

# CHARACTERIZATION OF EDIBLE CANNA (*CANNA EDULIS* KER) STARCH-BASED BIOCOSMOSITES REINFORCED WITH SORGHUM (*SORGHUM BICOLOR* (L.) MOENCH) STALKS FIBER

Achmad Nandang ROZİAFANTO\*<sup>1</sup>  
İmas SOLİHAT<sup>1</sup>

<sup>1</sup>Food Nanotechnology Department, Politeknik AKA Bogor, Bogor, Indonesia

Gönderilme Tarihi / Received: 20.08.2024  
Kabul Tarihi / Accepted: 08.06.2026

**ABSTRACT:** This study investigates biocomposite films made from edible canna starch (CS) and alkali-treated sorghum stalk fiber (SF) as a reinforcing agent. The addition of treated SF (0–10 wt.%) significantly improved tensile strength, Young's modulus, and water resistance. Alkali treatment removed lignin, hemicellulose, wax, and oils from the fiber surface, increasing hydrophilicity and enhancing compatibility with the starch matrix. FTIR and SEM analyses confirmed structural modifications and better fiber dispersion. The resulting films exhibited strong hydrogen bonding and reduced water absorption due to improved interfacial adhesion. This work introduces a novel combination of high-amylose canna starch and underutilized sorghum fiber, contributing new insight into biodegradable packaging materials. The study not only valorizes agricultural waste but also supports the development of sustainable biocomposites with promising application in eco-friendly packaging. Future research should explore large-scale processing, long-term stability, and performance optimization to advance commercial viability.

**Keywords:** Biocomposites, Canna edulis Ker, Starch, Sorghum, Fiber, Alkali treatment

## SORGHUM (*SORGHUM BICOLOR* (L.) MOENCH) SAP ELYAFI İLE GÜÇLENDİRİLMİŞ YENİLEBİLİR KANNA (*CANNA EDULIS* KER) NIŞASTA BAZLI BİYOKOMPOZİTLERİN KARAKTERİZASYONU

**ÖZ:** Bu çalışma, takviye maddesi olarak sorgum sapı lifinin (SF) eklenmesiyle matris malzemesi olarak yenilebilir kanna nişastasından (CS) oluşan biyokompozitleri araştırmaktadır. Sonuçlar, alkali ile muamele edilmiş SF konsantrasyonunun sistematik olarak ağırlıkça %0'dan %10'a yükseltilmesinin, biyokompozit filmlerin hem gerilme mukavemetini hem de Young modülünü önemli ölçüde artırdığını ortaya koyuyor; bu da matris ve elyaflar arasındaki etkili stres iletimini gösteriyor. SF'nin alkali muamelesi, hemiselüloz, lignin ve balmumu ve yağlar gibi kirlenici maddeleri uzaklaştırarak selüloz maruziyetini artırdı ve hidroksil ve karboksil gruplarından dolayı hidrofilitenin artmasına yol açtı. Bu artırılmış hidrofilitiklik, işlenmiş SF ve CS arasındaki uyumluluğu geliştirerek üstün su direnci kapasitesine sahip biyokompozit filmler elde edilmesini sağladı. Biyokompozitler, çoğunlukla kimyasal yapılarının uyumluluğuna ve benzerliğine atfedilen, lifler ve matris arasında güçlü moleküller arası hidrojen bağları sergiler. Genel olarak, elde edilen biyokompozit filmler olumlu mekanik, fiziksel ve suya dayanıklılık özellikleri sergiler. Sonuç olarak bu çalışma, sorgum lifi ile güçlendirilmiş yenilebilir kanna nişastasının ambalaj uygulamalarında kullanılması potansiyelinin altını çizmektedir. Gelecekteki çalışmalar, biyoambalaj malzemelerinin daha da geliştirilmesi çok önemli olduğundan, fiber yüklemelerinin biyokompozit filmlerin fiziksel ve mekanik özellikleri üzerindeki etkisini derinlemesine araştırılabilir.

**Anahtar Kelimeler:** Biyokompozitler, Canna edulis Ker, Nişasta, Sorgum, Lif, Alkali işlem

\*Sorumlu Yazarlar/Corresponding Authors: anandangr@yahoo.com

DOI: <https://doi.org/10.7216/teksmuh.1536012>

[www.tekstilmuhendis.org.tr](http://www.tekstilmuhendis.org.tr)

## 1. INTRODUCTION

Fossil fuel-based polymers are widely used in the packaging industry, which has raised long concerns for the global ecosystem. This study aims to address the environmental concerns associated with the widespread use of fossil fuel-based polymers in the packaging industry. Extensive quantities of ecologically detrimental plastic trash have prompted many researchers to investigate polymers derived from natural, renewable, sustainable, and biodegradable substances. Several previous studies have explored biopolymers as possible alternatives to fossil-based plastics in order to address the increasing environmental concerns associated with non-biodegradable plastics [1-3]. Consequently, various thermoplastic composites have been used in the production of environmentally friendly packaging items. Based on sustainability point of view, this approach facilitates the emergence of environmentally favorable materials, leading to the development of biocomposites. Basically, biocomposites are the types of material created by reinforcing a matrix with natural fibers, which used here as the fillers. The use of natural fibers derived from agricultural leftovers as eco-friendly filler materials has gained significant popularity in the manufacturing industry. This is mostly owing to their cost-effectiveness, low density, and ability to minimize tool wear [4, 5]. The biocomposite production generally utilizes natural fibers derived from various sources, such as hardwood (*Carpinus betulus*, *Ceratonia siliqua*), flax, hemp, hemlock, jute, nettle, sisal, sugarcane bagasse, oil palm empty fruit bunches, sorghum, and date palm [6-14].

Biocomposites has been highly favored in material engineering owing to its advantageous properties, such as their lightweight nature, energy efficiency, biodegradability, sustainability, practicality, and eco-friendliness [15-17]. As one of the most abundant natural fibers, lignocellulosic fibers offer several benefits as reinforcing agents in biocomposites production, but they also possess certain flaws. The water-attracting properties and strong bonds of cellulosic fibers hinder their interaction with the matrix material, resulting in inadequate connection and poor mechanical strength [18]. Consequently, biocomposites with these properties often exhibit restricted dimensional stability when it comes to contact with water. For this reason, several techniques had been carried out to enhance the compatibility between lignocellulosic fibers and the matrix [19]. Besides, surface changes are also frequently employed to improve the performance of cellulosic fibers in order to facilitate stronger adhesion between the fibers and the matrix, such as starch [20, 21].

Edible canna (*Canna edulis* Ker), a plant belonging to the Cannaceae family, has tubers that are rich in starch, fiber, and carbohydrates content [22]. The cultivation of this crop is primarily concentrated in South East Asia (Indonesia, Vietnam, Thailand), South America, and China [23]. Edible canna starch, in addition to its high content (ranging from 70 to 80%), is also characterized by several exceptional properties, namely it is highly digestible and able to form gel-like structures. Moreover, edible canna starch also possesses the amylose content of up to

35%, the highest among that of other starches-containing crops, such as cassava, wheat, yam, sweet potato, potato, and taro, all of which only contain amylose ranging from 10.1 to 32% [23-25]. Several previous studies had demonstrated that the amount of amylose in starch has a notable effect on its ability to form films. Starch with higher amylose content can lead to stronger and more rigid films due to the hydrogen bonds that formed between the linear chains of amylose [26-28]. Therefore, edible canna starch, with a significant amount of amylose, is considered highly potential for biopolymer composites production.

The utilization of starch generally can produce more robust biopolymer films compared to those of made from other alternative natural substances [29, 30]. However, the resulting biopolymer films often exhibit certain drawbacks, such as low tensile strength and lack of water resistance [31]. To overcome these constraints, it is particularly necessary to improve several mechanical and physical properties of edible canna starch. The combination of starch and natural fiber is intended to produce superior outcomes. Sorghum (*Sorghum bicolor* (L.) Moench) is considered the fourth most significant cereal crop worldwide, behind maize, wheat, and rice. By weight, one individual of sorghum plant consists of 15% grains, 10% leaves, and 75% stalk [32]. Sorghum stalk, similar to that of sugarcane, contain a substantial amount of glucose, which has great potential as a substitute raw material for bioethanol production. Nevertheless, the bioethanol production process often generates significant quantity of bagasse byproduct. Sorghum bagasse has a fibrous nature and possesses a significantly greater cellulose content of around 49%, higher than that of sugarcane and rice straw [4, 33, 34]. For this reason, sorghum bagasse, with its abundant cellulose content, might be useful as a reinforcing agent to improve the properties and crystalline structure of biopolymer composites [6, 35].

This study examines the utilization of glycerol as a substance that increases the flexibility of a material, known as a plasticizer, and sorghum bagasse fiber (SF) as a reinforcing agent for edible canna starch (CS), which serves as a matrix in biocomposites. In a larger context, this study proposes that adding sorghum bagasse fiber as a filler component in edible canna starch-based biocomposites can increase the worth of the product and strengthen the capacity of the composites to function as fully environmentally friendly packaging materials. Currently, there have been no existing studies published on the use of sorghum bagasse fiber to reinforce biopolymer composites made from edible canna starch. Therefore, this study aims to reinforce edible canna starch with sorghum bagasse fiber to optimize the mechanical, physical, and water resistance properties of the biopolymer composite film.

## 2. MATERIALS AND METHODS

### 2.1. Extraction of Sorghum Fiber

The stalk of sorghum was acquired from a field plant breeding program at the Center for Research and Technology of Isotopes and Radiation Application, which is part of the National

Research and Innovation Agency of Indonesia. The SF had an ethanol:benzene extractive content of  $7.02 \pm 0.06\%$ , a lignin content of  $23.95 \pm 0.27\%$ , a holocellulose content of  $64.22 \pm 1.77\%$  and an ash content of  $4.35 \pm 0.38\%$  where ethanol:benzene extractive was analyzed according to the TAPPI T 204 cm-97 method, lignin content by the NREL LAP 003 Standard method, holocellulose content by Wise method (1946) and ash content by the TAPPI T 211 om-02 method. The SF were broken into pieces at around 4 cm in length, then crushed until they could pass through screens with a mesh size of 40–60. Subsequently, the sorghum fibers were rinsed using distilled water, then exposed to sunshine in a shelter covered with plastic for 5 days to facilitate drying. The dehydrated SF samples were then treated with NaOH solution at two different concentrations, namely 1 and 5% (w/w), under agitation for 3 hours at a temperature ranging from 70 to 90°C, as described in Ismojo, et al. [36]. The resulting pulp was rendered at chemically neutral condition by rinsing it using distilled water until it reached a pH of 7. Then, the pulp was filtered and dried at room temperature. Finally, the chemical composition of the resulting SF samples was analyzed using FTIR.

## 2.2. Preparation of Biocomposites

The schematic of biocomposite preparation is described in Figure 1. The edible canna starch (CS) powder (commercial food grade, 27% amylose, <10% water content) used in this study was purchased from a local market under the brand name “Lingkar Organik”. Biocomposites were fabricated using the traditional solution casting technique. A film-forming combination was prepared by dispersing 10 gr of CS in 180 ml of distilled water. Then, 3 gr of glycerol (by weight, based on dry starch) was added to the mixture. Untreated and treated SF were added into the mixture as reinforcement agent, with concentration ranging from 0 to 10 wt.%. The solution was heated at a temperature of 80°C while being stirred slowly for 15 minutes. Prior to casting, the liquid used to make the biocomposite was chilled and placed in a vacuum desiccator to prevent the formation of air bubbles. Subsequently, 50 gr of each solution was poured into petri plates measuring diameter of 120 mm. The cast plates were placed in a temperature-controlled oven with airflow at 50°C for 24 hours in order to remove any moisture. Subsequently, the desiccated samples were subjected to a 48-hour conditioning period at a temperature of 25°C. Following this, the samples were separated from the plates. Prior to characterization, all film samples were placed in a desiccator for one week.

## 2.3. Characterization of Biocomposites

### 2.3.1. The Thickness of Biocomposite Films

The thickness of the biopolymer composite films was measured using a micrometer (Mitutoyo-co, Kawasaki, Japan) with a precision of 0.01 mm. This technique based on the ASTM method F2251 was employed to determine the thickness of each replication of the film sample, then the average value was calculated to obtain the real thickness.

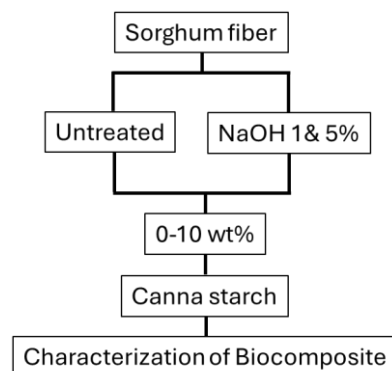


Figure 1. Process flow chart of biocomposite fabrication.

### 2.3.2. Water Contents (WC)

The water content of three replications of each sample was measured using the ASTM D664-07 standard. The samples' initial weight ( $W_i$ ) was measured, then they were dried at a temperature of 105°C for 24 hours. After the drying process, the samples' final weight ( $W_f$ ) was measured. Equation (1) was employed to calculate the water content of each film sample.

$$W_C (\%) = \frac{W_i - W_f}{W_i} \times 100\% \quad (1)$$

### 2.3.3. Water Absorption (WA)

Water absorption (WA) was measured using the ASTM D 570-98 standard. First, the samples were dehydrated at a temperature of 50°C for 24 hours. Then, they were placed in a desiccator in order to cool down and determine their stable weight. The samples' initial weight ( $M_i$ ) was subsequently measured and submerged in distilled water at a temperature of  $23 \pm 2^\circ\text{C}$ . During a specific time period, the submerged samples were cleaned using a dry cloth, then the samples' final weight ( $M_f$ ) was measured. As seen in Equation (2), both the final and initial weight values were included to calculate the water absorption of each film sample.

$$W_A (\%) = \frac{M_f - M_i}{M_i} \times 100\% \quad (2)$$

### 2.3.4. Fourier Transform Infrared (FTIR) Spectroscopy

The Fourier transform infrared spectra of the samples were determined using an Agilent ATR Carry 6300 spectrometer to analyze the presence of functional groups in the untreated and treated SF samples. Each film sample was analyzed using 32 scans in the wavenumber range of  $4000\text{--}650\text{ cm}^{-1}$  with a spectral resolution of  $2\text{ cm}^{-1}$ .

### 2.3.5. Thermal Properties

A Differential Scanning Calorimetry (DSC) Setline Setaram analysis was conducted on the samples, where they were heated from 35°C to 200°C at a rate of 10°C per minute. The sample

was conditioned under controlled parameters of 60% relative humidity (RH) and a temperature of 25°C.

### 2.3.6. Mechanical Properties

The mechanical properties of each film sample were determined at room temperature under ambient conditions using a 5 kN Testometric UTM, based on the ASTM D882-02 standard. The sample, measuring  $70 \times 10 \text{ mm}^2$ , was securely positioned between the tensile clamps. At first, the sample's gauge length was set at 30 mm and the machine's crosshead speed was set at 2 mm/min. The tensile strength, tensile modulus, and elongation values of 10 replications of each sample were measured. The mechanical qualities were then assessed by calculating the average of the data obtained.

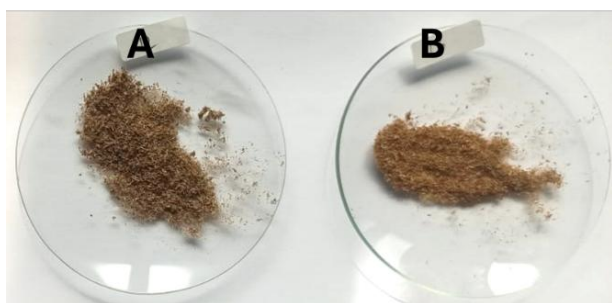
### 2.3.7. Morphological Analysis

A field emission scanning electron microscope (FESEM) FEI Inspect F50 was utilized to analyze the morphological characteristics of the biocomposite film. To prevent unwanted charging, the specimens were entirely coated with a thin layer of gold using the sputtering technique. The FESEM analysis was conducted at an acceleration voltage of 3 kV.

## 3. RESULTS AND DISCUSSION

### 3.1. Chemical Composition of Sorghum Fiber

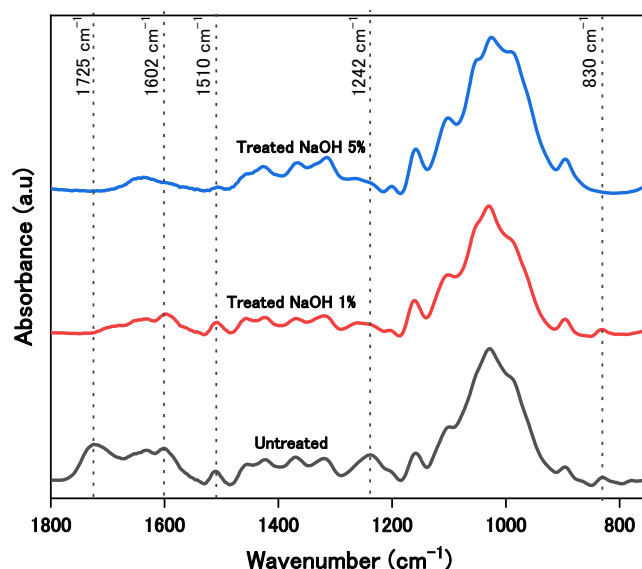
The pictorial view of SF fiber untreated and after treated with NaOH are shown in Fig. 2. The alkali treatment in this study aims to eliminate a significant portion of the lignin, which, due to its non-crystalline structure, decreases the tensile strength of sorghum fiber and other lignocellulosic biomass in general. Lignin naturally coats cellulose fibrils, serving as a protective barrier against chemical reactions. The interwoven arrangement of lignin and cellulose produces stable covalent cross-links, thereby ensuring a strong and lasting connection between both polymers [2, 6, 9, 19].



**Figure 2.** Pictorial view of SF fiber untreated and after treated with NaOH

The FTIR spectra of both untreated and treated SF are depicted in Figure 3. The spectra are displayed as the absorbance (%) plotted against the wavenumber ( $\text{cm}^{-1}$ ) in the range of  $1800 \text{ cm}^{-1}$ – $750 \text{ cm}^{-1}$ . The untreated SF exhibit a prominent peak at  $1725 \text{ cm}^{-1}$ , which can be attributed to the stretching of carbonyl groups

in carboxylic and aldehyde functional groups present in hemicellulose [37, 38]. The peaks seen at  $1602 \text{ cm}^{-1}$  and  $1514 \text{ cm}^{-1}$  can be attributed to the existence of the C–O bonds in lignin and hemicellulose, respectively. The peak at  $1242 \text{ cm}^{-1}$  is indicative of the presence of the C–C aromatic ring in lignin [39–41]. The observed peaks around  $830 \text{ cm}^{-1}$  likely correlate to the C–H out-of-plane deformation on the benzene ring in lignin, as suggested by [38, 42]. Lastly, the spectra of the treated SF at  $1725 \text{ cm}^{-1}$ ,  $1602 \text{ cm}^{-1}$ ,  $1514 \text{ cm}^{-1}$ , and  $830 \text{ cm}^{-1}$  vanished, suggesting a reduction in the lignin and hemicellulose content in sorghum fiber. The alkali treatment resulted in the elimination of these peaks, suggesting that the lignin aromatic chain was destroyed by the alkaline solution [14, 42, 43]. From our previous study, Ismojo et. al. [32], the morphological by SEM analysis indicates that the alkali treatment process effectively enhances the fibre's structure by eliminating non-cellulosic components such as hemicellulose, lignin, wax and pectin. As a result, the treated fibre exhibits a smoother and brighter appearance, with improved separation of individual fibres. This modification can potentially enhance the fibre's mechanical properties, adhesion in composite materials, and overall performance in various applications. The hydrophilicity of alkali-treated SF was also enhanced due to the presence of hydroxyl and carboxyl groups in cellulose, leading to increased exposure [14]. In turns, this enhanced hydrophilicity improves the compatibility between the treated SF and CS.



**Figure 3.** FTIR spectra of untreated and treated SF

### 3.2. Thicknesses of Biocomposite Film

As seen in Figure 4, the thickness of biopolymer composite film increased considerably when the SF concentration was added. The dry mass per unit area of the film-forming solutions was precisely regulated during the casting process, leading to varying thickness values of both control and biocomposite films. The biocomposite

film sample containing SF concentration of 10 wt.%, which was treated with 1% of NaOH, exhibit the greatest thickness of 580  $\mu\text{m}$  compared to that of all other samples. Both the inclusion of treated and untreated SF increased the thickness of the biocomposite in a similar linear trend. This was resulted mainly due to the intermolecular interaction between sorghum fiber as the filler and edible canna starch as the matrix. Increasing the concentration of SF leads to more porous structure and lower relative density of the material tested, resulting in thicker and coarser biopolymer films. Several previous studies also reported similar findings on the effects of fiber addition on the thickness and density of biocomposite film [44-46].

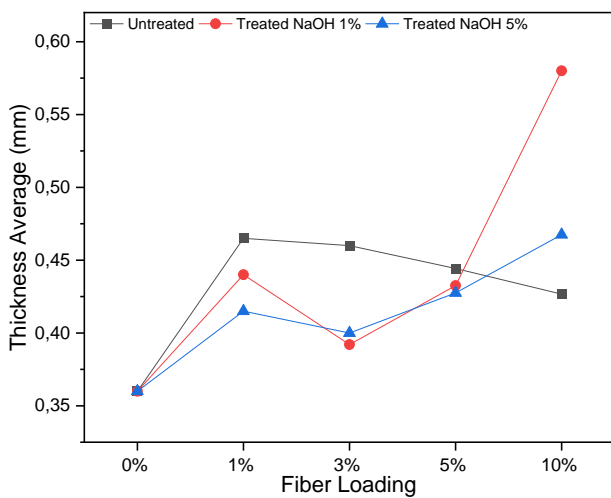


Figure 4. Thickness of biocomposite films

### 3.3. Water Contents

Despite being hydrophilic, the treated SF reduced the moisture retention in biocomposite films, as illustrated in Figure 5. The water contents of biocomposite films containing treated SF significantly decreased from 12% to 8% when the fiber loadings were increased from 1% to 10%. On the other hand, at the same condition, the water content of biocomposite films containing untreated SF significantly increased from 9% to 12%. It is also revealed that at SF concentration of 10 wt.%, the treated SF films exhibit much lower water content compared to that of untreated SF films. The little augmentation in the water content of biocomposite films is mainly thought to be due to the relatively lower water content of SF compared to that of CS. The enhancement is ascribed to the hydrophilic characteristic of CS, which is a polymeric compound with many hydroxyl groups, namely three hydroxyl groups per monomer. Furthermore, there was a modest decrease in the moisture content of biocomposite films when the concentration of SF was increased from 1% to 10 wt.%. As a result, the hydrophilicity of the biocomposite films reduced due to the addition of SF concentration. This phenomenon is most likely resulted by robust hydrogen bonds formed between the fibers and the matrix, which hinders the incorporation of water into the starch molecules [47, 48].

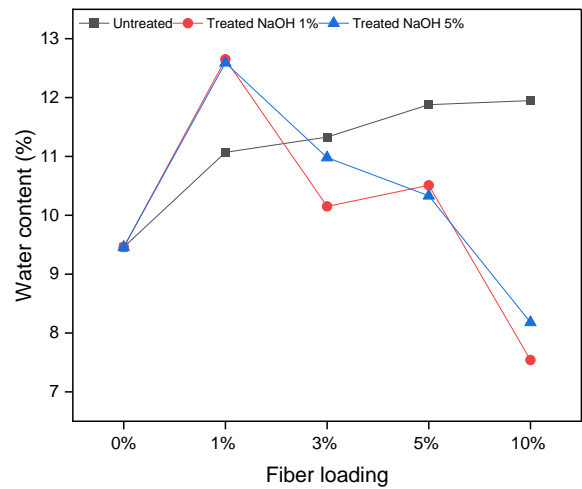


Figure 5. Water content of biocomposite films

### 3.4. Water Absorption

In the packaging industry, it is considered necessary to have films that are highly resistant to water in order to protect and maintain the quality of food products. To achieve this, it is important to assess the water absorption characteristics of biopolymer-reinforced natural fiber composites. These kinds of material tend to easily absorb moisture from the surrounding environment or other external sources, and this will affect their usage performance. For this reason, water absorption analysis is frequently performed to determine the quantity of water absorbed by a composite material during certain period of time. Water absorption capacity of a composite material is not only affected by its fiber content, temperature, and orientation [49], but also by several other factors, such as surface protection, diffusivity, and exposed surface area of the material. Figure 6 presents the results of the water absorption analysis on CS-based biocomposite films when exposed to distilled water for different durations (30 min, 60 min, and 90 min). As per Fick's law, the rate of weight increase caused by water absorption stabilizes after one hour of immersion. The ability of biocomposite films to absorb water varies each other depending on the pretreatment and fiber loading. The hydrophilicity of the biocomposite films can be decreased by pretreating the fiber and increasing the fiber loadings [50, 51]. The control CS film and untreated SF biocomposite, largely known for their hydrophilic nature, originally demonstrated significant water absorption. Nevertheless, the inclusion of treated SF substantially decreased the water absorption, resulting in biocomposite films that exhibit greater resistance to water absorption compared to that of control and untreated SF films. After 30 minutes, the biocomposite films containing untreated SF produced the highest rates of water absorption, while the lowest rates were resulted by the films containing treated SF. Similar trends were also resulted in the rates of water absorption at both the 60 minute and 90-minute intervals. The robust interface between the treated SF and the matrix, together with hydrogen bonding interactions inside the biocomposite film, enhances its ability to resist water absorption.

These results indicate that increasing the amount of treated SF in biocomposites can improve their ability to resist water [52, 53]. This also makes CS-based biocomposite reinforced with SF

acceptable for wide use in packaging applications, especially when it comes to contact with high humidity environment.

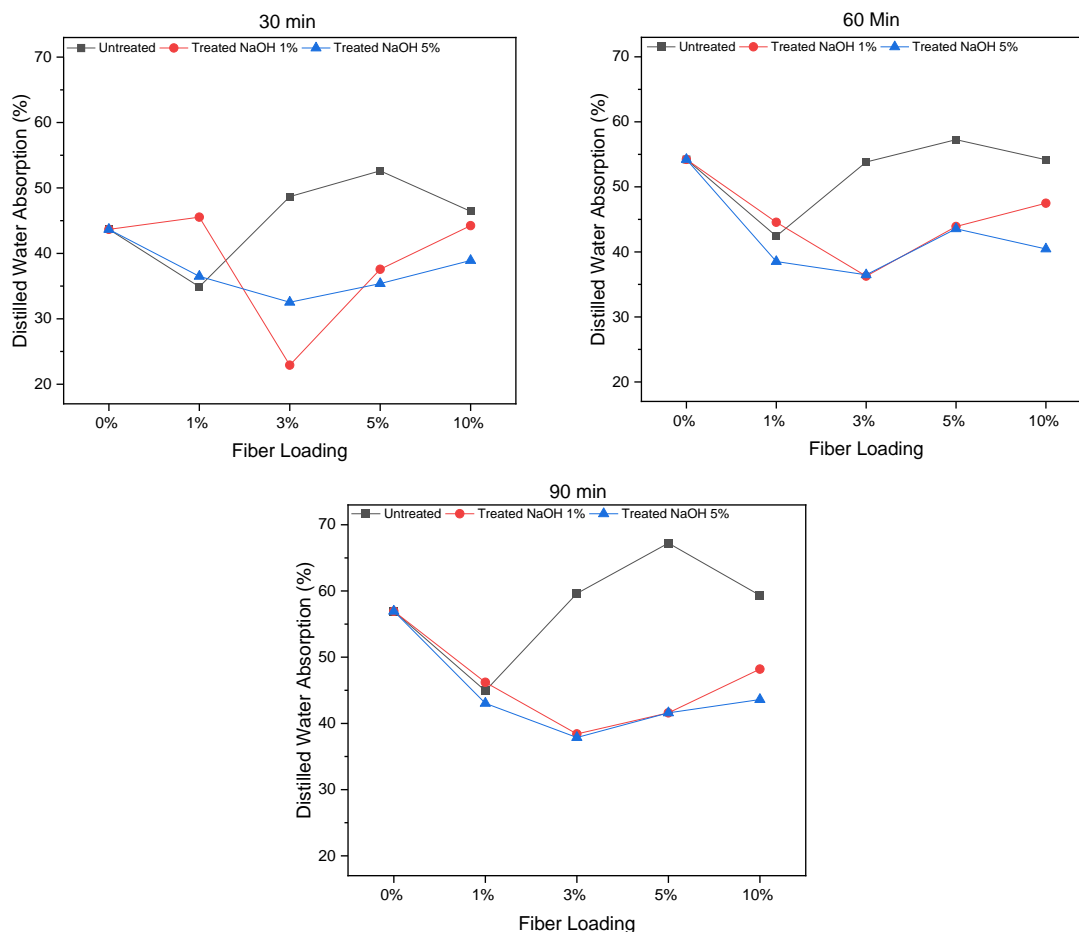


Figure 6. Water absorption capacity of biocomposite films

### 3.5. Thermal Properties

In the Differential Scanning Calorimetry (DSC) analysis of starch films, the glass transition temperature ( $T_g$ ) is a crucial factor influencing thermophysical transitions. As noted by Tarique et al. [54],  $T_g$  represents the temperature at which bound amylose and amylopectin begin to loosen, allowing significant movement of the starch molecules. Figure 7 presents the DSC thermogram of *Canna edulis* biocomposite before and after the incorporation of SF treated fiber. Prior to the addition of SF treated fiber, the thermogram exhibits two endothermic peaks at approximately 82,89°C and 118,12°C. However, following the incorporation of SF treated fiber, these peaks merge into a single endothermic peak at around 80,06°C. The addition of natural fibers can alter the thermal stability and heat transfer characteristics of the biocomposite, leading to changes in the DSC peaks [55, 56]. The integration of natural fibers into the biofilm matrix can improve heat transfer due to better thermal conductivity of the fibers compared to the biofilm alone. This can result in a more uniform heat distribution and a single, more

pronounced endothermic peak [57]. The addition of natural fibers can induce chemical and structural changes in the biofilm matrix. These changes can affect the thermal transitions observed in the DSC thermogram, such as melting or recrystallization processes [56, 58]. The fibers may act as nucleating agents, promoting more uniform melting behavior and leading to a single endothermic peak [56].

### 3.6. Mechanical Properties

The mechanical properties of biocomposite can be improved by adding natural fiber as a reinforcing agent in the polymer matrix. Figure 8 illustrates the impact of SF addition on the mechanical characteristics of CS-based biocomposite films. Tensile testing was performed to assess the tensile strength, Young's modulus, and elongation at the point of fracture. The results reveal that treated SF biocomposite films exhibit higher tensile strength and Young's modulus when the concentration of treated SF was increased, as compared to those of control and untreated SF films. This enhancement can be attributed to the increased compatibility resulting from the processing of SF, also due to the

structural resemblance of cellulose between SF and CS. In addition, SF impeded the mobility of starch molecules and improved the interaction between CS and SF, resulting in more effective transmission of stress [59, 60]. Nevertheless, the addition of SF concentration generated a contrary impact on the elongation at break of CS-based biocomposites, namely it diminish the capacity of the biocomposites to undergo deformation and stretching. In terms of biocomposite film reinforcement, the addition of SF concentration here from 0% to

10 wt.% increased the thickness of the film, leading to a reduction in elongation at break, as depicted in Figure 8. The decrease in film elongation as the fiber loadings were increased can be attributed to the formation of additional hydrogen bonds between SF and CS, resulting in stronger intermolecular interactions. These interactions impede the movement of the polymer chains, resulting in enhanced stiffness and decreased pliability of the biocomposite films [61-63].

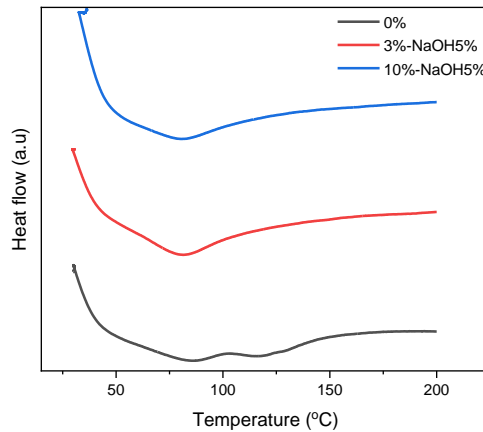


Figure 7. DSC test results for the biocomposite films

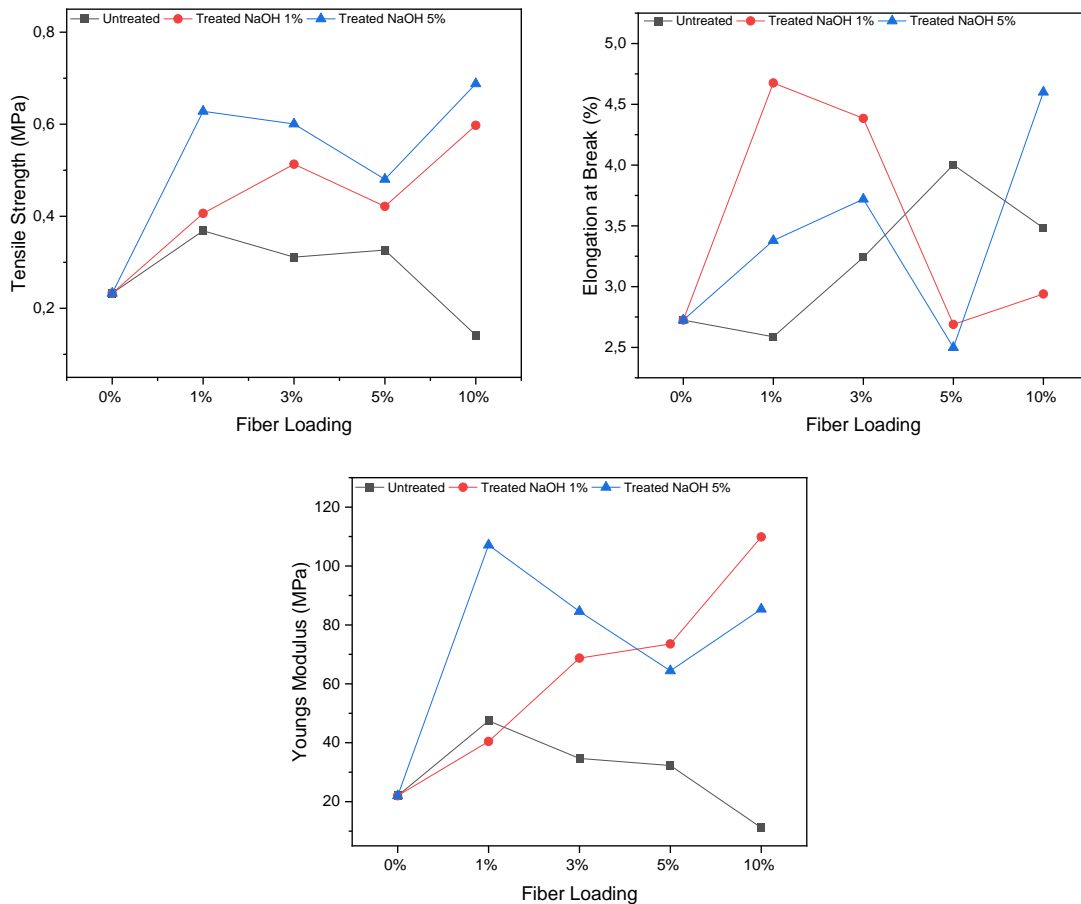
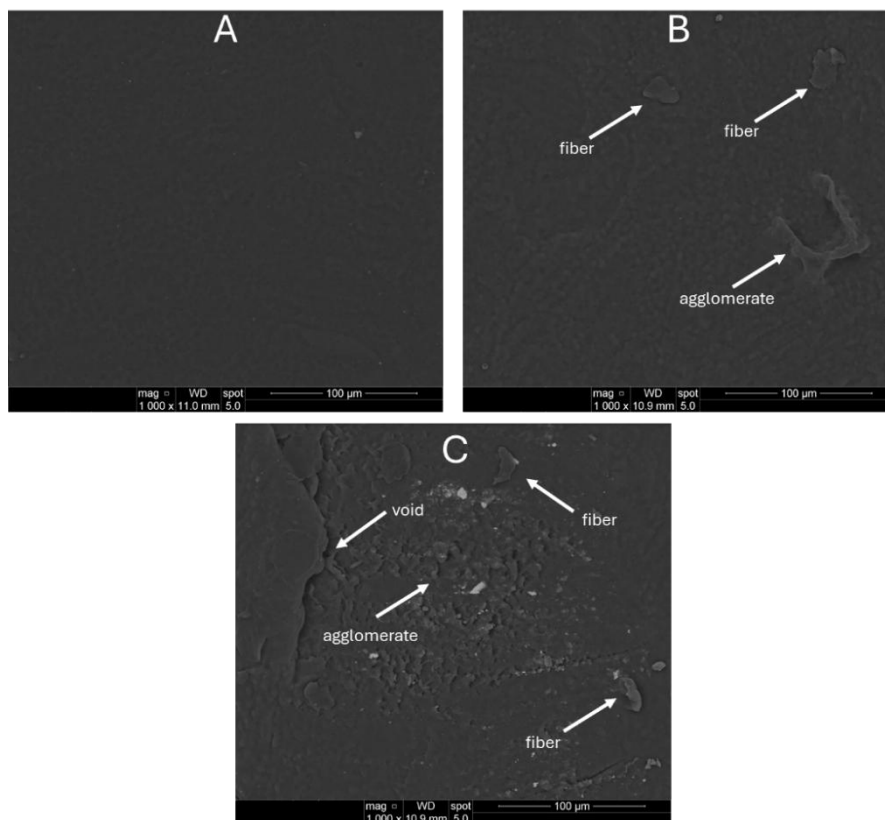


Figure 8. Mechanical properties of biocomposite films

### 3.7. Morphological Analysis

Figure 9 presents FESEM micrographs comparing the control biofilm and the biocomposite film. The surface of the control film appeared smooth, with no visible starch granules or cracks, and no noticeable aggregation. This observation aligns with findings by Tarique et al. [54] and Sanyang et al. [64] who reported similar surface characteristics in natural fiber biocomposite films with starch matrix. While the control film maintained a smooth and uniform appearance, the biocomposite film displayed an irregular surface. This irregularity was attributed to the increased concentration of SF fiber, leading to noticeable agglomeration within the matrix. The uneven structures suggest weak interfacial adhesion between the fiber and the matrix. Similar findings were reported by Ibrahim et al. [65] and Tarique et al. [54], where a film containing a high fiber concentration, void formations were observed, and the fracture surfaces became rougher and more rigid. This indicates that lower SF fiber concentrations integrate

more effectively with the matrix, leading to better dispersion, whereas higher concentrations promote aggregation. Additionally, reinforcing the biocomposite with 10% SF fiber contributed to the formation of voids and a coarser fracture surface. Starch agglomeration was also observed on the surface, possibly indicating crosslinking interactions at both intra- and intermolecular levels between SF fibers and the matrix, which may contribute to an increase in the molecular weight of the film. The incorporation of SF fibers into starch matrices can lead to significant interactions. These interactions can be both covalent and non-covalent, such as hydrogen bonding and hydrophobic interactions, which contribute to the overall stability and mechanical properties of the composite films [59, 60]. The presence of natural fibers can cause agglomeration on the surface of the starch matrix. This agglomeration is indicative of strong interactions between the fibers and the starch, which can lead to an increase in the molecular weight of the film due to the formation of a more complex network structure [60, 66, 67].



**Figure 9.** The SEM images of control biofilms (a) and biocomposite films with SF fiber 5% -NaOH5% (b) and 10%-NaOH5% (c)

### 4. CONCLUSION

Biocomposite films with favorable mechanical, physical, and water resistance properties have been successfully fabricated using edible canna starch (CS) as the matrix and alkali-treated sorghum fiber (SF) as reinforcement. Increasing the concentration of treated SF from 0% to 10 wt.% significantly enhanced tensile

strength and Young's modulus, indicating effective stress transfer within the composite. Alkali treatment improved fiber-matrix compatibility by removing hemicellulose, lignin, wax, and oils, thereby increasing surface hydrophilicity and enabling stronger hydrogen bonding with CS. The biocomposites also showed superior water resistance, attributed to robust intermolecular

interactions that limited water uptake. These improvements highlight the synergy between high-amylose starch and treated sorghum fiber in producing functional, biodegradable films. This study makes a novel contribution to the literature by introducing sorghum stalk fiber, an underutilized agricultural byproduct, as a reinforcement for edible canna starch, which has not been previously reported. The findings demonstrate strong potential for these biocomposites in sustainable packaging applications and provide new insights into fiber treatment and matrix interaction mechanisms. Future research should explore scalability, long-term performance, and alternative treatments to optimize biopackaging materials.

## REFERENCES

- Handayani L., Aprilia S., Arahman N., and Bilad M. R., (2024) Assessment of fibers from different part of the *Calotropis gigantea* biomass as a filler of composites foam PVA/PVP, *South African Journal of Chemical Engineering*.
- Ayyachamy M., Cliffe F. E., Coyne J. M., Collier J., and Tuohy M. G., (2013) Lignin: untapped biopolymers in biomass conversion technologies, *Biomass Conversion and Biorefinery*, vol. 3.
- Santos F. et al., (2017) Productive potential and quality of rice husk and straw for biorefineries, *Biomass Conversion and Biorefinery*, vol. 7.
- Roziafanto A. N., Furqon M., Sofyan N., and Chalid M., "Microfibrillated cellulose prepared from *Sorghum bicolor* (L.) moench by TEMPO-mediated oxidation treatment," in *International Conference on Chemical Science and Engineering*, 2021: Springer, pp. 9-16.
- Mahesh V., Mahesh V., Joladarashi S., and Kulkarni S. M., (2023) Experimental study on two-body and three-body abrasive wear behaviour of jute-natural rubber flexible green composite, *Journal of Thermoplastic Composite Materials*, vol. 36, no. 4.
- Roziafanto A. N., Lazuardi D. R., Ghozali M., Sofyan N., and Chalid M., (2023) Hydrothermal treatment of sorghum (*Sorghum bicolor* (L.) Moench) stalks for enhanced microfibrillated cellulose production, *Materials Research Express*, vol. 10, no. 9.
- Mansur I., Arrosid A., Yuanita E., and Chalid M., "Comparison study of chemical treatment of OPEFB fiber in the manufacture of microfibrillated cellulose," in *AIP Conference Proceedings*, 2024, vol. 2710, no. 1: AIP Publishing.
- Sathish S. et al., (2021) A review of natural fiber composites: Extraction methods, chemical treatments and applications, *Materials Today: Proceedings*, vol. 45.
- Oushabi A., Sair S., Hassani F. O., Abboud Y., Tanane O., and El Bouari A., (2017) The effect of alkali treatment on mechanical, morphological and thermal properties of date palm fibers (DPFs): Study of the interface of DPF–Polyurethane composite, *South African Journal of Chemical Engineering*, vol. 23.
- Zininga J. T., Puri A. K., Dlangamandla N., Wang Z., Singh S., and Permaul K., (2023) Integrated biorefinery of *Mucor circinelloides* biomass and sugarcane bagasse for application of high-value biopolymers, *Biomass Conversion and Biorefinery*.
- Altay L. et al., (2023) The Effect of Wastes of Nettle Fiber on Mechanical and Thermal Properties of Polypropylene Composite, *Journal of Natural Fibers*, vol. 20, no. 1.
- Atagur M. et al., (2020) Mechanical and thermal properties of *Carpinus betulus* fiber filled polypropylene composites, *Polymer Composites*, vol. 41, no. 5.
- Atagur M. et al., (2020) Evaluating of reinforcing effect of *Ceratonia Siliqua* for polypropylene: Tensile, flexural and other properties, *Polymer Testing*, vol. 89.
- Seki Y. et al., (2018) Surface modification of new cellulose fiber extracted from *Conium maculatum* plant: a comparative study, *Cellulose*, vol. 25.
- Andrew J. J. and Dhakal H., (2022) Sustainable biobased composites for advanced applications: Recent trends and future opportunities–A critical review, *Composites Part C: Open Access*, vol. 7.
- Gondaliya A., Alipoormazandarani N., Kleiman M., and Foster E. J., (2023) Sustainable compressed biocomposite: Review on development and novel approaches, *Materials Today Communications*.
- Kumar S. and Saha A., (2022) Utilization of coconut shell biomass residue to develop sustainable biocomposites and characterize the physical, mechanical, thermal, and water absorption properties, *Biomass Conversion and Biorefinery*.
- Mohit H. and Arul Mozhi Selvan V., (2018) A comprehensive review on surface modification, structure interface and bonding mechanism of plant cellulose fiber reinforced polymer based composites, *Composite Interfaces*, vol. 25, no. 5-7.
- Yang J., Ching Y. C., and Chuah C. H., (2019) Applications of lignocellulosic fibers and lignin in bioplastics: A review, *Polymers*, vol. 11, no. 5.
- Fazeli M., Florez J. P., and Simão R. A., (2019) Improvement in adhesion of cellulose fibers to the thermoplastic starch matrix by plasma treatment modification, *Composites Part B: Engineering*, vol. 163.
- Bhatia J. K., Kaith B. S., and Kalia S., (2016) Recent developments in surface modification of natural fibers for their use in biocomposites, *Biodegradable Green Composites*.
- Vu T.-H., Peng K.-C., and Chung R. H., (2019) Evaluation of environmental efficiency of edible canna production in Vietnam, *Agriculture*, vol. 9, no. 11.
- Zhang J. and Wang Z.-W., (2013) Soluble dietary fiber from *Canna edulis* Ker by-product and its physicochemical properties, *Carbohydrate polymers*, vol. 92, no. 1.
- Gabriel A. A., Solikhah A. F., Rahmawati A. Y., Taradipa Y. S., and Maulida E. T., (2021) Potentials of Edible Canna (*Canna edulis* Kerr) Starch for Bioplastic: A Review, *Industria: Jurnal Teknologi dan Manajemen Agroindustri*, vol. 10, no. 2.
- Pudjihastuti I., Sumardiono S., Supriyo E., and Kusumayanti H., "Quality analog rice composite flour: Modified starch, *Colocasia esculenta*, *Canna edulis* Ker high protein," in *AIP Conference Proceedings*, 2018, vol. 1977, no. 1: AIP Publishing.
- Wang K. et al., (2017) Mechanical and barrier properties of maize starch–gelatin composite films: Effects of amylose content, *Journal of the Science of Food and Agriculture*, vol. 97, no. 11.

27. Romero-Bastida C., Bello-Perez L. A., Velazquez G., and Alvarez-Ramirez J., (2015) Effect of the addition order and amylose content on mechanical, barrier and structural properties of films made with starch and montmorillonite, *Carbohydrate Polymers*, vol. 127.
28. Menzel C. et al., (2015) Improved material properties of solution-cast starch films: Effect of varying amylopectin structure and amylose content of starch from genetically modified potatoes, *Carbohydrate Polymers*, vol. 130.
29. Van N. K. et al., (2023) Preparation and characterization of achira starch oxidized by different concentration of sodium hypochlorite, *Vietnam Journal of Chemistry*, vol. 61.
30. Ávila-Martín L., Beltrán-Osuna Á. A., and Perilla J. E., (2020) Effect of the addition of citric acid and whey protein isolate in *Canna indica* L. starch films obtained by solvent casting, *Journal of Polymers and the Environment*, vol. 28, no. 3.
31. Rhim J.-W. and Ng P. K., (2007) Natural biopolymer-based nanocomposite films for packaging applications, *Critical reviews in food science and nutrition*, vol. 47, no. 4.
32. Qi C., Yadama V., Guo K., and Wolcott M. P., (2013) Thermal conductivity of sorghum and sorghum-thermoplastic composite panels, *Industrial Crops and Products*, vol. 45.
33. Alves J. A. A. et al., (2019) Sorghum straw: Pulping and bleaching process optimization and synthesis of cellulose acetate, *International Journal of Biological Macromolecules*, vol. 135.
34. Kusumah S. S., Umemura K., Yoshioka K., Miyafuji H., and Kanayama K., (2016) Utilization of sweet sorghum bagasse and citric acid for manufacturing of particleboard I: Effects of pre-drying treatment and citric acid content on the board properties, *Industrial Crops and Products*, vol. 84.
35. Handayani S., Husnil Y. A., Handayani A. S., and Chalid M., "Application of waste sorghum stem (*Sorghum bicolor*) as a raw material for microfibre cellulose," in *IOP Conference Series: Materials Science and Engineering*, 2019, vol. 509, no. 1: IOP Publishing, p. 012015.
36. Ismojo I., Ammar A. A., Ramadhita G., Zulfia A., and Chalid M., (2018) Influence of chemical treatments sequence on morphology and crystallinity of sorghum fibers, *Indonesian Journal of Chemistry*, vol. 18, no. 2.
37. Husnil Y. A., Yuanita E., Ramadhani N., and Chalid M., "Study on the effect of bleaching treatment on the mechanical properties of kenaf fibers," in *Materials Science Forum*, 2020, vol. 1000: Trans Tech Publ, pp. 278-284.
38. Namondo B. V., Etape E. P., Foba-Tendo J., Yollandev F. C., Nsom M. V., and William N., (2019) Extraction and Physicochemical Characterization of Lignin from Cameroon's Three Raffia Palm Species (*Raffia Farinifera*, *Raffia Hookeri* and *Raffia Vinifera*) and Africa Oil Palm (OPEFB), *Journal of Materials Sciences and Applications*, vol. 5, no. 2.
39. Roziafanto A. N., Alfariis F., Ramadhan T., and Chalid M., "Preliminary Study of Modified Lignin Compatibility in Polypropylene-Modified Bitumen," in *Macromolecular Symposia*, 2020, vol. 391, no. 1: Wiley Online Library, p. 1900158.
40. Roziafanto A. N., Dwijaya M. S., Yunita R., Amrullah M., and Chalid M., "Synthesis hybrid bio-polyurethane foam from biomass material," in *AIP Conference Proceedings*, 2019, vol. 2175, no. 1: AIP Publishing LLC, p. 020068.
41. Ayyash A., Varol E. A., Kılıç M., and Özsin G., (2024) Influence of aging on the rheological behavior and characteristics of bio-oil produced from olive pomace via slow pyrolysis, *Biomass Conversion and Biorefinery*, vol. 14, no. 8.
42. Sudiyani Y., Tsujiyama S.-i., Imamura Y., Takahashi M., Minato K., and Kajita H., (1999) Chemical characteristics of surfaces of hardwood and softwood deteriorated by weathering, *Journal of wood science*, vol. 45, no. 4.
43. Lehto J., Louhelainen J., Kłosińska T., Drożdżek M., and Alén R., (2018) Characterization of alkali-extracted wood by FTIR-ATR spectroscopy, *Biomass Conversion and Biorefinery*, vol. 8.
44. Venegas R., Torres A., Rueda A. M., Morales M. A., Arias M. J., and Porras A., (2022) Development and Characterization of Plantain (*Musa paradisiaca*) Flour-Based Biopolymer Films Reinforced with Plantain Fibers, *Polymers*, vol. 14, no. 4.
45. Edhirej A., Sapuan S., Jawaid M., and Zahari N. I., (2017) Cassava/sugar palm fiber reinforced cassava starch hybrid composites: Physical, thermal and structural properties, *International journal of biological macromolecules*, vol. 101.
46. Lenhani G. C. et al., (2021) Application of corn fibers from harvest residues in biocomposite films, *Journal of Polymers and the Environment*, vol. 29.
47. Chavan P. et al., (2022) Nanocomposite starch films: A new approach for biodegradable packaging materials, *Starch-Stärke*, vol. 74, no. 5-6.
48. Zhang J. et al., (2023) Effects of different sources of cellulose on mechanical and barrier properties of thermoplastic sweet potato starch films, *Industrial Crops and Products*, vol. 194.
49. Vilay V., Mariatti M., Taib R. M., and Todo M., (2008) Effect of fiber surface treatment and fiber loading on the properties of bagasse fiber-reinforced unsaturated polyester composites, *Composites Science and Technology*, vol. 68, no. 3-4.
50. Bouhank S., Nekkaa S., and Haddaoui N., (2016) Water absorption, biodegradation, thermal and morphological properties of *Spartium junceum* fiber-reinforced polyvinylchloride composites: effects of fibers content and surface modification, *Journal of Adhesion Science and Technology*, vol. 30, no. 13.
51. Mittal M. and Chaudhary R., (2018) Experimental investigation on the mechanical properties and water absorption behavior of randomly oriented short pineapple/coir fiber-reinforced hybrid epoxy composites, *Materials Research Express*, vol. 6, no. 1.
52. Narkchamnan S. and Sakdaronnarong C., (2013) Thermo-molded biocomposite from cassava starch, natural fibers and lignin associated by laccase-mediator system, *Carbohydrate Polymers*, vol. 96, no. 1.
53. Kargarzadeh H., Johar N., and Ahmad I., (2017) Starch biocomposite film reinforced by multiscale rice husk fiber, *Composites Science and Technology*, vol. 151.
54. Tarique J., Sapuan S., and Khalina A., (2021) Effect of glycerol plasticizer loading on the physical, mechanical, thermal, and barrier properties of arrowroot (*Maranta arundinacea*) starch biopolymers, *Scientific reports*, vol. 11, no. 1.
55. Neto J. S., de Queiroz H. F., Aguiar R. A., and Banea M. D., (2021) A review on the thermal characterisation of natural and hybrid fiber composites, *Polymers*, vol. 13, no. 24.

56. Jordá-Vilaplana A., Carbonell-Verdú A., Samper M.-D., Pop A., and Garcia-Sanoguera D., (2017) Development and characterization of a new natural fiber reinforced thermoplastic (NFRP) with *Cortaderia selloana* (Pampa grass) short fibers, *Composites Science and Technology*, vol. 145.
57. Oberparleiter S. and Lackner R., (2023) New insight into the melting process of PA 6 fibers: Determining tension-induced changes of enthalpy by HTA-assisted DSC analysis, *Polymer Testing*, vol. 117.
58. Ezekiel N., Ndazi B., Nyahumwa C., and Karlsson S., (2011) Effect of temperature and durations of heating on coir fibers, *Industrial Crops and Products*, vol. 33, no. 3.
59. Kallu S., Kowalski R. J., and Ganjyal G. M., (2017) Impacts of cellulose fiber particle size and starch type on expansion during extrusion processing, *Journal of food science*, vol. 82, no. 7.
60. Balakrishnan P., Sreekala M., Kunaver M., Huskić M., and Thomas S., (2017) Morphology, transport characteristics and viscoelastic polymer chain confinement in nanocomposites based on thermoplastic potato starch and cellulose nanofibers from pineapple leaf, *Carbohydrate polymers*, vol. 169.
61. Hazrol M., Sapuan S., Ilyas R., Zainudin E., Zuhri M., and Wahab N. A., (2022) Morphology and selected properties of kenaf fiber/cornhusk reinforced corn starch hybrid biocomposites, *Polimery*, vol. 67, no. 11-12.
62. Fu Z. Q., Wu H. J., Wu M., Huang Z. G., and Zhang M., (2020) Effect of wheat bran fiber on the behaviors of maize starch based films, *Starch-Stärke*, vol. 72, no. 11-12.
63. Corradini E., Agnelli J. A., Morais L. C. d., and Mattoso L. H., (2008) Study of properties of biodegradable composites of starch/gluten/glycerol reinforced with sisal fibers, *Polímeros*, vol. 18.
64. Sanyang M., Sapuan S., Jawaid M., Ishak M., and Sahari J., (2016) Development and characterization of sugar palm starch and poly (lactic acid) bilayer films, *Carbohydrate polymers*, vol. 146.
65. Ibrahim M., Sapuan S., Zainudin E., and Zuhri M., (2019) Potential of using multiscale corn husk fiber as reinforcing filler in cornstarch-based biocomposites, *International journal of biological macromolecules*, vol. 139.
66. de Oliveira J. P. et al., (2025) Effect of eucalyptus nanofibril as reinforcement in biodegradable thermoplastic films based on rice starch (*Oryza sativa*): Evaluation as primary packaging for crackers, *Food Chemistry*.
67. Guimarães I. C. et al., (2016) Cellulose microfibrillated suspension of carrots obtained by mechanical defibrillation and their application in edible starch films, *Industrial Crops and Products*, vol. 89.