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# Energy valorization of sludge from the wastewater treatment plant of Boumerdes by biogas product

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## Abstract
This work represents only a beginning in the experimental study of the management of sludge from a biological method that aims to produce energy and reduce the risks associated with land application of sewage sludge and mineral fertilizers. Several pathways exist for the disposal of sludge, but the choice must depend on the cost of installation, the origin of sludge, the added value of the resulting product and the potential impact of the industry accepted on the environment. The landfill (also called storage) is a low-rewarding and legally prohibited in many countries. However, application of composts obtained should not be done without confirming their hygienisation, their stabilization and maturity. In addition, the sludge compost should be free of phytotoxicity, with concentrations of heavy metals (Cu, Zn, Cd, Hg, Cr ...) and organic micro levels below international standards. Our work was performed at the National Sanitation Office of Boumerdes, the objective is, to assess the ability of the sludge treatment, to determine what treatments they undergo, estimate the risk of pollution and finally know their reusability for energy purposes. For this we conducted a study on the physico-chemical and bacteriological sludge and an analysis reports for three years ago, then an experimental study for an anaerobic digestion to produce biogas, finally, the analysis by the chromatogram and the spectra in SCAM mode of the biogas composition products, shows that methane is present with the bigger rate. Our results showed that organic matter is not negligible in most cases it is greater than 40%, favoring the development of pathogens. In this case, sludge requires a stabilization step to reduce rate of organic matter. The statistical study reports the purification plant of Boumerdes between the years 2008 and 2010, based on the volume of sludge produced by the station and organic matter content showed that energy efficiency is important if the biogas is used. Especially in the period beginning the month of March until September when the sludge is concentrated in organic matter.

*Key words:* sludge, biogas, anaerobic digestion, green technology, fermentable organic matter, bio energy.

*Nomenclature*

-D: dilution factor or dilution considered;
-V: volume of inoculums;
- N: number of colonies;
- X: number of seeds per ml or g of product
-P0: weight of empty crucible;
-P1: weight (cup + wet sludge);
-P2: weight (crucible + dry mud);
-P3: weight (+ crucible ignited)

## 1. Introduction
Several pathways exist for the disposal of sludge, but the choice must depending on installation cost, the origin of sludge, the added value of resulting product and potential impact of the industry accepted on environment. The landfill (also called storage) is a low-rewarding and legally prohibited in many countries. However, application of composts obtained should not be done without confirming their hygéinisation, their stabilization and maturity [1]. In addition, the sludge compost should be free of phytotoxicity, with concentrations of heavy metals (Cu, Zn, Cd, Hg, Cr ...) and organic micro levels below international standards. Our work was performed at the National Sanitation Office (NSO) of Boumerdes, the objective is study of sludge produced during the process of water treatment at the wastewater treatment plant, to assess the ability of the sludge treatment, to determine, what treatments they undergo, estimate the risk of pollution and finally know their reusability for energy purposes. For this we conducted a study on the physico-chemical and bacteriological sludge and a study reports three years.

## 2. Materials and methods

Dewatered sludge samples were made to the wastewater treatment plant (Algeria) every day (about 42 samples) for physicochemical analysis, two samples for microbiological analysis. Thickened sludge sampling is done at the valve thickening and those dehydrated at the belt filter sludge samples were ground until the stock suspension (figure 1)

**Figure 1:** Sample collection

*2.1 Microbiological analysis:*

*a. Detection and enumeration of total and fecal coliforms :* From the dilutions be aseptically 1 ml in a Petri dish empty and sterile prepared for this purpose and number. We then completed with about 15 ml of agar Desoxycholate melted and then cooled to 45°C. Make a circular motion and then back and forth in the shape of "8" to allow the inoculum to mix with the agar. Let solidify on bench. The plates were incubated at 37°C for 24 to 48 hours for total coliforms and 44°C for 24 hours at 48°C for fecal coliforms, making a first reading after 24 hours.

*b.Detection and enumeration of total and fecal streptococci:* Streptococci belong to the family of lactobacteriaceae which are gram negative aerobic or facultative anaerobes.

Identification of streptococci is done by two steps:

❖ *Presumptive test on the Rothe broth (sodium N):* 50 ml of stock solution are inoculated in a vial containing 50 ml of medium Rothe double concentration. In a second step 5 Rothe double tubes of medium concentration were inoculated with 10 ml of stock solution and five tubes of medium Rothe simple concentration were inoculated with 1 ml of stock solution. Incubation is at 37°C for 48 hours.

The tubes with microbial disorder are considered positive, they contain streptococci and total streptococci may contain, they must be submitted to the confirmatory test.

❖ *Confirmatory test on the EVA medium (ethyl violet sodium azide):* This test is to identify and number the positive tubes on medium Rothe which will be a subculture (2-3 drops) on medium sodium acid ethyl violet. Incubation is at 37°C for 24 hours. The presence of fecal streptococci, results in a disturbance and microbial pellet violet or white, at the bottom of the tube. The number of faecal streptococci per 100 ml is calculated by the method of Most Probable Number.

❖ *Detection and enumeration of Staphylococcus aureus:* Transferring solution stock using sterile pipette (0.1 ml); an agar Surface Chapman plate. Carefully spread the inoculum to the agar surface, trying not touching the edges of the box with a sterile triangle. The box will be incubated at 37 °C for 48 hours. Staphylococcus aureus colonies appear on the middle yellow, bright, vaulted surrounded by a clear halo. To confirm the presence of Staphylococcus aureus some biochemical characteristics of the species are made. Results are expressed as number of seeds with "ml" or "g" of product.

*Detection and enumeration of sulphite-reducing Clostridium (CSR):* Insert 2x5 ml of the stock suspension in sterile empty tubes 2 and also 1 ml of the latter into another tube which will be completed later with 4 ml of sterile saline. These three tubes are heated in a water bath at 80°C for 10 minutes to eliminate the vegetative forms and leave only the spores. The tubes are cooled as soon tap water before pouring the agar aseptically Beef Liver melted and cooled to 45°C with added sodium sulfite (5 ml) and iron alum (2 ml) tubes are again cooled

to room air and incubated at 37°C for 72 hours. The colonies of sulfite-reducing Clostridium appear black. The results are expressed by the presence or absence of germ in formula (1):

$$X = N.\ 1/D\ .1/V \quad (1)$$

❖ *For Salmonella*: Introduce 25 ml of the sample to be analyzed in 100 ml of medium lactose broth Mannitol, buffer that will be incubated at 37°C, for 24 hours. Take 1 ml of medium pre-enrichment and seed it in 10 ml of Selenite Broth Sodium Acid. Incubate at 37°C for 24 hours. From the mid Acid Sodium Selenite positive, inoculate by streaking a Petri dish containing agar Hecktoen. Incubation is at 37°C for 24 hours. Salmonella are in the form of colonies of 2 to 4 mm in diameter and greenish-blue color with or without a black center. The results are expressed by the presence or absence of germ.

*2.2 Physical-chemical analysis:*

*a. Determination of the dried solids:* The sludge is made up of solids and water. The determination of the dried solids is through the removal of moisture at 105 ° C. The dryness is expressed in% by formula (2):

Mud =water +dry matter, 100%= moisture (%) +Solid content (%) (2)

- Weigh the empty crucible (P0), Place in a porcelain crucible weight (P0) a quantity of mud, then reweigh the crucible filled: it is the weight (P1), the difference (P1-P0) is the weight of wet mud, then place the crucible in the oven at 105 ° C until complete evaporation of water, usually for 24 hours, after cooling, place the crucible in a desiccator for 15 to 20 minutes to absorb the moisture produced by sublimation at transition temperature of 105°C to room temperature. Re-weigh: the weight (P2), the difference (P2-P0) determines the weight of dry matter (DM), the dryness percentage is the ratio of dry weight of the wet mud.

❖ *Dry matter concentration:* Is a mud g / l of DM (concentration). It is generally assumed that 1 liter of mud weighs approximately 1 kg then X g / l DM are contained in one liter ie d in 1000 g of sludge (Operating Instructions of National Sanitation Office). It can be calculate through following equations:

$$\text{Solid content } \% = [(P_2-P_0) \div (P_1-P_2)] \times 100 \quad (3)$$

$$DM = VMS + MM \quad (4)$$

$$X\ g/l \longrightarrow X \times 10^{-1}\ \% \text{ solid content.} \quad (5)$$

$$\% = X\ (g/l)\ /1000\ (g\ sludge) \quad (6)$$

❖ *The content of volatile matter in suspension VMS:* The dry matter consists of organic matter (MVS) and mineral matter (MM). Organic materials have the property of being mineralized at high temperatures:
Organic matter (C, H, O, N) CO2 + H2O + NH3 + H2S ... ... ..
The molecules produced by calcinations of organic matter at high temperatures are in gaseous form and will therefore evaporate, which is why we determine the organic matter by calcinations at 550 ° C. It expresses the concentration of VMS% compared to the DM.

❖ *Conducting an experiment of methane:* The experimental procedure involves following steps: - Fill the bottle of 5 liters, closing bottle; (figure 2)- Monitoring control parameters of digestion: temperature, PH, rate of organic matter.

*b. Chemical Processing and Analysis:*

❖ *Gas composition analysis:* 50 µL of the head space of the digester device was taken with tight gas syringe and was analyzed by using a HP 5890 gas chromatograph coupled with a HP 5970B mass-selective detector (Hewlett- Packard, Palo Alto, CA, USA). A polyethylene glycol-type capillary column (HP-inn wax, 0.25 mm i.d., 60 m length, and 0.25 µm film thicknesses) was chosen for the separation, and helium as the carrier gas. The injection port temperature was maintained at 250 °C. The column temperature was run according to the following program: T1 = 40 °C, 10 min, and then 5 °C min-1 up to T2 = 150 °C, finally 10 min isothermal. The MSD was operated in electron impact mode with the following conditions: ionization potential = 70 eV; source temperature = 230°C; transfer line temperature = 280 °C, m/z scan range = 20 ÷ 250, frequency = 3 scans per second. The signals were acquired and processed by dedicated software purchased from Hewlett-Packard. The quadruple detector was operated in electron impact; both SCAN and SIM (Selected Ion Monitoring) mode were used.

**Figure 2:** Experimental device to produce biogas.

The compounds identification was carried out through comparing the features of peaks eluted with those of authentic analyze standards. GC retention times, and mass spectra derived from reference (NIST) or home-made libraries were used for the purpose. Whenever necessary the mass spectrometric fragmentation patterns helped in peak identification. Analyses were performed in scan and then in SIM mode. The ion fragments corresponding to m/z = 16 was diagnostic for identification and quantification of methane.

## 3. Results and Discussion:

*3.1. Microbiological test results:* The results confirmed the presence of a large microbial diversity, and are only examples of many pathogenic micro-organisms existing in the sludge, which are the source of wastewater.
Despite the non-existence of a regulation that limits the number of micro-organisms in the sludge because of the lack of binding studies of epidemics in sewage sludge, the health risk is still possible even if the micro-organisms are not absorbed by plants, they can be transmitted by air or by binding to injuries of some plants such as vegetables, which cause serious health risks for individuals who are in the vicinity of the land covered by sludge spreading or consume raw foods of plant origin [2]. Therefore, we need ways to eliminate these pathogens that are sometimes sporulating and / or thermo tolerant, which makes the treatment of sludge by drying or by dehydration such inefficient.

*3.2.Physical and chemical analysis:* The production of sludge is important for two months in March and April: 1073 and 1535 $m^3$/month, which poses the problem of storage. The organic matter content ranges from 48.58% to 58.82% DM for March 2011 and from 43.18% to 65.46% for April 2011(figure3).

The rate of organic matter is an index of non-stability (OM %> 40%) and represents the rate of fermentability and the power energy. Theoretically, only half of the organic matter can be degraded by anaerobic digestion, we obtain a biodegradable organic matter 11 660.89 kg to 14 021.77 kg in March and April (figure 4).

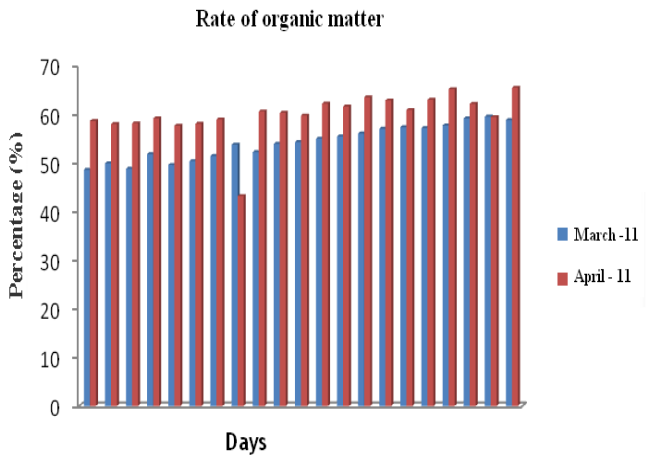



**Figure 3:** Rate of organic matter in the thickened sludge during March and April 2011

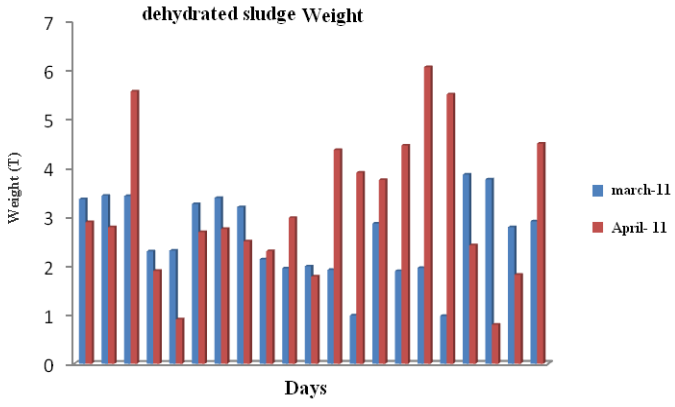



**Figure 4:** dewatered sludge Daily weight during March and April 2011

For biogas, 1kg of biodegradable organic matter gives between 900 and 1000 liters of biogas [3]. So we can produce minimum 10 494.8 $m^3$ of biogas for March and 12 619.59 $m^3$ for April (figure5).
Methane is the main constituent of biogas with 60% minimum, a value of 6 296.88 $m^3$ for March and 7 571.75 $m^3$ for April. Methane can replace different energy sources as shown in (Table 1).

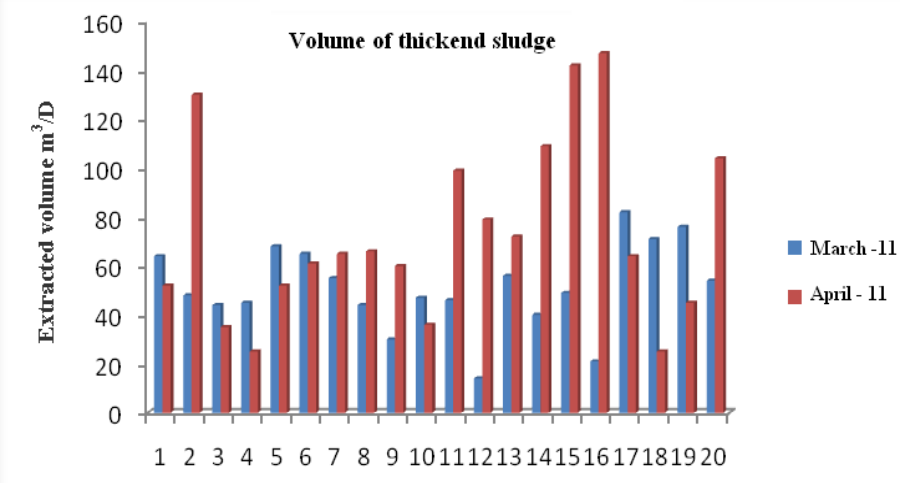



**Figure 5**: thickened sludge daily volume during March and April 2011

**Table1:** power equivalent of methane

| | $CH_4$ ($m^3$/mont) | Mazout (l/month) | Wood (kg/month) | Essence (l/month) | Charbon (kg/month) | Alchool (l/month) | Natural Gaz ($m^3$/month) | Electrical power (KW/h) |
|---|---|---|---|---|---|---|---|---|
| **March** | 6296,88 | 6296,88 | 13223,44 | 7241,41 | 8185,94 | 10704,69 | 5919,06 | 61079,73 |
| **April** | 7571,75 | 7571,75 | 15900,67 | 8707,51 | 9843,27 | 12871,97 | 7117,44 | 73445,97 |

*3.3. Analysis of reports (2008, 2009, 2010):*
*a. Heavy metals concentration of dewatered sludge:* The results show that heavy metal concentrations do not exceed the standard values, but the contribution of repeated sludge spreading could cause long-term accumulations incompatible with the quality of crops, in addition, the growing plants fast as vegetables (lettuce, spinach, carrots) specifically accumulate some metals, which justifies that the sludge can't be applied to this crop. The influence of food consumption plays an important role in the accumulation of some metals in the human body, causing serious health risks [4].

*b. Study of physico-chemical analysis reports of the sewage sludge from 2008 to 2010:*
The results of our study are summarized in (table 2): These tables include the results of physicochemical analysis of sludge from the WWTP for the last three years, and the statistical results of the approximate energy study have been done.

**Table 2:** anaerobic process result

| | Organic Matter (Kg/month) | Organic Matter Biodegradable (kg/month) | Biogas ($m^3$/month) | $CH_4$ ($m^3$/month) |
|---|---|---|---|---|
| March 2011 | 23321,79 | 11660,89 | 10494,8 | 6296,88 |
| April 2011 | 28043.55 | 14021.77 | 12619.59 | 7571.75 |

(Figure 6) presents the change in volume of thickened sludge produced. This amount varies each year and even each month in the same year. Volume of: 14 189 $m^3$ in 2008, 16 934 $m^3$ in 2009 and 16 222 $m^3$ in 2010.
The volume is low in the months of rainfall compared to other months where rainfall decreases. (Figure 7) represents the weight of the dewatered sludge; it's 765.108 Tons in 2008, 765.394 Tons in 2009 and 760.37 Tons in 2010. It is proportional to the volume extracted. (Figure 3) shows the percentage of total organic matter in the thickened sludge.

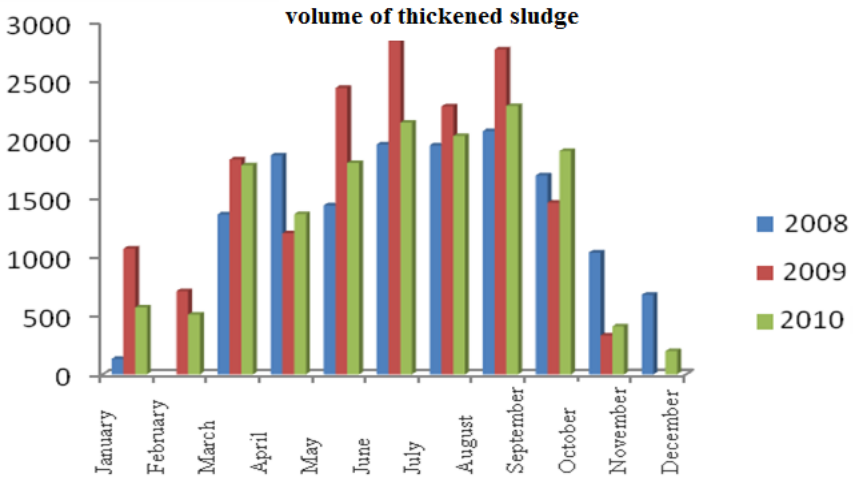



**Figure 6:** thickened sludge volume

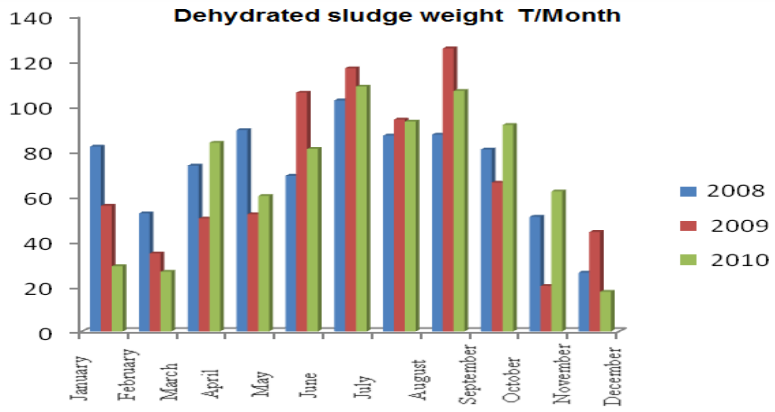



**Figure 7:** Dehydrated sludge weight

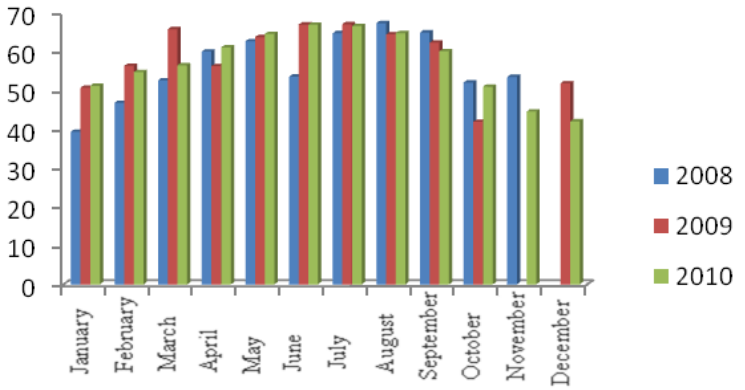



**Figure 8 :** thickened sludge‘s organic matter rate

The organic matter content (figure 8) is moderately high, 56.27% in 2008, 59.05% in 2009 and 57.14% in 2010. It varies from 39.45% to 67.50% for the year 2008, from 42% to 67.26% for 2009 and 42.13% to 67.12% for 2010.

The months that have an organic matter content <50%: January, February and December in 2008; October 2009; November and December in 2010 (figure 9). Wastewater in these months is less concentrated organic pollutants compared with other months as a result of rainfall. Indeed, the sludge produced is less loaded and is almost stable. As a result, the energy efficiency of anaerobic digestion is weak. Biodegradable organic matter is organic matter that can be reduced by biogas.

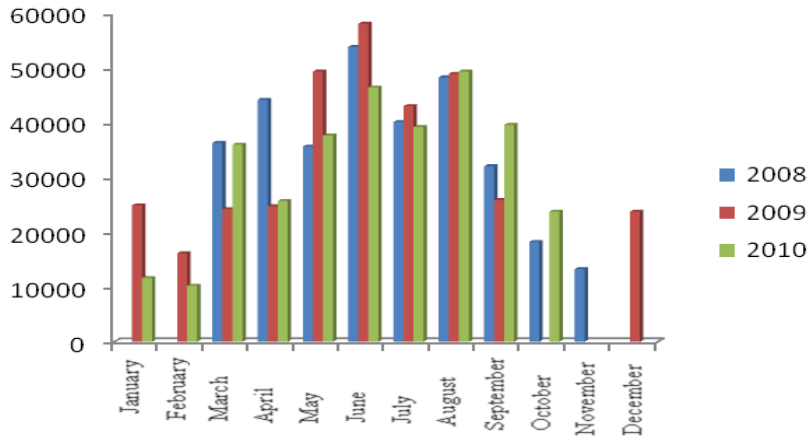



**Figure 9:** Variation of organic matter rate

Considering that half of the organic matter is degraded during the digestion. By ignoring the months when the concentration of organic matter is low, we obtain a total of 142 453.46 kg organic matter in 2008, 137 319.35 kg in 2009 and 137 180.59 kilograms in 2010 (Figure 10).

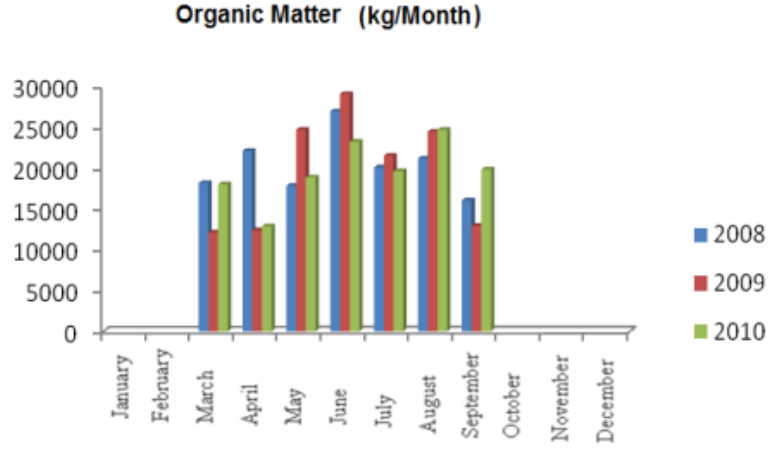



**Figure 10:** biodegradable organic matter rate

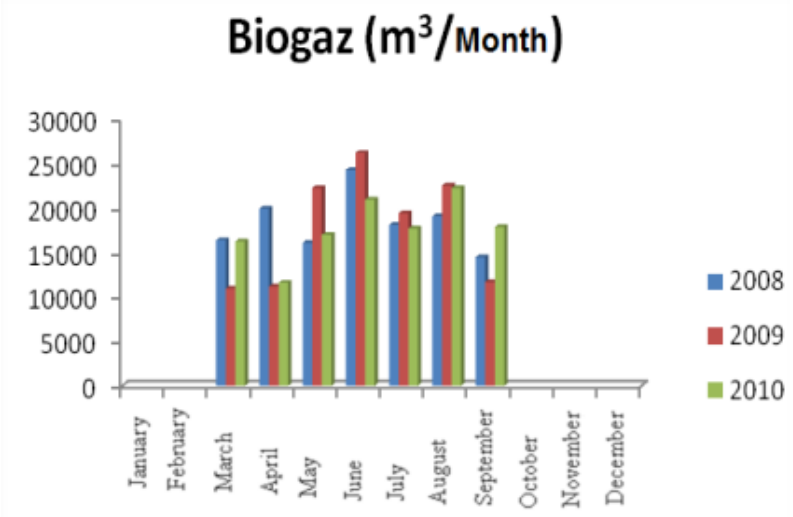



**Figure 11:** biogas quantity produced

Theoretically, according [3], 1 kg of OM makes about 900 liters (0.9 $m^3$) of biogas. Applying this definition, we have the amount of biogas in each month in each year: 128 208.11 $m^3$ in 2008, 124 037.98 $m^3$ in 2009 and 105 616.67 $m^3$ in 2010 (figure 11).

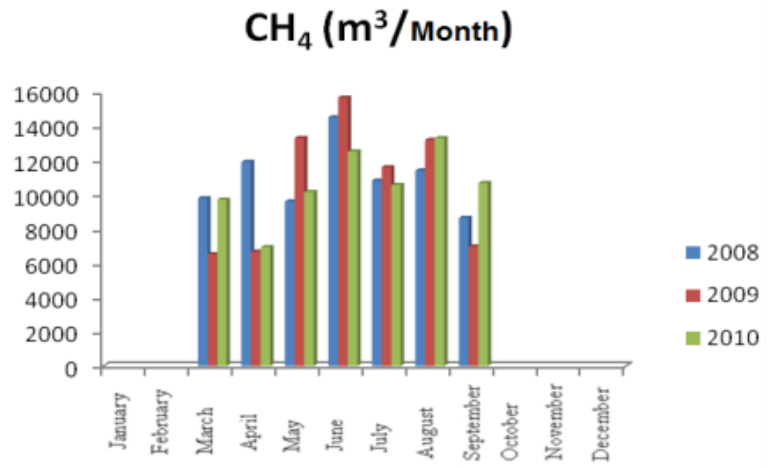



**Figure 12:** rate of methane**.**

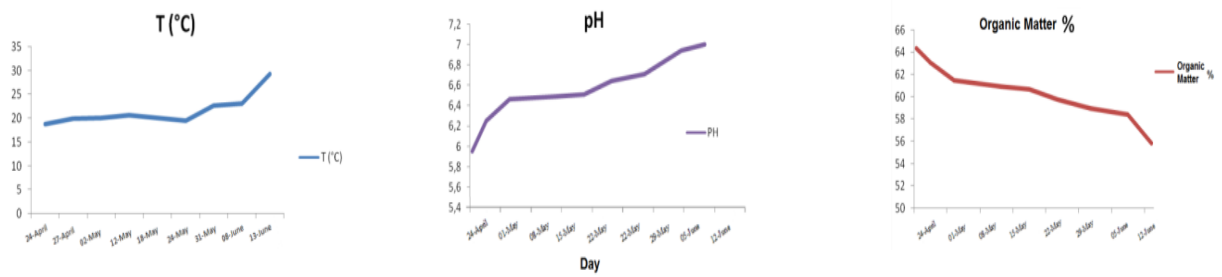
If we consider that our biogas contains minimum 60% methane, the amount of methane in each month will be as it is shown in (figure12). The total methane produced will be: 76 924.86 $m^3$ in 2008;74 422.78 $m^3$ in 2009 and 63 370 $m^3$ in 2010.

*3.4. Process of anaerobic digestion:*

Temperature is key parameter of biogas success. The experiment was carried out in ambient conditions, medium temperature; sludge temperature measurements are recorded in (figure 13(a)), pH changes are presented in (figure13(b)) ; Anaerobic bacteria including methanogenic bacteria are sensitive to changes in pH. The optimum pH of digestion process is 6.7 to 7.4; [5]. The accumulation of volatile fatty acids leads to rapid drop in pH values below 5.0. Then pH increases with consumption of acids in step of acetogenesis [6]. Based on our results, we note that pH was 5.94 at beginning and increases to stabilize around 7. This explains why methane has already begun in thickener. In general, there are three phases of pH evolution during anaerobic digestion: Acidification of substrate, this phase was spent in thickener is to say, before experiment start Substrate alkalinization. The evolution time of slowest pH is between 1 day and 37 days of experiment, pH values increased from 5.94 to 6.7. Stabilization of substrate pH [6]. From 47 day of experiment, there is pH value stabilization around 7.

*a. Evaluation of organic matter degradation:*

Note that organic matter degradation is very slow, during a residence time of fifty days; the rate of organic matter is degraded to 8.55%. Rate of organic matter degradation during the experiment duration is determined by temperature, high temperature favors the hydrolysis reactions, which will decrease the reaction time[7]. Temperature is the key parameter that accelerates fermentation reactions, we obtained 8.55% degradation in 50 days, under the same conditions and to degrade half of organic matter from beginning, we need about 188 days. By cons, if using a heat source, digestion can take a shorter time. In the end of digestion process we have obtained 21.92% of dry matter and 3.39% of organic matter (Figure 13).




**(a)** **(b)** **(c)**

**Figure 13:** variation of experimental parameters of anaerobic digestion process:
**(a): temperature; (b): pH; (c): organic matter degradation**

*b. Gas composition analysis:*

(figure 14A & 14B) show the chromatogram and the spectra in SCAM mode of the biogas composition products, (figure 15A & 15B) show the chromatogram and the spectra in SIM mode, the fragment ion m/z = 16 was chosen of the methane identification**.**

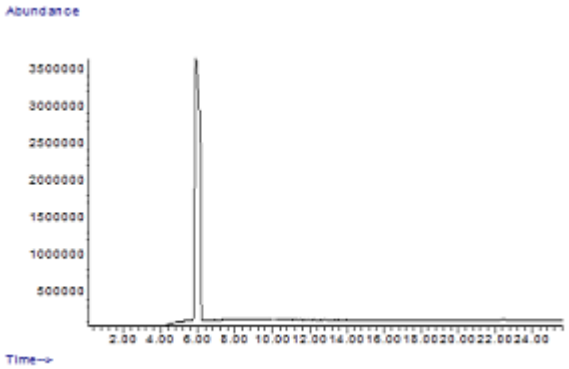



**Figure 14 A:** Chromatogram GC/MS in SCAN mode

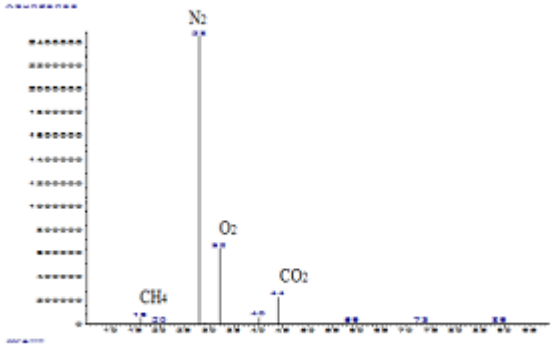



**Figure 14 B:** Spectra m/z ions fragments

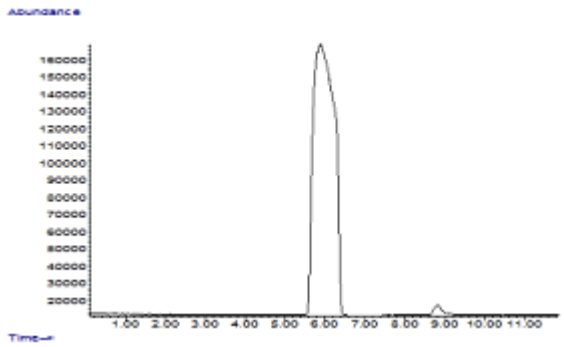



**Figure 15 A:** Chromatogram GC/MS in SIM mode

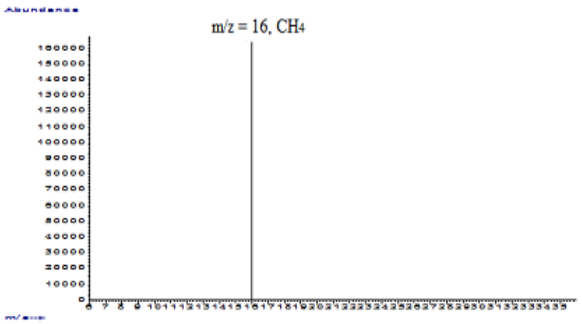



**Figure 15 B:** Spectra m/z of methane

*c. Timing Diagram Water Splash-Biogas*

Our proposal is to integrate a chain of biological stabilization in wastewater treatment plant's process as follows:

* Thickening prior to digestion: to send sufficiently concentrated sludge digestion to reduce digester volume.
* Digester: also allows sludge storage instead of storing them in thickener. At the WWTP, and because of lack of means to achieve dehydration every day, the sludge is stored in the thickener for a period exceeding two days, causing sludge degradation so a disruption of thickener operation.
* Biogas storage before upgrading to adapt its production for later use.
* Sludge dewatering after digestion, it can making an easier dehydration. Indeed, digestion reduces volatile content, and hydrophilic colloidal sludge. It saves one to three points of dryness from fresh sludge, when the volatile content decreases to 50%; reduce amount of sludge to be dewatered, electricity consumption and polymers; reduce odors in dehydration plant. Digested sludge (digestat) can also be post-composting, without producing odors and makes less use of synthetic fertilizers on agricultural land.

## Conclusion

The aim of this work is the production of biogas, especially the bio methane by conversion of organic matter present in the sludge by anaerobic fermentation. The organic matter present in the sludge was greater than 40%, favoring the development of pathogens. In this case, the sludge requires a stabilization step to reduce the rate of organic matter. Biogas as byproduct of anaerobic digestion was confirmed by gas chromatographic coupled with mass spectrometer, operated in both SIM and SCAN modes.

The physical and chemical analysis of the degradable organic matter shows that, this latter was lower than (3.39%), indicates that the high yield of the conversion process.
In addition, biogas, a byproduct of anaerobic digestion, can be recovered and valued. It is mainly composed of methane, a major greenhouse gas (GHG) and its enhancement with the objectives of the Kyoto Protocol, to reduce GHG emissions**.**

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