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# KINETIC STUDY OF THERMOPHILIC ANAEROBIC DIGESTION OF CATTLE MANURE IN A CONTINUOUSLY STIRRED TANK REACTOR UNDER VARYING ORGANIC LOADING RATE

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**ABSTRACT**

This study established a kinetic model for predicting biogas production using a Continuously Stirred Tank Reactor (CSTR) digesting cattle manure under varying Organic Loading Rate (OLR) at Thermophilic Temperature (TT) of $56 \pm 1^{o} C$. The daily reactor loading was commenced at an OLR of 1.0 g.$_{oTS}$/L.d. This (OLR) was increased fortnightly by 0.5 g.oTS /L.dtill an OLR of 5.0 g.oTS /L.dwas attained. At this point, the biogas and methane yields were no longer commensurate with the feeding and the reactor was adjudged failing. The volume of biogas produced was measured and analysed for $CH_4$, $CO_2$, $H_2S$ and CO using an Infrared gas detector (PRONOVA). The experiment was run continuously for about 140 days. The biogas yields corresponding to OLR of 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 g.oTS /L.d were 0.420, 0.370, 0.420, 0.450, 0.430, 0.430, 0.400, 0.380 and 0.360 L/goS respectively. The highest biogas yields of 0.45 L/goS was recorded at an OLR of 2.5 goTS/l.d. The average methane content was 57%. The kinetic model developed related biogas yield with OLR and could be used to dimension CSTR experiment digesting animal manure.

**Keywords:** kinetic model, organic loading rate, thermophilic temperature, biogas yields, cattle manure.

**Nomenclature**

| Parameter | Meaning | Parameter | Meaning |
|---|---|---|---|
| oTS | Organic Total Solid | UASB | Upflow Anaerobic Sludge Blanket reactor |
| TS | Total Solid | CSTR | Continuously Stirred Tank reactor |
| FM | Fresh Mass | FOSTAC | Volatile Organic Acid/ Buffer Capacity |
| HRT | Hydraulic Retention Time (days) | $y_{max}$ | Maximum biogas yield, L/goS |
| OLR | Organic Loading Rate (goTS/L.d) | Y | Biogas yield, L./goS |
| AD | Anaerobic Digestion | N | Nitrogen, mg. kg$^{-1}$ TS 60° |
| CF | Crude Fibre | P | Phosphorus, mg. kg$^{-1}$ TS 60° |
| $V_R$ | Volume of the reactor (L) | K | Potassium (%TS) |
| COD | Carbon Oxygen Demand | $M_o$ | Input and output mass flow |
| K<br>TT | first order reaction rate constant (d1)<br>Thermophilic Temperature ($^{o}$C) | $c_o$ | Input substrate concentration,oTS (g/kg) |

## 1. INTRODUCTION

Anaerobic Digestion (AD) is the process by which bacteria acts on biological material to produce biogas. Biogas, an alternative energy source contains methane ($CH_4$), carbondioxide ($CO_2$) and varying amounts of water, oxygen, hydrogen sulphide ($H_2S$) and other compounds (Madu and Sodeinde, 2001, Adebayo *et al.*, 2015). AD of organic matter to biogas has advanced to the point of seeing methane production as a carrier of energy rather than just a way of treating waste (Karlsson *et al.,* 2014). Biogas production takes place inside an anaerobic digestion reactor which could be batch digestion reactor (digester), plug low reactor or a Continuously Stirred Tank Reactor (CSTR). For this study, a factory made CSTR was chosen because it is the most common for commercial agricultural biogas plants (Terboven, *et al*., 2017).

Biogas is beginning to gain ground in most developing countries especially in Africa and efforts are being put together to establish the effects of various factors on biogas yields of different organic materials. Studying these to quantify how effectively they can be used as alternative energy sources. There is a vast energetically available agricultural residues in Africa especially in Nigeria which can be processed into biogas (Jekayinfa and Scholz, 2013 a, b). Researchers have used various agricultural residues and manure to produce biogas (Chanakya and Sreesha, 2012; Silvestre *et al*., 2015; Wobiwo *et al*., 2017; Andriamanohiarisoamanana *et al.,* 2017).

Several factors influence the quality and yields of biogas. Among these factors are temperature, feedstock properties, Organic Loading Rate (OLR), pH, Hydraulic Retention Time (HRT), co-digestion (mix-ratio) and type of digester. Organic Loading Rate (OLR) and Hydraulic Retention Time (HRT) form part of the important parameters that influence AD. The effect of various parameters on biogas yields have been studied by researchers (Riggio *et al.,* 2017, Babee and Shayegan, 2011, Chen *et al*., 2014, Adebayo *et al.,* 2013, Panyadee *et al*., 2013, Adebayo *et al.,* 2014 a, b, c, Adebayo *et al*., 2015 a, b, c, d, Jekayinfa *et al*., 2015, Qiao *et al.,* 2013, Mel *et al.*, 2015, Krishnan *et al.,* 2016). Trnovec, and Britz, (1998) studied the influence of OLR and HRT on the effectiveness of a bioreactor (UASB) digesting effluents and concluded that the removal of carbon oxygen demand ranged between about 90 to 93 % when the values of OLR were 9.8 and 10.95 kg $m^{-3}d^{-1}$(COD) with 10h HRT and feedstock pH of 5.5 using effluents from canning industry. Chen *et al*., (2014) studied the effect of OLR commencing from 5 and stopping at 17.5 kg. /$m^3$.day on the behaviour of an anaerobic two-phase digestion filter under pressure and recorded the best performance when the OLR was 12.5 kg./$m^3$.day.When the OLR was higher with HRT being shorter, the reactor was adjudged unstable.

There have been efforts for decades at modelling anaerobic digestion process which have produced models like, dynamic, monod (kinetic equations), unstructured models (segregated and non -segregation), particle swarm optimization, activated sludge model, first order model (Morris, 1976, Hill and Barth, 1978,Garcia-Ocha*et al*.,1999) and so forth. Interestingly, most of the developed models fall between simple and complex regions (Floride and Tassou, 2014). Simple models estimate biogas yield based on the source of the inputs like the number of cattles, sheep, chicken and so on. It is also termed complex because various physical, chemical and biological processes are involved. In CSTR experiments however, OLR and HRT are the most important parameters for dimensioning the reactor size and performances. Hence, kinetic models of anaerobic digestion of animal manure at thermophilic temperature using a continuously stirred tank reactor under varying organic loading rate has been developed which forms the basis of this paper.

## 2. MATERIALS AND METHODS

### 2.1 Materials and equipment

The animal manure (cattle slurry) used for this research was obtained from Grosskreutz, Germany. Other materials and equipment used included Continuously Stirred Tank Reactor (CSTR), gas measuring meter (RITTER), water bath with pump incorporated, gas bag (LINDE), thermometer, barometer, pH meter with conductivity probe, vertical stirrer (electrically operated) and timer for timing the operation of the stirrers. An Infrared Gas Detector (PRONOVA)was also used for the analysis of the gas compositions.

### 2.2 Analysis of the substrates

The sample of cattle slurry for this research was taken to the laboratory for prior analysis before the commencement of the experiment. The following parameters were determined; Total Solid, TS (%), Organic Total Solid, oTS (%TS), oTS (% Fresh Mass, FM), pH, TS ($60^o$-$105^oC$), conductivity, $NH_4$-N, Total acetic acid, ethanol, crude fibre, Phosphorus and Potassium. The inoculum which was obtained from previous anaerobic digestion experiment was also analyzed in the laboratory. After the commencement of the experiment, the reactor sample was taken on weekly basis to the laboratory for the determination of Total solid (TS), conductivity, $NH_4$-N, organic Total Solid (oTS), pH, oTS (%FM) and FOSTAC. This helps to monitor what was happening within the CSTR. The German standard guideline was followed in carrying out the analysis (VDI, 4630 of 2006).

The constituents of the gas produced ($CH_4$, $CO_2$, $H_2S$ and $O_2$), after measuring the volume using the connected gas meter (RITTER) were also determined every second day or third day using the infrared gas detector (PRONOVA). The standard procedures according to APHA, (1992), VDI, (2006) and Ramachandrra and Kamakshi, (2005) were used for the determination of the process parameters.

### 2.3 Method

Figure-1 is a pictorial view of the CSTR experimental set up.At the beginning of the experiment, the CSTR was filled with 8.0 litre of inoculum after which the addition of substrate commenced at a low OLR of 1.0 goTS$l^{-1}d^{-1}$ using Cattle slurry (Adebayo*, et al.,* 2015, VDI, 2006). The mass of the substrates was calculated (equation 1). The experiment was run at thermophilic temperature ($56 \pm 1^o C$ ) using cattle slurry. The temperature was sustained by the hot water being circulated through the hollow wall of the CSTR. The reactor was daily fed with seven feeding per week. The mass of substrate calculated for OLR of 1.0 goTS/l.dwas loaded for seven days. At the end of the seven days, the volume of the gas produced was measured, the compositions of the gas produced were determined and the effluent taken weekly from the reactor analysed in order to know what was taken place in the reactor. The reactor volume was maintained constant by taken the excess above the marked point before feeding. The reactor was stirred slowly at a speed of about 100 rev/min for fifteen minutes every hour (this was achieved with the installation of a timer).

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**Figure-1.** Pictorial view of CSTR investigational set up at thermophilic temperature.

$$M_s = \frac{OLR \times V_R}{C_s} \quad (1)$$

The experiment was repeated at OLR of 1.0 g oTS/L.d for another week making 14 days altogether with the same loading per day. The averages of the measured parameters were then noted. The OLR was then increased by 0.5g oTS/L.d fortnightly to have 1.5, 2.0. 2.5, 3.0, 3.5, 4.0, 4.5, and 5.0 g oDM/L.das recommended by VDI, 4630 with fourteen (14) per each OLR. When the experiment was in progress, the cattle slurry samplebeing used waskeptat 3$^{o}$C while the one to be used later was kept in the inner cold room at an average daily temperature of -21$^{o}$C in order to ensure that the substrate properties were kept constant.The feeding, drawing, gas measurement using a multi-chamber rotor gas meter, RITTER and gas analysis using an infrared gas detector popularly called PRONOVA continued for about 140 days while thereactor temperature was maintained at a thermophilic temperature of $56 \pm 1^{o} C$. The volume of biogas produced was averaged on weekly basis and recorded based on standard conditions (0$^{o}$C, 1013 mbar). The trend of biogas and methane yields were observed especially with increase in OLR. The feeding continued until the reactor began to show signs of failure at 5.0goTS/L.d (OLR). At this point, the experiment was terminated (at about 140 days).

**2.4 Development of kinetic model**

A Kinetic model was established for the CSTR experiment using the approach of Linke, 2006. The model predicts the biogas production fromthe CSTR maintained at thermophilic temperature.If the biogas mass is ignored and equal amount of input and output mass flow $M_o$ are considered, then the mass balance equation will be

$$V_R \frac{dc}{dt} = M_o.c_o - M_o.c + V_R.r(c) \quad (2)$$

r(c), the rate of removing substrate which is a function of the concentration (c) is expressed as kinetic of the first order and written as

$$-\frac{dc}{dt} = r[c] = -k[c] \quad (3)$$

$V_R = m_o.HRT\ at\ steady\ \ state\ \ for\ V_R$ .

k= first order reaction rate constant (d1)

$\frac{dc}{dt} = 0$, equations (2) and (3) can now be combined to give equation (4).

$$HRT = \frac{1}{k}.\left(\frac{c_o}{c} - 1\right) \quad (4)$$

ARPN Journal of Engineering and Applied Sciences



According to Linke, (2006), the decomposable portion of the complex organic feedstock is converted to biogas based on the following equation (5).

$$\frac{c_o - c(t)}{c_o} = \frac{y(t)}{y_{max}} \quad (5)$$

Equation (5) can be re-written as

$$\frac{c_o}{c} = \frac{y_{max}}{y_{max} - y} \quad (6)$$

By putting equation (6) in (4), the equation becomes

$$HRT = \frac{1}{k} . \left( \frac{y}{y_{max} - y} \right) \quad (7)$$

$$y = \frac{HRT . k . y_{max}}{HRT . (k+1)} \quad (8)$$

OLR and HRT are most desirable in CSTR experiments and substituting $OLR = c_o / HRT$ ,equation (8) becomes;

$$OLR = \frac{k \times c_o}{y / (y_m - y)} \quad (9)$$

The equation above (equation 9) can be re-arranged as:

$$y = y_m \frac{k.c_o}{k.c_o + OLR} \quad (10)$$

$y_m$ was the yield from a batch experiment which has the same properties with the cattle slurry used in this experiment. The constant k was obtained from the CSTR experiment by plotting the graph of y/($y_m$-y) against 1/OLR having k.$c_o$ as the slope.

## 3. RESULTS AND DISCUSSIONS

The chemical and thermal properties of the substrate and the inoculum as determined in the laboratory prior to the commencement of the experiment areas presented in Table-1. The Total Solid (TS), Organic Total Solid, oTS (% in TS), oTS(%Fresh Mass, FM), $NH_4$-N (g/kgFM), $N_{kjel}$ (g/kgFM), P (mg/kg TS 60$^o$C), K (%TS) and pH for the cattle slurry and inoculum used were foundto be 11.77%, 84.05% TS, 9.89% FM, 1.22 g/kgFM, 3.87 g/kgFM, 1082.7 mg/kg TS 60$^o$C, 2.05%DM, 6.56 and 2.23%, 59.31%TS, 1.32%FM, 0.779 g/kgFM, 1.47 g/kgFM, 306.6 mg/kg TS 60$^o$C, 15.50%TS and 7.95 respectively. Figure 2 shows the interrelationship between the biogas yields, biogas rate, methane concentration, retention time (day) and organic loading rate at thermophilic temperature throughout the period of the experiment (about 140 days). Biogas production rate (L./L.d) increased steadily from 0.42 L/L.dat OLR of 1.0goTS/L.dup to 1.79 L/L.d when OLR was 5.0 goTS/L.d. Biogas yields increased gradually after the reactor has attained stability from 0.37 L/goS at OLR of 1.5 goTS /L.d to 0.45 L/goS after which it began to decline until 0.36 L/goS was recorded at OLR of 5.0 goTS/L.d. at this point, the reactor was adjudged to have failed since gas production was no longer commensurate with the feeding. At this point, the experiment was terminated at about 140 days of operation. Similarly, the methane yield was also noticed to increase after reactor stability at OLR of 1.5 goTS/L.d from 0.208 to 0.21 L/goSat OLR of 5.0 goTS/L.d. The methane yield also peaked when the OLR was 2.5 goTS/L.d. The experiment revealed that as OLR increases, biogas and methane yields decrease. The methane concentration throughout the experiment was averaged at 57%. Figure-3 shows the relationship between the yields (biogas and methane) and the OLR. It revealed that the yields decreased with increase in OLR.

**Table-1.** Chemical and thermal properties of cattle slurry and inoculum.

| Parameters | Cattle slurry | Inoculum |
|---|---|---|
| Total Solid, TS (%) | 11.77 | 2.13 |
| oTS (%TS) | 84.05 | 48.25 |
| TS (%FM) | 9.89 | 1.03 |
| TS (60$^o$-105$^o$C) | 96.89 | - |
| $NH_4$-N (g.kg$^{-1}$$_{FM}$) | 1.22 | 0.31 |
| $N_{kjel}$, g.kg$^{-1}$$_{FM}$ | 3.87 | 1.47 |
| P mg. kg$^{-1}$ TS60° | 1082.7 | 306.6 |
| K %TS | 2.05 | 15.50 |
| CF(%TS) | 26.75 | - |
| pH | 6.56 | 8.16 |
| Conductivity (mS/cm) | 9.98 | 11.56 |
| Ethanol (g.l$^{-1}$) | <0.04 | <0.04 |
| Propanol | <0.04 | <0.04 |
| TAC | 8.12 | 0.33 |

ARPN Journal of Engineering and Applied Sciences



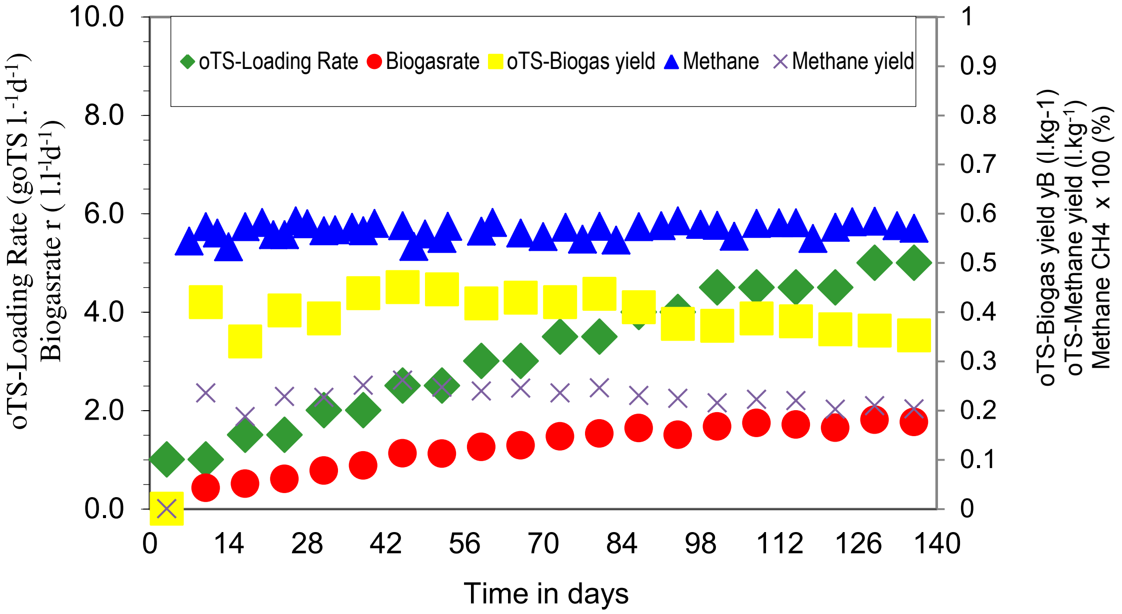


**Figure-2.** OLR, Biogas production rate, Biogas yield, methane yield and methane concentration atthermophilic temperature.

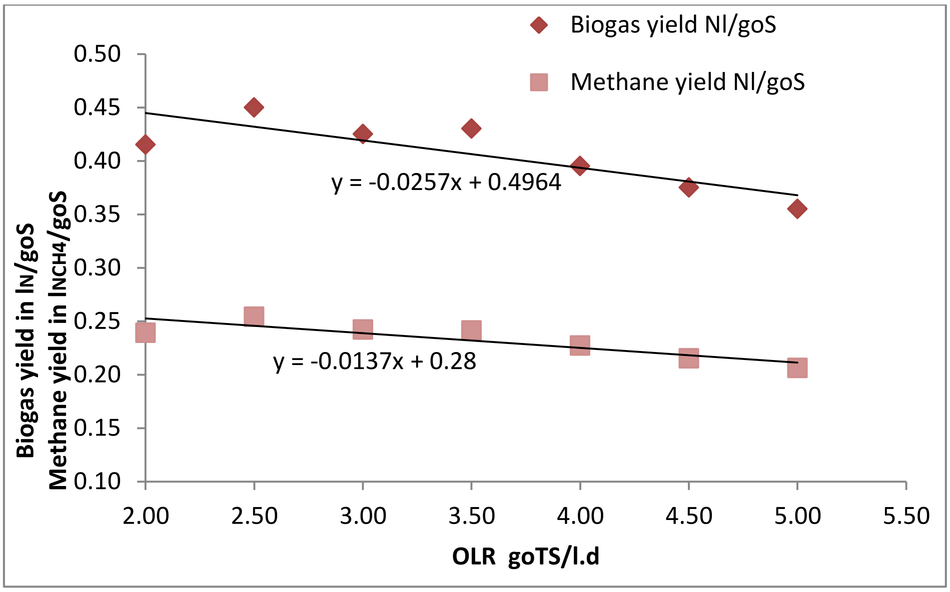


**Figure-3.** Plot of biogas and methane yields against OLR.

Figure-4 shows the relationship between the properties of the effluents (pH value, TS 105%,oTS %FM, conductivity, NH4-N, FOSTAC) and HRT. It revealed that the pH values ranged between 7 and 8 (between neutral and basic). This fell within the acceptable limit and hence the FOSTAC was kept within limit throughout the experiment. TS and oTS increased with increased in HRT and that led to the failure of the reactor because at high oTS, bacterial activities will slow down and hence affected biogas production.

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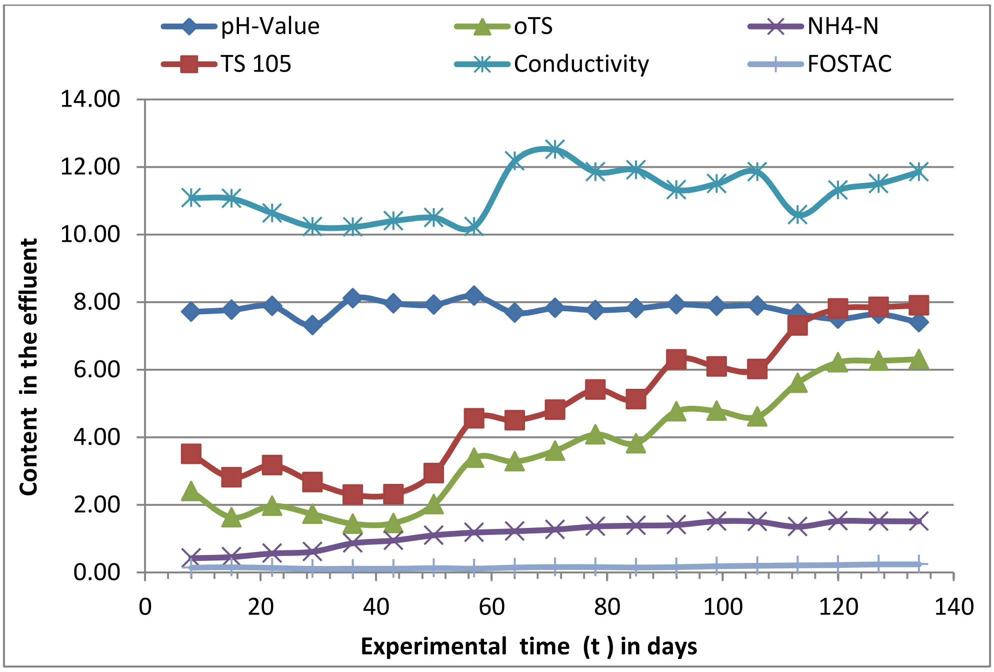


**Figure-4.** Analysis of reactor effluents.

**3.1 CSTR experiments and the kinetic model**

The important parameters involved in kinetic model development co, k and $y_{max.}$are very important. A plot of $y/y_{max}$-y on the abscissa against 1/OLR on the ordinate axes have the product of $c_o$ and k as the slope.The $c_o$ can be obtained from the average of the analysis of the substrate (cattle slurry) used for the experiment and thus k. The maximum biogas yield, $y_m$ was 0.490 l/goS. Figure-6 is a plot of the yield ratio (y/ym-y) versus the inverse of OLR. The average concentration of the samples (animal manure) used for the experiment was 84.27 g/kgFM.

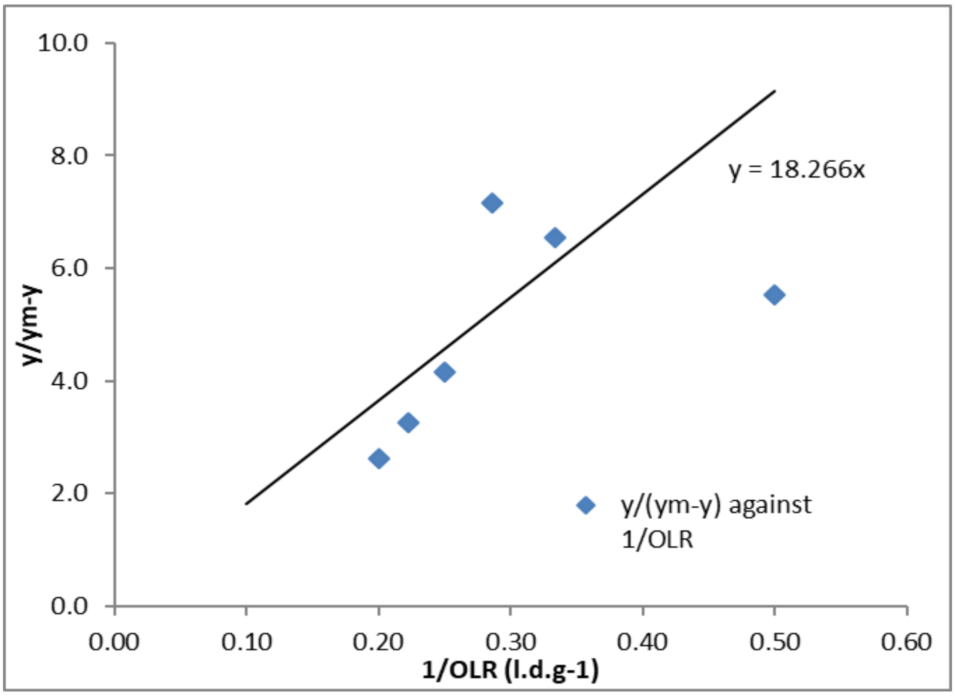


**Figure-6.** A plot of $(y/y_m$-y) vs (1/OLR).

The kinetic model established for the CSTR experimentations is of the form

$$Biogas\ yield\ (y) = y_{max} \frac{k.\ c_o}{k.\ c_o + OLR}\ (equation\ 10\ as\ above)$$

$$c_o.k = Slope = 18.26 \qquad (11)$$
$$\Rightarrow k = 0.217$$

The model of the first order (kinetic) for CSTR digesting animal manure at TT (56$^o$C)is now;

$$y = 0.11\frac{c_o}{0.22.c_o + OLR} \qquad (12)$$

## CONCLUSIONS

From the results obtained, the biogas and methane yields decrease as OLR increases. The maximum biogas and methane yields of 0.45 L/goS and 0.25 L$CH_4$/goS respectively were recorded at OLR of 2.5goTS/L.d. The average concentration of methane ($CH_4$) in a CSTR digesting cattle slurry under thermophilic condition was found to be 57%. The long-term experiment performed (140 days) using CSTR has enabled the establishment of kinetic model based on mass balance equation of the first order. The kinetic model developed would find great application in anaerobic digestion of animal manure using a CSTR at thermophilic temperature.

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