Hybrid Upflow-Anaerobic Filter (HU-AF) Integrated technology for bio-methane generation from vinasse with shorter hydraulic retention time

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Abstract. Research about Hybrid Upflow-Anaerobic Filter (HU-AF) integrated technology for bioenergy generation from vinasse was conducted. Modification of influent flow system, expansion of filter contact area and addition of gas accumulator for biogas collection were used to enhance the degree of vinasse biodegradation biogas generation, and yet to shorten the hydraulic retention time (HRT). Hybrid Upflow-Anaerobic Filter (HU-AF) integrated technology was a double dome reactor with sequential connection, with up-flow inlet distribution inside the reactor. Total volume of HU-AF was 60 m³. A mixing and a feeding chamber, with volume 3 m³, each were installed before HU-AF reactor. A reservoir chamber with volume of 6 m³ was added after HU-AF reactor. OLR was 2.04 kg COD/day. A methanogenic seed sludge derived from anaerobic lagoon. The experiment was carried out initially by seeding, acclimatization and adaptation of microorganisms inside the reactor. A feeding ratio of vinasse: water = 1:4, and initial pH of 6 were adjusted. Result showed that Full-scale HU-AF integrated technology was able to degrade COD with maximum degradation of COD 55.45% at pH 7.1, and shorten the HRT into 32 days. Biogas production was consisted of 57.21 % methane, 13.98% CO₂ and various gases 28.1% (H₂S, VOC, CO and H₂O). This experiment showed that HRT was shorter than conventional reactor.

Keywords. ethanol industry, vinasse wastewater, bioenergi, Hybrid Upflow-Anaerobic Filter, methane generation

1. Introduction

In 2020-2030, Indonesia is predicted facing energy crisis[1]. Industrial sector, which mainly depended to fossil fuel, was the one who mostly impacted. There is an urgency to find an alternative renewable energy to fossil fuel.

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Perpres No.5/2006 National Energy Policy have set National Energy Management for developing mix primer energy in 2025, where 23% national energy come from renewable sources, particularly 8.3% derived from bioenergy. Bioenergy potential could be from forest timber, crops, palm oil, pulp and paper, starch industrial waste etc.

Ethanol industry is one of industry that based on molasses as raw materials. Beside ethanol as main product, vinasse and stillage are considered to be by products of fermentation and distillation process. Vinasse has characteristic of high organic, acidic pH, high temperature and high volume. Vinasse is considered to be bioenergy source that yet to be explored immensely[2]. The issue related to vinasse is aquatic pollution, that come from poor management of wastewater. Vinasse has pollutant concentration as follows: $BOD_5 = 109.038$ mg/l, $COD = 353.797$ mg/l and $TSS = 7,200$ mg/l, pH 4 – 5 and temperature 40-50°C [2]. Regarding to that, vinasse has large potential to be utilized as raw material for biogas/bioenergy. Every 1 kg COD removal will generate 200-350 liter methane[3].

Technology to convert high organic waste into biogas still depend on anaerobic conventional technology[4]. Bottlenecks of anaerobic conventional technology are long retention time, spacious, and low methane generation. Furthermore, even though Hybrid Anaerobic Baffle Reactor (HABR) could remove COD up to 92%, but this technology still require long retention time[5]. Another example is Up-flow Anaerobic Sludge Blanket (UASB) which converts COD >86 kg COD/m³.days, with removal efficiency of 60% but retention time is unknown.

One of ethanol industries that have utilized its wastewater is PT. Indo Acidatama, located in Karanganyar-Solo. PT. Indo Acidatama has utilized vinasse, convert it into biogas energy using four units anaerobic facultative chambers, with total volume of 120,000 m³. The chambers were covered by polymer HDPE, and have total retention time of 40-50 days. This technology has weakness, such as spacious, long HRT, dangerous gas leakage, less biogas production etc. PT Madu Baru that is located in Yogyakarta, have not used its wastewater as bioenergy source but has converted half of its wastewater into liquid fertilizer, and treated the rest in wastewater treatment plant (using conventional anerobic-aerobic technology). PT Enero, one of ethanol industry located in Mojokerto, East Java, still use conventional technology with HRT 88 days. PT Enero use two SS vertical tanks with agitation, with total volume of 30.000 liters. However, biogas is not utilized yet.

As already described, based on its characteristic, vinasse has high potential for biogas production. For 1 kg COD reduction will produce 200-350 liters' methane. Assume that vinasse wastewater volume is 307,045 m³/days, and 75% COD removed, with conversion factor of 4.500 – 6.300 kcal/m³ calories per m³ wastewater. The total amount of biogas generation will be 1.381.703 – 1.934.383,5 kcal/days. If average energy need for ethanol distillation process is 2700 kcal/L, biogas production could be used to produce 511.7-716.4 m³/days. This scheme can be applied in alcohol small industrial center in Sukoharjo that still use woods as energy sources.

This research focus on innovative reactor model for high organic wastewater conversion into biogas namely Hybrid Upflow-Anaerobic Filter (HU-AF) that could shorten hydraulic retention time to 20 -25 days. HU-AF reactors models use filters and up-flow inlet feeding to expand contact area between substrates and microorganisms.

2. Material and Method

2.1 Reactor Set up

2.1.1 Lab scale

BMP (Bio-methanation Potential Test) test was conducted to analyze the degree of COD degradation, in line with methane generation. Referred to method that was developed by [6] (**Figure 1**). Table 1 shows vinasse initial inlet characterization.

Figure 1. BMP test analyzer

Table 1. Vinasse initial inlet parameter concentration

No.	Parameter	Concentration	Units
1.	BOD	92, 045	mg/l
2.	COD	261, 206	mg/l
3.	TSS	32,450	mg/l
4.	Total N	793.8	mg/l
5.	Total P	35.25	mg/l
6.	Sulfida (sbg S ⁻²)	18.25	mg/l
7.	Sulfat (SO4 ⁻²)	2,026	mg/l
8.	MLSS	91,900	mg/l
9.	MLVSS	10,850	mg/l
10.	pH	4.7	-
11.	Temperature	55	°C

2.1.2 Full-scale application

Figure 2. Full scale HU-AF reactor lay-out

Figure 3. HU-AF reactor details

- Legends:
- A : Mixing chamber
 - B : Feeding Chamber
 - C1 : Dome 1 (without filter)
 - C2 : Dome 2 (with filters)
 - D : Reservoir chamber

Hybrid Upflow-Anaerobic Filter (HU-AF) integrated technology contained of double dome reactors (**C1 and C2**) with sequential connection, with up-flow inlet distribution inside the reactor. Total volume of HU-AF was 60 m³. A mixing (**A**) and a feeding chamber (**B**), with volume 3 m³, each were installed before HU-AF reactor. A reservoir chamber (**D**) with volume of 6 m³ was added after HU-AF reactor.

2.2 Material

For both lab and full scale application:
Vinasse with initial characteristic written in table 1 was used. For feeding, vinasse was diluted by water in the ratio of 1: 4 = vinasse: water (final concentration is shown in table 2). Lime (CaO, technical grade) with concentration of 210 ml CaO per L vinasse was used to adjust the pH from initial ph of 3.6 to 6.0. Urea and TSP (technical grade) was added in the ratio of BOD: N: P= 100: 2.5: 0.1 [7]. Macro and micronutrients, referred to [8] and [9] were added. For microorganisms seed, sludge derived from anaerobic lagoon WWTP was used.

Table 2. Characteristic inlet after dilution (vinasse: water = 1:4 ratio)

No.	Parameter	unit	Concentration
1	pH	-	6.3
	COD	mg / L	34468
3	BOD	mg / L	10459
4	MLSS	mg / L	2830
5	MLVSS	mg / L	710
6	Total P	mg / L	0.105
7	Total N	mg / L	453.6
8	Sulfide	mg / L	39.7
9	Sulfate	mg / L	<0.26
10	Nitrate	mg / L	2.473
11	Nitrite	mg / L	<0.001

2.3 Operational Condition

- **Lab-scale application (Bio-methanation Potential test)**
BMP test was conducted in batch, with volume reactor was 5 L and run for 50 days. Periodically (every 2 or 3 days) COD and pH were collected and analyzed.
- **Full-scale application**
Seeding was done through adding sludge seed derived from anaerobic lagoon WWTP alcohol industry, with volume 20% from total reactor volume (around 6 m³ each dome). Nutrients (macro, micronutrients urea and TSP) were added and reactor was left for 7 days. After 7 days, reactor was fed with vinasse wastewater with 4 times dilution and pH 6 until it was full, then left from another 7 days. Afterwards, reactor was fed daily with vinasse wastewater (with flow rate 1.5 m³/day) and running for 34 days. COD, pH and gases (CH₄, CO₂) were analyzed.

2.4 Analysis

Gases were measured **using** Gas Chromatography and COD, BOD, TSS, pH, temperature, MLSS, MLVSS, N total, P total, Sulfate (SO₄⁻), alkalinity, nitrite, nitrate, Total Plate Count (TPC) and sulfide were analyzed using APHA AWWA.

3. Result and discussion

3.1 Lab scale

BMP test (bio-methanation potential test) was conducted to know the maximum anaerobic organic degradation, which theoretically in line with potential methane production. In BMP test, two different substrates were used, Diluted inlet vinasse wastewater (4 times diluted with water) and non-diluted inlet vinasse wastewater. Diluted inlet had initial COD 38499 mg/L (OLR = 0.19 kg COD /day) and non-diluted had COD 168,516 mg/L (OLR = 0.8 kg COD/day).

Figure 4 shows organic degradation profile of both diluted and non-diluted substrate. Organic degradation was indicated by COD reduction during 50 days of BMP test. For non-diluted substrate, initial COD was 168,516 mg/L. COD was decreased to 113,844 mg/L after 27 days, and was stagnant. The maximum COD removal of non-diluted substrate was 33.52% (**figure 5**). On the other hand, diluted substrate with initial COD of 38,499 mg/L could reduce COD value into 13,213 mg/L and had maximum organic degradation of 65.68 % with HRT of 21 days (**figure 5**). Low organic degradation in non-diluted substrate was might be because of accumulation of VFA (volatile fatty acid) such as propionate acetate, butyrate that accumulated in the system. The accumulation of VFA made the pH in the system drop to below 6.0 (**Figure 5**) and turn to be slightly acidic. The accumulation of VFA and pH drop made the system unbalance, that made acidogenesis microorganisms dominant in the system and inhibit the growth of methanogens microorganisms. Methanogens growth were strictly correlated with pH, with optimum pH growth was 6.5 -7.5 [10]. In contrary, **figure 5** also shows that pH in diluted substrate system increased from 5.8 to 7.6, indication of balanced system in anaerobic condition. Thus the organic degradation could get a maximum value of 65.68%.

Figure 4. COD reduction profile during 50 days BMP test

Diluted (COD_{rem} =65.68%)

Figure 5. COD removal vs pH profile during BMP test from 2 different substrates, diluted and non-diluted substrates.

3.2 Full-scale application

Results from BMP test was then used as a basis for full scale application. Full scale application was taking place in traditional small scale industry, in Polokerto, Sukoharjo Central Java. The operational condition was divided into 3, namely seeding, acclimatization, and continuous running. Seeding was done by adding 20% sludge seed into the reactor (around 12 m³ total for 2 domes). Urea, phosphate, Macro and micronutrients were added, then reactor was left for 7 days. Acclimatization was done by filling the reactor to full with mixing of water and vinasse wastewater at the ratio 4:1. pH was adjusted to 6 using CaO. Afterwards, reactor was left for another 7 days. During seven days period, gas production was monitored using manometer that was attached to the reactor. When gas formation was indicated, continuous run was started. The total daily flowrate for vinasse wastewater was 1.5 m³/day. The daily feeding was divided into 3 times period; each was 0.5 m³ using submersible pump. Continues running was done by mixing 0.5 m³ vinasse with CaO to achieve pH 6, in mixing chamber. Afterward, vinasse wastewater was flown to the feeding chamber and pumped into the reactor, and was done three times a day. OLR was 2.04 kg COD/day. The continuous running was run for 34 days. Figure 6 showed COD reduction and COD removal profile during continuous running.

Figure 6 shows that maximum COD reduction occurred at days 34 with maximum COD reduction from 34,468 mg/L to 15,355 mg/L (55.54%). This result was lower than BMP test result, due to indication of leakage inside the reactor. Figure 7 shows that maximum methane production was 57.21% and CO₂ 13.98%, achieved at day 33. Figure 7 also shows that COD removal, methane and CO₂ generation were correlated with pH. Methane generation and COD removal increased when pH rise from 6.3 to 7.1. In contrary, the CO₂ production decreased as pH increased. This is a normal phenomenon in anaerobic biodegradation pathway using mix cultures microorganisms. Anaerobic biodegradation has 4 stages, namely hydrolysis, acidogenesis, acetogenesis, and methanogenesis[11]. High concentration of CO₂ with pH< 6.5 indicate that stage is still in the acidogenesis/fermentation phase. Normally in acidogenesis/fermentation stage, VFA (propionate, acetate, butyrate), H₂ and CO₂ are generated pH was also slightly acidic because the production of VFA (at pH around 5.5 – 6.5). To obtain the maximum methane production, the production of acetate should be

maximized. VFA must be oxidized to acetate and H_2/CO_2 also have to be synthesized to form acetate by homoacetogen. This stage is known by acetogenic phase. The increased in pH to 7.1 indicate of the release of some alkalinity in the system to buffer the pH, thus able to balance condition. The condition favors the growth of homoacetogen and methanogens. Acetate was optimally produced then consumed to form methane.

Figure 6. COD degradation and COD removal through period of experiment

Figure 7. Correlation between CH₄, CO₂ production, and COD removal with pH

4. Conclusion

BMP test showed that to get the maximum organic degradation/removal, vinasse wastewater must to be diluted. Four times dilution, make organic degradation increased from 33.52% to 65.68 %. BMP test acted as a basis for full scale application. Full scale application of methane production from vinasse was using Upflow-Anaerobic Filter (HU-AF) Integrated technology with total volume of 60 m³. Full scale application showed that maximum COD degradation occurred at days 33 when pH increased from 6.3 to 7.1. Methane production was 57.21% and

CO₂ 13,98%, the experiment showed that Hybrid Upflow-Anaerobic Filter (HU-AF) Integrated technology is able to shorten the HRT to 33 days with maximum methane generation 57.21%.

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