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Mesophilic Digestion Kinetics of Manure Slurry

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Abstract Anaerobic digestion kinetics study of cow manure was performed at 35°C in bench-scale gas-lift digesters (3.78 l working volume) at eight different volatile solids (VS) loading rates in the range of 1.11–5.87 g l^{-1} day^{-1}. The digesters produced methane at the rates of 0.44–1.18 l l^{-1} day^{-1}, and the methane content of the biogas was found to increase with longer hydraulic retention time (HRT). Based on the experimental observations, the ultimate methane yield and the specific methane productivity were estimated to be 0.42 l CH_4 (g VS loaded)$^{-1}$ and 0.45 l CH_4 (g VS consumed)$^{-1}$, respectively. Total and dissolved chemical oxygen demand (COD) consumptions were calculated to be 59–17% and 78–43% at 24.4–4.6 days HRTs, respectively. Maximum concentration of volatile fatty acids in the effluent was observed as 0.7 g l^{-1} at 4.6 days HRT, while it was below detection limit at HRTs longer than 11 days. The observed methane production rate did not compare well with the predictions of Chen and Hashimoto's [1] and Hill's [2] models using their recommended kinetic parameters. However, under the studied experimental conditions, the predictions of Chen and Hashimoto's [1] model compared better to the observed data than that of Hill's [2] model. The nonlinear regression analysis of the experimental data was performed using a derived methane production rate model, for a completely mixed anaerobic digester, involving Contois kinetics [3] with endogenous decay. The best fit

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values for the maximum specific growth rate (μ_{m}) and dimensionless kinetic parameter (K) were estimated as 0.43 day^{-1} and 0.89, respectively. The experimental data were found to be within 95% confidence interval of the prediction of the derived methane production rate model with the sum of residual squared error as 0.02.

Keywords Anaerobic · Digestion · Kinetics · Manure · Mesophilic · Mathematical model · Methane production

Introduction

Animal waste is a valuable biomass resource, which can be utilized as a renewable source of energy. However, it is often mishandled and underutilized, leading to numerous environmental problems such as surface and ground water contamination and greenhouse gas (methane) emissions. The United States alone produces approximately 230 million tons of dry matter animal waste every year that sometimes cannot be used as a local fertilizer [4]. Therefore, an efficient, economical, and sustainable approach to animal waste management is urgently needed. The most commonly applied animal waste management option is anaerobic digestion. Products of the anaerobic digestion process are nutrient-rich fertilizer and a sustainable distributed energy source in the form of methane. Therefore, large-scale animal waste digesters are receiving growing attention as a nonconventional energy-producing technology. Conversion of 50% of the annual animal waste generated is equivalent to 5% of the U.S. annual coal consumption, on an energy basis [4].

The performance of animal waste-fed anaerobic digesters is affected primarily by the biodegradable matter (volatile solids) present in the influent, influent feeding rate, and the hydraulic retention time. The combined effect of these three important parameters is sometimes represented by a single parameter called volatile solids loading rate (g l^{-1} d^{-1}). To maximize the energy output of anaerobic digesters, methane production rate and methane yield needs to be optimized. Methane production rate is most often represented as the volume of methane produced per unit volume of digester per unit time, and the methane yield is defined as the volume of methane produced per unit weight of volatile solids loaded.

The development of mathematical models to describe anaerobic digestion of animal waste started in the 1960s [5]. Since then, numerous different types of animal waste digestion models (structured/unstructured, segregated/unsegregated, empirical, and mechanistic) have been developed and reported in the literature [1, 2, 5–10]. The complexity of these models varies tremendously and can contain many parameters [10] or very few parameters [1]. Such complexity of the models is often a function of the reactor system described. Moreover, the accuracy of these model predictions varies from case to case because of the varying nature of animal waste, reactor type, and kinetics.

Although having some shortcomings as mentioned by Husain [9], steady-state anaerobic digestion models proposed by Chen and Hashimoto [1] and Hill [2] have been extensively used in the literature because of their simplicity. Chen and Hashimoto [1] proposed that the methane production rate (G) for an anaerobic digester can be given as:

$$G = B_{\mathrm{o}}L\left(1 - \frac{K}{\mu_{\mathrm{m}}\theta - 1 + K}\right) \tag{1}$$

where B_{o}=ultimate methane yield at infinite retention time [1 CH$_4$ (g VS loaded)$^{-1}$], K= dimensionless kinetic parameter, μ_{m}=maximum specific growth rate (day^{-1}), θ=hydraulic retention time (day), L=volatile solids loading rate (g l^{-1} day^{-1}).

As it is not easy to quantify the amount of active microorganisms inside a manure-fed digester, determination of kinetic parameters is a tedious exercise. Therefore, a temperature dependent empirical formula (Equation 2) was proposed by Hashimoto et al. [11] for the calculation of maximum specific growth rate (μ_m). The dimensionless kinetic parameter, K, was found to increase exponentially with influent volatile solids concentration [12], and thus, given by Equation 3.

$$\mu_{\mathrm{m}} = 0.013T(\mathrm{in}^{\circ}C) - 0.129 \quad (\text{for } 20 < T < 60) \tag{2}$$

$$K = 0.8 + (0.0016)e^{0.06S_0} \tag{3}$$

where S_0 is influent VS concentration.

Hill [2] derived an empirical kinetic model for a continuously fed digester. According to which the methane production rate ($\mathrm{l\ l^{-1}\ day^{-1}}$) is,

$$G = \gamma B_0 \sigma I \tag{4}$$

$$I = 0.5 + (1/2.95)\mathrm{Arctan}\left[(\tau - \sigma)/0.211\right] \tag{5}$$

where γ is the amount of methane produced per volatile solids consumed, B_0 is the biodegradability factor for the type of waste, σ is the loading rate (equal to the concentration of volatile solids in the feed divided by hydraulic retention time), and I is a productivity index. Hill [2] defined τ as a "stress index", and recommended that the values of γ, B_0, and τ are set to 0.5 l CH_4 (g VS destroyed)$^{-1}$, 0.483 [g VS (g VS)$^{-1}$], and 9.21 (g VS $\mathrm{l^{-1}\ day^{-1}}$) for confined beef cattle, respectively. However, these kinetic parameters are known to be affected by the composition of manure (which is a function of animal type and feeding practices), level of inhibition, digester type, etc., and thus, cannot be generalized.

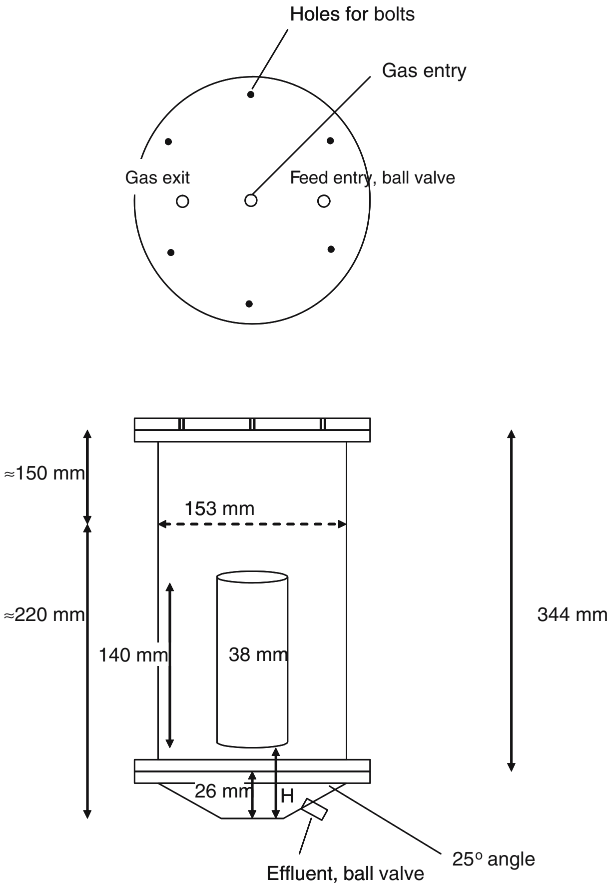
In this work, experimental results of mesophilic (35°C) digestion kinetics of cow manure slurry have been presented and discussed. The predictions of Chen and Hashimoto's [1] and Hill's [2] models, using their recommended kinetic parameter values, were evaluated against the experimental results. Later, a nonlinear regression analysis of the obtained experimental data was performed to calculate μ_m and K using a derived model, for the methane production rate of a completely mixed anaerobic digester, involving Contois kinetics [3] with endogenous decay.

Materials and Methods

Experimental Design

Six bench-scale anaerobic digesters were constructed from polyvinyl chloride plastic with a 3.78-l working volume. A schematic of a bench-scale digester is shown in Fig. 1. The digesters were housed in a temperature-controlled (35°C) area, and operated in a pseudo-continuous mode (i.e., fed every other day) with different feeding rates but with the same feed composition. The biogas generated in the digesters was collected in Tedlar gas bags. To provide gas-lift mixing, a part of the generated biogas was pumped back (1 l $\mathrm{min^{-1}}$) from the top to the lowest point of the draft tube inside the digesters. More details about the digester setup have been described elsewhere [13].

Fig. 1 Schematic of the bench-scale anaerobic digester. Circular body in the center of the digester is a hollow draft tube suspended from the top of the digester (H=40 mm)



The raw cow manure was collected fresh (less than 2 days old) from the University of Tennessee Institute of Agriculture, Tennessee and stored at 4°C until use. Preparation of the manure involved several steps, as described previously [14]. Initially, the digesters were operated under similar conditions with fresh feed added every other day at a rate of 1.64 l feed per week. Then, the feed quantities were changed, while keeping the feed composition constant, and the gas production and the gas composition were monitored every 2 days until steady state (determined as the equivalent of three residence times). Feeding rates were selected to achieve VS loading rates of 5.87, 3.33, 2.39, 1.68, 1.32, and 1.11 g l^{-1} day^{-1}. At steady state, liquid samples from three consecutive sampling events were taken and frozen for subsequent analyses. Digesters 1 and 2 were allowed to continue operation in a second phase, while the feed quantities were again changed to achieve VS loading rates of 3.91 and 1.96 g l^{-1} day^{-1}, respectively. Altogether, cow manure digestion study was performed at eight different VS loadings as given in Table 1.

Analytical Methods

Duplicate gas samples (150 μl) were collected using a gas-tight syringe and a sampling port in the gas collection bag. They were then injected into a gas chromatograph and analyzed as

Table 1 Experimental conditions, and methane production rate and methane yield of the digesters.

Digesters	Volume of feed added every other day (l)	Hydraulic retention time (day)	VS loading (g l^{-1} day^{-1})	Methane production rate (l l^{-1} day^{-1})	Methane yield [l (g VS loaded)$^{-1}$]
Digester 1	1.65	4.6	5.87	1.18	0.20
Digester 1 (2nd run)	1.11	6.9	3.91	1.12	0.29
Digester 2	0.94	8.1	3.33	0.96	0.29
Digester 3	0.67	11.3	2.39	0.81	0.34
Digester 2 (2nd run)	0.55	13.8	1.96	0.73	0.37
Digester 4	0.47	16.1	1.68	0.57	0.34
Digester 5	0.37	20.5	1.32	0.45	0.34
Digester 6	0.31	24.4	1.11	0.44	0.40

described elsewhere [13]. The gas volume in each bag at sampling time was determined by pumping out the gas contained in the bag through a wet gas test meter (GSA/Precision Scientific, Chicago, Ill).

Feed and effluent samples were analyzed for a variety of parameters. Total solids (TS), total suspended solids (TSS), volatile solids (VS), total volatile suspended solids (VSS), volatile fatty acids (VFA), total chemical oxygen demand (TCOD), dissolved COD (DCOD), and total nitrogen (TN) were determined as described elsewhere [13].

Anaerobic Digestion Kinetic Parameters

As it has been discussed in the following section, the experimental methane production rates observed during this study did not compare well with the predictions of Chen and Hashimoto's [1] and Hill's [2] models using their recommended kinetic parameters. Therefore, appropriate kinetic parameters for the observed experimental data need to be sought. As it is very difficult to quantify the amount of active microorganisms inside a manure-fed digester, the kinetic parameters are often estimated through nonlinear regression analysis of the experimental data with respect to kinetic and reactor-scale model predictions. In this work, a simple methane production rate model for a continuous stirred-tank reactor (CSTR) type anaerobic digester was derived assuming first-order substrate consumption rate. The microbial growth kinetics was assumed to follow the model of Contois [3].

$$\text{Specific growth rate, } \mu = \frac{\mu_{\mathrm{m}} S}{(bX + S)} \tag{6}$$

where μ_{m} is the maximum specific growth rate (day^{-1}), X is the cell mass concentration (g l^{-1}), b is called Contois constant, and S is the substrate concentration (g l^{-1}).

Rate of microbial growth, $r_{\mathrm{m}}=(\mathrm{d}X/\mathrm{d}t)=\mu X$, and rate of substrate consumption can be written as: $r_{\mathrm{s}}=-k'\mathrm{S}$, where k' = first-order rate constant (day^{-1}). Substrate mass balance for a CSTR can be written as:

$$\begin{gathered}\text{Accumulation} = \text{Input} - \text{Output} + \text{Production} \\ V\frac{\mathrm{d}S}{\mathrm{d}t} = QS_0 - QS + r_s V\end{gathered} \tag{7}$$

where V is the digester working volume, Q is the flow rate (day^{-1}), S_0 is the influent substrate concentration (g l^{-1}), and S is the effluent substrate concentration (g l^{-1}).

At steady-state, $\frac{dS}{dt} = 0$, and therefore, effluent substrate concentration (g l^{-1}),

$$S = \frac{S_0}{(\theta k' + 1)} \tag{8}$$

where, $\theta = V/Q$= hydraulic retention time (day).

Now, if Y_{ms} is the specific methane productivity [l CH_4 (g VS consumed)$^{-1}$], methane production rate (l l^{-1} day^{-1}) is,

$$G = \frac{Y_{\mathrm{ms}}(S_0 - S)}{\theta} \tag{9}$$

Substituting Equation 8 in Equation 9,

$$\frac{S_0}{G} = \frac{\theta}{Y_{\mathrm{ms}}} + \frac{1}{Y_{\mathrm{ms}}k'} \tag{10}$$

Based on the above equation, a plot of θ vs S_0/G can be drawn to estimate the specific methane productivity (Y_{ms}). Mass balance equation for the net microbial growth can be given as follows:

$$V\frac{\mathrm{d}X}{\mathrm{d}t} = QX_0 - QX + Vr_{\mathrm{m}} - VXK_{\mathrm{d}} \tag{11}$$

where, X_0=influent microbial concentration (g l^{-1}), which is assumed zero, X=effluent microbial concentration (g l^{-1}), K_{d}=endogenous decay constant (day^{-1}).

At steady-state, $(\mathrm{d}X/\mathrm{d}t)=0$, and therefore,

$$\mu - K_{\mathrm{d}} = \frac{1}{\theta} \tag{12}$$

Substituting Equation 6 in Equation 12 gives the effluent substrate concentration at steady state,

$$S = \frac{S_0}{\left[\frac{\mu_{\mathrm{m}}\theta}{K(1+\theta K_{\mathrm{d}})}\right] + \left(\frac{K-1}{K}\right)} \tag{13}$$

where K is a dimensionless kinetic parameter=Yb, Y is microbial yield = $X/(S_0-S)$, and b is Contois constant.

Substituting Equation 13 in Equation 9 gives the methane production rate (l l^{-1} day^{-1}),

$$G = \frac{Y_{\mathrm{ms}}}{\theta}\left[S_0 - \frac{S_0}{\left[\frac{\mu_{\mathrm{m}}\theta}{K(1+\theta K_{\mathrm{d}})}\right] + \left(\frac{K-1}{K}\right)}\right] \tag{14}$$

By performing nonlinear regression analysis of the observed experimental data against the results of Equation 14, using Y_{ms} from Equation 10 and K_d=0.03 day^{-1} [15], maximum specific growth rate (μ_m) and dimensionless kinetic parameter (K) can be estimated.

Results and Discussion

In this study, confined cattle manure slurry was digested at 35°C and the steady-state performance data were acquired. For all loading conditions, digesters were operated for a period more than three HRTs. The observed results for the feed and effluent from the digesters under steady-state conditions are shown in Tables 1 and 2. As expected, more biogas (and methane) was produced in reactors that received a larger quantity of feed. It is evident from Fig. 2 that the methane content of the biogas was dependent on the hydraulic retention time. Shorter retention times resulted in a lower concentration of methane in the biogas. Normally, in a digester methane formation takes place via two routes [9]. In the first case, acetoclastic methanogens convert acetate to methane and carbon dioxide. This pathway accounts for about 70% of the total methanogenesis. The second route involves conversion of H_2 and CO_2 to CH_4 by hydrogen-utilizing methanogens. This pathway accounts for about 30% of the total methanogenesis. Observation of biogas with lower methane content at shorter retention times suggest that the conversion of H_2 and CO_2 to CH_4 by hydrogen-utilizing methanogens gets hampered at shorter HRTs.

A plot of the inverse of HRTs vs observed volume of CH_4 produced per unit mass of VS loaded (methane yield) is shown in Fig. 3. One of the ways of estimating ultimate methane yield [B_0, l CH_4 (g VS loaded)$^{-1}$] is to extrapolate the curve to 1/HRT=0. For the present study, ultimate methane yield was determined as 0.42 l CH_4 (g VS loaded)$^{-1}$ (Fig. 3). Based on Equation 10, a plot of θ vs S_0/G was drawn to estimate the specific methane productivity (Y_{ms}) as shown in Fig. 4. The slope of the curve was used to calculate the specific methane productivity as 0.45 l CH_4 (g VS consumed)$^{-1}$.

The results presented in Table 2 show that about 17–59% of the total COD and 43–78% of the dissolved COD present in the feed were consumed at 4.6–24.4 days HRTs. Volatile fatty acids concentration in the effluents at HRTs longer than 11 days were observed to be below detection limit. Maximum VFA concentration of 0.7 g l^{-1} was observed at 4.6 days

Table 2 Different measured parameters for feed and digester content at steady state.

Parameters	TS (g l^{-1})	VS (g l^{-1})	TSS (g l^{-1})	VSS (g l^{-1})	VFA (g l^{-1})	TCOD (g O_2 l^{-1})	DCOD (g O_2 l^{-1})	TN (g l^{-1})
Feed	50	27	36	23	1.7	58	14	1.6
Digester 1	40	18	34	16	0.7	48	8	1.9
Digester 1 (2nd run)	34	16	24	14	0.1	32	5	2.1
Digester 2	34	15	28	16	0.1	34	4	1.8
Digester 3	31	13	19	11	<0.1	26	3	1.9
Digester 2 (2nd run)	37	17	23	14	<0.1	32	5	2.2
Digester 4	28	13	22	13	<0.1	28	3	2.0
Digester 5	24	11	18	11	<0.1	21	3	1.9
Digester 6	32	14	17	10	<0.1	24	3	2.0

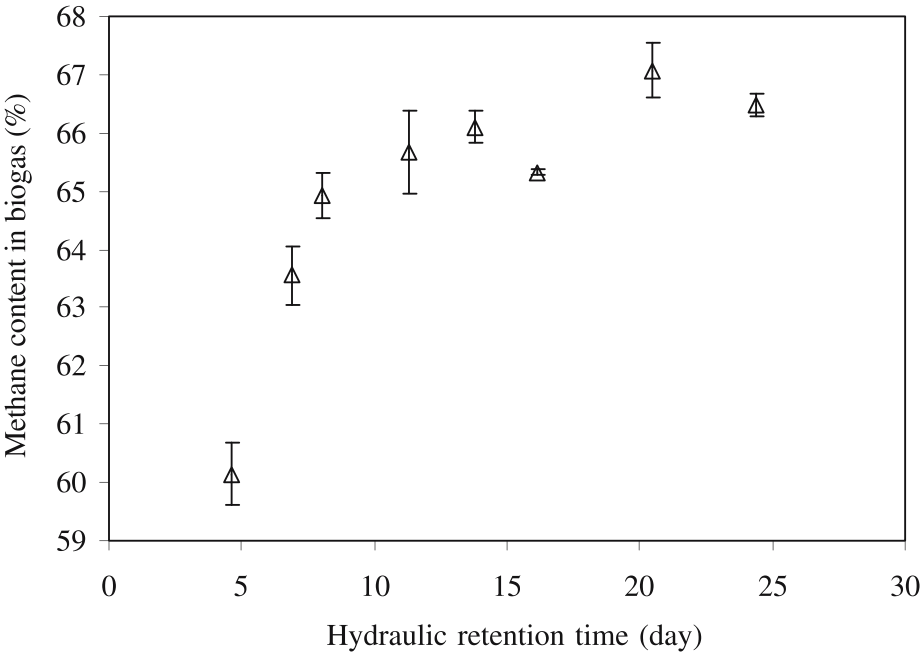


Fig. 2 Methane content in biogas as a function of the hydraulic retention time. *Error bars* are showing the standard deviation values for triplicate measurement at each condition

HRT. The pH of the feed and the content in the digester was 7.36–7.45, essentially constant.

A comparison of the experimental methane production rate with the Chen and Hashimoto [1] and Hills [2] models is shown in Fig. 5. The figure shows that the Chen and Hashimoto model [1] very closely predicts the experimental data except for the last two data points. This indicates that as per Chen and Hashimoto [1], methane production rate should drop at HRTs shorter than 7 days. However, for the cow manure slurry used in this study, there was no significant drop in the methane production rate, which was observed even at 4.6 days HRT. Figure 5 also shows that Hill's model [2] under-predicted the methane production rates for all VS loading except in the case of 5.87 g l^{-1} day^{-1} VS loading. As per Hill's model [2], the methane production rate should increase linearly with the increase in the VS loading until the digester would fail at about 8.5 g l^{-1} day^{-1} VS loading. However, the data observed during this study indicates a nonlinear behavior especially at higher VS loadings, as shown in Fig. 5.

Kinetic Parameters Estimation

Nonlinear regression analysis offers a means for estimating model parameters, which minimize the squared differences between predicted and experimentally observed values of a dependent variable. In the method, a set of experimental values of independent and dependent variables are given as input to a nonlinear regression analysis code, which returns the values for the model parameters corresponding to the least square criterion. Initially, the derived model (Equation 14) predictions were calculated using $Y_{\mathrm{ms}}=0.45$ l CH_4 (g VS consumed)$^{-1}$ (observed value), $K_{\mathrm{d}}=0.03$ day^{-1} [15], and guess values for K and μ_{m}.

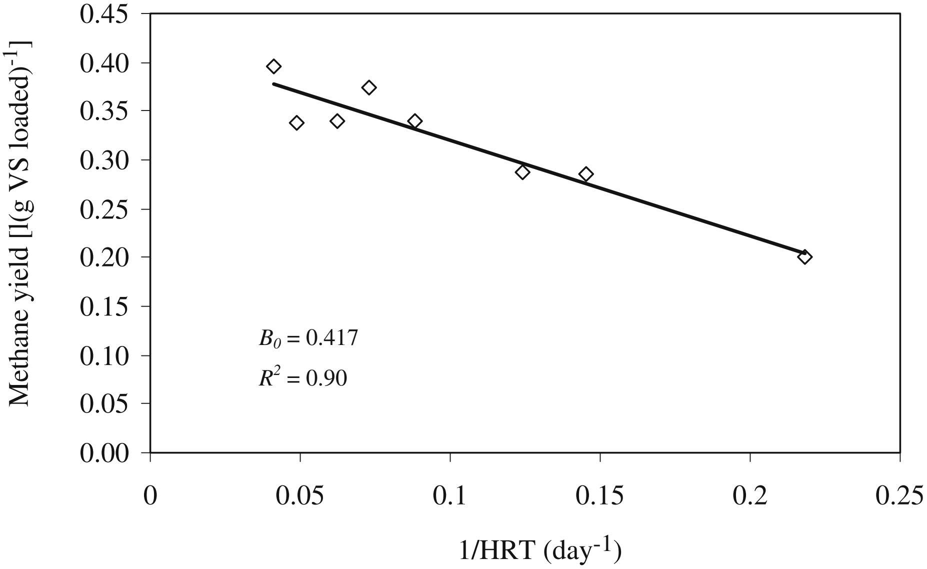


Fig. 3 Plot of methane yield vs 1/HRT for determining ultimate methane yield (B_0)

Thereupon, nonlinear regression analysis of the experimental data and the derived model predictions was performed using SOLVER function in the Microsoft Excel by varying the values of the dimensionless kinetic parameter (K) and the maximum specific growth rate (μ_m). The values of K and μ_m corresponding to the least square criterion were found as

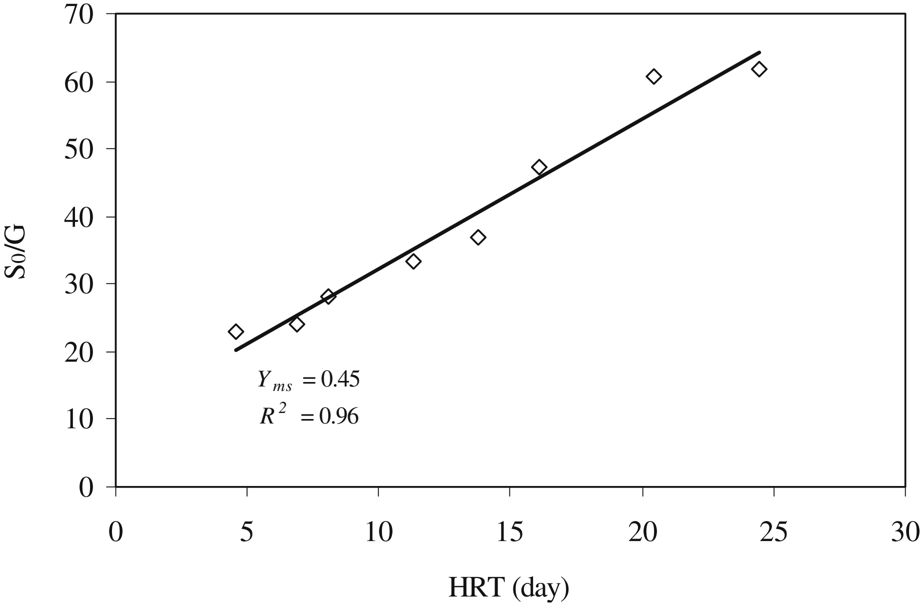


Fig. 4 Plot of S_0/G vs HRT, for calculating specific methane productivity, Y_{ms}

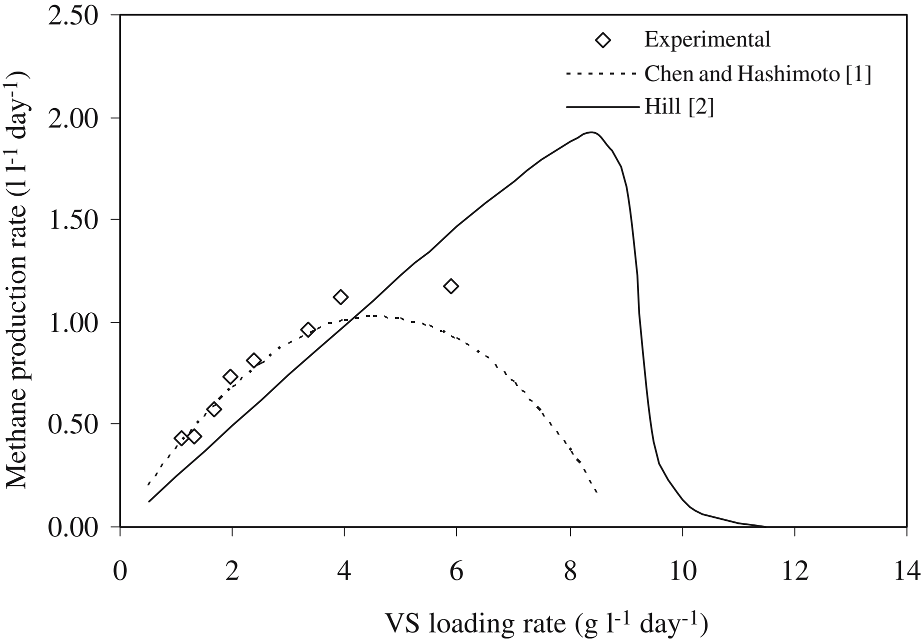


Fig. 5 Comparison of the experimental methane production rates with the predictions of Chen and Hashimoto [1] and Hill [2] models

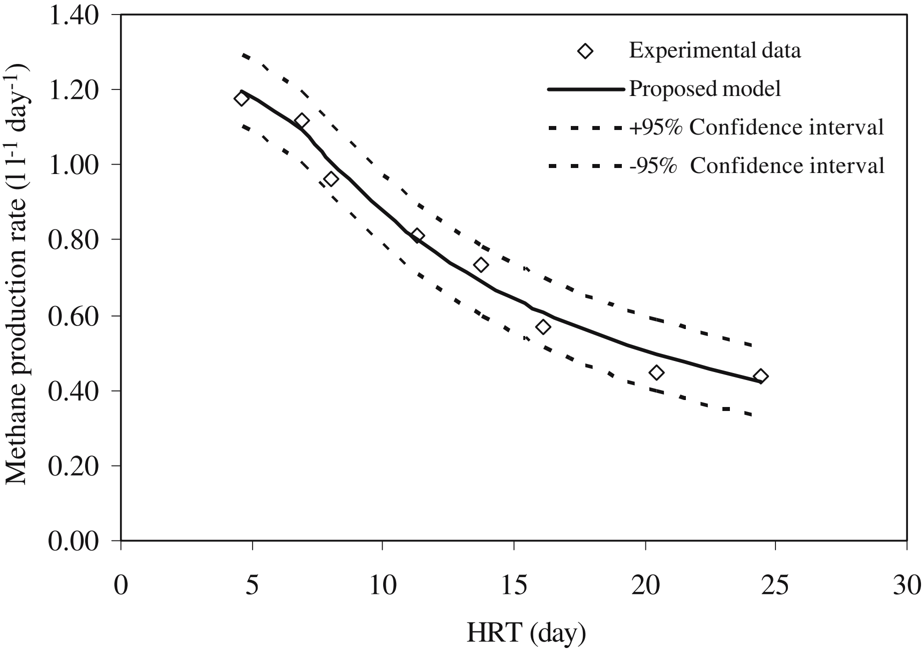


Fig. 6 Plot shows the fit of the derived model (Equation 14) to the experimental data points and the 95% confidence interval

0.89 and 0.43 (day^{-1}), respectively. The curve fit along with 95% confidence interval and the experimental data points have been shown in Fig. 6.

To compare the goodness-of-fit of the observed experimental data to Chen and Hashimoto's [1] and Hill's [2] models, coefficient of determination (R^2) and the sum of residual squared error (SRSE) were calculated. A summary of the of R^2 and SRSE values for the three cases: (1) Chen and Hashimoto's [1] model with their recommended kinetic constants, (2) Hill's [2] model with his recommended kinetic constants, and (3) Equation 14 with the estimated kinetic constants, have been provided in Table 3. The table shows that the derived model offers a valid description of the manure slurry digestion, as it maps the performance of the digester with a tight 95% confidence interval of 0.09 $\text{l l}^{-1}\ \text{day}^{-1}$ and had a coefficient of determination of 0.99. The Chen and Hashimoto [1] model prediction, using their recommended model parameters, showed an R^2 value of 0.86, while the prediction of Hill [2] had a poor correlation (R^2=0.51) with the experimental results. These findings suggest that rather indiscriminately, using the manure digestion models and the recommended parameters may lead to significant error in the methane production rate prediction.

Remarks

Anaerobic digestion of cow manure at 35°C in bench-scale gas-lift anaerobic digesters produced methane at the rates of 0.6–1.5 $\text{l l}^{-1}\ \text{day}^{-1}$ at volatile solids loadings of 1.2–5.5 $\text{g l}^{-1}\ \text{day}^{-1}$. The observed methane production rates did not compare well with the predictions of Chen and Hashimoto's [1] and Hill's [2] models (Fig. 5). However, under the studied experimental conditions, the predictions of Chen and Hashimoto's [1] model fitted better to the observed data than that of Hill's [2] model (Table 3). The experimental methane production rates were found to be within 95% confidence interval of the prediction of the derived methane production rate model involving Contois kinetics [3] and endogenous decay. The best fit values for the maximum specific growth rate (μ_m) and dimensionless kinetic parameter (K) were found as 0.43 day^{-1} and 0.89, respectively. The findings of this study suggest that rather indiscriminate use of the manure digestion models and the recommended kinetic parameters may lead to significant error in the methane production rate prediction. Therefore, it is strongly recommended that each manure digestion system should be individually analyzed and designed to efficiently serve its purpose.

Table 3 Summary of the goodness-of-fit.

Models	Model parameters	R^2	SRSE*
Chen and Hashimoto [1]	μ_m=0.326 day^{-1} K=0.81 B_0=0.42 l (g VS loaded)$^{-1}$	0.86	0.074
Hill [2]	τ=9.21 g VS $\text{l}^{-1}\ \text{day}^{-1}$ γ= 0.5 l CH_4 (g VS destroyed)$^{-1}$ B_0=0.483	0.51	0.566
Derived Model (Equation 14)	μ_m=0.43 day^{-1} K=0.89 K_d=0.03 day^{-1} Y_m=0.45 l CH_4 (g VS consumed)$^{-1}$	0.99	0.024

*The sum of residual squared error = $\sum_{i=1}^{n}\left((\text{fitted} - \text{measured})/\text{measured}\right)^2$, n is the number of data point

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