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Comparative study of the functioning and the efficiency of two experimental biogas measuring devices

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Abstract – Several wastes can be treated by Anaerobic Digestion (AD) to minimize their adverse effects. Different parameters can affect the wastes valorization such as their origin, as well as the used processes and devices. The purpose of this study is to compare two experimental devices for biogasmeasurement and to select the best for its biogas production and laboratory scale operation.
The substrate chosen for this study is glucose with the addition of poultry digestate as an inoculum. The use of erlenmeyer as gasometer filled with wateris often inconvenient for various reasons, namely gas solubility in water and water evaporation and also the difficulty of passing the produced gas especially with low flow rate up to the gasometer. Also, we followed the kinetics of the volume of biogas produced by each device as a function of time and the degradation of Organic Matter (OM).
The use of the device with the gasometer formed by reverse graduated burette filled by the barrier solution occur the best results in terms of maximum amount of biogas produced, minimization of the reduction of the pH during the AD and the biodegradation of the OM a recorded an important value which confirms the good performance of this device.
These results allow us to conclude that the choice of the second digester for starting experimental AD can minimise gas solibility and promotes the stabilization of optimal conditions of AD.

Keywords: Experimental device, Anaerobic Digestion, Glucose, Biogas

1. Introduction

The technology of biogas production by AD is very little knownand poorly applied in Tunisia. Indeed, biogas isso neglected among the main sources of energy in the country. The use of biogas technology can solve many ecological and economic problems.
Thanks to AD, waste becomes a source of wealth. Bioenergy is estimed to be the fourth largest energy resources in the world (Chen and Lee, 2014), and is nearly GHG-neutral replacement for fossil huels (Haberl et al. 2012) due to its renewable and widely applicable characteristics and its abundance.This technology is becoming essential in the process of reducing waste volumes and biogas production, which is a renewable energy source that can be used in the production of electricity and heat. AD is a biochemical process whereby complex OM is degraded under anaerobic conditions by consortia of bacteria (Al Seadi 2008). It is an eco-friendly process (Horvath et al. 2016) and one of the most efficientmethods for conversion of biomass to Methane.
To develop such an optimal control strategy, it is critical to monitor AD processes as closely and accurately as possible to enable estimation of process states and to detect unstable process states in time (Wolf et al. 2009). Whatever the valuation methodenvisaged, the operating conditions and the conduct of a methanogenic fermenter must be chosenin order to optimize the amount of energy recovered through the biogas. The operator will focus onoptimize the production of biogas by controlling its quantitiesand its quality.
AD can be determined as the volume of biogas produced, or the amount of substrate depletion or the formation of intermediates and end products by the different micro-organisms groups. Techniques for measuring the rate and volume of biogas produced from anaerobic biodegradability assays include;

lubricated syringes, volume displacement devices, pressure manometers or transducers. Most of the techniques have been manually operated. The improvement of the biogas production, in terms of flow, is primarily due to the good management of the process and also to the choice and efficiency of the bioreactor used. According to several previous works, there is a common method of measuring biogas by displacing liquid, like the displacement of a liquid or a tap water in the test tube into a pool of water (Shankar et al. 2013; Vindis et al. 2008) and others with the use of acid and salt solutions (Sponza 2003; Schonberg et al. 1997). Finally, we found also the measure by using of syrings (Elasri and Afilal, 2016). As part of a research project that wanted to focus on the opportunity of waste recovery by AD or biomethanation approach as an alternative way of biological treatment of fermentable organic waste.
The experimental approach at the laboratory scale has made it possible to evaluate the methanogenic potential of waste, as well as the possibilities of optimizing AD kinetics by studying several parameters. Among these parametersis the choice of an effective and reliable experimental device for the measurement of biogas produced which is the objective of this article.

2. Materials and Methods

2.1. Substrates

The sustrates used in this paper are the glucose ($C_6H_{12}O_6$) as a biodegradable organic substratewith the addition of the inoculum which is a poultry digestate.
This inoculum was taken from an old digester fed by fresh poultry droppings recovered from a recovery carpet and after wet AD at 35°C. Before mixing the substrates and starting the AD, the inoculum is pre-incubated in a water bath for two to three days at 35°C. A combustion test was carried out for the biogas produced by the inoculum; which consists of linking the digester with a benzene beak. The presence of a blue flame indicates the methane productionand subsequently it can be deduced that our ferment contains methanogenic bacteria, which facilitates the smooth running of AD and promotes the production of the biogas.

2.2. Preparation of digesters and caracterization of fermentation medium

In our study, AD was used in batch mode. At the end of the digestion, when the release of the biogas drops or becomes zero, the reactor is emptied and a new batch is introduced (Ostrem 2004). It is in a wet wayat 8% DM, when the solids content is less than 15%. The concentration of 8% is the optimal for the AD of glucose, acording to (Budiyono et al. 2010; Balsam 2002; Zennaki et al. 1996). It is also in mesophilic mode between 30 and 40°C, with an optimal operating temperature of 35°C. It is the most used mode, because of its stability and good biogas production (Chabalier 2006).
In our work, we studied the glucose degradation with experimental AD using two different devices. To prepare the batchs, we distributed inoculums in digestersand we add the quantitesof glucose. All batchs are incorporated at constant temperature of 35°C in a water bath and each test is performed in duplicate. In this research, we characterized the fermentation medium before, during, and after AD. Among the parameters tested are: pH, Dry Matter (DM) and the Organic Matter (OM).
We measured the initial pH, during the AD (every week) and the final pH. The determination of DM is carried out according to ISO 11465 AFNOR X 90-029 1994.The samples were taken and put in an oven at 105 ° C. for 24 hours. The respective DM levels are obtained by successive weighing of the samples, before and after drying in an oven. The organic matter (OM) is measured by reference to (NFU 44 160 1985). The previously dried samples are calcined in a muffle furnace at 550°C for 4 hours. The loss of mass, relative to the amount of DM, corresponds to the OM level.

2.3. Experimental devices for measuring daily biogas

The majority of laboratory volumetric gas meters are based on the liquid displacement method. Gasometers are the classical gas measuring unit which can be constructed with simple materials like glass/plastic jars or cylinders. We are interested in the comparison between two experimental devices for measuring biogas, one used in our laboratoryand the other inspiredby (El Asri et al. 2015).

- **Device 1**: Quantitative monitoring of biogas produced daily was used in an experimental digester, which consists of an erlenmeyer flask closed with a perforated silicone plug. This digester was connected to a gasometer (erlenmeyer) where it was built manually in the laboratory. The quantitative monitoring system consists in connecting the digester by the gas channel to from a pipe while being careful not to leak. The gas produced from degradation of

biodegradable material by microbial biomass will exert pressure on the water (used as displacement liquid) where the latter will be discharged from the water pipe to be recovered in a graduated beaker. So the volume of water that will be released into the beaker is equivalent in volume of biogas produced. The digester is linked to an entonoire to measure the pH of the floated fermentation medium.

- **Device 2**: The second type of reactor is an erlenmeyer connected using a silicone tube to a gasometer formed by reverse graduated burette filled with acidified saturated NaCl solution (citric acid 5 % and 20 % NaCl). The Summit of the gasometer is occupied by a valve and a syringe to adjust the level of measurement. When the biogas is produced, it pushes guard solution down the gasometer in the direction of an erlenmeyer of recovery. The batch is equipped with a syringe to take up the samples to measure the pH. Among the practices used for the experimental trial is manual stirring twice a day.

2.4. Correction and standardization of cumulative production of biogas

All the daily quantities produced in ml must be corrected and standardized by the correctionequations which take into account the temperature and the pressure as standard conditions 0° C and 101.325 kPa (McNaught and Wilkinson 1997). In each uncorrected volume there is a dead volume that varies depending on the temperature and ambient pressure of the laboratory, this variation influences directly the corrected product biogas volume. So, the standardized product volume of biogas (V_f) is deducted according to the following equation and it will be expressed in « Standard ml or Nml » and by the following equation (McNaught and Wilkinson 1997):

$$\mathbf{(1)} \qquad \mathbf{Vf} = \frac{\mathbf{273.15}}{\mathbf{Tamb \times Pamb}} (\mathbf{Vu \times 1013.25 - (hi - hf) \times d}) - \big(\mathbf{Vd \times 1013.25 - (hi \times d)}\big)$$

With Vu = Vd + (hf x Œ).
Vu: Uncorrected volume (ml),Vd: Volume died (ml), Œ:Calibration coefficient of the gasometer measured in ml/cm.
T_{amb}: Ambient laboratory Temperature at the time of the measurement in K (°C + 273.15).
P_{amb}: Ambient laboratory pressure at the time of measurement in hpa.
d: Density of the liquid.
h_f: Measurement of height.
h_i: Initial height (height of reference).

In our study, we used two types of liquids of displacement which are the water for the first device and the guard solution for the second. Among the correction parameters of the biogas produced in the equation is the density of the displacement medium that is measured using a densimeter. Water and Guard solution are introduced in a transparent specimen of a size appropriate to the densimeter. Just before the measurement, we move well the solution with a glass rod to eliminate layers of density and temperature (GK 800 2009). One proper densimeter above the scale is immersed in the liquid, once the densimeter well balanced and floating freely without touching the wall of reads the density in Baume (°B). The correspondence between the density and the Baume degrees is as follows:
For liquids heavier than water:

$$\mathbf{(2)\ d = 145 \div (145\text{-}dB°)}$$

For liquids lighter than water:

$$\mathbf{(3)\ d = 140 \div (dB° + 130)}$$

2.5. Biodegradability and mineralization

The characteristics of the biodegradation of anysubstrate by AD in a digester can be chosen from at least one of the following characteristics: the cumulative production of methane as a function of time by the methanizer; the composition of the biogas produced; the concentration of intermediates and inhibitors; and the percentage of Chemical Oxygen Demand (COD) of biodegraded substrate. For our study, biodegradability was estimated from the determination of the difference between initial and final OM and also from theoretical and experimental biogas production.

Still assuming total mineralization of OM, the potential for methane and carbon dioxide can be determined from the carbon, hydrogen and oxygen content of matter through the equation of Buswell (Buswell and Mueller, 1952). The elemental analysis of the substrates allows the determination of the overall stoichiometric formula of their OM and the estimation of their theoretical methanogenic potentials, from the Buswell equation.

Based on the production of biogas is 0.746 l/g of graded glucose or the theoretical potential of 746 mL (biogas)/g OM (Angelidaki 2002). Thus, the percentage of mineralization (% M) can be calculated by:

$$(4)\quad \%\ M = P_{Biogas} x\ 100/746$$

3. Results and discussions

3.1. Kinetics of biogas production

One of the best indicators of proper digester operation is the gas volume produced per day. According to the curves of the kinetics of production of Biogas by the two devices, we notice that the largestproduction of biogas is registered with the use of device 2 with a maximum equal to 210 Nml. For the first device, we found a maximum daily production of the order of 177 Nml.This result is due to the design of the digester with the graduated burette directly attached to the gasometer (connection with distance reduced between them by silicone tube) and also to the use of the barrier solution. This solution is saturated with the salt which reduced the maximum of dissolved CO_2. The presence of dissolved solids led to an almost total hydration of the solute, which leaves a less free solvent available for the absorption of gases (Umbreit et al. 1964). Thus the high ionic force prevents gases from dissolving in the moved liquid (Guwy and A.J, 2004).

Regarding the device 2 which is equipped with a gasometer filled with water as a displacement medium, this water allows the solitization of the fraction of CO_2 present in the biogas produced. CO_2 has a stronger solubility in water than methane.

Water is classified as a biogas purification method before its use and / or recovery (Ryckebosh et al. 2011). Other authors confirm the elimination of H_2S and CO_2 by physical absorption in water (Schomaker et al. 2000; Strevettet al. 1995). The biogas scrubbing techniques mentioned above also make it possible to remove CO_2 biogas; given the solubility of CO_2, which is much higher than that of methane, the biogas can be bubbled through a solvent bath to trap CO_2.

Collection of gas is usually done with the use of vessels containing a suitable liquid which is displaced as the gas gets collected. This technique is simple, economic and it can work for a long period of time without maintenance and it used by several researchers (Liu et al. 2004; Angelidaki et al. 1992; Beaubien et al. 1988).The preservation and collection of gases is the most important operation for any liquid displacement gasometer (Bunsen and Roscoe, 1857).In our study, the found results can be confirmed by those announced by (Walker et al. 2009) in which the comparison of different barrier solutions are shows that all of the other barrier solutions performed better than tap water, The resistances to the diffusion of CO_2 in 20 % saturated NaCl solutions was very weak comparing with other percentages: The loss of CO_2 in 40 % saturated NaCl was 49.4% and for other it was 27.3%. Similarly, the loss of CO_2 in 60 %, 80 %, and 95 % saturated solutions was 46.9%, 27.4% and 7.4%, respectively (Parajuli 2011). For this reason, it can be deduced that the barrier solution used showed a better performance in the preservation of gas products especially CO_2. But, it remains very weak to reach the performances recorded by 95% of saturation.

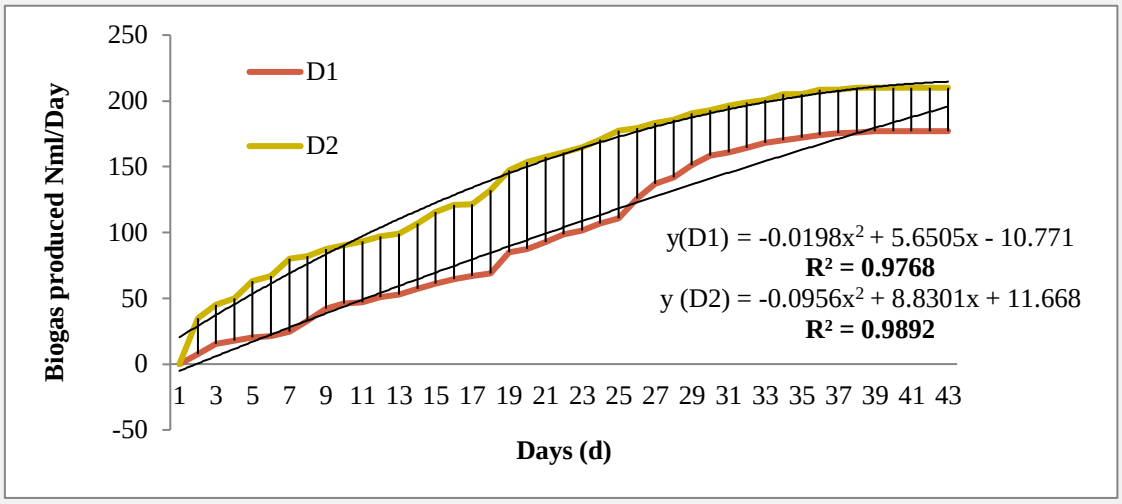

Figure 1. Kinetics of production of Biogas by the two devices

In most cases where the experimental conditions were optimal, the curves of cumulative production of biogas were exponential. In our test we did not use exponential trend curves that are generally useful when data values increase or decrease more rapidly. When we tried trend curves with this type, we found curves that do not describe well our measures. In addition, we can not create an exponential trend for data that contains null or negative values as in the first day. The trend curves used in our study are of the second order polynomial type which are generally used to represent data fluctuations used also by Cornell 1981, Misi and Forester 2001) to describe some effects of optimization related to 2-component co-digestion. Figure 1 shows two curves that illustrate the relationship between time and biogas production. The coefficient of determination (R^2) helps to determine how well the regression equation is adapted to describe the distribution of points. In our curves they are 0.9768 and 0.9892 successively for D1 and D2. They can indicate thegood correspondence between curves and data.Moreover we can deduce in our case that the rate of variability expressed by the set regression is very close to that of total variability. The biogas monitoring produced is well represented by the trend curves and also the regressions equations express well the quantities of biogas produced.

3.2. Biogas produced by the two devices

The biogas produced can also be expressed by the amount introduced into the substrate and subsequently the percentage of OM. It canbeexpressed by the formula below:

(5) Biogas yield = biogas volume/quantity of substrate

As illustrated in Figure 1, the experimental response of biogas production for both devices is almost without latency or " point of inflection "under optimal conditions of equilibrium of the test, through the use of glucose, a simple organic component that is easily metabolized. Under these conditions, on particulate matter, the kinetically limiting phase is recognized as hydrolysis (Mata-alvarez et al. 2000; Vavilin et al. 1996; Pavlostathis and Giraldo-Gomez, 1991).

For this type of daily quantitative monitoring, we can studythe degradations of OM by using three parameters: the half-life ($T_{1/2}$) which is the time taken by a substance (molecule, drug or other) to lose half of its physiological activity, k; a speed constant inversely proportional to a time and the potential of biogas and the %M. The decomposition and the biodegradation of the OM during the AD are not instantaneous but it is made as a function of the time, the half-life characterizes this decay by indicating the duration at the end of which the quantity of OM is diminished of half. It is also called "half-reaction time". The D1 records the weakest $T_{1/2}$ (10.78 d) and the largest k (0.064 d^{-1}), we can say that in D1, the quantity of glucose is transformed quickly in biogas than in D2 ($T_{1/2}$=14.55). According to Garcia-Heras (2003), the hydrolysis rates, expressed as a first-order coefficient k, for carbohydrates are between 0.5

and 2 d^{-1}. This helps us to conclude that the speeds found for both devices are slow. This can be explained by the quantitative and qualitative presence of the initial methanogenic biomass in digesters.

Table 1. Productions of biogas measured with the two devices and the time of half time ($T_{1/2}$)

	Biogas produced (Nml)	OM_d(g)	Biogas produced (Nml/g OM)	M (%)	$T_{1/2}$(d)	K (d^{-1})
D1	177	0.60	295	43.10	10.78	0.064
D2	210	0.60	350	56,30	14.55	0.047

3.3. Evaluation of pH during AD by both devices

pH is not always easily controllable since it is related to multiple other parameters of the process of AD. It is a very interesting indicator in the stabilization and the good progress of the AD. AD processes are strongly influenced by pH, it takes place from optimally in the neighborhood of neutrality with an optimum value between 6.5 and 7.5 (Gourdon 2012) or between 6 and 8 by others authors (Batstone et al. 2002). A pH difference in this range is usually a sign of a bad operation of the digester, and an accumulation of acids or alkalines compounds.

According to fig2, we found that the pH values of D2 throughout the monitoring period belong to the range indicated by the literature. After 35 days, the pH measurement showed that there is a reduction especially for the first device from 8 to 4.86 and also for the second device from 8.05 to 6.20. The activity of the methanogenic bacteria starts to become inhibited with pH equal or lower than 6.6 (Kuria 2008).

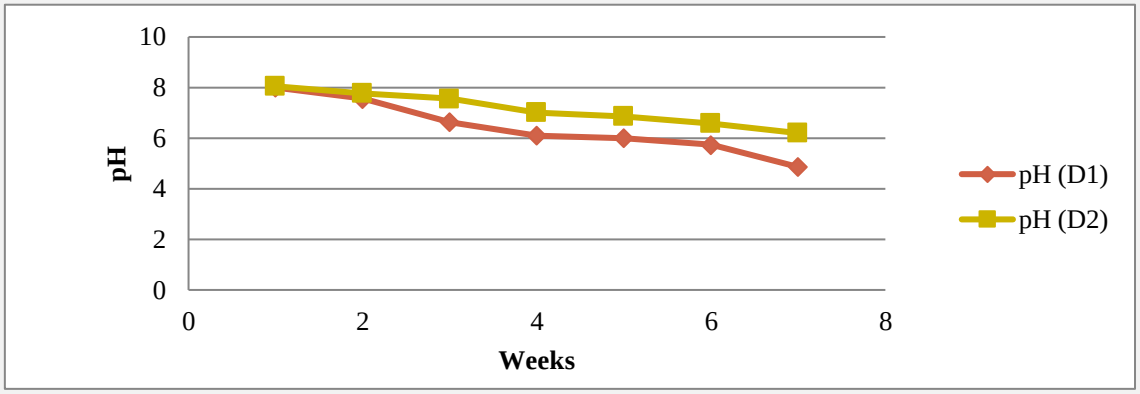

Figure 2. Difference between pH evolutions during the entire period of AD

3.4. Biodegradability of the substrate and effect of the retention time

The second device records the highest produced potentiel (350 Nml/g OM) and also the important percentage of mineralization which is 56.30%. When compared with the other device that produced 295Nml/g OM of biogas and with 43.10% mineralization, it is possible to say that the D1 is more reliable to producethe maximum biogas. The quantities of biogas found by the two devices are lower than the theoretical potential (746 ml/g OM). This result is confirmed by other author indicating that a portion of glucose (5-10%) is used to promote bacterial synthesis and the other portion is transformed into biogas.

In our case, the retention time is 42 days.This parameter is very important in the design of the anaerobic digester since often participates in the evaluation of the economic feasibility of technology, because it affects the efficiency of one type of anaerobic digester, with regard to the degradation OM and the specific production of biogas, depending on the composition of the substrateand the temperature of the system (Mata-alvarez 2002). Longer retention time requiresgenerally a larger volume of digester, at the same time it increases the potential for acclimation of the microflora and minimizes the effects due to toxicity (Yadvika et al. 2004). D2 has shown that it is easy to use even for long follow-up times. On the one hand, it even detects the small amounts of biogas produced, it is easier to fill the gasometer with the solution each time it becomes empty by the syringe attached to the top and still it is more convenient for installation and operation at the laboratory level.

4. Conclusion

This study demonstrated that the use of the device 2 increased the production of biogas (210 Nml). Several factors favored this increase; let us first mention the use of the guard solution rich on salt. This measurement technique reduces the absorption of biogas (CO_2) in the gasometer. For the pH evolution, this device has maintained generally optimal values for the production of biogas and the proper functioning of microorganisms. Regarding the biodegradability and mineralization of organic matter, it can be concluded that this device has the highest percentage of degradation that can be confirmed by the best amount of biogas recorded.

Finally, these results allow us to say that this type of device is recommended because of its features: it is easy to configure and use even for long durations, robust and inexpensive, its design promotes the detection of biogas produced without being lost through the long pipe linked by the gasometer. Also, it shows the optimal conditions of AD (pH, %M) and the better performance in the preservation of gas products.

Although much attention is given to the bio-chemistry and physical characteristics of AD and also the biogas production by different devices, this work must be complished by the study of the biogas production by the device with water and remplace it by the same barier solution used in the second device to more justify his use and have more knowledge about the comparaison and the efficacity.

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