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ENHANCING METHANE PRODUCTION FROM RICE STRAW CO-DIGESTED WITH BUFFALO DUNG BY OPTIMIZING EFFECT OF SUBSTRATE RATIO, ALKALINE DOZE AND PARTICLE SIZE

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ABSTRACT

The purpose of this study was to enhance the methane generation from the rice straw by anaerobically co-digestion with the buffalo dung. The study was carried out into three phases and it includes determination of ratio of rice straw and buffalo dung, specific mass of sodium bicarbonate ($NaHCO_3$) as buffer and alkaline pretreatment agent and appropriate size of the rice straw particle size that yields maximum methane. The higher generation of methane from anaerobic co-digestion of rice straw and buffalo dung could be achieved by taking the ratio of rice straw and buffalo dung as 3:7 on the basis of volatile solids, the specific mass of $NaHCO_3$ of 0.5 g/gVS and the rice straw particle size of 2 mm. Considering the above parameters, methane generation was obtained about 184 NmL/gVS at anaerobic biodegradability of about 48%. By using particle size of 4mm, methane generation was obtained about 172 NmL/gVS. However, because of about double energy consumption to produce rice straw of particle size of 2 mm than to the particle size of 4 mm, thus on the basis of energy consumption later one is suggested as appropriate particle size.

Keywords: methane, buffalo dung, rice straw, specific mass of sodium bicarbonate, particle size.

INTRODUCTION

In Pakistan various crop generates a huge amount of residue and because of the improper management, its considerable quantity is being wasted that leads to a solid waste disposal problem (Mahar *et al.*, 2012). Out of the wasted crop residues, rice straw is among the unexploited crop residues from energy point of view. The co-digestion of crops and their byproducts with the animal dung is being obtaining more attention because of the energy obtained from them is carbon neutral and is the best alternative to the fossil fuels (Sahito *et al.*, 2012). Methane generation from crops and their byproducts has considerable economic and environmental advantages. Additionally, conversion of animal waste into biogas decreases methane emissions, which is caused because of the dung storage. In previous study, it was recommended that the anaerobic digestion of the crop residue could be a disposal way for conversion of the wasted crop residues into the energy (Mahar, 2010).

Crop residues consist of three different types of polymers including cellulose, hemicellulose and lignin. Among the three, lignin is a most complex chemical compound. It improves the mechanical strength of the cell wall and is difficult to degrade (Sahito *et al.*, 2013). Pretreatment of crops' residue improves the digestibility and could be done mechanically by reducing the particle size of the crop residue, thermally by supplying heat to the substrate, chemically by adding some alkaline or acidic chemical, or biologically by employing certain microorganisms (Liew *et al.*, 2011). Mechanical and biological pretreatment processes are cost extensive (Lin *et al.*, 2009), but somewhat mechanical pretreatment is essential in order to even the substrate inflow and outflow within the anaerobic digestion reactor. Out of the thermal and chemical pretreatment processes, alkaline pretreatment is the most feasible process as it increases internal surface area, causes destruction of the structure of lignin, reduces the degree of inhibition during anaerobic digestion process and does not require any energy (Kumar *et al.*, 2009; Ward *et al.*, 2008). Moreover, in anaerobic digestion process adequate alkalinity is necessary in order to control the pH. The methane producing bacteria need bicarbonate alkalinity, and among the chemicals that release bicarbonate alkalinity, sodium bicarbonate ($NaHCO_3$) is the best chemical (Gerardi, 2003). Besides, the $NaHCO_3$ not only provides the bicarbonate alkalinity, but it also works as the pretreatment chemical.

The objective of this study was to enhance the methane production from the rice straw by anaerobically co-digestion with the buffalo dung. The study was carried out into three phases and includes determination of ratio of rice straw and buffalo dung, specific mass of $NaHCO_3$ as buffer and alkaline pretreatment chemical and appropriate size of the rice straw particle size that yields maximum methane generation. Moreover, anaerobic biodegradability and energy to reduce the particle size of the rice straw were also considered as the assessment criteria.

MATERIALS AND METHODS

Features of the substrates: The features of the substrates used in the present study i.e. rice straw and buffalo dung including weight fractions of carbon (C), oxygen (O), hydrogen (H), nitrogen (N) and sulfur (S), percentages of total solids (TS), moisture content (MC) and volatile solids (VS) and pH values were found out as stated in a previous study (Sahito *et al.,* 2013). Moreover, the bulk density of the each particle size of rice straw was also determined by dividing mass of rice straw with its volume.

Experimental protocol: This study was carried out in three phases, to enhance the methane generation from the co-digestion of rice straw and buffalo dung. All the three phases are illustrated in Fig. 1. In the first phase, the ratio of rice straw to buffalo dung was taken into consideration and was aimed that at which ratio of rice straw to buffalo dung, the maximum methane can be achieved. During the first phase, the pH values in each batch reactors was upheld at about 8.0 by the addition of the 2M $NaHCO_3$ solution, while the less than 1 mm particle size of the rice straw was used. Based on the grams of VS present in the substrates, six dissimilar ratios of rice straw to buffalo dung were used in this study i.e. 1:9, 2:8, 3:7, 4:6, 5:5 and 6:4 and are labeled as ratios R1 to R6 correspondingly.

In succeeding phase, the ratio of rice straw to buffalo dung that yields maximum methane was further taken into consideration for the utmost appropriate quantity of the sodium bicarbonate $NaHCO_3$, which not only works as the pretreatment chemical to increase the efficiency of the anaerobic digestion process but also works as the buffer. On the basis of percentages of VS content in the substrate mixture, six dissimilar quantities of $NaHCO_3$ were employed i.e., 0.2, 0.3, 0.4, 0.5, 0.6 and 0.7 g $NaHCO_3$/gVS and were labeled as alkaline doze D1 to D6 correspondingly. The retaining time of the alkaline dozes was 24 hours. Moreover, less than 1 mm particle sized rice straw was used.

The third phase of the study was about the selection of utmost appropriate particle size of the rice straw. For this, ratio of the rice straw and buffalo dung and the most appropriate quantity of $NaHCO_3$ from the first and second phases respectively were used. The particle size is among the significant factors, which influences the competence of the anaerobic digestion process. In this study, six dissimilar particle sizes of the rice straw were used i.e. less than 1 mm, 2 mm, 4 mm, 6 mm, 8 mm and 10 mm and are labeled as sizes S1 to S6 correspondingly. Moreover, the rice straw size was reduced by employing hammer mill fitted with the appreciated shredding plate of hole size 2 mm, 4 mm, 6 mm, 8 mm and 10 mm size, while the less than 1 mm particle size was achieved by crushing 6 mm particle sized rice straw through coffee grinder.

Preparation of batch experiments: Each of the biochemical methane potential (BMP) tests was run as the duplicate and their average values were considered as the final result. The tests were conducted on automatic methane potential test system (AMPTS). The maximum sizes of the test reactors were 500 mL and were made of glass. Moreover, the operating temperature of the test was 37 °C, which is utmost promising temperature for the methane forming bacteria (Krishania *et al.*, 2013). Each batch reactor was filled with a blend of rice straw and buffalo dung comprising of 5 g of VS of the substrates. In addition an amount of 20 mL of inoculum was also added in each reactor, which was taken from the lab scale anaerobic reactor. Subsequently, by addition of distilled water, each reactor was top upped to 400 mL and was sealed with rubber stopper. The oxygen present in the reactors was expelled out by passing nitrogen gas.

Standard Error: In present study the batch assays were run as duplicate and for statistical significance the average values were taken as the final results. The standard error of the methane production was estimated by using Eq. 1, where SE is the standard error, SD is the standard deviation and n is the number of observations.

$$SE = \frac{SD}{\sqrt{n}} \quad (1)$$

The standard deviation was calculated by Eq. 2, where x represents the methane production and n is the number of observations.

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x}_i)}{(n-1)}} \quad (2)$$

Anaerobic Biodegradability: The estimation of substrates' anaerobic biodegradability (AB) in terms of percentage was made by employing Eq. 3 (Heo *et al.,* 2004). The BMP_E is the experimental biochemical methane potential that was achieved throughout the incubation time of substrates, while the BMP_T is the theoretical biochemical methane potential that theoretically estimated.

$$AB = \frac{BMP_E}{BMP_T} \times 100 \quad (3)$$

The BMP_T was calculated by using Eq. 4 given by Bushwell and Mueller (1952), where the inferiors *v, w, x, y* and *z* are the moles of the *C, H, O, N* and *S* correspondingly.

$$C_vH_wO_xN_yS_z + \frac{(4v - w - 2x + 3y - 2z)}{4}H_2O \longrightarrow \frac{(4v + w - 2x - 3y - 2z)}{8}CH_4 + \frac{(4v - w + 2x + 3y + 2z)}{8}CO_2 + yNH_3 + zH_2S \quad (4)$$

Substituting the atomic masses of *C, H, O, N* and *S* in Eq. 4 results Eq. 5, where BMP_T is in NmL and *C, H, O, N* and *S* are the dry weight percentages of respected element present in the substrate.

$$BMP_T = \frac{930 \times C + 2790 \times H - 350 \times O - 600 \times N - 175 \times S}{C + H + O + N + S} \quad (5)$$

Furthermore, BMP_T for the dissimilar ratios of the rice straw and buffalo dung utilized in this research work were estimated on the basis of their quantities set in each ratio.

Coefficient of multiple determination: In the present study the coefficient of multiple determination (R^2) was used to determine the relation between the rice straw particle size and its bulk density and was calculated by using Eq. 6. The R^2 is a comprehensive factor to reckon the correctness of the relation and represents the strength of the linear or non-linear association between the two variables. The R^2 was also used to determine the relation between the rice straw particle size and the electrical energy consumption used for its shredding.

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{n}(y_i - \bar{y}_i)^2} \quad (6)$$

RESULTS AND DISCUSSION

Features of the substrates: The features of the substrates including the elemental composition, MC, TS, VS, pH values, and the BMP_T are presented in Table 1. The BMP_T for buffalo dung is only 391 NmL/gVS whereas for rice straw it is 430 NmL/gVS. The BMP_T for different rice straw to buffalo dung ratios were estimated on the basis of their quantities and are presented in Table 2. It was perceived that increase in the percentage of rice straw is increasing the BMP_T.

The dissimilar particle sizes along with their bulk densities for the rice straw are presented in Table 3. Bulk density was determined within the range of 45 to 246 kg/m^3 for particle sizes 10 mm to less than 1 mm correspondingly, which are comparable to the values given by the Mani *et al.* (2003). Additionally, the coefficient of determination (R^2) between the rice straw particle sizes and their bulk densities was also calculated as 0.85, which depicts that the there is an inverse linear relationship between them.

Effect of rice straw to buffalo dung ratios on methane production (Outcome of phase one): The cumulative methane generation and its flow rate at different rice straw to buffalo dung ratios is shown in Fig. 2. Methane generation was started from day one and was increasing till it reaches to maximum value. The maximum methane generation was observed as 722.8 NmL for ratio R3 (30% rice straw and 70% buffalo dung) followed by 706.2, 689.1, 651.3, 586.4 and 461.9 NmL for ratios R4, R5, R6, R2 and R1 respectively. Before the methane generation terminated, it exhibits quite a few small peaks. The maximum flow rate was detected as 65.9 NmL/day for ratio R5 followed by 65.7, 60.3, 58.2, 43.9 and 33.1 NmL/day for ratios R4, R6, R3, R2 and R1 correspondingly. The results showed that, increasing the rice straw to buffalo dung ratio up to 3:7 increases the methane generation and decrease the time to achieve the peak of the cumulative methane. Besides, further increase in the rice straw to buffalo dung ratio, decreases the methane generation. This decrease is might be due to the acidic nature of the rice straw (Sahito *et al.,* 2013).

The efficiency of the anaerobic digestion process is also influenced by parameters, like temperature, pH, volatile fatty acids (VFA) and alkalinity. Sufficient alkalinity is essential in order to avoid the pH decrease, which decreases due to accumulation of VFA (Lesteur *et al.,* 2010; Chen *et al.,* 2008). Depending on the substrate to decompose, anaerobic digestion process is stable within the range of 2000 to 18000 mg $CaCO_3$/L of alkalinity (Cuetos *et al.,* 2008; Gelegenis *et al.,* 2007) and generally stable in the pH range 6.5–7.5 (Stronach *et al.,* 1986). The anaerobic digestion process is stable up to the ratio of VFA to alkalinity of 0.5, while a ratio that exceeds beyond 0.6 is considered as the indication of overloading (Lin *et al.,* 2009). At the end of phase one, the effluent of batch reactor was analyzed for pH, alkalinity and VFA. The result of pH, alkalinity and VFA at the end of phase one is given in Table 4. The results shows that the batch reactors were within the stable range of pH ranging from 6.8 to 7.4, alkalinity was observed in the range of 625 to 2625 mg $CaCO_3$/L, whereas the values of VFA were low and ranging from 240 to 360 mg CH_3COOH/L. The ratio of VFA to alkalinity was observed as less than 0.5, thus no inhibition of the anaerobic digestion process was observed.

The graph between the dissimilar ratios of rice straw and buffalo dung, BMP_E along with the standard error and AB is presented in Fig. 3. It reveals that as the ratio of the rice straw was increased from R1 to R3, the BMP_E was increasing but further increase causes the decrease BMP_E. Similarly, AB was increasing with the increase of the rice straw ratio from R1 to R3 but then it declines. Furthermore, the maximum AB was observed as 37.7% for ratio R3 followed by 36.1, 34.5, 32.0, 31.2 and 25.1 % for ratio R4, R5, R6, R2 and R1 correspondingly. As the maximum BMP_E and AB were discovered for ratio R3, thus ratio R3 was designated as the utmost appropriate ratio of rice straw and buffalo dung that enhances the methane generation.

Effect of different alkaline dozes on methane production (Outcome of phase two): The cumulative methane generation and its flow rate at optimized rice straw to buffalo dung ratio R3 with different alkaline dozes of $NaHCO_3$ is illustrated in Fig. 4. Generation of methane from reactor bottles containing alkaline dozes D1 to D4 was started from day one, while from dozes D5 and D6 it was started from day five because of the higher alkalinity present in the reactor bottles (Sahito *et al.*, 2013). The highest methane generation was achieved as 889.1 N mL by using 0.5 g $NaHCO_3$/gVS i.e. doze D4 followed by 846.8, 844.2, 837.2, 819.1 and 768.3 N mL for dozes D3, D5, D6, D2 and D1 correspondingly. Before the methane generation terminated, it exhibits quite a few peaks. This fluctuation of the methane flow rate is due to the dynamic balance between the acidogenic and methanogenic phase of anaerobic digestion process (Song *et al.*, 2013).The maximum flow rate was detected as 59.2 NmL/day in the batch reactor containing $NaHCO_3$ of 0.6 g $NaHCO_3$/gVS i.e. doze D5 followed by 59.0, 56.7, 54.2, 53.1 and 47.7 NmL/day for dozes D4, D2, D3, D6 and D1 correspondingly. The results showed that, increasing the quantity of $NaHCO_3$ up to 0.5 g/gVS increases the methane generation and decrease the time to achieve the peak of the accumulative methane. Besides, further increase in the quantity of $NaHCO_3$, decreases the methane generation because of the higher alkalinity (Sahito *et al.*, 2013). Song *et al.* (2013) also reported that excessive alkalinity is toxic for anaerobic digestion.

At the end of phase two, the effluent of batch reactor was analyzed for pH, alkalinity and VFA. The result of pH, alkalinity and VFA at the end of phase two is given in Table 5. The result shows that the pH of batch reactors was within the range of 7.2 to 8.1, which is slightly higher than to the optimum value especially for doze 0.7 g $NaHCO_3$/gVS. The alkalinity was observed in the range of 750 to 2425 mg $CaCO_3$/L, whereas the values of VFA were low and ranging from 120 to 360 mg CH_3COOH/L. The ratio of VFA to alkalinity was observed as less than 0.5, thus no inhibition of the anaerobic digestion process was observed.

The chart between the dissimilar quantities of $NaHCO_3$, BMP_E along with the standard error and its AB is presented in Fig. 5. It reveals that as the quantity of the $NaHCO_3$ was increased from D1 to D4, the BMP_E was increasing but further increase causes the decrease BMP_E. Similarly, AB was increasing with the increase of the rice straw ratio from D1 to D4 but then it declines. Furthermore, the maximum AB was observed as 46.4% for doze D4 followed by 44.2, 44.0, 43.7, 42.7 and 40.1% for dozes D3, D5, D6, D2 and D1 correspondingly. As the maximum BMP_E and AB were observed for doze D4, thus doze D4 was selected as the utmost appropriate alkaline doze that enhances the methane generation of the rice straw and buffalo dung.

Effect of particle size of rice straw on methane production (Outcome of phase three): The cumulative generation of methane and its flow rate employing optimized rice straw to buffalo dung ratio R4 and alkaline doze D5 with different particle sizes of the rice straw are presented in Fig. 6. Generation of methane started from day one and was increasing till it reaches to maximum value. The highest methane generation was detected as 918.7 NmL for particle size 2 mm i.e. size S2 followed by 886.5, 860.0, 837.7, 805.2 and 796.6 NmL for particle sizes S1, S3, S4, S5 and S6 correspondingly. Before the methane generation terminated, it exhibits quite a few small peaks. The peak flow rate was observed as 83.8 N mL/day by batch reactor containing particle size S1 followed by 82.3, 74.9, 60.1, 66.1 and 48.5 N mL/day for particle sizes S2, S3, S5, S4 and S6 correspondingly. The results showed that, decreasing the rice straw particle size increases the methane generation and decrease the time to achieve the peak of the accumulative methane, except for particle size less than 1 mm, where methane generation decreases.

At the end of phase three, the effluent of batch reactor was analyzed for pH, alkalinity and VFA. The result of pH, alkalinity and VFA at the end of phase three is given in Table 6. The result shows that the pH of batch reactors was within the range of 7.8 to 8.0, which is slightly higher than to the optimum value. The alkalinity was observed within the stable range of 1920 to 2130 mg $CaCO_3$/L, whereas the values of VFA were low and ranging from 420 to 900 mg CH_3COOH/L. The ratio of VFA to alkalinity was observed as less than 0.5, thus no inhibition of the anaerobic digestion process was observed.

The chart between the dissimilar particle sizes of the rice straw, BMP_E along with the standard error and its AB is presented in Fig. 7. Except for rice straw particle size less than 1 mm, the present study reveals that as the particle was decreases, the BMP_E was increasing. Similarly, AB was increasing with the decrease of the rice straw particle size from S2 to S6. Furthermore, the maximum AB was observed as 47.9% for particle size S2 followed by 46.2, 44.8, 43.7, 42.0 and 41.5% for size S1, S3, S4, S5 and S6 correspondingly. The maximum BMP_E and percentage AB were observed for particle size S2, thus particle size S2 could be the best rice straw particle size that enhances the methane generation from rice straw and buffalo dung.

In contrast, as rice straw particle size is decreased, the energy consumption increases. In the present study, the rice straw was shredded by using hammer mill (Condux, D6451; Wolfang Bei Hanau; LHM 20/16; 1976). The result of the electrical energy consumption utilized for shredding of the two different particle sizes of the rice straw i.e. 4 mm and 2 mm are given in Table 7. The results show that the specific energy consumption by the above said model of the hammer mill for shredding the rice straw particle sizes 4 mm and 2 mm was 3.3 and 6.5 kWh/ kg respectively. It reveals that to produce particle size 2 mm, about double energy is required in comparison to the 4 mm particle size, thus rice straw particle size of 4 mm is recommended.

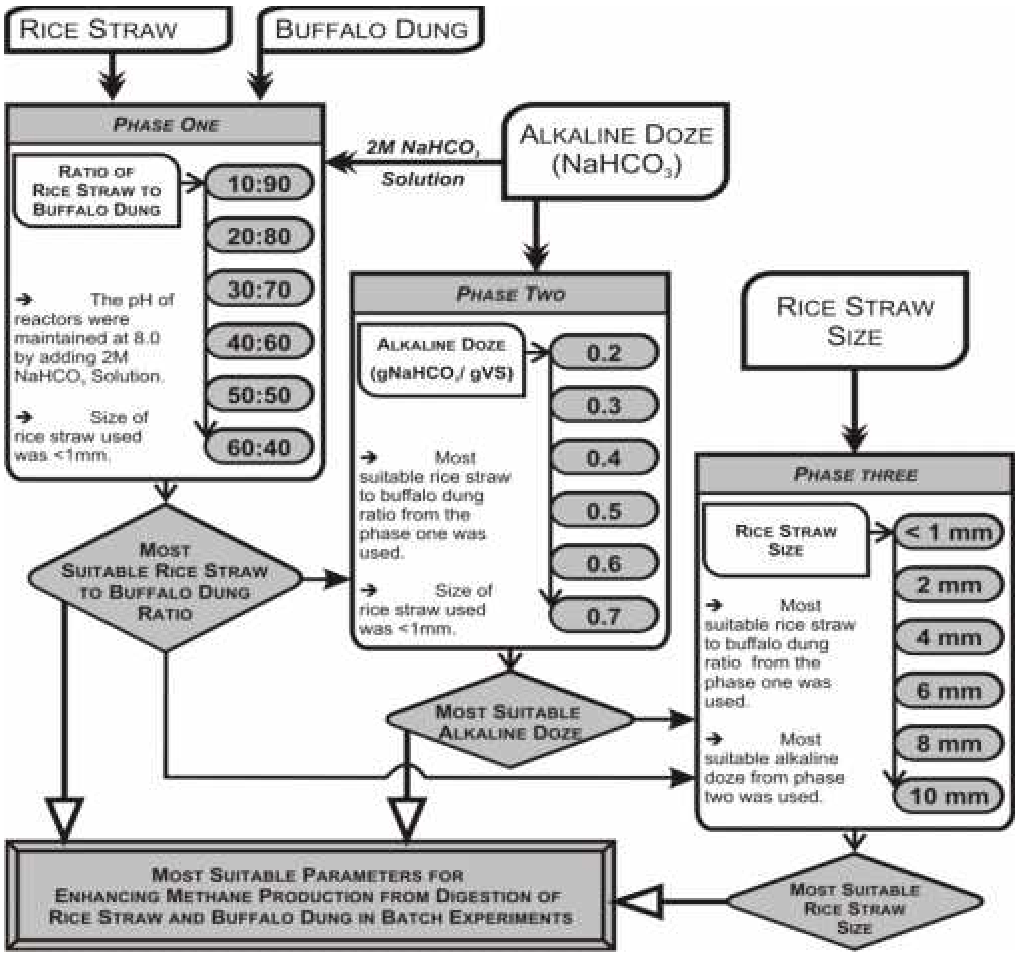

Fig. 1 The three phases of study

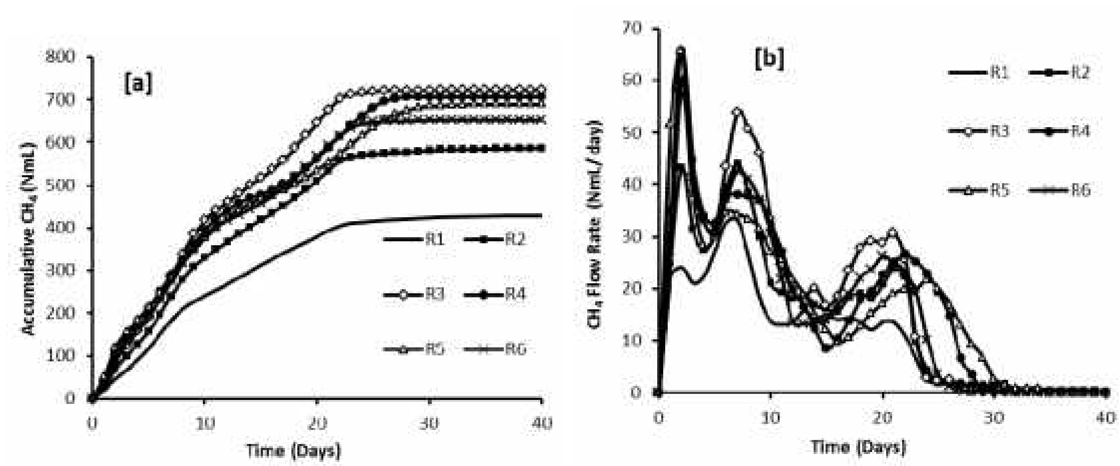

Fig. 2 Generation of methane at dissimilar ratios of rice straw and buffalo dung [a] accumulative methane [b] methane flow rate

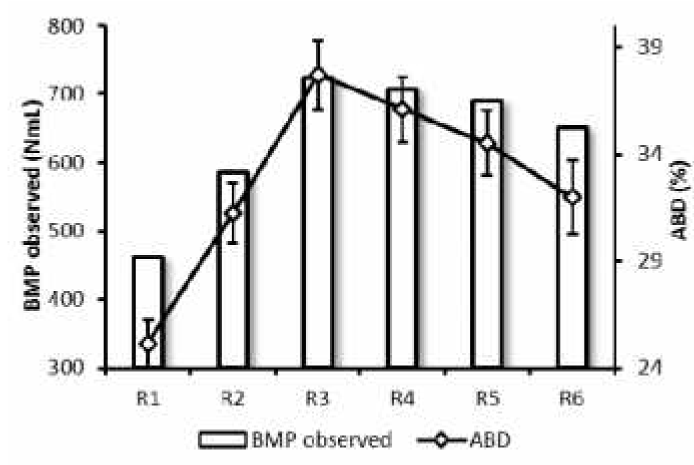

Fig. 3 Association of BMP_E and AB at dissimilar ratios of rice straw and buffalo dung

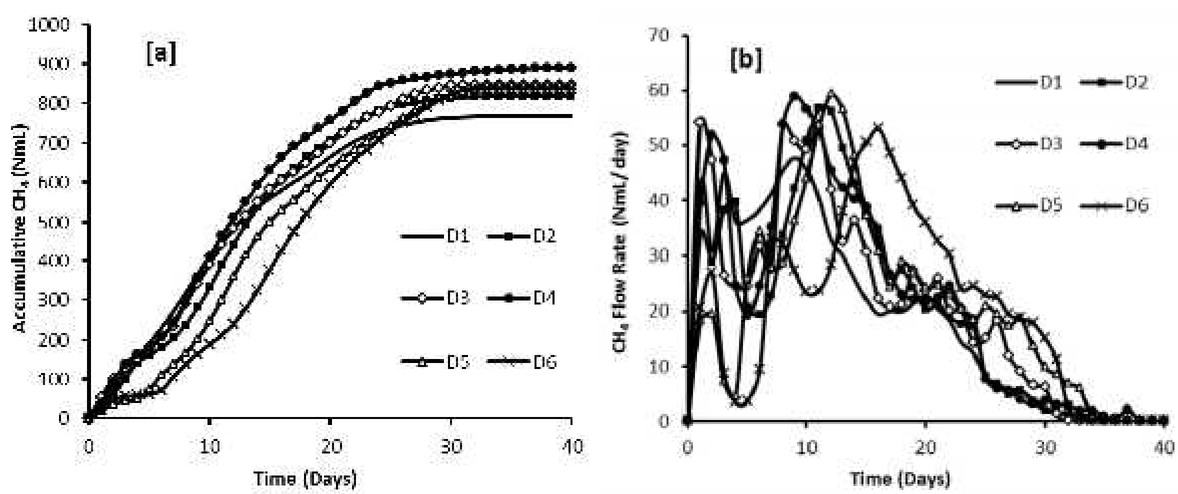

Fig. 4 Generation of methane at dissimilar alkaline dozes [a] accumulative methane [b] methane flow rate

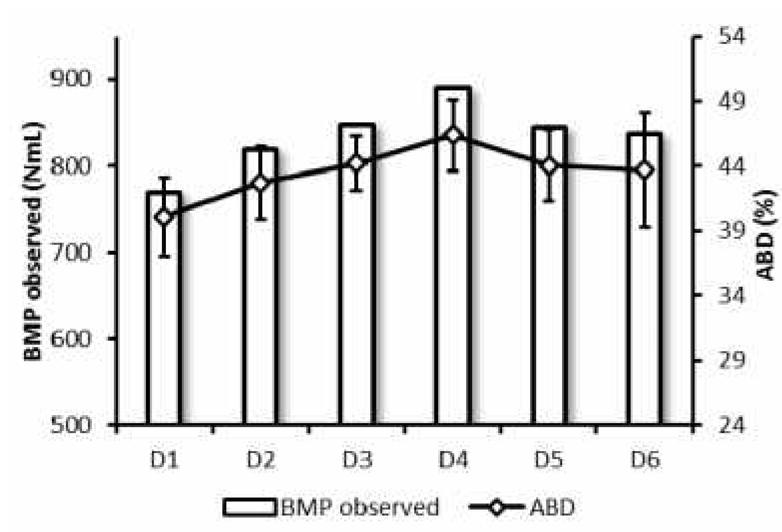

Fig. 5 Association of BMP_E and AB at dissimilar alkaline dozes

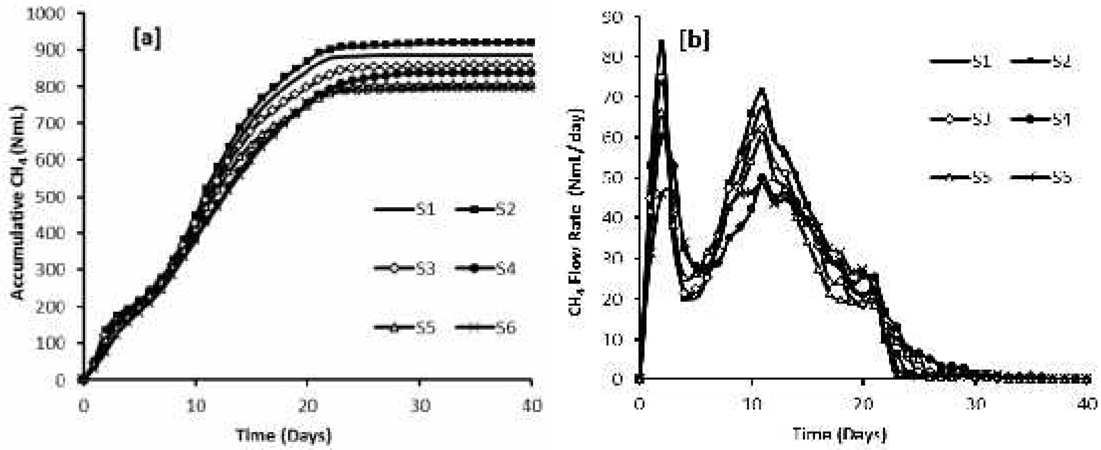

Fig. 6 Generation of methane at dissimilar rice straw particle sizes [a] accumulative methane [b] flow rate of methane

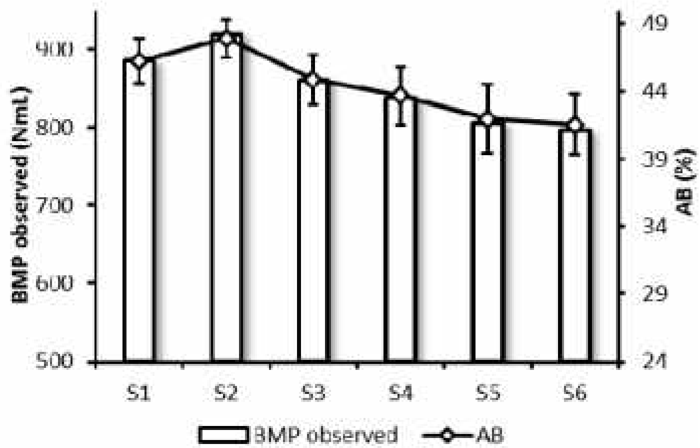

Fig. 7 Association of BMP_E and AB at dissimilar particle sizes of rice straw

Table 1. Features of the substrates.

Substrate	% weight (dry basis)					pH	MC (%)	TS (%)	VS (% TS)	BMP_T (NmL/gVS)
	C	H	N	O*	S					
Rice straw	37.6	4.8	1.0	37.0	0.2	6.0	2.1	97.9	83.4	430
Buffalo dung	38.6	4.3	1.3	40.1	0.2	7.5	80.5	19.5	71.8	391

* oxygen was calculated by difference

Table 2. BMP_T for different rice straw to buffalo dung ratios.

Rice straw to buffalo dung ratio (gVS/gVS)	R1	R2	R3	R4	R5	R6
	1:9	2:8	3:7	4:6	5:5	6:4
BMP_T (NmL)	395	399	403	407	411	414

Table 3. Bulk density of rice straw at dissimilar particle sizes

Rice straw particle sizes	S1	S2	S3	S4	S5	S6
	< 1 mm	2 mm	4 mm	6 mm	8 mm	10 mm
Bulk density (kg/m^3)	246	177	109	66	63	45

Table 4. Result of pH, alkalinity and VFA at the end of phase one

Ratios (gVS/gVS)	pH	Alkalinity (mg $CaCO_3$/L)	VFA (g CH_3COOH/L)	VFA/ Alkalinity
1:9	6.8	625	360	0.58
2:8	7.0	1000	360	0.36
3:7	7.2	1500	240	0.16
4:6	7.2	1875	240	0.13
5:5	7.3	2375	240	0.10
6:4	7.4	2625	360	0.14

Table 5. Result of pH, alkalinity and VFA at the end of phase two

Dozes (g $NaHCO_3$/ gVS)	pH	Alkalinity (mg $CaCO_3$/L)	VFA (g CH_3COOH/L)	VFA/ Alkalinity
0.2	7.2	750	120	0.16
0.3	7.3	1075	120	0.11
0.4	7.4	1425	360	0.25
0.5	7.4	1775	240	0.14
0.6	7.7	2050	240	0.12
0.7	8.1	2425	240	0.10

Table 6. Result of pH, alkalinity and VFA at the end of phase three

Particle Sizes (mm)	pH	Alkalinity (mg $CaCO_3$/L)	VFA (g CH_3COOH/L)	VFA/ Alkalinity
< 1 mm	7.8	2130	900	0.42
2 mm	7.8	2090	600	0.29
4 mm	7.9	2030	420	0.21
6 mm	8.0	2100	480	0.23
8 mm	8.0	1920	360	0.19
10 mm	8.0	1970	480	0.24

Table 7. Result of energy consumption used for shredding of rice straw

Rice Straw Size	Specific Energy (kWh/kg)
4 mm	3.3
2 mm	6.5

Conclusions: This study was carried out to enhance the methane generation from the rice straw by anaerobically co-digesting it with the buffalo dung. The study was carried out into three phases and it includes determination of ratio of rice straw to buffalo dung, specific mass of $NaHCO_3$ as buffer and alkaline pretreatment chemical and appropriate size of the rice straw particle size that yields maximum methane. The outcome of study reveals that the efficiency of the co-digestion of the substrates at issue is apparently acted upon by the three parameters under discussion. Among the three parameters, rice straw to buffalo dung ratio was more significant factor followed by addition of sodium bicarbonate and rice straw particle size respectively, that enhances the methane generation. Besides, that the maximum methane generation from the co-digestion of the rice straw and buffalo dung could be achieved by taking rice straw to buffalo dung ratio of 3:7 on the basis of volatile solids, the specific mass of $NaHCO_3$ of 0.5 g/gVS and the rice straw particle size of 2 mm. Considering the above parameters the methane generation was obtained about 184 NmL/gVS at anaerobic biodegradability of about 48%. By using particle size of 4mm, methane generation was obtained about 172 NmL/g of VS. However, because of about double energy consumption to produce rice straw of particle size of 2 mm than to the particle size of 4 mm, thus on the basis of energy consumption later one is suggested as appropriate particle size.

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