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# LIGNIN DEGRADATION OF KAPOK FIBER (*Ceiba pentandra*, L) WITH DIFFERENT TIMES OF PULPING

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***Abstract**. Lignin reduced the adhesion between the polymeric matrix and natural reinforcement in composite materials by its hydrophobic characteristic. Removal of lignin can be a solution to improve fiber function in composite use. This study aimed to determine the degradation of lignin at different times of pulping. The soda process was conducted at 60, 70, 80, 90, and 100 minutes of pulping using heat in a room atmosphere. Yields, lignin content, and lignin spectrum (FT-IR analysis) were measured. The result showed the highest degradation of lignin was in 100 minutes of pulping with 80.34% lignin degradation and 3.57% lignin left in fibers. However, the yield of fibers decreased with increasing pulping time due to lignin removal. 100 minutes of pulping time showed the highest loss of fiber yield. This study showed the best pulping time for kapok fiber is 100 minutes with 3.57 % lignin remaining in the pulp.*

***Keywords:** degradation; kapok fiber; lignin; pulping; yield*

## 1. Introduction

The utilization of lignocellulosic materials gained attention as a promising approach for the production of environmentally friendly products. Lignocellulosic materials, known for their abundance, affordability, and biodegradability, have attracted interest. However, the presence of lignin in these materials posed a challenge to their use in fuel and chemical production (Jamaldheen *et al*., 2022). It has been reported that the mechanical fibrillation process during the production of lignocellulose nanofibers is prolonged by lignin (Zhang *et al*., 2022) and that enzymatic hydrolysis is inhibited through the formation of non-productive cellulose binding by lignin (Yoo *et al*., 2020; Yuan *et al*., 2021). Additionally, non-productive adsorption could be induced by residual lignin in lignocellulosic materials due to disparities in molecular weight and hydroxyl groups (Yao *et al*., 2022).

Lignin is a phenylpropanoid polymer composed of aromatic alcohols called monolignols with three primary monolignols: p-coumaryl alcohol, coniferyl alcohol, and sinapyl alcohol. Lignin is formed through highly crosslinked polymerization of these monolignols which involve various chemical bonds, such as C-C, C-O, and ether linkages (Xu *et al*., 2020). Lignin exhibits

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potential application in the production of benzene (Meng *et al*., 2021), as well as antioxidant and hydrophobic membranes (Fontes *et al*., 2021). However, in the biocomposite field, the presence of lignin inhibits the hydrolysis process and reduces adhesion on composite, which can negatively affect product quality (Arslan *et al*., 2020).

Lignin's complex bonds, including aryl ether bonds, can be broken to eliminate the presence of lignin in lignocellulose materials. Aryl ether bonds are relatively easy to break due to non-condensable bonds within lignin, and this process could be accomplished through various methods (Jung *et al*., 2018). The Removal of lignin can be achieved through the utilization of enzymes or chemical treatment. One widely employed method for lignin removal from lignocellulosic materials is the alkaline treatment using sodium hydroxide (NaOH) (Modenbach & Nokes, 2014; Hassan & Badri, 2014; Jung *et al*., 2018). NaOH breaks the bonds within lignin, consequently impacting the lignin within the materials. Lignin elimination from the fibers can be achieved by hydrolyzing the ester bonds in lignin structures through NaOH treatment at low temperatures and low pressure (Liu *et al*., 2014). NaOH is used as the alkaline agent for lignin removal at various concentrations based on the raw material (Ciftci *et al*., 2018; Jung *et al*., 2018; Jiang *et al*., 2020).

Kapok fiber is one of the tropical plants that is grown in Indonesia. This fiber is locally used as a stuffing material for dolls and pillows (Sangalang, 2021). Kapok fibers consist of approximately 64% cellulose (Wang *et al*., 2012; Bozaci, 2019) and served as potential sources of cellulose. Nevertheless, the high lignin content, up to 21% (Dong *et al*., 2015) in kapok could pose a challenge. While kapok could be used as a reinforcement material in composites, the presence of lignin in kapok could inhibit composite matrix and reinforcement surface adhesion due to its hydrophobic nature. This results in the creation of a barrier on the composite, consequently impairing its quality. To further the utilization of kapok fiber, this study aims to determine the degradation of lignin at different times of pulping.

## 2. Methods

### 2.1. Material

Kapok fibers were collected directly from the farmers in the Kapok production center, Pati, Central Java, Indonesia. The collected fibers were treated in soda process conditions with different times of pulping (60, 70, 80, 90, and 100 minutes). The soda process used sodium hydroxide (NaOH, 3.5%) that was purchased from Merck, Germany.

### 2.2. Pulping treatment

Kapok weight 15 g and then placed in 1000 ml Erlenmeyer. NaOH 3.5% was then added into Erlenmeyer at kapok to NaOH solution ratio of 1:15 using dry weight of fibers. Subsequently, Erlenmeyer was heated on a hotplate using 105°C temperature under room atmosphere conditions.

The soda process was completed over different times of pulping (60, 70, 80, 90, and 100 minutes). The resulting fibers were rinsed with distilled water until neutral pH was attained. The treated pulp was treated was then adjusted for moisture content and prepared for lignin content and FT-IR analysis.

### 2.3. Lignin content

2 g dry-weight fibers were placed in 1000 ml Erlenmeyer. 72% NaOH solution t was prepared before pouring 25 ml into Erlenmeyer. The solution was then placed in the acid room for a period of 2 hours. Furthermore, the solution was placed on a hot plate with a condenser to prevent water evaporation and was heated for an additional 4 other hours to complete the reaction. The resulting solution was then washed in distilled water until pH neutral was achieved. The fiber was then dried in over with 105°C temperature until a constant weight was reached. The Lignin content was determined using the Equation (1).

$$L\ (\%) = \frac{I_w}{R_w}\ x100\% \qquad (1)$$

L is lignin content, $I_w$ for dry weight of fiber, and $R_w$ resulted in weight after the reaction was done.

### 2.4. Fourier Transform Infrared Spectroscopy (FT-IR) analysis

The surface chemistry of the fiber was implemented By Fourier Transforms Infrared Spectrophotometer using prepared pulped fibers. The dried fibers were examined from 4000 to 410 by FT-IR spectrophotometer (IRTracer-100, SHIMADZU, Japan) with KBr/pellet ratio (99/1).

## 3. Results and Discussion

### 3.1. Yield

The degradation of lignin in kapok fiber affects the yield of kapok fiber. Figure 1 showed that the yield of kapok fiber is reduced due to the addition of NaOH. The condition of 60 minutes of pulping showed 79.72% yield, which further decreases to 78.95% at 70 minutes, 73.94% at 80 minutes, 76.74% at 90 minutes, and 71.22% at 100 minutes. Lignin degradation was responsible for the removal of lignin from lignocellulosic materials, resulting in a decrease in fiber yield (Melro *et al*., 2020). This reduction was attributed to the degradation of certain C-C, ether, and C-H bonds through heat and NaOH reactions. The reaction with alkaline affected the structure of lignin, leading to the release of smaller and consequently reduced lignin content in the fibers (Xu *et al*., 2020). The various duration of the pulping process for fiber impacted the yield of lignin degradation. The breakage of the lignin bonds in the cell wall stopped when the pulping time is halted, and as a result, lignin degradation is not fully optimized.

### 3.2.Lignin content

The lignin content of kapok fiber from Pati is 18.16% and it decreased after being treated with pulping time. Figure 3 showed the lignin content of kapok fiber decreased from 60 minutes of pulping with 7.37% lignin content, 70 minutes with 6.39%, 80 minutes with 4.49%, 90 minutes with 4.37%, and 100 minutes with 3.57% of residual lignin in the pulp. Lignin content in fibers decreased due to reaction of lignocellulosic material with NaOH solution at a high temperature, leading to lignin degradation.

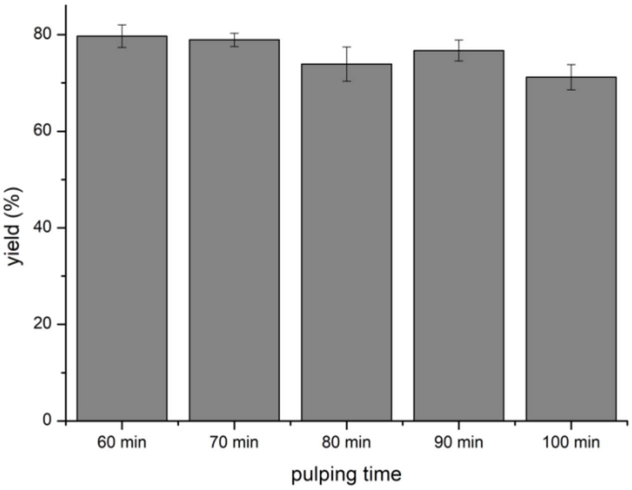


Figure 1. The yield of kapok fiber with different pulping time

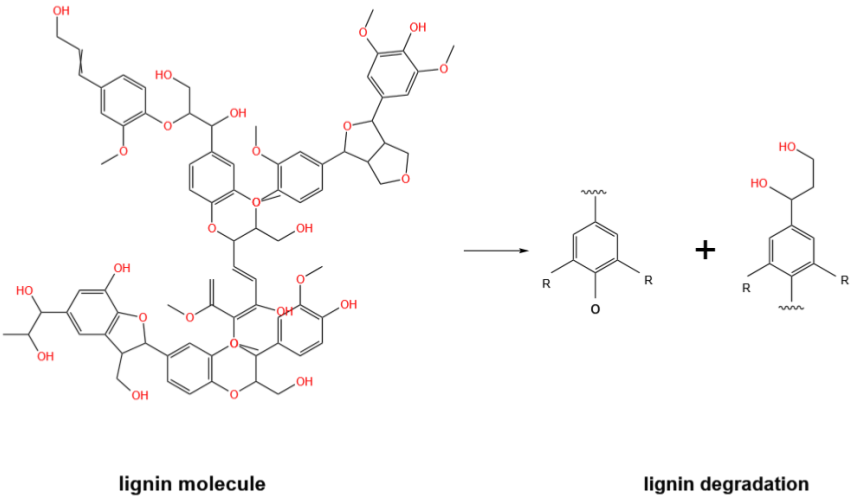


Figure 2. Degradation of lignin

During the alkaline pulping process, lignin is treated with an alkaline solution which causes the lignin to undergo cleavage reactions. One of the most common reactions is the cleavage of ether linkages between lignin monomers, forming lignin fragments (Ding *et al.*, 2021). The hydroxide ion (OH-) from the NaOH attacks the ether linkage between two phenylpropane units in the lignin model compound, leading to the cleavage of the bond and the formation of a lignin fragment. The resulting lignin fragment has a carbonyl group (C=O) and a carboxyl group (COOH) that were formed from the cleaved ether linkage. The resulting lignin fragments are much smaller and simpler in structure than the original lignin polymer (Figure 2). Additionally, the alkaline conditions can cause the formation of new carbonyl and carboxyl groups on the lignin fragments. The reaction affected the fibers, giving them better flexibility and making them softer due to swelling of the cell walls (Jin *et al*., 2021).

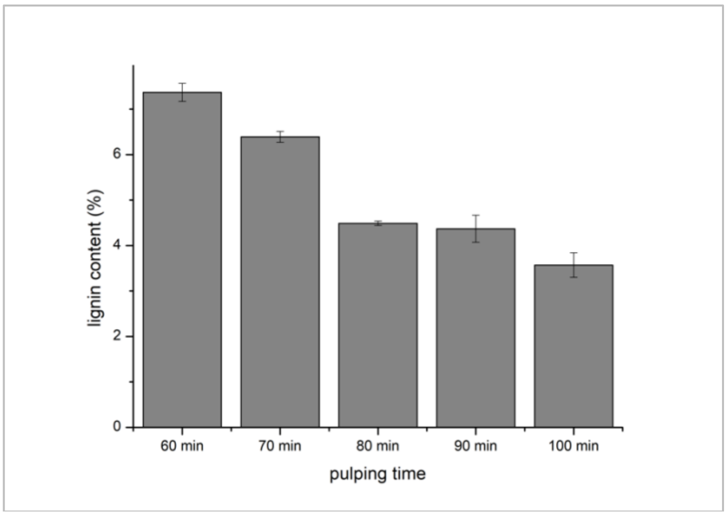
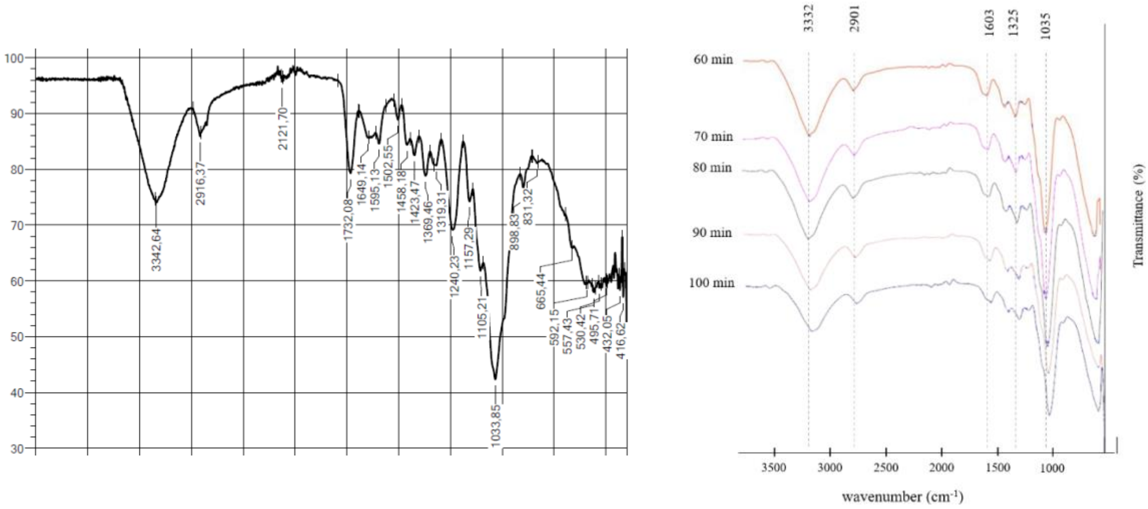


Figure 3. Lignin content of different pulping times of kapok fiber



**(a)** **(b)**

Figure 4. FT-IR spectra of (a) untreated kapok fiber and (b) different pulping times of kapok fiber

## 3.3. FT-IR analysis

Lignin is an aromatic polymer derived from aromatic alcohol sub-unit by incorporation onto the (-OH) groups of aromatic rings of phenylpropanoid monomer units (p-hydroxyphenyl, guaiacyl, and syringyl units) (Queiroz *et al*., 2015). FT-IR analysis showed the pulping reaction caused changes in lignin. The peak at 1603 cm$^{-1}$ (60 mins pulping), 1601 cm$^{-1}$ (70 mis of pulping), 1599 cm$^{-1}$ (80 mins of pulping), 1601 cm$^{-1}$ (90 mins of pulping), and 1599 cm$^{-1}$ (100 mins of pulping) (Figure 4b) corresponds to the aromatic ring of lignin. The pulping process on fiber led to the degradation of lignin as seen in Fig. 4 where the C-C stretch broadened after the pulping reaction. This finding is consistent with the works of (Gabhane *et al*., 2020).

It can be seen in the pictures (Figure 4a) that the C-H stretching was found at a peak of 2916 $cm^{-1}$ for kapok without treatment. Furthermore, the range of C-H stretching remained within the peak range of 2899-2904 $cm^{-1}$ after the pulping treatment. The presence of O-H group stretching was observed at 3342 $cm^{-1}$ and 3332 $cm^{-1}$ for untreated and treated fiber, respectively, which is consistent with findings from (Tolesa *et al*., 2020). The hydrolysis reaction during pulping leads to the breakdown of lignin bonds in lignocellulose, resulting in the alteration of functional groups with C-H and H-O bonds. Several fingerprints exhibit high intensity in the peak range of 1000 $cm^{-1}$ on untreated fiber. After the pulping treatment, new fingerprints become visible within the peak range of 500 $cm^{-1}$, indicated of a C=C stretching of alkene that was found due to the reaction of pulping with kapok fiber.

## 4. Conclusions

In this work, the degradation of lignin was observed with different pulping times. Different pulping durations led to a reduction of lignin content in kapok fiber. After 60 minutes of pulping, the yield was 79.72%, which decreased to 71.22% after 100 minutes of pulping. Alkaline treatment breaks the ester bonds of lignin, resulting in the degradation of lignin polymers and the removal of the bonds among lignin, cellulose, and hemicellulose. The result indicated that the optimal pulping time for kapok fiber is 100 minutes with 3.57 % lignin remaining in the pulp. The Pulping treatment on kapok fibers also generated distinct fingerprint peaks on FT-IR analysis due to the chemical reaction between the fiber and the alkaline solution used in the pulping process.

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