

## OPTIMIZATION OF ORGANIC LOADING RATE AND HYDRAULIC RETENTION TIME FOR MAXIMUM PRODUCTION OF METHANE THROUGH ANAEROBIC CO-DIGESTION OF CANOLA STRAW AND BUFFALO DUNG

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### ABSTRACT

In more than a few regions of Pakistan, crop residues are being wasted and are leading to a solid waste problem. Anaerobic co-digestion of crop residues along with animal dung is one of the appropriate method to convert wasted crop residues into the renewable energy. In the present study, the anaerobic co-digestion of canola straw long with buffalo dung was carried out in continuously stirred tank reactor (CSTR) for optimization of organic loading rate (OLR) and hydraulic retention time (HRT). The various organic loads were added in the range of 2-4 gVS L<sup>-1</sup>day<sup>-1</sup>, whereas various HRTs were kept from 20-35 days. Results reveal that on the increase of the OLR, the volumetric methane production increases, without significantly compromising methane yield. On the contrary, by increasing the HRT, the volumetric methane production decreases, whereas the specific methane production increases. The optimum OLR and HRT were observed as 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> and 30 days respectively, where maximum specific methane production was observed as 143 NmL g<sup>-1</sup>VS<sub>added</sub>. Moreover, the experimental data were simulated, which reveals that and the reaction kinetic follow the first order CSTR model efficiently.

**Keywords:** optimization, hydraulic retention time, organic loading rate, canola straw, buffalo dung.

### INTRODUCTION

Production of methane from agricultural waste is getting importance in recent years as it offers considerable environmental advantages (Chynoweth, 2004) and it is an additional source of earning for crop growers. For sustainable waste management, Asian countries are facing an uphill battle because of the increasing waste generation (Agamuthu *et al.*, 2007). In more than a few regions of Pakistan, crop residues including banana plant waste, cotton gin waste, rice straw, canola straw, sugarcane trash and cotton stalks are leading to a solid waste problem (Mahar *et al.*, 2012). On the other hand, there are around 70 million heads of buffaloes and cattle, and around 90 million heads of goats and sheep in Pakistan (Pandey, 2007). The ineffective management of the animal waste may lead to environmental hazards, such as water pollution and greenhouse gas emissions (Nasir, 2014). Anaerobic co-digestion (AD) of crop residues along with the animal dung could be a more suitable discarding way for the unexploited percentage of the crop residues and efficient management of animal dung considering especially the process stability, secondary contaminant generation (organic fertilizer), level of safety risk for workers and communities and noise levels near installation during operation (Mahar, 2010).

Financial competence of the effective AD process, not only driven by the investment and operating

cost of the biogas plant, but also significantly depends on the optimum plant parameters. In the previous study, the anaerobic co-digestion of the canola straw with buffalo dung was optimized for three parameters in batch experiments, which includes canola straw to buffalo dung ratio, alkaline chemical doze and canola straw particle size. It was observed that the most suitable canola straw to buffalo dung ratios based on volatile solids was 40:60, the most suitable alkaline chemical doze was 0.6 gNaHCO<sub>3</sub> g<sup>-1</sup>VS<sub>added</sub> and the most suitable canola straw particle size was 4.0 mm (Sahito *et al.*, 2014).

In order to operate the continuous AD plant, organic loading rate (OLR) and the hydraulic retention time (HRT) are principal parameters. OLR is the quantity of organic material added per unit volume of the AD reactor in a day. The mass of organic material can be expressed as volatile solids (Vesilind, 1998) or biological or chemical oxygen demand (Tchobanoglous *et al.*, 2003). Rate of methane generation is enormously dependent on the OLR. So as to get maximum yield from the specified size of the AD reactor, the OLR is to be kept as maximum, but at the greater concentration of OLR, the AD process may be inhibited because of the accumulation of volatile fatty acids that may cause a decrease of pH within the digester. HRT is an average time to which the feedstock remained inside the anaerobic digester (Rittmann and McCarty, 2001). Decrease in the HRT, upsurges the hazard of washout of the active bacterial population. On the contrary, the

increase in the HRT increases the capital cost of the reactor. In a similar way to the OLR, there should be an optimum HRT to keep the efficient operation of the AD plant.

In literature, no study was found regarding anaerobic co-digestion of canola straw along with buffalo dung in CSTR. Thus, the present study was carried out to optimize the OLR and HRT for the anaerobic co-digestion process using the canola straw and the buffalo dung as substrate. The optimization was carried out in continuous experiments by using the CSTR. Further, the kinetic coefficients at the varying OLRs for the co-digestion of canola straw and buffalo dung were also

estimated by using the first order CSTR model, and simulated for different HRTs.

## MATERIALS AND METHODS

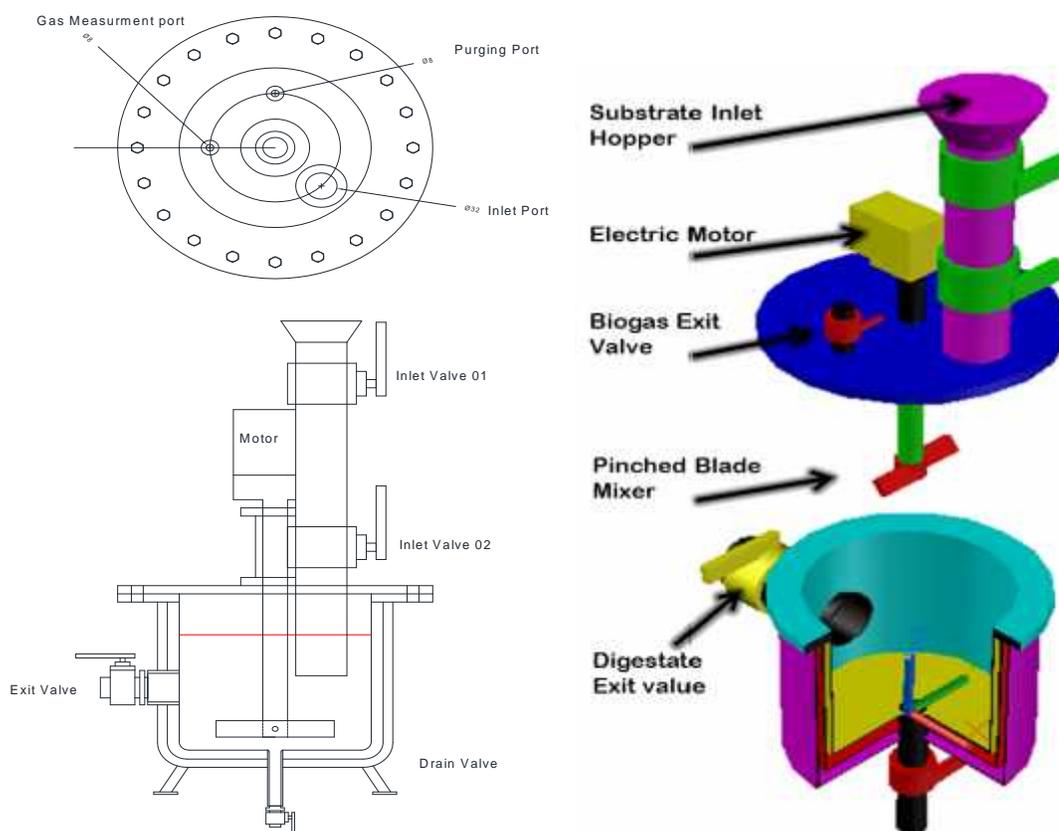
**Substrate characteristics:** The substrate used in the present study was the buffalo dung and the canola straw. The percentages of moisture content (MC), total solids (TS) and volatile solids (VS) were analyzed as per standard methods of (APHA, 1998), whereas the pH was analyzed by using the hydrogen ion sensitive pH meter. The substrate characteristics are given in Table 1.

**Table 1. Substrate characteristics**

Feedstock	pH	MC (%)	TS (%)	VS (% TS)
Buffalo dung	7.6 ± 0.1	80.50 ± 0.5	19.50 ± 0.5	70.05 ± 1
Canola straw	5.5 ± 0.1	6.56 ± 0.5	93.44 ± 0.5	89.08 ± 1

**Anaerobic digester:** Anaerobic digester used in the present study was the CSTR. The digester was a round container, made up of SS-304 and has the overall volume of 5.0 liters, beyond which working volume was 4.0 liters and the residual 1.0 liters was the headspace. The digester was designed as the triple shell as shown in Fig. 1. The

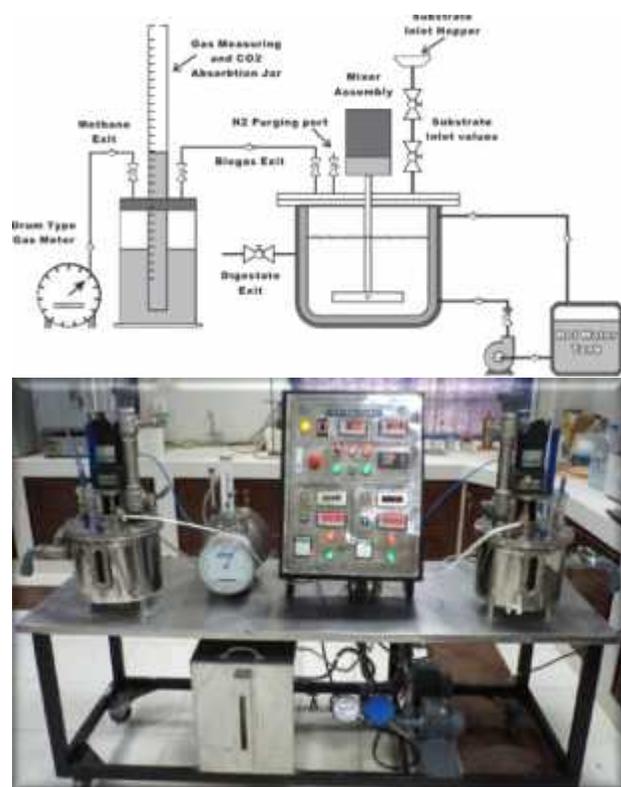
inner shell carries the substrate, while the middle shell carries the hot water. The hot water was heated through the electric element of 0.5 kW and circulated through the pump. So as to reduce loss of heat, the middle shell was enfolded by glass wool insulation.



**Fig. 1: Orthographic and sectional isometric projection of CSTR**

Digester has the top cover contains substrate inlet port, biogas withdrawal port, N<sub>2</sub> purging port and mixer shaft housing. The digester was air tightened by using butylene rubber O-ring between the top cover and digester flange. Additionally, a mechanical seal was used between the shaft and its housing. The twin pinched blade mixer was connected to the shaft that was driven by the motor and gearbox assembly. Double inlet valves were employed to avoid outflow of biogas from head space.

**Measurement of methane:** Biogas mainly composed of methane and carbon dioxide (CO<sub>2</sub>). In the present study setup, in order to get only the methane, the biogas was initially passed through the CO<sub>2</sub> absorption jar that carries a 3M NaOH solution. The quantity of methane was measured through drum type wet gas meter (Ritter TG-05). The schematic diagram and graphic view of developed CSTR setup is shown in Fig. 2.



**Fig. 2 Schematic diagram and graphic view of developed CSTR assembly**

The biogas was also analyzed in the gas chromatograph for its composition. Gas chromatographic analysis was done by using thermal conductivity detector and employing the capillary column. A sample of 500  $\mu$ L was charged by means of the gas tight micro syringe. As per the results of chromatographic analysis, biogas comprises of about 99% of methane. The measurement of methane was converted to normal pressure and temperature as proposed by Amon *et al.* (2007).

**Digester startup and its stability:** AD process was commenced with the addition of fresh buffalo dung and its digestate. The digestate was obtained from another laboratory scale CSTR, which was anaerobically treating buffalo dung at the temperature of 37 °C. The slurry was prepared with the ratio of 80:20 for fresh buffalo dung and its digestate correspondingly, and then total solid concentration was decreased to 5% by adding tap water. The slurry was filled in the AD reactor up to the volume of 4.0 liters. Subsequently, the AD reactor was linked with the CO<sub>2</sub> absorption jar followed by the drum type wet gas meter as presented in Fig. 2. The substrate was feed in the digester through the “substrate inlet hopper”, while the digestate was discharged through the “digestate exit”. So as to ensure anaerobic condition, reactor was purged with N<sub>2</sub> for about 5 minutes. The stirrer was adjusted at 60 rpm for mixing the substrate, and its timer was set to 5 minutes and 30 minutes as running and resting times respectively. After seven days of a startup, the digester was charged on a daily basis with the slurry of fresh buffalo dung. The TS concentration in the digester was constantly kept at 5%.

The steady state condition of the digester was tested by frequently examining the digestate for pH, total alkalinity (TA) and volatile fatty acids (VFA). The AD process is stable at TA in the range of 2000 to 18000 mg CaCO<sub>3</sub>L<sup>-1</sup> of (Cuetos *et al.*, 2008; Gelegenis *et al.*, 2007); pH in the range of 6.5 to 7.5 (Stronach *et al.*, 1986); and the ratio of VFA to TA of 0.5 (Lin *et al.*, 2009). The pH was measured with a hydrogen ion sensitive electrode, whereas the TA and VFA were investigated rendering to the Standard Methods (APHA, 1998).

**Optimization of OLR And HRT:** After achieving the steady state condition of the digester, the OLR optimization of the canola straw with the buffalo dung was started. The OLRs used in the present study are given in the Table 2. The OLRs used in the present study were in the range of 2 to 4 gVS L<sup>-1</sup>day<sup>-1</sup>. So as to make the slurry of required OLR, the ratio of the canola straw and buffalo dung was set as per results of the previous study, i.e. 40:60 based on VS (Sahito *et al.*, 2014). The HRT was fixed to 20 days. On the pre-fixed HRT and pre-specified volume of digester, the total volume of inlet slurry was calculated as 200 mL. In correspondence to the range of OLR and the inlet volume of the slurry, the TS concentration was kept in the range of 5.1 to 10.2 % and presented in the Table 2. At the HRT of 20 days and OLR of 5 gVS L<sup>-1</sup>day<sup>-1</sup>, the TS concentration was 12.76%. Because of the constrain of about 10% of TS concentration of the inlet feedstock that a pump can handle, the experiments in the present study were restricted to OLR of 4 gVS L<sup>-1</sup>day<sup>-1</sup> (Hobson *et al.*, 1981; Igoni *et al.*, 2008). Furthermore, each OLR was charged after successful digestion of 28 days.

**Table 2. Various OLRs for co-digestion of canola straw and buffalo dung.**

OLR (gVS L <sup>-1</sup> day <sup>-1</sup> )	HRT (day)	Canola Straw (g)	Buffalo Dung (g)	TS (%)
2	20	3.771	34.278	5.1
3	20	5.657	51.418	7.7
4	20	7.542	68.557	10.2

Afterwards, the various HRTs were used by maintaining the TS concentration to about 10%. The HRTs used in the present study are presented in the Table 3. The HRTs were in the range of 20 to 35 days, whereas the corresponding OLRs were 4 to 2.29 gVS L<sup>-1</sup>day<sup>-1</sup>. Furthermore, each HRT was changed after successful digestion of 15 days.

**Table 3. Various HRTs for co-digestion of canola straw and buffalo dung**

HRT (day)	OLR (gVS L <sup>-1</sup> day <sup>-1</sup> )	Canola Straw (g)	Buffalo Dung (g)	TS (%)
20	4.00	7.542	68.557	10.21
25	3.20	6.034	54.846	10.21
30	2.66	5.015	45.590	10.21
35	2.29	4.310	39.175	10.21

**Calculating kinetic coefficient:** The universal material balance equation for an AD reactor is “Accumulation = Inflow – Outflow + Generation”. A perfect CSTR is always in steady state condition, i.e. the rate of substrate inflow equals to the rate of digestate exit, and thus the variation of rate of substrate regarding time is zero. As per the Linke (2006), the HRT of the CSTR can be represented by Eq. 1.

$$HRT = \frac{1}{k} \left( \frac{G_{max}}{G_{max} - G(t)} - 1 \right) \quad (1)$$

Where  $G(t)$  is the methane production at any time  $t$  in NmL;  $G_{max}$  is the maximum quantity of methane in NmL and  $k$  is the first order kinetic coefficient in day<sup>-1</sup> (Mata-Alvarez and Labrés, 1992). At the given HRT, Eq. 1 was used to estimate the value for  $k$  for the co-digestion of the crop residue and buffalo dung. As per the results of the previous study (Sahito *et al.*, 2014), the  $G_{max}$  from the co-digestion of canola straw and buffalo dung, combined at the optimized ratio of 40:60 based on VS; and using the canola straw particle size 4 mm was 172 NmL g<sup>-1</sup>VS<sub>added</sub>. Eq. 1 can be repositioned as Eq. 2, where the ratio  $G(t)/G_{max}$  is the efficiency of the AD process (Linke, 2006) and here it is denoted by  $y$ . Eq. 2 was used to establish the relation between the HRT and the efficiency of the AD process.

$$y = \frac{G(t)}{G_{max}} = \frac{1}{1 + \frac{HRT \times k}{k_1}} \quad (2)$$

**Statistical Analysis:** The estimated values of  $k$  were confirmed by calculating the error between the experiments and calculated values of methane. Three types of errors were calculated namely, standard deviation of residuals (*SDR*), mean absolute error (*MAE*) and mean bias error (*MBE*). The *SDR* was calculated by using Eq. 3 (Sahito *et al.*, 2013) and is a measure of the average variance between methane ascertained experimentally ( $G_{asc}$ ) and the methane calculated ( $G_{mod}$ ) by using a mathematical model. In Eqs. 3-5,  $n$  represents the number of data days. The *MAE* was calculated by using Eq. 4, while the *MBE* by Eq. 5 (Parikh *et al.*, 2007; Shen *et al.*, 2010).

$$SDR = \sqrt{\frac{\sum_{i=1}^n (G_{asc} - G_{mod})^2}{n}} \quad (3)$$

$$MAE = \frac{1}{n} \sum_{i=1}^n |G_{mod} - G_{asc}| \times 100 \quad (4)$$

$$MBE = \frac{1}{n} \sum_{i=1}^n (G_{mod} - G_{asc}) \times 100 \quad (5)$$

Further, the optimum experimental HRT and OLR were also simulated by using the first order CSTR model. In order to simulate the HRT and OLR, first interpolation was carried out to determine the kinetic coefficient at the optimum OLR by using Eq. 6.

$$k_2 = \frac{(OLR_2 - OLR_1)k_3 + OLR_1k_1}{OLR_2 - OLR_1} \quad (6)$$

Where  $k_2$  is the unknown kinetic coefficient at the optimum  $OLR_2$ ;  $k_1$  and  $k_3$  are the kinetic coefficients estimated by using Eq. 1 at the  $OLR_1$  and  $OLR_3$  respectively.

## RESULTS AND DISCUSSION

**Optimized OLR and HRT:** The cumulative and specific methane production and pH, TA and VFA of the digestate from the co-digestion of canola straw and buffalo dung at the different OLRs are represented in Fig. 3. The results are in accordance with the interpretations of the Lindorfer *et al.* (2008) that by increasing OLR, the volumetric methane production increases without compromising specific methane yield. Wang *et al.* (2013) had also observed that by increasing VS load, initially biogas yield increased and then becomes stable.

Moreover, the dissimilarity in the cumulative methane was detected, which might be because of the oscillations of making of food for methanogenic microorganisms (Song *et al.*, 2013).

The maximum mean specific methane production was detected from 4 gVS L<sup>-1</sup>day<sup>-1</sup> i.e., 101 NmL g<sup>-1</sup>VS<sub>added</sub> followed by 96 and 98 NmL g<sup>-1</sup>VS<sub>added</sub> from 2 and 3 gVS L<sup>-1</sup>day<sup>-1</sup> correspondingly. The optimum OLR is that beyond, methane yield will not be increased (Yadvika *et al.*, 2004). In the present study, the pH of the digester was 7.0, which upsurges to 7.3 by addition of pre-optimized doze of the NaHCO<sub>3</sub> i.e., 0.6 g NaHCO<sub>3</sub>g<sup>-1</sup>VS (Sahito *et al.*, 2014). Through the charging of second OLR i.e., 3 gVS L<sup>-1</sup>day<sup>-1</sup>, the pH value was increased to 7.4 and thus to maintain the pH, the amount of NaHCO<sub>3</sub> added was reduced to 0.5 g in the set slurry of 200 mL (i.e., 0.03125 g NaHCO<sub>3</sub>g<sup>-1</sup>VS).

Throughout the first few days of 2 gVS L<sup>-1</sup>day<sup>-1</sup>, the TA in the AD reactor was lower than 1500 mg CaCO<sub>3</sub>L<sup>-1</sup> that was increased to 2000 mg CaCO<sub>3</sub>L<sup>-1</sup>. At the second OLR, the TA was increased beyond 2500 mg CaCO<sub>3</sub>L<sup>-1</sup> and then reduced to around 2000 mg CaCO<sub>3</sub>L<sup>-1</sup> as the quantity of NaHCO<sub>3</sub> was decreased. The values of TA at the OLR of 4 gVS L<sup>-1</sup>day<sup>-1</sup> were kept almost constant, which showed the steadiness of the process. The VFA at 2 gVS L<sup>-1</sup>day<sup>-1</sup> was ranging from 120 to 240 mg CH<sub>3</sub>COOH L<sup>-1</sup>. This wide range of VFA was because of the lower quantity of TA in the AD reactor and later VFA was stabilized. Furthermore, throughout the 84 days of OLR optimization, the VFA to TA ratio was lower than 0.2 and consequently expressing AD reactor as under loaded (Lin *et al.*, 2009).

The cumulative and specific methane production and the pH, TA and VFA of the digestate from the co-digestion of canola straw and buffalo dung at the different HRTs are represented in Fig. 4. Outcomes reveal that with the increase of HRT, the volumetric methane production decreases, whereas the specific methane production increases. The production of methane was significantly influenced by HRT of the reactor which is in accordance to the observations of Takin and Dalgic (2000). For the HRT of 20 and 25 days, the pH diverse in the range of 7.2 to 7.3, but for the HRT of 30 and 35 days the pH persisted to 7.3. The TA was in the range of 2000 to 2175 mg CaCO<sub>3</sub>L<sup>-1</sup> for all the HRTs that represents the steady state condition of the AD reactor. Moreover, in the first few days of, the VFA were higher as 240 mg CH<sub>3</sub>COOH L<sup>-1</sup>. The VFA were almost constant to 180 mg CH<sub>3</sub>COOH L<sup>-1</sup> and then reduce to 120 mg CH<sub>3</sub>COOH L<sup>-1</sup> at the HRT of 35 days. However, during the period of HRT optimization (60 days), the VFA to TA ratio was lower than 0.2 and it represents that the digester was not overloaded (Lin *et al.*, 2009).

Most of the energy crops cannot be digested completely at HRT less than 20 days (Wolf, 2013). In present study results show that maximum mean specific methane was detected from HRT of 30 days i.e., 143 NmL g<sup>-1</sup>VS<sub>added</sub> followed by 138, 124 and 103 NmL g<sup>-1</sup>VS<sub>added</sub> from HRT of 35, 25 and 20 days, respectively. Thus, the HRT of 30 days is the optimum for the co-digestion of the canola straw and the buffalo dung. On the other hand, the corresponding OLR is 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> is the optimum value. The optimized OLR is in agreement with the findings of Wolf (2013) that the OLR of 2.5 kgVS m<sup>-3</sup>day<sup>-1</sup> is the utmost appropriate value for the anaerobic co-digestion process. At the higher OLR the decrease in methane potential may be due to inadequate quantity of inoculum, as suggested by Eskicioglu and Ghorbani (2011).

**Kinetics of the co-digestion of canola straw and buffalo dung:** The calculated values of *k* at dissimilar OLRs for co-digestion of canola straw and buffalo dung along with errors are represented in the Table 4. The coefficient *k* was in the range of 0.093 to 0.112 day<sup>-1</sup>, while the corresponding *SDR* values were low and in the range of 3 to 7 NmL g<sup>-1</sup>VS<sub>added</sub>. Regarding *MAE*, the values of *k* calculated from the data at OLR of 2 gVS L<sup>-1</sup>day<sup>-1</sup> and 3 gVS L<sup>-1</sup>day<sup>-1</sup> have higher correctness than to 4 gVS L<sup>-1</sup>day<sup>-1</sup>. Concerning the OLR of 2 gVS L<sup>-1</sup>day<sup>-1</sup> and 4 gVS L<sup>-1</sup>day<sup>-1</sup>, the consistent *k* values underestimates the methane yield, while the *k* values from the OLR of 3 gVS L<sup>-1</sup>day<sup>-1</sup> overestimates.

An interactive consequence of HRT on the efficiency of the co-digestion of canola straw and buffalo dung at the different OLRs is shown in Fig. 5. Results reveal that the efficiency of AD process was improved with increasing HRT and highest efficiency was achieved at the OLR of 3 gVS L<sup>-1</sup>day<sup>-1</sup>. On the other hand, at the OLR of 2 and 4 gVS L<sup>-1</sup>day<sup>-1</sup>, the efficiency of the AD process was decreased. From the experimental data, it was observed that the optimum HRT and OLR for the maximum methane production from the co-digestion of canola straw and buffalo dung were 30 days and 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> respectively. This observation was validated by interpolating the value of the kinetic coefficient at the OLR of 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> and is given in the Table 4. The efficiency line of the 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> is very near to the efficiency line of 3.0 gVS L<sup>-1</sup>day<sup>-1</sup> (Fig. 5). Moreover, the values of *SDR*, *MAE* and *MBE* considering the methane production data of OLR of 3.0 gVS L<sup>-1</sup>day<sup>-1</sup> are also lower for OLR of 2.66 gVS L<sup>-1</sup>day<sup>-1</sup> (Table 4). Thus, the estimated kinetic coefficients are supporting the optimized OLR that was obtained experimentally. Furthermore, it also establishes that the co-digestion of canola straw and buffalo dung follows the first order CSTR model efficiently.

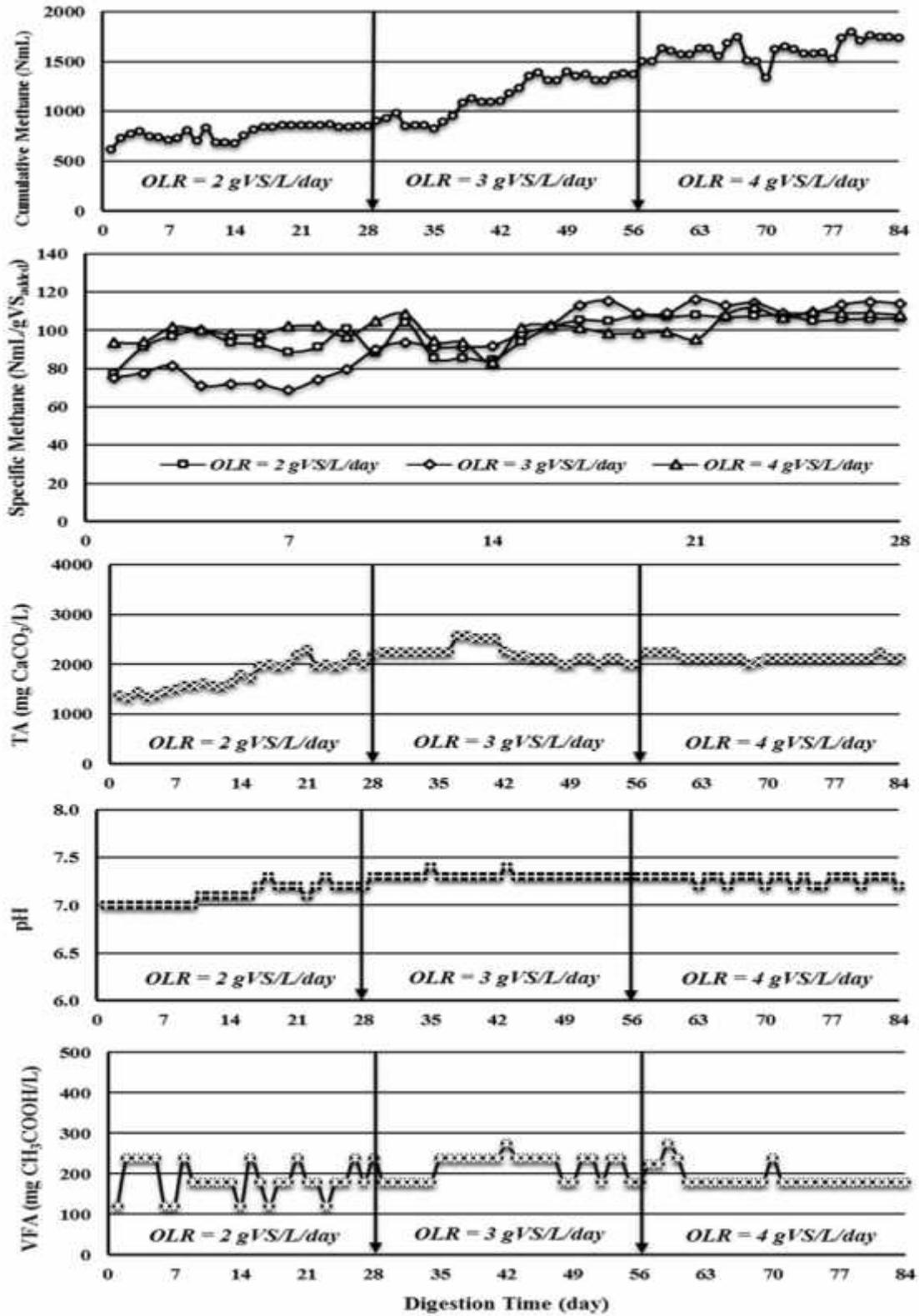


Fig. 3. The cumulative and specific methane production and pH, TA and VFA of digestate from co-digestion of canola straw and buffalo dung at different OLRs

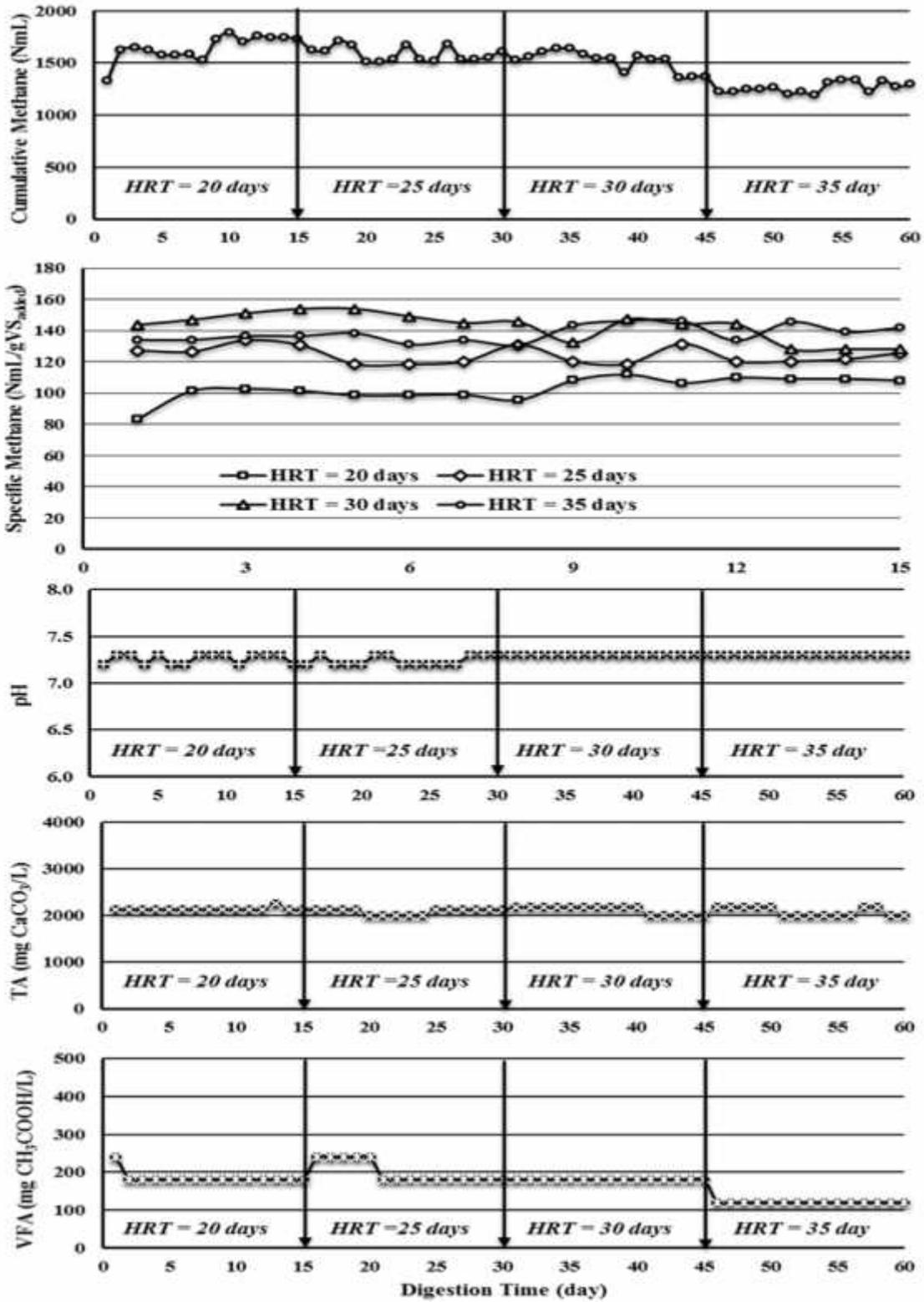


Fig. 4. The cumulative and specific methane production and pH, TA and VFA of digestate from co-digestion of canola straw and buffalo dung at different HRTs

Table 4. Kinetic coefficients at various OLRs

OLR (gVS L <sup>-1</sup> day <sup>-1</sup> )	HRT (day)	k (day <sup>-1</sup> )	SDR (NmL g <sup>-1</sup> VS <sub>added</sub> )	MAE (%)	MBE (%)
2	30	0.095	3.76	2.79	-0.03
3	30	0.112	3.04	2.64	2.20
4	30	0.093	6.89	5.56	-5.56
2.66 (Interpolated)	30	0.106	2.01	1.39	0.60

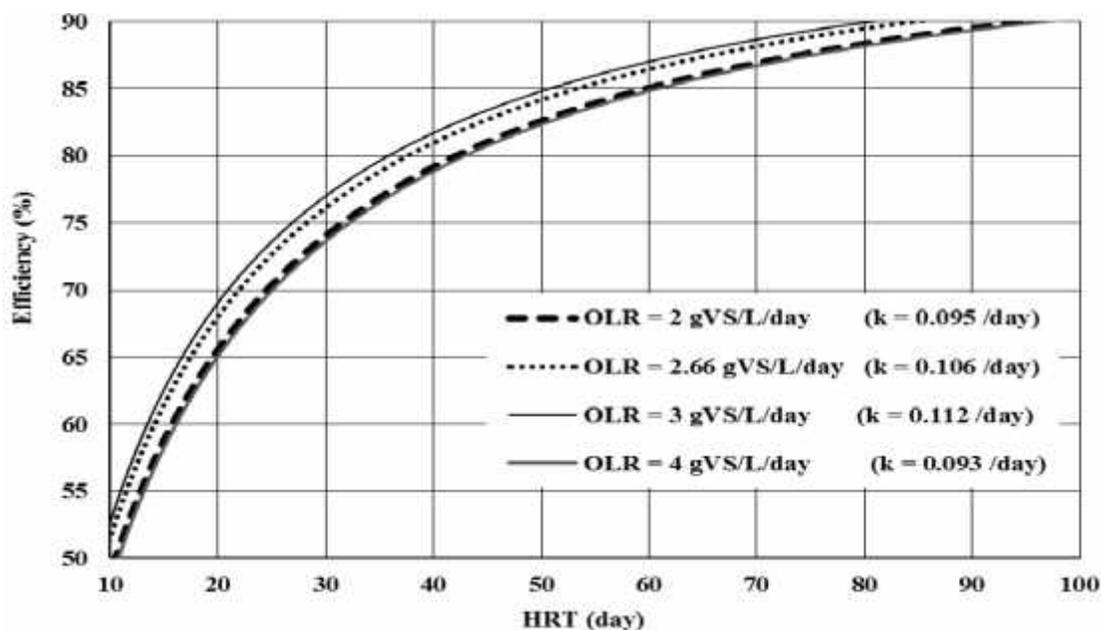


Fig. 5. An interactive effect of HRT on efficiency of the co-digestion of canola straw and buffalo dung at the different OLRs

**Conclusions:** The OLR and HRT, both has significant effect on the methane yield from the anaerobic co-digestion of the canola straw along with buffalo dung. The optimum value of OLR was 2.66 gVS L<sup>-1</sup>day<sup>-1</sup>, while the optimum HRT was 30 days. In order to maintain the steady state condition of the reactor, only 0.03125 g NaHCO<sub>3</sub> g<sup>-1</sup>VS is required, which is 95% lower to the quantity of NaHCO<sub>3</sub> used at startup. Additionally, the co-digestion of canola straw and buffalo dung follows the first order CSTR model efficiently.

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