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The Study of Slurry Recirculation to Increase Biogas Productivity from *Jatropha curcas* Linn. Capsule Husk in Two Phase Digestion

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Abstract

The purpose of this study was to obtain the best slurry recirculation treatment for two-phase digester system which produced the highest biogas production in the DH-JcL substrate. The study was conducted in the laboratory using 750 mL and 2 L of plastic digester and biofilter plastic as growth immobilize. The slurry recirculation treatment consisted of the recirculation of 50 % slurry to methanogenesis digester, 50 % slurry to hydrolysis digester, and 25 % slurry to hydrolysis digester combined with 25 % to methanogenesis digester. The highest daily biogas production was obtained from the recirculation of 50 % slurry to hydrolysis digester. However, the best slurry recirculation treatment, which was more stable in longer period, was 50 % slurry to methanogenesis digester.

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Keywords: Biogas; dried husk *Jatropha curcas* Linn.; slurry recirculation; two-phase digester system

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| Nomenclature | |
|----------------|---|
| DH-JcL | Dried husk <i>Jatropha curcas</i> Linn. |
| JcL | <i>Jatropha curcas</i> Linn. |
| wk | week |
| d | day |
| VFA/alk | volatile fatty acids/alkalinity |

1. Introduction

Jatropha curcas Linn. (JcL) is one of biofuel crops. The product of JcL is jatropha oil - PPO (pure plant oil), which is only 14 % of the total harvest volume, while the rest are in the form of capsule husk, seed cake, and seed coat [1,2], which accounted for 86 % of the total harvest volume and have not been well utilized.

Nolten [2] recommended capsule husk as a source of biogas. However, in previous studies, DH-JcL (dried husk JcL) was not successfully used as substrate for biogas because it contained organic material with high buffering capacity [3], especially high ratio of VFA/alk [4], thus the digester became unbalanced [5,6]. In the unbalanced digester system, there was an accumulation of short-chain organic acids or volatile organic acids or evaporated fatty acid, which includes acetic acid, propionic acid, butyric acid, valeric acid, isovaleric acid [7], caproic acid, and enanthic acid [8]. The impact of this organic acid accumulation was inhibition of methane-producing archaea activity [5,9] resulting in low biogas production.

Several methods can be used to increase biogas production in the digester system [10,11], i.e.

- Utilization of additives,
- Slurry recirculation,
- Variation of operational parameters such as temperature, hydraulic retention time (HRT) and substrate particle size, and
- Utilization of fixed film/biofilter.

During slurry recirculation, microbial population was added to the system in order to replace the washed out microbial populations [10]. Previous study proved that 50 % hydrolysis slurry recirculation with DH-JcL substrate could balance two-phase system (with glass wool as anorganic growth immobilize), thus increasing biogas production [12,13]. This paper is reporting further study on recirculation with DH-JcL substrate for two-phase digester with special plastic pieces designed as anorganic growth immobilizer. The study was important because there was an indication that recirculation didn't improve total biogas production [10,14] and there was a possibility that the method with addition of ammonia inhibited anaerobic digestion [15,16]. The study was aimed to obtain the best slurry recirculation treatment for highest biogas production.

2. Material and method

This experiment was conducted in the research laboratory of PT Bumimas Ekapersada, Bekasi, West Java, from March to June 2013. In two-phase digester system (Fig. 1), a 2 L plastic digester was used as methanogenesis digester and a 750 mL plastic digester as hydrolysis digester (Fig. 2a). The experiment was arranged in a complete randomized design (CRD) with three replications in 32 °C water bath. Biofilter plastic (special plastic pieces designed with > 95 % void space), which used as growth immobilize, was placed into the methanogenesis digester (Fig. 2b). This study used DH-JcL JatroMas toxic cultivar and similar slurry starter of DH-JcL digester.

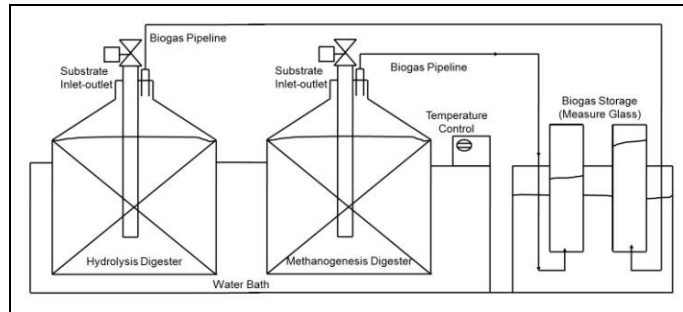


Fig. 1. Schematic of two-phase digester (adapted from [17]).

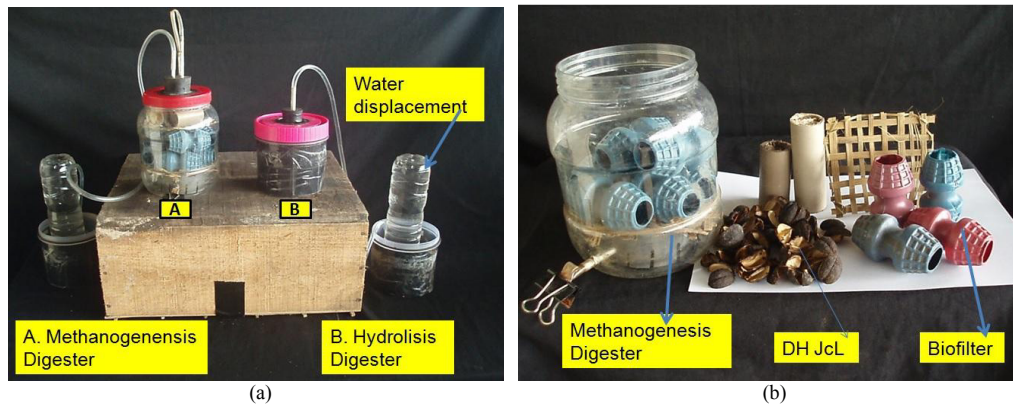


Fig. 2. (a) Two-phase digester; (b) methanogenesis digester with biofilter plastic.

DH-JcL was used as hydrolysis digester substrate. The amount of DH-JcL in control treatment was 35 g and 52 g in other treatments. Substrate in hydrolysis digester was mixed with water solvent with the ratio of 1 : 8 [18]. Feeding was conducted every day with draw and fill method [19]. Digester hydraulic retention time in this study was set for 12 wk. Every 4 wk (one period), hydrolysis digester contents were removed and replaced with new substrate. The observed variables were biogas production volume using water displacement method [20], pH and temperature in the effluent using pH meter and digital thermometer, and acetic acid content using titration method.

Four treatments were used in this study:

- Treatment A: Recirculation of 50 % slurry to methanogenesis digester
- Treatment B: Recirculation of 50 % slurry to hydrolysis digester
- Treatment C: Recirculation of 25 % slurry to hydrolysis combined with 25 % to methanogenesis digester
- Control

3. Results and discussion

3.1. Temperature and pH observation

This study was performed with the temperature range of 30.85 °C to 33.12 °C in the hydrolysis digester and 31.43 °C to 32.68 °C in the methanogenesis digester. The ideal temperature range for mesophilic microbial growth is 30 °C to 35 °C [17] so the temperature range used in this study was ideal.

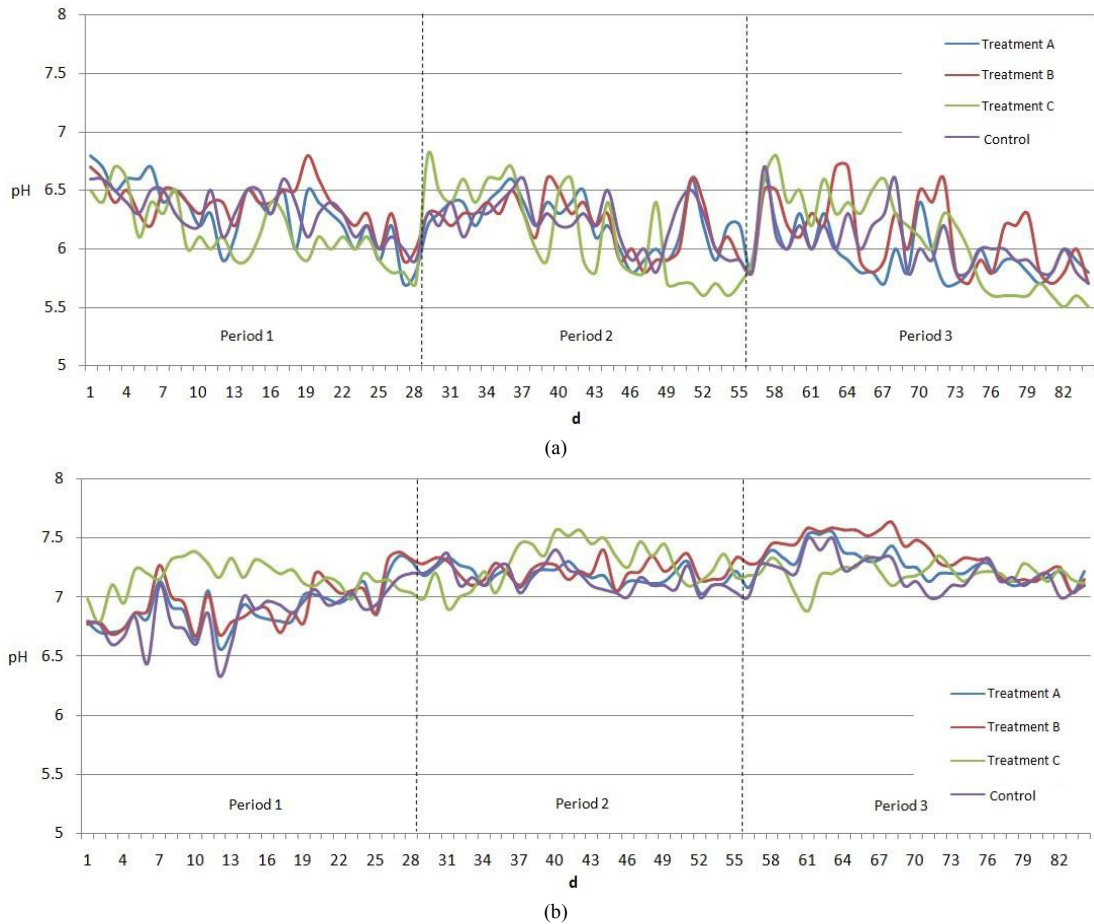


Fig. 3. The dynamic of slurry pH in (a) hydrolysis digester & (b) methanogenesis digester.

The anaerobic degradation process is highly pH dependent because each microbial group involved in the reactions has specific pH range for optimal growth [15]. The ideal pH in hydrolysis digester is ranging from pH 5.0 to pH 7.0 and the ideal pH in methanogenesis digester is pH 6.0 to pH 8.5 [17], so the range of pH used in this study was normal (Fig. 3a and Fig. 3b).

Fig. 3a showed that in the hydrolysis digester, pH was decreasing from the beginning to the end of one period. The occurrence of this dynamic was indicated by acidogenesis activity in the hydrolysis digester. After the removal and replacement of the hydrolysis digester content with new substrate at the end of each period, the pH increased suddenly. Generally, pH in the methanogenesis digester was increasing (Fig. 3b). The pH dynamic phenomenon was due to acetic acid content decrease after the change from acetic acid to methane.

3.2. Biogas production

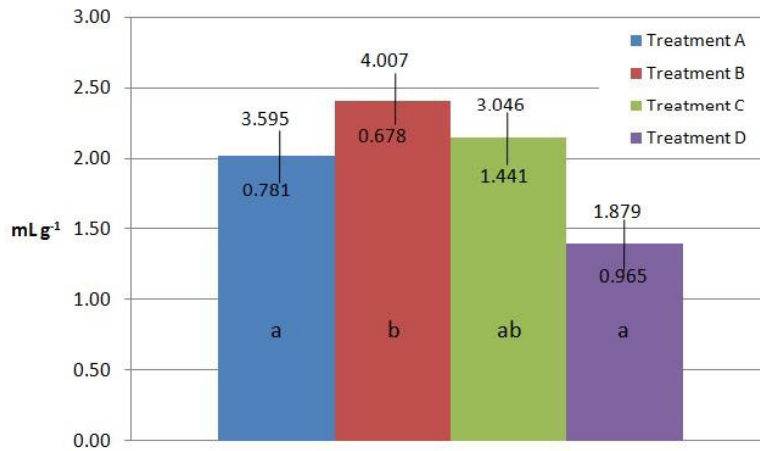


Fig. 4. Daily biogas production ($\text{mL} \cdot \text{g}^{-1}$) in methanogenesis digester.
Note: treatments with the same letter were not significantly different according to Duncan test 5 %.

Fig. 4 showed that treatment B has the highest daily biogas production while the control produced is the lowest. Based on this data, there were two assumptions for this condition:

- Recirculation treatments can increase biogas production, with slurry recirculation (to methanogenesis digester) means augmentation - adding archaea [10,21] and/or by the addition of ammonia (to hydrolysis digester) as a buffer so that the process is more stable [22,23]. The result supports previous studies [12,13].
- pH is a poor indicator. Even though the results of section 3.1 showed that pH was in normal condition, biogas productions still varied. This was because DH-JCL is an organic material with high buffer capacity, thus the change in pH value will be relatively small [3,24]. The result supports previous studies [18,25].

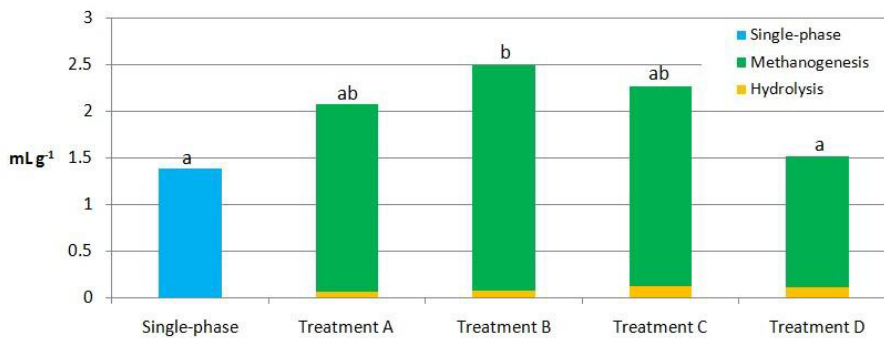


Fig. 5. Daily biogas production ($\text{mL} \cdot \text{g}^{-1}$) two-phase vs. single-phase.
Note: treatments with the same letter were not significantly different according to Duncan test 5 %.

Fig. 5 shows that the daily biogas productions of the four treatments (two-phase digester system) were higher compared to single-phase digester system. Daily biogas production in Treatment A, C, and control were not significantly different with single-phase digester system. Treatment B produced the highest biogas production and was significantly different from the single-phase digester system and the control.

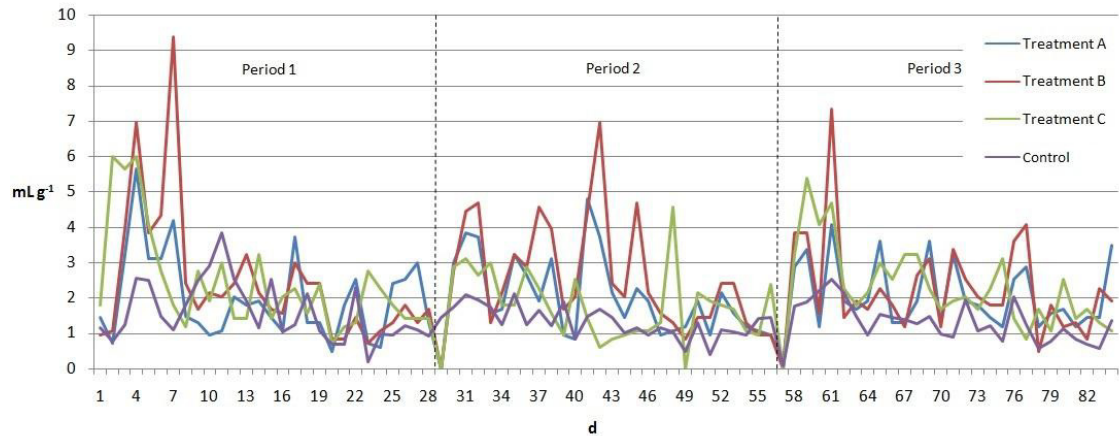


Fig. 6. Daily biogas production ($\text{mL} \cdot \text{g}^{-1}$) in methanogenesis digester.

Recirculation treatment had other positive impact, such as the peak of daily biogas production in treatment A, B and C were higher and tended to be earlier than the control (Fig. 6). Exception was in 2nd Period, the peak of daily biogas production in the control was earlier than the other treatments. Daily biogas production curve in the 2nd Period, which was fluctuated in the beginning and middle of the period and decreased at the end, differed from the 1st and 3rd Periods where the daily biogas production curve was increased and decreased precipitously at the beginning and then stabilized in the middle and decreased at the end of the period.

In three periods it appeared that biogas production tended to be high in the beginning and decreased at the end of the period. This was because DH-JcL raw material was only added once into the two-phase digester system for this period.

3.3. VFA observations

VFA content is recommended as indicator for high buffered material [8]. Approximately 85 % of the VFA content of anaerobic digester is acetic acid [26], therefore VFA content can be represented by acetic acid content. Acetic acid content in hydrolysis and methanogenesis digesters can be seen in Table 1.

Table 1. Acetic acid content ($\text{g} \cdot \text{L}^{-1}$) in hydrolysis and methanogenesis digester.

| Treatment | | Period 1 | | | | Period 2 | | | | Period 3 | | | |
|-------------|----------------|----------|--------------|--------------|--------------|----------|--------------|--------------|--------------|----------|--------------|--------------|--------------|
| | | 7 | 14 | 21 | 28 | 7 | 14 | 21 | 28 | 7 | 14 | 21 | 28 |
| Treatment A | Hydrolysis | 2.93 | 2.69 | 0.73 | 0.61 | 0.4 | 0.56 | 0.72 | 0.48* | 4.62 | 4.02 | 1.22 | 1.83 |
| | Methanogenesis | 2.32 | 0.85 | 0.61 | 0.61 | 0.4 | 0.4 | 0.32 | 0.56* | 0.97 | 0.73 | 0.49 | 0.85 |
| Treatment B | Hydrolysis | 4.88 | 2.32 | 0.61* | 3.3 | 0.56 | 0.8 | 0.48 | 0.64 | 1.46 | 0.61* | 0.73* | 0.85* |
| | Methanogenesis | 0.98 | 1.71 | 0.85* | 0.61 | 0.32 | 0.56 | 0.32 | 0.48 | 0.61 | 0.85* | 1.70* | 1.1* |
| Treatment C | Hydrolysis | 5.6 | 1.84 | 0.48 | 0.8 | 2.07 | 1.22* | 2.07 | 1.58* | 0.33 | 0.24* | 0.11 | 0.1* |
| | Methanogenesis | 0.64 | 0.48 | 0.48 | 0.48 | 1.70 | 1.95* | 1.95 | 1.83* | 0.19 | 0.34* | 0.09 | 0.11* |
| Control | Hydrolysis | 2.32 | 1.71* | 0.61 | 0.73* | 0.48 | 0.48* | 0.48* | 0.24* | 1.58 | 3.41 | 0.97 | 1.83 |
| | Methanogenesis | 1.34 | 2.08* | 0.61 | 0.85* | 0.32 | 0.64* | 0.64* | 0.4* | 0.61 | 0.85 | 0.61 | 1.58 |

Note: bold numbers with star: acetic acid accumulation.

Accumulation of VFA as an indication of unbalance process is shown with acetic acid content in the methanogenesis digester which exceeded the one in the hydrolysis digester (Table 1). These data support the result in section 3.2., that pH is not a good indicator, which is visible from the normal pH (Fig 3a and 3b). In addition, it

appeared also that the accumulation of acetic acid was higher in the control than Treatment A, B, and C (Table 1).

To simplify the value, acetic acid accumulation in Table 2a and Table 2b are counted from Table 1. Acetic acid accumulation was obtained from the total number of weeks when acetic acid accumulation occurs, divided by the total number of weeks in observation period. In all three periods, there was 42 % acetic acid accumulation in the control, which was higher than in Treatment A, B and C, which were respectively at 8 %, 33 %, and 33 % (Table 2a). In two periods (1st Period and 2nd Period), accumulation of acetic acid in Treatment A, B, C and the control were 13 %, 13 %, 25 %, and 63 % (Table 2b). When the results of Table 2a and 2b were compared, there was no additional accumulation of acetic acid in the 3rd Period of treatment A and the control (percentage of acetic acid accumulation in three periods < two periods) while in Treatment B and C, there was additional accumulation of acetic acid (percentage of acetic acid accumulation in three periods > two periods).

Table 2a. Acetic acid accumulation (%) in three periods.

| Treatment | Acetic acid accumulation (%) |
|-----------|------------------------------|
| A | 8 |
| B | 33 |
| C | 33 |
| Control | 42 |

Table 2b. Acetic acid accumulation (%) in two periods (Period 1 and Period 2).

| Treatment | Acetic acid accumulation (%) |
|-----------|------------------------------|
| A | 13 |
| B | 13 |
| C | 25 |
| Control | 63 |

Data in Table 2a and 2b supports the results in section 3.2. Due to its highest percentage of acetic acid accumulation, the control had the lowest daily biogas production (Fig. 4 and Fig. 5). Treatment A and B in terms of acetic acid accumulation had the highest biogas production potential.

Table 3a. VFA reduced efficiency in four two-phase digester treatments in two periods (Period 1 & 2).

| | Treatment | Total acetic acid content ($\text{g} \cdot \text{L}^{-1}$) | | | VFA reduced efficiency (%) |
|---------|----------------|--|----------|-------------|----------------------------|
| | | Period 1 | Period 2 | Total 1 & 2 | |
| A | Hydrolysis | 6.96 | 2.16 | 9.12 | 33.44 |
| | Methanogenesis | 4.39 | 1.68 | 6.07 | |
| B | Hydrolysis | 11.11 | 2.48 | 13.49 | 57.10 |
| | Methanogenesis | 4.15 | 1.68 | 5.83 | |
| C | Hydrolysis | 8.72 | 6.94 | 15.66 | 39.27 |
| | Methanogenesis | 2.08 | 7.43 | 9.51 | |
| Control | Hydrolysis | 5.37 | 1.68 | 7.05 | 2.41 |
| | Methanogenesis | 4.88 | 2.00 | 6.88 | |

VFA reduced efficiency is the percentage of VFA in hydrolysis digester that converted into methane in methanogenesis digester [5]. The highest VFA reduced efficiency is in Treatment B (Table 3a). This data also supports the results in section 3.2. because Treatment B has highest biogas production (Fig. 4 and Fig. 5), while the control with the lowest efficiency has the lowest biogas production.

However, the conclusion from Fig. 4 and Fig. 5 data that Treatment B is the best treatment requires caution. Further study of Table 3a was indicated in Table 3b.

Table 3b. VFA reduced efficiency in four two-phase digester treatments in total three periods.

| | Treatment | Total acetic acid content ($\text{g} \cdot \text{L}^{-1}$) | | | | VFA reduced efficiency (%) |
|---------|----------------|--|----------|----------|------------------|----------------------------|
| | | Period 1 | Period 2 | Period 3 | Total 1, 2 and 3 | |
| A | Hydrolysis | 6.96 | 2.16 | 11.69 | 20.81 | 56.22 |
| | Methanogenesis | 4.39 | 1.68 | 3.04 | 9.11 | |
| B | Hydrolysis | 11.11 | 2.48 | 3.65 | 17.24 | 41.47 |
| | Methanogenesis | 4.15 | 1.68 | 4.26 | 10.09 | |
| C | Hydrolysis | 8.72 | 6.94 | 0.78 | 16.44 | 37.71 |
| | Methanogenesis | 2.08 | 7.43 | 0.73 | 10.24 | |
| Control | Hydrolysis | 5.37 | 1.68 | 7.79 | 14.84 | 29.04 |
| | Methanogenesis | 4.88 | 2.00 | 3.65 | 10.53 | |

The VFA reduction efficiency, when followed through the three periods (Table 3b), was differed from the results in Table 3a, with the highest efficiency in Treatment A, while Treatment B was ranked 2nd. This was indicated by

the accumulation of acetic acid that occurred in the 3rd period in Treatment B (Table 1 and 2a). The problem was apparently due to the accumulation of ammonia [15,16,27]. Treatment A was more stable in longer period than Treatment B, which was indicated by having the lowest acetic acid accumulation in the three periods (Table 2a).

4. Conclusion

Three slurry recirculation treatments created more balanced two-phase digester system, which increased daily biogas production. The highest daily biogas production was obtained on the recirculation of 50 % slurry to methanogenesis digester. However, the best recirculation treatment, which was more stable in longer period, was the recirculation of 50 % slurry to methanogenesis digester.

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References

- [1] Hanratty P. *Jatropha – The real potential fuelstock international*. Amsterdam; March 15th, 2010.
- [2] Nolten W. Waterland working in synergy with the people and elements of nature. In: *Jatropha Summit*. Kuala Lumpur. February 16th-17th, 2008.
- [3] Schmidt T. Anaerobic digestion of *Jatropha curcas* L. press cake and effects of an iron-additive. *Waste Management & Research* 2011; 29(11):1171–1176.
- [4] Hendroko R, Adinurani PG, Wahono SK, Nindita A, Sasmito A, Liwang T. Performance capsule husk *Jatropha curcas* Linn. as substrate biogas in single phase anaerobic digestion. [Article in press].
- [5] Wohlgenut O. Co-Digestion of hog manure with glycerol to boost biogas and methane production. [Thesis]. The University of Manitoba, Canada; 2009.
- [6] Björnsson L, Mattiasson B, Henrysson T. Effects of support material on the pattern of volatile fatty acid accumulation at overload in anaerobic digestion of semi-solid waste. *Applied Microbiology and Biotechnology* 1997; 47:640–644.
- [7] Meilany D, Setiadi T. Pengaruh pH pada produksi asam organik volatile dari stillage bioetanol ubi kayu secara anaerobic [Effect of pH on the production of volatile organic acids from the cassava bioethanol stillage anaerobically]. In: *Proceeding of Seminar Nasional Rekayasa Kimia dan Proses Universitas Diponegoro*. Semarang. July 27th–28th, 2009. [Bahasa Indonesia].
- [8] Labatut RA, Gooch CA. Monitoring of anaerobic digestion process to optimize performance and prevent system failure. Department of Biological and Environmental Engineering. Ithaca, New York: Cornell University; 2012.
- [9] Ahring BK, Sandberg M, Angelidaki I. Volatile fatty acids as indicators of process imbalance in anaerobic digestors. *Applied Microbiology and Biotechnology* 1995; 43: 559–565.
- [10] Yavdika, Santosh, Sreekrishnan TR, Kohli S, Rana V. Enhancement of biogas production from solid substrates using different techniques—a review. *Bioresource Technology* 2004; 95(1):1–10.
- [11] Nasrin K, Beyza A. Two Experimental practice in order to increasing efficiency of biogas production by treating digestate sludge. [Thesis]. University College of Borås, Sweden; 2007.
- [12] Salafudin, Adinurani PG, Leopold ON, Yosephianus S, Liwang T, Hendroko R. Study biorefinery capsule husk from *Jatropha curcas* Linn. waste crude jatropha oil as source for biogas. *International Conf and Exhibition on Renewable Energy and Energy Efficiency*. World Renewable Energy Congress. Bali – Indonesia, October 17th–19th, 2011.
- [13] Hendroko R, Wahono SK, Wahyudi A, Adinurani PG, Liwang T, Salafudin, Sasmito A. Studies on augmentation: Recirculation slurry and EM4 to boost biogas production from *Jatropha curcas* Linn. capsule husk in two stage digestion. In: *Proceeding of Seminar Nasional Kimia UGM*. Yogyakarta, May 18th 2013. 1(1): 91–99. [Bahasa Indonesia].
- [14] Johannesson S. Evaluation of anaerobic CSTRs operated in series. [Master of Science Dissertation]. Lund University; 2008.
- [15] Kerroum D, Mossaab BL, Hassen MA. Production of biogas from sludge waste and organic fraction of municipal solid waste. In: Kumar S, editor. *Biogas Chapter 8*. [Internet] Accessed on April 5th, 2012 from <http://www.intechopen.com/books/biogas/production-of-biogas-from-sludge-and-organic-fraction-of-municipal-solid-waste>. 2008.
- [16] Griffin LP. Anaerobic digestion of organic wastes: The Impact of operating conditions on hydrolysis efficiency and microbial community composition. [Thesis]. Colorado State University; 2012.
- [17] Hendroko R. Biogas - bahan ajar mata kuliah teknologi konversi biomassa. [Biogas - a teaching materials of biomass conversion technologies course]. Pasca sarjana energi terbarukan, Universitas Darma Persada Jakarta; 2013. [Bahasa Indonesia].
- [18] Praptiningsih GA, Hendroko R, Wahono SK, Sasmito A, Nelwan LO, Nindita A, Liwang T. Optimization of concentration and EM-4 augmentation for improving bio-gas productivity from *Jatropha curcas* Linn Capsule Husk. *Int. Journal of Renewable Energy Development* 2014; 3(1):73–78.
- [19] Velmurugan B, Ramanujam RA. Anaerobic digestion of vegetable wastes for biogas production in a bed-batch reactor. *Emerg. Sci.* 2011; 1(3):478–486.

- [20] Budiyo, Kusworo TD. Biogas production from cassava starch effluent using microalgae as biostabilisator. J. of Sci. and Eng. 2011; 2(1):4–8.
- [21] Monnet F. An introduction to anaerobic digestion of organic wastes. Remade. Scotland; 2003. p.48.
- [22] Pind PF, Angelidaki I, Ahring BK, Stamatelatou K, Lyberatos G. Monitoring and control of anaerobic reactors. In: Ahring BK, editor. Berlin: Biomethanation II. Springer; 2003. p. 135–182.
- [23] Lee GAP, Duncan JM. Anaerobic digestion, gasification, and pyrolysis. Waste management and minimization. [Internet] [n.d.] Accessed on April 5th, 2014 from <http://www.eolss.net/sample-chapters/c09/e4-13-04-03.pdf>
- [24] Ojong POT. Investigation of the effects of co-digesting of biodegradable waste and swine manure on the biogas process. [Thesis]. Linköping University; 2011.
- [25] Hendroko R, Wahono SK, Adinurani PG, et al. The study of optimization hydrolysis substrate retention time and augmentation as an effort to increasing biogas productivity from *Jatropha curcas* Linn capsule husk at two stage digestion. In: Praptiningsih GA, Anggi N, Agus SY, Andi S, editors. Conf. and Exhibition Indonesia Renewable Energy & Energy Conservation 2013. Energy Procedia 2014;47: 255–262.
- [26] Gerardi MH. The microbiology of anaerobic digesters. wastewater microbiology series. New Jersey: A John Wiley & Sons, Inc.; 2003.
- [27] Shahriari H, Warith M, Hamoda M, Kennedy KJ. Effect of leachate recirculation on mesophilic anaerobic digestion of food waste. Waste Management 2012; 32:400–403.