RESEARCH ARTICLE OPEN ACCESS

ANAEROBIC CO-DIGESTION OF AGRICULTURAL BIOMASSWASTE FOR BIOGAS PRODUCTION

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Abstract:

The Philippines is an agricultural country with a vast amount of agricultural wastes. The country's initiatives to reduce dependence on fossil fuels and greenhouse gas emissions through the Biofuels Law (RA 9367) have encouraged the use of bio-based fuel sources. Biogas is a renewable, high-quality fuel which can be produced from various organic raw materials. This study investigated the production of biogas from the anaerobic co-digestion of cattle manure with various feedstock such as rice straw, coconut shell, and sewage sludge. Results showed that the methane production increased by 162% from the co-digestion of cow manure with sewage sludge and co-digestion of cow manure with coconut shell and rice straw undergoes ammonia inhibition. With these, the anaerobic co-digestion of cow manure with sewage sludge has the best effect on the treatment of biomass feedstocks of rice straw coconut shell and sewage sludge at various treatment. This experiment would give a viable estimate of the possible methane production from co-digestion of these resources. The enhancement of the biogas yield was attributed to the improvement of biodegradability of cow manure through the addition of sewage sludge. These results are contributing to develop a feasible biogas production from rice straw, coconut shell, and sewage sludge..

Keywords — **Anaerobic co-digestion, biogas, coconut shell, methane, rice straw, sewage sludge, treatment.**

I. INTRODUCTION

Lack of power sources, especially in rural areas of developing countries, particularly Mindanao, Philippines necessitates the search for efficient power producing devices for small-scale operation. Also, the depleting fossil fuel based energy resources necessitate the search for new and renewable energy resources.

With a growing population and with increasing energy consumption coupled with higher living standards, there is a huge challenge in limiting the emissions of pollution and greenhouse gases. Moreover, the available fossil fuels are limited; consequently, with increasing prices, we need alternative energy sources to replace the fossil fuels. The commercially renewable vehicle fuels available today are ethanol, biogas, biodiesel, and electricity produced from renewable energy sources.

Renewable energy produced from lignocellulosic materials (i.e. agriculture and forest residues), non-food crops (i.e. algae and grasses) or industrial waste and residue streams have considerably lower greenhouse gas emissions (GHGs) due to its closed carbon cycle. The use of second generation feedstocks will diminish the concern over competition between food versus fuel and will lessen the potential impact on food supply.

At present, feedstocks from the municipal solid waste, industry, food processing plants and wastewater treatment plant, are limited; thus, new renewable sources are sought after. With the abundant availability of agricultural biomass in the Philippines makes it a highly interesting feedstock sourced from a variety of crops like palay, corn, sugarcane, cassava, and coconut; and livestocks such as carabao, cattle, hog, goat and dairy, that can be utilized for biogas production. These sources can serve as fuel for power generation or as feedstock for advanced biofuels production. Given the abundant supply of biomass raw materials in the country, it would be advantageous for the Philippines to exploit these resources to address the country's energy dependence, mitigate climate change and eventually achieve economic growth and prosperity.

Biogas is a renewable, high-quality fuel which can be produced from various organic raw materials and used for various energy services. Biogas technology has been developed and widely used over the world because it has several advantages – reduction of the dependence on non-renewable resources, high energy-efficiency, environmental benefits, available and cheap resources to feedstock, relatively easy and cheap technology for production, and extra values of digestate as a fertilizer. But the current status of biogas production and utilization varies largely among continents.

Biogas as renewable source of energy is becoming increasingly important since biogas has some ecological advantages mainly being CO2 neutral; hence, it reduces the formation of greenhouse gases. Furthermore, biogas represents a meaningful way of both waste use and waste disposal as agricultural, commercial and municipal waste from biogenic sources used for the production of different types of biogas.

TABLE I
TYPICAL COMPONENTS OF A BIOGAS [1]

Component	Concentration
Methane	40 – 75%
Carbon Dioxide	25 – 55%
Water (Steam)	0 – 10%
Nitrogen	0 – 5%
Oxygen	0 – 2%
Hydrogen	0 – 1%
Hydrogen Sulfide	0 – 1%
Ammonia	0 – 1%

The main components of biogas are methane (CH_4) and carbon dioxide (CO_2); it also contains significant quantities of undesirable compounds. Table I shows typical compounds and their concentrations in a biogas. For the exploitation of energy gained from biogas, the amount of methane is essential: the higher the amount of methane, the higher the output of energy from biogas. The other components are mostly useless for the energy production such as nitrogen or water. It is even possible that a negative impact on engine reliability can be provoked by trace components such as hydrogen sulfide (H_2S), ammonia (NH_3) and siloxanes. The trace components can be very harmful as the aggressive substances formed may destroy the engine, e.g., due to corrosion [1] – [2].

The existence and amounts of these contaminants depend on the sources of the biogas – landfill, anaerobic fermentation of manure or wood gasification. As they are aggressive to the engine, they should be removed before the gas is combusted in the engine using a biogas purification step. An important step during the purification process is to desulfurize the gas in particular by removing hydrogen sulfide. Further steps include drying it to remove water from the biogas or mechanical filtering to remove dust and other particles [2] – [4].

This study aimed to develop a zero-waste technology that optimizes resource recovery of agricultural wastes to produce bioenergy efficiently and in an environment-friendly approach. This could address the issues of power shortage being observed in the Mindanao, Philippines by utilizing as a fuel for the stationary non-road engines and engine generator units.

II. THEORETICAL METHOD OF DETERMINING BIOGAS POTENTIAL

The theoretical or stoichiometric production of methane and carbon dioxide in anaerobic digestion can be calculated according to Buswell's formula [14]. In this case, the elementary composition of the substrate must be known, and the production of methane and carbon dioxide can be calculated by using the following formula:

$$C_aH_bO_c + \left(a - \frac{b}{4} - \frac{c}{2}\right) H_2O \rightarrow \left(\frac{a}{2} + \frac{b}{8} - \frac{c}{4}\right) CH_4 + \left(\frac{a}{2} - \frac{b}{8} + \frac{c}{4}\right) CO_2$$

This calculation is performed with the assumption that all energy goes to the production of methane and that the energy consumed for the growth of the microorganism can be neglected.

TABLE III
THEORETICAL METHANE POTENTIAL ACCORDING TO BUSWELL'S FORMULA

	Chemical Formula	Units (NmL_{CH4}/g_{VS})
Carbohydrates	$(C_6H_{10}O_5)_n$	415
Fats	$C_{57}H_{104}O_6$	1014
Proteins	$C_5H_7NO_2$	496

Furthermore, if the elemental composition of the substrate is unknown, a component analysis can be used to predict the methane potential of a substrate. This presumes that the concentration of carbohydrates, proteins, and fats are known. In Table II, a theoretical composition estimation of the above-mentioned components can be found, as well as the calculated theoretical yield from the respective groups according to Buswell's formula [5].

III. METHODOLOGY

A. Biogas Digester Set-up

The laboratory setup consisted of 8 reactors, each with approximate working volumes of 6 L. Digesters was developed in the Texas A&M University Bio-Energy Testing and Analysis Laboratory. The digesters were constructed of clear PVC pipe with an inside diameter of 15.2 cm (6 in.) and with lengths of 30.5 cm (1 ft). Clear PVC allowed for visual investigation of the digesters while the experiment was being conducted. A properly sized 15.2 cm PVC cap was used to seal the reactor on the bottom. A threaded PVC fitting was used on the top of each reactor, with a 15.2 cm threaded PVC plug sealing the reactor. PVC cement was used to seal all fitting on the reactor, not including the top threading, allowing for opening. A sealing compound putty was used in the threaded connection to prevent gas leaks from the pressure inside the reactors.

The digester caps were drilled to allow for outlets. The bottom of each reactor consisted of a 0.635 cm valve to allow for liquid samples to be taken. The top was drilled for two outlets; one consisted of a 0.635 cm valve to allow for sample feeding, while a second outlet used a 0.635 tube connection to transfer gas into the connected gas collector. All fittings were sealed using a thread sealant tape to prevent leakage.

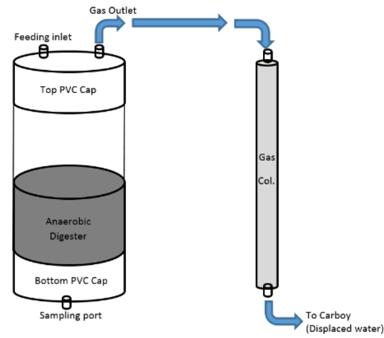

Fig. 1a Digester and Collector set-up

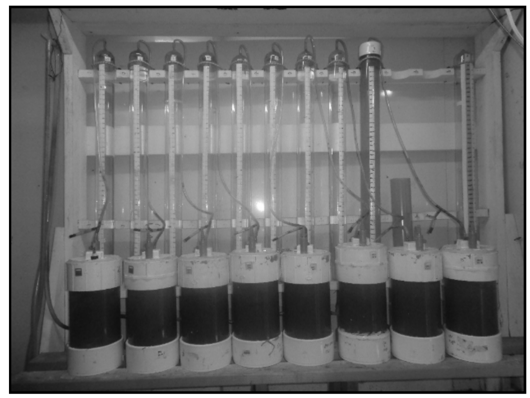

Fig. 2b Reactors inside environmental chamber.

Gas collectors were primarily built as glass containers used in previous anaerobic digestion studies; some collectors had to be replaced with constructed PVC pipe collectors. All collectors were measured to have an internal diameter of 7.6 cm (3 in.) and 122 cm (48 in.) long. Tube connections on both ends were measured to be 0.635 cm (0.25in) in diameter on the top of the collectors, and 1.27 cm (0.5 in.) on the bottom. The PVC gas collectors used fitted caps, drilled to allow for tube connections of similar sizes to the glass collectors, and sealed with PVC cement.

Gas collectors were connected in parallel using 1.27 cm plastic tubing along the bottom connections. The main line was connected to two carboys in the experiment: an overhead refilling carboy and an overflow carboy below the collectors.

The refilling carboy was used to fill the collectors with water initially to a zero-level. When the digesters were sealed from the atmosphere and the overflow carboy opened, any biogas produced displaced water into the overflow carboy, allowing for gas measurements. After several days, when the water levels of the collectors neared empty, gas samples were taken to release pressure as the overhead carboy refilled the collectors to repeat the process.

The experiment was conducted in an environmental chamber to allow for control of atmospheric conditions. Humidity and pressure remained constant throughout the experiment, however the heating element in the chamber was non-functional. A space heater was used to maintain a temperature of 35° C in the room.

B. Digestion Trials

Two batch trials were performed to investigate the effects of different treatments. For each batch trial, reactors were filled with cattle manure and various combinations of biomass like sewage sludge, rice straw and coconut shell. Control reactors containing only cattle manure and tap water were maintained during each batch trial. Feed compositions used for the batch trials are given in Table III. Each reactor had a total working volume of approximately 4.8 L, and duplicate reactors were used for each feed composition. The trials were performed in an environmental chamber maintained at 35°C. The digesters were mixed daily by turning them upside-down and shaking for about 20 seconds

As shown in Table III, the co-digestion of cattle manure and coconut shell (CM + CS) shows the greatest amount of carbon-to-nitrogen ratio at 36:1 and the co-digestion of cow manure and sewage sludge is the least carbon-to-nitrogen ratio at 25.9:1. A higher volume of feed and higher organic load were expected to require longer digestion times for the said trials. The duration of the batch trials was 30 days. The digestion trials ended when biogas production ceased.

TABLE IIIII
EXPERIMENTAL CONDITIONS IN ANAEROBIC CO-DIGESTION EXPERIMENTS

Treatment	Digestion Type	Treatment Mixture	C/N	OLR
1	Mono-digestion	Cow Manure	29.6	0.5
2	Co-digestion	Cow Manure + Sewage Sludge	25.9	0.5
3	Co-digestion	Cow Manure + Rice Straw	30.0	0.6
4	Co-digestion	Cow Manure + Coconut Shell	36.0	0.6

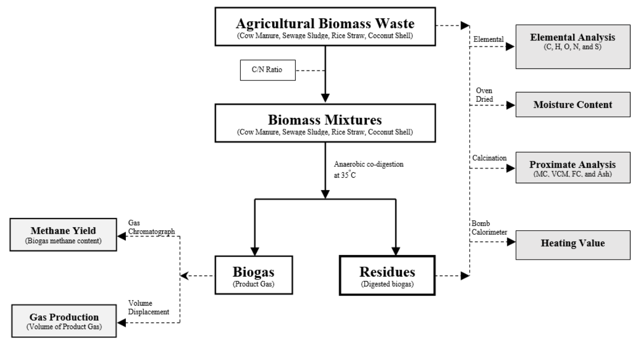

Fig. 2. Schematic diagram of the anaerobic co-digestion experimental procedure conducted in this study

C. Analysis Techniques

Analysis of the biomass utilized in the experiment consisted of two types: proximate and ultimate analysis. Proximate analysis yield results giving the volatile solids content, combustible matter, and ash content of the organic compounds used, while ultimate analysis gives the carbon, hydrogen, nitrogen, and sulfur contents as a percentage of dry matter.

The proximate analysis was conducted in several parts. Firstly, biomass samples are dried in a heating oven at 105° C for at least 12 hours, to ensure minimal moisture. The comparison of the weight before and after this process yields the moisture content of the raw sample. Dried samples were then measured in 1-g samples in metal crucibles and capped. They were first placed in a muffle furnace at 950° C purged with nitrogen gas for 15 minutes. This results in the biomass vaporizing, but not undergoing full combustion. The weight difference yields the volatile combustible matter. The remaining samples were then placed in a muffle furnace uncapped at 550° C for 4 hours. This resulted in complete conversion of volatile matter, and gave results of total volatile solids as a percentage of the original dry matter, as well as the ash content when the remaining residue was measured.

Ultimate analysis was conducted utilizing a VarioMICRO Cube Elemental Analyzer. The machine uses a combustion tube and reduction tube to combust 2mg samples under a gaseous environment of oxygen and helium, and is calibrated to give calculations for the percentage content of carbon, nitrogen, oxygen, and sulfur. The machine is first run with several blanks to ensure a zero-starting point. Sulfuric acid samples are then used as a standard and compared to normal content values. When the samples meet the standard requirements, digester samples are run.

Initial biogas measurements are taken as the full amount of gas produced within the collectors. Samples are taken to measure the exact nature of the gas, primarily the amount of methane found. Gas samples were analyzed with a model 8610C gas chromatograph (SRI Instruments, Torrance, Cal., USA) to determine O_2, N_2, CH_4, and CO_2 concentrations. The columns used were a 1.8-m (6-ft) silica gel packed column and a 1.8-m molecular sieve 13X packed column, both supplied and installed by the GC manufacturer. The temperature of the GC oven was held at 37°C for the first 4 minutes after injecting the sample and then ramped up from 37°C to 220°C at a rate of 20°C min–1; the run was complete after 15 minutes. The carrier gas used was helium with a flow rate of 20 mL min–1 at 27 psi. A thermal conductivity detector was used.

The GC was calibrated using various standard gas mixtures containing O_2, N_2, CH_4, and CO_2. The standard mixtures had an accuracy of 2%. The calibration curve for each gas had three points except CO_2 which had only two points. The GC was calibrated approximately twice monthly using the GC manufacturer's software, and low variation was observed in peak areas for the calibrated gases.

pH was analyzed using an ion meter calibrated with samples measuring at pH 4,7 and 10. Samples were mixed with a magnetic stirrer when measured, and replaced after measuring. When pH required balancing, a 5M NaOH solution was prepared using a mixture of solid sodium hydroxide pellets and deionized water.

IV. RESULTS AND DISCUSSIONS

D. Characterization of Biomass Feedstock

The anaerobic co-digestion experiment used mixtures from different biomass such as Cow Manure, Sewage Sludge. The experimental design was completely randomized to attain a carbon-to-nitrogen (C/N) ratio of the mixture close to 30 (Table IV). A control run was done using cow manure as sole substrate. All the experiments were performed in duplicates using methane production and biogas yield as response variables.

TABLE IVV
EXPERIMENTAL CONDITIONS AND RESULTS IN BATCH TEST

Treatment	Digestion Type	Treatment Mixture	C/N	OLR	Biogas Production	Methane Content	Total Methane
					L	%	L
1	Mono-digestion	CM	29.6	0.5	4.5	28.2	1.0
2	Mono-digestion	CM	29.6	0.3	3.2	21.7	0.5
3	Co-digestion	CM + SS	25.9	0.5	2.9	60.9	1.1
4	Co-digestion	CM + SS	25.9	0.5	5.0	60.3	2.0
5	Co-digestion	CM + RS	30.0	0.6	3.0	21.0	0.4
6	Co-digestion	CM + RS	30.0	0.6	1.8	12.7	0.2
7	Co-digestion	CM + CS	36.0	0.6	1.9	13.5	0.2
8	Co-digestion	CM + CS	36.0	0.6	1.5	15.0	0.2

Table V summarizes the characterizations of the cow manure, sewage sludge, rice straw and coconut shell biomass wastes. Based on the characterization results, the coconut shell and rice straw potentially be used as a substrate for the biogas production since it has lower nitrogen content (0.9% and 0.1%), which is a factor to consider in order to avoid ammonia inhibition of the digestion process.

TABLE V
CHARACTERISTICS OF THE BIOMASS MATERIALS USED IN THE ANAEROBIC DIGESTION EXPERIMENTS CARRIED OUT IN THE STUDY

Property	Cattle Manure	Sewage Sludge	Rice Straw	Coconut Shell
Moisture (% fresh matter)	86.7 (0.22)	85.8 (0.18)	7.9 (0.69)	14.3 (0.60)
TS (% fresh matter)	13.2 (0.25)	13.9 (0.08)	92.9 (0.67)	84.6 (0.89)
VS (%TS)	9.0	6.9	92.3	84.6
Ash (%TS)	4.2	6.9	0.6	0.1
TKN (%TS)	7.8	34.9	5.7	0.9
pH	6.1	7.4	6.8	6.0
C (%TS)	37.0	32.3	32.9	54.1
H (%TS)	4.9	4.5	4.7	6.3
O (%TS)	56.7	57.3	61.4	39.5
N (%TS)	1.2	5.6	0.9	0.1
S (%TS)	0.1	0.3	0.1	0.0
C:N Ratio	29.6	5.8	36.1	380.9
Heating Value (MJ/kg)	17.0	15.0	14.4	19.3

The analysis shows the rice straw was oven-dried, and the total solids content was 92.9%, followed by coconut shell (84.6%), sewage sludge (13.9%), and cattle manure (13.2%) having the less total solids. The feedstock in this study had a wide range of TS and VS content, which were likely a result of the chemical and biological structure of the feedstocks which correlates to the digestibility of the biomass material. Similarly, high volatile combustible solids content (VS) was found in a coconut shell (92.30%) and followed by rice straw (84.60%), and the volatile solids found in cattle manure (9.0%), and sewage sludge (6.9%) is significantly lower, demonstrating the spent microbial activity from the original process. Cattle manure (86.70%), and sewage sludge (85.80%), showed the disparity of moisture contents, with coconut shell (14.3%), and rice straw (7.9%) for having a low content level. Further, the coconut had high carbon content and low nitrogen content and the C/N ratio was 380.9, which was much higher than the optimal range of 25-30 [6]. The biomass characterization results presented in this study were closely similar to those in other reports [7] – [8].

E. Biogas Production

Figure 3 shown is an average cumulative gas production curve for the 4 groups of digestion trials with 2 replicates based on treatments. Gas production was compared based on the different levels of balanced on carbon-to-nitrogen ratios and plotted for gas production. In all systems, the cumulative gas production treatments of cattle manure with sewage sludge producing the most methane and the cow manure with coconut shell mixture producing the least. This shows evidence that sewage sludge helps to increase the biogas production due to its organic solids.

The four treatments were internally compared at the varying carbon-to-nitrogen ratios and plotted for gas production. This gives results on the carbon balancing potentially having an impact on cumulative gas production. Results from the experiment show that for the cattle manure and sewage sludge mixtures, balancing at a ratio of 25:1 results in highest potential biogas production.

This suggests that the anaerobic co-digestion of cow manure and sewage sludge produces biogas more efficiently than mono-digestion of cow manure for the methane production process, and is most probably due to the better degradation and utilization of organics in the wastes [9].

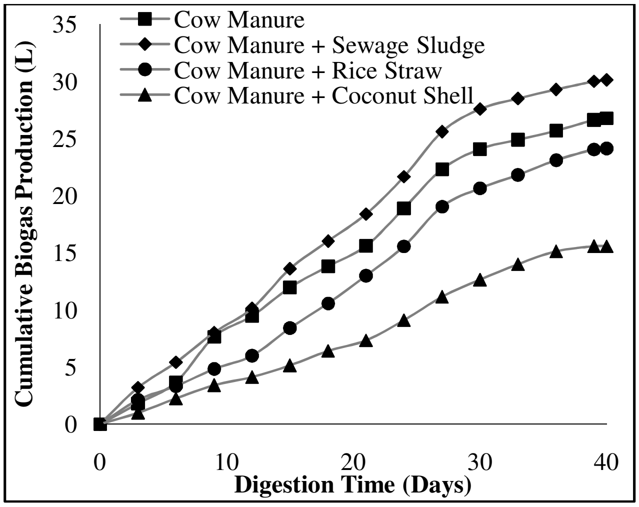

Fig. 3. Variation of biogas production during the anaerobic co-digestion process: cumulative biogas production

F. Biogas Composition and Methane Yield

Gas samples were collected and analyzed, primarily to check methane and carbon dioxide content. As some collectors contained less biogas than others when samples were collected, biogas concentration was lower by default. To account for this, a CH_4:CO_2 ratio was made for all samples. In biogas, a theoretical ratio of methane to carbon dioxide is approximately 1.5:1. Tables IV show the average compositional analysis for the gases detected in the chromatograph.

Oxygen was detected in the gas chromatograph system, contradicting the presence of methane found from anaerobic digestion. Samples taken in the Tedlar bags may have provided a leakage possibility, and coupled with potential air pockets in the digesters during refilling, oxygen contamination would have occurred during analysis. Values were normalized to accommodate for the desired oxygen-less environment, and standard values are shown in table IV.

The highest methane concentration was overall found in the cattle manure and sewage sludge mixture balanced at 25:1 carbon-to-nitrogen ratio. The concentration ratios, however, are too far with the other mixtures (coconut shell, rice straw). With the exception of the control mixtures, carbon balancing shows in increase in methane concentration with respect to carbon dioxide.

This may be due to the smaller samples taken from the control mixtures due to less biogas produced from these reactors overall. Likewise, reactors containing the mixtures of cattle manure and sewage sludge showed a short startup time to produce methane in comparison to the other digesters.

Also, the co-digestion of cow manure with agricultural waste (rice straw and coconut shell) was less stable compared with the co-digestion of cow manure with sewage sludge. This disparity could be observed due to the poor homogeneity of the mixture [10] and consequently, more variations were observed in the daily biogas production.

Because biogas produced by AD consists only of CH_4 and CO_2 along with trace amounts of H2S and other gases that were not measured, the composition of the biogas being produced by microbial activity was calculated assuming it consisted of only the CH_4 and CO_2 contents measured by GC.

The fraction of CH_4 in the biogas for several reactors during the test is shown in Figure 4 and 5.

Cumulative CH_4 production for each of the batch trials is shown in Figures 3 through 5. To calculate cumulative volumetric methane production, it was assumed that the volume of biogas that accumulated in the gas collectors between readings was composed only of CH_4 and CO_2 as indicated above. The trends for cumulative CH_4 production were similar to those for biogas production shown in Figures 3.

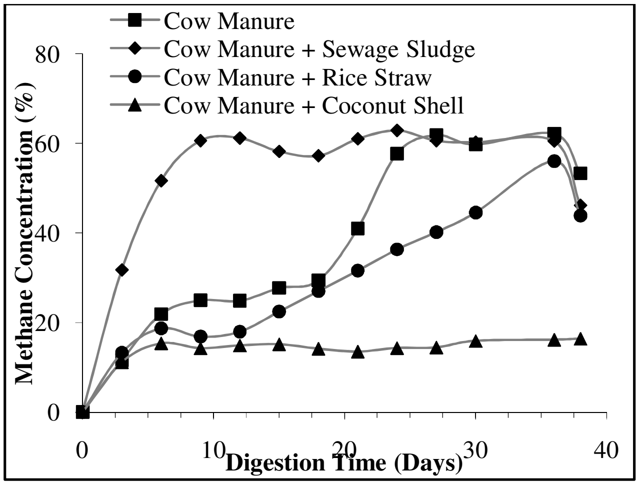

Fig. 4. Variation of biogas production during the anaerobic co-digestion process: methane content

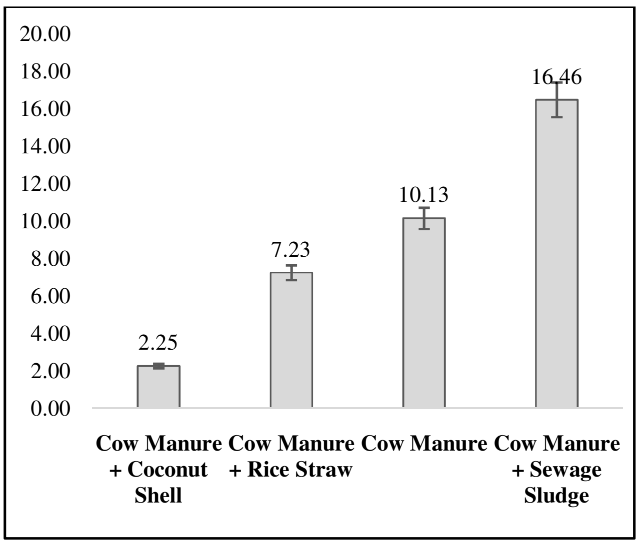

Fig. 5. Variation of biogas production during the anaerobic co-digestion process: cumulative methane production. [Y-axis is the methane production, in liters (not shown)]

G. Methane production on various biomass treatment

The daily methane production rate and methane yield of Cattle Manure (CM), CM + Sewage Sludge (CM+SS), CM + Rice Straw (CM+RS) and CM + Coconut shell (CM+CS) are shown in Fig. 4.4. For CM+SS, only one peak of the daily methane yield was observed, which occurred on day 4 (1,180 mL). For CM, two peaks of the daily methane yield were observed, which occurred on day 10 and day 24 (820 mL) (Fig. 4.4). The occurrence of the peaks of the daily methane yield of CM+SS may be due to the higher soluble COD content [11], which could be rapidly biodegraded in the first few days. Also, it was pointed that the degradation rate of crude protein is lower than that of carbohydrates. For the CM, the peak of the daily methane yield would be attributed to the subsequent degradation of crude protein and macromolecular substances, such as lignocelluloses.

At the end of the 30-days digestion, the cumulative methane yield obtained from CM+SS (16.22 L) was about 1.5 times higher than that from CM (9.62 L). CM+SS has been demonstrated in many cases as an excellent substrate for anaerobic digestion. The methane yield of rice straw and coconut shell in this experiment was relatively lower than the methane yield (6.74L, 2.18L), but Gu, et.al (2014) presented a similar methane yield.

However, CM+RS and CM+CS is slightly poorly biodegradable because of its special cell structure, which limited the hydrolysis of organic matter [12]. The lower methane production may have resulted from lignin structure which was shown to be an inhibiting factor for methane fermentation.

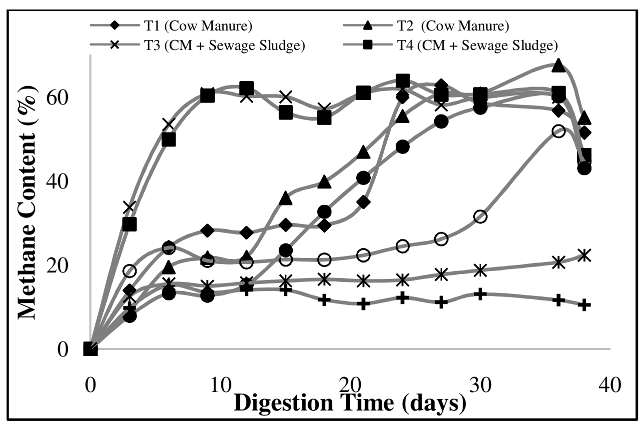

Fig. 6. Methane yields for the individual biomass treatments

V. CONCLUSIONS

This research focuses on the possible means of increasing methane production potential from anaerobic co-digestion of agricultural biomass waste.

The results and findings of the conducted research are summarized as follows:

1. Results of biomass treatment strategies as well as carbon balancing show to have a positive influence on digestion. The cumulative methane production for the co-digestion mixtures were 33.12, 54.68, 24.17 and 14.03 L for cow manure (control), cow manure + sewage sludge (CM+SS), cow manure + rice straw (CM+RS) and cow manure + coconut shell (CM+CS), with carbon balancing at ratios for carbon-to-nitrogen of 30:1, 26:1, and 30:1, 36:1 respectively.
2. Anaerobic co-digestion of the different ratios of biomass feedstock is promising and generated a significant amount of biogas (methane gas).

ACKNOWLEDGMENT

The authors would like to thank Bio-Energy Testing and Analysis Laboratory of Texas A&M University, College Station, Texas, USA for offering me the opportunity to conduct this research. Also, the Engineering Research and Development for Technology (ERDT) under the Science Education Institute (SEI) of Department of Science and Technology in the Philippines and the College of Engineering, MSU-IIT for their financial support to this project.

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