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ANAEROBIC DIGESTION OF COW MANURE: INFLUENCE OF HYDRAULIC RETENTION TIME ON BIOGAS YIELD AND PROPERTIES OF DIGESTATE

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Abstract

This research paper assessed the influence of hydraulic retention (HRT) time on biogas yield and the properties of digestate during anaerobic digestion. Anaerobic digestion of cow manure was simultaneously digested in a custom built 20L digester at different HRT's of 10, 18, 28, 38, 50 and 60day. The study showed HRT had substantial influence on biogas yield and on the properties of digestate. An average biogas yield of 1.6m^3/KgVS$_{added}$ to 3m^3/KgVS$_{added}$ was obtained. The elemental content of carbon, hydrogen, and nitrogen of the resultant digestate reduced from 35.6w/w% to 19.9w/w%, 6.3w/w% to 2.8w/w%, and 2.1w/w% to 0.8w/w%, respectively, after a 60day HRT. Consequently, the higher heating values reduced from 15.1MJ/kg to 3.5MJ/kg. Also, the carbon-to nitrogen ratio was between 17.9 and 24.8 when compared to that of cow manure, while the hydrogen-to carbon ratio was in the range of 1.4 to 1.8. The digestate could be used as a fertilizer, alternatively as a precursor for biofuel production.

Keywords: Anaerobic digestion, Biogas, Digestate quality, Hydraulic retention time

INTRODUCTION

The production of biogas through anaerobic digestion (AD) of organic waste has continued to attract interest in recent years. This could increase the use of renewable fuels and simultaneously increase several countries renewables portfolio. It could also provide local economic stability and environmental benefits, thus creating jobs, reducing air, water pollution and greenhouse gas GHG emissions (Kuo and Dow, 2017).

AD is a multistep biological process for converting organic waste such as sewage, agricultural waste, municipal waste, animal manure, industrial effluent to useful valuable end products (Angelidaki *et al*., 2009; Alvarez *et al*., 2000; Zinganshin *et al*., 2012). AD produces biogas containing ~50% to 70% methane, 25% to 50% carbon dioxide, trace amounts of hydrogen sulphide, and a semi-solid effluent, generally referred to as digestate (Alburquerque *et al*., 2012). The resultant digestate contains partially degraded organic/inorgamic matter and microorganisms such as *Aspergillus* sp., *Basillus* sp., *Pseudomonas* sp., *Samonella* sp. and *klebsiella* sp. Importantly, AD is a low cost process and the gas produced could be used to generate energy; heat, electricity and liquid transportation fuels (Li *et al*., 2019).

The use of biogas technology would help to achieve the Millennium Development Goals (MDG) of United Nations, which aim to eradicate poverty and hunger (Alfa *et al*., 2014). Although, large-scale application of conventional chemical fertilizers improves farm produce, it negatively affect soil quality, leads to eutrophication, and environmental degradation (Owamah *et al*., 2014). In contrast, the use of digestate do not only reduces land pollution but also the digestate contains microorganisms and with higher proportion of available macronutrient for plant growth and development compared to the untreated organic waste (Alfa *et al*., 2014; Risberg *et al*., 2017). Such approach would improve socio-economic human health and environment of communities, especially in the Sub-Saharan Africa. Moreover, AD is an important technology to meet 20% of European Commission energy consumption from renewable sources by the year 2020 (EREC, 2008; Perez-Camacho *et al*., 2019). Although, AD is a promising technology, it has not yet been widely utilized (Xu *et al*., 2018).

A review of scientific literature showed that the hydraulic retention time (HRT) could influence the amount of biogas yield and digestate quality (Azzahrani *et al*., 2018; Baati *et al*., 2018; Shi *et al*., 2017). These aforementioned reports investigated effects of HRT's on AD separately for 25, 30, 40, 60 and 150 HRTs using different feedstocks at varying reaction temperatures of 37^{o}C, 45^{o}C, 55^{o}C and 60^{o}C. However, these studies were performed using semi continuous anaerobic digestion process. In addition, the properties of digestate were not reported. Although, Alburquerque *et al*., (2012) and Garfi *et al*., (2014) assessed the quality and potential of digestate as biofertilizers, the effect of HRT on biogas yield and digestate were not reported. Moreover, Alfa *et al*., (2014) and Shi *et al*., (2017) suggested that further research investigations in increasing HRTs for AD is necessary, in order to provide more information on digestate properties. Therefore, the aim of this study is to elucidate the influence of HRT on biogas yield and on the quality of the digestate.

MATERIALS AND METHOD

Cow manure was used as substrate in the present study. The manure was collected from a local livestock farm at Thandalam, Tamil Nadu, India. The AD experimental studies were conducted using six (6) 20L batch digesters, denoted as Digester A, Digester B, Digester C, Digester D, Digester E and Digester F.

Each digester (Digester A to Digester F) was loaded with 12.4kg of manure slurry containing 8%w/w dry solids and 400ml of inoculum obtained from manure based biogas plant at the pilot plant of Aban Infrastructure Pvt, Biotechnology division, Chennai, India. AD operates efficiently with solids <10% (Lee *et al*., 2019). Then the digester was sealed with a rubber cork, equipped with control valve and tubing to facilitate gas collection. Thereafter, the digesters were allowed to stand for up to 60 day HRT under room temperature. Typically, Digester A was operated for a 10day HRT, 18day HRT for Digester B, and 28day HRT for Digester C, while Digester D, Digester E and Digester F were operated for 38day HRT, 50day HRT and 60day HRT, respectively. During experimentation, the digesters were agitated for 2min every day, in order to improve the contact between substrates and bacteria. This would also prevent accumulation of substrates and fatty acids. The amount of biogas produced was estimated by water displacement method, and readings were collected in interval of 2days. After completion of HRT for each digester, the digestate (effluent) was dewatered for further analysis. A schematic view of the anaerobic digestion experimental setup is shown in Fig. 1.

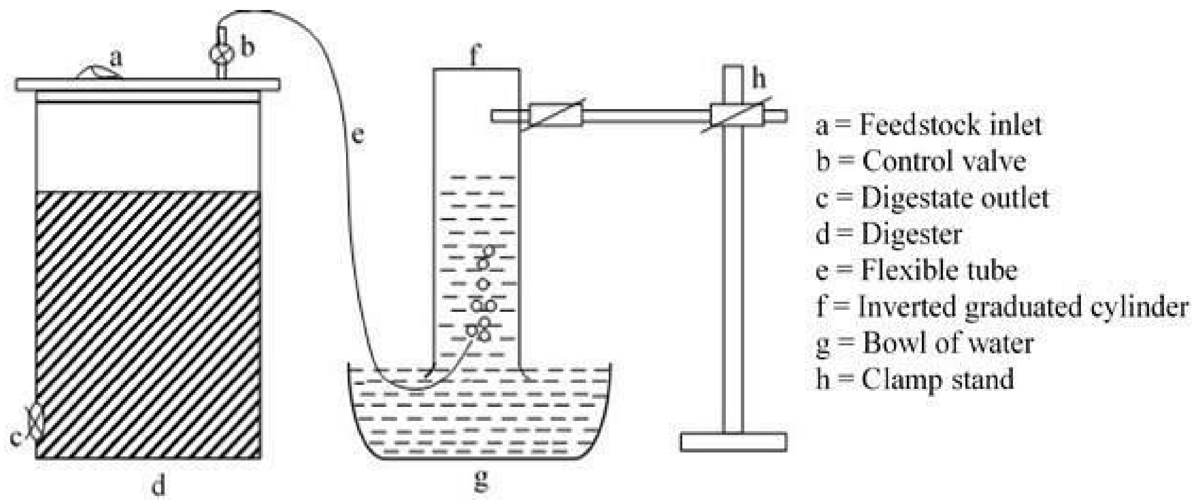

Fig. 1: Schematic view of anaerobic digestion experimental setup.

Analysis

The composition of elemental carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) of the cow manure and digestates were determined using VarioEL III elemental analyser system GmbH in accordance to the ASTM D-5291 and D-3176 methods. The elemental oxygen (O) content was determined by subtraction from combined mass of C, H, N and S. The higher heating value (HHV, MJ/kg) was determined using a unified correlation equation (Eq. 1) proposed by Channiwala and Parikh, (2002).

$$HHV\left(\frac{MJ}{kg}\right) = 0.3491C + 1.1783H + 0.1005S - 0.1034O - 0.0151N - 0.0211A \quad (1)$$

where C is the mass of carbon, H the mass of hydrogen, and N mass of nitrogen. S, O and A are the respective mass of sulfur, and oxygen, while A is ash on a dry weight basis.

The ratios of hydrogen-to-carbon, oxygen-to-carbon and nitrogen-to-carbon ratio were determined according to Eq. (2), Eq. (3) and Eq. (4), respectively (Eboibi *et al*., 2019). The atomic carbon-to-nitrogen ratio was estimated by division of the percentage weight of C and N obtained from the elemental analysis.

$$H/C = \frac{H(\frac{W}{W}\%)\ x\ MW_C}{C(\frac{W}{W}\%)\ x\ MW_H} \quad (2)$$

$$O/C = \frac{O(\frac{W}{W}\%)\ x\ MW_C}{C(\frac{W}{W}\%)\ x\ MW_O} \quad (3)$$

$$N/C = \frac{N(\frac{W}{W}\%)\ x\ MW_C}{C(\frac{W}{W}\%)\ x\ MW_N} \quad (4)$$

where $H(\frac{w}{w}\%)$ represents weight percentage of hydrogen, $C\left(\frac{w}{w}\%\right)$ weight percentage of carbon, and $N(\frac{w}{w}\%)$ is the weight of nitrogen. MW_C , MW_H and MW_N is the molecular weight of carbon, hydrogen and nitrogen, respectively.

Metallic composition substrate and digestates were obtained using Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), Agilent 7500 series. Samples for analysis were prepared in accordance to method explained by Eboibi *et al*., (2014). The pH was analysed using a laboratory pH meter directly without dilution.

RESULTS AND DISCUSSION

Biogas yield

The biogas yields from anaerobic digestion of cow manure from all the digesters are presented in Fig. 2 to Fig. 7. Generally, three days lag phase was observed for all the digesters. The biogas yield from Digester A is presented in Fig. 2. As illustrated in Fig. 2, there were no readings for day 1 to day 3, which was referred to as lag phase as mentioned previously. There were gradual increase in biogas yield from $0.1m^3/KgVS_{added}$ for 4day HRT to $2.2m^3/KgVS_{added}$ for 8day HRT. This data is relatively within an average biogas yield of 0.6L/d to 0.9 L/d reported by Haryanto *et al*., (2018).

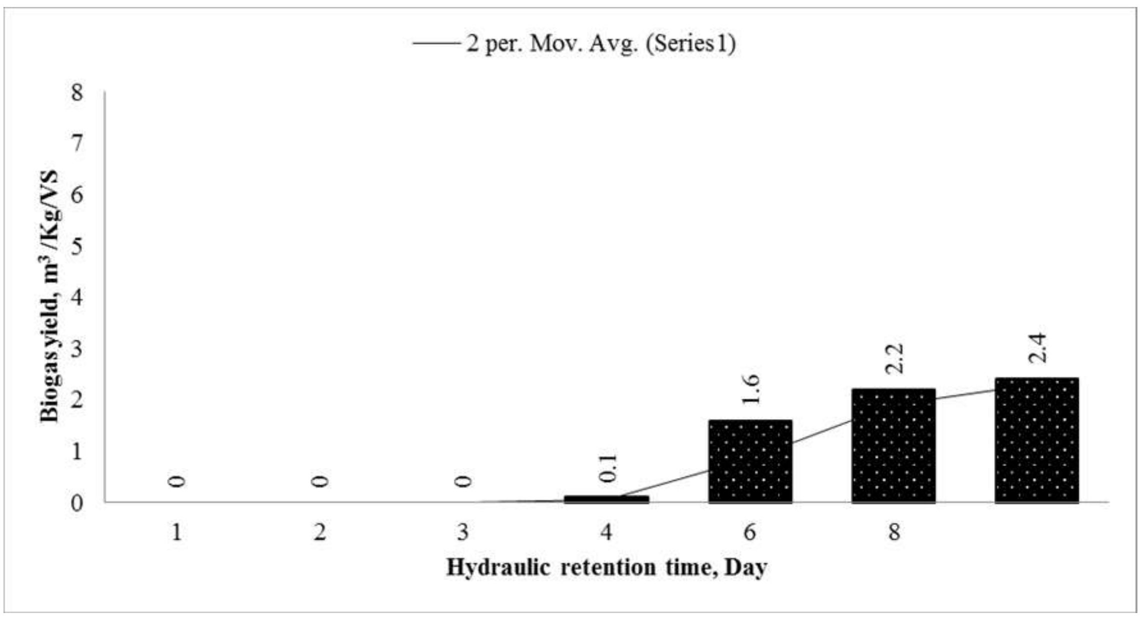

Fig. 2: Biogas yield from Digester A during anaerobic digestion of cow manure

Fig. 3 shows the biogas yield for Digester B. As shown in Fig. 3, there was an increase in biogas yield up to 8day HRT, which was followed by decrease in 10day HRT. Then an increase in biogas yield of $2.2m^3/KgVS_{added}$ from 12 day HRT to $7.6m^3/KgVS_{added}$ for 16day HRT. It was found that the trend in biogas production for Digester B differed to that of Digester A. This is because Digester B had longer HRT of 16day, though the trend were similar between 4day HRT and 8day HRT. Despite being in different digesters the substrate could have undergone similar degradation. Also, it could be confirmed the substrates possess easily biodegradables, hence were easily digested initially. However, microbial activities could differ hence the variation in biogas yields, as shown in Fig. 3.

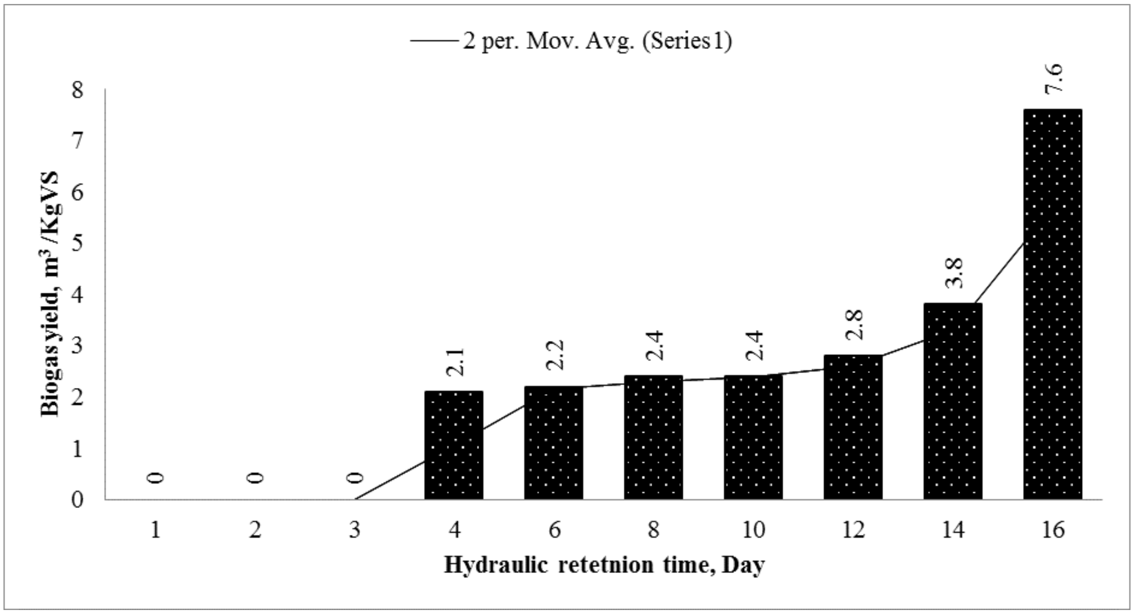

Fig. 3: Biogas yield from Digester B during anaerobic digestion of cow manure

The decrease in biogas yield at 10day HRT could be due to a number of reasons. Firstly, it could be due to inactivity of microbes, and secondly could be due to exhaustion of easily biodegradables of the substrate. Due to the exhaustion, the microorganisms require time to feed on the substrates, hence the lower biogas yield. The increase in biogas yield from 10day HRT to 16day HRT could be referred to as the exponential phase. Previous reports (Hansen *et al*., 1998; Otero *et al*., 2011) have shown that the exponential phase led to maximum yield in biogas. In this present study, maximum biogas yield of $7.6m^3/KgVS_{added}$ was obtained at the exponential phase (16day HRT), which is similar to $7.2m^3/KgVS_{added}$ average biogas yield reported by Harryanto *et al*., (2018).

The yield in biogas obtained from Digester C is presented in Fig. 4. As shown is Fig. 4, the AD of the substrate was monitored for 28day HRT. Maximum biogas yield of $7.3m^3/KgVS_{added}$ was obtained at 16day HRT (similar to Digester B), minimum of 1.9 $m^3/KgVS_{added}$ at 10day HRT. The maximum yield at 16day HRT could be due to: suitable condition for substrate utilisation, perhaps temperature fluctuations which might have led to increase digestion of substrates. Though, the experiment was conducted at room temperature, changes in weather condition led to increase in temperature. The sharp increase in biogas yield at 16day HRT was also observed for Digester C (Fig. 4) and D (Fig. 5) . Although all digesters were wrapped with black polythene, Digesters B, C and D were closer to widow, hence sunlight rays could have influenced the digestion. The trend in biogas production was found to be similar to that of Digester B, with an initial increase from 4day HRT to 8day HRT. Then a decrease in biogas production in 10day HRT, followed with an increase in production. The phases of lag, exponential, stationary and decline for growth and death of microorganism was found to have occurred in Digester C, as shown in Fig. 4. This finding suggests that a 30day HRT could be enough for AD of manure.

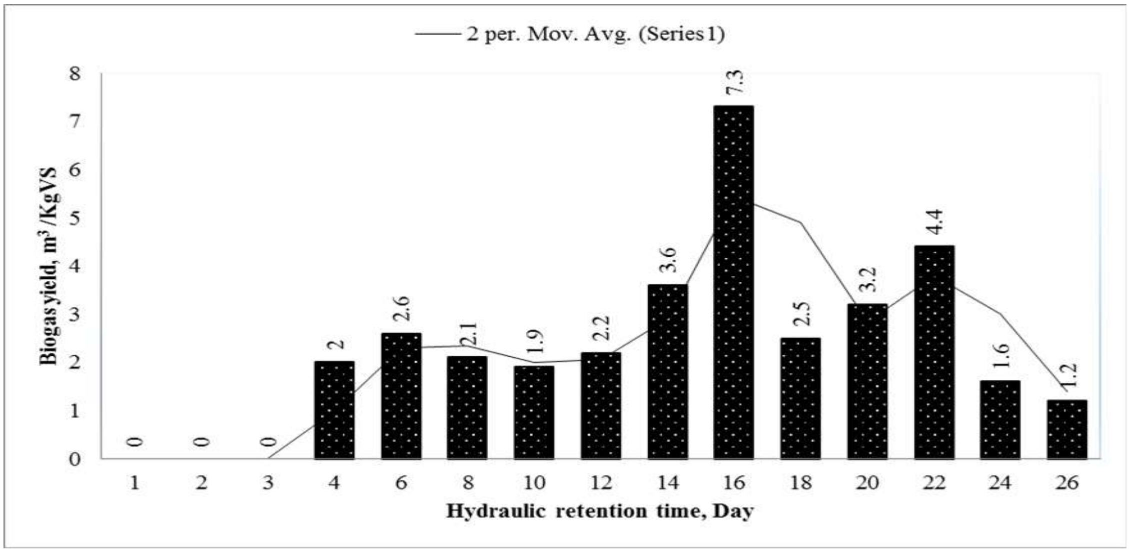

Fig. 4: Biogas yield from Digester C during anaerobic digestion of cow manure

The biogas yield obtained from Digester D, Digester E and Digester F during digestion of the substrates is presented in Fig. 5, Fig.6 and Fig. 7, respectively. As shown in Fig. 5, biogas production from Digester D, was of similar trend with previous digesters (C and B). Again, a maximum biogas yield ($5.8m^3/KgVS_{added}$) was obtained at 16 day HRT, though lower when compared to $7.3m^3/KgVS_{added}$ and $7.6\ m^3/KgVS_{added}$ for Digester C and Digester B, respectively. Interestingly, the decrease in biogas yield at 18day HRT for Digester D also occurred at similar day for Digester C and Digester E, except Digester F. This finding suggests the digesters operated under similar operating conditions.

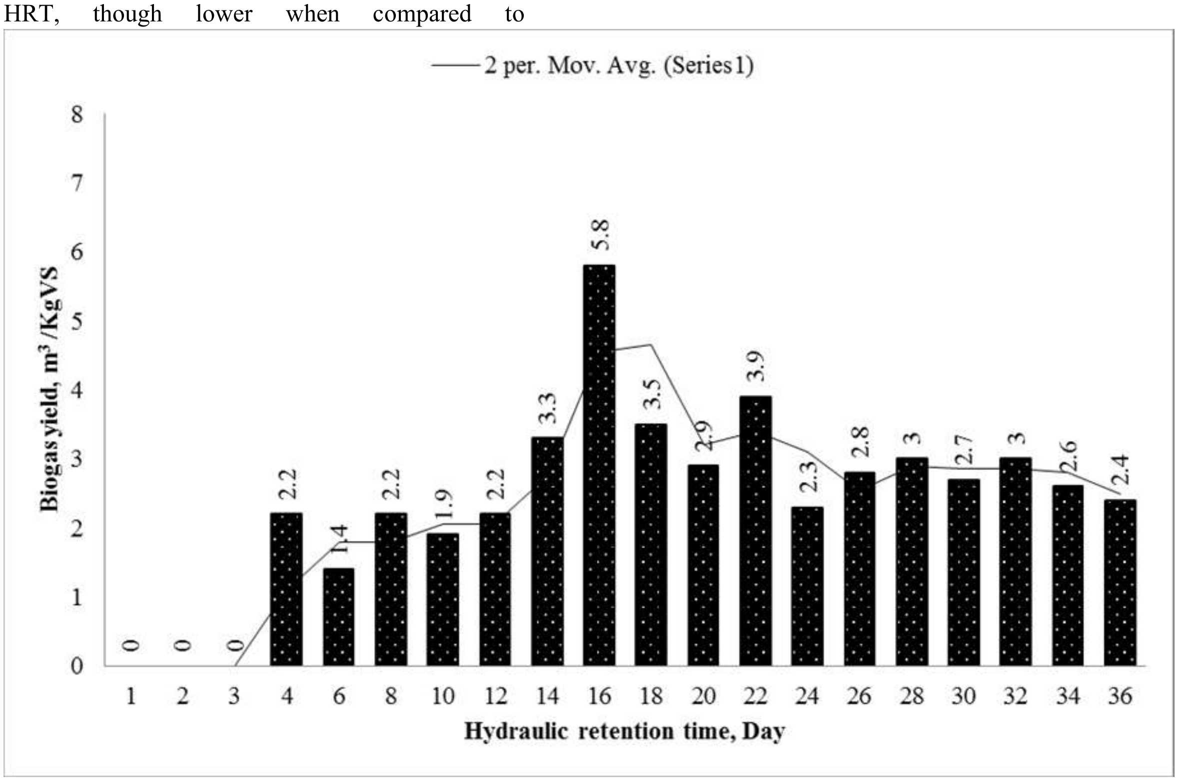

Fig.5: Biogas yield from Digester D during anaerobic digestion of cow manure.

For Digester E, a maximum biogas yield of 7.6m^3/KgVS$_{added}$ was obtained at 12day HRT, while minimum of 0.4 m^3/KgVS$_{added}$ was achieved at 20day HRT. These values were found to be similar to the data obtained for previous digesters. Based on the data presented in Fig. 6, it is clear that microbes underwent different growth phase, leading to variation in biogas yields.

Comparison of Digester E (Fig. 6) with Digester D (Fig.5), showed that both digesters had similar biogas yield between 4day HRT and 36day HRT. Digester E had biogas yield of ~50m^3/KgVS$_{added}$ with an average yield/day of about 2 m^3/KgVS$_{added}$, while Digester D achieved ~49m^3/KgVS$_{added}$ and 2.46m^3/KgVS$_{added}$ for biogas yield and an average yield/day, respectively. This finding suggests that the degradation of substrates by microbes in the different digesters could be similar. As shown in Fig. 6, there was a decrease in biogas yield between 14day HRT and 34day HRT, however, this was followed with gradual increase in yields from 40day HRT.

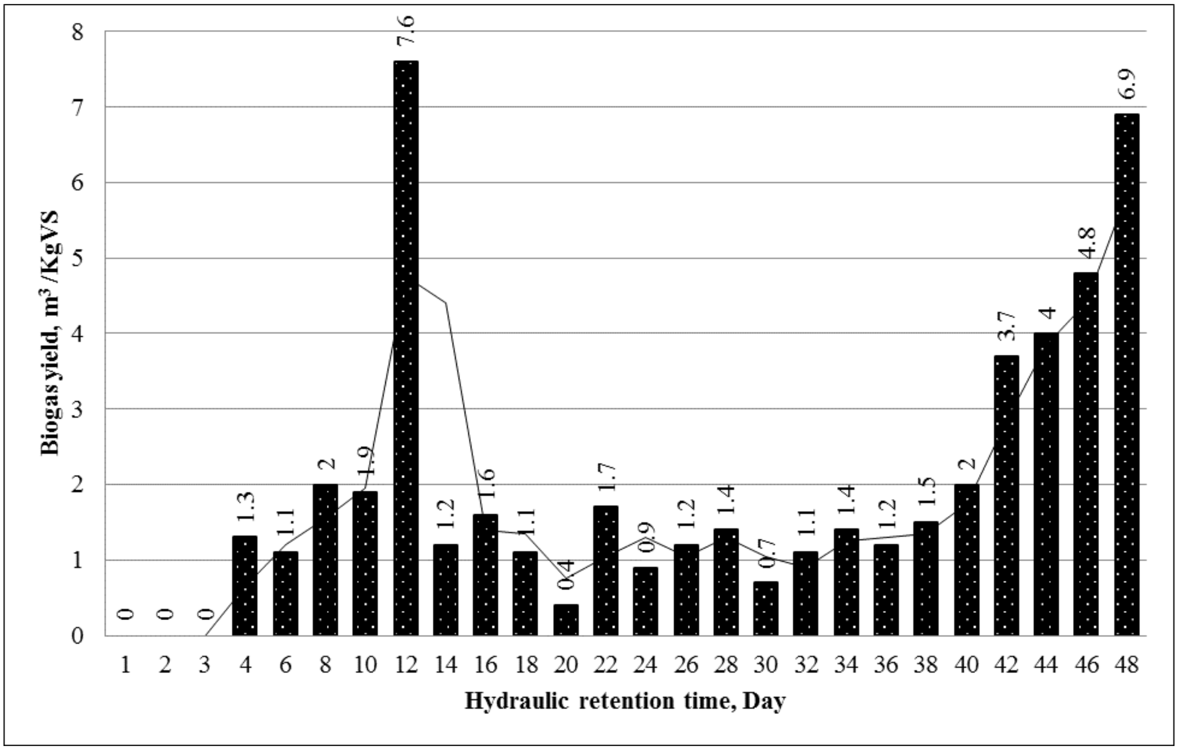

Fig. 6: Biogas yield from Digester E during anaerobic digestion of cow manure.

Fig. 7 shows real life scenario of the digestion process for a 60day HRT period, with high peaks at two regions. First, at 20day HRT, and the second at 42day HRT. As shown in Fig. 7, maximum biogas production was achieved at 42day HRT, then followed with gradual decrease in yield. The decrease in biogas yields could be due to adaptation of methanogenic organism, characterized by the adaptation periods for degradation of long-chain fatty acids and volatile fatty acids. This study has shown that longer HRT is required for degradation of lignocellulosic biomass high in cellulose and hemicellulose. Also, high levels of ammonia may inhibit activities of microorganisms in such environment, as evidence with high pH and ammonium (Table 1). However, substantial amount of ammonium content in digestate is desirable, as it enhances its fertilizers quality (Risberg *et al*., 2017).

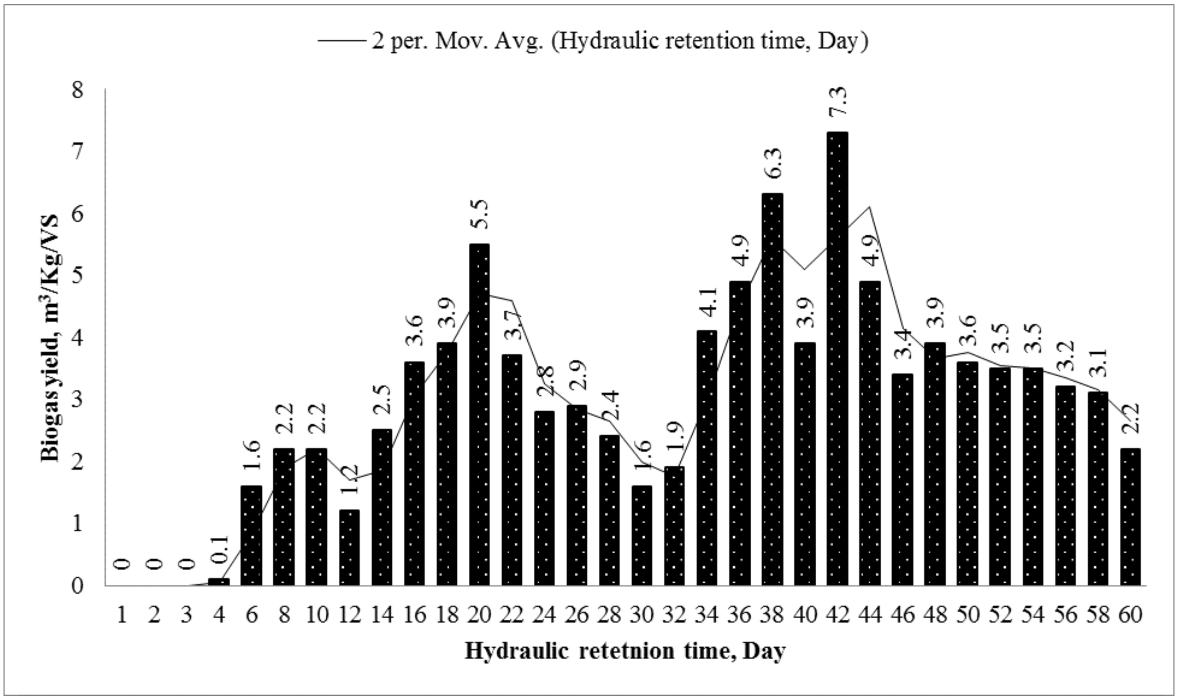

Fig. 7: Biogas yield from Digester F during anaerobic digestion of cow manure

The cumulative biogas yields from all digesters is presented in Fig. 8. As shown in the figure, the coefficient of determination (R^2) increased with increasing HRT, except for digester F. The R^2 values obtained were 0.632, 0.913, 0.926, 0.929, 0.964 and 0.948 for DA, DB, DC, DD, DE and DF, respectively. The R^2 values of present study were found to be in general agreement with those previously reported. For example, Browne and Murphy, (2013) reported 0.86 to 0.99, Fleck *et al*., (2017) obtained 0.80 and 0.94, while 0.876 to 0.997 were reported by Li *et al*., (2013).

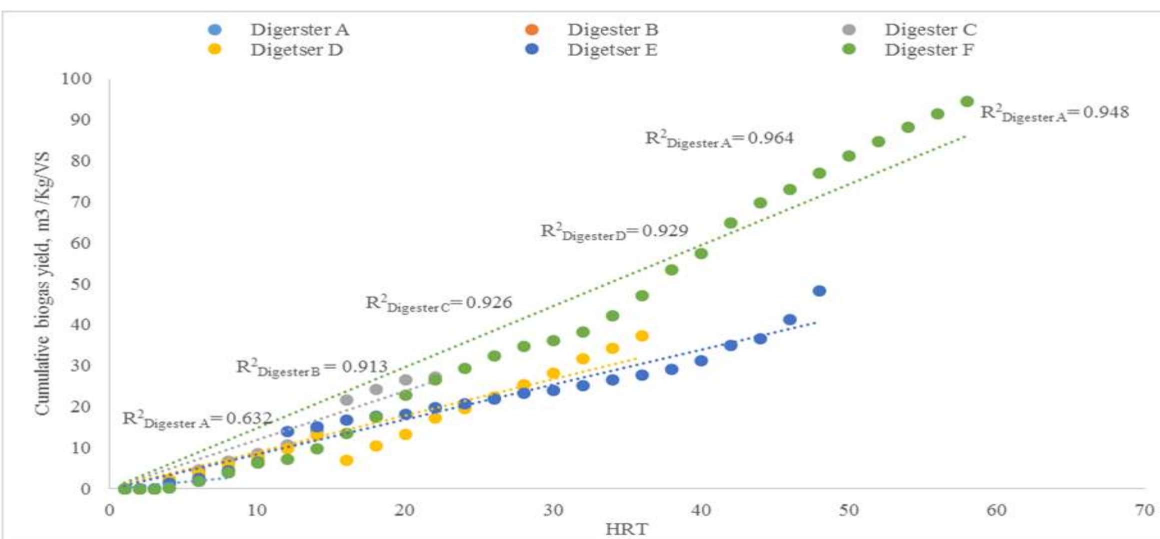

Fig. 8: Cumulative biogas yields for all digesters

늑A: Digester A. DB: Digestr B. DC: Digester C. DD: Digester D. DE: Digester E. DF: Digester F.

Digestate quality

Knowing the quality of digestate is important before its further use, potentially as an alternative fertilizer and an energy source. In this study, the digestate quality including that of the cow manure was assessed based on its elemental and metallic compositions. The amount of element in digestate is shown in Fig. 9. As presented in Fig. 9, there were general reduction in the elemental carbon, hydrogen, nitrogen and sulfur except oxygen. Apparently, reduction of these elements led to an increase in the oxygen content. The carbon content reduced from 35.6w/w% (cow manure) to 19.9w/w% of digestate after a 60day HRT. Similarly, the hydrogen content decreased from 6.3w/w% to 2.8w/w%, while the nitrogen content decreased from 2.1w/w% to 0.8w/w%. There was no substantial variation in sulfur content, as it fluctuates between 0.6 w/w/% and 0.7w/w/%. The reduction in elemental content could be due to the uptake by microorganisms for growth during decomposition period. AD of cow dung and poultry droppings led to reduction in elemental content of carbon, hydrogen and oxygen (Alfa *et al*., 2014), though this study shown there was an increase in oxygen content.

As expected, the decrease in elemental contents had effect on the energy density of the digestates. Comparing with the cow manure, the digestate HHVs reduced from 15.1MJ/kg to 3.1MJ/kg, after a 60day HRT. Confirming that substantial amount of energy was evolved during the digestion period. This also reaffirmed that AD technology produces an energy-rich biogas for generation of electricity.

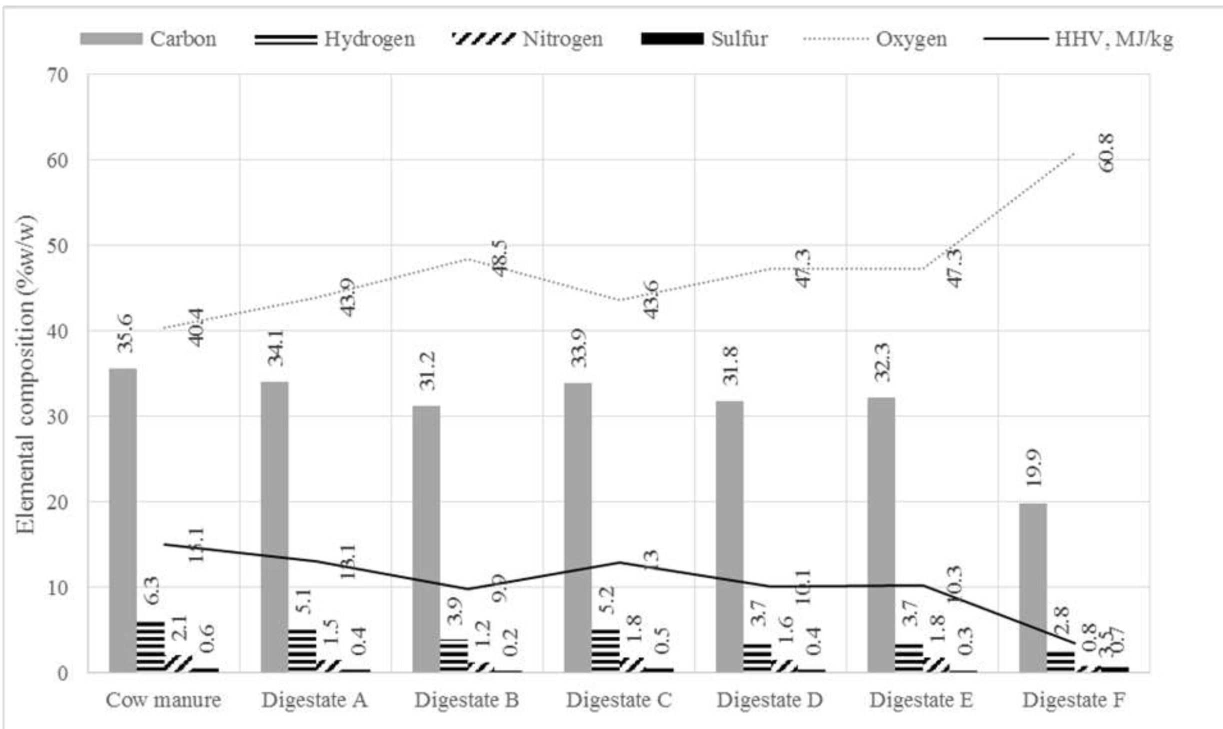

Fig. 9: Elemental composition of digestates following AD of cow manure

Importantly, the carbon-to-nitrogen ratio of the digestate was found to be between 17.9 and 24.8 and higher when compared to 16.9 for cow manure. However, there was decrease in atomic ratio of hydrogen-to-carbon (H/C) when compared to that of cow manure (Table 1). Also shown in Table 1, the H/C of digestates were within range of 1.39 to 1.79, lower than 2.12 for cow manure. The lower H/C ratio of the digestates were as a result of energy loss towards biogas production, as mentioned previously. The oxygen-to-carbon ratio for the digestates were in the range of 1.0 to 2.29, numerically higher than 0.85 of cow manure. Due to usage of carbon by microorganisms during digestion, the digestate were characterised with lower nitrogen-to-carbon ratio (0.03-0.04) when compared to 0.05 for cow manure.

Table 1: Atomic ratios of digestates following anaerobic digestion of cow manure

Feedstock	HRT	H/C	C/N	O/C	N/C
Cow manure	-	2.12	16.9	0.85	0.05
Digester A	8	1.79	22.7	0.96	0.03
Digester B	16	1.50	26	1.16	0.03
Digester C	28	1.84	18.8	0.96	0.04
Digester D	36	1.39	19.8	1.11	0.04
Digester E	48	1.27	17.9	1.10	0.04
Digester F	60	1.68	24.8	2.29	0.03

Beside the digestate being potential biofertilizers, they could also be used as feedstocks for biofuel production. As the aforementioned ratios were within range of data used as feedstock for biofuel production, for example through hydrothermal liquefaction (Eboibi *et al*., 2015).

The metallic composition of the digestates including that of cow manure is presented in Table 2. Although, there some variation in the elemental content, there were no substantial changes in the metallic composition of the digestates when compared with that obtained from cow manure. Some of the little changes were found in potassium (K), magnesium and zinc, apparently these metals could have been used by the microorganisms during methanogenic reaction. This finding suggests that the digestate could also be used as fertilizers on farm, since the digestate and cow manure relatively still contained similar amount of microelements and minor elements. This is in agreement with Koszel and Lorencowicz, (2015) who investigated use of digestate obtained from biogas digesters as alternative fertilizers. They reported that resultant digestate and bovine manure contain similar amount of macroelement and heavy metals.

Table 2: Metallic composition of digestates after anaerobic digestion of cow manure

Parameter (g/KgWW)	Cow manure	Digestate A	Digestate B	Digestate C	Digestate D	Digestate E	Digestate F
Ca	2.4	2.4	2.1	2.0	2.4	2.3	2.4
Cr	10.2	10.6	10.0	14.2	13.9	14.0	11.1
Cu	20.6	20.2	21.3	21.4	22.1	20.0	22.2
Cd	0.4	0.4	0.3	0.3	0.4	0.3	0.3
K	2.8	2.6	2.8	3.0	3.4	3.0	3.0
Mg	0.6	0.6	0.6	0.8	0.8	0.8	0.6
Mn	40.4	38.8	38.2	40.8	42	43.4	44.1
Zn	8	8.2	9.4	8.6	8.2	7.8	8.6
pH at 23°C	7.8	8.2	7.2	8.6	8.0	7.4	8.8
Salinity (ppm)	345.0	345.0	340.0	338.0	334.0	340.0	325.0

Based on the data presented in Table 2, pH is an important factor that affects AD.As shown in Table 2, the pH slight increase from 7.8 to 8.2 for Digester A and 7.2 for Digester B. Then decreased to 8.6 and 8.0 for Digester C and D, respectively. Thereafer decreased to 7.4 for Digester E and 8.8

for Digester F. Similar trend in pH was found for AD of organic solid waste (Tai *et al*., 2007). In present study, it was found that the pH values coressponed with either a low or high biogas yield. Low pH value <5 or >8 inhibits methanogenic bacteria ativities responsible for biogas production (Garba and Sambo, 1992). A pH of 6.8 to 7.0 was reported by Haryanto *et al.,* (2018) after AD of cow dung in a semicontinus anaerobic digester. In this present study, pH values above neutral condition led to low biogas yields. The decrease in pH could be due to prodcution of acidic metabolites, while increased could mostly be as a result of assimilation of the acidic compounds.

CONCLUSION

Hydraulic retention time (HRT) impact on biogas yield and digestate have been evaluated based on biogas yield and properties of the resultant digestate after anaerobic digestion of cow manure. The result of the study showed that HRT has substantial effects on biogas yield and the digestate. Higher HRT reduced the elemental composition (C H N S), energy density (HHV, H/C) and to an extent the coliform content of the resultant digestate. However, with no substantial changes in the metallic content of the digestate when compared with that of the cow manure. Based on data obtained from this study, the digestate could be used as suitable as feedstock for biofuel precursors.

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