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EFFECT OF ALKALINE CONCENTRATION TREATMENTS ON THE CHEMICAL, PHYSICAL, AND THERMAL CHARACTERISTICS OF CELLULOSE FROM TAPIOCA SOLID WASTE

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Abstract. *Tapioca solid waste (TSW), as a source of natural fiber, is produced in abundance, but its utilization is minimal and even has the potential to pollute the environment. Unfortunately, TSW fiber has low physical, mechanical, and thermal characteristics that limit its application. Therefore, one way to improve the characteristics of TSW so that the fiber can be applied in various fields is by using the modified alkalization method. This study aims to determine the effect of alkali concentration on cellulose's chemical, physical, and thermal characteristics from TSW. Alkali treatment used NaOH solution of 0%, 5%, 10%, 15%, and 20% (v/w). The chemical characteristics of the fiber were analyzed for moisture, starch, lignin, hemicellulose, and cellulose content. Fiber surface morphology was analyzed by scanning electron microscopy (SEM), functional group changes with Fourier transform infrared (FTIR), degree of crystallinity with X-ray diffraction (XRD), and thermal stability with thermogravimetric analysis (TGA). The results showed that alkaline treatment affected changes in the chemical, physical, and thermal characteristics of cellulose from TSW. The increasing concentration of NaOH causes the water and cellulose content to increase, while the starch, hemicellulose, and lignin content decrease. Surface morphology is getting rougher, fiber dimensions increase to 10% NaOH concentration, but at higher concentrations, it causes a decrease in dimensions. FTIR analysis showed that the intensity of the hemicellulose and lignin functional groups decreased with increasing NaOH concentration. The degree of crystallinity and crystal size increased until the NaOH concentration was 10%, but at higher concentrations, it tended to decrease. Meanwhile, d-spacing increased with increasing NaOH concentration. The thermal stability of the fiber tends to decrease with increasing NaOH concentration. Alkalized cellulose from TSW has the potential to be used in a wider field, such as adsorbent and composite reinforcing agent.*

Keywords: *alkaline treatment; cellulose; cellulose characteristics; tapioca solid waste.*

1. Introduction

Currently, lignocellulosic waste has enormous potential to be utilized as a source of natural fiber because it is abundant and does not affect the availability of food crops [1]. Utilization of various types of lignocellulosic biomass sources as a source of natural fiber has been widely

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reported, such as corn husk [2], rice husk [3], sago frond [4], cocoa pod husk [5], and coconut husk [6]. In addition to these sources, in Indonesia, there is much tapioca solid waste (TSW) as a by-product of processing cassava into tapioca. Cassava is produced at 20-21 million tons/year and makes 20-30% of TSW [7]. So far, TWS has been used as an ingredient for bioethanol [7], briquettes [8], biogas [9], and animal feed [10]. TSW was also reported to have cellulose and lignin content of 20.60% and 4.08% [11], 19.27% and 4.11% [12], respectively. Thus, TSW has promising potential in providing natural cellulose fibers in large quantities with significant economic benefits, while its utilization is still limited.

Natural fibers have been widely used as an alternative raw material for synthetic fibers. Natural fibers have been applied as a matrix reinforcement agent for composite panels in the construction, infrastructure, and manufacturing industries [13]. However, the characteristics of natural fibers have weaknesses, including being hydrophilic, high water adsorption, less reactive, and less compatible with polymer matrices, thus affecting the mechanical characteristics of the composite [14]. This characteristic weakness of natural fibers can be corrected by modifying the fiber surface. Various modification methods have been reported by many researchers, such as alkalization [15], acetylation [16], acrylation [17], and permanganate [18]. However, among these methods, the alkalization method is more popular and widely applied because it is cheaper and has a lower negative impact on the environment than other chemical modification methods [1], [19]. The alkalization method has also been reported to increase the interfacial adhesion between natural fibers and the composite matrix [17].

Senthamaraikannan et al. reported 5% NaOH treatment on *Coccinia grandis*. L fiber increased the tensile strength of the composite from 273.0 to 316.3 MPa and thermal stability from 213.4 °C to 220.6 °C [20]. The 5% NaOH concentration in banana fiber gave the best fiber characteristics [21]. Using 0.5-8% NaOH solution on corn husk fiber increased the tensile strength from 224.05 to 368.25 MPa and the degree of crystallinity from 49.85 to 59.49% [2]. Using 10-15% NaOH concentration in bamboo fiber was reported to cause a significant increase in tensile and flexure strength. However, higher alkali treatment decreased the tensile and flexure strength [22]. The concentration of NaOH 0-0.7% in palm wood fiber can increase the tensile strength from 0.90-4.28 MPA, but 1% NaOH decreases its tensile strength [23]**.** The concentration of 4% NaOH in fiber jute can increase tensile strength up to 30% [24]. This study's results indicate that NaOH treatment concentration varies greatly, and there is no information on the optimum alkali concentration treatment to improve cellulose-based natural fibers' mechanical and thermal properties for composite applications.

Besides improving natural fibers' physical, mechanical, and thermal characteristics, alkali treatment with NaOH is also reported to be very effective in increasing fiber purity by degrading fiber impurities such as lignin, hemicellulose, and extractive compounds [25]. In general, the effect of qualitative alkali treatment has been reported to cause the fiber structure to swell and the surface morphology to become rough [20], [26]. However, based on the researcher's knowledge, quantitatively, the effect of variations in NaOH concentration on changes in fiber dimensions and fiber roughness levels, especially in TSW, has never been reported. In addition, structural changes, the distance between cellulose straight chains (d-spacing), and crystallite size of TSW fibers as supporting data on swelling and roughness levels have not been comprehensively reported.

Based on the problems described, this research was conducted to determine the effect of alkali concentration on the chemical, physical, and thermal characteristics of TSW cellulose. Chemical characteristics were analyzed for changes in water content, starch, lignin, hemicellulose, and cellulose. SEM analyzed the surface morphology, then determined the fiber surface profile, dimensions, and roughness. FTIR analyzed changes in functional groups. Changes in crystallinity degree, d-spacing, crystallite size by XRD, and thermal stability by TGA.

2. Methods

2.1. Materials

Research materials include NaOH (Merck), α-amylase enzyme (Novozyme), and distilled water. TSW was obtained from a tapioca factory in Tanah Baru Bogor, West Java. TSW was dried in the sun until the moisture content reached about 12%, then ground and filtered through a 40 mesh sieve.

2.2. Alkaline Treatment Process

This research was conducted through two stages: starch hydrolysis and alkaline treatment. Starch hydrolysis was carried out enzymatically; TSW (30% w/v) was gelatinized at a temperature of 95-100^{o}C. The α-amylase enzyme was added at a concentration of 1.2 mL/kg (600 U/mL), stirred periodically for 1 hour, and pressed to separate the fiber from the liquid fraction. Next, the TSW fiber was dried to a moisture content of about 10% w/w. Alkaline treatment was carried out using NaOH solution at various concentrations, namely 0% w/v (control), 5% w/v, 10% w/v, 15% w/v, and 20% w/v. TSW fiber (10 g) was added with 100 mL of NaOH solution according to the treatment, heated at 100^{o}C for 2 hours, and stirred periodically. Furthermore, the fibers were rinsed until the pH was neutral and dried using an oven at 50±2^{o}C for 24 hours to produce delignified fibers.

2.3. Fiber Characterization Analysis

2.3.1. Chemical Characteristics

The chemical characteristics of the fiber were determined by the method developed by Van Soest, consisting of Neutral Detergent Fiber (ADF), Acid Detergent Fiber (NDF), cellulose, hemicellulose, and lignin. NDF is an indigestible component consisting of cellulose, hemicellulose, and lignin, while ADF is a digestible component consisting of cellulose and lignin. Hemicellulose content is calculated by the difference between the content of NDF and ADF [27].

2.3.2. Scanning electron microscopy (SEM)

The surface morphology of TSW fibers from each treatment was characterized using SEM (Zeiss EVO MA10). The sample's surface was coated with a thin layer of gold with a sputtering time of 60 seconds and a sputtering current of 20 mA. Furthermore, the surface morphology of the fiber from the SEM image was analyzed for roughness using Image-J. Hardness is expressed by the Roughness average (Ra), as reported by Liu et al. [28].

2.3.3. Fourier Transform Infrared (FT-IR) Spectroscopy

The chemical functional groups of the samples were analyzed by Fourier transform infrared spectroscopy (FTIR Bruker-Tensor 37). Samples (2 g) were mixed with potassium bromide (KBr) until they reached a weight of 200 mg and pressed into pellet form. Measurements were made at wavenumbers from 4000-500 cm^{-1} with a resolution of 4 cm^{-1}.

2.3.4. X-ray diffraction (XRD)

XRD analysis was performed using Bruker Advance D8. The sample was irradiated with Cu Kα (λ=0.15406 nm) at 40 kV and 35 mA with a reflection symmetry geometry 2θ between 5-40^o with a step of 0.02^o. The degree of crystallinity was calculated by the method presented by Segal et al. [29].

$$CrI\ (\%) = 100 \times [(I_{200}-I_{am})/I_{200}]$$

I_{200} is the intensity of the reflection [200] (2θ=21-23^o), and I_{am} is the intensity of the amorphous portion of cellulose (2θ=18^o). The size of d-spacing cellulose was calculated using Bragg's equation, and the crystallite size was calculated using Scherrer's equation [30]. Bragg's equation is $N\lambda = 2d \sin\theta$, and Scherrer's Equation is $L = 0.9\lambda/(H \cos\theta)$. Where L is the crystallite size, is the X-ray wavelength (0.15418 nm), H is full width at half maximum (FWHM) in radians, and is Bragg's angle.

2.3.5. Thermogravimetric analysis (TGA)

Thermal stability was analyzed using Differential Thermal-Thermogravimetric Analyzers (TGA) Shimadzu DTG-60 Series. The sample (10 mg) was heated from 30 to 550ºC under a nitrogen atmosphere (50 mL/min) at a heating rate of 10ºC/min.

3. Results and Discussion

3.1. Fiber Chemical Composition

Treatment of alkali concentration on TSW fiber affects changes in the fiber's chemical composition. The chemical composition of the fiber during the alkaline treatment process at various concentrations of NaOH is presented in Table 1.

Table 1. Chemical composition of fiber by alkali treatment at various concentrations of NaOH

Chemical composition	Raw material	NaOH Concentration (w/v)				
		0%	5%	10%	15%	20%
Moisture	12.32	9.89	10.31	10.77	10.65	11.76
Starch	60.10	-	-	-	-	-
Lignin	2.53	25.21	17.65	10.98	6.84	5.27
Hemicellulose	8.89	24.54	12.09	11.3	4.72	3.35
Cellulose	10.04	35.45	44.38	55.57	66.31	65.49

Tapioca solid waste (raw material) contains starch up to 60.10 %, but the starch content was not detected in the fiber after the enzymatic hydrolysis process. It was suspected that the starch had hydrolyzed into glucose. The water content of the fiber tends to increase with the increasing concentration of alkaline treatment. Similar conditions also occur in fiber cellulose content. The cellulose content of the fiber with 20% w/v alkali treatment increased by 84.76%, while the lignin and hemicellulose content decreased by 79.10% and 86.35%, respectively the fiber without alkali treatment (0% NaOH). The water content increased due to the reduced hemicellulose and lignin content bound to the cellulose, followed by the fiber structure's swelling due to the effect of increasing alkali concentration. As a result, the free hydroxyl groups and the water adsorption ability were also higher. The higher the fiber cellulose content, the higher the alkali concentration, and the higher the ability to degrade lignin and hemicellulose. The content of cellulose, hemicellulose, and lignin from several lignocellulosic sources showed differences from the results of this study, such as sugarcane bagasse containing 36.32% cellulose, 24.7% hemicellulose, and 8.14% lignin [31], abaca fibers contain 56–64% cellulose, 25–29% hemicelluloses, and 11–14% lignin [32], agave contains 78.65% cellulose, 8.47% hemicellulose, and 4.65% lignin [19], and pineapple fibers contain 70 –82% cellulose, 44–60% hemicellulose, 8–13% lignin [21]. Differences in chemical composition are due to differences in fiber sources and treatment methods for cellulose insulation.

3.2. Surface Morphology

The alkali concentration increased from 0-5% w/v resulting in an increase in fiber diameter of 8.43% compared to no alkali treatment (0% NaOH). However, if the alkali concentration is increased to 20% w/v, the fiber diameter tends to decrease.

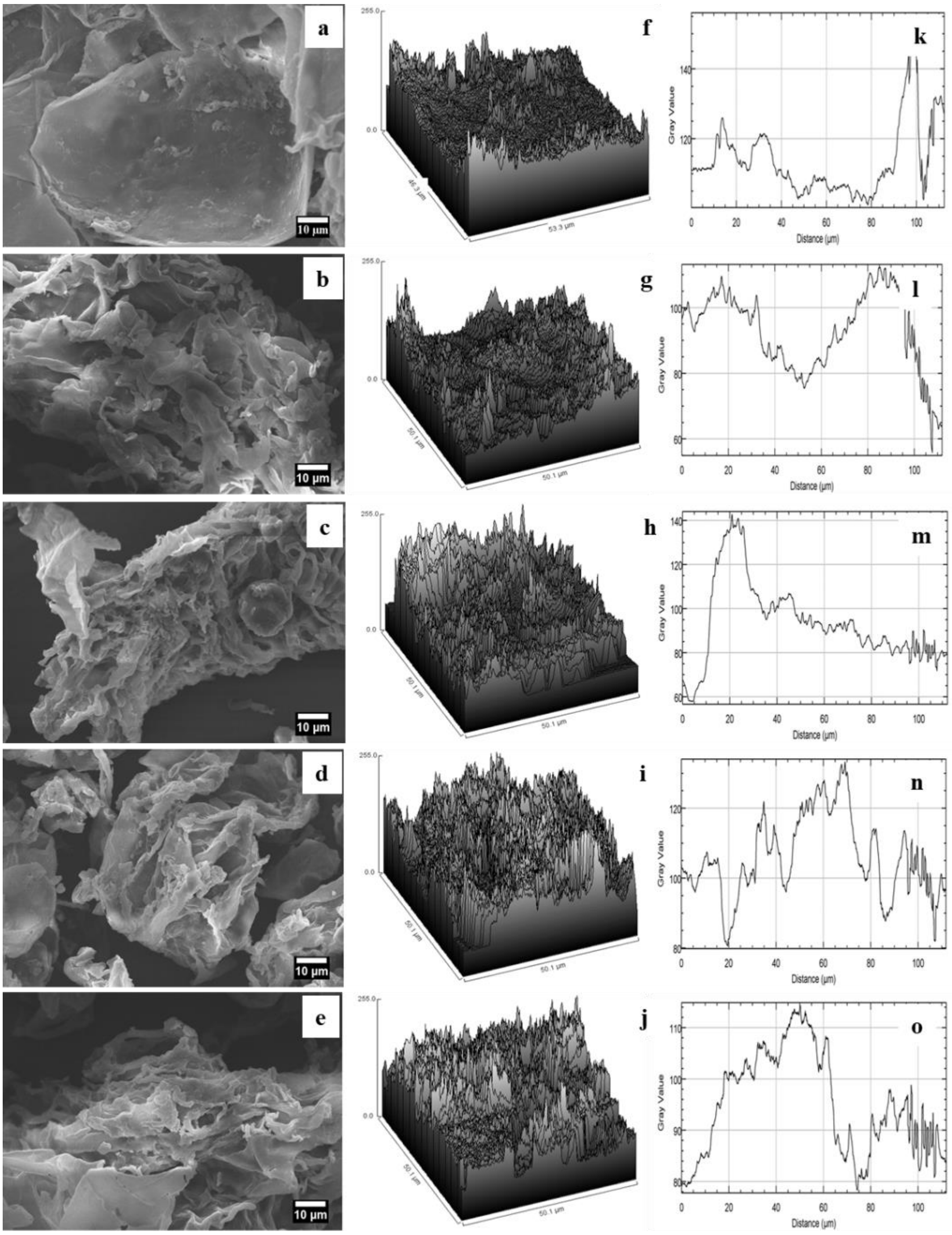

Figure 1. Fiber surface morphology by SEM analysis with 500 X magnification (a-e), surface plot (f-j), and roughness measurement (k-o) of the fiber at concentrations of NaOH 0%, 5%, 10%, 15%, and 20% w/v, respectively.

The alkali concentration treatment affected the changes in diameter and surface roughness of the fiber, as presented in Table 2. The alkali concentration from 0% to 5% is thought to only be able to swell the fiber structure so that the fiber diameter increases. Meanwhile, the alkaline concentration of more than 5% w/v not only caused the fiber structure to swell but also the fiber bundles were separated from each other due to the degradation of the lignin and hemicellulose structures as fiber binders and resulting in a porous fiber structure. The surface morphology of these fibers is confirmed in Figures 1a-e.

Table 2. The Diameter and Roughness (Ra) of fiber by alkali treatment at various concentrations of NaOH.

NaOH concentration	Diameter (µm)	Ra (µm)
0%	87.01	0.53
5%	94.35	0.93
10%	64.17	0.95
15%	47.59	1.06
20%	56.03	1.24

The degradation of the fiber structure is due to the ability of alkaline solutions to dissolve lignin, hemicellulose, pectin, and wax compounds [25]. Meanwhile, the increasing alkali concentration causes the fiber roughness value to increase, as shown from the surface plot profile in Figure 1f-j and fluctuations in the gray value of each image in Figure 1k-o. The higher the gray value, the higher the surface roughness of the fiber. This increase in roughness is caused by the structure of the fiber getting swollen and porous as the alkali concentration increases. This study's results align with the statement of Chen et al., which states that the dimensions of bamboo fiber increase at low concentrations and increase at higher concentrations [33]. It is also reported that the surface roughness of bamboo fibers increases with increasing alkali concentration [15].

3.3.Functional Group

The alkali concentration treatment affected the intensity of the FTIR spectra and the shift in the wavenumber of several fiber functional groups. The FTIR spectra of the fiber at various alkali concentrations are presented in Figure 2a. Changes in the spectra intensity were determined by calculating the difference between the intensity values of the spectra on the fiber with 0% alkali treatment against the intensity of the spectra with 5-20% alkaline concentration (Figure 2c). In general, the vibration of the hydroxyl group (-OH) of the fiber is located at a wave number of 3417 cm^{-1} [34]. The vibration of the C-H group with 0% alkali treatment appears at a wave number of 2931cm 1, but the 5-20% alkali treatment causes a shift in the wave number to a smaller direction, namely 2898 cm^{-1} [35]. The intensity of these spectra is increasing and getting wider, which indicates the fiber has more abundant hydroxyl groups. An increase also followed the increase in the intensity of the hydroxyl group spectrum at wave numbers around 1629-1647 cm^{-1} (Fig. 2b) [36].

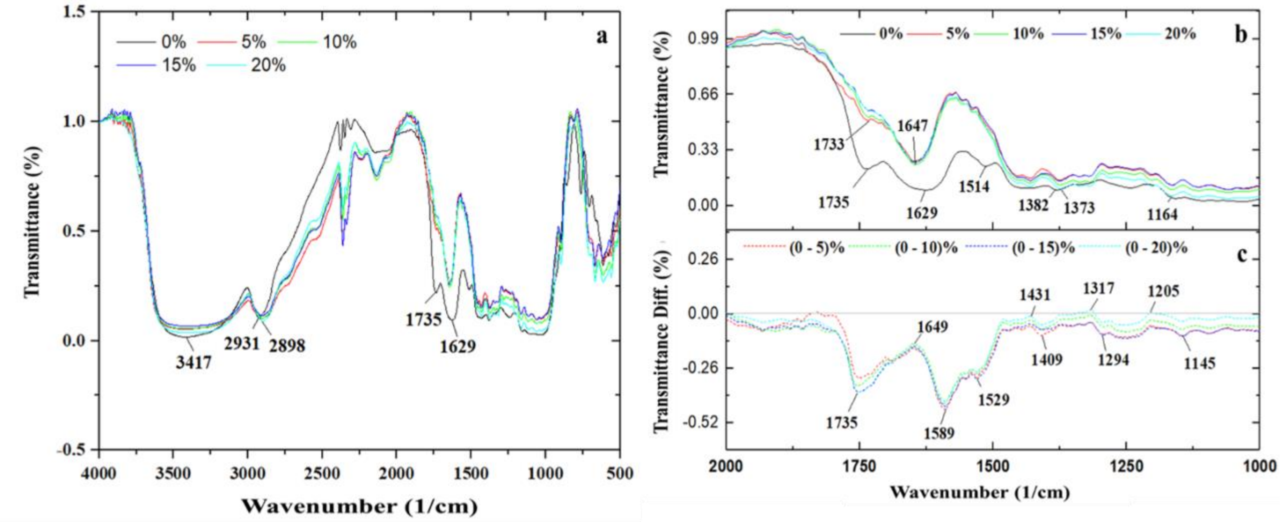

Figure 2. FTIR spectra at various concentrations of alkali treatment at wave numbers (a) 4000-500 cm^{-1}, (b) 2000-1000 cm^{-1}, and (c) intensity differences (transmittance diff.) spectra without alkali treatment with alkali concentration treatment.

Fiber with 0% alkali treatment had sharp peak spectra of 1735 cm^{-1}, 1629 cm^{-1}, and 1514 cm^{-1}. This peak is a stretching vibration of C=O acetyl and carbonyl groups in hemicellulose or carboxylate groups in lignin [37], [20]. The intensity of these spectra decreases and shifts to a wave number of 1733 cm^{-1} after the alkali concentration treatment is increased. The treatment with 20% alkali concentration had the highest intensity difference, which means that 20% alkali concentration had a higher ability to degrade lignin and hemicellulose than the lower alkali concentration. The decrease in hemicellulose and lignin content causes the purity of the fiber to be higher. This decrease is indicated by the difference in the spectra intensity at wave numbers 1431 cm^{-1}, 1317 cm^{-1}, and 1205 cm-1 [38], which is greater at 20% alkaline concentration. The peaks of 1164 cm^{-1} and 896 cm^{-1} are C-O-C stretches on the α-1,4 glycosidic bonds of cellulose [37], [12].

3.4.Degree of Crystallinity

Treatment of alkali concentration affects the degree of crystallinity of the fiber. The alkali concentration from 0% to 10% causes the degree of fiber crystallinity to increase but decreases after increasing the alkali concentration by more than 10%. In this condition, the structure of cellulose also changes from cellulose I to cellulose II.

Figure 3 shows that cellulose I has a crystallographic plane (1-10), (110), and (200) at 14.61^{o}, 16.99^{o}, and 22.50^{o}, respectively. Meanwhile, cellulose II has a crystallographic plane (1-10), (110), and (020) at 12.06^{o}, 1.91^{o}, and 21.70^{o}, respectively [39]. The value of d-spacing and crystallite size in the plane (200) also increased, then decreased in the plane (020) when the alkali concentration was more than 10%. The value of d-spacing, crystallite size, and degree of crystallinity at various alkali concentration treatments are presented in Table 3.

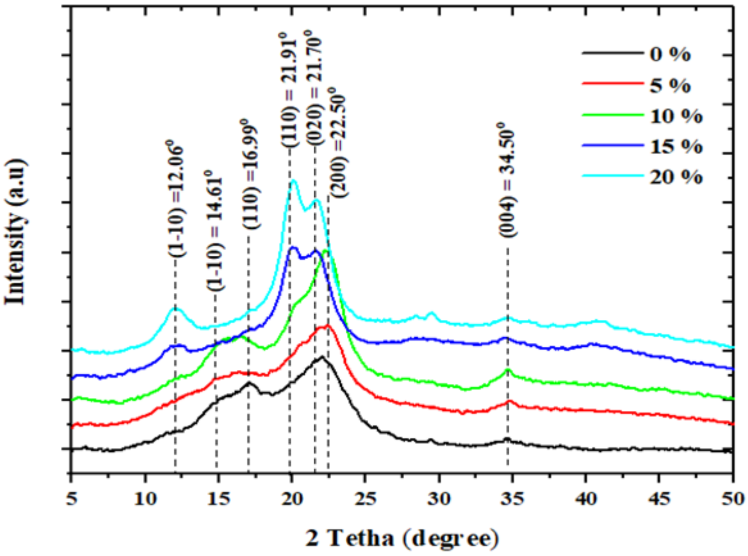

Figure 3. XRD spectra of fiber at various alkaline concentrations.

Table 3. Value of d-spacing, crystallite size, and degree of crystallinity of fibers at various alkali concentrations.

NaOH	d-spacing (nm)				Crystallite size (nm)				Crystallinity (%)
	(1-10)	(110)	(200)	(020)	(1-10)	(110)	(200)	(020)	
0%	0.61	0.52	0.39	-	1.9	1.44	1.7	-	35.10
5%	0.62	0.54	0.39	-	1.85	1.24	2.02	-	37.90
10%	0.60	0.54	0.41	-	2.24	1.59	3.12	-	40.10
15%	0.74	0.45	-	0.41	4.34	2.1	-	2.84	31.50
20%	0.73	0.45	-	0.41	8.1	2.81	-	2.45	30.90

The increasing d-spacing value indicates that the distance between the linear chains of cellulose fibers is getting wider [40]. This condition can be used to prove that the cellulose fiber has swelled due to alkaline treatment.

3.5.Thermal Stability

The alkali concentration treatment affects the thermal stability of the fiber. Figure 4a shows that the degradation of fiber components consists of 3 phases, namely the evaporation process of water content (30-150°C), degradation of cellulose and non-cellulose components (>150-425°C), and the oxidation process of decomposition of charcoal residue (>425°C) [12]. In the first stage, the fiber from the 15% and 20% alkaline concentration treatment experienced the highest degradation, with the maximum degradation temperature ranging from 79.89-91.65°C and weight loss of about 12-13% of the initial weight (Figure 4b).

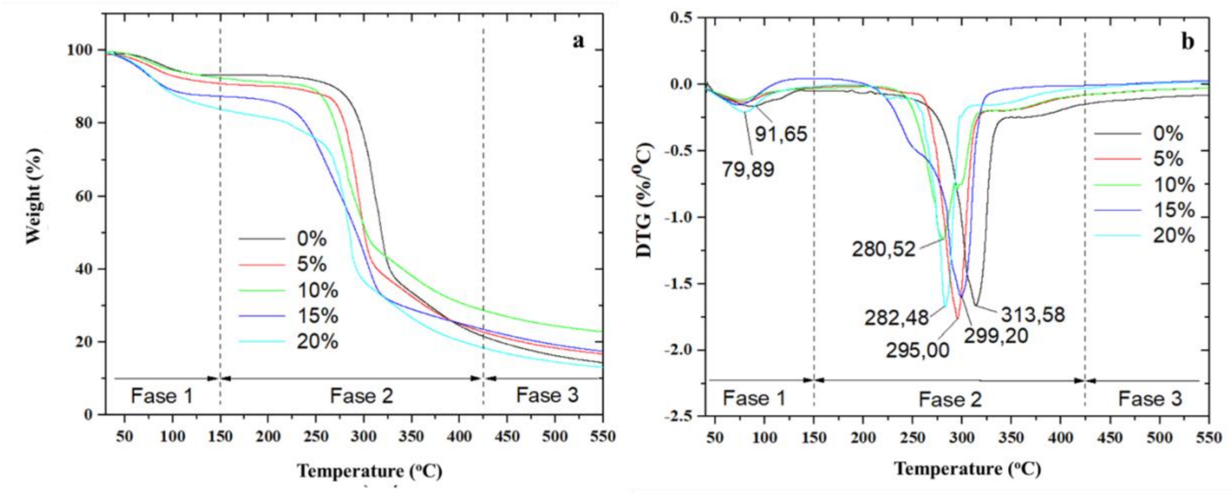

Figure 4. Thermal fiber stability at various alkaline concentrations by analysis of TGA (a) and Derivative Thermogravimetry-DTG (b).

The water content in the fiber is relatively higher, so it evaporates faster. The second phase is the degradation process of hemicellulose, cellulose, and lignin components, with the maximum degradation temperature varying for each fiber. Fiber from 0% alkali treatment had the highest degradation temperature of 313.58^{o}C with a weight loss of 42.51% due to the fiber's relatively high lignin content. Lignin has been reported to have higher thermal stability than cellulose and hemicellulose [6]. Meanwhile, 15% and 20% NaOH concentration treatment fibers had relatively lower thermal stability with relatively high weight loss, 55.05%, and 58.84%, respectively. The high weight loss in the fiber causes the charcoal residue produced at a temperature of 550^{o}C to be relatively low, as presented in Table 4.

Table 4. The initial temperature of degradation (T_{onset}), a maximum temperature of degradation (T_{max}), weight loss (WL), and charred residue (CR) of the fiber at various alkaline concentrations.

NaOH	Fase I			Fase II			Fase III
	T_{onset} (^{o}C)	T_{max} (^{o}C)	WL (%)	T_{onset} (^{o}C)	T_{max} (^{o}C)	WL (%)	CR (%)
0%	52.38	91.65	6.61	178.54	313.58	42.51	14.45
5%	42.73	80.02	9.01	158.06	295.00	41.1	16.87
10%	41.8	74.61	9.79	157.49	280.52	38.82	22.98
15%	42.22	73.68	12.57	152.03	299.20	55.05	17.61
20%	41.93	79.89	12.84	153.01	282.48	58.84	13.19

The high weight loss is due to the swelled cellulose structure and the low degree of fiber crystallinity. This result is in line with the statement of Lee et al., who reported that cellulose with a low degree of crystallinity and small particle size tends to have low thermal stability [37]. Each fiber material has different thermal degradation characteristics, such as the maximum degradation temperature of sugarcane bagasse fiber of 300.0^{o}C with weight loss. 13.5% [14], and bamboo at 335.5^{o}C with a low weight loss of 6.6% [41]. Differences in thermal stability can be influenced by different types and sources of materials, methods, and conditions of the insulation process, which ultimately affect the composition of chemical compounds, such as the content of cellulose, hemicellulose, and lignin.

4. Conclusions

Alkali treatment with various concentrations of NaOH on TSW fibers has been carried out and reported in this article. The results showed that the alkaline concentration treatment improved the chemical, physical, morphological and thermal characteristics of TSW fibers. The 10% NaOH concentration resulted in better TSW fiber characteristics than the 5% and 15% NaOH concentrations. The 10% NaOH concentration treatment reduced the hemicellulose and lignin content of TSW fiber by almost 60%, and increased the cellulose content by almost 60% in TSW fiber without alkali treatment. The diameter of the fiber decreased, while the roughness increased almost two times compared to the fiber without alkali treatment. The purity level of TSW fiber increased, which was indicated by the reduced intensity of the hemicellulose and lignin functional groups in the FTIR spectra. The d-spacing value increased from 0.39 to 0.41 nm, crystallite size from 1.70 to 3.12 nm, and crystallinity degree from 35.10 to 41.10%. However, the maximum temperature of degradation decreased from 313.58 to 280.52°C. Thus, alkali treatment has been able to improve the characteristics of TSW fiber so that this fiber has the potential to be applied in a very wide range of fields, such as as a reinforcing material in polymer composites, and as an adsorbent in waste water treatment.

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