

Comparison of Lignin Degradation in Empty Oil Palm Bunches Biomass with Alkaline Pretreatment

Felix Tayon^{1,2} Roni Maryana¹, Dita Ariyanti^{2,*}, Ori Yansa Garia³

¹ Department of Chemistry, Faculty of Military Mathematics and Natural Sciences, The Republic of Indonesia Defense University (Universitas Pertahanan Republik Indonesia), Bogor 16810, Indonesia

² Research Center for Chemistry, National Research and Innovation Agency, Building 452 KST BJ Habibie, Serpong Tangerang Selatan, Banten, Indonesia

* Corresponding Author: ditafansuri@gmail.com

Abstract

Lignocellulose is an abundant polysaccharide component in nature and consists of cellulose, hemicellulose and lignin. Lignin degradation is a key step in the processing of lignocellulosic biomass from empty oil palm fruit bunches to produce bioenergy and valuable chemicals. Alkaline pretreatment method has become an effective approach in lignin degradation. This study aims to compare alkaline pretreatment with other methods in terms of lignin degradation efficiency. The initial treatment was carried out by cooking using a bench scale reactor at a temperature of 150 oC for 30 minutes. Characteristics before and after alkaline pretreatment were analyzed using Fourier Transform Infrared (FTIR). The research results showed the lowest lignin content was 7.71% with alkali pretreatment using a reactor.

Keywords: alkaline pretreatment; lignin; lignocellulose

Introduction

Oil palm is a tropical plantation crop that produces oil which is widely used in industry. Apart from producing oil from fresh bunches, palm oil produces solid waste in the form of empty palm fruit bunches. Empty oil fruit bunches consist of fiber containing lignocellulose which is often used in energy applications from biomass [1].

Lignocellulose is a complex mixture of the two main components in plant cell walls, namely lignin and cellulose, along with other polysaccharide polymer components such as hemicellulose. It is one of the main components in many types of biomass, including wood, rice husks, straw, and various types of fiber crops [2]. Lignin is one of the building blocks of lignocellulose. Lignin is a strong and complex polymer. This provides additional structural support to the plant, especially to the wood. Lignin also provides resistance to various environmental conditions and attacks by microorganism. However, lignin also makes biomass difficult to decompose or use in bioenergy processing processes, such as bioethanol production. Lignin must be broken down so that cellulose and hemicellulose can be easily accessed for further processing [3].

Lignocellulose breakdown process can be carried out with pretreatment. Pretreatment that can be used is delignification to separate lignin and cellulose [4]. Delignification is the process of dissolving lignin from lignocellulosic materials to obtain purer cellulose. The delignification process can be carried out using several methods such as thermal, physical, chemical and enzymatic processes [5]. This research uses chemical pretreatment with an alkaline solution as a lignin solvent.

The aim of breaking down lignocellulose is to separate lignin from cellulose. The presence of lignin can inhibit the performance of microorganism in the process of converting cellulose into sugar for bioethanol production. High cellulose content will result from optimal treatment. This research aimed to obtain high levels of cellulose by degrading lignin using alkaline pretreatment.

Materials and Methods

Materials

The materials used in this research were NaOH 10% (b/v), H₂SO₄ 72%, CaCO₃ powder, H₂SO₄ 4%, empty oil palm fruit bunch fiber and aquades. The instruments used are test tube, Erlenmeyer, scott bottles, water batch, autoclave, magnetic stirrer, vacuum filtration, beaker, bench scale reactor, crucible, furnace, oven and syringe filter.

Material preparation and alkaline treatment

Empty palm fruit bunch fiber was ground to 200 mesh and weighed 500 g. Put the sample in the reactor then add 2,5 L NaOH. Cooking was carried out for 30 minutes at a temperature of 150 °C and pressure 1 bar. Samples before and after cooking will be analyzed for cellulose and lignin levels using the component analysis. 0,3 g sample was added with 3 mL H₂SO₄ 72% and hydrolyzed for 2,5 hours in a water batch at a temperature of 30 °C. After hydrolysis, the sample was put in a scott bottle with additional 84 mL distilled water. Place the scott bottle in the autoclave for 2 hours at 121 °C. After hydrolysis the sample is separated between filtrate and solid. The filtrate will be analyzed by high performance liquid chromatography (HPLC) and spectrophotometer at wavelength of 205 nm. The solids will be analyzed for ash content. Ash content analysis and spectrophotometer are used to calculate lignin content and HPLC analysis is used to obtain cellulose content.

Oil palm empty bunches characterization

Characterization of functional groups of empty oil palm fruit bunches before and after pretreatment was carried out using a Fourier-Transform Infrared (FTIR) spectrometer in the frequency range 4000-500 cm⁻¹ with a resolution of 2.0 cm⁻¹.

Result and Discussion

Alkaline pretreatment is one of the steps in the processing of organic materials, such as lignocellulosic biomass, before the materials can be used in various applications, such as the production of bioethanol, bioenergy, and biochemical products. Alkaline pretreatment involves treating organic materials with alkaline solution such as NaOH to decompose the cellulose and lignin levels of empty bunches before and after pretreatment. Based on table 2, the lignin content of empty bunches before delignification was 34,9% and cellulose was 19,55%. After delignification the lignin content decreased to 7,71% and cellulose increased to 58,11%. This shows that lignin that lignin levels were removed by 77,84%. Table 3 compares several studies regarding pretreatment with alkaline with this experiment. The results of this experiment were that alkaline pretreatment with NaOH using a bench scale reactor resulted in higher lignin removal compared to other conventional pretreatment. Interestingly, alkaline pretreatment with a bench scale reactor can effectively remove lignin in a very short time compared to other conventional pretreatment.

Table 1. Composition of palm oil waste

No	Component	Content (%)
1	Empty bunch	23
2	Shell	6,5
3	Palm mud	4
4	Fiber	13
5	Liquid waste	50

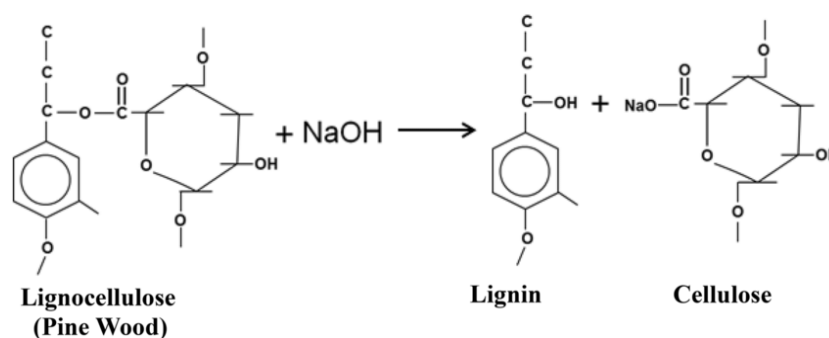


Figure 1. Lignocellulose breakdown reaction with NaOH

Alkaline pretreatment increases the conversion of lignocellulosic biomass into bioenergy products or chemicals by reducing the resistance caused by lignin and increasing the accessibility of cellulose and hemicellulose. Factors such as alkali type, concentration, temperature, contact time, and biomass-alkali ratio greatly influence the alkali pretreatment efficiency and final biomass conversion results. The factors that influence the rate of reduction in lignin levels in pretreatment include: the concentration of alkali used.

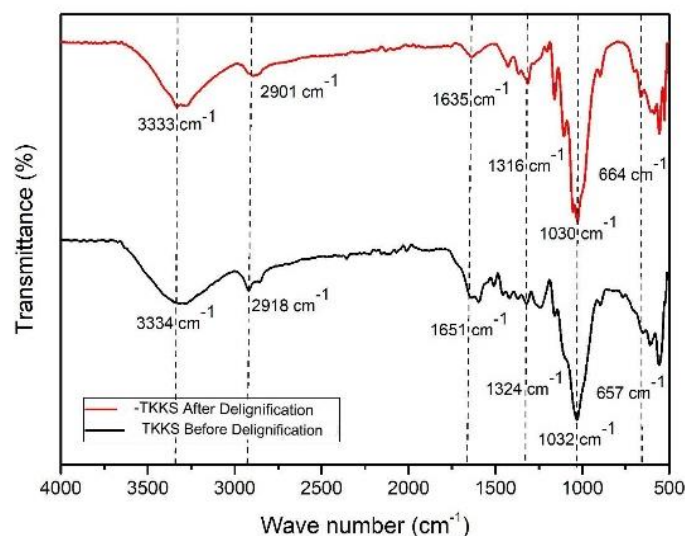


Figure 2. IR spectrum before and after delignification

Table 2. Component analysis before and after pretreatment

Treatment	Content (%)	
	Lignin	Cellulose
Before	34,9	19,55
After	7,71	58,11

Table 3. Comparison of alkaline pretreatment

Pretreatment Condition	Lignin Removal	Reference
Conventional pretreatment with 10% NaOH at 70° C	47,81%	[6]
Microwave-assisted NaOH pretreatment at 840° C	73,75%	[7]
Conventional pretreatment with 10% NaOH at 150° C	47,31%	[8]
Conventional pretreatment with NaOH at 100° C	59,1%	[9]
Conventional pretreatment with 10% NaOH at 150° C	77,84%	This Research

High concentrations of alkali can degrade lignin, but concentrations that are too high can damage cellulose. Apart from that, temperature and cooking time are key in lignin degradation. The same thing with concentration, the higher the temperature can damage the cellulose. Based on Figure 2, there is a difference between the spectrum of empty bunches before and after delignification. The wave number 3333 cm^{-1} indicates OH stretching of hydrogen bonds and the peak at 2901 cm^{-1} comes from CH stretching

[10]. The peak appearing around 1316 cm^{-1} indicates OH bending in cellulose and the peak at 1030 cm^{-1} in the spectrum indicates COC stretching of the β -1,4-glycosidic bond. an increase in the peak at wave numbers $500\text{--}1100\text{ cm}^{-1}$ indicates an increase in cellulose content.

Conclusion

Alkaline pretreatment with cooking for 30 minutes using a reactor obtained optimal results when compared with the results of previous research. Furthermore, cellulose levels increased from 19,55% to 58,11%. This was followed by a decrease in lignin levels from 34,9% to 7,71%. The increase in cellulose content after pretreatment was detected by FTIR spectrum.

Acknowledgements

Thank you to the BRIN Advanced Chemical Research Center for research funding and Mr. Roni Maryana for technical support.

References

- [1] Agustini, L., & Efiyanti, L. (2015). Pengaruh Perlakuan Delignifikasi Terhadap Hidrolisis Selulosa Dan Produksi Etanol Dari Limbah Berlignoselulosa. *Jurnal Penelitian Hasil Hutan*, 33(1), 69–80. <https://doi.org/10.20886/jphh.v33i1.640.69-80>
- [2] Asmoro, N. W., Hidayat, C., Ariyanto, T., & Millati, R. (2023). Cellulose Isolation From Oil Palm Empty Fruit Bunch Using Different Pretreatment Processes. *Journal of Applied Science and Engineering (Taiwan)*, 26(11), 1513–1520. [https://doi.org/10.6180/jase.202311_26\(11\).0001](https://doi.org/10.6180/jase.202311_26(11).0001)
- [3] Barlianti, V., Dahnum, D., Hendarsyah, H., & Abimanyu, H. (2015). Effect of Alkaline Pretreatment on Properties of Lignocellulosic Oil Palm Waste. *Procedia Chemistry*, 16, 195–201. <https://doi.org/10.1016/j.proche.2015.12.036>
- [4] Darojati, H. A., Purwadi, R., & Rasrendra, C. B. (2020). Proses Fraksionasi Biomassa dari Tandan Kosong Kelapa Sawit melalui Metode Organosolv Etanol dengan Penambahan Katalis. *Jurnal Selulosa*, 10(02), 73. <https://doi.org/10.25269/jsel.v10i02.303>
- [5] Devi, D., Astutik, D., Cahyanto, M. N., & Djaafar, T. F. (2019). Kandungan Lignin, Hemiselulosa Dan Selulosa Pelepah Salak Pada Perlakuan Awal Secara Fisik Kimia Dan Biologi. *Jurnal Ilmiah Rekayasa Pertanian Dan Biosistem*, 7(2), 273–282. <https://doi.org/10.29303/jrpb.v7i2.148>
- [6] Fakhriansyah, M., Fathimahhayti, L. D., & Gunawan, S. (2022). G-Tech : Jurnal Teknologi Terapan. *G-Tech : Jurnal Teknologi Terapan*, 6(2), 295–305.
- [7] Maryana, R., Ma'rifatun, D., Wheni, I. A., K.w., S., & Rizal, W. A. (2014). Alkaline pretreatment on sugarcane bagasse for bioethanol production. *Energy Procedia*, 47, 250–254. <https://doi.org/10.1016/j.egypro.2014.01.221>
- [8] Sindhuwati, C., Mustain, A., Rosly, Y. O., Aprijaya, A. S., Mufid, M., Suryandari, A. S., Hardjono, H., & Rulianah, S. (2021). Review: Potensi Tandan Kosong Kelapa Sawit sebagai Bahan Baku Pembuatan Bioetanol dengan Metode Fed Batch pada Proses Hidrolisis. *Jurnal Teknik Kimia Dan Lingkungan*, 5(2), 128–144.

- <https://doi.org/10.33795/jtkl.v5i2.224>
- [9] Zuhri, F., Arbianti, R., Utami, T. S., & Hermansyah, H. (2016). Effect of methylene blue addition as a redox mediator on performance of microbial desalination cell by utilizing tempe wastewater. *International Journal of Technology*, 7(6), 952-961. <https://doi.org/10.14716/ijtech.v7i6.1795>
- [10] Rahmawati, F., Basuki, R., Fahri, M., Apriliyanto, Y. B., Kurniadi, T., Nareswari, V. A., Azzahra Sandri & Istiqomah, T. (2024). Reaction Mechanism in Standardized α -Cellulose Content Test: Study from Boehmeria nivea Fiber. *Indonesian Journal of Chemical Studies*, 3(1), 22-27. <https://doi.org/10.55749/ijcs.v3i1.45>