Methane Production By Anaerobic Co-Digestion Of Sewage Sludge And Wheat Straw Under Mesophilic Conditions

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Abstract: This study investigated the possibilities of improving methane yield production from, the anaerobic co-digestion of wheat straw (WS) and primary sludge (PS). The batch experiment was conducted under mesophilic conditions. Different mixtures of WS and PS depending on its C/N ratio were carried out to investigate the optimum C/N ratio for effective methane production. The cumulative methane yields (CMYs) for co-digestion of PS with WS at C/N ratios of 35, 25, 20, 15 and 10 were 1.29, 1.62, 1.33, 2.44 and 2.16 time than digesting PS alone, respectively. The maximum CMYS was observed at C/N ratio of 15 with an increase of 89 %, 50.93 %, 83.61 % and 13.12 % compared with the other C/N ratio of 35, 25, 20 and 10 respectively. This result showed the positive synergy of co-digesting of PS and WS for methane production caused by improving the C/N ratio of the feed stock.

Index Terms: Anaerobic co-digestion, C/N ratio, Primary sludge, Methane potential, Wheat straw.

I. INTRODUCTION

One of the most urgent problems facing many countries is the disposal of their sewage sludge (SS) [Rizzardini et al. 2014] and crop residues. The amount of SS has been increased worldwide as a result of an increase in the amount of wastewater being treated [Smith S. R.]. Anaerobic digestion, consider one of the most suitable treatment technique for sewage sludge and other organic wastes. It converts sewage sludge [Zhang 2014], animal wastes [Moller 2004] and crop residues [Lee et al. 2013; Saheto et al. 2013 and Zhang 2013] into methane-rich biogas, which is can be used for heating and electricity [Whiting et al. 2014]. In addition as a result of global warming, increases in waste disposal and energy costs and the need for environmentally sustainable waste management, this technology has received great attention, especially in rural areas of developing countries [Wang 2012].

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Anaerobic digestion is the most common treatment technique for sludge stabilization [Wang et al. 2008], resulting in a reduction in the amount of volatile solids (VS) with biogas production by a variety of microorganisms degrade organic matter into several intermediate products that are converted into a renewable energy source known as methane (CH4) [Mussoline 2014]. In the other side sewage sludge is a poor feed source for anaerobic digestion as it is high in nitrogen and low in carbon (Kim et al. 2004). Thus as a result both of the paucity of carbon and the ammonia released during digestion the methane yield is reduced, which may cause inhibitory to the process. As a consequence it would prove beneficial to supply the additional feed needed to utilise the extra capacity, as an easily degradable, high carbon substrate. Co-digestion in admixture with sewage sludge would then further enhance the methane yield [Horan et al 2011]. There are a lot of potential substrates that could be used in admixture during digestion. For example crop residues such as wheat straw [Wang et al. 2009 and Wang 2012], rice straw [Kim et al. 2012; Komatsu et al., and Mussoline et al 2014], and corn stalks [Jorge et al. 2013] are produced in large quantities in over the world every year, which, due to its organic nature, can be a valuable alternative feedstock for biogas production. The advantages of co-digesting plant materials with organic waste as sewage sludge lie is that sewage sludge can provide buffering capacity and improved balance of nutrients [Sosnowski et al. 2003], while the added plant materials with high carbon content can improve the C/N ratio of the feedstock, thereby decreasing the risk of ammonia inhibition to the digestion process [Hills and Roberts 1981]. These positive synergistic effects were considered providing potential for higher methane yields [Mata-Alvarez et al., 2000]. Crop residues certainly deserve more research attention for being used as a feedstock for co-digestion with organic waste as swine manure, because of its large unexploited benefits for biogas production via anaerobic digestion [Zhu J.]. The C/N ratio is an important indicator for controlling biological treatment systems. It appears that the ideal C: N ratio is waste specific over a range from 9 to 30 [Horan et al 2011]. Jun Zhu used swine manure with crop residues mixtures and reported optimal operation with a C: N ratio of 20:1 with increases up to 11 folds in cumulative total biogas production observed for corn stalks, as compared to the control. Rizk et al. (2007) used FVW and sewage sludge and found this was optimal at a C: N of 20:1. Wang et al 2009 observed higher methane production from co digestion of swine manure with wheat straw. However, pretreatment of wheat straw was not effective in increasing the methane yield. Kim et al., 2012, studied that co digestion of rice straw and sewage sludge in batch digester, the results show high biogas production by adding rice straw to sewage sludge. Komatsu et al., showed that co digestion of sewage sludge and rice straw in mesophilic thermophilic digester, the results indicated that increased methane production by 66 to 82 % in mesophilic digesters and by 37 to 63 % in thermophilic digester. Abdul Razaque S., 2013 reported the co digestion of crop residues and buffalo dung, the result showed that the maximum biogas production was obtained from co digestion with wheat straw. Wu et al., (2010) studied the co-digesting of swine manure with three crop residues (corn stalks, oat straw, and wheat straw), and reported that all crop residues increased biogas production and net CH4 volume at all C/N ratios. Zhang et al. 2013 reported that the Combination of goat manure with corn stalks or rice straw significantly improved biogas production at all carbon-to-nitrogen (C/N) ratios. In most of the studies about co-digestion of sewage sludge with agricultural residues, they usually used rice straw and corn stalks as co substrates to improve biogas production, but there are fewer studies about co digestion of sewage sludge with wheat straw. The overall goal of this study is to expand the knowledge about co-digestion of sewage sludge and wheat straw to apply the final result in my country (EGYPT). Specific research objectives are to evaluate the optimized C: N ratio enhancing the highest methane yield, rate of methane production and anaerobic biodegradability.

II. METHODOLOGY

A. Preparation of Substrate

Wheat straw (WS) was dried at room temperature. The size of the dried wheat straw was reduced by using hammer mill followed by the coffee grinder and brought up to the size of <1.0mm. The recommended size range of straw was 0.3 to 1 mm for the economical and energy-saving consideration (Yong et al 2015). The sample was placed in to the plastic bag for later determination of its characteristics and BMP. Sewage sludge taken from a full scale municipal wastewater treatment plant located in NANTES operated with the activated sludge method, after that the PS dried to stabilize for the future using.

B. Inoculum

Inoculum used in this study was fresh cow manure obtained from the small farm located near NANTES. In order to ensure degradation of easy degradable organic matter still present in the inoculum and remove dissolved methane, the inoculum was stored with an anaerobic headspace, for readapting the inoculum to 37 _C, the inoculum was putted in the water path in 37 _C for three days before the BMP test starting [Hansen et al. 2004].

C. Analytical Methods

Total solids (TS), volatile solids (VS) and pH were determined according to the APHA Standard Methods [APHA]. Total nitrogen (TN), total carbon (TC), total oxygen (TO), and total hydrogen (TH) were estimated with a thermal conductivity detector by FLASH EA 1112 Series CHN Analyzer. The produced biogas was measured daily by water displacement method and its composition was measured by Clarus 500 Gas Chromatograph (Perkin Elmer).

D. Biochemical methane potential (BMP) test

Anaerobic batch digestion tests were carried out in duplicate under mesophilic conditions at 37 ± 1°C, which is most favorable temperature to methanogenic microorganisms [Krishania et al. 2013]. The 500 ml glass bottles were used as reactors with effective volume of 400 ml and the gas phase was 100 ml. To obtain the best mixing ratio of the co-digestion of primary sludge (PS) and wheat straw (WS) for effective biogas productions, five different mixing C/N ratios at 35.55 (R1), 25.31 (R2), 20.12 (R3), 15.38 (R4) and 10.08 (R5) were tested as shown in Table 1. Each bottle was partially filled with 150 ml of inoculum and an appropriate amount of digesting substrate with a VSinoculum/VSsubstrate ratio of 2 to minimize diffusion limitation and to avoid acidification or toxicity inhibition [Rico, C., et al. (2014)]. The total organic load of feed-stock in each bottle was 7.73 g VS/L (3.09 g VS/400 ml). Tap water was added to all the bottles to reach the working volume of 400 ml. The initial pH level for all bottles was adjusted by using 6 M NaOH to a value of 7±0.1 [Horan et al. 2011]. Unmixed PS (R6 (PS: WS=100:0)), WS (R7 (PS: WS=0:100)) and only inoculum were anaerobically digested as controls. The bottles were sealed with silica gel stoppers, and the air was purged with N2 to produce absolute anaerobic conditions. All reactors were mixed manually for 1 min once a day before the gas volume was measured [Zhang et al. 2014] and were immersed up to half of their height in a temperature controlled using water path, kept at a constant temperature of 37 ±1°C by Polystat 23 (Bio Block Scientific). The quantity of methane produced was measured by water displacement (Fig. 1). Each reactor have a cape with two plastic tubing, one end was connected to the CO2 separating bottle, while the other was closed by installing plastic tubing clamps strikethrough piece of rubber. Methane produced from the inoculum was subtracted from the sample assays. The observed methane gas production was obtained by using Equation (1),

$$BMP_{observed} = \frac{V_{(ino+feedstock)} - V_{ino}}{\mathrm{mVS}_{feedstock}} \quad (1)$$

Where BMP observed is the observed biochemical methane potential (ml CH4/g VSadd), V (ino + feedstock) is the volume of methane produced by inoculum and substrate (ml CH4), Vino is the volume of methane produced by inoculum alone (ml CH4) and mVS feedstock is mass of volatile solids in substrate (g VSadd). The experiments were stopped when there was no methane production observed. After methane production stopped, the digestate were finally sampled to determine TS, VS and pH to measure the VS removal rate and biodegradability of the feedstock.

Fig. 1. *BMP test set up*

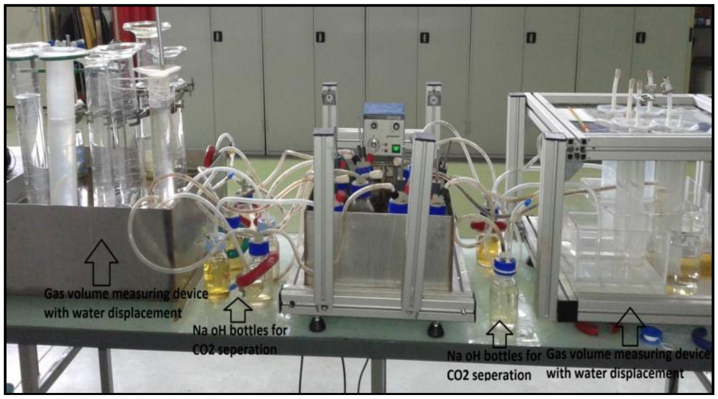


Table 1

Experimental design: different amount of substrates and inoculum in co-digestion of PS with WS. Details of batch experiments design w

Reactor Number	R1	R2	R3	R4	R5	R6	R7
C/N ratio	35.55	25.31	20.12	15.38	10.08	5.96	158.73
PS (g vs)	0.43	0.65	0.85	1.15	1.80	3.09	0
WS (g vs)	2.66	2.44	2.24	1.94	1.29	0	3.09

E. Setting CO2 Separation Unit

The easy method to separate the methane from biogas by passing biogas from the solution of sodium hydroxide (NaOH) [Horan, N.J., 2009]. The 3M NaOH solution was used to absorb CO2 [Saheto et al. 2013]. The prepared solution was then filled in 250 ml glass bottle up to 230 ml. The bottles were sealed with plastic cap having two plastic tubes. One end of the CO2 separating bottle was connected to the reactor bottle and the other to the gas measuring device with water displacement method. The complete arrangement of the methane potential test setup is shown in Fig. 1.

III. RESULTS AND DISCUSSION

A. Characterization of Substrates

One of the most important steps for successful anaerobic digestion is the characterization of the substrates to ensure that it is balanced in terms of carbon, nitrogen, TS and VS content and where waste are to be blended, to ensure the optimum blend. The characterization results of feed stock and inoculum are shown in Table 2. As expected, wheat straw (WS) was rich in carbon content and organics (VS) at 47.62 % and 95.64 % respectively (on dry basis), while had low nitrogen content at 0.3 %. In contrast primary sludge was characterized by high nitrogen content at 6.70 % (on dry basis) and low carbon content at 39.90 %. Also WS had a higher C/N ratio than PS at 158.73% and 5.96% respectively. It should thus be easy to adjust the optimal digestion C/N ratio to improve the biogas production.

Table 2

Characteristics of substrates and inoculum used in BMP test.

Characteristics	Primary sludge	Wheat straw	Inoculum
VS (%)	82.50	95.64	73.28
TS (%)	81.70	90.82	5.59
C (dry wt.%)	39.90	47.62	ND
N (dry wt.%)	6.70	0.30	ND
O (dry wt.%)	28.30	43.37	ND
H (dry wt.%)	5.40	6.10	ND
C/N ratio	5.96	158.73	ND
pH	ND	ND	6.75

ND = Not determined.

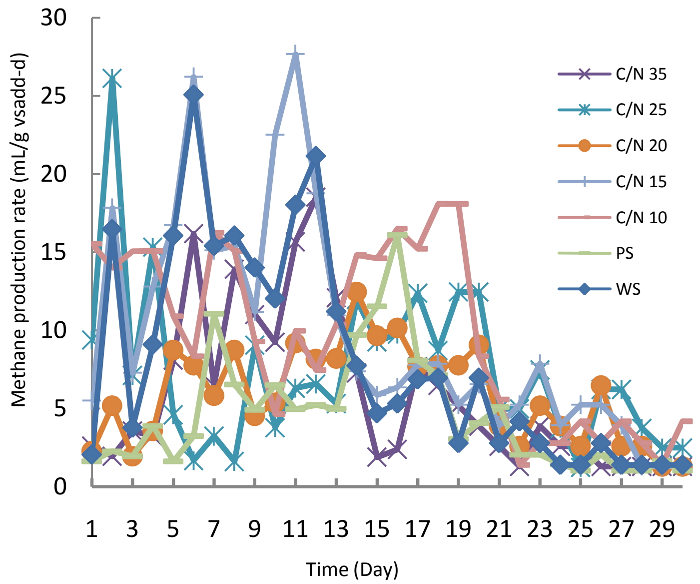
B. BMP test Assays

The daily methane production from co-digestion of primary sludge (PS) and wheat straw (WS) obtained at the incubation of 30 days under mesophilic conditions at 37±1°C is shown in Fig. 2. It can be observed that the production rate of methane decreases gradually after 20 days of incubation. The methane production for the first 15 days of the test was ranging from 55-79%, whereas within 20 days it accounts from 80-91% of the total methane produced within 30 days of BMP test. The digester retention time is the key process design constraint that is selected to ensure that the microorganisms in the reactor have adequate time to grow and reproduce [Li and Fang, 2007]. While it is important for economic success to ensure that the digester is operated at the maximum rate of gas production. For the co-digestion of PS and WS the 20 days retention time may be kept.

C. Methane production of PS and WS

The methane production rate and cumulative methane yield of PS and WS are shown in Fig. 2. For PS, the maximum peaks of the daily methane yield were occurred on day 16 (16.10 mL/g VS-d) and on day 7 (11.10 mL/g VS-d). For WS, the maximum peak of the daily methane yield were occurred on day 6 (25.10 mL/g VS-d) and on day 12 (21.20 mL/g VS-d) (Fig. 2). The two peaks of the daily methane yield of WS is more earlier and higher than the peaks of the PS, This may be due to the small particle size of the WS which is very easy for micro organisms to digestate it, and the high carbon content on WS than PS. At the end of the 29-days digestion, the CMY obtained from WS (243 mL CH4/g VSadded) was about 1.78 times higher than that from PS (136.80 ml CH4/g VSadded). WS has been demonstrated in many cases as an excellent substrate for anaerobic digestion [Wang et al. 2009 and Wang et al. 2012]. The methane yield of WS alone in this experiment was higher than the methane yield (121.20 CH4/g VS add) reported by Wang et al. 2012 and lower than the methane yield (322 CH4/g VSadded) achieved by Saheto et al. 2013, However, The methane yield observed from PS in our study (136.80 mL/g VSadded) was lower than 239 mL/g VSadded to 267 mL/g VSadded reported by Komatsu et al., The lower methane production may be due to the dried matter of the primary sludge.

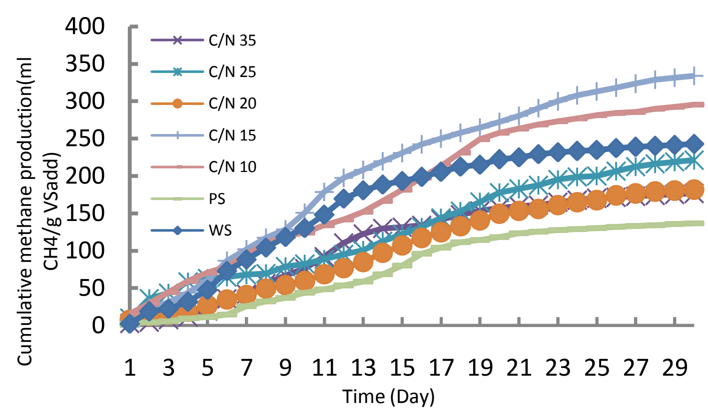
Fig. 2. *Daily methane production from the co-digestion of PS and WS with different C/N mixing ratios.*



D. Methane production from the anaerobic co-digestion of PS and WS

As shown in Fig. 2, For the co-digestion of PS and WS, the maximum peaks of the daily methane yield at C/N ratio of 35, 25, 20, 15 and 10 were occurred on day 12 (18.80 mL/g VS-d), day 2 (26.10 mL/g VS-d), day 14 (12.50 mL/g VS-d), day 11 (27.70 mL/g VS-d) and on day 18 (18.10 mL/g VS-d) , respectively. The cumulative methane yields (CMYs) for co-digestion of PS with WS at C/N ratios of 35, 25, 20, 15 and 10 were 176.70, 221.30, 181.90, 333.90 and 295.20 mL/g VSadded, respectively (Fig. 2), which showed a higher methane yield of 1.29, 1.62, 1.33, 2.44 and 2.16 time than digesting PS alone, respectively, and a higher methane yield of 1.37 and 1.22 time than digesting WS alone at C/N ratio of 15 and 10. However, for the other C/N ratio of 35, 25 and 20 showed a decrease in CMYs of 0.73, 0.91 and 0.75 time digesting WS alone, respectively. This may be occurred as a result of the high percent of carbon content (from WS) and low percent of primary sludge in these C/N ratios than C/N ratios of 15 and 10. Biogas is more easily produced from primary sludge than from excess sludge from activated sludge process with biological nutrient removal. Primary sludge is easily bio-degradable since it consist of more easily digestible carbohydrates and fats [E. Levlin], With the increase in C/N ratio, the CMYs initially increased to a peak at a C/N ratio of 15:1 and then declined (Fig. 3). The maximum CMYS was observed at C/N ratio of 15 with an increase of 89 %, 50.93 %, 83.61 % and 13.12 % compared with the other C/N ratio of 35, 25, 20 and 10 respectively. Similarly, Horan et al. 2011, studied the co-digestion of sewage sludge and industrial food waste and reported that Optimum methane yield occurred at a C:N ratio of 15 [Horan et al 2011]. Also, Sievers and Brune (1978) used sewage sludge and paper pulp mixtures and showed optimal operation with a C: N ratio of 16:1. The second maximum C/N ratio occurred at C/N ratio of 10 with an increase of 67.07 %, 33.42 % and 62.31 compared with the C/N ratio of 35, 25 and 20 respectively. In most of the studies about co-digestion of sewage sludge with agricultural residues, they usually used rice straw and corn stalks as co substrates to improve biogas production, but there are fewer studies about co digestion of sewage sludge with wheat straw. The maximum CMYs observed was 334.90 mL/g VSadded, which was higher than reported by Wang et al. 2012 (234.7 mL/g VS- co-digestion of dairy, chicken manure and wheat straw), Nallathambi et al. 1997 (300.93 mL/g VSadded), Komatsu et al. (311 ml/g VSadd-Co-digestion of sewage sludge and rice straw) and Saheto et al. 2013 (322 CH4/g VSadded) with an increasing of 42.29%, 10.97, 7.38% and 3.71%, respectively. The second highest BMP observed was 295.20 mL/g VSadded for C/N ratio of 10, which was 25.78% higher than reported by Wang et al. 2012. Also the high CMY in C/N ratio of 15 and 10 may be due to they contain a low percent of WS than other C/N ratios and the WS composed with a high percent of lignin (Wang et al., 2009). In addition, crop residues are primarily composed of cellulose, hemicelluloses, and lignin, among which lignin is the least degradable material in anaerobic digestion [Wu et al. 2010].

Fig. 3. *Cumulative methane yields production from the co-digestion of PS and WS with different C/N mixing ratios.*



E. VS removal rate and pH characteristics of the digestates

As shown in Fig. 3, the volatile solids (VS) removal rate in digesting WS alone is higher than digesting PS alone with a value of 50.58% and 55.88%. This may be due to the small particle size of WS which it is easy to digestate by micro organisms. Besides, the special cell structure of PS could be another reason for the lower VS removal rate [Nges and Liu 2009]. The maximum VS removal rates for the co-digestion of PS and WS occurred at C/N ratios of 10 and 15 were 63.59% and 61.68% (Fig. 3) and this value is more than digesting PS or WS alone, whereas the minimum value was occurred in C/N ratio of 20 and 35 with value of 56.38% and 57.20% VS. This means that the micro organism transfer the Carbone content to methane during the methanisation process so that the maximum CMYs occurred on the same C/N ratio of 15 and 10. This result showed the positive synergy of co-digesting of PS and WS caused by providing balanced nutrients and improving the biodegradability of the feed stock. It is important to maintain pH in desired range for efficient gas production [Ward et al. 2008], because it directly affects the growth of microbes [Krishania et al. 2013], so the initial pH level was adjusted by using 6 M NaoH to a value of 7±0.1. The systems maintained within a stable neutral range 7.21 to 7.66 as shown in Fig. 3 and table 3. Similarly, Wang et al. 2012 also found that the final pH of digestate varied from 6.44 to 7.82. Methanogenic bacteria work effectively at the pH range of 6.5 and 8 [Sibiya and Muzenda]. The results showed that the co-digestion of PS with WS enhanced the buffer capacity of the AD system.

Fig. 4. *Volatile solids removal rate and pH for the co-digestion of PS and WS with different C/N mixing ratios.*

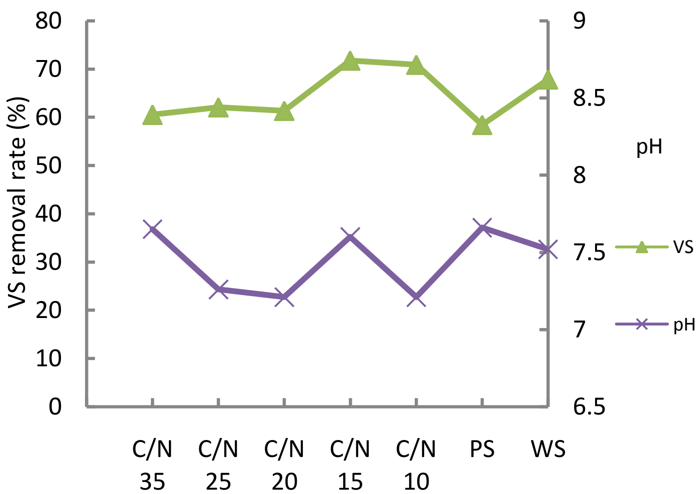


Table 3
Cumulative methane yields, volatile solids removal rate and pH for anaerobic co-digestion of PS and WS

	C/N 35	C/N 25	C/N 20	C/N 15	C/N 10	PS	WS
VS removal rate (%)	57.20	59.80	56.38	61.68	63.59	50.58	55.88
pH	7,65	7,26	7,21	7,60	7,21	7,66	7,52
Cumulative methane yields (mL CH4/g VS)	176.70	221.30	181.90	333.90	295.20	136.80	243

IV. CONCLUSIONS

Co-digestion of PS and WS depending on its C/N ratio increases both daily and cumulative methane yields (CMYs) produced during anaerobic digestion. The cumulative methane yields (CMYs) for co-digestion of PS with WS at C/N ratios of 35, 25, 20, 15 and 10 showed a higher methane yield of 1.29, 1.62, 1.33, 2.44 and 2.16 time than digesting PS alone, respectively, and a higher methane yield of 1.37 and 1.22 time than digesting WS alone at C/N ratio of 15 and 10. However, for the other C/N ratio of 35, 25 and 20 showed a decrease in CMYs of 0.73, 0.91 and 0.75 time digesting WS alone, respectively. The highest CMYS was observed at C:N 15 with an increase of 89 %, 50.93 %, 83.61 % and 13.12 % compared with the other C/N ratio of 35, 25, 20 and 10 respectively. The highest removal rate of volatile solids of 63.59 and 61.68% were achieved at C:N of 10 and 15 and this value is more than digesting PS or WS alone, while the minimum value was occurred in C/N ratio of 20 and 35 with value of 56.38% and 57.20% VS. This result showed that the co-digesting of PS and WS can enhance the methane production by improving the C/N ratio of the feedstock. The system maintain in a stable condition by enhancing the buffer capacity of the AD system and improving the biodegradability.

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