ENHANCING BIOGAS PRODUCTION BY THERMAL PRETREATMENT OF AGRICULTURAL WASTES

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ABSTRACT

Developing countries are suffering from many problems such as shortage in energy and electricity in addition to increasing wastes that cause high pollution. Biogas production is gaining increasing attention worldwide to avoid global warming, increasing energy security and the need for sustainable waste management. So the aim of the present study was enhancement of biogas production by using thermal pretreatment for the agricultural wastes. A laboratory scale bioreactor made from plastic, 8 liter capacity, was used in this research. Three different mixtures were used (100% cattle dung (CD), 50% CD + 50% BS, and 100% bagasse (BS)). The samples were heated in a furnace at 175 ^oC, and 200 ^oC. The samples were subjected to heat for three different durations 20, 40 or 60 min. Also samples were partially heated at four levels, 25%, 50%, 75% and 100%. The results indicated that the maximum daily biogas production, accumulative biogas production and methane production related to volatile solids mass (VS) were 732.4,15475.5 L/kg VS, and 577.9 LCH_4/Kg VS respectively for the second fermentation mixture (50% CD+ 50% BS) and 60 min heating time using temperature of 175 °C. But the maximum calorific value was 7241.7 kCal/ m^3 for the third fermentation mixture (100% BS) using temperature of 200 °C for 60 min and 100% heating of samples.

Keywords: *Biogas, thermal pretreatment, bagasse, heating time, methane production, calorific value.*

INTRODUCTION

Egypt generates huge amounts of farm wastes yearly. The removal of those wastes is a major concern and considered one of critical environmental challenges. Those wastes have coarse plant byproducts, farm yard manure, perished fruits and vegetables, and other wastes which chemically low in protein and fat contents.

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Most of farm wastes are high in lignin and cellulose contents. In addition, energy demand is increasing rapidly worldwide and resulted in a rapid depletion of fossil fuel resources. It is of great importance to find other sources of clean energy that cause minimal damage to the environment and reduce the greenhouse gases (GHGs) emission to the atmosphere (**IPCC 2000**).

Biogas generated by digestion has been proven to be the most energy efficient and environmentally beneficial technology for bioenergy production **(Fehrenbach *et al*. 2008).** Anaerobic digestion (AD) is used to produce biogas. AD is a biochemical process where different types of anaerobic microorganisms decompose complex organic matter (biomass) to smaller components, in the absence of oxygen. Biogas is not a pure chemical substance. It consists of 50-70% methane, 30-50% carbon dioxide and other minor ingredients such as, ammonia, hydrogen sulfide, siloxane and halides **(Bakhov *et al*. 2014).**The proportion of each of these components depends on the ingredients of the raw substrates and the process setting used during the digestion **(Bond** and **Templeton, 2011).** Methane content is the limiting factor of biogas calorific value. One cubic meter of biogas contains 60% methane with calorific value of 21.5 MJ equivalents to 5.97 kWh of electricity at standard temperature and pressure (STP).

The procedure of anaerobic digestion occurs through four progressive stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The anaerobic digestion process is reliant on the cooperation between the different microorganisms that can complete the four previously mentioned stages**.** However, some substrates are decomposed very slowly due to the inhibitive effect of some of their chemicals to the growth and activity of the microorganisms. Also, their molecular configuration is intricate to the degree that microorganisms and their enzymes become unable to decompose **(Montgomery** and **Bochmann 2014).** To overcome some of these problems and increase degradability of complex molecules such as sugars and lignocelluloses, various chemical, mechanical and thermal pretreatment technologies have been developed. Those techniques include but not limited to thermal treatments and enzymatic processes. In thermal pretreatment, the wastes are heated (on average 125 to 220 °C) under pressure and kept at that temperature for the desired time. This can be conducted with autoclaves, ovens or microwave devices. Water must be added to dry wastes before thermal treatment to prevent the hydrogen bonds that linked with crystalline cellulose and the lignocelluloses complexes, causing the biomass to swell **(Garrote *et al*. 1999).** Several investigators stated that heating of wastes before anaerobic digestion has improved methane production and volatile solids reduction **(Bharathiraja *et al*. 2018).** Many studies stated that the optimal thermal range of 160–180 °C was suitable for hydrolysis of wastes to increase methane production during anaerobic digestion **(Jayashree *et al*. 2014)**. **Qiao *et al*. (2011)** conducted several experiments that include thermal preheating at 170 °C for one hour. They reported that the biogas production increased significantly for different substrates used in the study**. Bougrier *et al*. (2008)** classified thermal pretreatment into two groups. The first used temperatures between 70 and 121 °C led to enhancing biogas yield about 20-30%. The second group is between 160-180 °C which led to more biogas production enhancement.

Biogas is a form of renewable energy sources. Biogas is used for replacement of fossil fuels for power and heat production purposes. Also, it can be used as gaseous vehicle fuel. Methane-rich biogas can be used instead of natural gas as a feedstock for producing chemicals and materials **(Weiland 2010).** So the aim of this study is to examine the effects of thermal pre-treatment by varying temperature, exposure time, and heated proportion of two farm wastes and their mixture on enhancement of biogas production, methane percentage, and calorific value.

MATERIALS AND METHODS

This study was carried out between June and August 2018 at Faculty of Veterinary Medicine, Zagazig University, Egypt to evaluate the influence of two heating temperatures as thermal pretreatments, three heating times and partial heating of substrate samples using two different agricultural wastes and their mixture on biogas production.

1. Materials

1.1. Two types of biomass lignocelluloses-rich, cattle dung (CD) and bagasse (BS) were used in this study. The bagasse wastes were fine ground into particles with an average size of 1.0 mm then the ground wastes were classified into three groups according to mixing ratio (A: 100% CD, B: 100% BS and C: 50% CD + 50% BS) and analyzed for chemical characteristics as shown in Table (1). Bagasse components were determined as 39% cellulose, 25% hemicelluloses, 26% lignin, and 10% minerals and other components. The mixtures of organic wastes were mixed at a ratio of 1: 2 w/w with water to arrive at the desired solid content. Fresh rumen extract was used as a starter of anaerobic digestion.

Table 1: Chemical analysis of agricultural wastes used in the experements.

	Protein, %	N, %	C/N	pH	ASH, %	TVS, %	TMC, %	TS, %
Cattle Dung (CD)	13	2.5	29	6.9	14.1	85.9	87.9	12.1
Bagasse (BS)	7.5	1.1	31	6.6	16.8	83.2	84.5	15.5
Mixture (50% CD + 50% BS)	11.7	1.9	30	6.7	15.9	84.1	86.4	13.6
Starter	29	4.3	20	6.8	37.2	62.8	90.2	9.8

TVS = Total Volatile Solids, TMC = Total Moisture content, and TS = Total Solids.

1.2. A number of laboratory scale bioreactors used in this research. They were 8 liter capacity plastic prototype reactors as shown in **Fig.1**, with 6 liter as a filling volume and 2 liter as a headspace volume. Each reactor was connected with a plastic tube with diameter of 0.5 cm just above the slurry. After the fermentation started, the gas bubbled up entering the tube which led to a flask that half filled with a brine solution to work as pressure regulator. The end of the tube was immersed near the bottom of the flask. Another tube exiting from the top of the flask connected the flask to the gas collection setup. The gas collection setup consisted of a water path and an inverted graduated cylinder of 10 litter capacity filled with water. The tube is inserted under the inverted cylinder so the gas would bubble up displaces a volume of water equivalent to the volume of biogas produced.

1.3. Top loading balance (50kg “Five goat” model Z051599), Digital pH meter, thermometer, electric oven and electric muffle were used with all experiments.

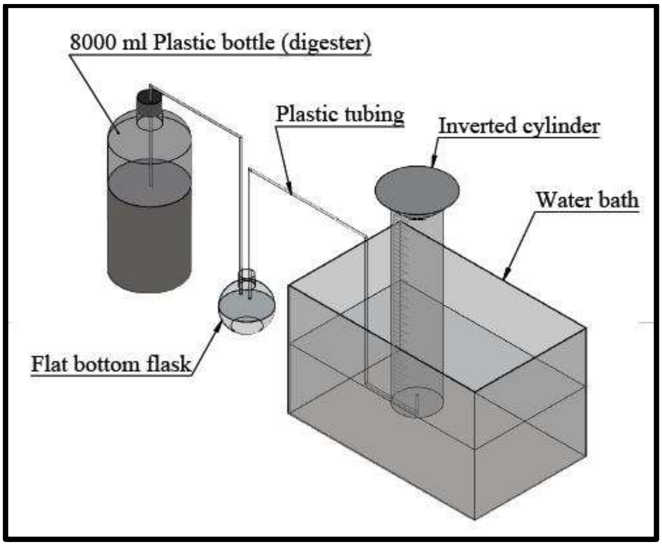

Figure 1: Schematic diagram of the experiment layout.

2. Methods

2.1. Experimental conditions

All experiments were conducted under the following parameters:

- Three different fermentation substrates, 100% CD, 100% BS, and 50% CD + 50% BS.
- Two different temperatures of (175 and 200 °C) as a thermal pretreatment.
- Three different durations of thermal pretreatment (20, 40 and 60 min).
- Four different levels of wastes partial heating (25%, 50%, 75% and 100%).

2.2. Thermal pretreatment

The mixed waste samples were put in 2 liter tempered glass flask and mixed with distilled water at ratio 1: 4 w/w to prevent dry biomass from burning during pretreatment. The flasks were covered by aluminum foil and put in a high temperature furnace (Model SX-5-12, made in China). The temperature regulator was set at 175 and 200 °C. The temperature increased gradually until reached the set point. Once temperature was reached the set point, the furnace was held for the selected durations of time (20, 40 or 60 min) for each temperature of thermal pretreatment. After the different pretreatments were carried out, the flasks were collected from the furnace and left to cool down at room temperature. The pretreated samples at different temperatures were placed in the bioreactors with distilled water at a ratio 1: 2 w/w.

2.3. Analysis and calculations

Standard analytical methods (**Tran, 2017**) were employed to determine total solids (TS), volatile solids (VS) and total Kjeldahl nitrogen (TKN) of sugar cane bagasse and cattle dung before pretreatment. Total organic carbon (TOC%) was calculated by dividing the value of volatile solid content (VS%) by 1.8 according to **Adams *et al*., 1951.** Produced biogas was measured by a wetted displacement method according to **Gosch *et al.* (1983)**

2.4. Measurements

All treatments were evaluated taking into consideration the following indicators:

2.4.1 Daily biogas production

The volume of produced biogas was equal to the volume of displaced water from the inverted graduated transparent 10 L plastic cylinder as used by **Gosch *et al*. (1983)** then corrected to account for temperature variations using the following equation:

$$V = \frac{V_m \cdot \mathrm{T}}{\mathrm{T}_\mathrm{m}}$$

Where: V: biogas volume at STP, ml, V_m: biogas volume at room temperature, ml, T: standard temperature, 0 °C (273 °K), and T_m: room temperature in °K.

The following equation was used to estimate biogas production based on the amount of volatile solids consumed per day:

$$V_{BG} = m_{VS} \times Y_{BG}$$

Where V_{BG}: is the estimated biogas production rate associated with feed material (m^3 biogas/day), m_{VS}: is the mass flow rate of volatile solids contained in feed material (kg volatile solid/day), and Y_{BG}: is the biogas yield of feed material (m^3 biogas/kg volatile solid).

2.4.2 Methane and carbon dioxide content

The volumes of CH_4 and CO_2 in biogas were estimated by the concentrated alkaline absorption method using the potassium hydroxide (KOH, 40%). The KOH device consists of glass U-tube shaped with 12 mm internal diameter filled with KOH **(Ezekoye** and **Okek, 2006)**. The U-tube attached with tap in order to adjust the solution level with atmospheric pressure after CO_2 removal. The tube was installed with samples injection as a biogas inlet and with gas outlet to release gases after CO_2 removal. The percentage of CO_2 in biogas was calculated as follows:

$\boldsymbol{CO_2}\% = \frac{V_1 - V_2}{V_1} \times \mathbf{100}$, (**Abdel-Hadi** and **Abd El-Azeem 2008**).

Where V_1: biogas volume before removal of CO_2, ml, and V_2: volume of the other gases after removal of CO_2, ml.

$\boldsymbol{CH_4}\% = \mathbf{100}\% - [\boldsymbol{CO_2}\% + \mathbf{3}\%(\boldsymbol{H_2S}\ \textit{and other Gases Vol.})]$, (**Abdel-Hadi** and **Abd El-Azeem 2008**). Where: 3% is the average content of H_2S and other gases in biogas corresponding to **(GTZ, 1999).**

$V_{CH_4} = V_{BG} \times C_{CH_4}$

Where**:** $\mathbf{V_{CH4}}$ is the methane production rate of feed material (m^3 CH_4/day), and $\mathbf{C_{CH4}}$ is the biogas content associated with feed material.

2.4.3 Calculation of Calorific Value (CV) of Biogas

Calorific value of biogas depends on many factors which are mainly the percentage of methane, moisture content and temperature.

CV of biogas = CV of pure methane × methane percentage.

Where, CV of pure methane = 8560 kCal/m^3, (**Salunkhe *et al.* 2012)**

RESULTS AND DISCUSSION

The biogas production from each treatment was measured daily and the accumulative biogas production was obtained from summing out the daily production. It is interesting to notice the difference in results for different treatments on short and long-term runs of the experiments.

Table 2: Results summary for control treatments.

Type of substrate	Daily Biogas Production, L/kg VS	Accumulative Biogas Production, L/kg VS	Methane production, L/kg VS	Calorific Value kCal/m^3
100% C.D.	258.5	4280.0	162.4	5392.8
50% C.D. + 50 BS	291.0	4630.0	189.0	5564.0
100% BS	216.3	4020.0	151.8	5992.0

The control treatments (no heating) results are shown in Table 2. It is visible that the mixed substrate gave the highest daily and accumulative biogas production, while the pure bagasse gave the highest calorific value of produced biogas due to the increased content of methane. The results summarized in Table 2 will be used as a basis for comparison throughout the discussion. The retention time (duration from start to end date) varied slightly among different substrates used in the experiments. The 100 % cattle dung treatments took 31 day but the mixed (50% cattle dung + 50% bagasse) took 36 day while the 100% bagasse treatments took the longest retention time of 40 day.

1. **Effect of heating times and partial heating under temperature of 175 °C**

Results for daily and accumulative biogas production at different heating times and partial heating for different fermentation mixtures at constant temperature of 175 ^{o}C are shown in **Fig 2**. The results showed that by increasing heating, daily biogas production increased.This result is attributed to the effect of heat on degradation of protein and complex carbohydrates bonds as well as chemical oxygen demand (COD) solubilization that led to enhancing biogas production. The results also indicated that increasing heating time to 40 min, daily biogas production increased by 47.6, 42.3, and 20.6% for 100% C.D., 50% C.D. + 50% bagasse and 100% bagasse respectively comparing to control treatment.

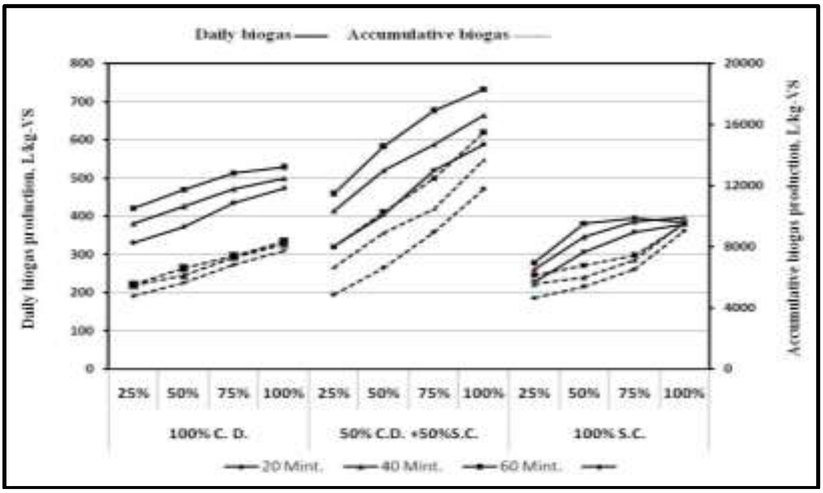

Figure 2: Effect of heating time and partial heating of samples on daily and accumulative biogas production at 175 °C

Meanwhile, increasing heating time to 60 min, led to increase daily biogas production by 62.9, 57.6 and 29.4% under the same condition. This result is similar to **(Pillia *et al*. 2015)** where they said the optimum pretreatment time for increasing biogas production are in the range from 30 to 60 min.

It can be noticed that by increasing partial heating percentage to 50%, daily biogas production increased. These values reached 470.4, 582.1 and 381.5 L/kg VS for 60 min heating time. The results revealed that heating 75% of the samples boosted biogas production to 98.8, 130 and 83.4% for 60 min heating time compared to control. This is due to dispersion of macromolecules that led to increase the surface area of the substrates that causing increase in biogas production (**Protot *et al*., 2011**). It can be noticed that the maximum daily biogas production values for each mixture were 528.6, 732.4.5 and 383.9 L/kg VS for 100% CD, 50% CD + 50% BS and 100% BS under 60 min heating time and 100% percentage heated because at higher temperatures, the substrate particles become finer. As a sequence of this, the surface area increases that led to better contact between substrate particles and the microorganism's population, thus more organic matter is converted into bio-methane.

Regarding the accumulative biogas production as shown in **Fig. 2**, the results showed that maximum accumulative biogas yield was 4860.4, 6616.6, 8958.9 and 11779.2 L/kg VS for 50% CD + 50% BS and decreased to 4673.8, 5402.8, 6548.2 and 9065.1 L/kg VS for 100% BS under the four heating percentages and 20 min heating time. The reason for this increasing lignin content and lipid in bagasse made digestion activities more difficult for the microorganisms. Reduction in digestion activities of the microbes led to slow biogas production (**Adelekan** and **Bamgboye 2009**). The results indicated that accumulative biogas values increased to 43.6, 92.4 and 49% for 100% CD , 50% CD + 50% BS and 100% BS under 40 min heating time compared to control for percentage heated samples 50%. This result is similar to **(Qiao *et al*. 2011).**

2. Effect of heating times and heated percentage under temperature of 200 °C:

It was proved that increasing temperature from 175 to 200 °C, daily biogas production increased for all treatments. The results indicated that the daily biogas production values were higher for 50% CD + 50% BS compared to 100% CD at 20 min heating.The 100% BS gave the least values under the same conditions.

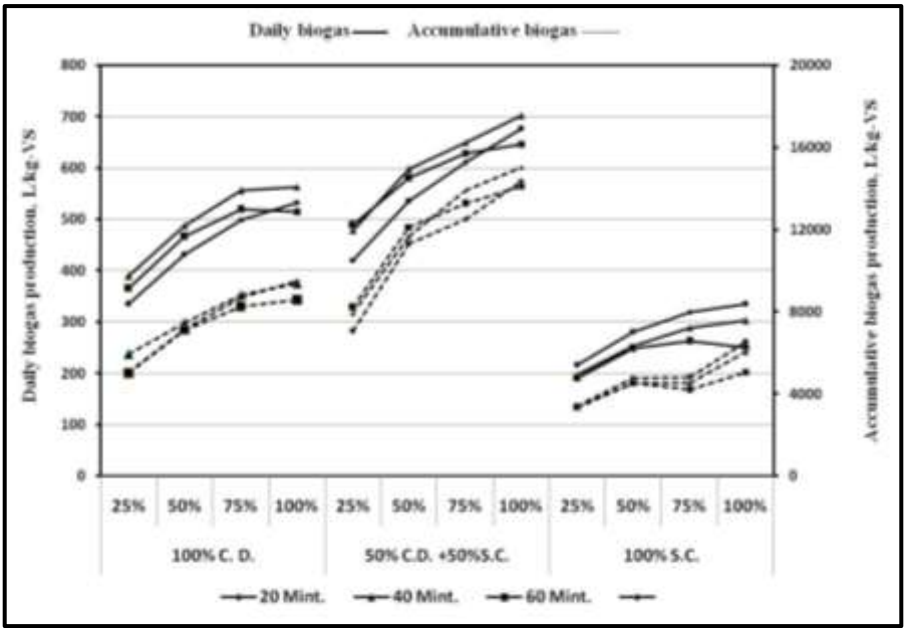

Figure 3: Effect of heating time and partial heating of samples on daily and accumulative biogas production at 200 °C

The results ascertained that increasing thermal pretreatment temperature to 200 °C, increased daily biogas production for 100% CD and 50% CD + 50% BS because higher temperature enhanced the biodegradable organic carbon by cracking the cell wall and releasing the intercellular matter in aqueous phase, which improves the digestion rates, reduces the hydraulic retention time, and increases the biogas production **(Pilli *et al*., 2011).** But these percentages decreased for the third mixture under the same previous conditions. This is because formation of toxic compounds and change in color produced that led to decreasing biogas production. In the other hand, the results showed that the values of daily biogas production under the second fermentation mixture decreased for 60 min heating time. This is due to high temperature with longer time responsible for the production of unmanageable soluble organics or toxic/inhibitory intermediates during the pretreatment process **(Wilson** and **Novak 2009)**.

Fig (3) shows accumulative biogas production for different fermentation mixtures and different heating time under thermal temperature of 200 °C. The results showed increasing treatment temperature led to increase accumulative biogas values. The second mixture gave the highest values followed by 100% CD while the 100% BS gave the least values. The results indicated that increasing heated percentage to 100% accumulative biogas increased compared to other partial heating treatments.

3. Effect of heating time and heated percentage on daily methane production and biogas calorific value under temperature of 175 °C:

Daily methane production and biogas calorific values for different fermentation mixtures under different heating time and heated percentage of samples are shown in **Fig. 4.**

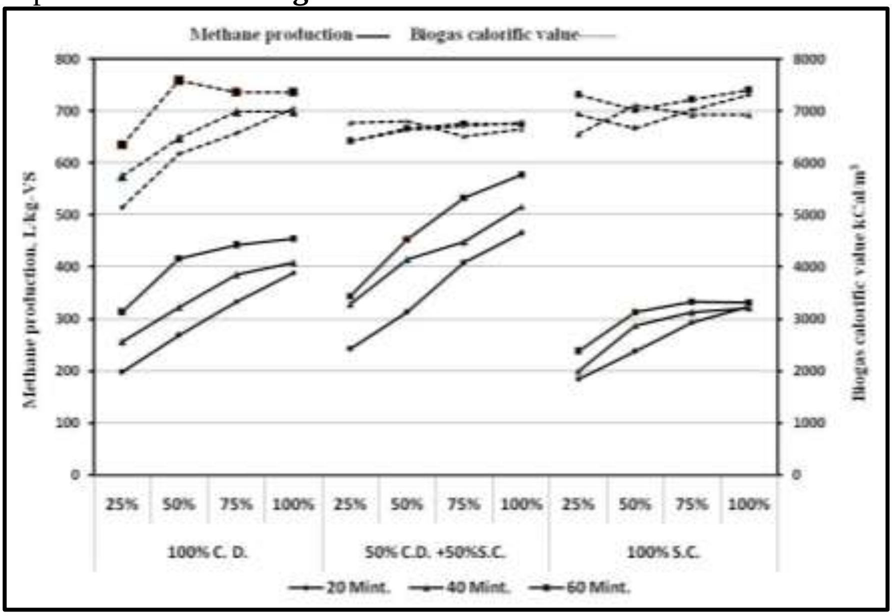

Figure 4: Effect of heating time and heated percentage of samples on daily methane production and biogas calorific value at 175 °C

The results indicated that mixing bagasse with cattle dung has a good impact on methane production. Also the results indicated that increasing heating time affected daily methane production positively. The 100% BS treatment gave the least values. The reason for this low value in methane production is probably due to the fact that significant quantities of carbon dioxide and other non-combustible gasses were produced in the digestion of bagasse.

4. Effect of heating time and heated percentage on daily methane production and biogas calorific value under temperature of 200°C:

The results illustrated in **Fig. 5** indicate that there was positive effect on daily methane production when thermal temperature increased to 200° C. That is because of high thermal temperature increasing volatile dissolved solids meaning the maximum percentage of substrate COD that is converted to methane.

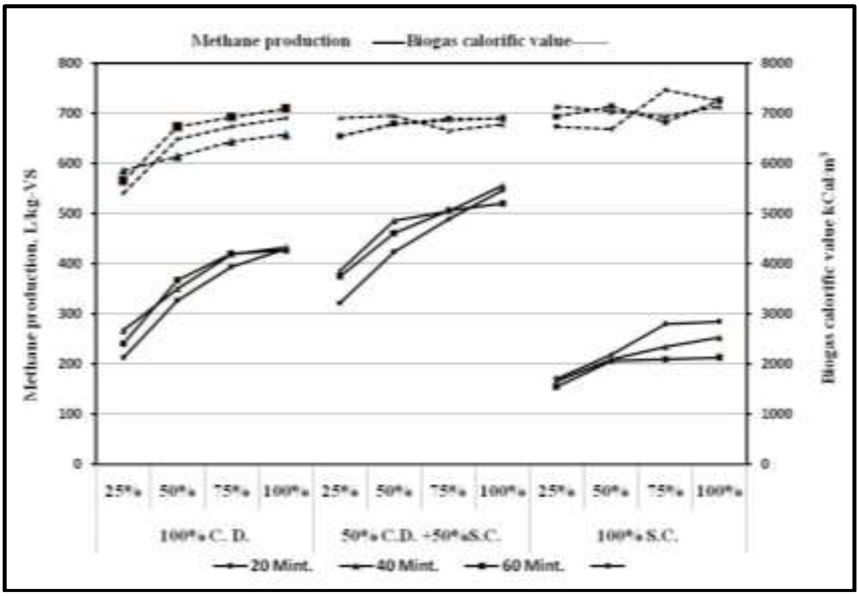

Figure 5: Effect of heating time and heated percentage of samples on daily methane production and biogas calorific value at 200 °C

It can be noticed that daily methane production ranged from 155.2 to 555.9 L CH_4/kg VS. Results also indicated that increasing heating time to 60 min, daily methane production decreased. This is in agreement with (**Bougrier *et al*. 2006**) who reported that thermal pretreatment at high temperatures greater than 170° C led to the creation of chemical bonds and resulted in the agglomeration of the particles.

It is also worthy to notice that the biogas calorific value was highly affected by the thermal pretreatment of substrate. As **Fig. 5** depicts the effect of substrate type and heated percentage on the produced biogas calorific value at 200 °C, the maximum calorific value was 7241.7 kCal/m^3 for the third fermentation mixture (100% BS) using temperature of 200 °C for 60 min and 100% heating of samples.

CONCLUSION

From the obtained results and their discussion, it can be concluded that the maximum daily biogas production values for each mixture were 528.6, 732.4 and 383.9 L/kg VS for 100% CD, 50% CD + 50% BS and 100% BS under 60 min heating time and 100% percentage heated samples. Also the maximum calorific value was 7241.7 kCal/m^3 for the third fermentation mixture (100% BS) using temperature of 200 °C for 60 min and 100% heating of samples.

According to the obtained results, it is recommended to use the 50% CD + 50% BS with 60 min thermal treatment to produce maximum biogas yield. Also, it is recommended to use thermal pretreatment of wastes by a convenient method. It was suggested to build a suitable oven with enough capacity to thermally treat the wastes. This oven should use a cheap source of energy such as burning farm wastes.

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الملخص العربى

تحسين إنتاج الغاز الحيوى عن طريق معاملة المخلفات الزراعية حرارياً

عبدالله قشطة*، ريهام صبري فيض الله** و علاء عونى**

تعانى الدول النامية من مشاكل كثيرة مثل مشكلة نقص الطاقة وانقطاع الكهرباء بالإضافة إلى زيادة نسبة المخلفات وما لها من تأثير سيء على البيئة. لذا فإن تكنولوجيا الغاز الحيوى هى الحل الأمثل لهذه المشكلات بإعادة تدوير هذه المخلفات لإنتاج الغاز الحيوى كمصدر للطاقة الحرارية. لذا كان الهدف من البحث إجراء بعض المعاملات الحرارية لتحسين جودة إنتاج الغاز الحيوى من المخلفات الزراعية ودراسة تأثير خلط المخلفات على تحسين آداء المخمرات.

تم إجراء بعض التجارب المعملية بكلية الطب البيطرى جامعة الزقازيق حيث تم تصنيع مجموعة من المخمرات من البلاستيك بسعة ٨ لتر وتم استخدام ثلاثة مخاليط مختلفة من المخلفات (١٠٠% روث مواشى ، ٥٠% روث مواشى +٥٠% مصاصة قصب و١٠٠% مصاصة قصب) وكذلك تم تسخين المخلفات تحت درجتى حرارة ١٧٥ و ٢٠٠°م كمعاملة حرارية لفترات زمنية مختلفة (٢٠، ٤٠ و ٦٠ دقيقة) قبل وضعها فى المخمرات وتم تسخين جزئى للعينات بنسب (٢٥%، ٥٠%، ٧٥% و ١٠٠%) من الحجم الكلى للعينات كما تم إجراء التجربة الحاكمة بدون معاملات حرارية. تم تقييم آداء المخمرات وذلك بقياس حجم الغاز اليومى وحجم الغاز الكلى وكذلك جودة الغازمن خلال قياس حجم الميثان. كذلك تم تقدير القيمة الحرارية للغاز الناتج. وكانت أفضل النتائج التى تم الحصول عليها هى:

١- أعلى معدل لحجم الغاز اليومى وحجم الغاز الكلى وحجم الميثان كان ٧٣٢,٤ لتر غاز/كجم، ١٥٤٧٥,٥ لتر غاز/كجم و ٥٧٧,٩ لتر ميثان/كجم منسوبة للمادة الصلبة المتطايرة عند المخلف الثانى (٥٠% روث مواشى + ٥٠% مصاصة قصب) ومعاملة حرارية ١٧٥°م وفترة تسخين ٦٠ دقيقة ونسبة العينة المسخنة ١٠٠% من الحجم الكلى.

٢- أعلى قيمة حرارية كانت ٧٢٤١,٧ ك كالورى/م^3 عند المخلف الثالث (١٠٠% مصاصة قصب) ومعاملة حرارية ٢٠٠°م وفترة تسخين ٦٠ دقيقة ونسبة العينة المسخنة ١٠٠% من الحجم الكلى.

لذلك يُوصى باستخدام المعاملة الحرارية للمخلفات من أجل تحسين آداء المخمرات اللاهوائية لإنتاج الغاز الحيوى. ويُوصى ببناء أفران حرارية لهذا الغرض تعمل باستخدام مصدر رخيص للطاقة مثل حرق المخلفات أو مصاصة القصب.

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