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# Analysis of the Use of Modified Coal Bottom Ash as a Media for Immobilization of Microorganisms for the Production of Biogas from Macroalgae

(Penggunaan Bottom Ash Batu Bara Termodifikasi Sebagai Media Immobilisasi Mikroorganisme Untuk Produksi Biogas dari Makroalga)

Elli Prastyo \*<sup>1</sup>, Puji Astuti Ibrahim <sup>2</sup>, Dian Farkhatus Solikha <sup>3</sup>

<sup>1, 2, 3</sup> Teknik Kimia, Institut Teknologi Petroleum Balongan - Jl. Soekarno-Hatta, Pekandangan, Indramayu

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# ABSTRACT

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\*e-mail corresponding author : elli.prastyo@gmail.com

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This is an open access article under the **Creative Commons Attribution-ShareAlike 4.0 International License.** Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. CC–BY-SA Biofuel from macroalgae is referred to as the third generation biofuel because it does not require fertile land in the production process. Therefore, it is not included in the debate on the food sector. One of the process engineering in biogas production is the addition of immobilization media in anaerobic reactors. Coal combustion produces bottom ash solid waste containing 39.70% carbon (C) and 46.99% silica dioxide (SiO2) as well as other trace metals that have the potential to be used as absorbent materials or immobilization media in anaerobic decomposition. The purpose of this study was to determine the effect of the use of coal bottom ashbased immobilization media in the production of macroalgae biogas Spadina sp. The fixed variable in this study was the use of immobilization media of 30 grams, 40 grams, and 50 grams. Test parameters in the form of sCOD, VFA, biogas volume, and methane levels were used in this study. The production of macroalgae biogas Spadina sp is carried out in a fixed bed reactor with a capacity of 2000 mL with semi-batch conditions. The results showed that the highest biogas production was 204.8 mL/day with the use of immobilization media of 40 grams. The lowest biogas production is 20.5 mL/day with the use of 50 grams of immobilization media. The use of 50 grams of immobilization media is the best media to produce biogas with a high level of purity with an average of 57.48% followed by the use of 40 grams of immobilization media with an average of 41.35%, and the use of 30 grams of immobilization media with an average of 21.96%.

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## 1. INTRODUCTION

Climate change and current energy-related environmental problems encourage using coal energy to be reduced and replaced gradually with new renewable energy. One of the negative impacts of the use of energy sourced from coal is bottom ash because production continues to increase and utilization has not been maximized. Coal combustion produces bottom ash solid waste containing 39.70% carbon (C) and 46.99% silica dioxide (SiO2) and other trace metals that have the potential to be used as absorbent materials or microorganism immobilization media in anaerobic decomposition [1].

Anaerobic digestion offers great potential to reduce the negative effects of coal or fossil energy use. As a consequence of the use of new renewable energy, biomass resources and supply have become major problems leading to competition for land between the food and energy sectors. Therefore, biomass produced from marine commodities can be a profitable alternative. Macroalgae have been considered potential aquatic energy crops due to their high growth rates [2].

Indonesia is an archipelagic country with the second longest coastline in the world. This is certainly a great potential in the production of marine commodities, one of which is macroalgae. Macroalgae production in Indonesia has now increased significantly. Macroalgae production in 2021 reached 9.12 million tons, an increase of more than three times [3]. Until now, its utilization is still very lacking, especially in the processing of new renewable energy. Macroalgae have a high carbohydrate and water content and are low in lignin compared to terrestrial plants so they are more easily degraded. Furthermore, macroalgae have been widely used as raw materials for the food and pharmaceutical industries. However, only a small percentage of the total population and types of macroalgae have been utilized by the industrial world. Therefore, making macroalgae as raw material in making biogas is one solution to increase the utilization of macroalgae in Indonesia [4]. Through proper research and processing, macroalgae have great potential as raw materials for biogas production.

Most studies have been conducted using the macroalgae type Ulva lactuca which produces methane values ranging from 120 to 270 L.kg.VS<sup>-1</sup> [5]. Methane gas production potential has also been measured for various brown macroalgae, such as Sargassum muticum (120 - 380 L kg.VS<sup>-1</sup>) [6], *Laminaria saccharina* (220 - 300 L kg.VS<sup>-1</sup>) [7], And several studies discuss methane production from Gracilaria verrucosa macroalgae (130 – 400 L.kg.VS<sup>-1</sup>) [8].

Biofuel from macroalgae is referred to as third-generation biofuel because it does not require fertile land in the production process. Therefore, it is not included in the debate on the food sector. In addition, the gross energy yield produced by macroalgae per hectare per year is shown to be high in the range of 199-365 GJ ha<sup>-1</sup>.yr<sup>-1</sup> [9] depending on the species, location, and variety of macroalgae [10]. Furthermore, macroalgae contain carbohydrates (19.06%), protein (5.53%), water (11.71%), ash (34.57%), and crude fiber (28.39%) [11]. Macroalga also contains other polysaccharides, namely cellulose which ranges from 23.97% – 35.22% [12]. Macroalgae also contain a C: N ratio of up to 30: 1 which is included in the good category for biogas feedstock [13].

A problem that often occurs in biogas production using anaerobic decomposition is the low growth rate of microorganisms, so biogas production using conventional anaerobic reactors with large volumes results in less economical processing. In addition, for waste flow at high speeds, the reactor is unable to prevent the occurrence of the washout phenomenon, where bacterial populations are carried out with the reactor outlet.

The solution to the constraints of anaerobic decomposition, especially for complex and non-ideal raw materials, is to use an immobilized anaerobic system, one of which is a fixed bed reactor [14]. Fixed bed reactors can accommodate high waste flow rates to reduce the need for reactor volume and simple processing [15]. The addition of immobilization media that functions as a place to grow microorganisms in forming biofilms can avoid wash-out, and increase the cell density of microorganisms and cell concentrations which directly have a good effect on the activity of microorganisms [16].

The scientific literature rarely discusses the production of biogas from macroalgae, especially regarding anaerobic processing using immobilization media. The majority of scientific literature discusses the potential of biomethane at the laboratory batch scale without any process modification for optimization in biogas production [17]. Macroalgae processing using slurry has been reported in several studies [18] [19]. However, this study does not establish the optimization of key parameters in the process of processing biogas from macroalgae. Biogas production from macroalgae is also carried out by [20] Using an anaerobic batch reactor without immobilization media which only discusses the production of biogas produced without considering other influential variables.

The current review aims collectively to present biomethane production scenarios from macroalgae using coal bottom ash immobilized anaerobic reactors. Several strategies for the amount of immobilization media applied to increase biogas production from macroalgae biomass are also considered. The use of fixed bed-type anaerobic reactors that operate semi-batch using coal bottom ash is a novelty in the research conducted.

## 2. RESEARCH METHODS

This research was carried out in the microbiology laboratory of the Chemical Engineering Study Program, Balongan Institute of Petroleum Technology from August 2022 to March 2023. Field observations were carried out to determine the location of the research material collection (macroalgae) and bottom ash and to determine the condition of the field. Macroalgae were obtained from Cemara Village, Cantigi District, Indramayu Regency. The type of brown macroalgae (Spadina sp) was selected based on availability in the field during field observations.

#### **Preparation of Substrate and Inoculum**

Padina sp macroalgae obtained from Cemara Village, Cantigi District, Indramayu Regency are then cleaned to remove dirt that sticks to the surface using running water. Padina sp macroalgae that have been cleaned are then carried out the drying process using an oven. The macroalgae substrate is prepared from dried macroalgae Padina sp soaked for 48 hours using clean water to reduce salt levels. Padina sp macroalgae that have been soaked are then filtered from soaking water, then mashed using a blender with a ratio of adding water to macroalgae 2: 1 [13]. The macroalgae substrate that has been made is then

analyzed with the value of Chemical Oxygen Demand (COD) using APHA 2005, Total Suspended Solid (TSS), and pH standards.

In this study, the fixed bed reactor inoculum was obtained from an active cow dung digester located in Sudimampir lor Village, Kayen RT 17 / RW 04 block, Balongan District, Indramayu Regency. The active effluent digester is then pressed and filtered to obtain an effluent solution that is free of solids. The inoculum solution that has been taken is then analyzed by Chemical Oxygen Demand (COD), Total Suspended Solids (TSS), and pH. The ratio of inoculum use to macroalgae substrate was 1:2 (v/v) in this study.

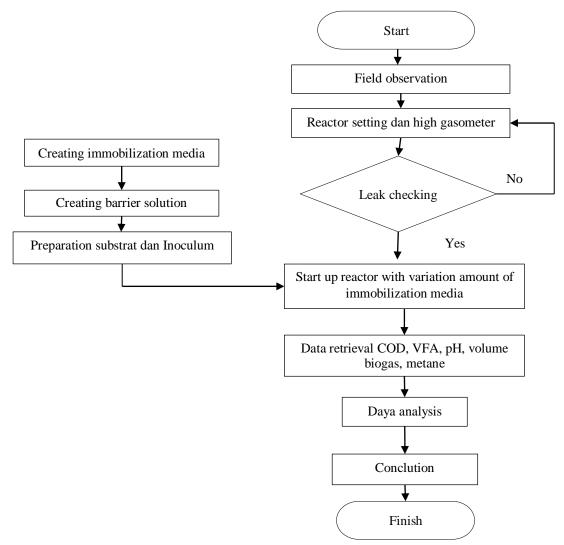


Figure 1. Research flow chart

#### **Creation of Immobilization Media**

Making bottom ash-based immobilization media obtained from the PLTU located in Sumuradem Village, Sukra District, Indramayu Regency. The bottom ash obtained is then filtered using a 100-mesh sieve to separate impurities. The filtered bottom ash is then mixed with bentonite with a ratio of 1: 1 (w / w). The mixture is then added water little by little until it forms a fairly fluffy dough. The ready dough is then molded using an extruder in the shape of a raschig ring. The molded material is dried at room temperature for 24-48

hours to dry, then oven at 1100C for 12 hours. The dry immobilization media is then calcined at 7000C for 1 hour using a furnace Thermolyne Tube Heater Type F21100. Variable amounts of immobilized media of 30 grams, 40 grams, and 50 grams were selected in this study. The bottom ash media immobilization technique is carried out by conditioning the reactor under batch conditions until biogas does not form at an early stage. This process aims to form an initial biofilm on the surface of the media and accelerate the adaptation process of microorganisms. The next step is to condition the reactor to operate in a semi-batch.

## **Research Analysis**

Analysis in the form of COD, VFA, pH, and methane levels in biogas was carried out in this study. Sampling is carried out every day for each variable determined. Gas samples are taken through a rubber septum in one of the holes in the upper liquid of the reactor using a high-pressure gas syringe. The samples were analyzed for methane levels using Gas Chromatography Shimadzu GC 8A Japan.

During the anaerobic decomposition process, the reactor operates in a semi-batch where there is no input but there is an output i.e. test sampling. The anaerobic decomposition process is stopped when there is no gas production and there is no decrease in COD. High gasometers are used to measure biogas production during the anaerobic decomposition process. The following equation is used to determine the volume of biogas measured using a high gasometer. A high Gasometer is a closed cylinder or column that is partially submerged in an open container containing 75% salt water saturated with pH = 2 as a barrier solution [21].

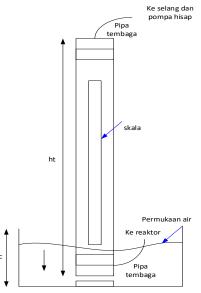


Figure 2. Scheme High Gasometer

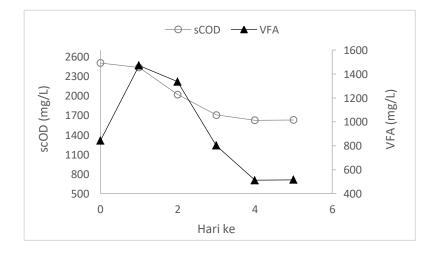
Measurement of the volume of biogas produced using the following equation: [21]

#### 3. RESULT AND DISCUSSION

Immobilization media plays an important role in the growth of microorganisms in anaerobic decomposition by forming a biofilm layer on the surface of the media. Biofilm formation allows anaerobic reactors to achieve steady-state conditions faster and reduces the potential for wash-out phenomena due to high organic loads or inhibition contained in the substrate. This extracellular polymer matrix protects the biofilm against shear stress in the reactor. Bacteria in aqueous environments tend to aggregate to form biofilms rather than in the free state [22]. The aggregation is done so that individual bacteria can compete for nutrients, and space and increase cell density and resilience. Variations in the amount of immobilization media potential for biogas formation from macroalgae are studied through a decrease in sCOD, VFA formation, biogas production, and methane levels formed from each variable.

#### The effect of decreasing COD on VFA production on various variables

Macroalgae padina sp is a substrate in this study which is fed into a fixed reactor with semi-batch operating conditions. The performance of substrate decomposition by microorganisms can be seen from how much COD decreases. The decomposed COD then forms volatile fatty acid (VFA) compounds in the reactor. Volatile fatty acid (VFA) is an intermediate product in the anaerobic decomposition process which will be converted by methanogen bacteria into biogas. The following figure shows the profile of COD reduction followed by VFA formation in a fixed-bed reactor.



(a)

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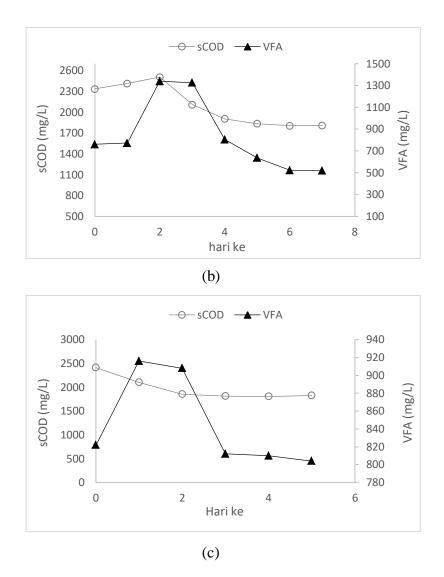


Figure 3. Experimental Data Decrease in sCOD Value to VFA Value (a) immobilization media 30 gr, (b) immobilization media 40 gr, (c) immobilization media 50 gr

Figure 3 shows the profile of the effluent COD value to the overall VFA effluent value correlated during the anaerobic decomposition process. The VFA effluent value increased at the start of start-up across all variables, By the stage of anaerobic decomposition where the initial stage is the hydrolysis process by acidogenesis bacteria which produce simple organic compounds consumed by acetogenesis bacteria and produce VFA. Therefore, acetogenesis bacteria will be able to grow and develop properly when the VFA value is available in the reactor. VFA values reached a stable state at 500 mg/L for all variables. This indicates that there is no longer a substrate converted into VFA in the reactor system, so the implication is that the VFA effluent value will tend to be stable following the COD effluent value.

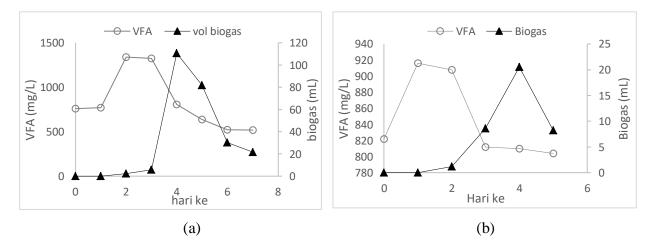
Figure 3 shows that the overall COD effluent value profile decreased for all variables. The use of immobilization media of 30 grams showed an increase in COD effluent at the beginning of the process until the third day then reached a steady state at a value of 1810 mg / L. The increase in COD value at the beginning of the process showed that acidogenic

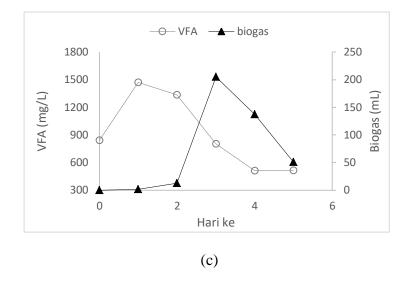
bacteria needed time for the adaptation process. This is by research conducted by [23] which shows that an increase in the value of effluent sCOD early in the process is natural for anaerobic decomposition because microorganisms require recovery time in new conditions until they reach a stable state again. Different things are shown by the use of immobilization media of 40 gr and 50 gr where a decrease in sCOD value occurs early in the process. A decrease in the value of sCOD effluent at the beginning of the process may indicate the use of porous immobilization media which has a greater impact on the rapid adaptation process of microorganisms to new conditions. Immobilization media containing trace elements such as Si, Al, Fe, and other compounds in their constituents can be a positive catalyst for microorganisms for the process of adaptation and degradation of organic compounds [24]. This is by the statement [25] Where the presence of trace metal compounds in the media can increase the degradation capacity of organic compounds by microorganisms.

The effluent COD value profile shows the increase at the end of the effluent COD decomposition process for all variables. The increase in COD effluent value indicates the death of microorganisms due to reduced amounts of organic compounds in the reactor. The death of these microorganisms is then read as sCOD effluent in the reactor. The microorganism batch reactor will enter the stationary phase where the growth phase begins to decline until it enters the death phase. In the stationary phase some microorganisms die and lysis events occur (cell rupture and cell fluid exit), dead microorganisms will affect the content of organic compounds [26]. The remaining microorganisms that die in the solution in this reactor are measured as COD so that the COD value of the solution rises.

#### The effect of VFA value on biogas production on various variables

One of the successes of the anaerobic decomposition process is shown by the production of VFA and the formation of biogas. VFA values that experience fluctuations are the impact of the metabolic processes of microorganisms in decomposing organic compounds. The VFA value profile of effluent and biogas production is shown in Figure 4.





# Figure 4. Results of VFA Value Reduction Experiment on biogas production (a) immobilization media 30 gr, (b) immobilization media 40 gr, (c) immobilization media 50 gr

Figure 4 shows the profile of VFA effluent value on brown algae biogas production padina sp for all correlated variables where the higher the VFA effluent value, the biogas production will decrease. The value of VFA increased at the beginning of the start-up which indicates that acetogenesis bacteria can grow and develop well in the reactor system, but methanogen bacteria have not been able to grow and develop properly. Methanogen bacteria take longer than other bacteria in the anaerobic decomposition process to work optimally, so after the 3rd day it is predicted that methanogens have grown and moved well [27]. Good activity against methanogenic bacteria is characterized by a decrease in the VFA value in the reactor and an increase in biogas production. Overall, VFA values showed a decrease for all variables where a decrease in VFA values was indicative of the metabolic process of methanogen bacteria in reducing VFA and producing biogas [23].

The production of macroalgae biogas Spadina sp showed the highest value occurred in the use of immobilization media of 40 grams with the highest peak occurring on the 3rd day of 204.8 mL. The presence of trace metals in the metabolism of microorganisms is believed to increase the rate of biogas production [28]. Furthermore, the lowest biogas production occurred using 50 grams of immobilization media. This indicates that the higher use of calcined bottom ash-based immobilization media makes biogas production decrease. The use of excess immobilization media can absorb complex organic compounds on the substrate which has an impact on decreasing VFA production and biogas production [29].

#### Comparative study of COD removal efficiency and methane concentration

A comparative study on the use of immobilization media aims to determine the effect of the resulting product, in this case, biogas methane levels. The energy contained in biogas depends on the concentration of methane gas (CH4), the higher the methane content, the greater the energy content in biogas The effect of the use of immobilization media on methane levels in the biogas produced is presented in Table 1.

		Kadar Metana	
Time	media imobilisasi	media imobilisasi 40	media imobilisasi
	30 gram	gram	50 gram
0	0	0	0
1	0	44.757	53.194
2	16.005	43.71	50.685
3	22.795	42.908	51.344
4	28.244	40.232	75.715
5	32.283	35.186	52.172
6	22.445		
7	24.026		

Table 1. Methane Rate Comparison

Table 1 shows that on days 0 and 1 of start-up there were no methane levels because biogas production had not yet emerged at the beginning of start-up. Biogas production from macroalgae padina sp appeared on day 2 which overall produced the highest methane levels on day 4 with the use of immobilization media of 50 grams. The immobilization media of 50 grams is the media producing the highest methane levels compared to other variables. This is by biogas production data where the highest biogas production is formed on day 4. High biogas production and methane levels due to the availability of substrates in the form of VFA in the reactor system. Furthermore, the growing use of immobilized media indicates an increase in methane levels. This indicates that the larger the immobilization medium can increase the purity of the biogas produced. Immobilized media with a calcined bottom ash base can be an adsorbent for certain compounds, in this case, CO<sub>2</sub>. This is due to the nature of adsorbents in absorbing carbon, whereas activated charcoal derived from bottom ash is hydrophobic and nonpolar which can bind carbon. The use of activated carbon as a biogas adsorbent can bind CO<sub>2</sub> compounds to biogas and increase methane concentrations significantly [30].

## 4. CONCLUSION

The use of Padina sp macroalgae has great potential as a substrate for the production of immobilized anaerobic biogas reactors based on coal bottom ash. The use of bottom ash-based immobilization media from coal affects the rate of biogas production and the level of purity of methane produced. The highest biogas production was 204.8 mL/day with the use of 40 grams of immobilization media. The lowest biogas production is 20.5 mL/day with the use of 50 grams of immobilization media. The use of 50 grams of immobilization media are biogas with a high level of purity with an average of 57.48% followed by the use of 40 grams of immobilization media with an average of 41.35%, and the use of 30 grams of immobilization media with an average of 21.96%.

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