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# ENHANCEMENT OF WATER RESISTANCE IN ABACA BANANA STALK FIBER MOLDED PULP USING ALKYL KETENE DIMER (AKD) FOR FOOD PACKAGING

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**ABSTRACT**
The present study investigates the fabrication and characterization of molded pulp derived from abaca banana stalk fibers with the incorporation of Alkyl Ketene Dimer (AKD). The addition of AKD aims to enhance the hydrophobicity of the molded pulp, thereby improving its water resistance and overall material performance. The research stages include preliminary preparation of abaca banana stalk fibers, processing of abaca banana stalk fibers into pulp, mixing of AKD with the pulp, molding of the pulp, and analyzing its mechanical properties and performance evaluation. The pulp production process was conducted using steam treatment and acid-base hydrolysis reactions. The results indicated that the printed pulp with AKD additive exhibited the following characteristics: a paper thickness of 1.55 mm, a grammage value of 633.27 g/m², a paper density ranging from 404.5 × $10^3$g/$m^3$to 419.6 × $10^3$ g/$m^3$, and a moisture content between 10.34% and 29.21%. The FTIR analysis revealed interactions between cellulose and AKD, characterized by a decrease in the intensity of the –OH absorption band at 3287.5 $cm^{-1}$, with the peak becoming broader and less sharp. Additionally, the CH-aliphatic absorption shifted from 2892.4 $cm^{-1}$ to 2914.8 $cm^{-1}$, with the single absorption peak splitting into a weaker double peak. Surface morphology analysis using SEM revealed changes in the molded pulp's surface, characterized by a reduction in small pore volume, resulting in a smoother surface morphology. These findings indicate a decrease in the water absorption capacity of the molded pulp. This study demonstrates that modifying abaca banana stalk pulp with AKD enhances the material's hydrophobic properties, thereby providing strong potential for its development as a water-resistant and sustainable biomass-based food packaging material.
**Keywords:** Pulp; Fiber**;** Water-Resistance; Mechanical-Properties; Cellulose.
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## INTRODUCTION

The problem of plastic waste from food packaging continues to escalate annually, prompting the development of more environmentally friendly packaging materials. One promising alternative is the utilization of lignocellulosic biomass, such as abaca banana stem fiber, as a raw material for biodegradable molded pulp.[1] However, cellulose-based materials generally exhibit low water resistance, necessitating improvements to their hydrophobic properties. One widely used method in the paper industry is the addition of AKD as a sizing agent, which enhances water resistance by forming β-keto ester bonds with the hydroxyl groups of cellulose.[2,3] Although the effectiveness of AKD has been widely reported in conventional paper and paperboard systems, its application in molded pulp derived from non-wood fibers, particularly abaca banana stem fibers, remains limited and has not been extensively studied, especially for biodegradable food packaging applications.[4,5] Abaca is a natural fiber resource characterized by its high tensile strength, attributed to the unique architecture of its cell walls. The cell walls consist of pectin (0.5-1%), hemicellulose (19-25%), lignin (5-13%), water-soluble substances (1.4%), fats and waxes (0.2-3%), and primarily cellulose (56-68%).[6] Regarding the difference in fiber position within the pseudostem, variations in biochemical composition can be expected.[7] The international production of abaca fiber is 65,000 tons per year, while the demand has reached 85,000 tons per year, resulting in a shortage of about 20,000 tons annually. Abaca fiber is widely used as a raw

material for paper pulp.[8] Abaca-derived pulp and paper exhibit several beneficial properties, one of which is their strong resistance to tearing. Once converted into paper, it is difficult to counterfeit, and the paper produced is used for security papers that are hard to replicate, such as stamps, certificates, diplomas, and other important documents.[8,9] As a cellulose-based material, molded pulp often requires the incorporation of additives to enhance its physical and mechanical performance. These additives include adhesives, hardening agents, smoothing agents, antimicrobial agents, coating agents, fillers, and hydrophobic agents.[10,11] Hydrophobic additives are specifically used to modify the naturally hydrophilic properties of fibers into hydrophobic ones.[12] This is crucial to ensure that the molded pulp products exhibit water and moisture resistance, which is especially important for applications such as food packaging, electronic product packaging, or other uses that may involve liquid exposure.[13] Common hydrophobic additives used in molded pulp production include resins, wax emulsions (e.g., paraffin or natural wax), fluorochemical agents (e.g., perfluorooctanoate [PFOA] and perfluorooctane sulfonate [PFOS]), polyethylene, and AKD.[14,15] In-molded pulp production, AKD serves as a sizing agent that alters the cellulose fiber surface properties, making it more water-resistant. AKD is added to the pulp slurry before forming and drying the molded pulp product. This compound creates a protective layer that prevents water penetration into the paper or molded pulp.[16] AKD molecules are composed of two characteristic groups: (a) a long-chain alkyl group containing 12–20 carbon atoms that imparts hydrophobicity, and (b) a four-membered lactone ring that reacts with cellulose to generate β-keto ester linkages.[17,18] This reaction coats the surface of the paper, making it hydrophobic. The results indicate that AKD is highly effective as a hydrophobic additive for paper, ensuring resistance to water and moisture.[19,20]

This study aims to evaluate the effect of AKD on improving the water resistance of molded pulp made from abaca banana stem fibers. The novelty of this research lies in the application of AKD as a sizing agent in molded pulp derived from non-wood abaca fibers, which has rarely been reported. This study also analyzes the changes in the physical properties and water resistance of the material, thereby contributing to the development of biodegradable packaging based on natural fibers with improved functional performance. Furthermore, this research supports the achievement of the Sustainable Development Goals (SDGs), particularly SDG 12 (Responsible Consumption and Production), by promoting the utilization of renewable biomass resources and encouraging the development of environmentally friendly packaging materials as alternatives to conventional plastic-based products.

## EXPERIMENTAL

### Materials and Chemicals

The samples of Abaca banana stalk fibers are obtained from Peunaron District, East Aceh Regency, Aceh Province, Indonesia. The chemical materials used were Pro Analysis (pa) grade from E. Merck.

### Preparation of Abaka Banana Stalk Fibers

The abaca banana stalks are peeled and soaked in water for one week. After soaking, the stalks are washed with clean water, and the fibers are manually separated. The separated fibers are then dried for three days and are ready for further testing.

### Isolation of Cellulose from Abaca Banana Stalk Fibers

Preparation of pulp from Abaca Banana Stalk Fibers was carried out using the same method as in our previous study.[21,22]

### Preparation of Molded Pulp

The preparation of molded pulp was carried out manually. Abaca Banana Stalk Fibers pulp was dispersed in distilled water at room temperature to form a pulp slurry. Into the pulp slurry, 2% AKD was added. The pulp slurry mixture was then poured into molds. The formed pulp sheets were heated at 60–80°C. Figure-1 illustrates the schematic process of molded pulp production.

### Paper Grammage Test

Paper grammage was measured following the standard methods described in SNI 14.0439–1989 and ISO 536:2012 for the determination of grammage in paper and paperboard. The test is conducted by weighing

the paper sample using an analytical balance, and the weight is then divided by the fixed sample area of 10 x 10 cm. Each sample tested has the same area. The grammage value is calculated using Equation (1) below

$$\text{Grammage}(G) = m/A \times 10^6 \quad (1)$$

Where:
G: grammage of the paper (g/m²)
m: mass of the test sample, expressed in grams
A: area of the test sample (length × width)

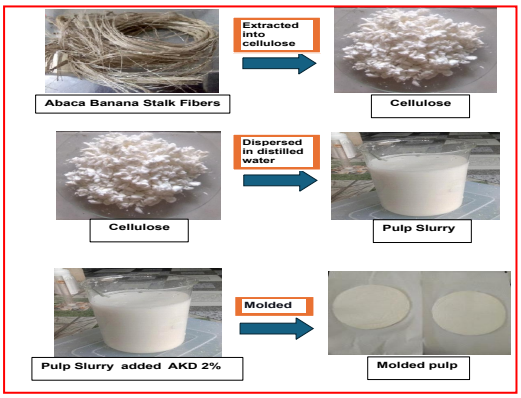


Fig.-1: Schematic Process of Molded Pulp Production

**Measurement of Paper Thickness**

The thickness of molded pulp samples was measured according to the ISO 534:2011 standard for paper and board, which outlines the procedures for determining thickness, density, and specific volume. Each sample was measured at five different points using a screw micrometre. The thickness values, initially recorded in millimeters (mm), were subsequently converted to centimeters (cm) by dividing the measurements by 10.

**Measurement of Paper Density**

The paper density is calculated by dividing the paper grammage by its thickness. The thickness measurement is performed using a digital caliper. The measurements were carried out under standard environmental conditions, with a relative humidity (RH) of 50% and a room temperature of 25 °C.

**Water Absorption Testing Method for Molded Pulp**

The water absorption testing method for molded pulp was conducted using the following procedure: First, the molded pulp was weighed using an analytical balance, and the obtained weight was recorded as the initial weight ($m_0$ ). Next, the molded pulp is immersed in distilled water with varying immersion times of 1, 2, 3, 4, 5, 6, and 7 h. After immersion, the molded pulp was removed and dried using clean tissue paper to absorb any residual water. The molded pulp samples were then dried and weighed, and the resulting value was recorded as the final weight ($m_1$ ). The water absorption capacity of the molded pulp was calculated based on Equation (2), as presented below.

$$\text{Sewelling ratio}(\%) = [(m_1-m_0)/m_0] \text{ X } 100 \quad (2)$$

Where:
$m_0$: the mass of molded pulp before immersion and expressed in grams
$m_1$: the mass of molded pulp after immersion and expressed in grams.

**Fourier Transform Infrared Spectroscopy (FTIR) Analysis**

FTIR spectra of molded pulp samples with and without AKD treatment were recorded using an Agilent Cary 630 FTIR spectrometer. Measurements were conducted in transmission mode with a spectral resolution of 4 $cm^{-1}$ across the wavenumber range of 4000–600 $cm^{-1}$.

**Sheet Morphology**

Surface morphology analysis of molded pulp samples with and without AKD incorporation was performed using a scanning electron microscope (SEM) (JEOL EO JSM-6510LA Version 1.0). The operating parameters included an accelerating voltage of 20.0 kV, probe current of 1.0000 nA, counting rate of $10^{11}$ cps, and an energy range of 0–20 keV. Prior to SEM observation, the samples were gold-coated to improve electrical conductivity and enhance image resolution.

## RESULTS AND DISCUSSION

**Abaca Banana Stalk Fibers**

Similar to other non-woody lignocellulosic residues, including rice straw, corn cobs, and sugarcane bagasse, Abaca banana stalk fiber is a renewable organic biomass that shows considerable potential as a sustainable raw material for molded pulp manufacturing.[23,24,25] Hydrolysis and bleaching reactions are used to extract cellulose from other compounds that bind it, such as hemicellulose, starch, wax, fats, and lignin found in organic biomass.[26] Generally, molded pulp produced from non-woody organic biomass has low mechanical strength, which is why the addition of additives, such as AKD, is necessary. AKD is a hydrophobic compound that interacts with cellulose fibers, forming a protective layer that reduces water absorption.[18,27] The results of this study show that the modification of cellulose using AKD compounds can enhance its physical properties, including paper grammage, paper thickness, paper density, hydrophobicity, and morphology. The use of Abaca banana stalk fiber as a raw material for food packaging products aligns with SDG-12: Responsible Consumption and Production.

**Paper Grammage**

Paper grammage is measured in grams per square meter (g/m²). The results of the paper grammage measurements before and after the addition of AKD are shown in Table-1. The grammage values of the paper before and after the addition of AKD are 615.48 g/m² and 6.27 g/m², respectively. The change in grammage values does not show a significant difference. Although the addition of AKD does not have a major impact on the increase in grammage, several studies explain that AKD is an additive compound that functions to improve the hydrophobic properties of molded pulp. A study conducted by Bildik *et al.* explains that adding AKD concentrations ranging from 0.1% to 5% can increase the contact angle of the paper from 19.080° to 116.170°. This indicates that although AKD does not significantly affect the change in grammage, this compound is effective in improving the hydrophobic properties of the paper.[28]

Table-1: Grammage Values of Molded Pulp Before and after the Addition of AKD

| Treatment | Measurement | | Mass (g) | Area ($cm^2$) | Grammage ($g/m^2$) | Average ($g/m^2$) |
|---|---|---|---|---|---|---|
| Before addition | 1 | | 6.1076 | 100 | 610.76 | |
| | 2 | | 6.1205 | 100 | 612.05 | 615.48 |
| | 3 | | 6.2363 | 100 | 623.63 | |
| After addition | 1 | | 6.1230 | 100 | 612.30 | 633.27 |
| | 2 | | 6.2870 | 100 | 628.70 | |
| | 3 | | 6.5882 | 100 | 658.82 | |

**Paper Thickness Measurement**

The thickness of the molded pulp was determined at five measurement points, namely right-top, right-bottom, center, left-top, and left-bottom. The samples were prepared with dimensions of 10 cm × 10 cm. The thickness of the molded pulp from three measurements is shown in Table-2. The paper thickness was measured at five measurement points (right-top, right-bottom, center, left-top, and left-bottom). The thickness measurements before the addition of AKD at the five measurement points showed a variation of 0.02 – 0.45 cm. This variation was higher compared to the measurements taken after the addition of AKD, which showed a difference of 0.02 – 0.11 cm. This indicates that the addition of AKD affects the distribution of fibers in the molded pulp, making it more uniform, as AKD helps reduce fiber gaps.[27]

Table-2: Paper Thickness Values before and after the Addition of AKD

| Treatment | Measurement | Thickness Values | | | | | |
|---|---|---|---|---|---|---|---|
| | | Right-Top (mm) | Right-Bottom (mm) | Left -Top (mm) | Left-Bottom (mm) | Center (mm) | Average (mm) |
| Before addition | 1 | 1.42 | 1.55 | 1.60 | 1.56 | 1.40 | 1.51 |
| | 2 | 1.85 | 1.52 | 1.60 | 1.20 | 1.50 | 1.53 |
| | 3 | 1.75 | 1.51 | 1.60 | 1.34 | 1.52 | 1.54 |
| After addition | 1 | 1.48 | 1.53 | 1.57 | 1.58 | 1.58 | 1.55 |
| | 2 | 1.50 | 1.54 | 1.58 | 1.57 | 1.58 | 1.55 |
| | 3 | 1.56 | 1.59 | 1.54 | 1.59 | 1.56 | 1.57 |

**Paper Density**

Paper density indicates how compact and thick the paper. Paper with high density is generally sturdier and has a smoother surface, whereas paper with low density tends to be lighter and more porous.[29] The paper density measurements before and after the addition of AKD are shown in Table-3. The paper density measurements show that the density of molded pulp after the addition of AKD was higher compared to before the addition of AKD. This was because the grammage and thickness of the molded pulp increased after the addition of AKD, although not significant. The addition of AKD can enhance density by binding the fibers more tightly, reducing porosity, and increasing hydrophobic properties, which contribute to the strength and stability of the paper.[30]

Table-3: Paper Density Value before and after the Addition of AKD

| Treatment | Measurement | Grammage ($g/m^2$) | Thickness (m) | Paper Density ($g/m^3$) |
|---|---|---|---|---|
| Before addition | 1 | 610,76 | $1,51 \times 10^{-3}$ | $404,5 \times 10^3$ |
| | 2 | 612,05 | $1,53 \times 10^{-3}$ | $400 \times 10^3$ |
| | 3 | 623,63 | $1,54 \times 10^{-3}$ | $404.9 \times 10^3$ |
| After addition | 1 | 612,30 | $1,55 \times 10^{-3}$ | $395 \times 10^3$ |
| | 2 | 628,70 | $1,55 \times 10^{-3}$ | $405,6 \times 10^3$ |
| | 3 | 658,82 | $1,57 \times 10^{-3}$ | $419.6 \times 10^3$ |

**Water Uptake Capability**

The AKD added acts as a hydrophobic agent[31], coating the surface of the fibers in the pulp and forming a water-resistant layer. The presence of this layer significantly reduces the interaction between cellulose fibers and water molecules. As a result, the molded pulp containing AKD was more resistant to water absorption, and the fiber structure absorbed less moisture. The water absorption capacity of molded pulp before and after the addition of AKD is presented in Table-4.

Table-4: Water Absorption Capacity before and after the Addition of AKD

| Treatment | | | |
|---|---|---|---|
| Before addition | | After addition | |
| Soaking time (h) | Water Absorption Capacity (%) | Soaking time (h) | Water Absorption Capacity (%) |
| 1 | 12,33 | 1 | 10,34 |
| 2 | 20,07 | 2 | 16.44 |
| 3 | 21,08 | 3 | 16,89 |
| 4 | 22,10 | 4 | 18,67 |
| 5 | 27,57 | 5 | 18,76 |
| 6 | 30,33 | 6 | 19,87 |
| 7 | 32,89 | 7 | 29,21 |

**FTIR Analysis**

The FTIR analysis of the molded pulp, both before and after the addition of AKD, is presented in Fig.-2. The results show that before the addition of AKD, the hydroxyl (–OH) groups' absorption in the region of 3311 $cm^{-1}$ exhibited a broad and sharp absorption peak. However, after the addition of AKD, the –OH

absorption peak at 3287.5 $cm^{-1}$ showed a decrease in intensity, with a broader and less sharp peak. This decrease suggests that most of the -OH groups have reacted with AKD, reducing the hydrophilic groups in cellulose and increasing the hydrophobic groups in the form of ester groups. Additionally, the aliphatic C–H absorption before the addition of AKD at 2892.4 $cm^{-1}$ appeared as a single weak peak, whereas after the addition of AKD, the aliphatic C–H absorption at 2914.8 $cm^{-1}$ displayed a weak double peak. The increased intensity of absorption around 2800–2900 $cm^{-1}$.[27,32] The characteristic aliphatic C–H absorption indicates the contribution of long alkyl chains from AKD.

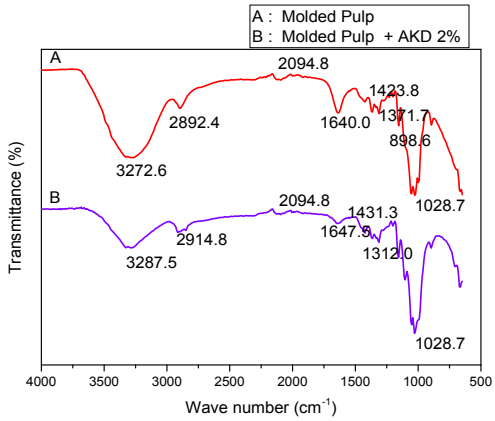


Fig.-2: Comparison of FTIR Spectra before and after AKD Addition

**Surface Morphology**

Figure-3 depicts the changes in the surface morphology of molded pulp resulting from the addition of AKD. In Fig.-3A, showing the surface of the molded pulp prior to AKD treatment, numerous small pores are visible across the surface, indicating a rougher texture. These pores are crucial as they act as sites for water absorption, which affects the pulp's ability to resist moisture. Following the addition of AKD, as illustrated in Fig.-3B, the fiber surface becomes noticeably smoother, accompanied by a significant decrease in the number of small pores. This change in surface structure indicates a decrease in the pulp's capacity to absorb water, which is a direct result of the AKD's influence on the fibers. The reduced pore volume suggests that the hydrophobic properties of the fibers have improved, making the molded pulp more resistant to water. This enhancement is beneficial for applications requiring moisture resistance, such as packaging materials.

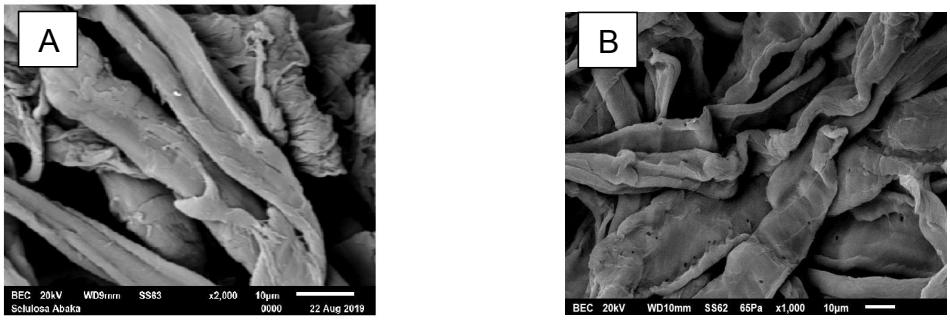


Fig.-3: Scanning Electron Micrograph at 1,000x Magnification of Molded Pulp: A dan B show the Condition before and after the Addition of AKD

## CONCLUSION

The research shows that adding AKD significantly affects the physical properties of molded pulp, including grammage, density, thickness, water absorption, functional groups, and surface morphology. AKD acts as a filler, improving the pulp's characteristics by filling the pores of the fibers, which results in a smoother surface. This pore closure reduces water absorption due to enhanced hydrophobicity, making AKD-modified pulp ideal for applications like food packaging that require water resistance and a smooth surface. The use of Abaca banana stalk fibers as a raw material for food packaging products aligns with SDG-12: Responsible Consumption and Production.

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## CONFLICT OF INTERESTS

The authors declare that they have no conflict of interest.

## AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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