-	13,5	-		-	1220,5	35°C	3525

Digester I : olive wastewater

Digester II : olive wastewater + poultry dung and straw

Digester III : olive wastewater + cattle dung

Fig. 2: Experimental set-up of the three digesters linked to the air tubes.

Once flammable, the biogas produced from each digester has been transported to chambers for analysis in the laboratory of Tunisian Society of Refining Industries (STIR), in Bizerte.

3. RESULTS AND DISCUSSIONS

3.1. PHYSICO-CHEMICAL ANALYSIS OF DIGESTERS BEFORE AND AFTER DIGESTION:

The table below summarizes the results of the different analyzes carried out, for the physicochemical parameters before and after the anaerobic fermentation of olive wastewaters, we noticed a change in pH, a decrease in

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organic matter, dry matter, total organic carbon, as well as mineral matter.

TABLE 4: Physico-chemical analysis of digesters before and after digestion.

Parametres	Digester I/I' : olive wstewater.			Digesteur II/II' : olive wstewater + poultry dun and et straw			Digesteur III/III' : wstewater + cattle dung.		
		DI	DI'		DII	DII'		DIII	DIII'
	Befor	After T35°C	After T52° C	Avant	After T35°C	After T52°C	Befor	After T35° C	After T52°C
pH	5,4	6,8	8	5,8	6,8	6,9	5,9	7	5,93
ST :MS(%)	12	8,50	9	7,28	5,87	6,8	6,95	5,8	5,99
MM(%)	8,85	47,31	46,7	18,82	30,68	30,57	17,33	33,33	30,5
MO(%)	91,15	52,69	53,30	81,18	69,32	69,43	98,67	66,67	69,50
CE ms/cm	18	15	9,8	17,9	9,6	5,86	18,6	17,5	16
COT %	50,63	29,27	29,61	45,10	38,51	38,57	54,81	37,03	38,61
NTK (en mg/g)	--	23,94	24,7	--	27,3	24,54	--	23,52	22,78
C/N	--	12,22	11,989	--	14,10	15,71	--	15,74	16,94
AGV (meq/L)	260	240	259	250	220	252	245	239	245

PH, ST (MS): total solid matter, dry matter, MM: mineral matter, MO: organic matter, EC: electrical conductivity, TOC: total organic carbon, NTK: Total nitrogen, AVG: Volatile fatty acids.

3.2. PARAMETERS TO MONITOR THE BIOGAS PRODUCTION:

The parameters considered are the pH, the AVG and the quantity and quality of the biogas produced in digesters.

3.2.1. Quantitative monitoring of gas production within the digesters

The volume of biogas produced has been measured using the displaced volume method [5,18]. Figures. 3, 4 and 5 show the evolution of the volume as a function of time for the two temperatures applied T = 35 ° C and T = 55 ° C.

The fermentation period in digesters I, II and III at a temperature of 35 ° was 40 days, the flammability of the gas produced has been noted from the seventh, eighth and ninth days with a production of 107, 127 and 130 Ml / day respectively; Whereas in the digesters I ', II' and III ', the fermentation of which was carried out at a temperature of 55 ° C, the flammability was obtained from the second day with volumes of 100, 105 and 185 ml / day respectively.

The kinetics of biogas production shows that the digesters III and III 'produce the largest quantities of biogas reaching a total volume of 4499 and 3981 ml successively, then the digesters II and II' with a production of 3961 and 1938 ml and finally the two digesters I and I' with volumes of 2688 and 1815 ml of biogas produced.

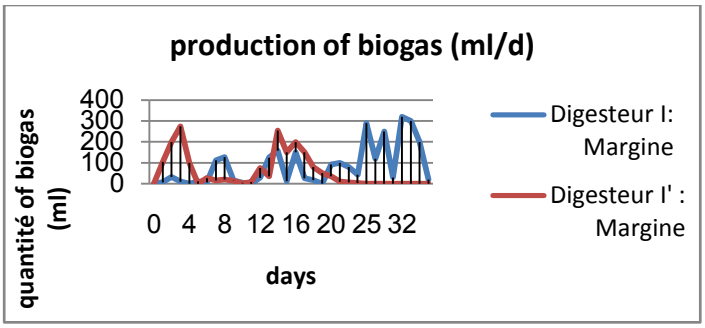

Fig. 3:Production of biogas (ml/d) in digesters I and I' at temperatures of 35 and 52°C.

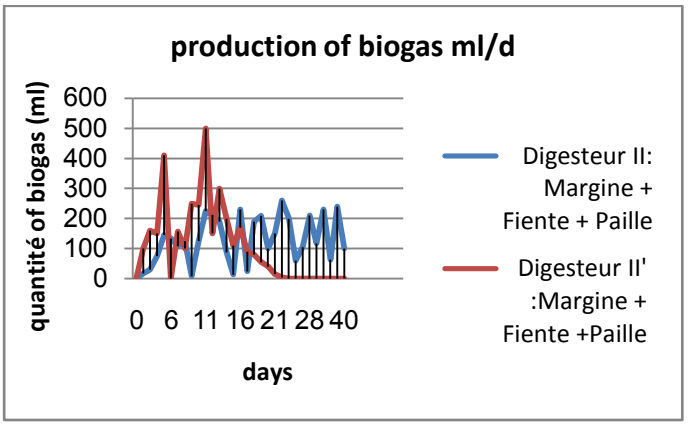

Fig. 4: Production of biogas (ml/d) in digesters II et II' at temperatures of 35 et 52°C.

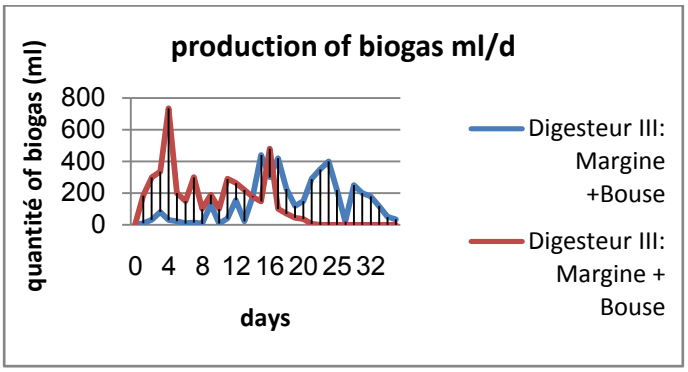

Fig. 5: Production of biogas (ml / d) of digesters III and III' at temperatures of 35 and 52 ° C.

The three figures clearly showed that the addition of fresh dung recovered from the rectum as an inoculum has improved biogas production. The choice of fresh dung from the rectum is mainly due to its freshness from a microbial point of view, since digestion at the level of the rumen is identical to that of anaerobic digestion. The microorganisms of the rumen are responsible for fermentations and ensure the production of volatile fatty acids (AGV) and gases (CO2 and CH4). It is necessary to mention that the bacteria in the rumen have passed through all the stages of anaerobic digestion to produce methane, that is why the fresh dung of cows still used as an inoculum.

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3.2.2. PH MONITORING

PH is a crucial parameter to stabilize and perform the anaerobic digestion. Anaerobic digestion processes are strongly influenced by pH. [17].

Through the figures (6, 7, 8), we observed that there is a decrease in the pH from 8.2 to 5 in the digesters. We have then adjusted the pH with sodium hydroxide (NaOH) 0.5N [16] over 15 days, in order to maintain its value in the neighborhood of 6.8 to 7. This value is adequate for development of methanogenic bacteria, [7].

PH is also of great importance, for the completion of methanogenic phase, since a decrease in pH results in inhibition of the methanization process.

The variation of pH depends on the evolution of the fermentation of the organic matter; pH decreases with the production of AGV during the acidogenic phase. The AGV produced during the acidogenesis; following the hydrolysis of organic matter. They have been degraded into H_2 and acetate. The latter has been transformed into methane during methanogenesis. The following table shows the evolution of AGV before and after methanization. [13].

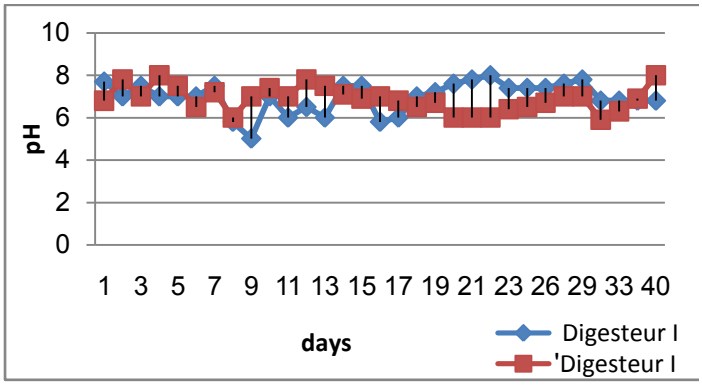

Fig. **6.** Curve of pH evolution in digesters I and I' at temperatures of 35 and 52 ° C.

It is noteworthy that the pH is slightly higher compared to that obtained in the mesophilic phase (pH = 8.5), this is probably due to a greater production of CaCO3 bicarbonates (increase in the buffering capacity of the system).

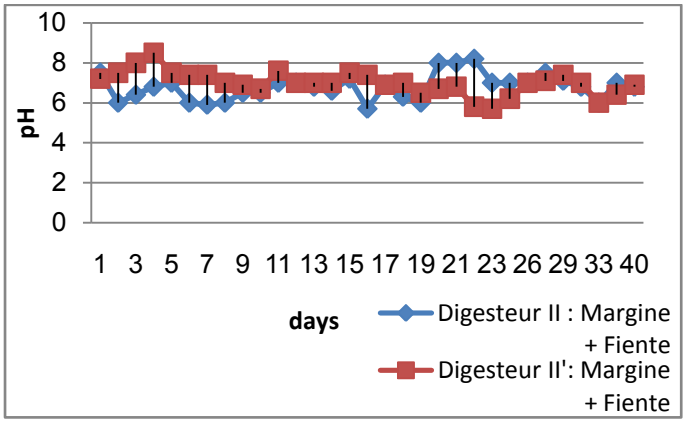

Fig. **7:** Curve of pH evolution in digesters II and II' at temperatures of 35 and 52 ° C.

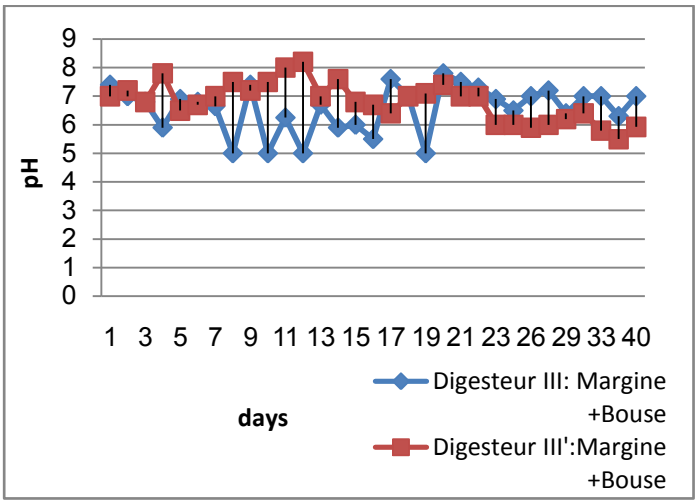

Fig. **8:** Curve of pH evolution in digesters III and III' at temperatures of 35 and 52 ° C.

3.2.3. STUDY OF THE EVOLUTION OF VOLATILE FATTY ACIDS "AGV"

The rate of "AGV"s is one of the most important parameters in the monitoring of anaerobic digestion and its performance since the accumulation of AGV is generally considered as an indicator of malfunction of the process [8] .

Indeed, the presence of AGV, including those with long chain, poisons the methanogenic cells and causes damage to the cell membrane [9].

AGV is the main intermediate in anaerobic digestion, and it accumulates under the action of the imbalance of the process. At low pH, the AGV becomes more toxic, this is due to the increase of its undissociated fraction.

The pH variation depends on the evolution of the fermentation of the organic matter; pH decreases with the production of AGV during the acidogenic phase.

The AGV produced during the acidogenic phase, due to the hydrolysis of organic matter have evolved over the time. They were degraded into H_2 and acetate. The latter has been transformed into methane during the methanogenese. [13]

The results of the AGV analyzes are presented in Figures 09, 10 and 11

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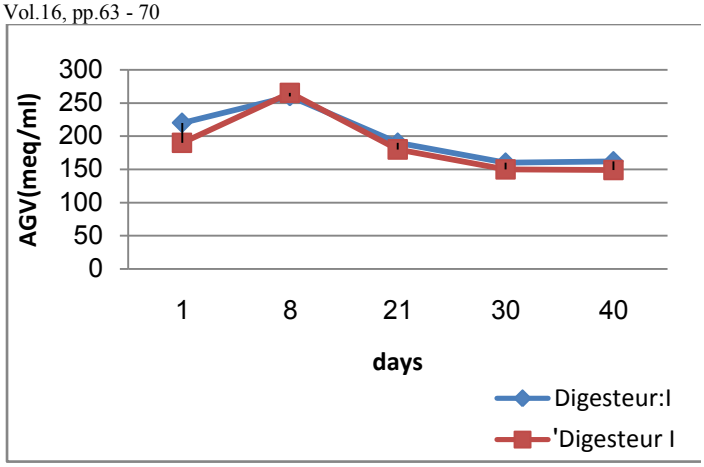

Fig. **9:** Evolution of the AGV concentration for both digesters I and I 'at temperatures of 35 and 52 ° C.

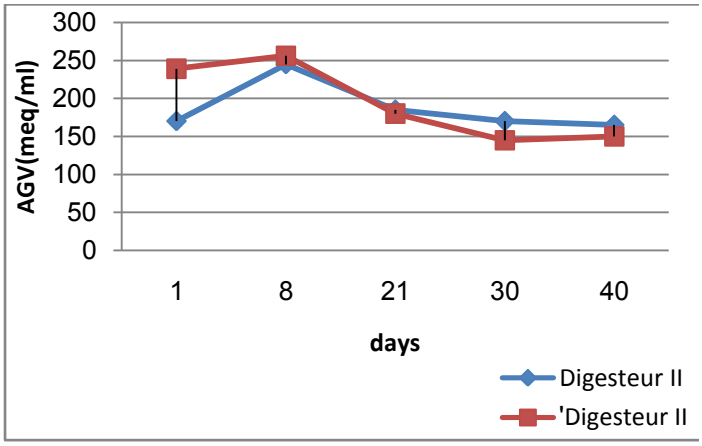

Fig. **10:** Evolution of the AGV concentration for both digesters II and II 'at temperatures of 35 and 52 ° C.

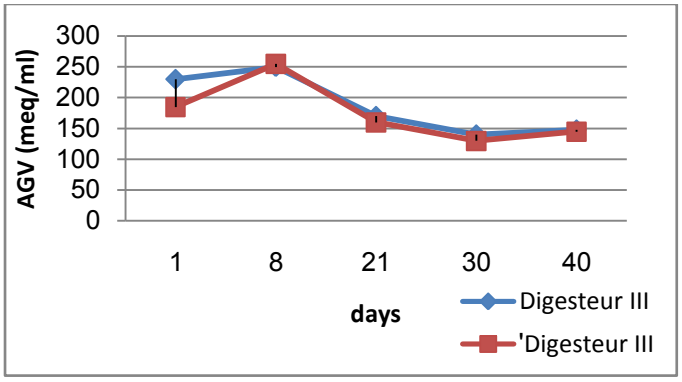

Fig. **11:** Evolution of the AGV concentration for both digesters III and III 'at temperatures of 35 and 52 ° C.

The concentrations of AGV are high, which can be related to the instability of the digester or the magnitude of the rate of hydrolysis, compared to that of methanogenes. Indeed, the hydrolytic and fermentative bacteria adapt more quickly than the abiogenes and methanogenes bacteria. This phase results in low biogas production. Subsequently, a decrease in the AGV concentration has been observed, as well as a slight stability. [10].

3.2.4. QUALITATIVE PERFORMANCE OF BIOGAS PRODUCED ON AN EXPERIMENTAL SCALE

The qualitative performances of the digesters have been followed after the analysis of biogas stored, using gas chromatography. The table 05 shows the contents of CH_4 H_2S, CO_2, O_2 and N_2 for each digester.
Biogas production is the most reliable criterion for anaerobic digestion [14]. The gas produced mainly contains methane ≥ (60%) [12], carbon dioxide ≥ 22%. And small amounts of hydrogen sulphide, oxygen, nitrogen, (Table 05).

TABLE **5:** Composition of biogas.

digester	CH_4 %	H_2S ppm	CO_2 %	O_2 %	N_2 %
Wastewater (35°C)	60,02	00	24,04	3,2	12,66
Wastewater +poultry dung (35 °C)	60,6	00	38,66	1,3	--
Wastewater + cattle dung (35 °C)	64,6	00	40,2	--	--
Wastewater (52°C)	62,58	2,8	34,85	--	--
Wastewater +poultry dung (52 °C)	63,36	10,2	25,99	--	--
Wastewater + cattle dung (52°C)	65,09	12	22,3	--	--

At the end of the experiment, it has been found that the methane content of the biogas produced is 60% Moletta (2008) [11], for the two temperatures applied. We have also noticed that the temperature does not influence the percentage of methane quantity in the gas, but it influences the activity of the microorganisms in each stage of fermentation development. The bacteria that work at the beginning are methanogenic agents, organized in groups where each performs a specific function. Hydrolytic bacteria begin to disintegrate complex organic matter into single small molecules. Presipermole bacteria and fermentative bacteria transform the molecules produced into intermediates (alcohols, organic acids, volatile fatty acids, acetic acid, H_2 and CO_2). Then another group of bacteria called acetogens starts to transform the alcohols and organic acids into acetic acid in addition to H2 and a small amount of CO2.

In this point, two actions of bacteria correspond to the formation of methane. Elastic acetic methanogenic microorganisms transform acetic acid into methane and CO_2 with 65% of CH_4, the other group of microorganisms called methanogen hydrogen-othropes uses H_2 and CO_2 to produce

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30% of CH_4; In this stage there is a production of H_2 in which, other types of bacteria are required; the sulfate reductants, which convert the sulfur contained in the primary material with H_2 into hydrogen sulphide; Finally we obtained methane, CO_2, hydrogen sulfuric and H_2O, this mixture is called biogas. After this presentation of the working steps of bacteria, it can be deduced that the temperature has an indirect influence on the volume of biogas produced. It is the factor that directs the activity of microorganisms and the reaction time.

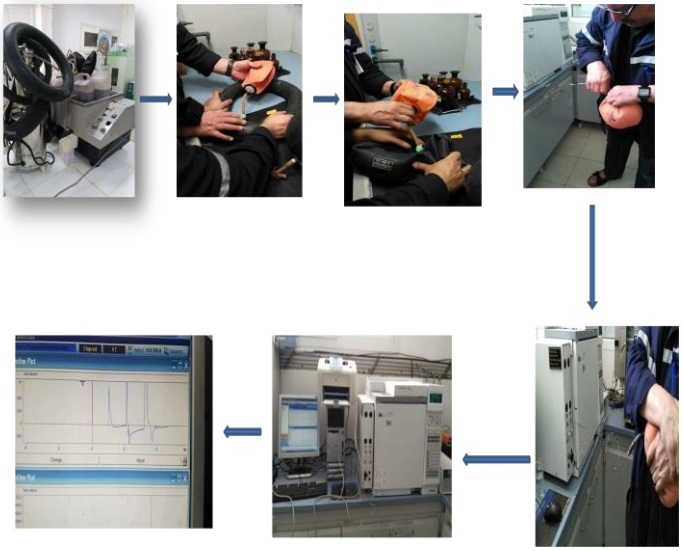

Fig. **12:** Steps to identify the composition of biogas.

3. USES OF GAS

Biogas can be used only for cooking food and lighting houses, (Fig. 13), especially in rural areas. [14]. As is the case of the rural digester of Hammam Sousse in Tunisia. This requires a sufficient and continuous biogas production.

Fig. **13:** Use of biogas as natural gas for cooking and lighting.

4. CONCLUSION

The study of biogas production by anaerobic digestion of olive margins has shown that the yield of biogas is very high ≥ (60%).

The results showed that the volume of biogas produced depends on the physicochemical parameters and the concentration of the initial organic matter. The period of production in the digester at a temperature of 35 ° was 40 days, the flammability of the gas produced was from the seventh, eighth and ninth day, with a production of 107, 127 and 130 ml / day respectively; While in the case of digesters I ', II' and III 'at a temperature of 55 ° C, the flammability was obtained from the second day with a volume of 100, 105 and 185 ml / day.

These results were obtaine, taking in consideration the bacterial activity which is much more intense at a temperature of 35 ° C. than 55 ° C. where the methanization process has been carried out at a low speed.

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