

# Comparison of Alkali Concentration to Enhance Utilizability of *Musa Sapientum* (Banana) Fibers for Possible Textile Applications

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## ABSTRACT

The objective of the present research was to investigate the efficiency of alkali treatment in degumming of banana fibers. The fibers were exposed to alkali aqueous solutions at different concentrations changing from 5 to 20 wt%. The efficiency of the alkali treatments was analyzed with the help of optical observations, fiber diameter measurement, single fiber tensile test and determination of pectin component. The chemical, crystalline, thermal and morphological properties of the fibers were also examined. FTIR analyses proved the removal of non-cellulosic components such as hemicellulose and lignin after alkali treatment. Fiber diameter decreased with increasing alkali concentration. Tensile properties and crystallinity index are correlated with alkali concentration. However, the cellulose structure of the banana fiber was altered with alkali treatments at 15 and 20 wt% concentrations. Microscopic observations revealed the appearance of single elementary fibers from the fiber bundle of the banana. These experimental findings suggested that alkali treatment can play a promising role to prepare lignocellulosic fibers for textile applications.

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## KEYWORDS

Degumming, banana fiber, lignocellulosic fiber, alkali treatment, *Musa Sapientum*

## 1. INTRODUCTION

The research on the usability of new lignocellulosic fibers for textile manufacturing requires identifying of physical, morphological and chemical characteristics of fibers [1]. Increasing concerns about sustainability and ecological responsibility drive researchers to investigate a variety of natural fibers in many applications including textiles [2]. Low cost, less tool wear during processing, biodegradability and ease of recycling are the major advantages of plant fibers [3, 4]. Lignocellulosic fibers

exist in bundles which are agglomeration of fibrils cemented together with pectin at middle lamellae. Thus, the lignocellulosic fibers are coarse and stiff in nature due to their lignin and pectin components. Fiber extraction is an important step in fiber quality since it determines the final features of a product. After harvesting the plants, the retting process is focused on the extraction of fibers from the stem [5]. The most often utilized procedures are dew and water retting. These procedures rely on microbial decomposition of the organic materials as the basis for the fiber extraction method [5]. In order to perform yarn spinning and improve

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the usability of lignocellulosic fibers in textile applications, it is necessary the extract elementary single fibers (micro-fibrils) from the bundles. This process is called cottonization or degumming. The degumming process is a chemical process to remove non-cellulosic content and release elementary single fibers. To commercialize natural fibers, removing non-cellulosic content in fiber is essential [6]. The cottonization process involves removing sticky and viscous parts of fibers to increase the machinability of natural fibers. It is possible to blend fine lignocellulosic fibers or namely “cottonized fibers” with cotton, wool and other textile fibers to add new features for alternative end-uses. For this purpose, alkali, acid, enzyme, steam explosion, plasma and ultrasonic technique applications are implemented to lignocellulosic fibers [1, 7, 8]. Also, the elimination of non-cellulosic fibers can increase the crystallinity, and tensile strength of natural fibers [9, 10].

Natural fibers including banana, jute, sisal and coir have been used to produce yarns, ropes, mats, matting hangings, tablemats, handbags, and purses for many years [11]. Due to promising properties of Banana fibers such as resistance to marine and UV conditions, lightweight, and high specific strength, they have been used extensively in many application areas such as textile, pulp, and paper industry, construction, and building industry, packing industry, and reinforcement material for composites [12]. It is known that the banana is one of the oldest culture plants among natural fibers. Banana belongs to the Musaceae family and Musa genus and the ‘banana’ word means ‘finger’ in the Arabic language [13]. Almost 120 million tons of banana fruits were produced in 2020 [14]. Banana is one of the major food crops in the world after rice, wheat, and maize with an average banana consumption of 12 kg per capita [15]. Banana fruit consists of two parts such as pulp and peel. The peeling part is about 40% of the total weight of the banana fruit. The banana peel was dumped as waste until recently, requiring high amounts of organic materials to be managed. Therefore, researchers have begun to focus on several possible applications including banana fiber-reinforced composites have emerged [11, 13, 15-19]. Developing the properties of banana fibers is very valuable in terms of their waste management. Due to their properties and availability, their composites have attracted attention in many fields such as automotive, aerospace, sports equipment, packing, and construction [20-23].

In recent papers, the cottonization of cellulosic fibers such as flax, hemp and pineapple leaf fibers via steam explosion, ultrasonic cavitation and decortication have been studied [1, 6, 7, 24, 25]. There are available resembling studies about alkali treatment on fibers from banana. Osorio et al. (2012) treated banana fibers with 5% NaOH solution at room temperature [26]. Vishnu Vardhini et al. (2019) modified banana fibers with aqueous NaOH solutions at 10, 15 and 20% concentrations at 80°C for 3h [27]. Parre et al. (2020)

treated the fibers from banana with 1, 3, 5, 7 and 9% concentrated sodium hydroxide solutions for 24h at room temperature [28]. Twebaze et al. (2022) performed degumming process for banana fibers via alkali treatment with the help of ultrasonic method [29]. Apart from these investigations on alkali treatment, 5 different alkali concentrations were implemented and pectin contents of the untreated and treated fibers from banana fibers were determined in this current study. The latest research was performed by Subash and Perumalsamy (2022) which consists degumming of banana fiber via enzyme treatment in green concept [30]. Degumming technique was also investigated for different types of cellulosic fibers. [31-33]. The primary interest of this current study is focused on enhance properties of fibers from banana to be utilizable for possible textile applications. Therefore, the purpose of this research to implement alkali treatment at different sodium hydroxide concentrations on banana fibers for degumming. The effect of alkali treatments on the physical, chemical, mechanical and morphological properties of the banana fiber was reported. On this basis, Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) analyses were performed. The efficiency of treatment is also elucidated by the determination of fiber diameter, pectin content, fiber tensile performance and morphological properties.

## 2. MATERIAL AND METHOD

### 2.1 Material

The fibers which were extracted from banana plants were provided from Antalya located in the southern part of Turkey. Commercial grade sodium hydroxide granules and EDTA were supplied from Merck Corp.

The fibers were cleaned with distilled water in two stages; for 4h at 40°C and then 20h at ambient temperature. Then, the fibers were oven-dried for 4h at 90°C. As an alkali treatment, the fibers were degummed with aqueous sodium hydroxide solutions at 5 different concentrations as given in Table 1 at 70°C for 30min. Then, the alkali-treated fibers were rinsed for several times with de-ionized water to neutralize. Finally, the fibers were exposed to the same drying process as implemented in the cleaning process.

**Table 1.** Sample codes according to the NaOH concentration

Sample code	NaOH concentration (wt %)
F1	0-Untreated
F2	5
F3	10
F4	12.5
F5	15
F6	20

## 2.2 Method

### 2.2.1 Fiber diameter

The diameter of the untreated and treated fibers was measured by using an optical method with Olympus BX 43 optical microscope. At least 30 readings were taken for each sample to measure the diameter at the magnification level of 20X and check for repeatability.

### 2.2.2 Tenacity

The tenacity of the untreated and treated fibers was measured by INSTRON 4411 universal testing machine equipped with 1 kgf load cell. The fibers (bundle) were tested at 20mm gauge length with 5mm/min loading rate. Pneumatic grips which were used for clamping the fibre have a pressure of 0.5 MPa pressure. All tests were performed at standard atmospheric conditions ( $20 \pm 2$  °C ambient temperature and  $65 \pm 4\%$  relative humidity) in accordance with ASTM D 3822 standard.

### 2.2.3 Determination of pectin component

Before the analysis of pectin content, the untreated and alkali treated fibers were dried at 100°C for 4h and then kept in a desiccator for half an hour and weighed ( $w_0$ ). The fibers were exposed to 0.5% w/v EDTA solution for 30min via using a magnetic stirrer at boiling temperature. After completing the process, the fibers were cleaned with de-ionized water, oven-dried at 100°C for 4h and then weighed ( $w_1$ ). The pectin content was computed by using the following formula (Eq.1) [17]:

$$\text{Pectin content} = \frac{w_0 - w_1}{w_0} \times 100 \quad \text{Equation (1)}$$

### 2.2.4 Fourier transform infrared spectroscopy (FTIR) analysis

Fourier Transform Infrared (FTIR) analysis was used to identify the effect of alkali treatments on functional groups of banana fibers with the aid of the Perkin Elmer Spectrum BX instrument. Data were recorded with a scan rate of 40 scans per minute and at a resolution of  $2 \text{ cm}^{-1}$  in the range of  $650\text{--}4000 \text{ cm}^{-1}$  wavenumber.

### 2.2.5 X-ray diffraction (XRD) analysis

The crystallinity of the untreated and treated fibers was examined with the help of the data obtained from X-Ray diffraction by using Rigaku Ultima 3 device with Cu-K $\alpha$  radiation. XRD device was set to 40 kV and 30 mA power, and scanning was done between  $5^\circ$  and  $65^\circ$  range with a scan rate  $2^\circ/\text{min}$  scan rate. The crystallinity index (CI) was determined by using the empirical formula (Eq.2) by Segal et al. [34].

$$CI = \left( \frac{I_{200} - I_{am}}{I_{200}} \right) \times 100 \quad \text{Equation (2)}$$

where  $I_{200}$  represents the peak at the maximum intensity that relates to the (200) lattice plane between  $22^\circ$  and  $23^\circ$ , and  $I_{am}$  is the minimum intensity value between the highest two peaks, which is at a  $2\theta$  angle between  $18^\circ$  and  $19^\circ$  [26, 27].

### 2.2.6 Thermogravimetric analysis (TGA)

To investigate the effect of alkali treatment on the thermal stability of the fibers from banana, thermogravimetric analysis (TGA) was performed by using a Shimadzu DTG-60H instrument. The investigation was performed from room temperature to 800 °C at a rate of 10 °C/min. To prevent oxidation, the analysis was conducted under a nitrogen atmosphere with a flow rate of 100 ml/min.

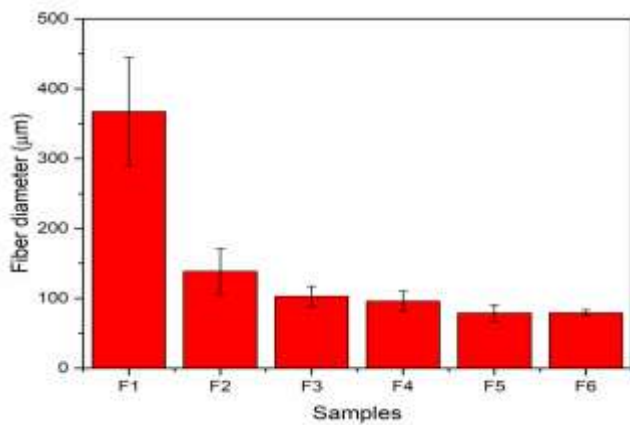
### 2.2.7 Scanning electron microscopy (SEM) observation

The morphological structure of the untreated and treated fibers was examined by FESEM FEI Nova Nanosem 650 model scanning electron microscope (SEM). SEM images were taken with an accelerating voltage of 5 kV. Prior to the examination, the fibers were coated with Au-Pd alloy by sputter coating.

## 3. RESULTS AND DISCUSSION

### 3.1 Fiber diameter

Fiber diameter is a vital parameter in spinning yarns. It plays a determinative role in the appearance and properties of textile materials in any form such as fiber, yarn and fabric. Fiber diameter can be used to realize the efficiency of the treatment on the extraction of fine fibers. Figure 1 depicts the fiber diameter values of the untreated and alkali-treated banana fibers. The diameter of the untreated banana fiber displayed a broad range of data. The fiber diameter of the untreated fiber dramatically dropped off with alkali treatment. The fiber diameter presents decreasing tendency with increasing alkali concentration. For the treated fibers at 15 and 20 wt% concentration, a narrow range of diameters can be observable in Figure 1. For the fibers treated with alkali at concentrations lower than 15 wt%, fiber diameter decreased probably due to the partial removal of non-cellulosic components. However, with increments in the concentration, fiber fineness came to exist with possible degradation of fiber integrity, which can release individual fibers [5, 33]. Twebaze et al. [29] accomplished 70.1% decrease in fiber diameter fiber from banana as an alkali treatment at 10 wt% concentration for 3h at 95°C. In the paper of Subash and Perumalsamy [30], enzyme degumming decreased fiber diameter of banana pseudemstem fiber from 1.11 mm to 1.02mm. In our study, we succeeded 72 and 74% decrease in fiber diameter of fiber from banana at 70°C for 4h, respectively.



**Figure 1.** Fiber diameter charts of the untreated and treated fibers from banana

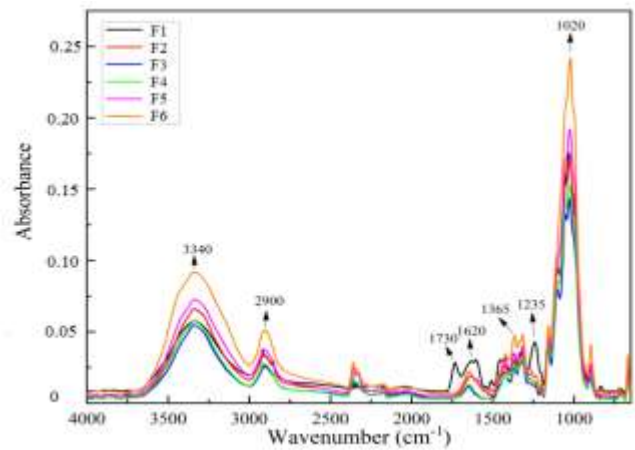
### 3.2 FTIR analysis

Figure 2 presents FTIR spectra of the untreated and treated fibers. Both the untreated and treated banana fibers presented the typical vibration bands of cellulose, hemicellulose and lignin [17]. The strong peaks at around 3500-3300 and 2900  $\text{cm}^{-1}$  can be correspond to intermolecular bonded hydroxyl (O-H) groups and C-H stretching vibration from the  $-\text{CH}_2$  group of cellulose and hemicellulose [35-38]. The bands at around 1730 and 1235  $\text{cm}^{-1}$  exhibit the presence of hemicellulose and lignin [39]. The peaks disappeared after alkali treatment due to the removal of these components [40]. The peaks observed at the absorption bands of 1630-900  $\text{cm}^{-1}$  are attributed to cellulose [38]. The intensity of the weak peak at around 1500  $\text{cm}^{-1}$  which is attributed to the C=C group stretching vibration of the carbonyl groups in hemicellulose decreased after alkali treatments. The intensity of the peaks at 1365, 1110 and 898  $\text{cm}^{-1}$  can be associated with the bending of C-H linkage, the vibration of ether groups (C-O-C) and the rocking vibration of C-H bonds, respectively, relating the native cellulose increased after alkali treatment [41]. Further, maximum intensity can be observed in F6 fiber which is coherent with chemical composition data [42]. The peak located at 1020  $\text{cm}^{-1}$  can be attributed to the skeletal vibration of the C-O-C pyranose ring and the intensity of this peak was the highest for F5 and F6 fibers [42]. This may be due to changes in the cellulose structures of these fibers.

### 3.3 Pectin analysis

Pectin is an adhering agent which exists in the primary cell wall of the plant and the fiber. It holds and binds up the microfibrils in the fiber which shows up in bundle form in lignocellulosic fiber. The main component of the gummy substances is pectin which is a very complex polysaccharide that behaves as an intercellular adhesive. As a result of alkali treatment, pectin can form lower molecular weight components which can dissolve in hot alkali solutions [43]. To perform the degumming of the hard

fibers, it is essential to remove the pectin component. To assess the efficiency of alkali treatment on pectin removal, the pectin content of the untreated and treated fibers was determined. The pectin fractions of the untreated and treated fiber from banana are given in Table 2. Alkali treatment is effective to remove non-cellulosic components such as hemicellulose, lignin, pectin and other components [44]. Therefore, the pectin constituent of the fibers from banana decreased after alkali treatment. The pectin content further decreased with increasing alkali concentration. However, the value increased after the treatment at 15 and 20 % alkali concentrations. The amorphous components of the fibers from banana which increased after treatments with 15 and 20 % alkali concentrations may be removed in addition to the pectin constituent. This possibly increased the weight loss of the fiber.



**Figure 2.** FTIR spectra of the untreated and treated fibers from banana

**Table 2.** The pectin contents the untreated and treated fiber from banana

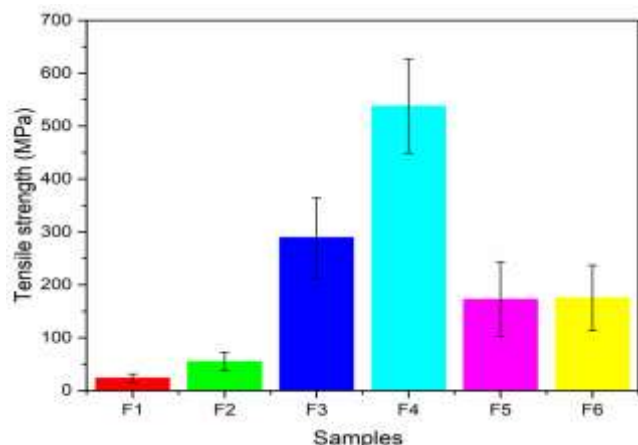
	Pectin content (%)
F1	10.50
F2	7.26
F3	2.70
F4	1.65
F5	4.44
F6	3.11

### 3.4 Tensile properties

Figure 3 presents the tenacity of the untreated and treated banana fibers. The tenacity of the untreated fibers initially increased with increasing alkali concentration but then decreased after the treatment with the 12.5 wt% alkali solution. Besides, the partial removal of the non-cellulosic components allows fibrils to rearrange along the tensile direction which may increase the tenacity [45]. Additionally, removal of these non-cellulosic components may increase the length/diameter ratio values which can enhance the tenacity of the banana fibers. At higher alkali concentrations, the change in fiber crystallinity and crosslinked network through hydrogen bonds between cellulose and non-cellulosic components may deteriorate fiber mechanical properties [46]. Thus, the loosely bound structure of fiber can result in higher elongation data (Table 3). Tensile properties of the banana fibers are consistent



with XRD data. Regarding to the tensile data, the banana fibers alkali-treated at 12.5 wt% concentration with fiber diameter lower than 100 $\mu$ m have a moderate textile grade quality and can be yarn spinnable [33, 47].

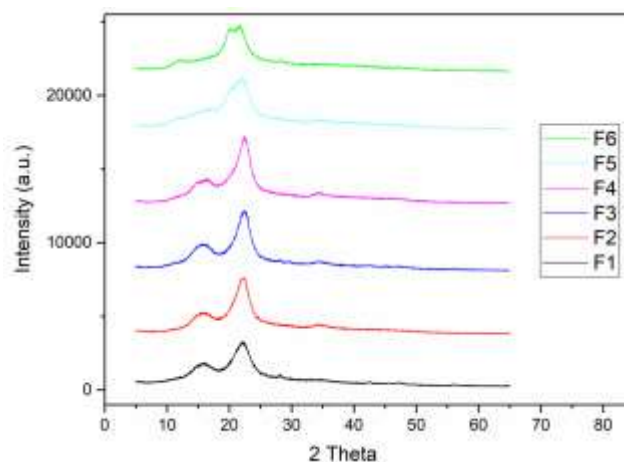


**Figure 3.** Tensile strength chart of the untreated and treated fibers from banana

### 3.5 XRD analysis

Alkali treatment of cellulosic natural fibers can remove non-cellulosic materials in an amorphous structure. Therefore, alkali treatment highly influences the crystallinity of the fibers [48]. Accordingly, the crystallinity of the banana fibers was investigated by XRD analysis and the graphs were drawn as shown in Figure 4. Two major peaks were identified for the untreated banana fiber at  $2\theta=22.5^\circ$  and  $15.6^\circ$  which are attributed to cellulose I structure with 110 and 200 crystalline phases, respectively. The untreated fibers and treated fibers treated with 5-12.5 wt% alkali concentrations represent a similar diffractogram. Then, the difference in the peaks became prominent for F6 banana fiber which was treated with 20% concentrated alkali solution. Three peaks were recorded for F6 fibers at  $2\theta=12^\circ$  (1-10),  $20^\circ$  (110), and  $22^\circ$  (200) which indicate cellulose II structure [49]. The alkali treated at high concentrations showed a transformation from cellulose I to cellulose II in crystalline structure [50-52]. The crystallinity index values of F1, F2, F3, F4, F5 and F6 fibers are computed as 58.6, 67.3, 68.0, 68.3, 48.7, 41.3%, respectively. The crystallinity index of the fibers increased with increasing alkali concentration until 12.5% and then it started to drop off. The maximum increment and maximum decrement in crystallinity index by 16.6 and 29.5% were recorded for F4 and F6 fibers. Alkali treatment can improve

the crystallinity of the banana fibers via removing amorphous components leading an increment in crystalline content [33]. However, alkalization at high concentrations can deteriorate the crystallinity of the banana fibers possibly due to the modification of cellulose structure from cellulose I to cellulose II (Figure 4).



**Figure 4.** XRD graphs of the untreated and treated fibers from banana

### 3.6 Thermogravimetric and derivative of thermogravimetric analysis (TGA/DTG)

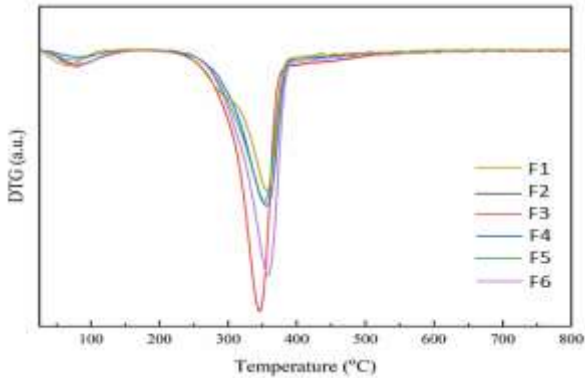
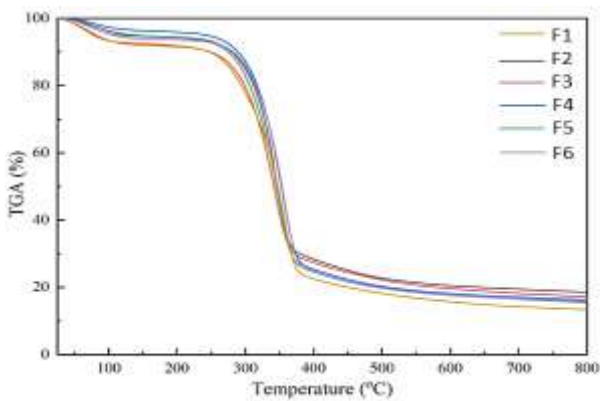
TGA/DTG of the untreated and treated banana fibers are presented in Figure 5 and Figure 6, respectively. Thermal degradation data derived from TGA are listed in Table 4. The first mass loss is recorded in the region of 2.95-6.98 % due to the dehydration of the banana fibers [53].  $T_1$  is the temperature at which hemicellulose thermally decomposes.  $T_1$  is also determined as the thermal resistance temperature [36]. The untreated and treated fibers showed thermal resistance temperatures between 171.28-183.77 $^\circ$ C. This may be due to the removal of hemicellulose and lignin after alkali treatments which plays a vital role on thermal degradation [35]. The major mass loss which shows a distinct peak occurred due to the decomposition of cellulose [54]. The maximum thermal degradation temperature decreased from 356.1 to 345.9, 339.3, 346.5 and 352.3 $^\circ$ C after alkali treatment at 5, 10, 12.5, 15 and 20 wt%, respectively. Removal of alkali-soluble agents may alter the thermal stability of the fiber from banana [52]. The char residue content of the untreated fiber which was measured as 13.35 % increased after all alkali treatments.

**T able 3.** The tensile properties of the untreated and treated fibers from banana

	Tensile strength (MPa)	Elongation at break (%)	Young's modulus (GPa)
F1	23.77 ± 7.87	6.99 ± 2.28	0.37 ± 0.16
F2	55.00 ± 17.66	7.44 ± 2.54	0.75 ± 0.12
F3	289.16 ± 75.67	10.89 ± 2.98	2.79 ± 2.05
F4	537.88 ± 89.08	19.85 ± 4.71	2.81 ± 0.71
F5	172.70 ± 70.57	20.13 ± 1.80	0.88 ± 0.44
F6	175.17 ± 61.91	13.17 ± 5.66	1.55 ± 0.79

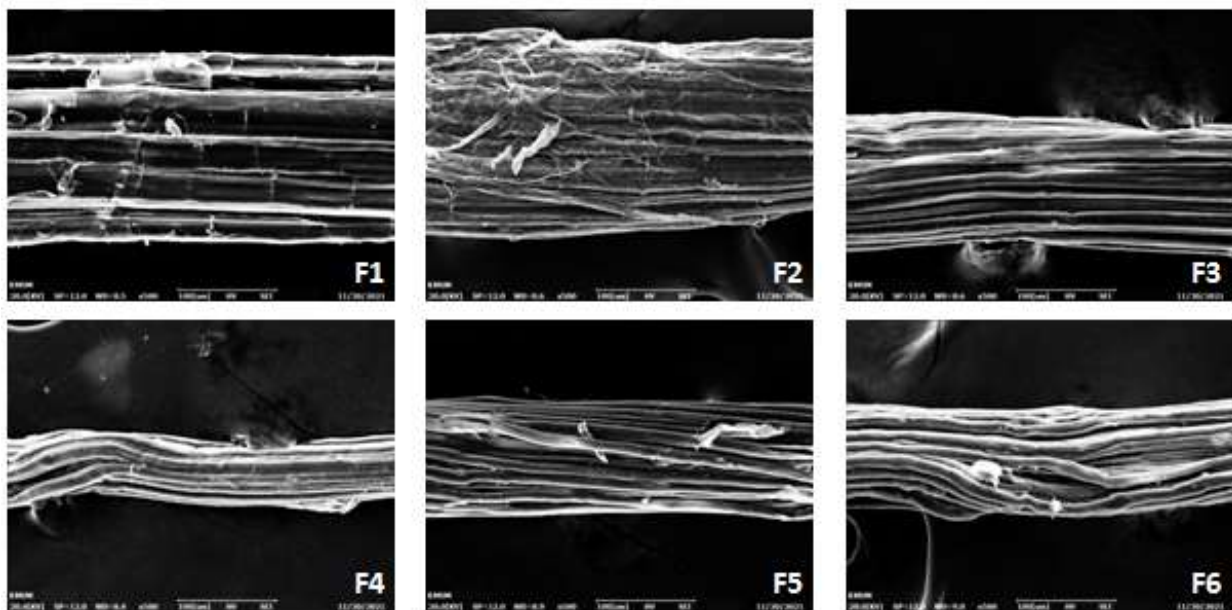
**Table 4.** TGA data of the the untreated and treated fibers from banana

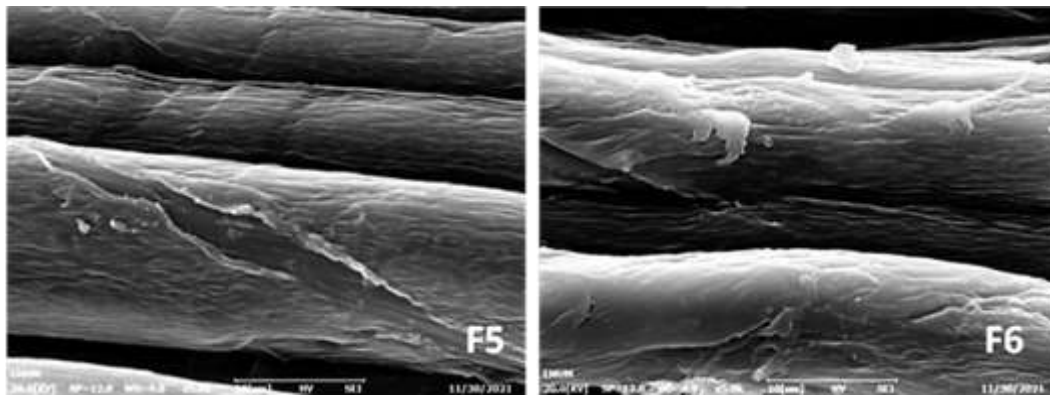
	Mass loss until 110°C (%)	T <sub>1</sub> (°C)	T <sub>max</sub> (°C)	Mass loss at T <sub>max</sub> (%)	Char Residue at 800°C (%)
F1	6.92	183.06	356.13	58.68	13.35
F2	4.23	183.51	345.89	49.03	18.58
F3	6.98	183.40	339.25	47.01	17.30
F4	2.95	177.27	346.47	43.06	16.29
F5	4.75	171.28	352.27	54.07	15.44
F6	5.14	183.77	357.96	54.97	15.87

**Figure 5** TGA graph of the untreated and treated fibers from banana**Figure 6.** DTG graph of the untreated and treated fibers from banana

### 3.7 SEM analysis

The changes on the surface of the banana fibers are visualized by SEM images which are presented in Figure 7. The impurities and non-cellulosic substances such as waxy and fatty acids are noticeable on the outer surface of the banana fiber. Besides, the surface of the fiber is smooth and uniform. It is determined that the fiber from banana like other lignocellulosic fibers exhibits a fiber bundle structure consist of several elementary fibers like fibrils bonded together with pectin and non-cellulosic components [2, 55]. The alkali treatment at 5 wt% concentration partially removed surface impurities (Figure 7). The treated fiber had a rougher surface and appeared jagged. However, a thin layer covering the surface of the fiber is still detectable and elementary fibers can be observed for F2 fiber. With increasing concentration, elementary fibers became more visible. Cleaner surfaces were obtained as a result of treatment at 10 wt% concentration. Fibers treated with stronger alkali solutions (12.5 wt %) present separation of elementary fibers from the bundle. For F5 and F6 fibers, the integrity of the bundle was quietly deteriorated reasonably by weakening the bond between fibrils [17, 39]. The fibers were separated into small units of fibrils. Additionally, surface peeling and engravings are observable on the surface of the fibers treated with alkali at 15 and 20 wt% concentrations in Figure 8 [56].

**Figure 7.** SEM images of the the untreated and treated fibers from banana (x500 magnification)



**Figure 8.** SEM images of the banana fibers treated at 15% and 20% concentrations (x5000 magnification)

#### 4. CONCLUSION

Industrialization of natural cellulosic fibers requires removing non-cellulosic components and separation of the fiber bundles to be utilizable in textile applications which can be performed by degumming. Via this method, the fibers with appropriate fineness and tensile strength can be blendable with short fibers to be spun into textile yarns. Accordingly, we investigated the effect of alkali treatment on degumming of banana fibers. Different concentrations were implemented for the alkali treatment of the fibers. Alkali treatment is selected due to its high effectiveness in the removal of non-cellulosic components from the fiber. The fiber diameter decreased with alkali treatment possibly due to the bonding materials and non-cellulosic constituents from the fiber. The pectin content significantly decreased with alkali treatment. However, it increased after the alkali treatment at 12.5% concentration. This may be due to the removal of some other amorphous materials based on the change in the fiber structure. SEM micrographs proved the removal of non-cellulosic fractions, separation of fine elementary fibers from the bundle and deterioration of the

fiber surface at higher alkali concentrations. Alkali treatment increased the crystallinity index and mechanical properties up to 12.5% alkali concentrations. Application of alkali solutions at 15 and 20 wt% concentrations altered cellulose allomorph and deteriorated crystalline structure of the banana fiber. According to all-experimental data recorded in this study, 12.5 wt% is determined as the optimum concentration for alkali treatment at 70°C for 4h for the banana fibers in this current study. The fiber from banana has app. fiber diameter of 100µm, Young's modulus of 2.81GPa and elongation at break of 20.13% can indicate its usability in textile applications such as manufacturing of sail and tent clothes, weft yarns in carpets and ropes and etc. The purpose of the degumming process is to obtain thinner, cleaner, flexible and strong fiber sufficient to tensile forces in yarn spinning processes. In a further study, treatment temperature, treatment duration and auxiliary agents can be examined as affecting parameters for obtaining finer and more qualified cellulosic fiber for possibly manufacturing of apparels and home textiles textile applications.

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