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# Biogas Production in the Anaerobic Digestion of Paper Sludge

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# Abstract

Pulp and paper industry generates large quantity of sludge, up to 1 m<sup>3</sup> /ton paper produced. Anaerobic digestion (AD) is a potential treatment to stabilize sludge and produce biogas for renewable energy. The aim of this study was to investigate the potential of AD of paper sludge (PS) generated from primary and secondary wastewater treatment and to compare the effect of cow manure to paper sludge. For the reactor with PS only, biogas production was observed starting on the 5<sup>th</sup> day with 6.3% of methane with a steady increase. The cumulative methane yield attained to 14.7 ml/g volatile solid (VS) until day 28. The second reactor containing PS and cow manure produced methane 269 ml/g VS until day 28. This study shows a more optimal AD process of paper sludge mixed with cow manure due to a more optimum C/N ratio and also higher VS.

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# 1. Introduction

Pulp and paper is considered as one of the most polluted industry in the world [1] and energy and water intensive [2]. These processes generate wastewater which are then treated using physical, chemical as well as biological treatment. Wastewater treatment eventually produces paper sludge (PS) from chemical and biological treatment in large quantities, ranging from 0,3 to 1 m<sup>3</sup> of PS/ton paper produced. The sludge generally contains chlorinated organics, pathogens and trace amount of heavy metals [3]. In Indonesia, land application and hazardous waste treatment are the typical choices for sludge stabilization. However, land

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application rarely pays attention to long-term effect to soil and hazardous waste treatment is very costly. Thus, on-site treatment of paper sludge must be considered in order to reduce production cost and also draw benefit from nutrients contained in the PS.

Anaerobic digestion (AD) is the most common method for sludge stabilization. Moreover, this digestion process using various anaerobic bacteria produces biogas including methane that can be an alternative source of energy. AD has been successfully used for sludge treatments of various kinds for example sewage sludge [4], waste activated sludge [5] and cow manure [6]. However, very few studies have applied AD for PS treatment. Lin et al [7] demonstrated that PS combined with monosodium glutamate waste liquor can produce up to 200 ml methane/g volatile solid (VS)<sub>added</sub>, with methane reaching up to 80% of the total biogas composition. However, this study was conducted under a maintained temperature of  $37^{\circ}$ C, a condition that can be costly for medium-scale industries. A local study by Soetopo et al [8] showed that the highest biogas methane content from AD of PS is 51.5% at the rate of 140 ml/g VS in 28 days. Nevertheless, this study was conducted using only sludge from secondary biological wastewater treatment. This is also less applicable considering that in most industries; PS originating from primary physical-chemical wastewater treatment can make up to 98% of sludge produced by the pulp and paper industry. Thus, this study aims to investigate the potential of biogas production derived from primary and secondary sludge of pulp and paper wastewater treatment under uncontrolled temperature, where both conditions are found to be more applicable to medium-scale pulp and paper industry.

# 2. Material and method

#### 2.1. Substrate

The sludge originated from a pulp and paper industry in East Java, Indonesia. After the production process, wastewater goes through primary physical treatment of sedimentation and secondary treatment using aerobic suspended growth tank. Afterwards, sludge volume from both the primary and secondary treatment is reduced through primary clarifier and belt press. Sludge used in this study was collected from the sludge holding tank from primary and secondary clarifier that is composed of both primary and secondary sludge. Samples were collected in jerry cans, conserved at 4°C before feeding the reactors the day after.

Two sets of experiments were conducted in two separate reactors run in parallel, labeled R1 and R2. Sludge characteristics on day one (feedstock) were measured and presented in Table 1. The first experimental set (Reactor 1-R1) is aimed to test the effectivity of AD of PS without any seeding and co-substrate. The second experimental set (Reactor 2-R2) combined paper sludge and cow manure as seed sludge as well as to adjust C/N ratio to fit the ideal 20-30 range. Total solid (TS) of cow manure was diluted using water to approximately 20% of total solid (TS) and afterwards combined with PS sludge with a 57% to 36% volumetric composition of PS and diluted cow manure, respectively.

#### 2.2. Experimental Devices

Anaerobic digestion was carried out in batch experiments using a 15 L capacity bioreactor. Empty reactors and gas bags was purged with nitrogen and afterwards filled quickly with substrate to the top. Continous mixing was performed using an 80 rpm impeller. Temperature and pH probes were installed for daily monitoring. Each reactor had two outlets equipped with valves. The first outlet was for substrate sampling and the second outlet was connected using a 3 mm hose equipped with a valve to a 1 L polypropylene gas bag (Tedlar Bag CEL scientific corp).

Substrate	TS ( g/L)	VS (g/L)	C (%)	N (%)	P (%)	K(ppm)
Paper Sludge – Reactor 1(R1)	110.2	66.6	36.15	0.95	0.013	66
Cow Manure	93.38	44.26	52.54	2.59	0.005	406
Paper Sludge + Cow Manure – Reactor 2 (R2)	84.68	41.28	38.56	1.19	0.007	86

Table 1. General characterization of substrate in the two separate reactors

#### 2.3. Sampling

30 mL of sludge was collected from each reactor twice a week to measure for chemical oxygen demand (COD) and volatile fatty acids (VFA). COD was measured using closed reflux method and VFA was measured using titration method [10] calculated through the total volume of NaOH titrated to take pH from 4 to 7. Biogas production was measured daily by disconnecting the gas bag and replacing consequent biogas collection using another set of gas bag, except when no biogas was detected. Meanwhile, biogas volume in the current gas bag was measured using a water displacement method [9]. Afterwards, gas composition was measured using the Shimadzu GC-8A. Argon was used as carrier gas at flow 40 mL/min. Standard curves were prepared using CH<sub>4</sub>, CO<sub>2</sub>, and H<sub>2</sub> for each component (100%, 80%, 60%).

### 3. Results and Discussion

Although temperature was uncontrolled, monitoring demonstrated steady temperature ranging from 29-32.5°C indicating mesophilic range (Figure 1). pH range of R1 ranged from 6.3-6.9 showing less fluctuation compared to R2 which ranged from 6.2-7.3.

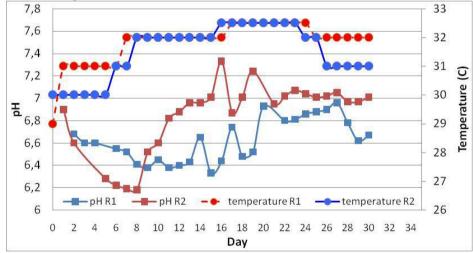


Fig. 1. Experimental pH and temperature data (R1=Reactor one containing only paper sludge and R2= reactor 2 containing paper sludge and cow manure)

Initial COD (Figure 2) of Reactor 1 containing only paper sludge was more than 5 times lower than initial COD of Reactor 2 containing paper sludge and cow manure. However, COD in reactor 1 constantly increased to five folds from 1760 to 8960 mg/L during the first 10 days of experiment, whereas COD in reactor 2

demonstrated an increase of 1.5 times during this period. The COD fluctuation trend for R1 was similarly observed for VFA production at a smaller extent. However, this similarity was less evident for R2. Considering the difference in COD and VFA trend as well as pH ranges, these results suggests that different processes dominated each reactors.

This was confirmed when the biogas production is observed (Figure 3). Both substrates did not produce any biogas until day 5. Methane production was slow until day 8 for R1.

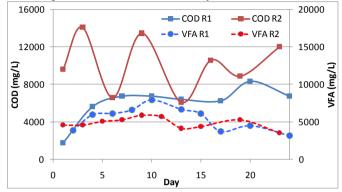


Fig. 2. Variation of COD and VFA value in the anaerobig digester pilot plant R1=Reactor one containing only paper sludge and R2= reactor 2 containing paper sludge and cow manure.

However, R2 showed significant biogas increase from day 7. Methane production peaks were observed at day 9 and day 16 for R1 at 303 and 218 mL respectively, whereas R2 demonstrated methane production peaks for day 7, 9, 13 and 18 at 387, 572, 1282, and 587 mL respectively. Methane production cumulative peaks were observed until day 28 for R1 at 1.5 L respectively, whereas R2 demonstrated methane production peaks for day 29 at 7.2 L respectively. Until day 22, cumulative methane production in R2 was 3.5 times more than R1. This is higher compared to results published by Prameswaran and Rittmann [10] where methane production from an anerobic digestion 1:1 PS and pig waste was at 0.8 L until day 80 and 0.1 L until day 40 from PS only. The cumulative yield attained to 269 mL/g VS for R2 which is higher than results shown by Lin et al. [7] with AD of sludge paper mixing with monosodium glutamate liquor waste.

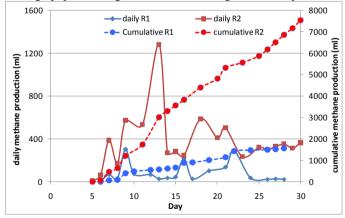


Fig. 3. Daily and cumulative methane production (R1=Reactor one containing only paper sludge and R2= reactor 2 containing paper sludge and cow manure)

Considering COD, VFA, pH and methane production, each reactor seemed to go through different pathways for methane production. VFA was considerably constant in R2 despite the fluctuation of COD as well as the high methane production. This suggested that methane production in R2 went from hydrolysis directly to methanogenesis in the first 9 days. Afterwards, methane was produced through the acetogenesis process, explained by the decrease of VFA in R2 from day 11 to 15 and an increase in methane on day 14. The slow production of methane for R1 as well as the increasing VFA suggested that R1 followed the typical AD pathway of hydrolisis, acidogenesis, acetogenesis and finally ending with methanogenesis.

# 4. Conclusion

Sludge generated from primary and secondary pulp and paper industry wastewater treatment demonstrated a high potential for energy recovery. Anaerobic digestion (AD) using sludge seeded with cow manure showed methane production of 269 mL/g volatile solid (VS) compared to AD using only paper sludge which produced 14.7 mL/g VS. Uncontrolled temperature and primary and secondary sludge combined are also two additional advantage of this process which makes it more applicable to medium industries.

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