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ENHANCEMENT OF BIOGAS PRODUCTION AND ORGANIC REDUCTION OF SLUDGE BY USING MICROWAVE PROCESSES

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ABSTRACT: Biogas produced from lignocellulosic biomass, e.g. animal manure, has the potential to be a promising renewable energy source. Anaerobic digestion (AD) is the most common process for dealing with sludge. Residual biomass after conventional anaerobic digestion contains varying amounts of cellulose and lignin. To the enhancement of biogas production and organic reduction of sludge by using a microwave (MW), processes are currently, the second generation from anaerobic digestion by using a microwave technique. Microwave is one of the non-mechanical methods of cell disruption in the field of sludge treatment. In the present work, the microwave treatment method is investigated in the Tractor and Farm Machinery Test and Research Station, Alexandria governorate to find the microwave effect on biogas production and volatile solid removal efficiency during anaerobic digestion. Low solids systems with a total solid (TS) content of about 10 % were analyzed at 40o C and the pH value is 8 in chemical treating processes. Microwaving is used to disrupt connections within cell walls, as well as to denature proteins. The objectives of the present work may be summarized as follows: - Evaluate the effect of microwaves on all performance indicators of the anaerobic digestion unit. - Determine the optimum conditions affecting the performance of the developed prototype. - Compared the results of the preliminary investigation of microwave (MW) irradiation pretreatments on the anaerobic digestion with an untreated treatment (control treatment). Low solids systems with a total solids (TS) content is 10.0% were analyzed at 120 rpm rotational speed of mechanical stirring. The inoculum to the bulk mass of substrates with water ratio was 10% (mass basis). The experimental conditions for pretreatments were as follows: - Three runs with microwave irradiation times 10, 20 and 30 min. -Three microwave irradiation power (100 , 450 and 800 W). It gives the best results to increase the rate of biogas production to reach the maximum value of 8.27 liters/day in 16 days of hydraulic retention time using a 450 W microwave for 30 min.

1. INTRODUCTION

Anaerobic digestion technologies have traditionally been employed to reduce of volume and weight of activated material and produce the corresponding amount of biogas. The slow degradation rate of activated material in an anaerobic digester is due to the rate-limiting step of activated material hydrolysis. This is caused by a low biodegradability of the cell walls and extracellular biopolymers inactivated material. It is important to reduce the amount of activated material produced and to reduce its residual organic content. The methods of improvement of the biodegradability of a particular substrate are mainly based on better accessibility of the substrate for microorganisms. Pretreatment of sludge-activated material by mechanical, chemical, or thermal disintegration can improve the subsequent anaerobic digestion. It is known that low-dose ultraviolet rays can stimulate bacterial growth and Microwave irradiation was found to have a positive effect on the overall anaerobic digestibility has improved the process of anaerobic is integration, solubilization, biological degradation, cell disruption of stabilized activated materials, the disintegration of bio-solids and the reduction of weight and volume. This review will discuss the role of microwaves in all these processes. Microwave irradiation pretreatment enhanced the solubilization of VS and COD and concomitantly increased the rates and cumulative production of biogas. The availability of energy sources has become a global issue due to the rapid depletion of fossil fuels (**Singh et al. 2011**) **[1]**. Many countries have begun to use alternative energy sources, especially clean energy sources such as geothermal energy, wind power, small hydropower, solar energy, biomass energy, tidal energy, and wave power. Clean energy is less polluting than fossil fuels and therefore has less impact on the environment. Biogas produced by anaerobic digestion is an interesting alternative to clean energy (**Esposito et al. 2012**)**[2]**. Anaerobic digestion is energy efficient and environmentally friendly energy production technology for sustainable energy production. It not only generates energy but also reduces the amount of organic waste and the area used to fill the earth (**Carrere et al. 2010**)**[3]**. Biogas is a mixture of methane and carbon dioxide (**Angelidaki et al. 2009**)**[4]**. Natural gas contains approximately 90-95% of methane, but biogas contains approximately 50-65% methane. Therefore, biogas is a low-grade natural gas. Biogas is produced by the decomposition of organic matter by anaerobic bacteria, leading to the production of methane. Biogas production requires the addition of bacteria to the organic fodder accumulated under anaerobic conditions and the appropriate temperature for the growth and propagation of methanotrophic bacteria (average temperature 30-35 °C and heat-loving temperature 50-60 °C) (**Chisti 2007**)**[5]**. Setting up the optimal initial OLR is also important especially during start-up periods because an increase in OLR is typically followed by a decrease in HRT, leading to improper degradation of a specific material under specific operating parameters (**Schnürer et al. 2017**)**[6]**. In Microwave processing, periodic sound pressure (ultrasound) is applied to a variable frequency to disassemble the cell wall from microcells or sludge blocks in the sludge (**Apul and Sanin 2010**)**[7]**. starting- up with 50% inoculation following an exponential feeding strategy was the best strategy for treating a source-sorted organic fraction of municipal solid waste (**Khoshnevisan et al. 2018**)**[8]**. Bring powerful turbulent vortices of about 5-100 µm volume around collapsed bubbles. High temperature and pressure developed within collapsed bubbles cause chemical physical effects (**Monnier et al. 1999**)**[9]**. In the first phase of the synthesis, the structure of the fine particles breaks down, and later, a fraction of the insoluble organic matter dissolves (**Shimizu et al. 1993**)**[10]**.

However, the peak height (max. rate of degradation) of cellulose peak for control at 341 °C is slightly affected and is shifted to 0.26 from 0.20% /°C due to MW pretreatment. This showed that Microwave destroyed the organic species and hemicellulose to a greater extent while the structure of cellulose was slightly altered and opened, which may account for increased biogas production. The anaerobic digestion of activated sludge has significantly improved the following waste of Microwave processing causing microbial cell degradation. There has been an increase in the degradation of volatile solids as well as an increase in the production of biogas. The increase in digestion efficiency was proportional to the degree of sludge disintegration. (**Tiehm et al. 2001**)**[11]**. In mesophilic reactors the test results showed only a small difference for the two cases, a correlation was found to exist between gas production and energy. Ultrasound therapy is one of the most promising techniques to disrupt aerobic bacteria. The Microwave treatment has many advantages, including operating reliability, no odor generation, no clogging problems, and good separation of water from the final sludge (**Bougrier et al. 2006**)**[12]**. The degradation efficiency of organic matter was increased from 38.0% to 50.7%, which is much higher than that with Microwave (42.5%) or with Na OH pretreatment (43.5%) in the subsequent aerobic digestion at the same retention time (**Jin, et al. 2009**)**[13]**. The microwave energy inputs in the range of 31–93 W h/L enhanced biogas production from differently composed substrates up to 71%. The highest increase was found for lignocellulosic-based materials and was related to improvements in solubilisation. Conversely, a lower biogas enhancement, in the range 3–23%, was found for protein-rich substrates. In this case, any relevant variation in soluble COD was observed after Microwave pretreatment (**Cesaroa, et al. 2014**)**[14]**. Thus, dependent upon the specific substrate composition, the relationship between solubilisation and anaerobic biodegradability was found to differ significantly and this evidence represents a key point for scaling up the combined Microwave/anaerobic digestion process.

This study aimed to study the effect of ultrasound doses on the efficiency of anaerobic digestion. by assessing the effects of ultrasound treatment on the concentration of suspended solids and the effect of Microwave processing on biogas production rate.

1. MATERIALS AND METHODS

The main experiments were carried out in the Power and Energy laboratory at Tractor and Farm Machinery Test and Research Station Alexandria Governorate. A model was manufactured and developed to determine the optimal configuration and the operating conditions for the effect of microwave irradiation on biogas production.

2.1. Fresh Cattle Dung

For trials, about 2 m^3 of fresh livestock manure was obtained from the dairy farm of the research station, faculty of agriculture, University of Alexandria, Alexandria Governorate. The cattle dung was taken directly after and analyzed to determine the chemical characteristics such as total solids, volatile solids, organic carbon, and total nitrogen that are listed in Table 1

Table 1 Chemical analysis of cattle dung

Parameters	Measured value
Total solids (T.S),%	30.46
Total volatile solids (T.V.S),%	70.83
Total organic carbon (T.O.C),%	40.69
Total nitrogen (T.N),%	1.82
Carbon / Nitrogen ratio (C/N ratio)	25.44: 1
pH	7.07

2.1. The Microwave Anaerobic Digester System

The Microwave Anaerobic Digester System used in this study consists mainly of an anaerobic digester unit, Microwave generator unit, and power amplifier unit.

2.1.1. Anaerobic digester unit

Six biogas digesters were manufactured for this research work. as shown in figures (1a, 1b). Each digester consists of a digestion chamber, an inlet and outlet tube, and a biogas tube.

Fig. (1a) Microwave Anaerobic Digester System.

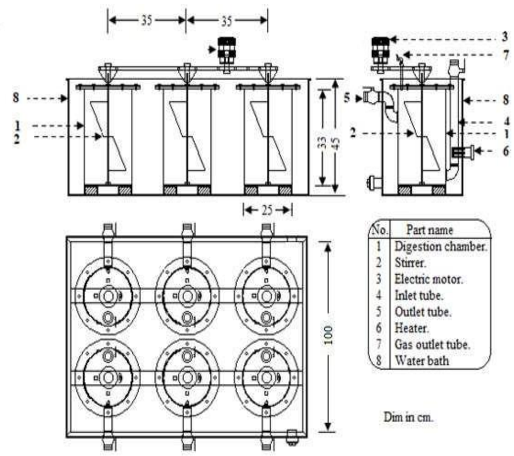


Fig. (1b) Sketch of Anaerobic Digester System.

a. Digestion chamber

The digestion chamber as shown in Fig. 2. was cylindrical in shape and made from a sheet of 2 mm thick stainless steel. It has gross dimensions of 33 cm high and 25 cm in diameter with a total and actual volume of 16 and 14 liters, respectively.

Fig. 2 The digestion chamber and biogas outlet pipes.

a. Biogas outlet pipes

biogas outlet pipe was a Plastic Flexible Pipe/Tube 1/4" in diameter joined by a 3 mm thick rubber gasket inside the hole perforated inside the upper caps of the digestion chamber.

2.2.2. Microwave oven unit

The Mechanical Control Microwave is shown in Fig.3. With 800 W Output, 6 power levels, Timer 30 min, 10 Seconds Turntable, and Dimensions (W*D*H) (439.5x355x258.2) Microwave. Was used in this study. Some of the microwave specifications are listed in Table 2.

Fig. 3 Microwave Oven.

Table 2 Microwave specification

Parameters	Measured value
Interior capacity,	20
Microwave power,	800 W, 2450 MHz
Oven size, (W*D*H),	439.5x355 x258.2

2.2. Laboratory Equipment

Several measurements were executed using different measuring devices.

2.2.1. Arduino methane measuring device

The percentages of methane gas in the production of experimental gas (at determined purity of methane gas) were measured using an Arduino methane measuring device, which consisted mainly of two main units, Arduino Nano board and methane sensor (MQ-4).

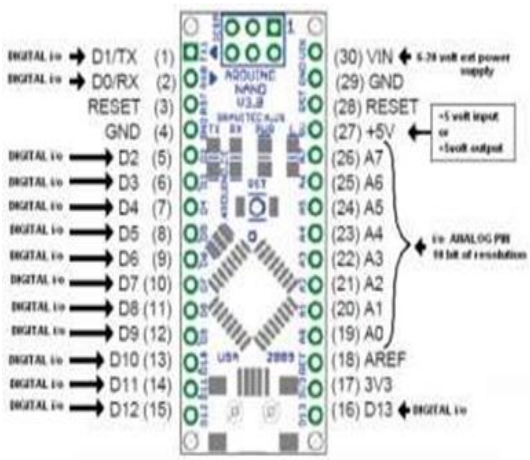
a. Arduino board

Arduino can provide an inexpensive open-source electronics prototyping platform based on flexible, easy-to-use hardware and software **(David et al. 2007)**[15] . The Arduino Nano (Fig. 4) is a single-board microcontroller, intended to make the application of interactive objects or environments more accessible. It has 14 digital I/O pins which can be used to interact with various peripherals. The Arduino also has six analog pins which are internally connected to an analog with a digital converter (ADC) to measure a range of analog voltages **(Kadimi and Dheeraj 2014)**[16]. It can be run by a laptop with a USB cable or by an AC-to-DC adapter or a 6 –20 V battery. Arduino-compatible custom sensor expansion boards, known as shields, can be developed to directly plug into the standardized pin headers of the Arduino board. They enable Arduino to connect to several sensors **(Ferdoush and Li, 2014; Hut, 2013)**[17]. Arduino plays a vital role in helping agricultural researchers and engineers by utilizing Arduino boards in different applications **Kadimi and Dheeraj (2014)**[18].

Fig. 4 Arduino Nano commercial microcontroller component.

MQ-4 Methane Sensor Circuit Built with an Arduino:

The methane sensor we will use is the MQ-4 sensor (Fig. 5). This is a sensor that is sensitive to the effects of methane. MQ-4 sensor coupled with a simple adjustable comparator circuit



using an LM393 IC. Detects Natural Gas & Methane, in the air. Designed to interface to an Arduino or other microcontrollers.

Fig.5 Methane sensor (MQ-4).

a. MQ-4 Methane Sensor Circuit Schematic

The methane gas sensor circuit was built with an MQ-4 sensor integrated with an Arduino as shown in Fig. 6. The connection point of the sensor is four leads. Two of them are for power. The Vcc terminal of the sensor connects to the 5V terminal of the Arduino board. The GND terminal of the sensor was connected to the GND terminal of the Arduino. This establishes power for the sensor. The other two connections (A out and D out) were connected to analog pin A0 and digital pin D8, respectively of the Arduino.

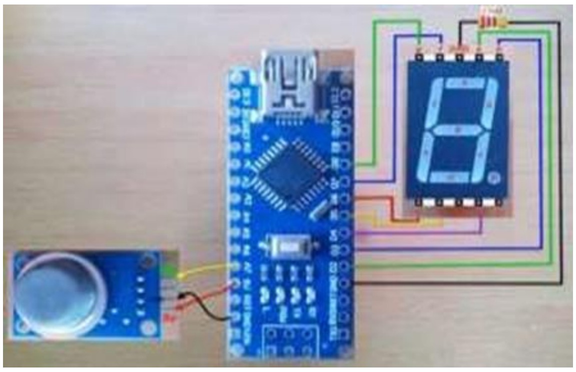


Fig. 6 MQ-4 methane Gas Sensor Circuit Schematic.

b. Arduino Sketch Code

The code needs to be uploaded to the Arduino so that it can measure methane gas levels, as shown in Fig. 7.

Fig. 7 Arduino Sketch Code.

2.1.1. *Arduino pH meter*

pH is a measure of the acidity or alkalinity of a certain solution. The pH scale ranges between zero and 14. The PH sensor kit includes a sensor probe and signal conditioning module that was connected with a BNC connector. The PH sensor is depicted in Fig. 8. The module has two potentiometers, one for adjusting the measured value offset and the other for adjusting the maximum measured value. Both are used for sensor calibration.

Specifications:

Supply Voltage: 5.V Measuring Range: 0 -14 PH

Measuring Temperature :0-60 ℃ Accuracy: ± 0.1 pH (25 ℃) Response Time : ≤ 5 sec

Fig. 8 Arduino PH meter.

2.1. Methods

Evaluation of the constructed digesters was carried out taking into consideration the following indicators

2.4.1 Gas yield

Monitoring of biogas production is the most common method and gives important information about the state of the anaerobic degradation process. To determine biogas production under digester operating conditions, the daily biogas production was volumetrically cumulated under laboratory conditions using water displacement a metering system as shown in **Fig. 9.** and correct non-temperature- or pressure-compensated biogas production measurements to standard conditions, the following relationship (the general gas law) should be used.

V2 = V1 * (T2/T1) * (P1/P2) **(1)**

Where: V1 = gas volume (m^3) at temperature T1 (°K) and pressure P1 (mm Hg) V2 = gas volume (m^3) at temperature T2 (°K) and pressure P2 (mm Hg)

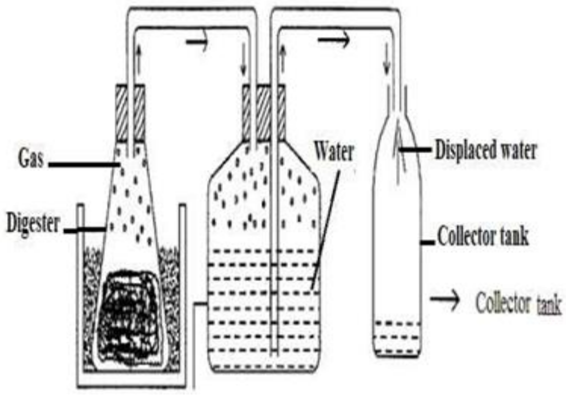


Fig. 9 Biogas measuring method using water displacement system.

2.4.2. Biogas compositions

The biogas compositions (methane content and others) were measured using a portable gas analyzer the methane gas sensor (MQ-4) with an Arduino board.

2.4.3. Chemical analysis

a. Total solids

The most important physical parameter of water is total solids content. The total solids content is composed of floating material, settleable matter, colloidal matter and matter in solution. Total solid content is the residue remaining after a water sample has been evaporated and dried at a specified temperature (103 to 105℃). The total solids of slurry were calculated as follows according to, (**APHA, 2005**)[19].

TS % = (Final weight/Initial weight) * 100 **(2)**

b. Modifying the moisture content

Water was added to the different prepared raw materials to form a slurry of desired total solids concentration (10 %). The amount of water required to adjust the total solids of the slurry was calculated as follows according to **(*Gómez et al. 2019*)**[20]**.**

$$\boldsymbol{D_w = R_m \left[\frac{TS_{rm} - TS_{dig}}{TS_{dig}}\right]} \qquad \textbf{(3)}$$

Where, D_w is dilution volume (liter), R_m is the amount of raw material added (kg), Ts_{rm} is the total solid of raw material (cow dung) and Ts_{dig} is the total solid of fermentation material.

C. Volatile solids

Volatile solids (VS) amount is the amount of combustible material in a sample. Is determined by an analytical method called "loss on ignition" the amount of volatile matter and incinerator from an exposed sample at 550 ° C for two hours. Organic matter (containing carbon) is lost and the remaining material is the mineral component or ash of the original sample. VS amount is usually reported as a percent of total solids (TS), where TS is the sum of the VS and ash components (**APHA, 2005**)[21] .

The volatile solids (VS) mass in kg was determined using the following formula (**Wittmaier, 2003**)[22].

$$VS\ (kg) = M_{fresh} \times VS\% \qquad \textbf{(4)}$$

D. Organic matter and organic carbon (O.M & O.C)

The percentage of organic matter was estimated from the percentage of ash using the following equations **(Kim et al. 2013)**[23]**.**

$$\text{Organic matter } (\%) = 100\ (\%) - \text{ash } (\%) \qquad \textbf{(5)}$$

Where: Ash (%) is the solid that remains after burning.

Organic carbon content can serve as an indirect determination of organic matter through the use of an approximate correction factor. The "Van Bemmelen factor" of 1.724 has been used for many years and is based on the assumption that organic matter contains 58 percent organic carbon.

The equation for the estimation of the organic matter according to this factor is the following one: OM (%) = 1.724 x OC (%) **(NRCS, 2011)**[24] **.**

$$\text{Organic carbon } (\%) = \text{Organic matter } (\%) / 1.7421 \qquad \textbf{(6)}$$

2.3. Experimental Setup and Procedure

In this study, a biogas laboratory was chosen at the tractors and farm machinery research and test station in Alexandria Governorate to assess the effect of microwave on all performance indicators of anaerobic microwave digestion including biogas production rate, energy requirements, and system efficiency. This is under different microwave power of:

- Three runs with MW irradiation for 10, 20 and 30 min.
- Three microwave irradiation power (100, 450 and 800 W).
- To study the enhancement of digestion, the blank test (control) was done.

2.3.1. The operation process

This experiment aims to achieve steady-state conditions under the batch fermentation process with a heating system of 40 ℃ and 120 rpm speed of mechanical stirring and 10% concentrations of total solids (TS). The batch operation period was about 25 days. The active volume of every digester was 14 liters. Before pre-treatment, samples were blended for 3-5 min to obtain a homogeneous mixture. From equations (2 and 3) Each digester was filled first by

12.6 liters of diluted cattle dung with 10.75% TS and 1.4 L of inoculum material (about 10% of active volume) with a concentration of 3.4 % TS. The final initial mixture had a 10 % of total solids. The inoculum was obtained from an actively operated biogas digester fed with cattle dung.

The volume of biogas was daily measured using the liquid displacement method and converted to the volume at the standard temperature and pressure conditions. In the water displacement method, the produced biogas is distributed into a gas collection bottle filled with acidified water saturated with salt, causing an equivalent volume of solution to be displaced to a graduated cylinder. From this displaced solution, one can easily record the volume of biogas produced during the day Yuan, et al. (2014). Liquid and the produced biogas samples were taken daily. The methane and carbon dioxide contents of the biogas were determined by the MQ-4 Methane Sensor. The pH was measured by a digital pH meter (Arduino PH meter). TS, volatile solids (VS), and ash were measured by the Standard Methods for the Examination of Water APHA, (2005).

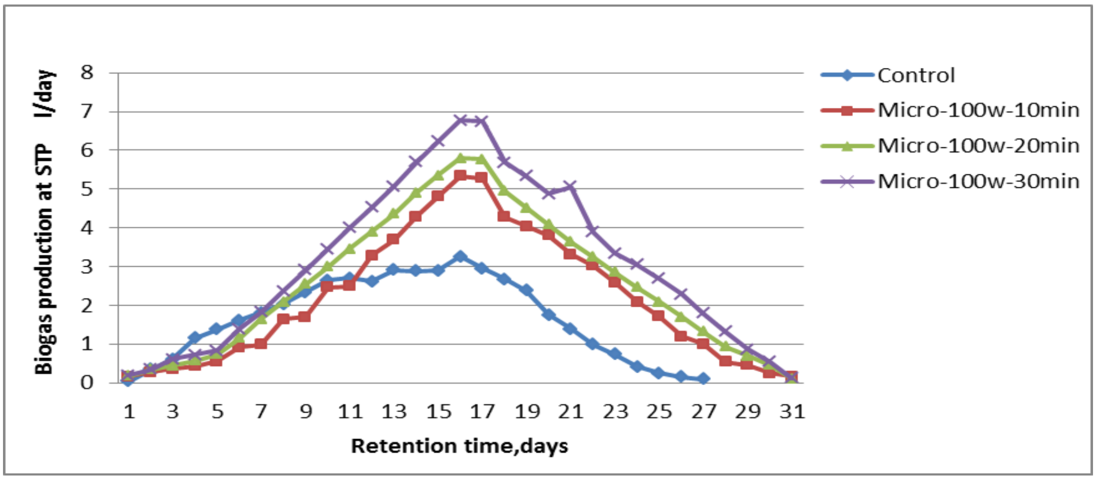
After loading the digesters with the organic wastes, the anaerobic reactors were sealed to maintain anaerobic conditions. All experiments were operated at pH within the range of (6-8).

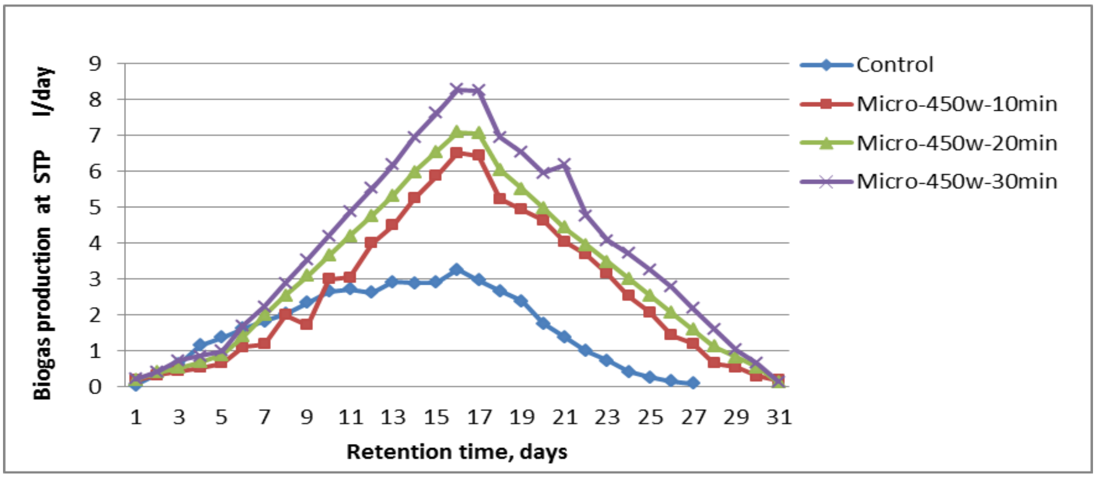
3. RESULTS AND DISCUSSION

This investigation aimed to evaluate the effect of microwave irradiation on biogas production rate, productivity and its content of methane (CH_4) during anaerobic digestion of cattle dung, at three different run times of microwave irradiation for 10, 20 and 30 min under 10 % total solids, at mesophilic (35-40 ℃) at 120 rpm stirring.

3.1. Effect of Using and Without Using Microwave on Biogas Production at STP

The daily biogas production rate (l/day) and cumulative (l) with and without using the microwave on cow manure biogas production at STP at 10% total solids (T.S.) are illustrated in Figures (10 and 11). The results revealed that the biogas was produced through the first days after the digesters feeding. This production may be due to the active inoculum added during the digester feeding with fresh cattle dung. The results illustrated in Fig. 10 showed that the biogas production rate increased with time until reached the maximum values of 3.61 l/day at 16 days, 5.92 l/day at 16 days, 5.8 l/day at 16 days, 6.78 l/day at 16 days, 6.51 l/day at 16 days, 7.08 l/day at 16 days, 8.27 l/day at 16 days, 5.42 l/day at 16 days, 6.41 l/day at 16 days, and 7.14 l/day at 16 days of hydraulic retention time without using microwave and with using 100 W microwave at 10 min, with using 100 W microwave at 20 minutes, with using 100 W microwave at 30 minutes, with using 450 W microwave at 10 min, using 450 W microwave at 20 min, using a 450 W microwave at 30 minutes, using an 800 W microwave at 10 minutes, using an 800 W microwave at 20 minutes, and using an 800 W microwave at 30 min, respectively.





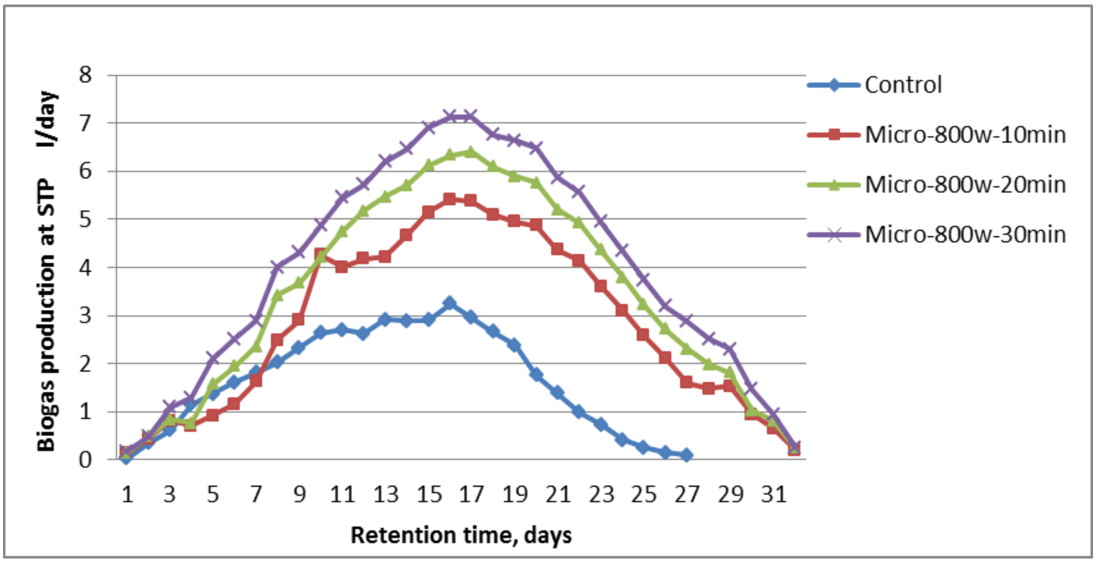


Fig. 10 Effect of using and without using microwave on cow manure daily biogas production (l/day) at STP.

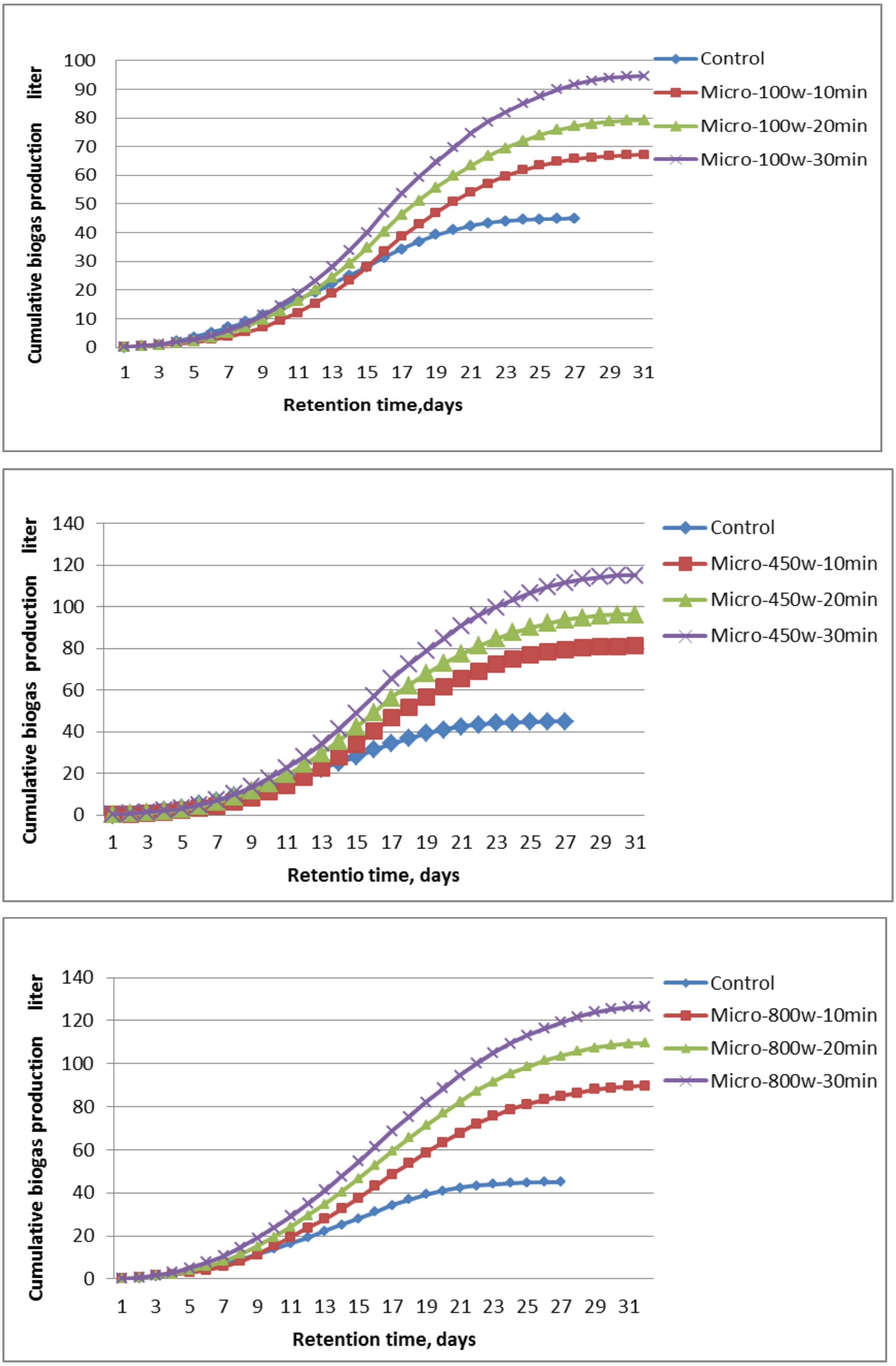


Fig. 11 Effect of using and without using the microwave on cow manure cumulative biogas production (l) at STP.

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