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Delignification of Cinnamon Bark (*Cinnamomum verum*) with Pre-treatment by NaOH to Increase Cellulose and Hemicellulose Recovery

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Abstract: *Bioethanol needs can be met from cellulose and hemicellulose biomass, for example cinnamon bark (Cinnamomum verum). Cellulose and hemicellulose are generally surrounded by a lignin structure. Therefore, pre-treatment is needed to degrade the lignin structure (delignification), so that the saccharification process of cellulose and hemicellulose can run optimally. The pre-treatment was carried out on the cinnamon bark with NaOH (Sodium Hydroxide). Cinnamon bark lignocellulose fractionation was carried out using the Chesson-Datta method. The results of the lignocellulose fraction obtained from samples without pre-treatment were 35,67% HWS (Hot Water Soluble), 24,33% hemicellulose, 14% cellulose, 25.96% lignin, and 0.04% ash. While the fraction results obtained from the sample with the addition of NaOH were 34,33% HWS, 35,67% hemicellulose, 18,33% cellulose, 11,52% lignin, and 0.15% ash. From these results it can be seen that the presence of pre-treatment in the addition of NaOH can increase the hemicellulose and cellulose fractions and reduce the lignin fraction.*
Keywords: *Cinnamon Bark; Lignin; Cellulose; Hemicellulose; NaOH*

Abstrak: *Kebutuhan bioetanol dapat dipenuhi dari biomassa selulosa dan hemiselulosa contohnya adalah kulit kayu manis (Cinnamomum verum). Selulosa dan hemiselulosa umumnya dilingkupi oleh struktur lignin. Oleh karena itu, dibutuhkan pra-perlakuan untuk mendegradasi struktur lignin (delignifikasi), sehingga proses sakarifikasi selulosa dan hemiselulosa dapat berjalan dengan optimum. Pra-perlakuan dilakukan dengan pada kulit kayu manis menggunakan basa NaOH (Natrium Hidroksida). Fraksinasi lignoselulosa kulit kayu manis dilakukan dengan metode Chesson-Datta. Hasil fraksi lignoselulosa yang didapatkan dari sampel tanpa pra-perlakuan penambahan NaOH adalah 35,67% HWS (Hot Water Soluble), 24,33% hemiselulosa, 14% selulosa, 25,96% lignin, dan 0,04% abu, sedangkan hasil fraksi yang didapatkan dari sampel dengan pra-perlakuan penambahan NaOH adalah 34,33% HWS, 35,67% hemiselulosa, 18,33% selulosa, 11,52% lignin, dan 0,15% abu. Dari hasil tersebut dapat terlihat bahwa dengan adanya pra-perlakuan berupa penambahan NaOH dapat meningkatkan fraksi hemiselulosa dan selulosa serta menurunkan fraksi dari lignin.*
Keywords: *Kulit Kayu Manis; Lignin; Selulosa; Hemiselulosa; NaOH*

INTRODUCTION

Energy needs are increasing with the increasing human population. Petroleum consumption in Indonesia reached 54% of the total energy used in 2009, while other forms of energy sources were below 30%. As a consequence, the price of fuel continues to rise, for this reason the government continues to seek other alternatives to save fuel (Prihandana *et al.*, 2007). Bioethanol can be used as an alternative energy substitute for gasoline that is environmentally friendly compared to fossil fuels. The advantages of bioethanol are cheap and environmentally friendly. Bioethanol is also a fuel that does not accumulate carbon dioxide gas (CO_2) and is relatively compatible with gasoline-fueled engines (Tan *et al*., 2008).

The second generation of bioethanol derived from lignocellulose includes agricultural and forestry waste products.

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Lignocellulosic biomass consists of cellulose, hemicellulose, and lignin with a complex three-dimensional structure (Faizal *et al.*, 2021). Its abundant availability makes this material a potential source of energy through conversion processes, both physical, chemical and biological processes. One of the most studied lignocellulosic conversion processes is the conversion process of lignocellulose into bioethanol which then be used to substitute gasoline for transportation purposes (Houfani *et al.*, 2020).

Optimization of bioethanol production through pre-treatment methods can increase the fraction of cellulose and hemicellulose so that the production of renewable energy can increase with the increasing number of substrates to be fermented. One of the pre-treatments is the addition of NaOH which aims to increase the surface area, reduce the degree of polymerization, reduce crystallinity, separate the structural bonds between lignin and carbohydrates, and damage the structure of lignin (Diaz *et al.*, 2015). The aim of this study was to increase the cellulose and hemicellulose fractions of cinnamon bark by pretreating with NaOH. With the increase in cellulose and hemicellulose fractions, the enzymatic hydrolysis process for bioethanol production will be easier.

RESEARCH METHOD

Preparation of cinnamon bark samples

Cinnamon bark was pounded with a mortar and pestle to make the structure softer. After that, the samples were ground in a blender until smooth. Samples were filtered with a 120 mesh sieve. Wet weight of the samples was weighed and then dried for 16 hours at 65°C in oven. The sample was then stored in a desiccator for one hour and the dry weight of the samples was weighed.

Cinnamon bark pre-treatment

For the pre-treatment variable, 10 grams of samples were taken and added with 100 mL of 1 M NaOH into 250 mL Erlenmeyer. The control variables were also prepared by adding 10 grams of sample to 250 mL of distilled water. The sample solution was then covered with aluminum foil and plastic wrap and put in an autoclave at temperature of 121°C and pressure of 1 atm for 30 minutes. In the control sample, the next step was measuring the pH and then filtering it using a Buchner funnel, while the pre-treatment sample was neutralized first using 1 M HCl to obtain a pH of 5-6 (according to the pH measurement of the control sample). The sample was dried in an oven for 24 hours at a temperature of 60-115°C. The dried sample was stored in a desiccator and then weighed as much as 1 gram of dry weight. This result is the value of a.

Cinnamon bark lignocellulose fractionation

A total of 1 gram of sample a was added with 150 ml of H_2O (distilled water) to the flask. The sample was then refluxed for 2 hours at 100°C. The residue was filtered off with 300 mL of hot distilled water using a Buchner funnel. The filtrate was dried to a constant weight using an oven for 24 hours at a temperature of 105°C. Dry filtrate was stored in a desiccator and then weighed in dry weight. This result was labeled as value b. Sample b added with 150 mL H_2SO_4 1 N into the flask then refluxed at 100°C for 2 hours. The residue was filtered off using a Buchner funnel while washing with distilled water. The filtrate was dried to a constant weight using an oven for 24 hours at a temperature of 105°C. Dry filtrate was stored in a desiccator and then weighed in dry weight. This result was labeled as value c. Sample c was added with 10 mL H_2SO_4 72% v/v at room temperature for 4 hours. The sample solution was diluted with distilled water to a concentration of 0.5 M into the flask. Then the sample was refluxed at 100°C for 2 hours. The residue was filtered off with 400 mL hot distilled water using a Buchner funnel. The filtrate was dried to a constant weight using an oven for 24 hours at a temperature of 105°C. Dry filtrate was stored in a desiccator and then weighed in dry weight. This result was labeled as value d. After that the sample d was ignited in a furnace at a temperature of 575 ± 25°C for 2 hours, this result was recorded as a value of e (Ma'ruf *et al.*, 2017).

$$HWS\ (\%) = \frac{a-b}{a} x\ 100\% \quad (1)$$

$$Hemicellulose\ (\%) = \frac{b-c}{a} x\ 100\% \quad (2)$$

$$Cellulose\ (\%) = \frac{c-d}{a} x\ 100\% \quad (3)$$

$$Lignin\ (\%) = \frac{d-e}{a} x\ 100\% \quad (4)$$

$$Ash\ (\%) = \frac{e}{a} x\ 100\% \quad (5)$$

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RESULT AND DISCUSSION

The process of cellulose hydrolysis in the lignocellulose complex structure will be much more difficult than the hydrolysis of free cellulose because lignocellulose is a very dense material so that under general conditions cannot be penetrated by water or enzymes. Therefore, pre-treatment was carried out with the addition of NaOH to hydrolyze lignocellulose. In the presence of NaOH, lignin and extractive substances will be dissolved. NaOH solvent is used because it can dissolve most of the lignin and can then be separated again easily so that it will increase the fraction of cellulose and hemicellulose. The fractionation results of each lignocellulose component of cinnamon bark are shown in Figure 1.

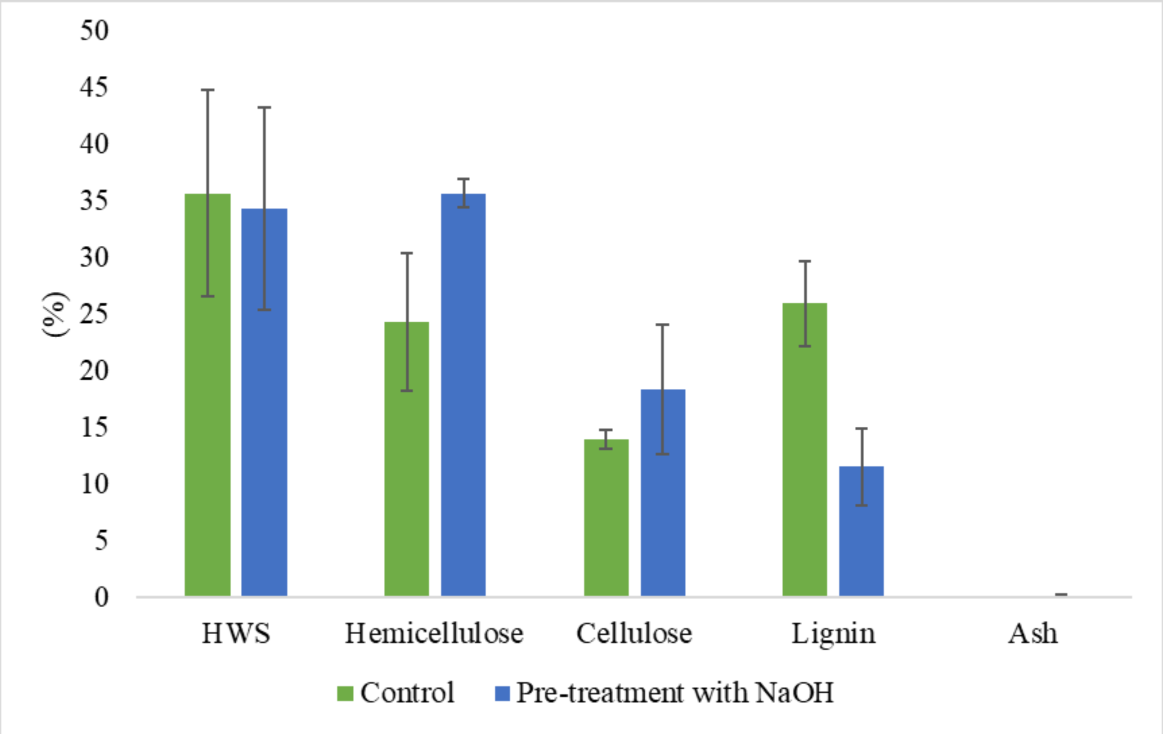

Figure 1. Lignocellulose fractionation with and without NaOH pre-treatment

The percentage of hot water soluble (HWS) fraction before pre-treatment was 35.67 ± 9.1% and after pre-treatment was 34.33 ± 8.96%. The hemicellulose fraction before pre-treatment was 24.33 ± 6.13% and after pre-treatment it was 35.67 ± 1.25%. The cellulose fraction before pre-treatment was 14 ± 0.82% and after pre-treatment was 18.33 ± 5.73%. The lignin fraction before pre-treatment was 25.96 ± 3.76% and after pre-treatment was 11.52 ± 3.36%. The ash fraction before pre-treatment was 0.04 ± 0.02% and after pre-treatment was 0.15 ± 0.06%. According to Gaspar *et al.* (2007), the alkaline treatment method is more effective at destroying the ester bonds between lignin, hemicellulose and cellulose and preventing the fragmentation of hemicellulose polymers. The use of NaOH as a delignifier can damage the lignin structure in the crystalline and amorphous parts. NaOH can extract hemicellulose by breaking down the amorphous structure of hemicellulose. NaOH can also break down lignin at temperatures less than 180°C, so the use of NaOH can destroy lignin as well as extract cellulose and hemicellulose in cinnamon bark.

Lignin levels decreased with the addition of NaOH. This is because the addition of an alkaline base in the form of NaOH will make it easier to break the bonds of the lignin compound. NaOH particles will enter the material and break down the lignin structure (Lutfi *et al.*, 2013) so that lignin dissolves more easily which results in a decrease in lignin levels. The decrease in the percentage of lignin in the cinnamon is also influenced by pH. Lignin will dissolve at high pH (alkaline) because the lignin phenolic hydroxyl group is in an ionized state to form its salt. This treatment will break down lignin into smaller particles (Arianie *et al.*, 2016).

CONCLUSION

The delignification process of cinnamon bark (*C. verum*) with NaOH pre-treatment can reduce the lignin fraction and increase the cellulose and hemicellulose fractions. The cellulose and hemicellulose fractions after NaOH pretreatment were 18.33 ± 5.73% and 35.67 ± 1.25%, respectively.

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