



## Characterization of nanocellulose from banana stem fiber and its bionanocomposite as a thermal insulation material

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### ABSTRACT

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Nanocellulose has garnered significant attention due to its unique properties and potential in various applications, including thermal insulation. The abundant banana stems found in Lombok Island generate considerable waste that can be utilized as a raw material for nanocellulose. This research aims to characterization of nanocellulose derived from Kepok banana stems and its composites as thermal insulation in solar panels. Nanocellulose was produced using acid hydrolysis with sulfuric acid ( $H_2SO_4$ ), and several treatments were carried out including Natrium hydroxide (NaOH), Sodium chlorite ( $NaClO_2$ ), Sulfuric acid ( $H_2SO_4$ ). The results show that nanocellulose from banana stems has a tensile strength of 13.374 MPa - 13.63 MPa. The addition of nanocellulose was found to increase the tensile strength of the composite reaching 13.374 up to 13.63 MPa, thermal conductivity 0,1692 up to 0,1940 W/mK and is quite heat resistant at a temperature of 200 °C. SEM photos of nanocellulose show surface roughness and produce interface strength between banana stem fiber nanocellulose and polyester resin. With solid bonding, good tensile strength, conductivity and thermal stability, bionanocomposites from banana stem can be used as thermal insulator (backsheets) materials for solar panels.

## 1. INTRODUCTION

Nanocellulose has garnered significant attention in recent years due to its unique properties and its potential applications across various fields. Several recent research articles and reviews have provided comprehensive explanations of nanocellulose, ranging from its production processes to its applications (Sofiah et al., 2023). Various types of nanocomposites have been widely used as coating materials for industrial machinery. One notable application is the modification of nanocellulose films, a type of modified nanocellulose coating, applied on glass, aluminum, and steel substrates. Nanocellulose-based nanocomposite coatings exhibit excellent properties, particularly in terms of air resistance, crack resistance, corrosion resistance, and water absorption and permeability (Sari et al., 2022). The use of natural fibers in various sectors, such as textiles, construction materials, and automotive industries, has great potential, especially when sourced from agricultural waste. In Lombok Island, most of the population are farmers, and many allocate part of their land to banana cultivation. After harvesting the fruit, farmers usually cut down the banana plants, which leads to the accumulation of banana stem waste. Banana stems, which are water-rich and decompose easily, pose a potential ecological hazard if not managed properly. The banana stem consists of a pseudo stem, made up of leaf sheaths, and the true stem, commonly known as the banana corm. The pseudostem is known not only for its high-water content but also for its rich polymer content, including cellulose, as stated by Castillo et al. (2023).

Several researchers have investigated the fibers derived from banana stems and their composites. Siahaan et al. (2021) studied the isolation of *banana pseudostem* fibers as reinforcement for biocomposites. They reported that degumming treatment using sodium carbonate was more effective than using xylanase enzymes, although the latter showed slightly higher tensile strength. Sodium carbonate degumming at a concentration of 30 g/L resulted in a 12.1% mass reduction, tensile strength of 18.84 g/Text, elongation of 21.74%, and water absorption capacity of 1310.33%. Thermal testing showed that the fibers degraded at around 360°C. These results suggest that *banana pseudostem* fibers can be applied as textile fibers and reinforcement for biocomposites.

Further research by Devi and Priatmoko (2024) explored the effect of adding nanocellulose and glycerol to enhance the mechanical properties of bioplastics made from banana corm waste. The nanocellulose was produced through acid hydrolysis, while the bioplastics were made using the casting method. Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) with a concentration of 50% was used for hydrolysis. The addition of nanocellulose increased the tensile strength of the bioplastics, while the addition of glycerol accelerated the biodegradation process. The bioplastics with nanocellulose and glycerol had a tensile strength of 1.83 MPa, elongation of 11.45%, Young's modulus of 48.12 MPa, and mass loss due to biodegradation of 64.44%.

Nasution et al. (2020) studied the optimal temperature and sulfuric acid concentration conditions for isolating nanocrystalline cellulose (NCC) from *banana pseudostems* to achieve the highest yield and water solubility. The *banana pseudostem* fibers were first delignified using NaOH at 80°C for 5 minutes, followed by bleaching with H<sub>2</sub>O<sub>2</sub> for 30 minutes (twice), and then acid hydrolysis with H<sub>2</sub>SO<sub>4</sub> for 1 hour, using varying hydrolysis temperatures (45°C, 50°C, 55°C, 60°C) and sulfuric acid concentrations (40%, 45%, 50%, 55%), with ultrasonication for 5 minutes. TEM analysis showed that the isolated NCC had sizes ranging from 125 to 144 nm. The highest NCC yield (26.75%) was achieved at 60°C with 55% sulfuric acid concentration. The lowest water solubility (0%) indicated the purity of the cellulose used for NCC isolation.

Nurjannah et al. (2020) suggested that *banana pseudostem* fibers can be utilized as biomaterials for clinical applications. The fibers were synthesized into methylcellulose (MC), which could be used to produce hydrogels beneficial for wound healing and skin irritation treatments. In another study by Tajalla et al. (2024), the thermal characteristics of composites made from polypropylene and *banana pseudostems* were examined. The results revealed that *banana pseudostem* fibers could serve as thermal insulation materials for housing due to their low thermal conductivity (0.1166 W/mK) when combined with a polypropylene matrix. The study also indicated that decreasing the fiber volume fraction led to lower thermal conductivity, and the arrangement of fibers and polymer matrix affected the composite's thermal conductivity.

Research on the utilization of *banana pseudostems* as nanocellulose and biocomposites for thermal insulation materials remains limited. Given the promising potential of *banana pseudostem nanocellulose* as an insulating material, further research is necessary to explore its characterization and composite applications for thermal insulation.

## 2. RESEARCH METHODS

The material used in this research is the pseudostem of the *\*Musa acuminata x balbisiana\** species, commonly known as kepok banana. The study consists of five stages: the first stage involves alkaline pretreatment using a 0.5% detergent solvent and 0.005% Na<sub>2</sub>CO<sub>3</sub>. The second stage is delignification, which uses a 17.5% NaOH solution. In the third stage, bleaching is conducted with a 0.2% NaClO<sub>2</sub> solution. The fourth

stage involves the production of nanocellulose through acid hydrolysis using a 60%  $\text{H}_2\text{SO}_4$  solution. Finally, in the fifth stage, composites are produced using polyester resin with concentration variations of 70% and 80%, where the ratio of nanocellulose to resin is 30:70 and 20:80, with a catalyst concentration of 2%.

The banana stem waste, cut into 10-15 cm pieces, was steamed for 20 minutes at  $100^\circ\text{C}$  and then extracted by combing the fibers with an iron comb. The fibers were subsequently cleaned under running water and air-dried in an enclosed room, shielded from sunlight, for 48 hours. The fibers were then processed using a detergent solution with a concentration of 0.5% and 0.005%  $\text{Na}_2\text{CO}_3$ . The fiber-to-solution ratio was 1:50, and the solution was heated to  $60^\circ\text{C}$  for 30 minutes. The fibers were repeatedly washed with distilled water, dried in an electric oven at  $105^\circ\text{C}$  for 1 hour, and then stored in a desiccator for 24 hours. The alkaline pretreatment process is illustrated in Figure 1.

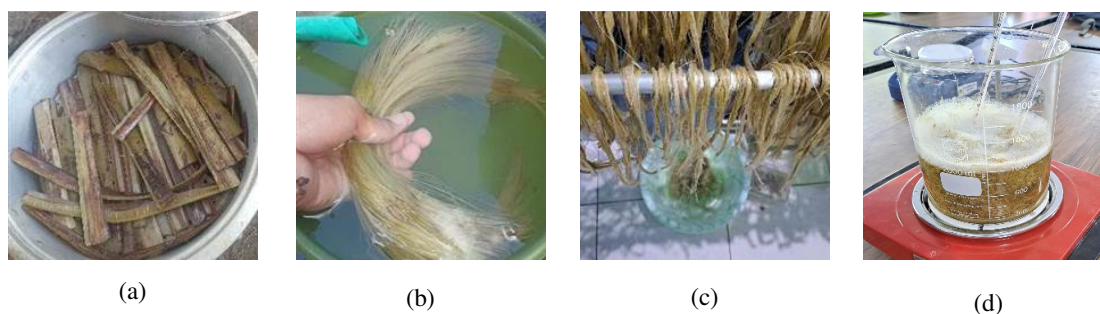


Figure 1. Pretreatment stages of *kapok banana pseudostem* fibers: (a) steaming process of the *banana pseudostem*, (b) fiber extraction and cleaning process, (c) fiber drying process in a closed room, (d) fiber soaking process using NaOH solution.

The delignification process was carried out using a 17.5% NaOH solution, with a material-to-solution ratio of 1:50, for 2 hours at  $40^\circ\text{C}$  under constant stirring. The resulting cellulose was then filtered and washed with distilled water until clean. The purified cellulose was subsequently dried in an electric oven for 1 hour at  $90^\circ\text{C}$ . The delignification process is illustrated in Figure 2.



Figure 2. Delignification process of *kapok banana pseudostem* fibers using NaOH.

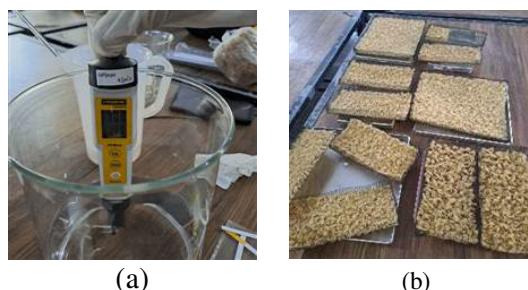


Figure 3. Bleaching process of *kapok Banana pseudostem* fibers: (a) pH measurement of the  $\text{NaClO}_2$  solution, (b) fiber drying process in a closed room.

The bleaching process was conducted using 0.2%  $\text{NaClO}_2$  dissolved in 1 liter of water. The pH was measured first using a digital pH meter, and then glacial acetic acid was added until the solution reached a pH of

4. The bleaching stage lasted for 2 hours, after which the cellulose was rinsed with water and air-dried at room temperature for 24 hours. The bleaching process was repeated once more following the same method. The bleaching stages are illustrated in Figure 3.

The nanocellulose production stage was carried out using the acid hydrolysis method. The cellulose was dried using a freeze dryer for 48 hours and stored in a desiccator to maintain its moisture content. The cellulose was then hydrolyzed with 60%  $\text{H}_2\text{SO}_4$  at  $40^\circ\text{C}$  for 30 minutes with constant stirring. Cold water (50 ml) was added to the solution to stop the hydrolysis process. The reaction mixture was cooled to room temperature and followed by centrifugation at 3500 rpm for 90 minutes. The nanocellulose suspension was obtained by stirring the solid fraction while gradually adding sufficient water. To preserve the nanocellulose suspension, absolute alcohol was added, and the suspension was stored in a clean and secure glass container. The nanocellulose production process is illustrated in Figure 4.

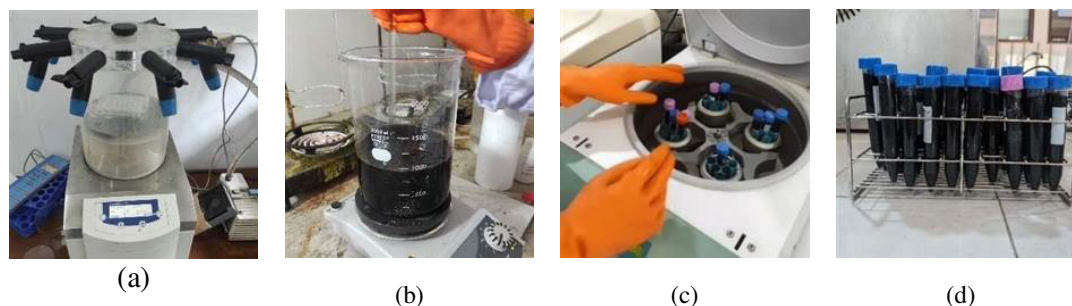


Figure 4. Nanocellulose production: (a) freeze-drying process, (b) hydrolysis process using  $\text{H}_2\text{SO}_4$ , (c) centrifugation process, (d) suspension storage process.

### Preparation of bionanocomposites

The production of bionanocomposites was carried out by calculating the volume composition of kepok *banana pseudostem* nanocellulose and polyester resin as the matrix. The composition of nanocellulose to resin matrix was varied at ratios of 20:80 and 30:70. The resin and catalyst were mixed until homogeneous. The seepok *banana pseudostem* nanocellulose was evenly placed into a silicone mold, and then the resin mixture was poured into an ASTM D638 type IV mold. The specimen was left to harden for approximately 24 hours. It was then carefully removed from the mold and allowed to dry at room temperature for 48 hours.

### Characterisations

The TGA (Thermogravimetric Analysis) test is conducted to determine the thermal characteristics of a material. This test is used to assess the mass changes of nanocomposite materials when heated across different temperature ranges. The result of this test is a thermogravimetric curve, providing information on phase transitions and thermal decomposition of the material. For this nanocomposite material, the TGA test was performed in the temperature range of  $25^\circ\text{C}$  to  $600^\circ\text{C}$ . The samples used included raw material, cellulose after bleaching, nanocellulose, and nanocomposites.

The SEM (Scanning Electron Microscopy) test was performed to analyze the surface morphology and size of the nanocomposite materials. SEM results provided surface images, and the surface area of the nanocomposite particles was measured using ImageJ software. The magnifications used for morphology identification were 250x, 500x, and 1000x. The samples analyzed included raw kepok *banana pseudostem* fibers, nanocellulose samples, and nanocomposite samples. For this SEM test, the sample weight was 3 g.

The tensile strength test was carried out to evaluate the mechanical properties of the material by applying tensile force or strain to it. Tensile strength was measured using a universal testing machine, RTG-1250 type. The sample preparation for the tensile strength test followed ASTM D638 Type IV specifications, with a specimen length of 115 mm and width of 19 mm. The ASTM D638 Type IV tensile test specimen can be seen in Figure 5.

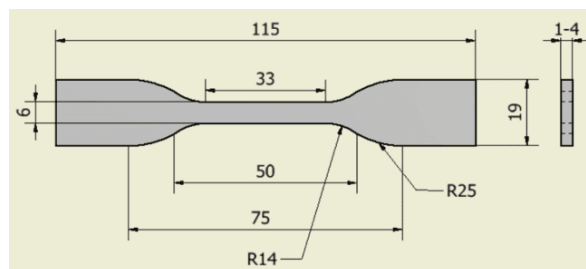


Figure 5. tensile test specimen of ASTM D638 Type IV

The color change test is used to demonstrate the occurrence of a reaction when the material undergoes chemical treatment. This test is conducted by observing the color changes in the material after each treatment. The thermal conductivity test is performed to determine the thermal conductivity coefficient of a material. The thermal conductivity coefficient is measured using the QTM-500 device with the hot wire method. The dimensions of the sample used are 100 x 50 x 2.8 mm. The test was conducted with three reference materials: polyethylene foam, silicone rubber, and quartz glass.

### 3. RESULTS AND DISCUSSION

TGA was conducted to assess material purity, decomposition, thermal degradation, and chemical reactions involving weight changes due to adsorption, desorption, and chemical kinetics. This characterization determines weight loss or weight gain (gas fixation) of the sample (Dewi et al., 2023). The TGA results for raw kepok *banana pseudostem* fibers and nanocellulose are presented in Figure 6. The test was performed using 3.45 g of raw fiber and 4.08 g of nanocellulose. The TGA curves show mass changes with increasing temperature, as depicted in Figure 6. Both samples began to decompose slowly at 25°C. Significant mass loss for the raw fiber sample occurred between 320°C and 362°C, while for nanocellulose, the extreme mass loss started at 295°C and continued until 480°C. At these points, most of the material decomposed completely by 600°C. The total weight loss for the raw fiber sample was 106.6%, while nanocellulose exhibited a weight loss of 104%.

The raw fiber demonstrated better thermal stability. Thermal degradation occurred in three stages: the first degradation at around 100°C was due to water evaporation; the second stage, between 300°C and 500°C, was the decomposition of the matrix; and the final stage at 500°C involved complete decomposition, leaving behind a charcoal residue (Nurazzi et al., 2021). These results align with previous findings by Nurazzi et al. (2021), who reported similar multi-step degradation in natural fiber-based materials. The comparison highlights that the use of kepok *banana pseudostem* fibers maintains consistent thermal degradation behavior in comparison to other lignocellulosic materials.

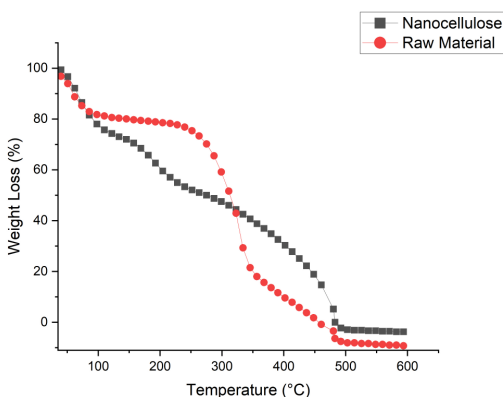


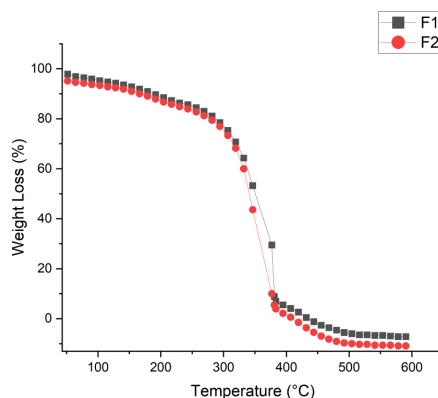
Figure 6. Thermogravimetric analysis of nanocellulose from *Musa acuminata* x *balbisiana* kepok *banana pseudostem*.

Table 1. Thermal stability of raw fiber and nanocellulose from kepok *banana pseudostem*

Sample	Residue (%)	T <sub>onset</sub> (°C)	T <sub>max</sub> (°C)
Raw Material	20.87	308.92	346.41
Nanocellulose	37.55	284.66	364.52



Figure 7 shows that decomposition in bionanocomposite samples began slowly at 25°C. Significant mass loss for the F1 bionanocomposite sample started at 368.97°C and continued until 416.48°C. For the F2 bionanocomposite, extreme mass loss occurred between 352.28°C and 386.19°C. Both samples completely decomposed by 600°C. The TGA results indicate that F1 bionanocomposites exhibit relatively higher heat resistance compared to F2. Additionally, the thermal stability of both F1 and F2 bionanocomposites aligns with the thermal stability of the backsheet in solar panels tested by Kumar and Bhargava (2018), with minimal mass loss observed in the 25°C to 350°C range.

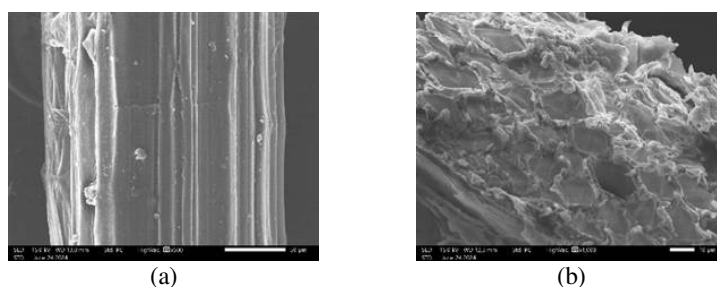


**Figure 7.** Thermogravimetric analysis of bionanocomposites from *Musa acuminata x balbisiana* kepok banana pseudostem.

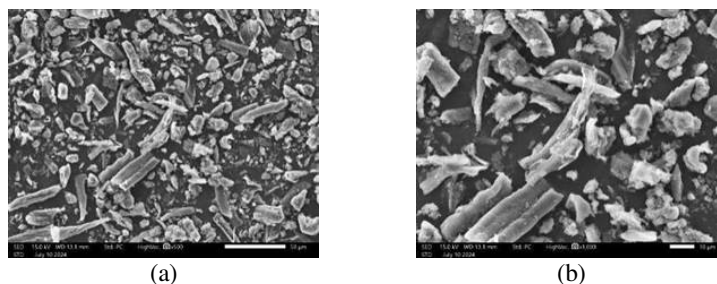
**Table 2.** Thermal Stability of Bionanocomposites

Sample	Residue (%)	T <sub>onset</sub> (°C)	T <sub>max</sub> (°C)
F1(20%)	2.88	358.97	416.48
F2(30%)	3.33	352.28	386.19

The morphological analysis of kepok *banana pseudostem* fibers was performed using SEM for both raw materials and nanocellulose samples. The magnifications used were 500x and 1000x, with the results shown in Figures 8 and 9. The longitudinal cross-sections visible on the fiber surface illustrate the fiber bundles composed of fibrils (Siahaan et al., 2021). In Figure 8(a), the raw material shows tightly bound fibers, while the irregular bonding observed in Figure 8(b) indicates that lignocellulosic bonds remain intact. In contrast, after acid hydrolysis, the nanocellulose fibers exhibit looser, rougher bonding, as shown in Figures 9(a) and (b). This indicates that treatments with 0.2% NaClO<sub>2</sub> and 60% H<sub>2</sub>SO<sub>4</sub> were effective in breaking the bonds between lignin, hemicellulose, and cellulose.

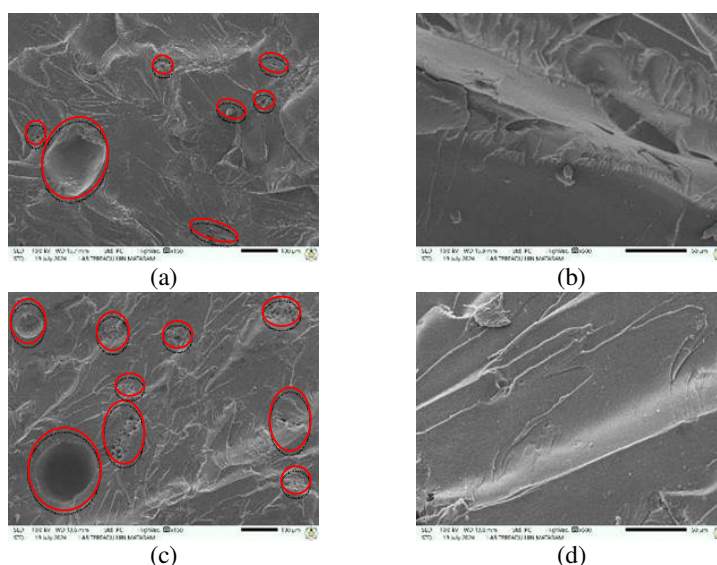


Figures 8. SEM images of raw kepok banana pseudostem fibers (a) 500x magnification (b) 1000x magnification.



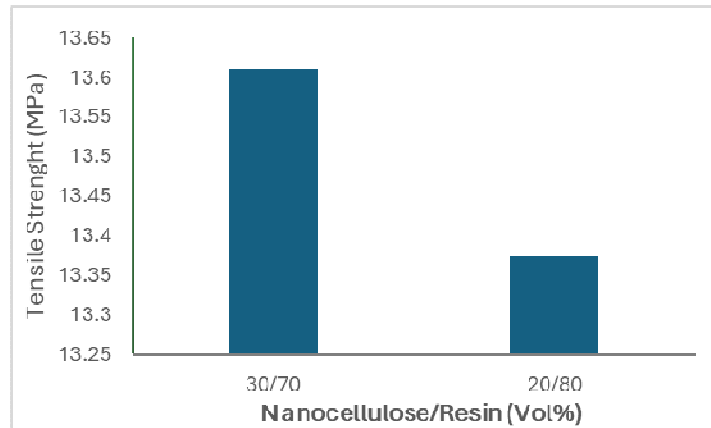
Figures 9. SEM images of nanocellulose from kepok banana pseudostem (a) 500x magnification (b) 1000x magnification.

The morphological analysis of bionanocomposite from kepok *banana pseudostem*, in 20:80 and 30:70 formulations, is shown in Figure 8. Figures 8 (a) and (b) present the morphological condition of the bionanocomposite with 20% nanocellulose and 80% polyester resin. In figure 10 (a), the parts circled with red circles are the layers and voids created throughout the surface of the 20:80 bionanocomposite. The voids formed are caused by air trapped during the mixing process when the material is in direct contact with air. Similarly, Figures 10 (c) and (d) illustrate the morphological conditions of the bionanocomposites with 30% nanocellulose and 70% polyester resin composition. In Figure 10 (c), the parts circled with red circles show voids uniformly distributed throughout the surface with smaller sizes than the voids in the bionanocomposites with 20:80 composition.

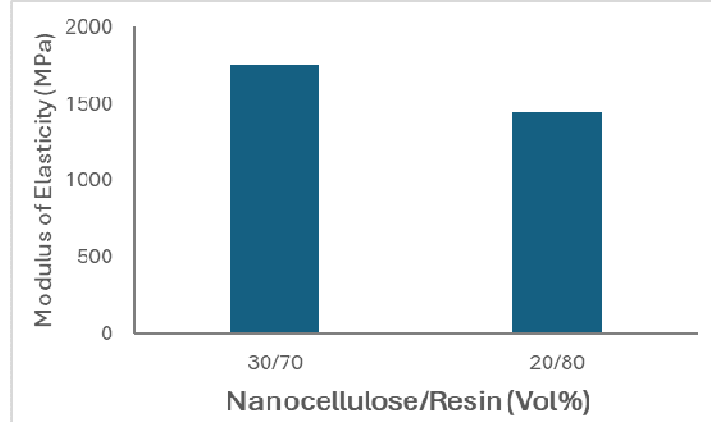


Figures 10. SEM images of bionanocomposites (a) 20:80 formulation at 150x magnification (b) 20:80 formulation at 500x magnification (c) 30:70 formulation at 150x magnification (d) 30:70 formulation at 500x magnification.

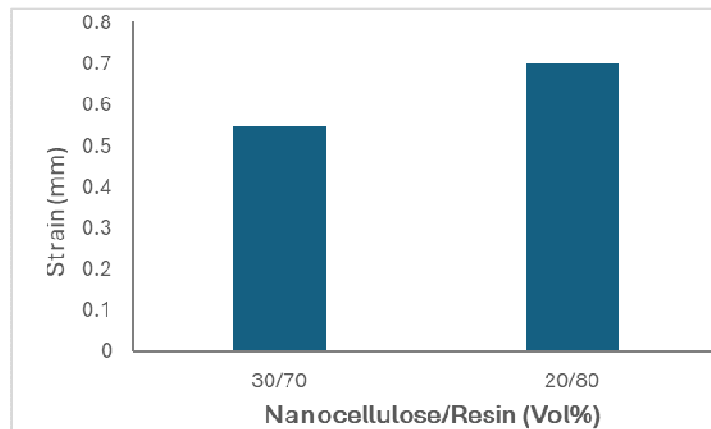
Tensile strength tests were conducted using a Universal Testing Machine (UTM) RTG-1250 model, following ASTM D638 Type IV standards. The results for the 20:80 and 30:70 bionanocomposite formulations are shown in the following figures 11. For the 20:80 bionanocomposite, the maximum load was 336.75 N with a maximum tensile strength of 13.374 MPa. For the 30:70 bionanocomposite, the maximum load was 340.5 N, with a maximum tensile strength of 13.61 MPa. The elastic modulus values for both composites are shown in Figure 9. The 20:80 composite had an elastic modulus of 1446.2 MPa, while the 30:70 composite had a modulus of 1751.4 MPa is shown in Figure 12. The maximum elongation capacities of both bionanocomposites are presented in Figure 13. The 20:80 composite exhibited a maximum elongation of 0.6993 mm, while the 30:70 composite had an elongation capacity of 0.5461 mm.



Figures 11. Tensile strength graphs for 20:80 and 30:70 bionanocomposites.



Figures 12. elastic modulus, and elongation graphs for 20:80 and 30:70 bionanocomposites.



Figures 13. elongation graphs for 20:80 and 30:70 bionanocomposites.



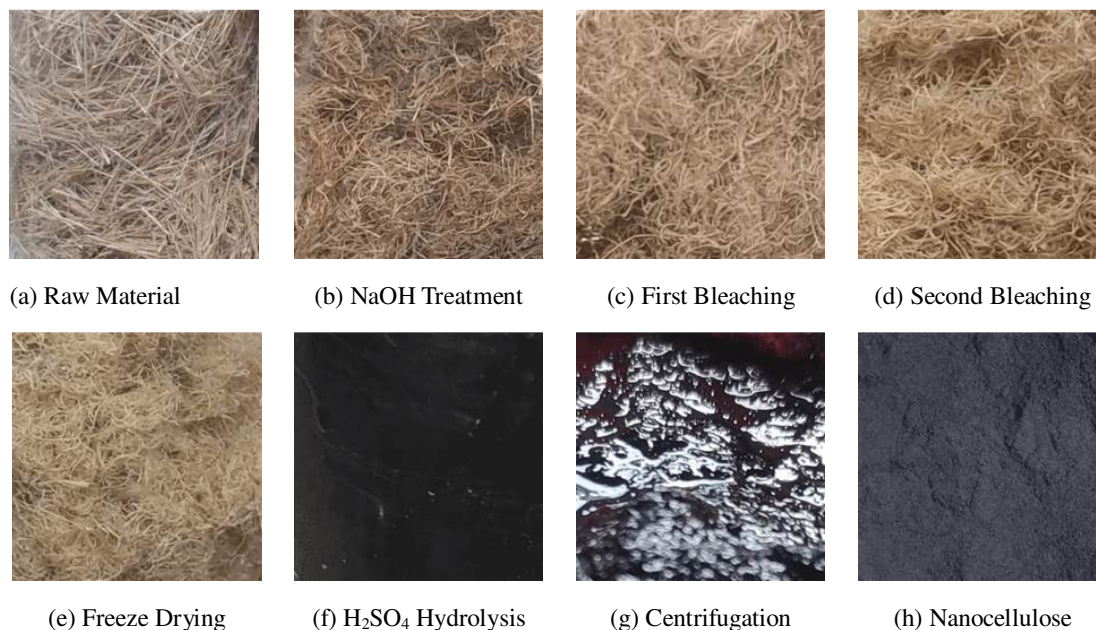


Figure 14. Color change of *banana pseudostem* fibers during various treatment stages

The analysis of color changes in the *banana pseudostem* from untreated fiber to nanocellulose is shown in Figure 14. In Figure 14(a), the raw *banana pseudostem* fibers are light brown. Figure 14(b) shows that after treatment with 17.5% NaOH, the fibers darken. Figure 14(c) shows the first bleaching stage, where the fibers lighten as lignin begins to separate from the cellulose (Nasution, Ellsworth and Wijaya, 2020). In Figure 14(d), after the second bleaching stage with 0.2% NaClO<sub>2</sub> for 2 hours, the fibers become even lighter and more translucent as non-cellulose components degrade. Figure 14(e) shows the fibers after freeze-drying, turning cream-colored. Figures 14(f) and (g) depict the fibers turning dark after 60% H<sub>2</sub>SO<sub>4</sub> hydrolysis. This significant color change is attributed to dehydration and carbonization, as noted by Leolovich in Pratama et al. (2019), when sulfuric acid concentrations exceed 60% at temperatures above 60°C. Finally, Figure 14(h) shows the final black-colored nanocellulose.

Thermal conductivity is shown in Figure 15 testing was conducted on two bionanocomposite samples with 20:80 and 30:70 nanocellulose to resin volume fractions. The thermal conductivity values obtained for the 20:80 and 30:70 formulations were 0.1940 W/mK and 0.1692 W/mK, respectively. The highest thermal conductivity value of 0.1940 W/mK for the 20:80 formulation surpasses the thermal conductivity of PET and EVA plastics in Adesina's (2021) study, which reported values of 0.150 W/mK and 0.084 W/mK, respectively.

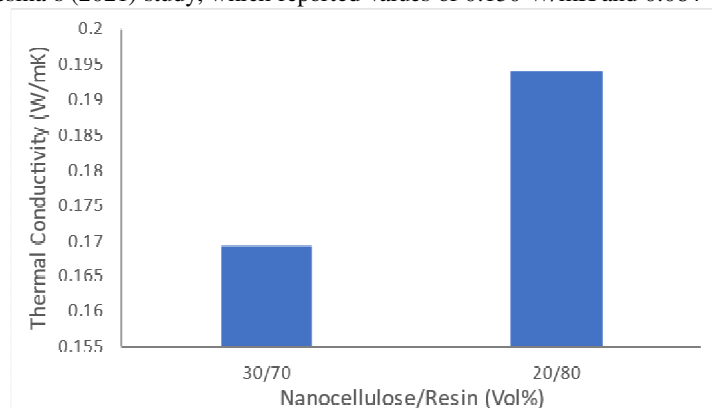


Figure 15. Thermal conductivity coefficient graph of bionanocomposites.

#### 4. CONCLUSION

This experimental study was conducted to characterize the tensile strength and thermal properties of nanocellulose from *banana pseudostem* fibers and its composites. The results indicate that nanocellulose produced through chemical processes using sulfuric acid ( $H_2SO_4$ ), sodium hydroxide (NaOH), sodium chlorite ( $NaClO_2$ ), and sulfuric acid ( $H_2SO_4$ ) possesses tensile strength, thermal properties, and a rough surface texture. The reinforcement of *banana pseudostem* nanocellulose in polyester composites increased the tensile strength to 13.61 MPa in the 30:70 variation, heat resistance up to 358.97°C, and thermal conductivity of 0.1940 W/mK in the 20:80 variation. The surface morphology of the composites indicates strong bonding with minor undulations in the 30:70 variation. With solid bonding, good tensile strength, thermal conductivity, and thermal stability, nanocellulose-based bionanocomposites from *banana pseudostem* fibers have the potential to be used as thermal insulating materials in solar panels.

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