Biogas production from waste activated sludge using cattle dung inoculums: Effect of total solid contents and kinetics study

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Abstract

Thermophilic anaerobic digestion of waste activated sludge (WAS) was experimentally studied in this research. WAS using cattle dung inoculums with total solids (TS) concentrations of 12.02, 17.58, 23.28, 26.75, and 35.2 g L⁻¹ were digested anaerobically in a batch digester at thermophilic temperatures (55°C) for a retention period of 15 days. Effect of TS concentration on the quality and quantity of the produced gas, pH variation, and the kinetics of biogas production were investigated. The results showed that biogas production potential and biogas production rate increased with an increasing TS concentration. The maximum biogas yields from TS concentration 12.02, 17.58, 23.28, 26.75, and 35.2 g L⁻¹ were 0.186, 0.189, 0.93, 0.213, and 0.231 L/(g VS)⁻¹, respectively. Modified Gompertz equation was employed to model the biogas production at different substrate concentrations. The equation gave a good approximation of the maximum biogas production (Rₘ) and the biogas yield potential (P) with correlation coefficient (R²) over 0.994. The digestion at TS concentration 35.2 g L⁻¹ gave the best results. The maximum biogas production reaches 0.856 L day⁻¹, and the biogas yield was 6.650 L at the end of the 13th day of the experiment. This amount of biogas with composition 72.59% of CH₄ and 23.6% of CO₂ is equivalent to 190 KWh of electricity. These results show that WAS mixed with cow dung is an effective feedstock for biogas production, giving a high cumulative biogas yield.

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1. Introduction

Activated sludge process (ASP) is used widely to treat both industrial and municipal wastewaters due to its great advantages such as its easy operation, high treatment efficiency and low operating cost. The production of large amounts of excess sludge, which is also called as “waste activated sludge (WAS)”, is the major drawback of the ASP. Since improper disposal of the WAS poses a significant threat for ecological systems because of its pathogen content and highly putrescible nature, the WAS should be treated prior to final disposal [1, 2]. As a result of the quantitative and qualitative expansion of wastewater treatment plants over time, the production of WAS has also increased. The expense for excess sludge treatment has been estimated to be up to 50% of the total operating cost of a wastewater treatment plant [3]. Although Waste activated sludge is rich in nutrients, it is not yet generally accepted for use as a fertilizer for agricultural purposes. The resistances from the farming industry concerns mostly fear of heavy metals and other presumably toxic compounds. Incineration is quite expensive and needs the treatment of flue gas in order to remove toxic compounds; it is thus highly debated. The main disposal route is land application (or agricultural use), but it is subject to reservations from farmers and consumers. Therefore, so, it is necessary to find more efficient treatment in order to reduce sludge production in the wastewater treatment plant [4]. Therefore, much attention in terms of both environmental and economical aspects has been focused on the sludge treatment processes for both reducing the amount of sludge produced and improving the stabilization degree of sludge. Biological methods such as anaerobic digestion is widely used for sludge stabilization, which not only reduces the quantity of sludge to be disposed off, but also produces valuable methane gas, improved dewatering properties of the digested sludge, high quality biosolids for land application, and as a carbon source for denitrification [5,6]. The anaerobic digestion process generally consists of four stage, hydrolysis, acidogenesis, acetogenesis and methanogenesis. In anaerobic digestion, the biological hydrolysis is identified as the rate-limiting step [7]. To reduce the impact of rate-limiting step, pretreatment of WAS is required such as thermal, alkaline, ultrasonic and mechanical disintegration [3,8-10]. These treatment can accelerate the solubilization of WAS and reduce the particle size, which subsequently improve the anaerobic digestion [11, 12]. Another option to increase biogas production from a WAS digester is inoculation with residues which present better digestibility, enhanced biogas production/methane yield arising from availability of additional nutrients [13,14]. The objective of this study was to determine the effect of total solid content of substrate on the biogas production from waste activated sludge inoculated with cattle dung at thermophilic conditions.

2. Material and methods

2.1. Material

The biological samples on which we work are the WAS and Cattle dung. The simpling of WAS was done in the urban wastewater treatment plants in Boumerdes, Algeria whose it’s retention time in the extended aeration process (sludge age) was 10 days. Then Cattle dung was obtained from bovine’s cattle farm, they characteristics are shown in table 1. The material has been homogenized in an electric blender. The samples have been stored at 4°C in a refrigerator until usage.

2.2. Anaerobic digestion

Microorganisms for anaerobic digestion consisted in start of those present in aerobic activated sludge inoculated with rumen microorganisms of cattle dung. The reactor for anaerobic digestion had a volume of 5 L and its working volume of 4.5 L. It was also equipped with gas and sludge sampling ports. When subtract was added, reactor was purged with helium gas to eliminate air from the reactor. The mixed sludge was stirred in the digester without oxygen contact. The reactor was incubated at 55 °C and the biogas volume generated was measured by liquid displacement (water, pH 2, NaCl 10%).

2.3. Analysis

Total solid (TS), volatile suspending solid (VS) and Chemical Oxygen Demand (COD) were determined according to Standard Methods APHA, 1995 [15]. Biogas samples were collected using a gas sampling injector and a sample of 100–200 μL was used for each run. The biogas composition (CH₄ + CO₂) was determined using a gas chromatograph(GC-HP 5890) equipped with a thermal conductivity detector (TCD) and stainless steel column that
was 2m long with a 5mm OD and 2 mm ID and contained Porapak Q 100 that had a mesh range from 80–100. The carrier gas was N\(_2\), and the analysis was carried out at a carrier gas flow rate of 30 mL min\(^{-1}\) with the injector, column, and detector temperatures at 120, 90, and 120 °C, respectively. The pH of the anaerobic slurry (sludge) was measured using a digital pH meter, which had an accuracy of ±0.1 pH unit. Phosphate was analyzed by the molybdenum blue method [16]. Molybdenum acid ammonium solution, 2.0 ml, and an L-ascorbic acid solution, 1.0 ml, were added to the sample solution. After 15 min, the absorbance at a wavelength of 700 nm with UV–visible recording spectrophotometer (UVmini-1240 SHIMADZU) using 10 mm matched quartz cells.

The kinetic data obtained from all assays were checked for the fitness of modified Gompertz equation [17, 18]. The modified Gompertz equation, that gives cumulative biogas production from batch digesters assuming that biogas production, is a function of bacterial growth. The modified Gompertz equation is given by (Eq 1).

\[
M = P \cdot \exp \left\{ - \exp \left[ \frac{R_m \cdot e}{P} (\lambda - t) + 1 \right] \right\} 
\]

(1)

Where M is the cumulative biogas production (L), P the biogas production potential (L), \(R_m\) the maximum biogas production rate (L d\(^{-1}\)), \(\lambda\) the duration of lag phase (day) and t is the duration of the assay at which cumulative biogas production M is calculated (day). The parameters P, \(R_m\) and \(\lambda\) were estimated for each of the digesters using POLYMATH software. The equations were integrated using the ODE45 solver, which is a built-in function in MATLAB.

3. Results and discussion

The profile of pH over the length of the digestion period at different TS concentration under thermophilic temperatures is shown in Fig. 1. The results indicated that the pH values seemed to vary with operation time in a similar way in the all samples; as seen, the pH started from the same initial pH (7.0 –7.1), and in the all samples it was dropped to 6.8 – 6.3. Dropped at first partly due to the heterogeneity of straw particles, subsequent hydrolysis process occurred in the reactors and the volatile fatty acids (VFA) accumulation, especially during the first three days. However, all the pH increased after 3 days operations, and reached around 6.5, and then gradually increased; finally, it reached a level about 7.8. The pH varied between 6.8 and 7.8 which nearly lied in the favorable pH range of 6.6–7.8 for methanogenic bacteria [19].

| Parameters             | Activated sludge waste | Cattle dung |
|------------------------|------------------------|-------------|
| TS (g/L)               | 24.1                   | 53.7        |
| VS(g/L)                | 10.71                  | 28.6        |
| T COD (mg/L)           | 780                    | 18720       |
| N-NH\(_3\) (mg/L)      | 18.8                   | 720         |
| N-NO\(_3\)- (mg/L)     | 3.6                    | 78.2        |
| P-PO\(_4\)- (mg/l)     | 42.3                   | 6.9 - 7.2   |
| pH                     | 7.2                    | 6.8         |
| Moisture content (%)   | 98.3                   | 46.3        |
| Total coliforms (MPN/100 mL) | 5.57 10\(^4\) | -           |
| Fecal coliforms (MPN/100 mL) | 8.83 10\(^4\)    | -           |
To study the effect of TS concentration on the performance of the anaerobic digestion process WAS with initial concentration of TS: 12.02, 17.58, 23.28, 26.75 and 35.8 g L$^{-1}$ at thermophilic temperature was digested. The TS content was presented in term of dry matter and the cumulative biogas maintained at room and ambient temperature along. The research was carried out in triplication. The data obtained from the study then is averaged and the cumulative volume of biogas production was observed during 13 days as showed in Fig. 2. The digestion was characterized without fluctuation of biogas production at the beginning. Degradation of substrate started almost immediately and proceeded without problems in all digestions and biogas production is significantly increased due to exponential growth of microorganisms and to their higher adaptation to the change of the concentration of substrate, except that for digestion with initial TS concentration of 12.02 g L$^{-1}$, it took about 2 -3 days for initiation of biogas production. The reason for this observation may be due to the lag phase of microbial growth. After 12 - 13 days observation, biogas production for all samples tend to decrease and this is predicted tends due to stationary phase of microbial growth [20].

The biogas yield, biogas produced per g organic solids (volatile solids) for different concentrations of substrata over a 13 day digestion time at thermophilic temperature (55°C) is shown in Fig. 3. The rates of biogas production differed appreciably according to the TS concentration. Furthermore, as shown in Fig. 3, the best performance for biogas production was the digester with 26.75 and 35.8 g L$^{-1}$ of TS, give biogas yield 0.231 and 0.213 L (gVS)$^{-1}$, respectively after 13 days observation. While, the other TS concentration of 12.02, 17.58, and 23.78 g L$^{-1}$ give the biogas yield 0.185, 0.189, and 0.186 L (gVS)$^{-1}$, respectively. This is similar with the information from Amani et al. [21] that the optimum solid concentration obtained for biogas production is in the range 30 - 32 g L$^{-1}$. The lower biogas yield indicated that there was an inhibition of methanogenic bacteria. It can be observed from Fig. 3, that bulk of substrate degradation takes place up to a period of 12 - 13 days suggesting that the digesters should preferably be run at a digestion time close to 12 - 13 days for optimum energy yield.

Milenko et al.[22] have reported data for batch thermophilic anaerobic digestion of waste activated sludge. They have obtained 0.565 L biogas (gVS)$^{-1}$ at retention time 10 days and concentration of solid content 15 %. This results presented on Fig.3 are lower compared to those reported by Milenko et al. but this result confirm that: the rates of biogas production varied noticeably according to the TS concentration, as the concentration 15% of Ts is higher compared to ours.

![Fig. 1. pH variations during SAW anaerobic digestion at different total solid concentration under the thermophilic conditions (55°C)](image-url)
The methane content of the biogas generated from the fermentation of studied substrate is shown in Fig. 4. It was in the range of 52–56% during the first 4–5 days of the digestion process and was observed to be in the range 62–72% after 13 days. The average methane content of the biogas generated from the fermentation of the WAS with initial TS concentration: 35.8, 26.75, 23.28, 17.58, and 12.02 g L$^{-1}$ was 86.7, 79.3, 78.2, 76.5, and 81.3 % respectively. The Fermentation of WAS at total solid concentration 35.8 g L$^{-1}$ had greater proportion of methane in the gas, and that increased as the concentration of total solid content increased.
Kinetic parameters of anaerobic digestion process are always used to analyze the performance of digesters and design appropriate digesters, which are also helpful in understanding inhibitory mechanisms of biodegradation[23]. With an assumption that biogas produced is a function of bacterial growth in batch digesters, modified Gompertz equation relates cumulative biogas production and the time of digestion through biogas yield potential (P), the maximum biogas production rate (Rm) and the duration of lag phase (\( \lambda \)). To analytically quantify parameters of batch growth curve, a modified Gompertz equation was fitted to the cumulative biogas production data. Values of parameters obtained are summarized in Table 2. It has been observed that the cumulative biogas production was fit well with the modified Gompertz equation as is evident from the correlation coefficient \( R^2 \) (0.996 - 0.998) between the experimental and predicted values along with the parameter estimates in Table 2. Lag phase (\( \lambda \)) was found 1.856, 1.915, 1.902, 2.565, and 4.622 day for TS concentration 35.8, 26.75, 23.28, 17.58, and 12.02 g L\(^{-1}\) respectively. Shortest lag phase (\( \lambda \)) was exhibited by TS concentration 35.8, 26.75 g and 23.28 L\(^{-1}\), 1.856 days, 1.915 and 1.902 respectively, which indicated a good acclimation of the organisms in the reactor. While the largest lag phase (\( \lambda \)) was exhibited by TS concentration 12.02 g L\(^{-1}\), 4.168 days. This lag phase might be due to low methanogenic activity and/or the number of methanogens, in the digesters, that could result in the accumulation of the volatile fatty acids (VFA) produced during the acidogenic step. High concentrations of volatile fatty acids could cause inhibition to methanogenesis [24]. However when TS concentration is higher the quantity and species of anaerobic bacteria enable to degrade more kind of substrate content in WAS and the yield is faster. The biogas production rate (Rm) for TS concentration 17.58 is the lowest of 0.353 L d\(^{-1}\) and the highest is shown by TS concentration 38.5 g L\(^{-1}\) with a value of 0.856 L d\(^{-1}\).

Therefore the amount of gas produced at the end of digestion period was highest for TS concentration 35.8 g L\(^{-1}\) (6.65 L). This could be because WAS is rich in nutrients and contains adequate amount of carbon, oxygen, hydrogen, nitrogen, phosphorous, potassium, calcium, magnesium and a number of trace elements which are very essential for the growth of anaerobic bacterium [25]. This could have optimized syntrophic interaction between acetogens and methanogens which is the most critical step in the biomethanation process [26]. However digesters for TS concentration 26.75, 23.28, 17.58, and 12.02 g L\(^{-1}\) produced 4.651, 3.779, 3.019, and 1.826 l of biogas respectively.
Table 2. Values of fitting functions and statistical measures for the kinetic model at different total solid content

| Total solid content (g L\(^{-1}\)) | Correlation coefficient (R\(^2\)) | Maximum biogas production (Rm), L day\(^{-1}\) | Biogas yield potential (P), L | Duration of lag phase (\(\lambda\)), day |
|-----------------------------------|----------------------------------|------------------------------------------|-------------------------|---------------------|
| 12.02                             | 0.997                            | 0.463                                   | 1.826                   | 4.622               |
| 17.56                             | 0.998                            | 0.353                                   | 3.019                   | 2.565               |
| 23.28                             | 0.997                            | 0.514                                   | 3.779                   | 1.902               |
| 26.75                             | 0.997                            | 0.596                                   | 4.651                   | 1.915               |
| 35.8                              | 0.996                            | 0.856                                   | 6.650                   | 1.856               |

4. Conclusion

Analyzing the experimental dataset it was found that, the production of biogas from WAS largely depends on the initial total solid concentration. If the amount of TS content was changed, the production of the gas was also changed. The maximum gas production was 0.231 L (g VS\(^{-1}\)) for TS concentration studied 35.8 g L\(^{-1}\) which gave the kinetic parameters of biogas production i.e. biogas production rate constants (P), maximum biogas production (Rm), and minimum time to produce biogas (\(\lambda\)) are 6.650 L; 0.856 L day\(^{-1}\); and 1.865 days, respectively. The graphs have verified that Modified Gompertz equation best describes cumulative gas produced as a function of retention time.

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