



DELIGNIFICATION OF OIL PALM EMPTY BUNCH USING PHYSICAL AND CHEMICAL COMBINATION METHODS

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ABSTRACT

Oil palm is a common plant in Sumatera Island Indonesia for the production of cooking oil. In the production of cooking oil, oil palm empty bunch is one of solid waste and the availability is in great amount. Oil palm empty bunch are commonly used as boiler, fertilizer and mulch as well as road hardeners. Oil palm empty bunch contain high lignocellulose, which is good for the main ingredient of ruminant feedstock after removing lignin from it using delignification process. Chemical method and combination of physical and chemical delignification were performed to investigate the process condition which result on the greatest lignin reduction. Sodium hydroxide solution and hydrogen peroxide in acetic acid solution were used as delignification agents. Physical methods such as sonication-microwave heating and direct heating were conducted after chemical treatment on the oil palm empty bunch. Delignification using 9% sodium hydroxide solution or 50% hydrogen peroxide in 15% acetic acid solution produced the greatest lignin reduction from 31.59% become 25.84% and 25.61%, respectively. Combination physical treatment (sonication and microwave or direct heating) and chemical treatment greatly reduced the amount of lignin when it using 50% hydrogen peroxide in 15% acetic acid solution. The amounts of lignin reduced become 3.11% and 1.46% for sonication-microwave and direct heating, respectively.

Keywords: oil palm empty bunch, delignification, sodium hydroxide, hydrogen peroxide in acetic acid.

1. INTRODUCTION

Indonesia is one of the countries with the largest number of oil palm trees (*Elaeis guineensis Jacq*) in the world. According to data from the Indonesian statistics agency, oil palm plantations in Indonesia are expanding from year to year [1]. The increasing demand for cooking oil causes the production of cooking oil from palm oil to increase and consequently the solid waste in the form of empty palm oil bunch is also available in very large quantities. Oil palm empty bunch are commonly used as boiler, fertilizer and mulch as well as road hardeners.

Oil palm empty bunch contain high lignocellulosic content (hemicellulose, cellulose, lignin). Considering the high amount of availability of oil palm empty bunch and high content of hemicellulose and cellulose in oil palm empty bunch; it is suitable for the raw material of ruminant feedstock. However, the lignin content in oil palm empty bunch must be removed before being used as raw material of ruminant feedstock. The delignification process will break down the lignin in crystalline and amorphous form so that the lignin is extracted out of the solid matrix in order to obtain hemicellulose and cellulose.

There are many types of delignification processes, such as physical, chemical, biological and its combinations [2-10]. Comparing those methods, chemical delignification has the shortest time and high lignin content reduction. In this study, physical, chemical and its modification of delignification process were performed in order to achieve the highest of lignin content reduction.

2. RESEARCH METHODOLOGY

2.1. Materials

Palm oil empty bunch used in this experiments were obtained from North Sumatera, Indonesia. Palm oil empty bunch was dried using oven (Memmert) at 50 °C for 24 h. Dried palm oil empty bunched was grinded (Janke dan Kunkel GmbH dan Co. KG) until become powder that passed 30 mesh sieving. Powder sample than keep in seal container for further experiments and analysis. The initial content of powder sample was moisture content 12% (Ohaus- MB35 Halogen), cellulose 27.8%, Hemicellulose 28.8% and lignin 31.4%. The amount of lignin, cellulose and hemicellulose was determined using Van Soest method [11].

Chemicals used for delignification process were sodium hydroxide, hydrogen peroxide, acetone, acetic acid, sulfuric acid and cetyl trimethylammonium bromide were analytical grade from Merck Germany.

2.2 Method

Chemical method of delignification of powder sample was performed using sodium hydroxide or hydrogen peroxide in acetic solution. 3g of powder sample was soaking in 150ml sodium hydroxide (5-10% concentration) or hydrogen peroxide in acetic solution (concentration of hydrogen peroxide was 50% and the concentration of acetic acid were 9-18%) for 90 min. After the soaking time, the sample was washed using distilled water until its pH was neutral and dried in oven (Memmert) at 105 °C for 16 h. The amount of lignin, cellulose and hemicellulose was determined using Van Soest method [11]. The chemical method then combined



with physical method (sonication and microwave or direct heating). Physical method was applied after soaking time; the time for sonication (Heraeus Labofuge 200) and microwave (National NN-S327 WF) was 30min, respectively, and direct heating time was 60min.

3. RESULT AND DISCUSSIONS

Chemical method of delignification was effective in reducing the amount of lignin from powder sample (oil palm empty bunch). Figure-1 shows the result of lignin,

hemicellulose and cellulose after delignification using sodium hydroxide. The amount of lignin decreased as the concentration of sodium hydroxide solution used in delignification process was increasing. The lowest lignin content was obtained when it was treated using 9% of sodium hydroxide solution become 25.84%. As the amount of lignin decreasing, the percentage of hemicellulose and cellulose in the processed sample increased as shown in Figure-1.

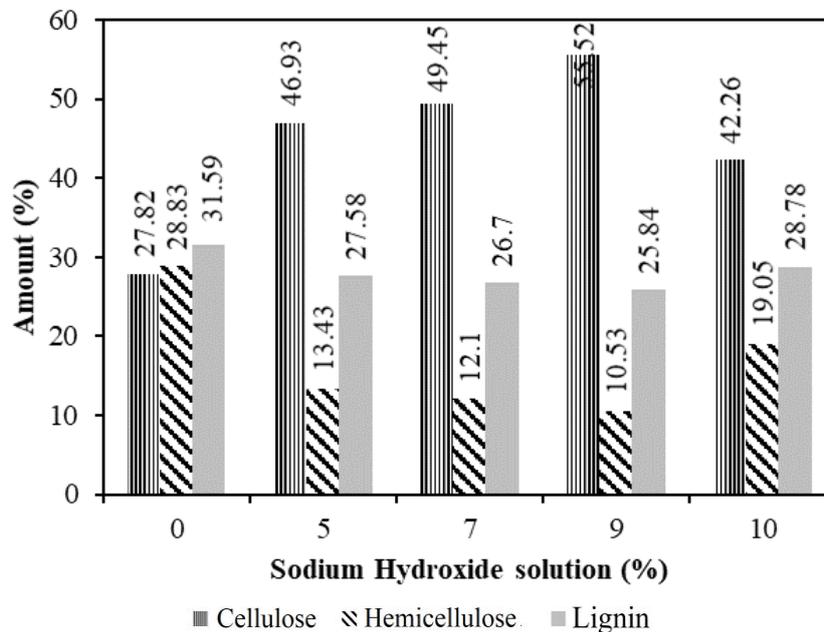


Figure-1. The content of oil palm empty bunch after delignification process using various sodium hydroxide solution with moisture content $12 \pm 0.6\%$

Delignification result using hydrogen peroxide in acetic acid solution was shown in Figure-2. In this experiment, the concentration of peroxide was constant (50%), while the concentration of acetic acid was varied from 9-18%. The lowest lignin content (25.61%) in processed sample was obtained using acetic acid concentration of 15%, which is similar to the result of delignification using sodium hydroxide solution 9%. These result showed that both chemical solution are potential for delignification agent, however considering the amount of lignin is still high, step process of delignification can be performed in order to eliminate all lignin from oil palm empty bunch.

Delignification agents (sodium hydroxide solution or hydrogen peroxide in acetic acid solution) used in these experiments was functioned to break the bonding in lignocelluloses, therefore, as the amount of delignification agents was increasing; the amount of lignin in sample was decreasing. However, extracted lignin may be deposited in the sample during filtration process when the delignification processes using higher concentration of 9% for sodium hydroxide solution and 15% for acetic acid.

Delignification using microwave or heating in the form of steam explosion or hot water have been explored in order to reduce the amount of lignin as much as possible [4-5, 8-10]. In the delignification process of oil palm empty bunch, microwave and sonication or direct heating had significant effect when it used together with hydrogen peroxide in acetic acid solution as delignification agent (Table-1).

The amount of lignin reduced from 31.59% became 3.11%. The effectiveness of direct heating had the same trend as sonication and microwave as shown in Table 1, even though direct heating reduced the amount of lignin slightly greater than sonication and microwave for the same delignification agent. However, in this experiment, sonication and microwave heating did not influence the reduction of lignin amount when it used sodium hydroxide solution for delignification (Table-1).

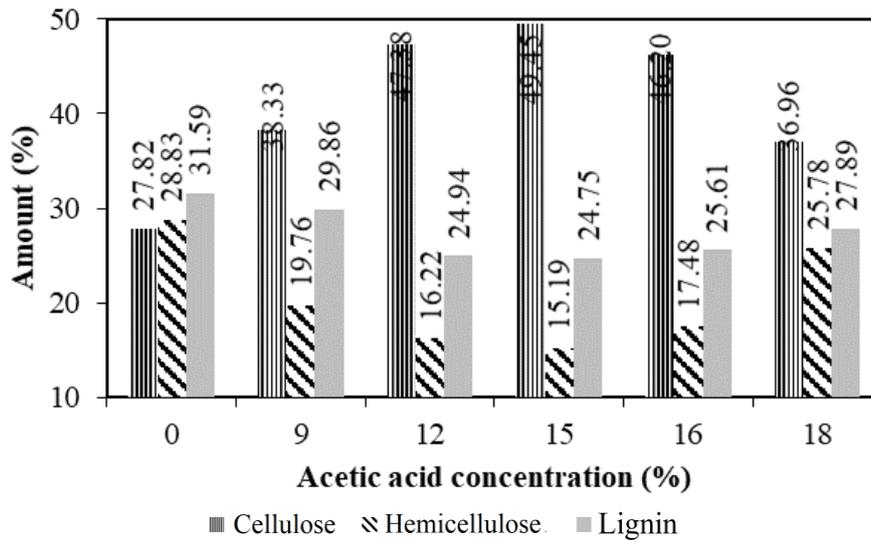


Figure-2. The content of oil palm empty bunch after delignification process using hydrogen peroxide 50% and various acetic acid solution with moisture content $12\pm 0.6\%$

Sodium hydroxide solution in low concentration caused swelling and leading to an increase surface area, a decrease in the degree of polymerization and crystallinity, separation of structural linkages between lignin and hemicellulose.

cellulosic, and disruption of the lignin structure [12]; therefore the effect of combining method with sonication and microwave or direct heating were slightly reduced the amount of lignin.

Table-1. The oil palm empty bunch content before and after delignification process with moisture content of $12\pm 0.6\%$.

Delignification method	Amount (%)					
	Lignin		Hemicellulose		Cellulose	
	A	B	A	B	A	B
Pristine oil palm empty bunch	31.59		28.83		27.82	
Sodium hydroxide solution (9%)	28.11	27.76	14.20	13.38	51.23	51.45
Hydrogen peroxide (50%) in acetic acid (15%) solution	3.11	1.46	14.62	10.29	70.21	79.71

A: followed by sonication 30min and microwave heating 30min

B: followed by direct heating at 165°C for 60min

Various type of delignification methods influenced the strength of lignocellulose bonding, which shown in the result of Fourier Transform Infra Red (FTIR) spectra (Figures 3 and 4). Both Figures 3 and 4 shown that delignification process caused shifting on stretching of O-H bonding ($3579\text{-}3728\text{ cm}^{-1}$), which is main functional bond in cellulose. Shifting of functional bonding in lignin

were shown in wave number $1442\text{-}1447\text{ cm}^{-1}$, which represent O-CH₃ bonding in lignin. The FTIR peak showed that the bonding strength was weaker after delignification processes. Wave number $1254\text{-}1260\text{ cm}^{-1}$ is indicating C-O-C (aryl-alkyl ether) bonding in lignin, and the FTIR peak showed that it was strong before delignification process.

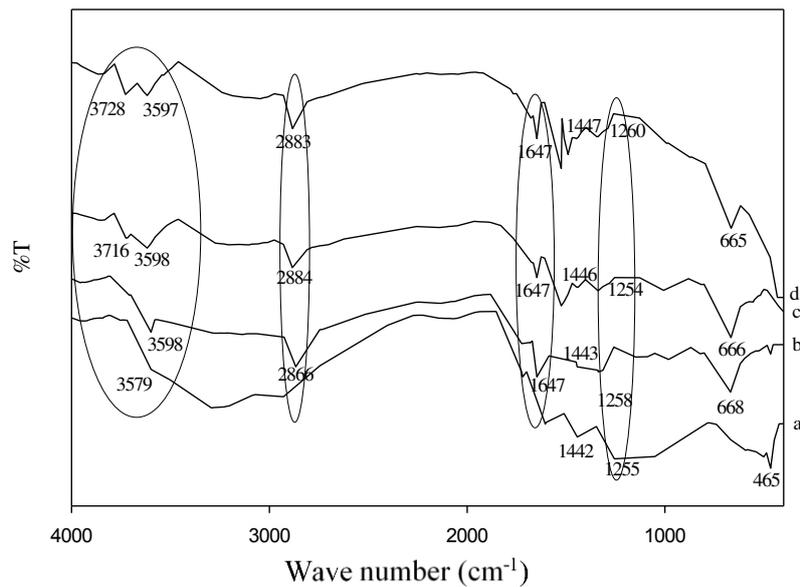


Figure-3. FTIR spectra of powder sample of oil palm empty bunch (a: pristine sample; b: 9% sodium hydroxide solution; c: 9% sodium hydroxide solution + sonication + microwave; d: 9% sodium hydroxide solution + direct heating).

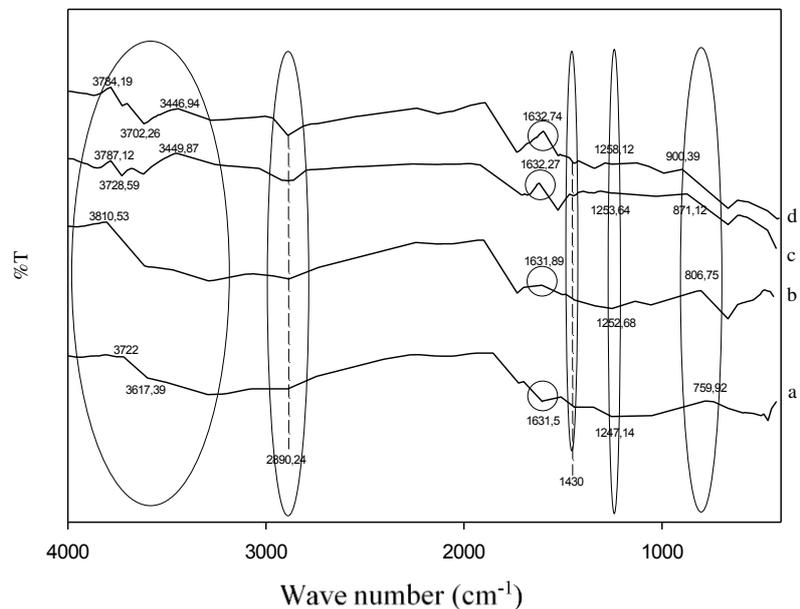


Figure-4. FTIR spectra of powder sample of oil palm empty bunch (a: pristine sample; b: 50% hydrogen peroxide in 15% acetic acid; c: 50% hydrogen peroxide in 15% acetic acid + sonication + microwave; d: 50% hydrogen peroxide in 15% acetic acid + direct heating)

4. CONCLUSIONS

The type of chemical solution was affecting the amount of lignin reduction. Delignification using 9% sodium hydroxide solution or 50% hydrogen peroxide in 15% acetic acid solution produced the greatest lignin reduction from 31.59% become 25.84% and 25.61%, respectively. Combination physical treatment (sonication

and microwave or direct heating) and chemical treatment greatly reduced the amount of lignin when it using 50% hydrogen peroxide in 15% acetic acid solution. The amounts of lignin reduced become 3.11% and 1.46% for sonication-microwave and direct heating, respectively.



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REFERENCES

- [1] Perkebunan D.J. 2015. Tree Crop Estate Statistics of Indonesia, M.O. Agriculture, Editor. Jakarta.
- [2] Hadrawi J. 2014. The Content of Lignin, Cellulose, and Hemicellulose of Oyster Mushroom (*Pleurotus ostreatus*) medium waste at different incubation period as feed, in Fakultas Peternakan. Universitas Hasanuddin Makassar.
- [3] Mosier N., *et al.* 2005. Features of promising technologies for pretreatment of lignocellulosic biomass. *Bioresource Technology*. 96: 673-686.
- [4] Barakat A., H.d. Vries and X. Rouau. 2013. Dry fractionation process as an important step in current and future lignocellulose biorefineries: A review.
- [5] Agbor V.B., *et al.* 2011. Biomass pretreatment: Fundamentals toward application. *Biotechnology Advances*. 29: 675-685.
- [6] Pedersen M., A. Viksø-Nielsen and A.S. Meyera. 2010. Monosaccharide yields and lignin removal from wheat straw in response to catalyst type and pH during mild thermal pretreatment. *Process Biochemistry*. 45: 1181-1186.
- [7] Silverstein R.A., *et al.* 2007. A comparison of chemical pretreatment methods for improving saccharification of cotton stalks. *Bioresource Technology*. 98: 3000-3011.
- [8] Wiman M., *et al.* 2012. Cellulose accessibility determines the rate of enzymatic hydrolysis of steam-pretreated spruce. *Bioresource Technology*. 126: 208-215.
- [9] Banerjee S., *et al.* 2009. Evaluation of wet air oxidation as a pretreatment strategy for bioethanol production from rice husk and process optimization. *biomas s and bio energy*. 33: 1680-1686.
- [10] Binta D., S. Wijana and A.F. M. 2013. The Influence Long of Curing to the Levels of Lignin and Cellulose Pulp (Bark and Midrib of Nypa) Using Biodegradator EM4. *Jurnal Industrial*. 2(1): 75-83.
- [11] Soest V.P.J. 1963. Use of detergents in the analysis of fibrous feeds. 2. A rapid method for the determination of fiber and lignin. *Journal of the Association of Official Agricultural Chemists*. 46: 829-835.
- [12] Sun Y., Cheng J. 2002. Hydrolysis of lignocellulosic materials for ethanol production: a review. *Bioresource Technology*. 83: 1-11.