International Journal of Sciences: Basic and Applied Research (IJSBAR)

ISSN 2307-4531
(Print & Online)

http://gssrr.org/index.php?journal=JournalOfBasicAndApplied

Alkaline Pretreatment of Sorghum Stalk and Co-Digestion with Sludge for Biogas Production

Purwoko[a]*, Muhammad Romli[b], Suprihatin[c], Liesbetini Haditjaroko[d]

[a,b,c,d]*Department of Agroindustrial Technology, Faculty of Agricultural Engineering and Technology, Bogor Agricultural University, IPB Darmaga Campus, West Java Indonesia*

**Email: p_purwoko @yahoo.com*

Abstract

Recycling of residual agriculture biomass using anaerobic digestion enables recovery of biomass carbon and nutrients as source of energy and fertilizer. The obstacles met include lignocellulosic structure of biomass tissue and its high carbon to nitrogen ratio. This research work evaluates alkaline pretreatment of sorghum stalk and co-digestion system of pretreated sorghum stalk with wastewater sludge. The sorghum stalk was pretreated by dilute sodium hydroxide solution of 0.5, 1.0, and 1.5% to improve bacterial accessibility. Digesters were fed on a mixture of sorghum stalk and sludge at ratios 90:10, 80:20, 70:30, and 60:40 (TS basis). Digesters were run in batch at 35^{o}C. Digesters performance was evaluated in terms of biogas production rate and yield. Digesters run with feed ratios of 70:30 and 60:40 showed shorter lag phase, higher biogas generation rate and higher biogas yield compared to those run with feed ratios 80:20 and 90:10. The highest specific biogas production of approx. 350 L/kg VS was achieved by digesters run at the ratios of 70:30 and 60:40. Digester run with ratio 90:10 resulted in the specific gas production of 150 L/kg VS, whereas that fed on feed ratio of 40:60 generated only 300 L/kg VS. Co-digestion of sorghum stalk and wastewater sludge at a proper ratio improved biogas production.

Keywords: alkaline pretreatment; sludge co-digestion; sorghum stalk; biogas production.

----

* Corresponding author.

E-mail address: p_purwoko @yahoo.com.

1. Introduction

Anaerobic degradation is the decomposition process of organic material in the absence of oxygen involving a consortium of anaerobic microorganisms that will degrade organic material into biogas [1]. The formed biogas consists of methane (CH_4), carbon dioxide (CO_2) and other gases in small quantities (H_2, H_2S, NH_3). Biogas can be used as biofuel. In addition to biogas, the anaerobic decomposition produces digestate and leachate. The digestate can be used as anaerobic compost to improve soil structure, while the leachate can be used as liquid fertilizer after appropriate pretreatment.

The transformation of organic matter into biogas undergoes four stages, namely hydrolysis, acidogenesis, acetogenesis, and methanogenesis involving millions of microorganisms [2] of the four stages, the hydrolysis stage is a critical stage in the anaerobic degradation of agricultural wastes such as rice straw, corn stalks, and sorghum stalks. The lignocellulosic waste contains cellulose, hemicellulose and lignin components that are relatively difficult to access by hydrolytic (enzymes) microorganisms leading to relatively low degradation and biogas production rates.

Anaerobic degradation technology so far has been successfully applied for the management of domestic sludge waste, livestock manure and sludge of domestic and industrial liquid waste, particularly liquid waste containing high organic material [2]. Organic solid wastes such as agricultural and agroindustrial biomass contains very high organic material that can be converted anaerobically into valuable products, such biogas, digestate and leachate.

Sorghum stalk is one of the potential stocks of agriculture biomass. Sorghum (*Sorghum bicolor* L.Moench) is an herbaceous plant which produces sorghum grain and residual biomass in the form of stalks and leaves. Sorghum stalk component is quite high compared to the main product of sorghum grain. Sorghum stalk yield is as high as 1.3 times of sorghum grain [3]. Assuming an average grain sorghum production of 5 tons/ha, it will generate sorghum stalk of 6.5 tons/ha. Sorghum stalk contains 88 percent of total solids consisting of 15 percent of lignin, and 61 percent of carbohydrates (cellulose and hemicellulose). The high cellulose and hemicellulose components can be converted to biogas.

Sorghum stalk degradation is constrained by the lignocellulose structure that is difficult to degrade due to cross bonds between polysaccharides (cellulose and hemicellulose] and lignin through ester and ether bonds [4]. Cellulose, hemicellulose, and lignin form microfibrils cellulose structure that serves to strengthen the crops [5]. Microfibril cellulose has two-thirds of crystalline regions difficult to access by hydrolytic enzymes and one third of amorphous region accessible by hydrolytic enzyme [6]. Another constraint in the degradation of sorghum stalk is the relatively low content of nitrogen in sorghum stalk. According to [7], the nitrogen content in dry sorghum stalk is only 0.74 percent.

Anaerobic digestion of sorghum stalk can be conducted in dry, semi-dry or wet fermentation process. A solid waste anaerobic degradation process with the total solid of more than 20 percent is known as dry process, while that with the total solid of less than 10 percent is known as wet process [8]. Dry process has the advantage of relatively small size of digester, but it requires a process of material stirring in the reactor that is quite difficult and expensive. Wet process with more amount of water requires large reactor volume. The advantage of wet process is that the digester can be operated without stirring; and if the raw material contains toxic on anaerobic microorganisms it can be diluted by water added.

This research work evaluates the potential of sorghum stalk to generate biogas and other anaerobic by-products, using semi-dry fermentation system of around 10% TS. In particular, the effects of alkaline pretreatment of the raw material and co-digestion with sludge were investigated. The biogas formation at various conditions was characterized using parameters of lag phase period, maximum biogas production rate and specific gas production.

2. Materials and Methods

2.1 Materials

The stalk of sweet sorghum was obtained from BIOTROP-IPB experimental field, Bogor. Cattle manure used as microbial seed was obtained from experimental farm in Darmaga Campus. Sludge was obtained from activated sludge wastewater treatment plant of PT, Van Melle Indonesia, Bogor.

2.2 Bioreactor

A set of 500 mL Erlenmeyer flasks was used as bioreactors or digesters. These flasks were put in a thermostat shaker water bath maintained at 35^{o}C; each flask was connected with tubing to a measuring cylinder used for measuring gas production by liquid displacement method.

2.3 Method

2.3.1 Preparation of raw materials

Sorghum stalk was sun dried. The dried sorghum stalk was cut into the size of 0.5-1.0 cm. The raw materials were analyzed to determine its chemical composition: water, ash, fat, protein, crude fiber and carbohydrate, carbon, nitrogen, and phosphate contents. The analysis was conducted according to the AOAC methods [9].

2.3.2 Sorghum stalk pretreatment using NaOH

The purpose of pretreatment is to improve the accessibility of hydrolytic anaerobic microorganisms (enzyme) on cellulose and hemicellulose. Dry sorghum stalk with the size of 0.5-1.0 cm was soaked in distilled water for 60 minutes until all parts of stalk got wet and then drained. The wet stalk was then soaked in different NaOH solutions of 0.5%, 1%, and 1.5% for 48 hours. The pretreated sorghum stalk was drained and washed with distilled water to remove the residual alkali from sorghum stalk. The pretreated sorghum stalk was analyzed for its TS, VS, lignin, cellulose, hemicellulose, ash, and nitrogen content.

2.3.3 Biogas Production

Biogas production was conducted in 500 mL Erlenmeyer as bioreactor with a total solid of 8-10 percent. Bacterial inoculum (cattle manure) used was 10 percent. Biogas production was observed daily for 60 days and substrate as well as digestate were analyzed for its TS (total solids), VS (volatile solids), ash, TKN (total kjeldahl nitrogen), and VFA (volatile fatty acids). The analysis was conducted according to the AOAC methods [9].

Co-digestion of sorghum stalk and sludge was conducted by mixing both materials at various compositions. Ratio of the pretreated sorghum stalk to sludge were 90:10, 80:20, 70:30, and 60:40. The same amount of inoculum (10%) was added to the mixture. The digester was set at pH 7 with the addition of dilute HCl or NaOH. Before digestion, the oxygen in the Erlenmeyer flasks was removed by sparging with nitrogen gas (N_2) to ensure the anaerobic condition throughout the fermentation process. Each erlenmeyer flask was covered with a rubber cap equipped with a swan neck glass pipe, the glass pipe was connected with plastic hose and connected to the inverted measuring cup filled with water to measure the gas formed. During the fermentation process, biogas production and composition were monitored by gas sampling.

3. Results and Discussion

3.1 Characteristics of sorghum stalk, cattle manure and sludge

Characterization of material is needed to enable the substrate formulation in the process of anaerobic digestion. The chemical characteristics of each material used are shown in Table 1. As shown in the table, sorghum stalk contains high volatile solids (86.64%). As agricultural biomass in general, the organic material is composed mainly from cellulose, hemicellulose and lignin. The high proportion of organics indicates that sorghum stalk has high potential as a raw material for biogas production. However, the ratio of C/N in sorghum stalk is very high, i.e.123. To be used as the substrate in the anaerobic degradation for biogas production, it will be lack of nitrogen content. According to [11, 12], the optimum C/N ratio for the biological anaerobic process should be in the range of 25-35. In comparison to other sources of inoculum, cattle manure contains millions of different anaerobic bacteria. Therefore, cattle manure was used as seed of anaerobic microorganisms. In contrast with sorghum stalk, the sludge show high nitrogen content. The low C/N ratio of sludge, i.e.8, indicates the suitability to be used as nitrogen source in a system of co-digestion with the sorghum stalk.

3.2 Characteristics of alkaline pretreated sorghum stalk

Table 2 shows the characteristics of sorghum after pretreatment using dilute NaOH. The alkaline pretreatment resulted in a slight decrease in cellulose content. Similarly, the hemicellulose content decreased slightly. Pretreatment with a NaOH concentration of 1.5% lead to a relatively higher reduction of cellulose and hemicellulose. In general, the lignin decreases with higher NaOH concentration. Soaking sorghum stalk in NaOH solution causes the physical structure of materials to become more loosely. According to [10], pretreatment using NaOH on rice straw affects the physicochemical structure of lignin-carbohydrate complex ester bond through hydrolysis reaction which releases the cellulose.

Table 1: Characteristic of sorghum stalk, cattle manure and sludge

No.	Parameter	Unit	Sorghum stalk	Cattle manure	Sludge
1.	Water	% wb	7.64	83.07	94.64
2.	Total solids (TS)	% wb	92.36	16.93	5.36
3.	Ash	% wb	5.72	4.60	1.94
4.	Volatile solids (VS)	% wb	86.64	12.33	3.42
5.	Carbon (C)	% wb	49.38	11.95	1.78
6.	Nitrogen (N)	% wb	0.40	0.38	0.22
7.	C/N Ratio	-	123	31	8
8.	Phosphate (as P)	% wb	1.33	-	-
9.	Cellulose	% wb	40.56	-	-
10.	Hemicellulose	% wb	10.18	-	-
11.	Lignin	% wb	15.07	-	-

Table 2: Characteristics of sorghum after pretreatment using NaOH

Pretreatment	Unit	Cellulose	Hemicellulose	Lignin	VS	Ash
Without NaOH	%	42.99	11.27	26.40	88.27	2.81
NaOH 0.5 %	%	41.05	10.18	24.71	89.02	3.04
NaOH 1.0 %	%	40.90	9.50	21.66	89.60	2.66
NaOH 1.5 %	%	40.56	6.87	15.07	89.67	1.95

3.3 Effect of alkaline pretreatment on biogas production

Biogas production was studied using pretreated sorghum stalk as substrate. The characteristics of the fermentation substrates for biogas production are presented in Table 3. Figure 1 shows the effect of alkaline pretreated sorghum stalk on biogas production. A higher biogas production can be observed at the end of fermentation, even though the difference in the gas generation rate is hardly noted. The alkaline pretreated digesters resulted in overall 14-16 higher of biogas production after 45-65 days of digestion. Pretreatment of sorghum stalk using an adequate NaOH concentration loosened the structure of lignin surrounding the cellulose and hemicellulose, thus degrading bacteria could access the cellulose and hemicellulose easier. In addition, the soaking in NaOH will dissolve lignin from sorghum stalk which is then removed during the washing process. The figure also shows that biogas production is not significantly different among the tested NaOH concentrations of 0.5 to 1.5 percent. Previous work by [2] on pretreatment of sorghum stalk using 0.5% NaOH concluded that the treatment reduced the dissolved carbohydrate content. Also, [2] reported that pretreatment of wheat stalk using 1% NaOH for 7 days increased the performance of anaerobic degradation process by 38-119%.

Table 3: Characteristics of the fermentation substrates for biogas production

Treatment	TS (%wb)	Ash (%wb)	VS (%wb)
NaOH 0 %	7.98	1.66	6.72
NaOH 0.5 %	8.52	1.38	6.72
NaOH 1.0 %	8.84	1.57	7.27
NaOH 1.5 %	8.75	1.54	7.20

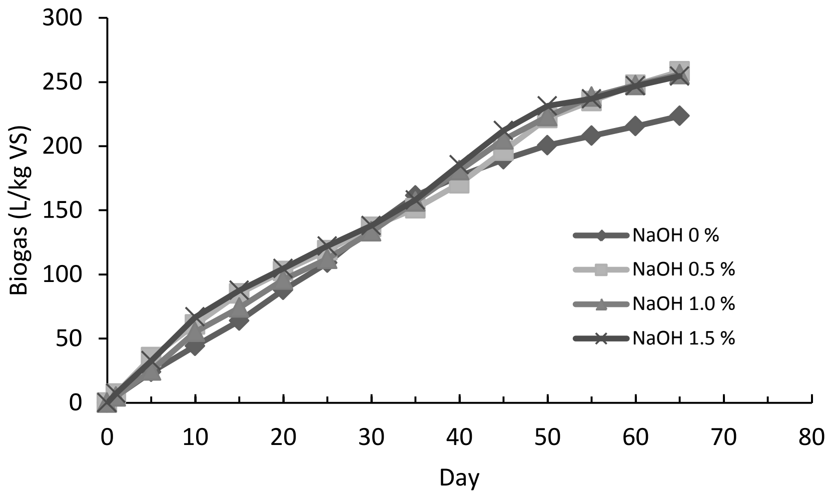

Figure 1: Effect of alkaline pretreatment of sorghum stalk on biogas production

3.4 Effect of sludge co-digestion on biogas production

The pretreated sorghum stalk with NaOH still results in high C/N ratios of 86-105. As stated by [11, 12], the anaerobic degradation process will be optimum in the substrate with C/N ratio of 25-35. Therefore, the addition of nitrogen is needed to improve the anaerobic process performance. Sludge addition to sorghum stalk can be seen as an alternative to increase the nitrogen content of sorghum stalk. Table 4 shows characteristics of substrate as a result of various levels of sludge addition. It is seen that the addition of sludge from 10 to 40% resulted in the C/N ratio in the range of 20-35. As shown in Table 4, the more the sludge is added, the lower value of C/N ratio can be obtained.

Table 4: Characteristics of substrate of sorghum stalk co-digestion and sludge

Sorghum stalk : sludge	TS (%wb)	Water (%wb)	Ash (%wb)	VS (%wb)	C/N Ratio
90:10	9.04	90.96	1.68	7.36	35
80:20	10.00	90.00	1.79	8.21	28
70:30	9.25	90.48	1.36	8.25	25
60:40	10.31	89.69	1.97	8.34	20

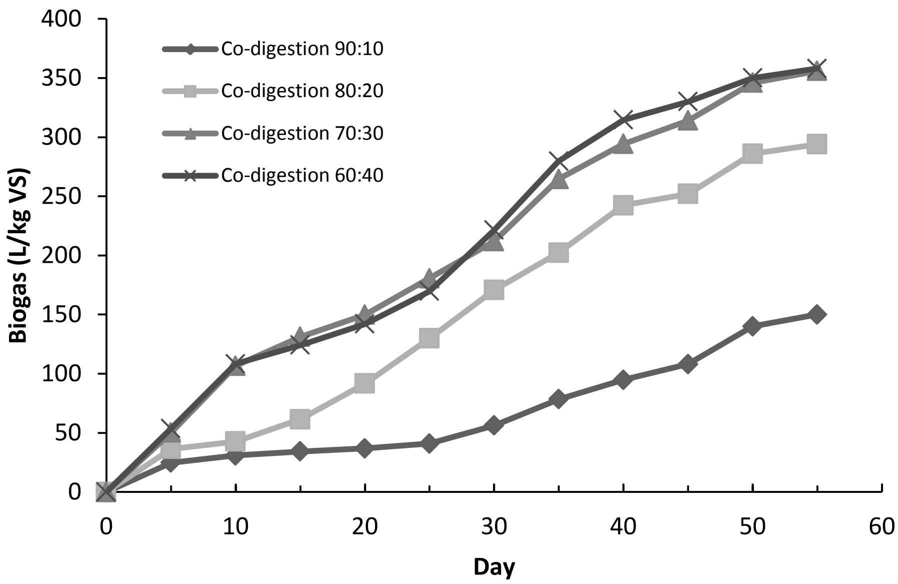

Figure 2: Effect of sludge co-digestion on biogas production

In this research work an experiment was also conducted in a larger digester, i.e. 25 liters. The experiment was carried out by co-digestion of sorghum and sludge with a ratio of 70:30 at room temperature and the result is presented in Figure 3. It shows that biogas production profiles directly on the exponential phase without lag phase. However, the rate of formation of biogas is slower than that in the 500 mL Erlenmeyer reactor. This is likely due to the lower operating temperature (26-30^{O}C) of the 25 L bioreactor. The 500 mL Erlenmeyer reactor were carried out at a higher temperature of 35^{O}C. The lower temperatures lead to less optimum condition for anaerobic microorganism's growth, and hence the biogas production.

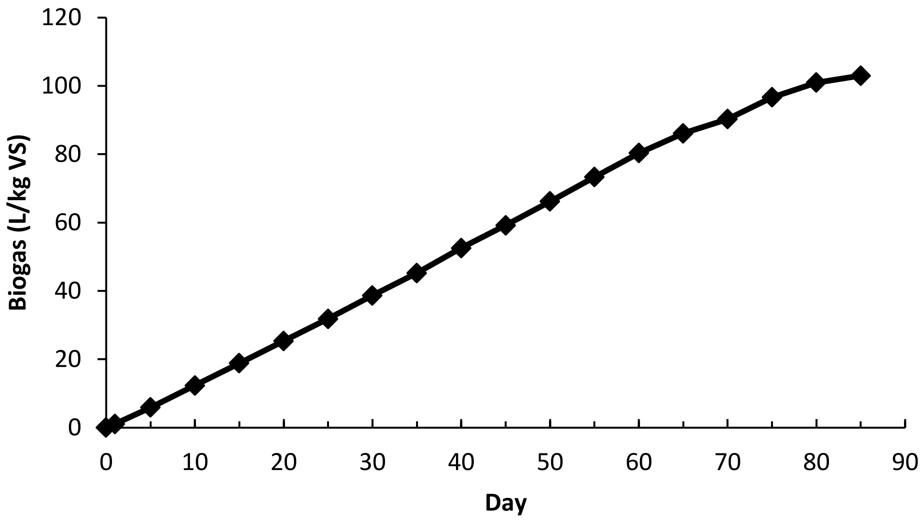

Figure 3: Profile of biogas production in 25 L bioreactor

3.5 Products of co-digestion of sorghum stalk and sludge

Abundant quantity of sorghum stalk is currently underexploited. In most cases, it even becomes burden to environment when treated improperly by burning or being left decomposed without control. The application of anaerobic digestion technology that simultaneously converts the organics of biomass into valuable products such as energy in the form of biogas and soil improver in the forms of liquid fertilizer and digestate is considered as a strategic approach. Recycling a proportion of nutrients and carbon back to land in the forms of organic fertilizer and soil improver can expectedly maintain high crop yields. Carbon recovery in the form of biogas will give another important benefit as an alternative energy to fossil fuel. The main content of biogas is methane and carbon dioxide. Table 5 shows the composition of methane and carbon dioxide in various proportions of sludge. The concentration of methane to carbon dioxide ranged from 68:32 to 79:21. In general, the portion of methane increases with increasing portion of sludge in the feed. This is consistent with the finding of [4] in their research on co-digestion of sludge and rice stalk that lower C/N ratio resulted in the increased portion of methane. The higher nitrogen content in the feed leads to the fulfillment of the needs of methanogenic bacteria to form CH_4 and CO_2. With such level of methane, the biogas produced is considered as inflammable gas. According [8], biogas is classified as flammable if the methane level is higher than 50 percent.

Table 5: Biogas composition in various proportions of sludge

	Ratio of sorghum stalk to sludge in the feed			
	90 : 10	80 : 20	70 : 30	60 : 40
CH_4 : CO_2	68 : 32	77 : 23	79 : 21	79 : 21

4. Conclusion

Alkaline pretreatment of sorghum stalk using 0.5 to 1.5 percent NaOH resulted in higher biogas production by 14-16 percent. Co-digestion system of sorghum stalk and sludge affected total production of biogas and its rate of generation. The ratios of stalk to sludge of 70:30 and 60:40 led to improved digester performance in terms of shorter lag phase, higher biogas generation rate, and biogas yield. This improvement was primarily due to better substrate composition, especially C/N ratio, but better supply of trace elements provided by sludge might also give important contribution. Biogas production of 350 L/kg VS could be achieved with fermentation time of approx. 55 days. This value of biogas production is still lower compared to the potential values. Other than feed composition, design and configuration of digester as well as its operating conditions affect digester performance. Co-digestion system with leachate recirculation is expected to improve the substrate removal efficiency and the biogas yield. The anaerobic digestion of the residual agriculture biomass enabled the recovery of organic carbon and nutrients of the biomass into biogas as source of energy and digestate and leachate as soil improver and fertilizer.

References

[1] Al Seadi, T., Rutz, D., Prassl, H., Kottner, M., Finsterwalder, T., Volk, S., & Janssen, R. (2008). Biogas handbook. University of Southern Denmark Esjberg, Denmark. http:www.sdu.dk

[2] Wu, X., Yao, W., Zhu, J., & Miller, C. (2010). Biogas and CH_4 productivity by co-digesting swine manure with three crop residues as an external carbon source. Bioresource Technology, Vol. 101, pp. 4042-4047.

[3] Kim, S., Dale, B.E. (2004). Global potential bioethanol production from waste crops and crop residues. J Biomass and Bioenergy. Vol. 26, pp. 361-375.

[4] Yang, Y., Zhang, Z., Zhao, R., & Sugiura, N. (2011). Development of a high-efficiency methane fermentation process for hardly degradable rice stalk. Symposium Paper JIFS, Vol.1, pp 8:61.

[5] Xiao, C., Bolton, R., & Pan, W.L. (2007). Lignin from rice stalk kraft pulping: Effects on soil annregation and chemical properties. Bioresour.Technol. Vol. 98(7), pp 1482-1488.doi: 10.1016/j.biortech.2005.11.014.

[6] Chandra, R., Takeuchi, H., & Hasegawa, N. (2012). Hydrothermal pretreatment of rice stalk biomass : A potential and promosing method for enhanced methane production. Applied Energy. Vol. 94, pp. 129-140.

[7] Chum, H.L., Douglas, L.J., Feinberg, D.A., & Schroeder, H.A., (1985). Evaluation of pretreatments of biomass for enzymatic hydrolysis of cellulose. Solar Energy Research Institute: Golden. Colorado, pp 1-64.

[8] Lissens, G., Vandevivere, P., De Barere, L., Biey, E.M., & Vestraete, W. (2001). Solid waste digestor: process performance and practice for municipal solid waste digestiland, on. Water Science and Technology. Vol.44 (8), pp. 91-102.

[9] AOAC, (1995). Official Methods of Analysis of The Association of Analytical Chemist. Washington DC:AOAC International.

[10] He Y, Pang Y, Liu Y, Li X, Wang K. 2008. Physcochemical characteristic of straw pretreated with Sodium Hydroxide in the solid state for enhancing biogas production. Energy and Fuel, Vol.xxx, No. xx, American Chemical Society.

[11] Deublein, D. & Steinhauser, A. (2008). Biogas from waste and renewable resource, Wiley-VCH Verlag GmbH & Co. KgaA, Weinheim.

[12] Igoni, H.I., Ayotamuto, M.J., Eze, C.L., Ogaji, S.O.T., & Probert, S.D. (2008). Designs of anaerobic digesters for producing biogas from municipal solid-waste. Applied Energy, Vol.85, pp 430-438.

[13] Yang, Y., Zhang, Z., Zhao, R., & Sugiura, N. (2011). Development of a high-efficiency methane fermentation process for hardly degradable rice stalk. Symposium Paper JIFS, Vol.1, pp.8:61.