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Development of Bio-CSTR Design For Bio-H$_2$ From POME As Renewable Fuel

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Abstract: Along with population growth and its activities, in business-as-usual approach, the energy needs to support these activities will be even greater. Up to this point, the fulfillment of energy sources is still dominated by fossil fuels. Therefore, innovation and technology development explore all potentials related to, especially, renewable fuel. Hydrogen (H$_2$) is a potential energy carrier with an energy content 2.75 times higher energy than hydrocarbon fuels. Previous research using Palm Oil Mill Effluent (POME) as a raw material has been carried out on 2.5 dm3 and 40 dm3 scales. Based on these results, a scaling-up system was designed as a bio–Continuous Stirred Tank Reactor (CSTR) for the production of H$_2$ from a capacity of 900 dm3 by modifying the existing reactor. The bio H$_2$ production system was designed by considering the feed stream will flow from the bottom and stream up through high concentration activated sludge which will decompose the organic content in POME. POME flow up and out through the overflow pipe. Meanwhile, the biogas, H$_2$ and CO$_2$, will flow through the upper pipe and be channeled to the biogas holder. POME feeding is designed to inflow up laminar so that POME decomposition occurs gradually as indicated by the decreasing COD and BOD values at the bottom and overflow. The difference in COD and BOD concentrations in the bio-CSTR shows a positive effect on the 1 m^3 bio-CSTR. The bio CSTR was equipped with impellers in 4 different levels to maintain uniformity at each level. Thus the form of POME flow is laminar and non stagnant. The result showed COD decreased between the bottom and the overflow reached 5280 ppm. In addition, the pH only changes to a maximum of 0.1. Both data indicated that biological processes working well and do not influence the operational condition

Novelty: This bio CSTR design for biogas H$_2$ production is a modification of an existing bio H$_2$ production system that uses POME as raw material and has a working volume of around 1 m3 (1000 liters). The previous system mixed with the bottom functioning on top by using circulation in the bioreactor. There hasn't been any decent data on H$_2$ biogas production until recently. Modification of the H$_2$ biogas production system is carried out by adding a stirring system that works in a laminar flow – non stagnant. Another added feature is the heating system for pretreatment, which can be used both for conditioning the seeding culture consortium biogas H$_2$ and for the preparation of feeding into bio-CSTR. A diaphragm pump that can work for sludge is also included in the system. Currently, research on the maximum H2 biogas production is being carried out on a 2.5 liter scale[1]. Novelty in this research is to design a bio CSTR on a scale of 1 m^3 which can also be utilized to produce H$_2$ biogas from POME.

Keywords: POME; bio H$_2$; design bio CSTR; renewable fuel; laminar and non-stagnant

1. Introduction

Indonesia has relatively large energy resources from fossil fuels; however, at some point, a crisis will occur when the resources are inevitably depleted by the increasing energy consumption [2]. National energy demand will continue to rise along with the progress of technology, civilization, lifestyle, and economic growth. The increasing need for energy becomes a great challenge to overcome immediately. Therefore, the government emphasizes the importance of new and renewable energy utilization, which has abundant potential. Utilization of industrial waste with oil bladders was also carried out in some global industries, such as olive pomace for the substrate to produce biogas in Tunisia [3]. Whereas in Indonesia, as the biggest palm oil producer in the world, Palm Oil Mill Effluent (POME) is liquid waste from palm oil mills, of course, it has become a problem since its high

amount which comes along as the side product of palm oil production. Since the available fuel and chemical are fossil-based that known to be non-renewable, finding a new resource which is independent of the fossil is more desirable. POME as biomass waste is a promising alternative for fuel and chemical sources [4].

For each tonne of palm oil produced, palm oil mill will generate approximately 5.5 – 7.5 tonnes of POME[5]. Raw POME is hot colloidal suspension, brownish, and acidic[6]. It has organic content including BOD (25.000 mg/L), COD (53.630 mg/L), and grease (8.370 mg/L) [7]. In advanced technology and big country like China has also struggled with pollution problems, which are mainly caused by the period of high economic growth. Therefore, effective treatment techniques and low cost should be established in order to achieve industrial activity without serious pollution like heavy metal[8]. In our case, POME waste has a very high COD, reaching 100,000 ppm or more, which means it needs serious pretreatment before being dumped into the river. Fortunately, this POME can be used as a substrate for bio-conversion into fuel, be it biogas or bio H_2. Especially for bio H_2, it has a high value on energy and the economy.

Moreover, The POME utilization into energy is the best solution to overcome the negative impact that could arise when the POME is streamed directly to the environment. POME contains organic matter that can be used as raw material in hydrogen production.

Hydrogen (H_2) is one of the promising energy resources in the future due to its environmentally friendly and renewable nature. Hydrogen can substitute fossil fuel since it has high energy content, 2.75 times bigger than hydrocarbon fuel. For a mass unit of hydrogen, the energy produced is 122 kJ/kg[9]. On the other side, hydrogen combustion with oxygen in the fuel cell system will only generate water as a side product. It is very beneficial because the emissions of gas that triggered the greenhouse effects will decrease[10]. POME has a COD and BOD concentration in the amount of 96.300 mg/L dan 53.200 mg/L, respectively[11] that it can serve as a substrate in biohydrogen production[12].

In anaerobic digestion whereas the process with the absence or limited supply of oxygen[13], hydrogen is produced as a side-product in the acidogenic step but in a low yield. Dark fermentation was used to increase hydrogen production, although the implementation in commercial scale was still challenging [14]. In dark fermentation, substrate pre-treatment was an important factor to suppress the methanogen bacteria activity and to improve hydrogen producing bacteria activity[15]. Factors affecting biohydrogen production via the fermentative route were substrate composition, nutrition availability, mode and operation reactor, and bacteria consortium[16].

The bio-CSTR used is a tank equipped with a mixer (agitator). The agitator function to broaden the contact area of biomass so the gas production will increase. POME waste utilization was conducted through the fermentation of POME using bacteria consortium at temperature 55°C and pH 6,0 to produce the biohydrogen. The process stages of fermentation begin with the POME degradation started with hydrolysis reaction, continued to acidification reaction, and then continued to the acetate acid formation. The hydrogen formation takes place in the acetate acid formation stage. The gas analysis result shows that the hydrogen concentration can reach up to 32%. In this process, the COD conversion is about 25%. The COD content in the fermentation residue remains high, in the amount of 75%[17]. Fig. 1 below shows the outline of the POME degradation process.
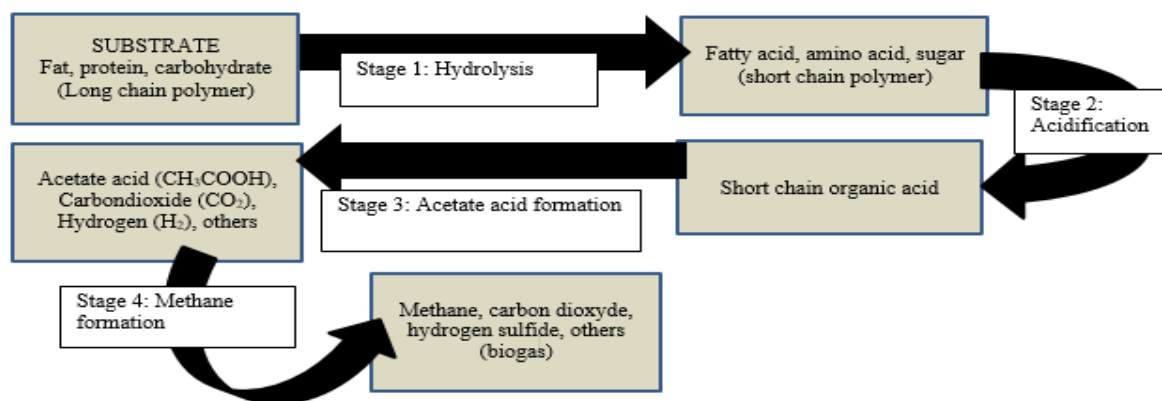

Fig. 1 The Process Stages of POME Degradation[17]

2. Material and Methods

In this study, the experiment was conducted at 1 m³ system bio CSTR located at Building 225, PUSPIPTEK, Serpong, South Tangerang. This work was supported by institution partner, PTPN VIII - a state-owned palm oil company- that has several palm oil industries in different locations like Kertajaya Ltd, Malimping and Cikasungka Ltd in Cikasungka.

2.1 Material

POME is the substrate, C source, which is used to produce biogas in this study was provided PTPN VIII,

taken from Cikasungka Ltd.- Cikasungka, which was the nearest location. In order to keep POME stay fresh, POME was taken in every month in amount for 1 m^3 volume. Before being put into bio-CSTR, POME must be filtered first to remove coarse impurities such as fiber and shell. Filtering must be done to avoid clogging of the dia-pump.

2.2 Methods

The design process of bio-CSTR consists of several steps. Determine the type of bio-CSTR tank, specify the design configuration. In this design, a cylindrical tank with an ellipsoidal top and a flat bottom lid is specified. Calculate the parameters to be sought in the equipment design calculation process corresponding to the need and desired goal using ideal data. Determine the reference, data input, and the calculation design process assumption. Design the actual parameter that affects the calculation of equipment dimension. Design testing and analyzing. In the final step, we conducted design testing. Pome as a raw material was pumped into bio-CSTR. The raw material is fed into the tank every day until overflow then we analyze it. The liquids (bottom and overflow area) were analyzed including COD, BOD, and pH values.

2.3 Chemical Oxygen Demand (COD)

Measurement is conducted using COD meter (Photometer system MD 100, Tintometer-Lovibond) and Thermoreaktor RD 125 Heater from Lovibond. Reactant used is COD kit vials COD / CSB 0 – 15.000 ppm that contain K2Cr2O7, HgSO4 dan H2SO4 61%. The sample is shaken in its bottles until homogeneous. Subsequently, dilute it with aquadest using measuring glass and an Erlenmeyer flask. The dilution is adjusted to the range of sample COD value so that the final dilution result is between 0 – 15,000 ppm. Therefore, it is necessary to know the range of COD values in the sample to determine the COD reagent used. If the COD value is not known, conduct the test using a reagent with the lowest concentration. Add 0.20 ml of the test sample into the COD kit vial using a micropipette and then homogenized. As a blank sample, add 0.20 ml of distilled water into the COD kit vial and homogenized it. Then place the vials into the heater that has been pre-heated at 150°C. Heat the vials in the heater for 120 minutes. After the heating process is complete, remove the vials and cool them down to room temperature. Measurement of COD value was carried out using a COD meter (Photometer system MD 100, Tintometer-Lovibond). The COD value of the test sample is the result of multiplying the COD value on the COD meter by the number of dilutions.

2.4 Biochemical Oxygen Demand (BOD)

The test aims to determine the amount of dissolved oxygen (in milligrams) required by aerobic microbes to digest carbon organic matter in 1 (one) liter of the test

sample for five days at a temperature of 20°C in a dark room. The BOD value is determined from the difference in dissolved oxygen concentration 0 (zero) days and 5 (five) days. As a standard control material in this BOD test, a glucose-glutamic acid solution was used. The materials needed are mineral-free water, concentrated H$_2$SO$_4$, NaOH, potassium iodide (KI), glacial CH$_3$COOH, starch, salicylic acid, Na$_2$SO$_3$, potassium dihydrogen phosphate (KH$_2$PO$_4$), ammonium chloride (NH$_4$Cl), MgSO$_4$.7H$_2$O, anhydrous CaCl$_2$, FeCl$_3$.6H$_2$O, microbial seed, glucose p.a., glutamic acid p.a., and nitrification inhibitor Allyl thiourea (ATU). The equipment used are the DO bottles, incubator, glassware, pH meters, calibrated DO meters, ovens, and analytical balances. The test sample is conditioned at a temperature of 20 ± 3°C. Then adjust the pH of the test sample to the range of 6.0 - 8.0 by adding a solution of H$_2$SO$_4$ or NaOH. The dilution level at pH adjustment should be less than 0.5%. Furthermore, remove the interfering substances from the test sample, including residual chlorine compounds, hydrogen peroxide, and supersaturated dissolved oxygen. The diluent solution is prepared by adding nutrient solutions consisting of a phosphate buffer solution, MgSO$_4$, CaCl$_2$, and FeCl$_3$ to oxygen-saturated mineral-free water. After that, add the microbial seeds and stir until homogeneous. Prepare the diluent solution immediately before use. The test sample that has been free from interfering substances is then conditioned to a temperature of 20 ± 3°C. Take a certain amount of the test sample and dilute with the diluent solution to a volume of 1 liter. Then put the test sample into 2 (two) DO bottles that have been prepared and marked with the notation A1 and A$_2$. Fill in the test sample until overflow, then close the bottle and shake it several times. Pour mineral-free water around the mouth of the enclosed DO bottle. Put the A$_2$ bottle in the incubator and keep it at 20 ± one °C for five days. Meanwhile, measure the dissolved oxygen levels in the A$_1$ bottle using a calibrated DO meter and record the result as zero-day dissolved oxygen (A$_1$). After five days, measure the dissolved oxygen level in the A$_2$ bottle and record the results as five-days dissolved oxygen (A$_2$). For blank measurement, fill the diluent solution without the test sample into 2 (two) DO bottles that have been prepared and marked with the notation B$_1$ and B$_2$. Subsequently, do the same way with the measurement of the test sample above. BOD$_5$ value of the sample is then calculated using the following equation:

$$BOD_5 = \frac{(A_1 - A_2) - \left(\frac{(B_1 - B_2)}{V_B}\right) V_C}{P} \qquad (1)$$

With BOD$_5$: BOD$_5$ value of the test sample (mg/L), A$_1$: The dissolved oxygen in the test sample at 0 days (mg/L), A$_2$: The dissolved oxygen in the test sample at 5 days (mg/L), B$_1$: The dissolved oxygen in the blank sample at 0 days (mg/L), B$_2$: The dissolved oxygen in the blank sample at 5 days (mg/L), V$_B$: The volume of microbe

suspension (mL) in blank DO bottle, the value is 0 if the test sample is not added with microbial seed, V_c: The volume of microbe suspension (mL) in the test sample bottle (mL), P: Volume ratio of the test sample (V_1) to the total volume (V_2)

3. Result and Discuccions

Reactor design approaches often do not allow designing entirely innovative reactors and considering the impact of uncertainties during the design procedure. Therefore a development design framework is proposed in this research. A cylindrical tank with an ellipsoidal top and a flat bottom lid is specified in the design process for this type of bio-CSTR tank. POME feed will be fed to the tank's bottom in the bio-CSTR tank. According to Fig.2, the fermented gas product will flow into the gas holder from the top of the bio-CSTR tank.
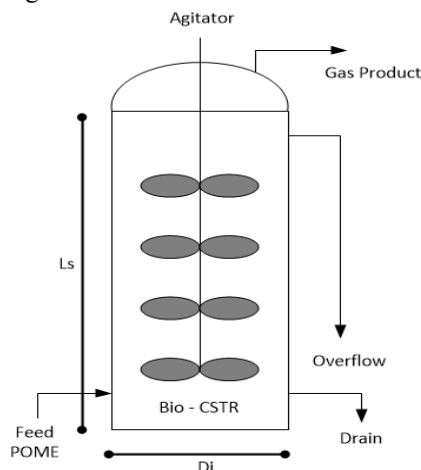

Fig. 2 Bio-CSTR System for Biohydrogen from POME

3.1 Bio-CSTR Dimensional Design

Bio-CSTR Dimensional Design
Working Vol.   = 1 m³
Safety Factor   = 10%
Total Volume   = 1.1 m3
Tank Volume[18]   = (π.Di2.Ls)/4
Ls   = 3 Di
Di   = 0.8 m
Ls   = 3 Di = 2.3 m
H Overflow   = 2 m
Lid Spesifications:
1. Ellipsoidal
2. Height of Lid = 0.19 m
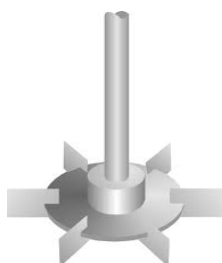
3.2 Bio-CSTR Impellers Design

Fig.3. Six Blade Turbine Impeller Design

The turbine impeller in the bio-CSTR design was chosen according to Fig. 3. Mixing system in a reactor design, including bioreactor, is one of the important factors that determine the performance of process.

Likewise, the improvement of the existing bio H_2 production system here, which was originally carried out by relying on a circulation pump, was modified by using a stirrer.

The reason for using the turbine impeller is because this type of impeller has many blades and smaller sizes, so it can be used at high speeds for fluids with a wide viscosity range. A turbine's diameter is normally 30 to 50 percent of the tank's diameter. Turbines impeller normally has four or six blades. A turbine impeller with six blades is used in this Bio-CSTR design. Radial flow is produced by a turbine's impeller with flat blades. This type is also suitable for gas dispersion because the gas produced by the microorganism process at the bottom will flow from the bottom to the top of the bio-CSTR, where it will be fragmented into gas bubbles. Flow patterns will vary depending on the type of impeller used[19]. This turbine impeller design is suitable for fluids with a viscosity of less than 100 cp. The six blades turbine impeller is an appropriate design for bio-CSTR because of the low viscosity of POME. The fluid flow pattern produced in the bio-CSTR using a six-blade turbine impeller is shown in Fig. 4.
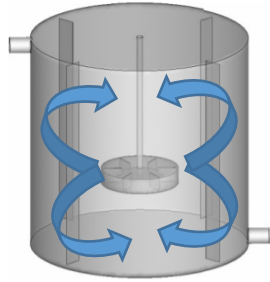
Fig. 4 Six Blade Turbine Impeller Flow Pattern[20]

The agitation system in bio-CSTR is designed to use multiple impellers as shown in Fig. 5. The purpose of the design is to maintain the effectiveness of the agitation process under changing conditions where the fluid height is greater than the tank diameter, hence four impellers design with a 450 mm space between impellers is utilized. According to Pauline M. Doran, in a low viscosity, if this model of the impeller is fitted with more than one design in a bio-CSTR with sufficient distance, each impeller has a radial discharge flow and produces large-scale
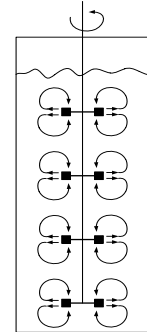
circulation loops that are independent. With a four impellers design, it is as if there are four separate stirred tanks arranged into one from top to bottom[21]. This design proved suitable for bio-CSTR biohydrogen with a large enough L/D tank design.

Fig. 5 Four Impeller Design and its flow direction in Biohydrogen Bio-CSTR from POME[21]

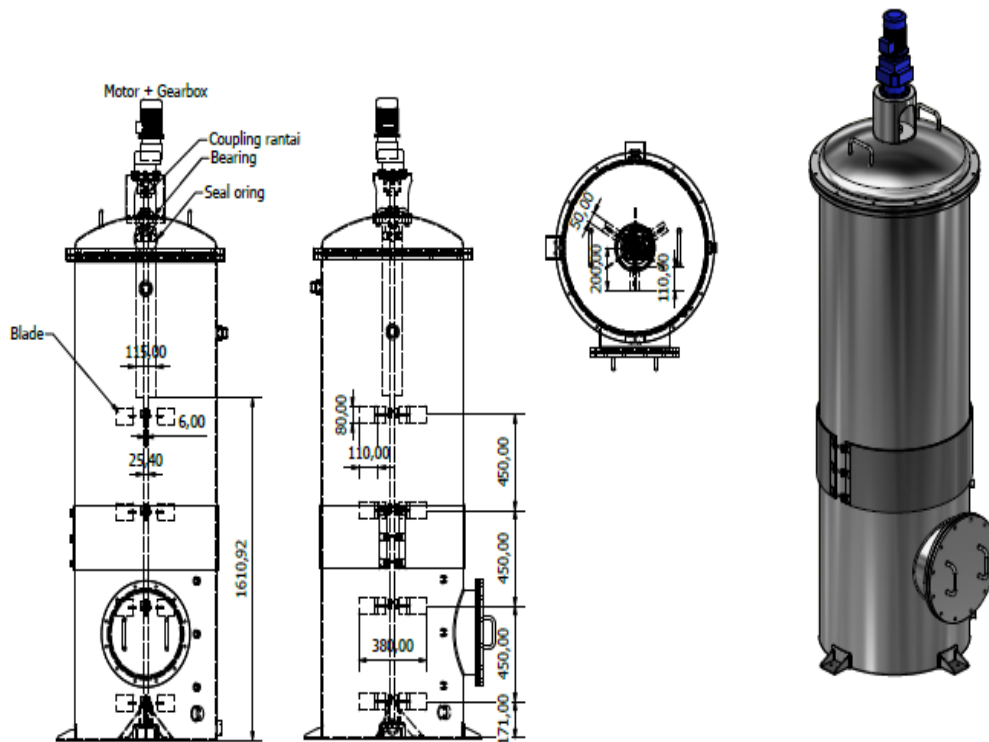## 3.3 Bio-CSTR Design Test for Biohydrogen Production from POME

Fig.6 Bio-CSTR Design for Biohydrogen from POME

Figure 6 showed the result of the bio-CSTR design. The test has been carried out according to the design. The initial stage is the preparation and conditioning of microorganisms which are then fed into the bio-CSTR tank. The next stage is the raw material (POME) obtained

from PTPN V is pumped into the bio-CSTR tank gradually until it overflows. This method uses a fed-batch system, in which feeds and nutrients are added regularly to the bio-CSTR tank.
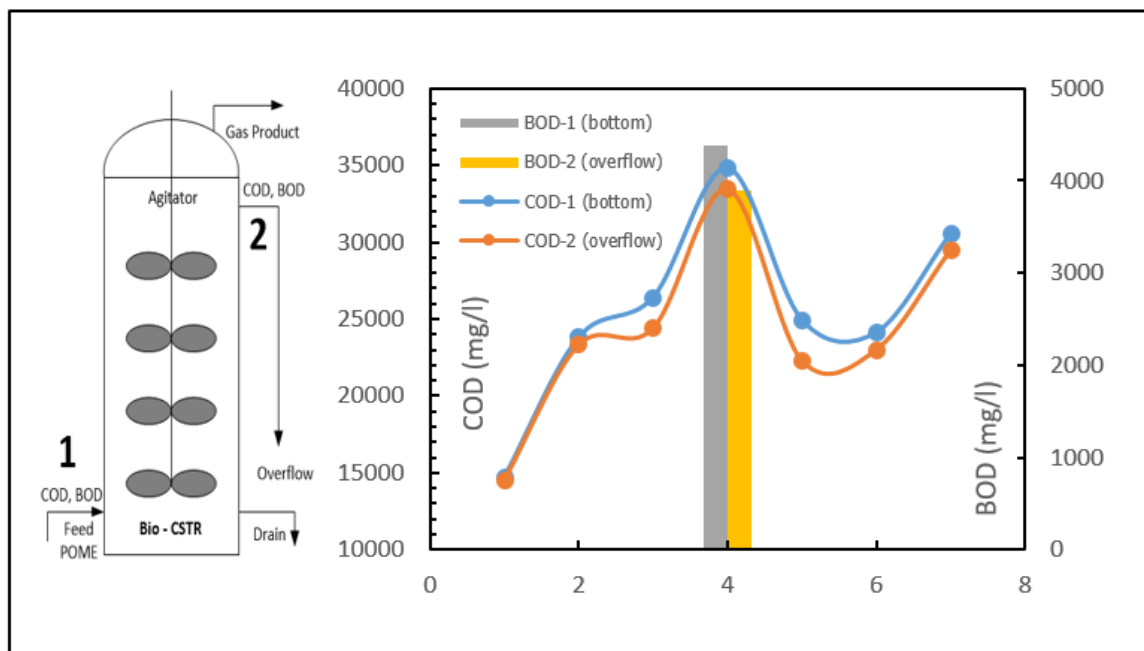
Fig.7 COD and BOD Analysis Results at Sampling Points 1 and 2 in Bio-CSTR (Bottom dan Overflow)

The effectiveness of converting POME to bio-H_2 was determined by measuring the reduction of COD (Chemical Oxygen Demand) and BOD (Biological Oxygen Demand). The COD and BOD were measured in the bottom and overflow. Effective management of POME can be also be utilized as a substrate to produce renewable energy, biogas, or bio H_2. This scheme can support environmental and energy policy implementers such as those implemented in Japan[22]. The effectiveness of the process in bio CSTR design was observed by decreasing COD and BOD concentration between in the bottom and overflow.

Based on Figure 7 the COD and BOD value at the bottom of the bio-CSTR are higher than at the overflow zone. It shows that there is a decomposition of POME raw material in the bio-CSTR tank. There is the degradation of organic components in POME raw materials by microorganisms therefore, the concentration is getting lower at the overflow zone[23]. This result also indicates that the homogeneity of microorganisms and POME is at the bottom, the gas production process is running well in the lower zone of the bio-CSTR. Microorganisms settle at the bottom of the bio-CSTR.

According to research by Jorge et al., this turbine impeller is suitable for the process of dispersing liquefied gases through mechanical agitation. This is not only because the impeller collects the gas underneath and forces it into the high shear zone near the blades where bubble formation occurs but also because it eliminates the flow instabilities shown by blade turbines[20]. The impeller design has a dominant radial flow pattern component where the impeller will cause the fluid to flow sideways and hit the wall then some turn to the middle and some turn down then back to the middle and so on and also have

a little tangential flow resulting from rotation impeller shaft. This turbine impeller is designed for high-speed operation and has a radial flow pattern, ensuring that microorganisms and nutrients are homogeneous only at the bottom and middle of the bio-CSTR, with no homogeneity at the top[24]

The correlation between BOD and COD for the production of both biogas and bio-H_2, in this case, is to examine how much biogas or bio-H_2 is produced to reduce the unit COD or BOD. However, this design is more focused on the uniformity of COD and BOD at each height, so it is expected that the BOD or COD in the overflow zone has a minimal value. The difference in COD and BOD concentrations in the bio-CSTR shows a positive effect on the 1 m3 (1000 L) bio CSTR design which indicates that the system is working laminar and non-stagnant. The trial of biogas production of H2 has also shown an increase in the concentration of bio-H2 to above 20%, increasing the concentration of H2 when producing biogas.

Besides COD and BOD, pH values were also evaluated. According to the data in Fig. 8. There was no significant difference between the pH values at points 1 and 2. This proves that the bio-CSTR design has the same operation condition at every point in the tank. The use of several impellers in a large L/D ratio tank design is very effective, as shown by the design of four stirrers in the bio-CSTR for biohydrogen from POME. This ensures that the process and conditions in the bio-CSTR tank are consistent from point to point, with no significant differences in pH between the overflow and the bottom area. As shown in Fig. 8, shows that the value was continuously increasing from day today. This is due to the addition of soda ash to maintain the pH conditions in the

bio-CSTR to keep microorganisms in optimal conditions. Each stage of POME degradation (hydrolysis, acidification, acetic acid formation and methane formation) produces fatty acids and other organic materials that are acidic. As a result, it is crucial to

maintain the pH conditions in the bio-CSTR throughout the process to remain in optimal conditions for microorganisms. By pH lower than 5 the metabolism of the methanogenic community could be easily hampered[25].
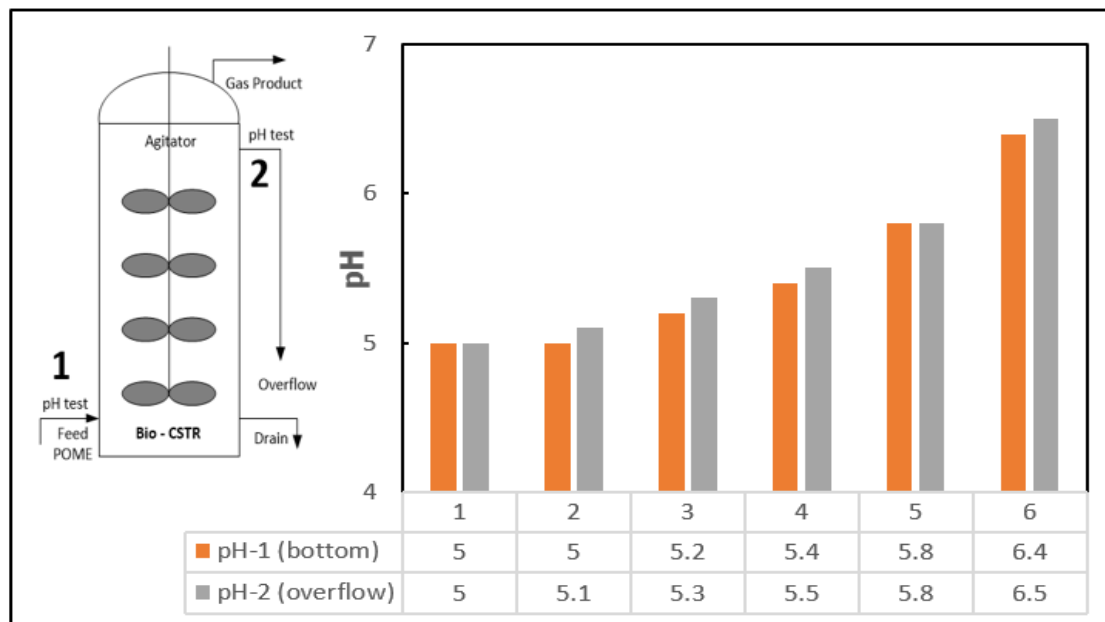

Fig.8 pH Analysis Results at Sampling Points 1 and 2 in Bio-CSTR (Bottom dan Overflow)

3.4 NRE calculation

$$N_{RE} = \frac{n\ D^2\ s\ g}{V}$$

Where n = impeller rotational speed, D = impeller diameter, s.g = material specific gravity, V = viscosity, Calculation N= 30 rpm, D= 0.7 m, s.g = 876, V = 2.5 cp. N_{RE} = 85.52 (laminar) [26].

The multiple impeller design in the bio-CSTR keeps the flow laminar and non stagnant. The NRE calculation result is 85.52, which means process agitation in the tank is still in the laminar zone. The design of multiple six blade turbine impellers resulted in appropriate process for the production of biohydrogen from POME.

4. Conclusion

The bio-CSTR design, which has a capacity of 1 m^3, dimension ratio Ls=3D, and is equipped with multiple six blade turbine impellers, was developed. The test has been carried out according to the design. The result showed that it could produce biogas with a biohydrogen content. The value of chemical oxygen demand (COD) and BOD (Biological Oxygen Demand) value at the bottom of the bio-CSTR is higher than at the overflow zone. It shows that there is a decomposition of POME raw material in the bio-CSTR tank. There is the degradation of organic

components in POME raw materials by microorganisms therefore, the concentration is getting lower at the overflow zone. The difference in COD and BOD concentrations in the bio-CSTR shows a positive effect on the 1 m^3 (1000 L) bio-CSTR design indicating that the system is working laminar and non stagnant to produce bio-H$_2$. Besides COD and BOD, pH values were also evaluated. The result showed that there was no significant difference between the pH values at the bottom and overflow point. This proves that the bio-CSTR design has the same operation condition at every point in the tank. Besides, the pH value was continuously increasing from day to day. This is due to the addition of soda ash to maintain the pH conditions in the bio-CSTR in order to keep microorganisms in optimal conditions. From NRE calculation result shows that the multiple impeller design in the bio-CSTR keeps the flow laminar and non stagnant. The design of multiple six blade turbine impellers resulted from the appropriate process for the production of biohydrogen from POME. However, these results still need to be reviewed to increase the concentration of bio-H$_2$ by minimizing the concentration of CH$_4$

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