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Utilization of Corn Husk Waste as Raw Material for Making Biobutanol

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ABSTRACT

Corn husk waste is a biomass raw material that can be processed into biobutanol. Making biobutanol to reduce the use of fossil fuels. This research used anaerobic microorganisms, namely *Clostridium acetobutylicum,* which can survive at low pH between 4,5 and 5 at a temperature of 30°C. Corn husk contains 36.218% cellulose; 25.212% hemicellulose; 15.807% lignin. The cellulose raw material content can be used for fermentation because it meets the fermentation content requirements of 36 – 50%. The research methods used include raw material preparation, delignification, dilute acid hydrolysis, and anaerobic fermentation. The research results showed that optimum condition was obtained at the 72-hour fermentation time with the addition of a 5% *Clostridium acetobutylicum* bacteria volume of 7.0160%. At the 72 hour fermentation time, it showed that the bacteria grew optimally and entered the stationary phase. Biobutanol levels after 72-hour decreased due to reduced nutrients in the fermentation media. The increase in the volume of bacteria affects the final results, the volume of bacteria is added, the final results biobutanol levels will decrease because the nutrients added have been used up. The results of the analysis of butanol levels were determined using Gas Chromatography, the standard for butanol formation obtained at a retention time of 3.598%. The low butanol content (7,0160%) does not require industry standards (96.5 – 99%) to be used as environmentally friendly fuel, due to obstacles in maintaining anaerobic fermentation conditions and not carrying out fermentation filtrate sterilization, which has an impact on the final result of biobutanol content.ly and requires improvement, particularly in the quest for zero defects.

Keywords: Corn husk, anaerobic fermentation, Clostridium acetobutylicum, biobutanol

Introduction

The current energy situation in Indonesia still involves imports, especially crude oil and fuel products, and total final energy use (without traditional biomass) in 2018. Where, the highest sector is dominated by transportation with 40% still using fossil fuels (DEN, 2019). These fossil fuels contain carbon and when burned they will form CO2 and unlike biomass, CO2 emissions are considered zero because it is assumed that they will be reabsorbed by plants (BPPT, 2019). The release of CO2 into the atmosphere from industrial, transportation, and other sectors can cause air pollution and have an impact on global warming. Therefore, in efforts to reduce dependence on fossil fuels, it is necessary to develop alternative fuel resources that are environmentally friendly biobutanol can be a solution to this problem because butanol is an environmentally friendly fuel and contains 25% more energy than ethanol which can be used as a fuel excellent sear (Cheng, 2018).

Several previous studies have been carried out regarding biobutanol, including the following: research conducted by (Tsai et al., 2020) with the title "Biobutanol from Lignocellulosic Biomass Using Immobilized *Clostridium acetobutylicum*". The raw materials used are rice straw, sugarcane bagasse, and microalgae using *Clostridium acetobutylicum* bacteria. The highest biobutanol yield was from rice

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straw. A yield of 13.8% was obtained. These results were obtained at a fermentation temperature of 37°C for 30 – 40 hours.

Research conducted by Valles et al. (2021) with the title "Optimization of Alkali Pretreatment to Enhance Rice Straw Conversion to Butanol. The raw material used is rice straw using *Clostridium beijerinckii*. The highest biobutanol yield was 10.01%. These results were obtained by adding a 5% volume of *Clostridium beijerinckii* at a fermentation temperature of 37°C for 72 hours.

Research conducted by Wechgama et al. (2017) with the title "Enhancement of Batch Butanol Production rom Sugarcane Molasses Using Nitrogen Supplementation Integrated With Gas Stripping For Product Recovery". The raw material used is sugar cane molasses using *Clostridium beijerinckii*. The optimum biobutanol yield was 13.9%. This result was obtained by increasing the volume of *Clostridium beijerinckii* by 5%, and urea nutrition by 0.81 grams, at a fermentation temperature of 37°C for 48 hours.

Based on several previous studies that have been carried out, biobutanol using biomass raw materials still has research opportunities. However, in previous research, there were still obstacles related to the use of fermentation time variables and the volume of bacteria added. In previous research, the fermentation time of 72 hours was not the best condition because the yield obtained was high when the fermentation time ended. The bacteria volume is only determined using a volume of 5% so it is not known that the volume used is in the best condition. Therefore, researchers propose to look for the best conditions for biobutanol levels from corn husks during fermentation and the volume of bacteria (*Clostridium acetobutylicum*) added. This research aimed to find the best conditions for biobutanol levels from corn husks during fermentation and the volume of bacteria (*Clostridium acetobutylicum*) added.

Material and Methods

Preparation of raw materials

The raw material of corn husks was obtained from Rungkut Market, Surabaya, East Java. The corn husks are washed with water and then dried for 3 days in the sun. Cut into smaller pieces, then blend for 8 minutes. Corn husk powder is obtained in a smaller size

Delignification

Put 20 grams of corn husk powder into an Erlenmeyer flask and add 200 ml of 2% NaOH. The Erlenmeyer was covered with aluminum foil and placed in an autoclave at 121°C for 15 minutes. The samples were cooled at room temperature (30°C). The samples were washed using distilled water to pH 7 and filtered. The precipitate was dried in an oven at 120°C to constant weight.

Dilute acid hydrolysis

The delignification results were put into a beaker glass by adding 250 ml of 2% H2SO4. Hydrolysis using a magnetic stirrer at a temperature of 100°C for 120 minutes. The samples were cooled at room temperature (30°C). Sample filtration to separate the filtrate and sediment. Then, adjust the pH to 5 using 50 ml of 40% NaOH. After the pH became 5, glucose levels were analyzed at the Nutrition Laboratory, Faculty of Public Health, Airlangga University.

Fermentation

The filtrate from hydrolysis containing glucose is put into a fermenter bottle of 100 ml. Next, 0.81grams of urea was added as well as variations in the volume of *Clostridium acetobutylicum* bacteria (3%; 4%; 5%; 6%; 7%). Fermentation is maintained at a temperature of 30°C and lasts for 24; 48; 72; 96; and 120 hours. Fermentation filtrate is filtered to separate it from the residue. The contents of the

fermentation filtrate were analyzed using Gas Chromatography at the Chemistry Laboratory at Semarang State University.

Results and Discussion

Delignification

Delignification is carried out to reduce lignin levels in lignocellulosic materials. Color changes during delignification and after delignification indicate that lignin has been released. The initial weight of corn husk powder before delignification was 20 grams, after delignification it decreased by 3.7995% grams. This reduction in the sample with research (Ayuni & Hastini, 2020) that delignification using NaOH can remove half of the initial sample weight. This happens because lignin is also dissolved in NaOH due to lignin binding to NaOH during delignification.

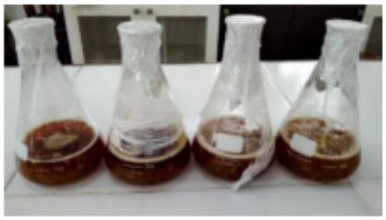

Figure 1. Color of corn husk powder during delignification

Figure 2. Color of corn husk powder after delignification

Dilute acid hydrolysis

The results of glucose analysis from the Nutrition Laboratory of Airlangga University were obtained at 2.17%. This affects biobutanol levels. If the glucose levels produced are low, the biobutanol levels produced will also be low (Wardefisni et al., 2020). The discrepancy between glucose levels and the standards used for fermentation (10 – 18%) (Herawati & Wibawa, 2019) because glucose is degraded into 5-hydroxymethylfurfural and furfural compounds so that the resulting glucose levels are still below existing standards.

Figure 3. Dilute acid hydrolysis process

Fermentation

Table 1. Biobutanol levels in fermented corn husks

Time (hours)	Bacteria volume (ml)				
	3%	4%	5%	6%	7%
24	0.8756	0.9814	1.0924	1.2762	1.3802
48	2.5581	3.3760	5.4764	3.9631	3.5346
72	5.8069	6.1677	7.0106	5.9709	5.4227
96	4.2142	5.8149	5.9274	5.3030	4.6011
120	3.2963	4.1909	4.8536	4.7506	3.5611

Based on the research that has been carried out, in table. 1, data on optimal biobutanol levels was obtained at 72 hours with the addition of 5% bacterial volume to obtain biobutanol levels of 7.0106%. The process of biobutanol formation begins to occur at 24 hours (lag phase) where bacteria use glucose to produce metabolic acids in the form of butyric acid, acetic acid, and ethanol. At 48 hours, bacteria grow (log phase) by converting butyric acid and acetic acid into butanol and acetone to reduce the toxicity of bacterial cells. Next, at 72 hours, the bacteria grow to their maximum state (stationary phase) (Khunchit et al., 2020). The tendency for the concentration of biobutanol to decrease from 96 to 120 hours is due to the reduced nutrients in the medium and the longer the fermentation process takes, causing a lot of the butanol that has been produced to be oxidized into organic acids and CO2 which are inhibitory compounds that cause death of microorganisms (Fitria et al., 2021).

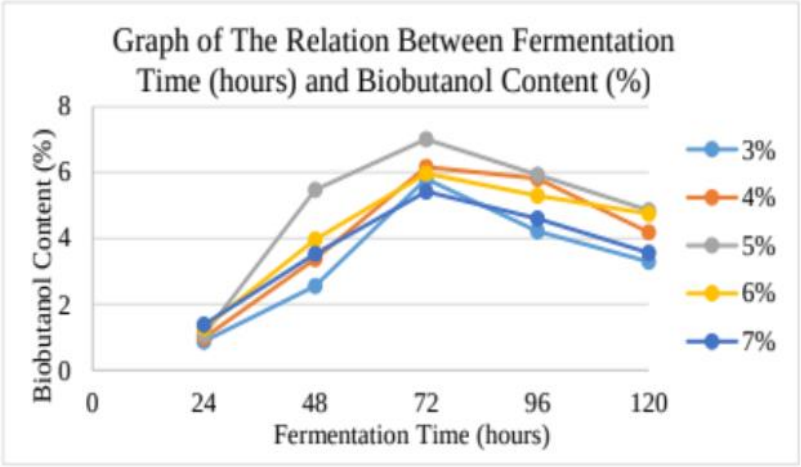

Figure 4. Relation between fermentation time (hours) and biobutanol content (%)

The things that influence this research are fermentation time and bacterial volume. Based on Figure 4, it can be seen that the longer the fermentation time, the more glucose levels are converted into biobutanol. However, in reality, the fermentation process has an optimum time, so that after the optimum time has passed the biobutanol levels will decrease. If the availability of food and nutrients is no longer sufficient for bacteria to live and reproduce, the biobutanol levels will decrease. Meanwhile, the volume of bacteria added affects the final results, where the more the added volume of bacteria, the more biobutanol levels will decrease because the nutrients added have been used up.

Retention time is a specific number of interaction times between compound molecules in the chromatography column (Ramayanti et al., 2021). Based on Figure 6, the retention time for butanol is

3.598 which shows that a compound will be completely separated from other compounds so that analysis using Gas Chromatography can separate compounds with high selectivity under optimum conditions. Figure 7 shows that the compounds: acetone (retention time 2.386), butanol (retention time 3.598), and furfural (3.162) were formed. These three compounds are formed after going through an acidogenic phase (*Clostridium acetobutylicum*) to produce a mixture of organic acids in the form of butyric acid, acetic acid, and lactic acid. Next, after the acid accumulates, the pH of the growth medium becomes more acidic, resulting in a shift towards the solventogenic phase when it switches to producing acetone and butanol. The formation of furfural as an inhibitor in the fermentation process causes delays in sugar absorption and decreases butanol production.

Figure 5. Anaerobic fermentation process

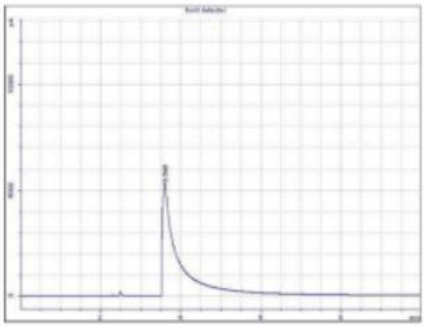

Figure 6. Gas chromatography analysis results (Standard butanol)

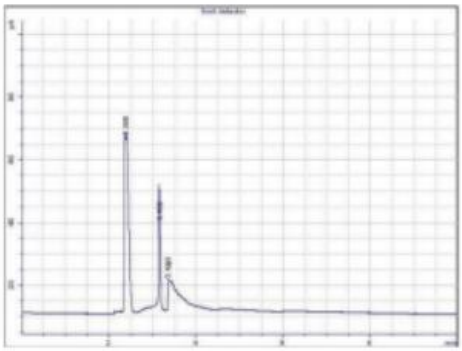

Figure 7. Gas chromatography analysis results (Compounds butanol)

The results of the biobutanol content were low (7.0106%) and did not require industry standards for biobutanol to be used as fuel (96.5% - 99%) from this research due to obstacles in maintaining anaerobic fermentation conditions. This condition is difficult to do because of the limitations of the tools used. This is because the nature of *Clostridium acetobutylicum* is an obligate anaerob. When

these bacteria are exposed to very low levels of air, the bacteria will die. Based on this situation, the fermentation process must be carried out in a vacuum, and in this condition is difficult to obtain the tools needed to maintain the anaerobic fermentation process so that it is not contaminated by outside air and sterilization of the fermentation filtrate is not carried out so that the filtrate is dirty and not yet sterile which results in the results of biobutanol levels the low one.

Conclusion

The best conditions for biobutanol levels from corn husks were found at the 72-hour fermentation time with an additional 5% volume of *Clostridium acetobutylicum* bacteria producing a biobutanol level of 7,0106%. Based on the graph, at the 72-hour fermentation, it is in the stationary phase.

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