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Original Paper

Effects of chemical treatments on morphological, physical, and chemical properties of okra (*Abelmoschus esculentus*) bast fibers

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ABSTRACT

In this study, bast fibers have been obtained from okra (*Abelmoschus esculentus*) plant stems via biological degumming process. The obtained fibers were subjected to some textile pretreatment processes such as scouring, alkalizing, oxygen bleaching and chlorine bleaching which are also utilized in cellulose nanoparticle production. Effects of these processes on their physical and chemical characteristics have been investigated and statistically analyzed. Treatments have been found to statistically affect physical properties of the fibers. They led to decrease in fiber linear density but increase in moisture content and water absorption capacity. Linear density reduction took place by fibrillation and elimination of impurities. Fibrillation has been evidenced by microscopy images. Prolonged submerging resulted in increment in water absorption. FT-IR spectra, SEM images and mass loss analysis present evidence for removal of lignin, hemicelluloses and vaxes upon chemical treatments. The ranges of the linear density, moisture content and water absorption of okra bast fibers are 6.3-20.1 tex, 2.9-6.0%, and 5.80 g/g to 11.8 g/g, respectively. Properties of okra bast fibers show similarities to conventional bast fibers such as flax, hemp and jute.

I. INTRODUCTION

Last decades, production industries have started to give prime importance to environmental sustainability. Biobased materials including natural fibers have been the subject of interest as an alternative to synthetic materials. Consequently, considerable demand has been formed for cellulosic fibers [1]. Among plant fibers, agro-residual fibers carry great opportunity due to the fact that they are renewable, biodegradable, cost efficient and readily available in vast regions of the world. Since they are produced as a byproduct of edible crop agriculture, they do not need extra arable land. Their utilization benefits agricultural economy, as well [2, 3].

Although traditional bast fibers were grown in Asia minor for around 4 millennia, the yearly production of flax fiber, which is the most common bast fiber, has decreased to 3 tons (2017) from 3700 tons (1969) there, because it could not compete with cotton and economic synthetic fibers [4]. Agro-residual biomass may be used to cater plant fiber demand which has been formed in these last decades. Studies continue to obtain fibers from residues of a number of agricultural plants including banana [5, 6], corn [7, 8], and okra [9, 10].

Within agricultural crops, okra takes an important role. Turkey was the 16th okra producer of the world in 2020 with 40,654 tons. Total okra production of the World that year was over 10.5 million tons. Over 75.7% of world okra production was carried out by low-income countries [11]. Moreover, residues of okra give bast fibers with properties like that of flax, hemp and jute [12].

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Effects of various chemical treatments on okra bast fibers have been studied in literature [7, 13, 14]. Khan et al. [13] investigated effects of $NaClO_2$ bleaching, acrylonitrile grafting and 10% NaOH alkalization on IR spectra, water absorption and tensile properties of okra bast fibers. They reported that alkalization was more effective in removal of extra cellulosic components, while acrylonitrile grafting led to highest tensile strength with lowest water absorption. Khan et al. [14] applied alkalization, $NaClO_2$ bleaching, maleic anhydrite and vinyl acetate treatments on okra bast fibers. They reported the lowest moisture content to belong to vinyl treated fibers, the greatest initial modulus to bleached fibers whereas the highest elongation at break to alkalized fibers. Khan et al. [7] applied alkalization at different concentrations of NaOH on okra bast fibers at boiling conditions. They found increase and then decrease of initial modulus, breaking tenacity, and breaking elongation as the concentration increases. They concluded the best concentration to be 3 g/L NaOH.

This study investigates effects of different chemical treatments such as scouring, alkalization, H_2O_2 oxygen bleaching and NaOCl chlorine bleaching which are commonly applied to cellulosic textiles for elimination of extra cellulosic substances and impurities, in addition to forming steps for cellulosic nanoparticle preparation. The innovative part of this study is investigating the effects of these chemical treatment steps on okra bast fibers with parameters suitable to obtain nanoparticles which have not been studied in the literature before as far as known by the authors. Effects of the chemical treatments on fiber morphology, linear density, moisture content, water absorption properties and FT-IR spectra have been investigated.

II. EXPERIMENTAL METHOD / TEORETICAL METHOD

2.1 Materials and Preparation Techniques

Okra plant (*Abelmoschus esculentus*) stems were collected from farms in Aegean Region in late autumn and were biologically degummed in aqua for 2 months in winter to obtain *raw* fibers. All chemicals used in this research were of analytical grade. Different chemical processes have been carried out in order to remove extra cellulosic components of okra bast fibers. A fraction of the raw fibers was scoured using 5 mg/L Na_2CO_3 and 5 g/L detergent at 75 °C for 40 min to produce *scoured* fibers. Some of the scoured fibers were treated with 17.5% NaOH at 34 °C for 2 h to get *alkalized* fibers. A portion of the alkalized fibers were bleached using 2% H_2O_2, 3.5 g/L Na_2CO_3 and 1.5g/L NaOH at 90 °C for 1 h to obtain oxygen-bleached (*o-bleached*) fibers. Some of the o-bleached fibers were bleached with 5% NaOCl for 90 min at 80 °C to get chlorine-bleached (*c-bleached*) fibers. 1:50 liquor ratio was adopted for all treatments which have been carried out using magnetic hot stirrer at 200 rpm and distilled water was used for preparation of all mentioned chemical solutions. Sample names and the treatment steps they have undergone are listed in Table 1 in consecutive order.

Table 1. Sample names and the treatment steps they have undergone in consecutive order

Sample name	1st treatment	2nd treatment	3rd treatment	4th treatment
Raw	-	-	-	-
Scoured	Scouring	-	-	-
Alkalized	Scouring	Alkalizing	-	-
O-bleached	Scouring	Alkalizing	Oxygen bleaching	-
C-bleached	Scouring	Alkalizing	Oxygen bleaching	Chlorine bleaching

2.1.1. Characterization of materials

Fiber micrographs were obtained via use of an Olympus SZ61 microscope with LCmicro software at 200X-400X magnifications at transmitted light. Linear density was measured according to ASTM D 1577-07 at 5 replicates with a minimum total length of a hundred centimeters length for each fiber bundle specimen using a Radwag precision balance and a millimetric ruler. Moisture content was determined according to ASTM D2495-07 at 3 replicates by using a Nuve FN 120 dry oven and the Radwag precision balance.

Time-dependent hygroscopicity of fibers was investigated by using a modified RILEM method, that was originally developed for plant-based construction materials [15]. Fibers were kept at 105 °C for 16 h and cooled in a desiccator and weighed (m_d). Fibers were soaked in water for 15 min, 60 min, and 120 min. They were taken out of water, centrifuged in a salad spinner for 50 secs at 2 rpm and weighed ($m(t)$). Three replicates were measured. Water absorption rate ($w(t)$%) as a function of duration was calculated as follows:

$$w(t)\% = \frac{m(t)-m_d}{m_d} \times 100 \quad (1)$$

Findings of physical characterization analyses are presented in Table 2 and time-functional water absorption in Table 3. Results of physical characterizations were subjected to statistical analysis at α:5%. Summary of statistical analyses is presented in Table 4. Fourier transform infrared spectra (FTIR) of the samples were recorded between 4000-400 cm^{-1} wavenumbers using a Perkin Elmer FT-IR Spectrophotometer Spectrum Two UATR-Two, US at attenuated total reflectance (ATR) mode keeping force gauge between 40-50 and accumulating 16 scans.

Scanning electron microscopy (SEM) images of okra bast fibers were obtained by using Phenom ProX, Thermofisher Scientific, at a voltage of 10 kV and magnification rates of 300x and 500x with back-scattered electron detection. Prior to capturing SEM images, fibers were subjected to gold coating at 5.8 nm thickness by use of a Cressington 108 auto sputter coater.

III. RESULTS AND DISCUSSIONS

Photographs and micrographs of the fibers are given in Fig 1 and 2, respectively. Linear density, moisture content, time-functional water absorption, FT-IR spectra and SEM images are presented in Figs 3, 4, 5, 6 and 7 in consecutive order.

Oxygen- and chlorine- bleaching increased whiteness and fineness of the okra bast fibers as seen in Fig. 1. Photo images and micrographs reveal that raw and scoured okra bast fibers are straight in shape, while getting crimpiness after being subjected to alkalization (Figs. 1 and 2). Raw okra bast fibers include the greatest amount of extra cellulosic substances. Scouring led to elimination of extra cellulosic particles. Alkalization led to crimpier and more slender fibers. A great variability of fineness is observed for all fibers. Separation into thinner fibers is observable in the treated fibers. Discoloration is seen in the bleached fibers. These findings agree with that of Khan

[14]. It is evident from all micrographs that okra bast fibers are technical fibers that are bundles of elementary fibers.

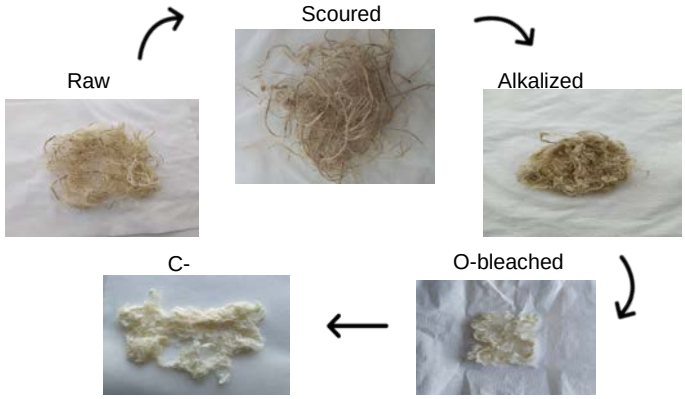

Figure 1. Photography images of raw, scoured, alkalized, o-bleached and c-bleached okra bast fibers

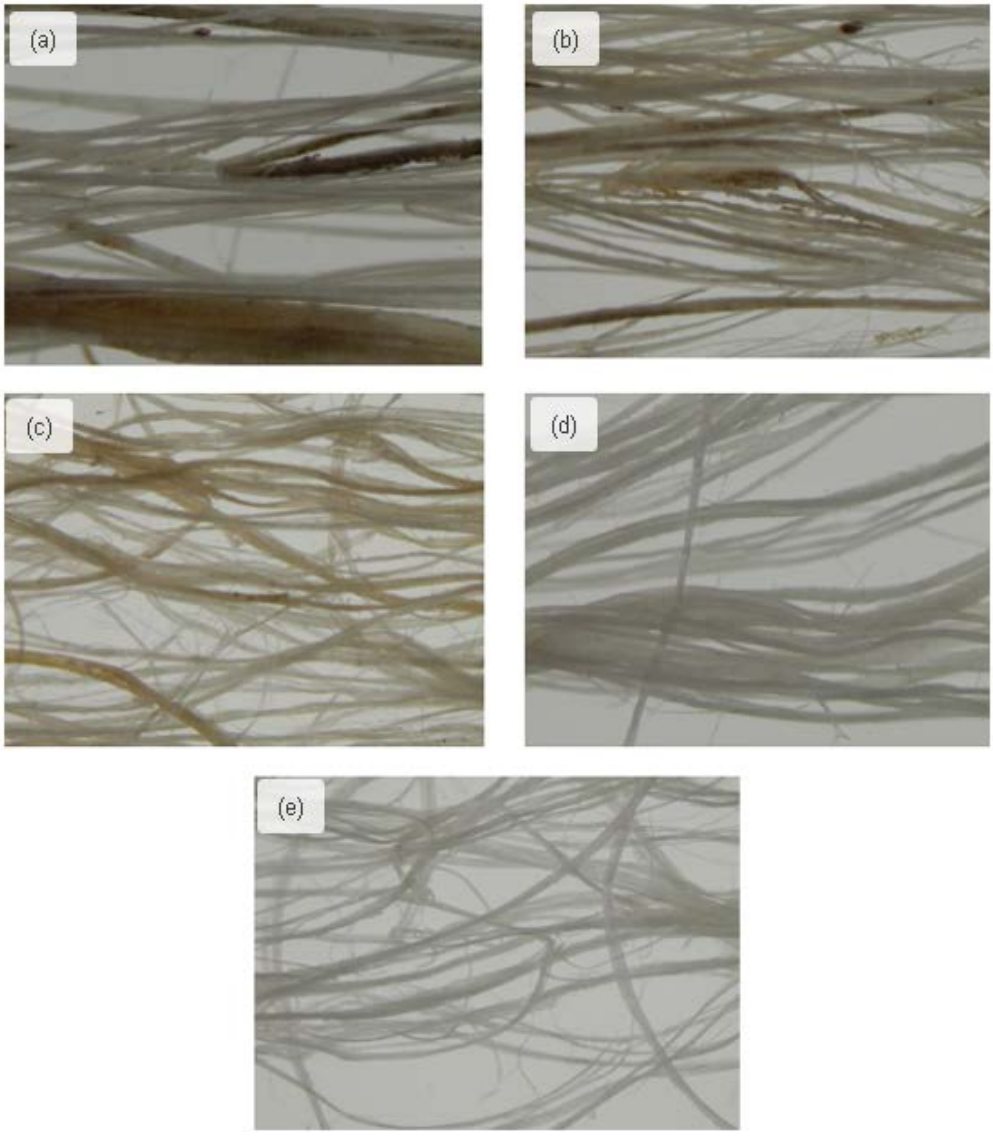

Figure 2. Microscopy images of **(a)** raw, **(b)** scoured, **(c)** alkalized, **(d)** o-bleached and **(e)** c-bleached okra bast fibers at 200x-400x magnifications

The applied chemical treatments have been found to have statistically significant effect on fiber linear density (p value 2.92×10^{-16}). The chemical treatments led to finer fibers as resulting in elimination of extra cellulosic components and in separation into fibrils (See Fig 3). Linear density analysis findings show similarity to optical images of the fibers.

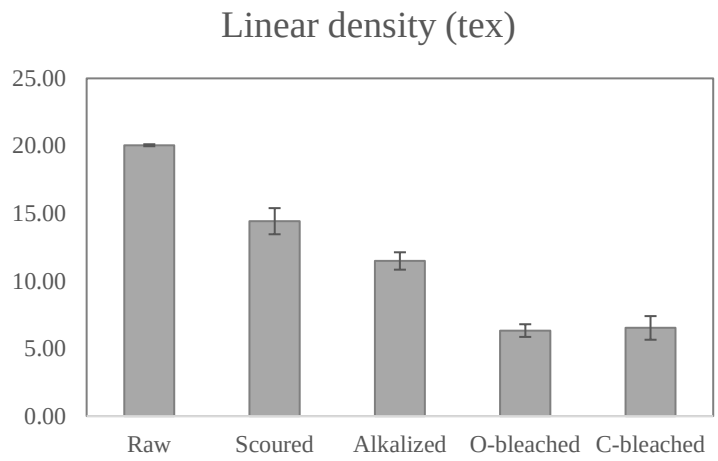

Figure 3. Effect of chemical treatments on okra bast fiber linear density

The applied chemical treatments have been found to have statistically significant effect on moisture content of the fibers (p value 3.2×10^{-4}). The chemical treatments resulted in higher moisture content as leading to elimination of extra cellulosic hydrophobic components to open hydrophilic cites; and separation of fibrils to get higher surface area to let water molecules to bind. Lignin, which is hydrophobic and soluble in hot alkali is eliminated in the alkalization process. Khan et al. [14] also reported higher moisture content upon alkalization of scoured okra bast fibers. Excessive chemical treatment led to decreased moisture content by eliminating water binding cites. Moisture content analysis show a trend that is reverse that of linear density. Fibers with high linear density show low moisture content. Moisture content analysis results are presented in Table 2.

Yield of the chemical treatments are given in Table 2. Scouring led to elimination of 8.9% of the total fiber mass. Alkalization was seen to show the most efficient extra cellulosic substance removal effect with 26.8% decrease in mass. Both lignin and hemicellulose are soluble in alkaline media [16, 17]. Chlorine bleaching is observed to be the least efficient in mass reduction: 2.7%. This might be due to the fact that c-bleaching was applied following o-bleaching. The cites to be attacked in the c-bleaching processes might have already been eliminated in the preceding o-bleaching step. Khan et al. [13] reported mass loss due to bleaching and alkalization were closer to each other; however, they applied alkalization after bleaching which is contrary to the current study.

Table 2. Effect of chemical treatments on physical properties of okra bast fibers

Sample	**Fiber yield** *(%)*	**Linear density** *(tex)*	**Moisture content** *(%)*
Raw	-	20.05±0.07	2.92±0.48
Scoured	91.6	14.43±0.97	4.58±0.39
Alkalized	73.2	11.48±0.64	4.82±0.77
O-bleached	87.6	6.33±0.47	6.00±0.74
C-bleached	97.3	6.53±0.88	5.10±0.30

Note: ± numbers depict 95% confidence intervals.

Table 3. Effect of chemical treatments on time-functional water absorption of okra bast fibers

Sample	Water absorption		
	15 min *(g/g)*	**60 min** *(g/g)*	**120 min** *(g/g)*
Raw	5.80±0.45	7.69±0.58	8.78±0.89
Scoured	6.52±2.63	9.45±1.32	11.81±2.91
Alkalized	8.28±1.30	9.60±1.38	9.41±0.49
O-bleached	10.55±2.53	9.45±0.88	11.05±0.67
C-bleached	8.34±0.18	9.26±0.65	9.52±0.54

Note: ± numbers depict 95% confidence intervals.

Table 4 (a). Summary of Single-factor ANOVA applied to linear density of okra bast fibers

Source of Variation	SS	df	MS	F	P-value	F critical
Between Groups	663.9882	4	165.997	221.7933	2.92×10-16	2.866081
Within Groups	14.96863	20	0.748431			
Total	678.9568	24				

Table 4 (b). Summary of Single-factor ANOVA applied to moisture content values of okra bast fibers

Source of Variation	SS	df	MS	F	P-value	F critical
Between Groups	0.001507	4	0.000377	14.94326	0.00032	3.47805
Within Groups	0.000252	10	2.52E-05			
Total	0.001759	14				

Table 4 (c). Summary of Two-factor ANOVA with replication applied to time-functional water absorption of okra bast fibers

Source of Variation	SS	df	MS	F	P-value	F critical
Soaking duration	36.90411	2	18.45205	11.56569	0.000189	3.31583
Samples	39.42471	4	9.856176	6.177823	0.000947	2.689628
Interaction	28.23183	8	3.528979	2.211954	0.055163	2.266163
Within	47.86237	30	1.595412			
Total	152.423	44				

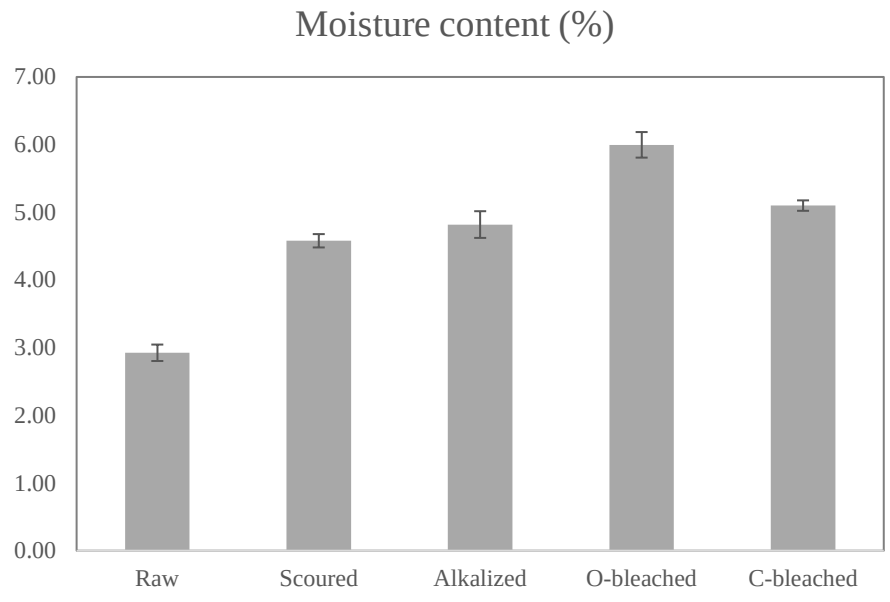

Figure 4. Effect of chemical treatments on moisture content of okra bast fibers. Error bars represent standard errors

The applied chemical treatments have been found to have statistically significant effect on water absorption (p value 9.47×10^{-4}) as does the soaking duration (p value 1.89×10^{-4}). Time-functional water absorption analysis shows that water absorption is generally increased with increment in soaking duration as presented in Table 3. As expected, raw fibers give the lowest water absorption values. Water absorption increases with chemical treatments, whereas excessive treatments led to reduction in water absorption (Fig 5). This finding agrees with that of Khan et al. [13]. Water absorption trend show similarity with that of moisture content. As in the case of moisture content, water absorption shows a reverse trend of linear density. Coarser fibers exhibit less water absorption. Obtained physical properties are within the range of those of common natural fibers such as flax, hemp and jute [12].

In the IR spectra presented in Fig 6, two major peaks are dominant: namely, one around 3400 cm^{-1} (corresponding to O-H due to hydrophilicity) and the other 1050-1000 cm^{-1} (to C-O in cellulose) [7]. Some secondary peaks include 2918–2855 cm^{-1} for C–H stretching in lignin and waxes, and 1732 and 1639 cm^{-1} of carbonyl (C=O) stretching vibration in lignin and hemicelluloses. Some peaks show differences in their strength and shifts in their locations due to the effect of chemical treatments on reactive groups. The peaks at 2918 and 1639 cm^{-1} lose strength in the chemically treated fibers. The ones at 2855 and 1732 cm^{-1} decrease at the scoured fiber and disappears after alkalization. The 1237 cm^{-1} peak decreases at the scoured fiber and partially disappears after alkalization. These findings show similarities to that of [14, 18, 19].

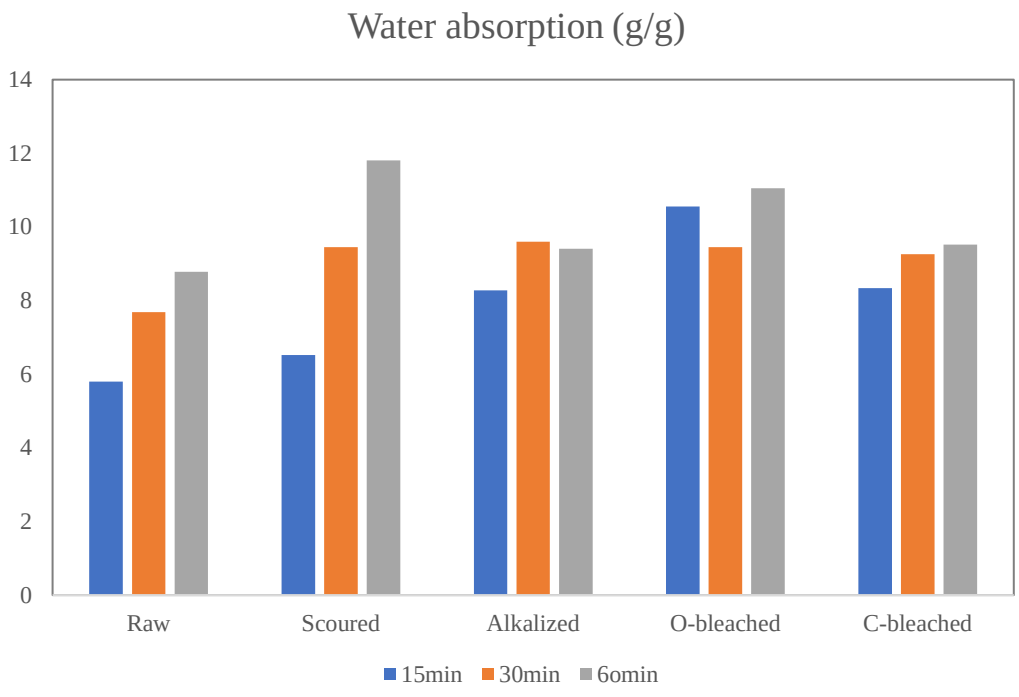

Figure 5. Effect of chemical treatments on time-functional water absorption of okra bast fibers

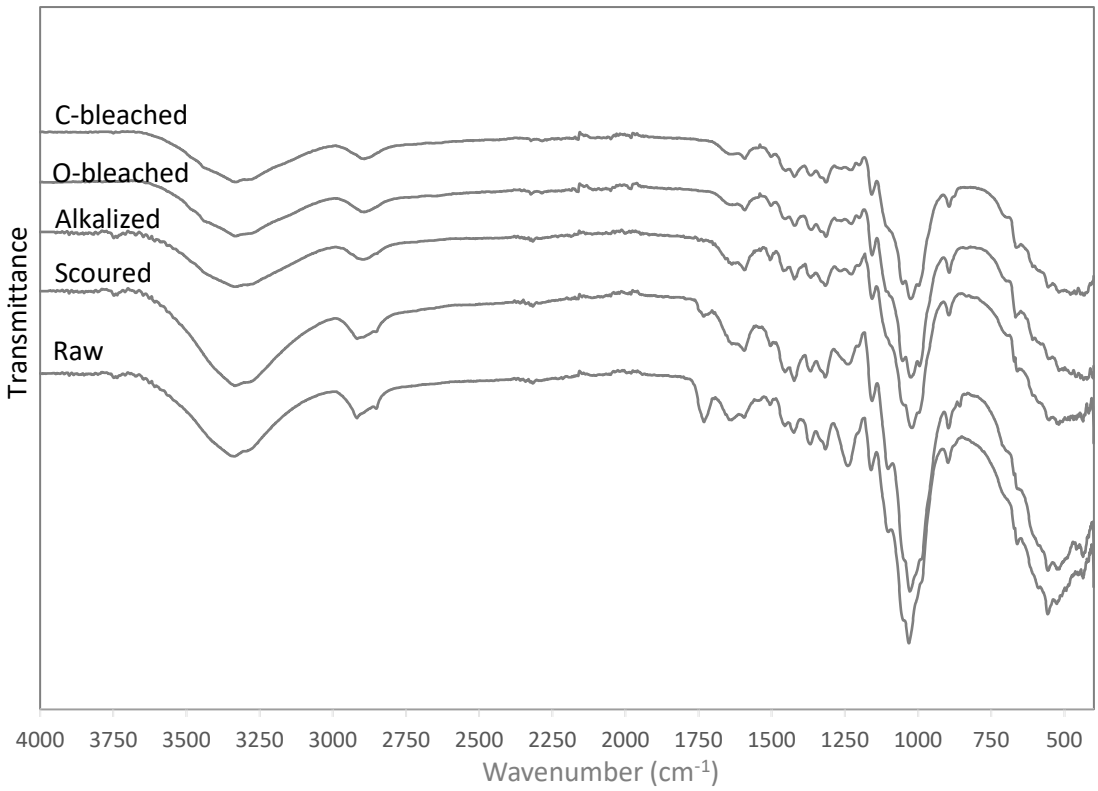

Figure 6. FT-IR spectra of okra bast fibers. Horizontal axis represents wave numbers (cm^{-1}), vertical axis transmittance (%)

SEM images are presented in Fig. 7. SEM images reveal that the okra bast fibers present longitudinal straight shapes. Fibers show highly variable diameters. It is observed that some substances take place on fiber surfaces. It is clearly seen that each fiber is a bundle of parallel elementary fibers stuck together. Chemical treatments result in separation of elementary fibers and decrease in observed fiber thickness. This finding agrees with the linear density analysis. It is also obvious that the treatments resulted in elimination of extra-cellulosic substances. Alkalized samples show some crimpiness complying to photo images. Excessive treatments led to damage on fibers as seen in Fig 7(j). SEM images show similarity to that of [14, 19].

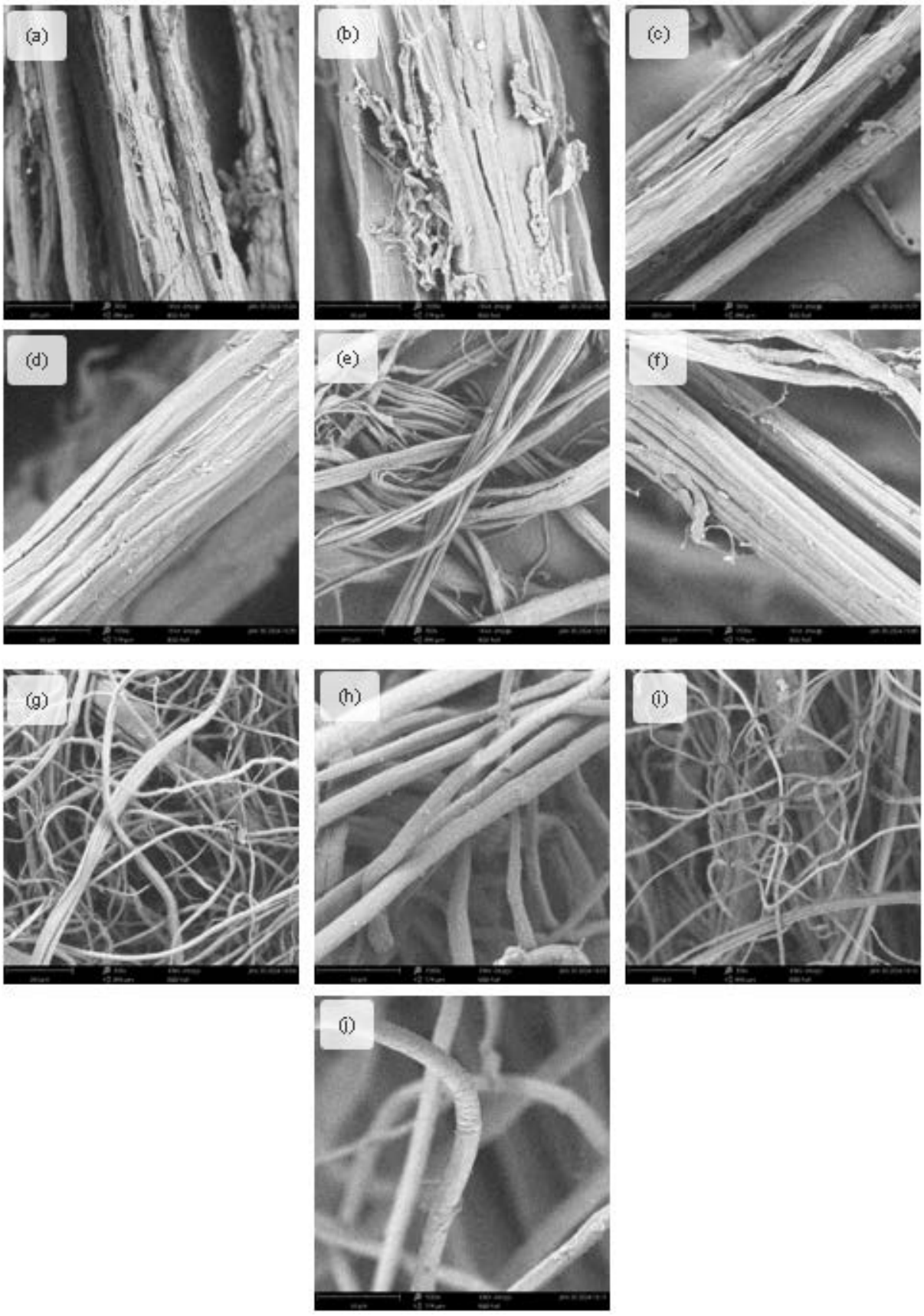

Figure 7. Scanning electron microscopy images of raw fibers at (a) 300x, (b) 1500x, scoured fibers at (c) 300x, (d) 1500x, alkalized fibers at (e) 300x, (f) 1500x, o-bleached fibers at (g) 300x, (h) 1500x and c-bleached okra bast fibers at (i) 300x and (j) 1500x magnifications.

IV. CONCLUSIONS

Bast fibers have been obtained from okra plant (*Abelmoschus esculentus*) stem via biological degumming process. The obtained fibers were subjected to some textile pretreatment processes such as scouring, alkalizing, oxygen bleaching and chlorine bleaching with parameters utilized in cellulose nanoparticle production. Effects of these processes on the physical and chemical characteristics of the fibers have been experimentally and statistically investigated. Treatments led to decrease of fiber linear density by fibrillation and elimination of impurities as evidenced by SEM microscopy and mass loss analysis but increase of moisture content and water absorption capacity. Prolonged submerging resulted in increased water absorption. FT-IR spectra suggest removal of lignin, hemicelluloses, and waxes upon chemical treatments. Chemical treatments have statistically significant effects on fiber properties. Characteristics of okra bast fibers show similarities to conventional bast fibers such as flax, hemp and jute.

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