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Effect of organic loading rate during anaerobic digestion of municipal solid waste

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HIGHLIGHTS

- Co-digestion of OFMSW with inoculum: an effective approach in methane production.
- COD removal affects methane production rate with respect to different pH.
- Maximum COD removal of 84.20% achieved with a CH_4 production of 9.22 L.
- Multiple regression analysis proved COD removal efficiency over CH_4 production.

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ABSTRACT

The effect of chemical oxygen demand (COD) and volatile solids (VS) on subsequent methane (CH_4) production during anaerobic digestion (AD) of organic fraction of municipal solid waste (OFMSW) was studied in a laboratory-scale digester. The experiment was performed in 2 L anaerobic digester under different experimental conditions using different input mass co-digested with inoculum and organic loading rate (OLR) for 27 days at 38 ± 2 °C. Three digesters (digesters 1, 2 and 3) were operated at initial loading of 5.1, 10.4 and 15.2 g/L COD_S per batch which were reduced to 77.9% and 84.2%, respectively. Cumulative biogas productions were 9.3, 10.7 and 17.7 L in which CH_4 yields were 84.3, 101.0 and 168.4 mL/g VS removal in digesters 1, 2, and 3, respectively. The observed COD removal was found to be influenced on variation in CH_4 production. Co-efficient of determination (R^2) was 0.67 and 0.74 in digesters 1 and 2, respectively.

1. Introduction

Anaerobic digestion (AD) is an attractive waste treatment technique in which both organic stabilization and energy recovery are achieved. Many agricultural and industrial wastes are also ideal candidates for AD because they contain high levels of easily biodegradable materials. Low methane (CH_4) yield and process instability are often encountered in AD process. A wide variety of inhibitory substances is the primary cause for failure of anaerobic digester since they are present in substantial concentrations (Chen et al., 2008).

Among various environmental conditions, pH is the most sensitive parameter. The pH of liquid effluent from the digesters indicates the stability of the system and its variation also depends on the buffering capacity of the system (Mata-Alvarez et al., 2000).

Volatile solids (VS) represents organic portion of the material solids that can be digested while the remainder of the solids is considered as fixed. The 'fixed' solids organic of the VS are non-biodegradable. The actual loading rate depends on the types of wastes fed into the digester (Mattocks, 1984). Food waste has low total solid (TS) and high content of soluble organic but it is much more easily degradable and has higher energy content per dry mass. Excess ammonia and volatile fatty acids (VFAs) accumulation are more with AD of high solid content food waste. Co-digestion of food waste with lignocellulosic biomass and/or two-phase operation was advocated to solve these problems (Mata-Alvarez et al., 2000; Chen et al., 2008, and Li et al., 2011).

Zhang et al. (2007) reported in their study that AD of food waste had average CH_4 yield of 435 mL/g VS after 28 days of digestion at 50 ± 2 °C. About 80% of CH_4 yield was obtained after the first 10 days of digestion giving a CH_4 yield of 348 mL/g VS.

Babaee and Shayegan (2011) used a 70 L reactor with a loading rate of 1.4 kg VS/(m^3/day) and reported stable performance of the

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digester with biogas yield (0.4 m^3 biogas/kg VS) and highest CH_4 yield (0.25 m^3 CH_4/kg VS) and VS reduction of around 88%. As the organic loading rate (OLR) was increased, the VS degradation and biogas production was decreased.

Mata-Alvarez et al. (1992) used a loading rate of 1.6 kg VS/(m^3/day) of fruit and vegetable waste in a reactor and reported highest biogas yield (0.47 m^3 biogas/kg VS) with VS reduction of around 88%.

Consistent CH_4 production, VFAs concentrations, pH and VS removal in different OLR of solid dairy manure indicated a stable and reproducible process with efficient performance in the range 6.0–8.0 g total chemical oxygen demand (COD_T)/kg inoculum/day (Saady and Masse, 2015).

Cow-dung contains soluble, particulate, and fibrous components. It contains carbohydrates (cellulosic and hemicellulosic fiber), lipids, fats, and proteins. Approximately 40–50% of the VS in dairy manure are biodegradable lignocellulosic biomass which can be converted to CH_4 (Jha et al., 2012). Angeriz-Campoy et al. (2015) demonstrated that food waste has been an appropriate co-substrate for the enhancement of hydrogen production in dark fermentation. Zhang et al. (2013) reported that the co-digestion of food waste with other wastes in a single digester became increasingly popular with the advantage of adjusting the C/N ratio.

Co-fermentation of different substrates could be beneficial due to dilution of toxic chemicals, enhanced balance of nutrients, and synergistic effect of microorganisms (Razaviarani and Buchanan, 2014). However, Demirel and Scherer, 2008 reported that AD of single highly biodegradable organic substrate might result in process failure in the absence of any buffering agent for pH adjustment and proper external nutrients addition.

It is also reported that high value of F/M might be toxic and low value might prevent induction of the enzyme necessary for biodegradation. The optimum value of F/M for AD ranged from 0.57 to 0.68. Decrease in cumulative CH_4 per unit particulate COD (COD_P) was also found to be minimum at F/M ration of 0.57. The two variables which influence CH_4 generation were compounded to derive a single parameter COD_P/volatile suspended solids (VSS) (Prashanth et al., 2006).

Many studies have been carried out on the effect of VS removal in CH_4 production during AD of MSW. But the information on the effect of COD reduction on subsequent CH_4 production is seldom scripted. Hence, the effect of COD and VS on anaerobic biotransformation of organics into CH_4 was investigated in detail and the findings are described in this paper.

The relationship between soluble COD (COD_S) reduction and CH_4 production was also examined by statistical approach.

2. Methods

2.1. Preparation of feedstock

Organic fraction of MSW (OFMSW) was used in the present experiment. Samples were collected from CSIR-NEERI colony. In laboratory, the waste sample was first crushed in a blender to reduce its particle size and then the sample was mixed thoroughly.

2.2. Experimental set-up

Batch experiments were carried out in a 2 L silicon septum anaerobic digester. In digester 1, OFMSW was taken as 200 g in 2000 mL water, and raw cow-dung slurry of about 50 g was incubated as active biomass. In digester 2, 100 g OFMSW was taken along with 1000 mL water and 1000 mL inoculum which was bought from a neighboring anaerobic digester plant. Digester 3 was used as control one in which raw OFMSW was taken as 200 g in 2000 mL of water.

After starting up, the digester was flushed with N_2 gas to ensure anaerobic condition in the headspace of the every digester. 30 mL sample was collected from both the digesters on every 3 days interval. All physico-chemical parameters with biogas volume and composition were analyzed. Biogas volume was measured by water displacement method. The volume of the displaced water is equivalent to the biogas produced in the digester. The experiment was conducted under room temperature (38 ± 2 °C). Inoculum obtained from an operating anaerobic digester was also added into the digesters.

2.3. Determination of physico-chemical properties of the sample

The pH, COD, TS, VS, VFA of the homogenized MSW mixture and the biogas volume and composition were estimated in order to analyze initial anaerobic conditions. Proximate analysis of MSW includes parameters, such as moisture content (MC), VS, fixed carbon and ash following standard methods (US EPA, 2009). The pH of the digesters was recorded from digital probes and the remaining parameters were analyzed following standard procedure. The MC, TS, VS was analyzed by gravimetric method. COD was analyzed by closed reflux method and VFA was analyzed by titration method following standard methods (APHA, 2005). CH_4 contents in the biogas were determined by Gas Chromatograph (Shimadzu GC2010) which was equipped with a thermal conductivity detector (TCD) and CH_4 standards (75%, 50%).

2.4. Statistical analysis

Linear regression analysis was applied to establish the relationship between COD (y) and the volume of CH_4 and pH (X_1 and X_2).

A data set of $\{y_i, x_i, \ldots\ldots, x_{ip}\}_{i=1}{}^n$ n statistical units, a linear regression model assumed that the relationship between the COD and the p-vector of regressor *i.e.*, volume of CH_4 and pH are is multiple linear. This relationship was modeled through a disturbance term or error variable ε_i, an unobserved random variable that adds noise to the linear relationship between the dependent variable and regressors. Thus, the model represented in the form as given in Eq. (1).

$$y_1 = \beta_1 x_{i1} + \ldots + \beta_p x_{ip} + \varepsilon_i = X_i^T\beta + \varepsilon_i \quad i = 1, \ldots, n \tag{1}$$

where T denotes the transpose, so that $X_i^T\beta$ is the inner product between vectors x_i and β.

n equations are stacked together and written in vector form as given in Eq. (2).

$$Y = X\beta + \varepsilon \tag{2}$$

The SS Regression is the variation explained by the regression line. The F-statistic is calculated using the ratio of the mean square regression (MS Regression) to the mean square residual (MS Residual). This can then be compared with the critical F value for 7 and 48 degrees of freedom (available from an F-table) to test the null hypothesis:

H_0: $\beta_1 = \beta_2 = \beta_3 = \beta_4 = \beta_5 = \beta_6 = \beta_7 = 0$

The p-value associated with the calculated F-statistic is probability beyond the calculated value. Comparing this value with 5%, for example, indicates rejection of the null hypothesis.

A statistical analysis was performed to support the relationship between CH_4 production and pH with COD_S reduction in AD, where, n and β denoted as degree of freedom (24) and COD removal efficiency, respectively. All the statistical evaluation was carried out using Microsoft Excel 2007.

3. Results and discussion

3.1. Characteristics of feedstock

The pH in digesters 1, 2 and 3 was initially low, which might be due to the formation of acids by acidogenic bacteria. However, it was increasing as the time of incubation increased. This might be due to digestion of VFA and nitrogen compounds through methanogenic bacteria (Mahanta et al., 2004).

The pH was in the range 6.4 and 7.5 in all the set-up. A neutral pH is favorable for biogas production as most of the methanogens grow best in the pH range 6.7–7.5 (Deublein and Steinhauser, 2008).

The characteristics of feedstocks as well as inoculum and raw OFMSW is presented in Table 1. The TS content of all the digesters (1, 2 and 3), inoculum and raw OFMSW were found to be 7.4%, 8.2%, 6.5%, 1.9% and 24.7%, respectively. The TS of digester obtained for biogas production is in the range 7–9%. Igoni et al. (2008) reported that TS content in waste feedstock would be between 4–10% and the percentage TS of MSW in a continuous AD process increases. Yavini et al. (2014) also reported that the amount of gas generated from these substrates decreases by decreasing and increasing the percentage TS concentration below and above the optimum value of 9% TS. The maximum volume of biogas generated for three substrates was 325, 468 and 680 mL in groundnut shell, maize cobs and rice straw, respectively.

The VS content of raw OFMSW, inoculum, and all the digesters (1, 2 and 3) were 78.6%, 76.9%, 84.3%, 94.2% and 80.4%, respectively. Potential of gas production can usually be estimated from the VS loading of the digester and the percentage of VS reduction through digestion. Waste characterized by a high concentration of VS is best suited to AD (Kayhanian, 1995). High TS and VS reduction showed higher specific biogas yield and a strong relationship between specific biogas yield and TS reduction ($R^2 = 0.97$) and VS reduction ($R^2 = 0.96$) has been found (Haider et al., 2015).

COD concentration was 25.7, 38.1 and 20.7 g/L in all the digesters 1, 2 and 3, and COD of raw OFMSW and inoculum was 21.4 g/kg and 35 g/L, respectively.

3.2. Digester's performance

3.2.1. Effect of pH

In digesters 1, 2 & 3 the final pH observed after 27 days of the start of the experiment was 6.9, 6.4, and 7.3, respectively. The pH profile of digesters 1, 2 and 3 during the time course study of the experiment is shown in Fig. 1. Maximum pH of 7.5 was observed in digester 3 throughout the study period. In digester 1, the final pH was observed as 6.4 which was found to be lesser than the final pH of digester 2, which shows that AD process inhibited due to higher VFAs concentration. During the course of the experiment, pH level in all the digesters were found to be increased but with different magnitudes.

During AD process, continuous hydrolysis of solid waste and the microbial conversion of biodegradable organic contents might lead to the production of intermediate VFAs at high concentrations, which could inhibit methanogenesis owing to the accumulation of VFAs and low pH (Sponza and Agdag, 2004; Ledakowicz and Kaczarek, 2002). Few researchers were in the opinion that optimum pH of one stage and two stage continuous stirred-tank reactor (CSTR) are 6.6–7.2, 3.2–5.5 (1st stage) 6.7–7.2 (2nd stage) (Ariunbaatar et al., 2015). Also, the pH of 7.5 was reported and CH_4 production was improved effectively by changing pH at the beginning of co-digestion of kitchen waste and cow-dung (Zhai et al., 2015).

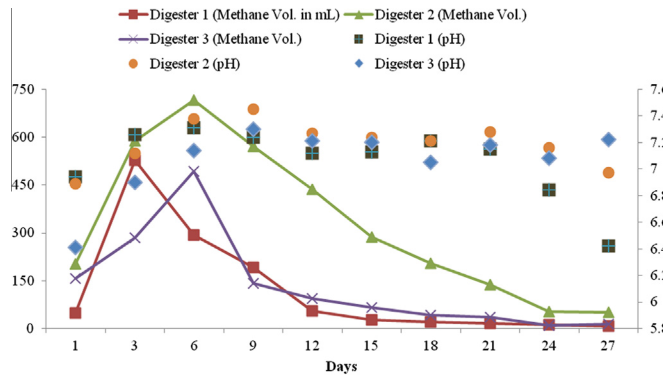

Fig. 1. Methane volume vs. pH in digesters 1, 2, and 3.

Table 1
Characteristics of feedstock in different digesters.

Sample name	MC (%)	TS[#] (%)	TVS[#] (%)	COD_T (g/L)	COD_S (g/L)
Raw OFMSW	75.3	24.7	78.6	21.3[*]	–
Inoculum	98.1	1.9	76.9	35.0	17.4
Digester 1	92.6	7.4	84.3	25.7	10.4
Digester 2	91.8	8.2	94.2	38.1	15.2
Digester 3	93.5	6.5	80.4	20.7	5.1

[#] Dry basis.
[*] g/kg.

3.2.2. Effect of retention time

The optimum retention time was ranged between 5 and 13 days in all the digesters. In digester 1, 4 day was optimum retention time while in digester 2, it was 7 days and it was 5 days for digester 3. Daily mixing of each feedstock during the experiment to pungent the biogas that forms and to prevent the formation of dry and inactive layers can also control the optimum retention time. It was observed that about 81% of biogas was produced in first 15 days in digester 2. Volumes of CH_4 and retention time of different digesters are shown in Fig. 2.

3.2.3. Biogas profile

The cumulative CH_4 production in all the digesters is presented in Table 2. Cumulative biogas and CH_4 production in digester 1 was observed as 10.7 L and 3.6 L, respectively whereas in digester 2, it was 17.7 L and 9.2 L, and 9.3 L and 3.2 L was observed in digester 3, respectively. Cumulative biogas and CH_4 production was higher in digester 2 than digester 1 and 3 due to the co-digestion of OFMSW with inoculum exceeding to high concentration of COD.

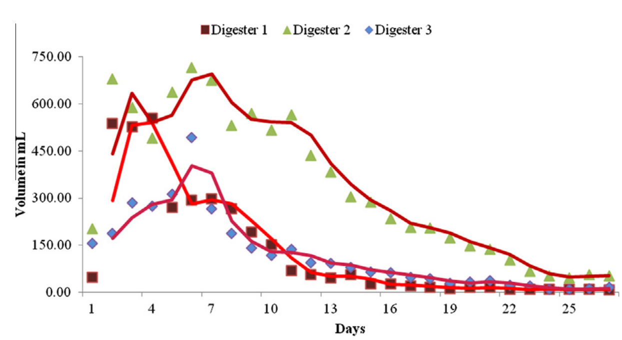

Fig. 2. Comparison of methane production in digesters 1, 2 and 3.

Table 2
Biogas production profile.

Different digesters	Cumulative biogas production (L)	Biogas yield (mL/g VS removal)	Cumulative CH_4 production (L)	CH_4 yield (mL/g VS removal)
Digester 1	10.7	303.4	3.6	101.1
Digester 2	17.7	323.2	9.2	168.4
Digester 3	9.3	276.8	3.2	84.3

3.2.4. VFA profile with respect to CH_4 production

It was observed that VFAs removal efficiency in Digester 2 was the best among all the digesters. The VFA profiles of all the digesters are shown in Fig. 3.

3.2.5. COD and VS removal with respect to biogas & CH_4 production

In digester 2, the maximum COD_S reduction efficiency was 84.2% followed by 77.9% in digester 1. Reduction profile of COD and the performance of digester 2 proved its superiority as compared to the digester 1. COD removal with respect to CH_4 production is shown in Fig. 4. The VS reduction was observed as 60.7% and 55.7%. From the experimental values, it was observed that biogas yield had a positive correlation with VS and COD reductions. Small increments in VS and COD reduction were observed with an increase in biogas production. The relationship between VS reduction with the production of CH_4 in digesters 1 & 2 is shown in Fig. 5.

It was reported that specific methane yields (SMY) of 166 and 194 L CH_4/kg VS fed from mesophilic (35 °C) dry (TS 15.2%) and wet (TS 7.7%) anaerobic digestion (DAD and WAD) of cow dung during 63 days of incubation, respectively whereas for the same incubation period, thermophilic (55 °C) operation yielded 188 and 226 L CH_4/kg VS fed for dry and wet operation, respectively. This was fed at an OLR of 10.7 g VS/kg inoculum/day (Jha et al., 2013). Jha et al. (2012) also reported SMY of 127.2 L CH_4/kg VS of cow-dung digested in mesophilic (35 °C) unmixed batch reactor during 35 days. They found that the mixing increased SMY and VS removal by 7.5% and 9.7%, respectively. Brown et al., 2012 concluded that volumetric productivity of DAD (18% TS) is 2–7 times greater as compared to WAD (5% TS) for lignocellulosic substrates based on 30 days batch incubation.

In the present case, CH_4 yield has been found to be 101.1, 168.4 and 84.3 mL/g VS removal, respectively in digesters 1, 2 and 3

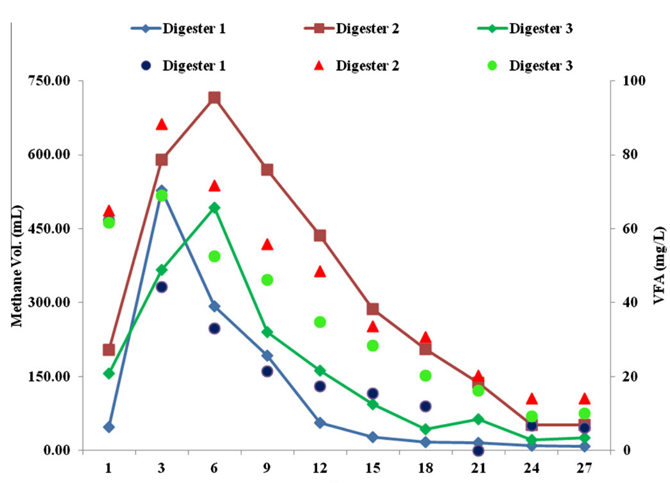

Fig. 3. VFA profile with methane production in digester 1, 2 and 3.

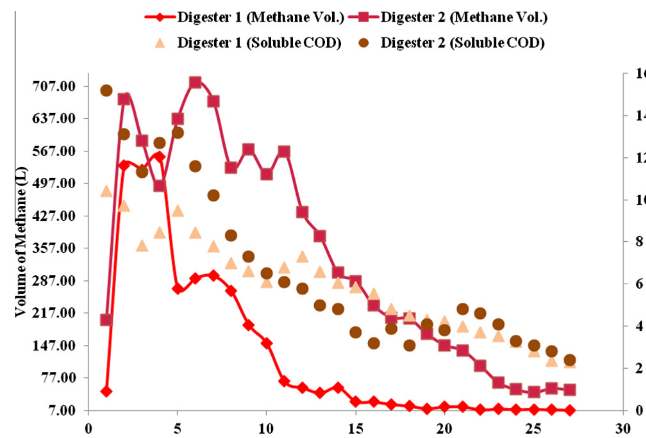

Fig. 4. Profile of methane production and COD_S in digesters 1 and 2.

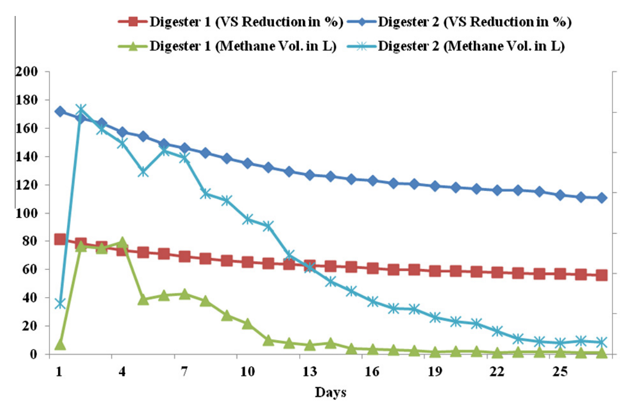

Fig. 5. Methane production v/s VS reduction in digester 1 and 2.

which corroborates the results observed by the above researchers (Jha et al., 2012, 2013; Brown et al., 2012).

3.2.6. Statistical analysis of results in both the digesters

Digester 1: The proportion of variation in COD removal is explained by variation in CH_4 volume, which is estimated by the co-efficient of determination (R^2). R^2 value of 0.67 was obtained indicating a very good fit. 67% of the variation in COD removal was explained by the independent CH_4 volume and pH in digester 1. The value of significance *F* was obtained as 0.021 which is less than 0.05. Hence, it is predicted that COD_S reduction is dependent on the independent CH_4 volume and pH in AD system. The *p*-value of the hypothesis test *i.e.*, H_0: $\beta_1 = 0$ is 0.013, which is less than 0.05 for intercept. Both CH_4 volume and pH was estimated to predict the reduction of COD_S, but CH_4 volume had good correlation with COD removal, because *p* value of CH_4 volume was lower than the pH ($0.017 < p < 0.018$).

Digester 2: Co-efficient of determination (R^2) value of 0.74 was obtained which indicates a very good fit. 74% of the variation in COD removal was explained by the independent CH_4 volume and pH in digester 2. The value of significance *F* was obtained as 0.009 which is less than 0.05. Hence, it is predicted that COD_S reduction is dependent on independent CH_4 volume and pH in AD system. The *p*-value of the hypothesis test *i.e.*, H_0: $\beta_1 = 0$ is 0.012, which is less than 0.05 for intercept. Both CH_4 volume and pH was estimated to predict the reduction of COD_S but CH_4 volume had good correlation with COD removal because *p* value of CH_4 volume was lower than the pH ($0.0035 < p < 0.0130$). The summary of regression output is presented in Table 3. Regression residual plots are shown in Fig. 6.

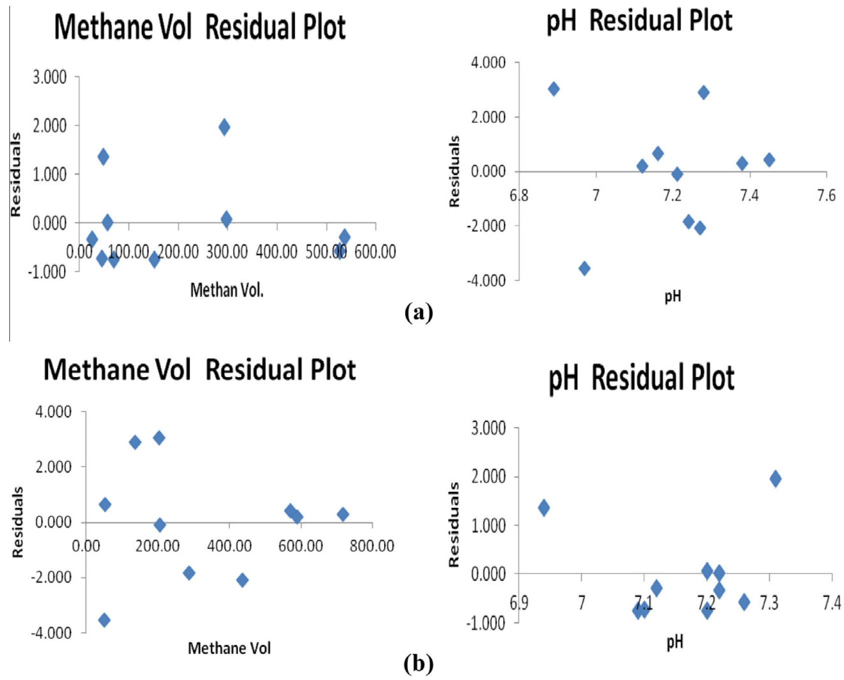

Fig. 6. Summary of multiple regression plot in (a) digester 1 and (b) digester 2.

Table 3
Summary of regression output for both the digesters 1 & 2.

Regression statistics of digester 1			Regression statistics of digester 2		
Multiple *R*		0.818	0.858		
R square		0.668	0.737		
Adjusted *R* square		0.574	0.661		
Standard error		1.062	2.348		
Observations		10	10		
	df	*SS*	*MS*	*F*	Significance *F*
ANNOVA results of digester 1					
Regression	2	15.923	7.961	7.054	0.021
Residual	7	7.901	1.129		
Total	9	23.824			
ANNOVA results of digester 2					
Regression	2	107.976	53.988	9.792	0.009
Residual	7	38.593	5.513		
Total	9	146.569			

4. Conclusion

AD for OFMSW is observed to be a feasible and effective solution to convert the waste mass into biogas production containing CH_4. Maximum CH_4 production was found in digester 2 with respect to organic loading, in which waste was co-digested. Maximum COD removal consequently revealed elevated amount of CH_4 and the data was also validated with statistical analysis. Detailed kinetic study including production and speciation of VFA during the digestion process and CH_4 generation is proposed to be investigated to optimize the organic loading rate in AD followed by application of statistical tools.

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