PAPER • OPEN ACCESS

Nonwoven production from agricultural okra wastes and investigation of their thermal conductivities

To cite this article: M N Duman *et al* 2017 *IOP Conf. Ser.: Mater. Sci. Eng.* **254** 192007

IOP Conf. Series: Materials Science and Engineering **254** (2017) 192007 doi:10.1088/1757-899X/254/19/192007

Nonwoven production from agricultural okra wastes and investigation of their thermal conductivities

M N Duman[1], E D Kocak[1], N Merdan[2] and I Mistik[1]
[1]Marmara University Technology Faculty Textile Engineering Department, Istanbul-TURKEY
[2]Istanbul Commercial University Faculty of Engineering and Design, Istanbul-TURKEY

E-mail: mnayci@marun.edu.tr

Abstract. Nowadays bio-based composite materials have been used in rising amounts and demanded widely in industrial uses, as they provide cost reduction and weight loss in the end use products. Agricultural cellulose based wastes can be a good alternative to synthetic fibers and can be used in natural fiber reinforced composite production, as there is a huge (more than 40 million tons) potential for natural cellulose production from agricultural wastes. Okra is one of the most grown vegetables around the world with stems left on the fields after harvest. When the similarity of mechanical properties of okra fibers with traditional bast fibers (flax, kenaf, hemp) are considered, from an economical and an environmental point of view this research emphasizes the potential of agricultural biomass for natural fiber production. In this study, okra stem wastes used for natural cellulosic fiber production and treated with 10% NaOH at 60^0C for 10, 20, 30 and 40 minutes. By alkali treatment, decrease in fiber diameter and weight, and increase in tensile strength and elongation % have been observed. Nonwoven production has been done from both the fibers with and without surface treatments. Thermal conductivity properties of both nonwovens have been investigated.

1. Introduction

The importance of studying on bio-based products increased due to rapid consuming of natural sources around the world and increasing environmental problems in the global scale. 50-65 million tons [1] plant wastes are coming out every year in Turkey. Most of them are not utilized and creating additional costs for collecting and destroying, and they are not converted to an economic value.

Okra is an agricultural plant that is easy and effortless to cultivate due to drought-tolerant formation and low water requirements. However, research and studies on the assessment of agricultural stem wastes have begun for the last fifty years [2] and studies on the fiber extraction from them have been done in the last decade [3]. Okra fibers are obtained from the stem wastes remaining on the fields after harvest of okra plants (Abelmoschus esculentus) from the family Malvaceae (Malvaceae). Okra stem waste fibers contain 67.5% a-cellulose, 15.4% hemicellulose, 7.1% lignin, 3.4% pectin, 3.9% fat and waxes and 2.7% water-soluble ingredients [3].

Plant fibers are natural, recyclable, renewable, degradable and sustainable materials. However, since they are hydrophilic materials, they can easily absorb moisture and this is an undesirable characteristic as it will cause decay. Surface modification is made to facilitate the industrial use of plant fibers and thus to increase it. The surface modification made as a pre-treatment can be applied by many methods to improve the mechanical properties of the fibers, such as strength, and to increase the absorbency of the subsequent treatments by providing surface roughness, some of which are alkalization with sodium hydroxide (NaOH) [4–12]or Sodium sulphate (Na_2SO_4) [12,13], bleaching with Sodium hypochlorite (NaClO) or Sodium chlorite ($NaClO_2$) [4,5,8,14], and acetylation with acetic acid [13–15]. Surface modification processes also remove as much of the structural material as lignin, hemicellulose, and pectin, allowing more reaction zones to occur in the cellulosic structure [13]. It has been observed that when the concentration of the chemical substance is too high, as well as being an important factor, it negatively affects the mechanical properties such as the strength of the fiber [12]. When pre-treated with alkaline, the content of cellulose increases to 75-80% due to removal of hemicellulose, lignin, pectin and other water-soluble substances in the okra stem fiber. The fiber becomes less dense and less rigid, which allows for more ability to the fibrillar structure to settle in the direction of tension [16]. The most common and widely used chemical in alkalisation is sodium hydroxide. Alkalisation process changes the natural structure of cellulose from cellulose I to cellulose II, the alkalization depolymerizes the molecular structure of natural cellulose I by forming short crystals [17].

As 36.000 tons/year okra grown in Turkey [1], utilization of the okra stem wastes has been studied according to 9th developments plan and region development visions of the State Planning Organization, while okra fibers have similar mechanical properties with the traditional fibers [3–5,14,18]. The aim of this study was utilizing the okra stem wastes and contributing them to the environmental biomass cycle by removing the wastes in their sources and by producing environmentally friendly, value added, a novel fiber/product. And it has the leading potential of production of the first fiber/product manufacturing from the agricultural wastes in Turkey.

In this study, okra stem wastes used for natural cellulosic fiber production and treated with 10% NaOH at 600C for 10, 20, 30 and 40 minutes. By alkali treatment, decrease in fiber diameter and weight, and increase in tensile strength and elongation % have been observed. Nonwoven production has been done from both the fibers with and without surface treatments. Thermal conductivity properties of both nonwovens have been investigated.

2. Materials and Method

Okra stem wastes have been collected from Mediterranean region. Fibers produced from okra stem wastes by using the specially designed machine (100 kg/hour capacity) in Marmara University. The fibers are left in the water-filled containers for 20 days so that the residues on the fibers separated from the plant extracts and the adhesive materials can be removed completely. In order to clean and fibrillate the surface of the fiber samples (each 2 g), surface modification has been applied with 10% NaOH at 60^{0}C in ratio of 1:20 g/ml for 10, 20, 30 and 40 minutes by referring Islam and Pickering's studies [17]. Then fibers first washed warm at 25°C, were neutralized with 5% acetic acid solution for 5 minutes and dried in the stove at 100°C for 2 hours. Finally, the fibers were conditioned in laboratory conditions (**65**±2% relative humidity **20**±2^{0}C).

Tensile strength and elongation% values of okra fibers have been tested with INSTRON 4411 testing machine (50 N load, 10 mm/min speed) according to ASTM D 3822 standards, their diameters have been measured with Projectina CH-9495 microscope, and their morphological views have been taken with JEOL JSM-T330 electron microscope in Physical Test Laboratory of Marmara University Faculty of Technology Textile Engineering Department. Then combing and needle punching processes have been applied to the fibers with and without surface modifications at the laboratuary based sampling nonwoven production line in ITA Laboratories in Istanbul/Turkey for forming nonwoven surfaces. In this line there are combing machine (CFS1/Feeding width-exit 1000 mm, 650m/min.), cross laying machine (FRT50, input-output 1000 mm, band 49 m/min, exit 6m/min.), needling machine ((MAG4000 roller AR95, preneedling–end needling 1200 mm, distance of the calenders 0-10 mm, roller: 1200 mm, needling speed 0-rpm-400rpm; oven 0-6,5 m/min., roller 0-10 m/min.) that can work seperately. The capacity of the machine is 50 kg/h and its width is 1000 mm (Cormatex S.R.L.). Thermal conductivity properties of both nonwovens have been investigated according to TS ISO 5085 standards in Marmara University laboratories with P.A. HILTON LTD.H940 testing machine.

3. Results

The fibers obtained from okra body wastes are amorphous due to their lignocellulosic properties and their most important ingredients are cellulose, hemicellulose and lignin. The characteristic properties of each component play a decisive role in all properties of the fiber. Okra body waste fibers are adhered to each other by materials such as lignin, gum and wax in the cells of the structure. After the fibers are separated from the plant extracts, it is necessary to apply the hydrophilization process in order to remove the water repellent substances like lignin. For this reason, in this study one of the methods used for surface modification to hydrophilize and fibrillate the okra stem waste fibers is the conventional method.

Factors affecting alkalization are the type and concentration of the chemical substances, the duration of the process and the heat. Alkali treatment removes lignin and hemicellulose from the fiber structure. By the alkali processes applied in this project, alkali-sensitive (OH) groups between the molecules reacted with water molecules as shown in equation (1) below to remove the H-bonds, so the amorphous structure and the amount of fibrillation which increases the amount of contact between the fiber and the matrix increased. The surface irregularity also increased with the rupture of the H-bonds, which leads to better mechanical bonding between the fiber and the matrix and to the tensile strength of the composite [19].

$$\text{Fiber} - \text{OH} + \text{NaOH} \longrightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O} \quad (1)$$

The stem diameter of the okra plant which grows in different regions in Turkey varies between 10-50 mm. Physical diameters (μm) (figure 1) of okra stem waste fibers obtained from different regions were determined for treated and untreated fibers.

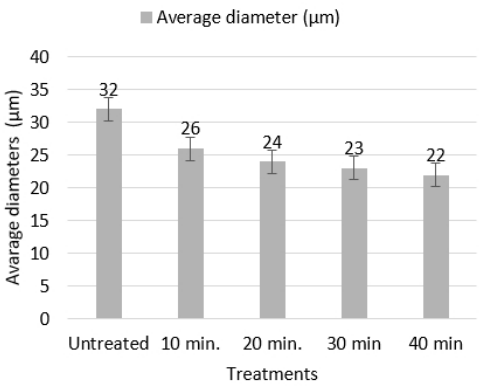

Figure 1. Diameters of okra fiber samples pre-treated with 10% sodium hydroxide.

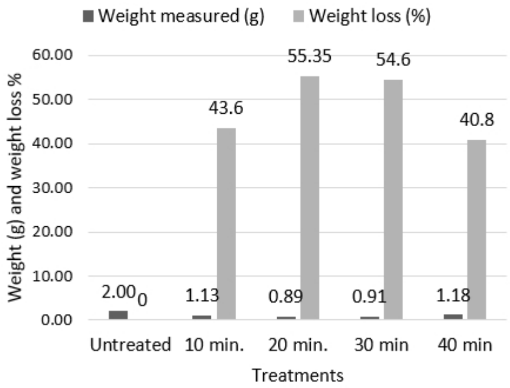

Figure 2. Measured weights and weight loss % of okra stem waste fibers treated with 10% sodium hydroxide.

In this study, the diameter of the okra stem fibers was measured from different points of the fiber and the average was taken. As shown in table 1, surface modified fibers showed some swelling due to mercerization and widening in cross-section. However, hemicellulose, lignin and other impurities are also removed during the surface modification process, and accordingly the diameter is reduced to some extent. When the cross-sectional and longitudinal views (figure 3) and diameter measurements of the okra stem fibers are examined, it is seen that there is a linear deformation [5] in the okra stem fibers as well as in all of the plant fibers.

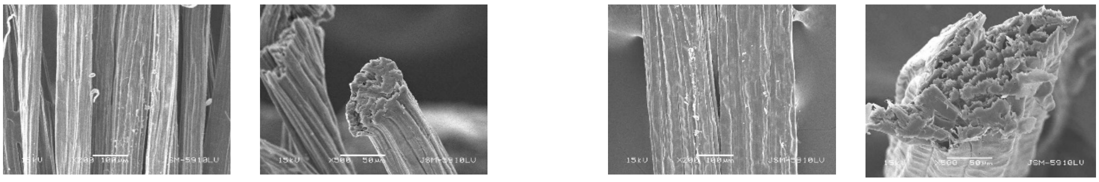

Figure 3. Longitudinal and cross-sectional SEM images of untreated and 10% sodium hydroxide treated okra stem waste fibers respectively.

It is seen that the results of the treatments with sodium hydroxide in the SEM images of okra stem fibers are effective. Inorganic materials, oils and waxes and non-cellulosic materials have been removed from the fiber surface. The chemical modification of the fiber surface appears to modify the surface of the fibers to give rise to roughness on the surfaces. As can be seen from SEM micrographs, it has been found that the conventional method is used to produce fibrillation on the surface of the okra stem waste fibers without damaging the molecular structure of the fiber and reducing its mechanical properties [20]. Because swelling is a physicochemical phenomenon of plant fibers in the context of water and chemicals, water molecules penetrate the gaps in the fiber and open hydrogen bonds, making the cellulosic fibers suitable for swelling. During the swelling process, the hydrogen bonds of the cellulose molecules break off and the water molecules are transported through hydrogen bonds. Thus, the water molecules bond with the OH groups of the cellulose fibers via hydrogen bonds, and as a result, the cellulose fibers begin to swell. It has been found that the degree of swelling is dependent on the number of free hydroxyl (OH-) groups in the fiber [21,22].

Tensile strength, elongation (%), Young's modulus (%) of okra stem waste fibers after surface modification with conventional method are given in figure 4. Its morphological structure, crystallinity, amorphous region ratio and orientation, physical and chemical properties affect the mechanical properties of lignocellulosic fibers. The moisture absorption capacity of lignocellulosic fibers varies depending on the fiber structure. Surface modification processes increase the roughness rate on the fiber surface and the ability to absorb moisture [23].

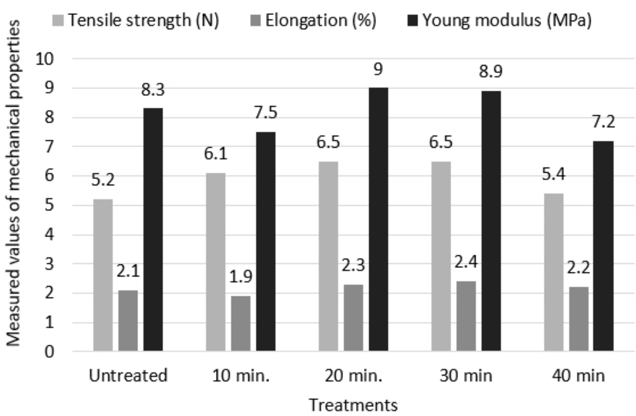

Figure 4. Mechanical properties of okra stem waste fibers treated with 10% sodium hydroxide.

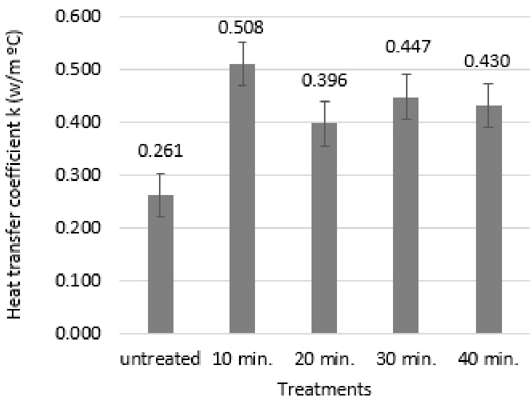

Figure 5. The calculated heat transfer coefficients of okra fiber nonwovens treated with 10% NaOH at different durations

As shown in figure 4, the increase in the tensile strength of the okra fibers with respect to the untreated fiber in the surface modification with sodium hydroxide according to the conventional method was determined, but decrease at tensile strength over 30 minutes was observed. After surface treatment, the okra stem waste fibers are weighed with a precision scale after preconditioning in lab conditions. The weight of each sample before treatment is 2 g. The weight loss of the fibers is calculated as a percentage by the following equation (2).

$$W_j = ((W_{before} - W_{after})/W_{before}) \times 100 \quad (2)$$

W_j: Weight loss%, W_{before}: Weight before treatment, W_{after}: Weight after treatment

Figure 2 shows the weight measurements and the weight loss (%) values of okra stem waste fibers after the treatment according to the pre-treatment. It can be said that 20 minutes of treatment with sodium hydroxide gives very good results and also allows the removal of the most purities. In addition, improvement in the brightness and stiffness of the okra stem waste fibers was observed.

After non-woven surface formation by needle punching method from treated and untreated okra waste fibers, the heat conductivity tests of these nonwovens have been done according to TS ISO 5085 standards. The heat transfer coefficient (K) of non-woven surfaces was shown in w/m ºC. The linear conduction module was used in the heat transfer device. The heat flow value (Q) from the digital display of the device was determined in watts. The heat transfer coefficient was calculated by using the eauqtion (3) as shown below.

$$K = (Q.d_x)/(A.\Delta T) \quad (3)$$

K: heat transfer coefficient, d_x: thickness of nonwoven surfaces, A: area of test area, ΔT: temperature difference, Q: heat flow value

The heat transfer coefficient K is determined at w/m ºC. The d_x thickness parameter in the equation has a very decisive role. To measure the heat transfer coefficient of non-woven surfaces obtained from the okra stem waste fibers, a material Δx with a diameter of 25 mm have been placed between the two brass plates at the T3 and T4 temperatures. The one side of the nonwoven surfaces have been adjusted to the temperature T3 and the other side of the surfaces to the temperature T4. According to Fourier Heat Conduction Law, while a heat transfer was occurring between the two media with temperature differences from the hot region to the cold region, the non-woven surfaces between these two-media had a certain heat transfer coefficient. According to the Fourier Law, the heat transfer coefficient of the material has been calculated with the equation (4) as shown below.

$$Q_x = - k.A.(\Delta T/ \Delta x) \quad (4)$$

After the measurement system started to function, temperatures T3 and T4 have been read from the digital display in every 10 minutes. The heat transfer coefficient of the surfaces has been calculated from the Fourier Law in the direction of the heat energy supplied to the system, sample thickness and area information.

As shown in figure 5, the okra fiber nonwoven treated with 10% NaOH at 10 minutes has the best heat transfer coefficient. Although the okra fiber nonwovens treated with 10% NaOH at 20, 30 and 40 minutes have better heat transfer coefficients than the okra fiber nonwoven which was produced from untreated fibers, their heat transfer coefficients are lower than the okra fiber nonwoven treated with 10% NaOH at 10 minutes. Also, it is seen from the graphic that there is no correlation with the heat transfer coefficients and the treatment durations.

4. Conclusion

Agricultural waste fibers and nonwovens obtained these fibers can have good mechanical properties and thermal conductivity values as the traditional fibers (both natural and synthetic) used in industry. Agricultural waste fibers are cost effective, lightweight, environmentally friendly, renewable, recyclable and degradable. As this study is a part of a TUBITAK (The Scientific and Technological Research Council of Turkey) research project, our studies are continuing. Nonwoven productions from okra stem waste fibers treated with different chemicals and enzymes will be done, and their heat transfer coefficients will be measured and compared.

Acknowledgements

Authors are highly thankful to TÜBİTAK (The Scientific and Technological Research Council of Turkey) for aiding our project #215M984. This study is a part of this project.

References

[1] "*Türkiye İstatistik Kurumu*" [Online]. Available: http://www.tuik.gov.tr/PreTabloArama.do. [Accessed: 09-May-2017].

[2] FRANKLIN W. MARTIN, 1982, "*Okra, Potential Multiple-Purpose Crop for the Temperate Zones and Tropics,"* Econ. Bot., **36**(3), pp. 340–345.

[3] Alam, M. S., and Khan, G. M. a., 2007, *"Chemical Analysis of Okra Bast Fiber (Abelmoschus Esculentus) and Its Physico-Chemical Properties,"* J. Text. Apparel, Technol. Manag., **5**(4), pp. 1–9.

[4] Khan, G. M. A., and Alam, S., 2013, *"Surface Chemical Treatments of Jute Fiber for High Value Composite Uses,"* J. Mater. Sci., **1**(2), pp. 39–44.

[5] De Rosa, I. M., Kenny, J. M., Puglia, D., Santulli, C., and Sarasini, F., 2009, *"Morphological, Thermal and Mechanical Characterization of Okra (Abelmoschus Esculentus) Fibres as Potential Reinforcement in Polymer Composites,"* Compos. Sci. Technol., **70**, pp. 116–122.

[6] Fortunati, E., Puglia, D., Monti, M., Santulli, C., Maniruzzaman, M., Foresti, M. L., Vazquez, A., and Kenny, J. M., 2013, *"Okra (Abelmoschus Esculentus) Fibre Based PLA Composites: Mechanical Behaviour and Biodegradation,"* Journa Polym. Environ., **21**, pp. 726–737.

[7] Arifuzzaman Khan, G. M., Shaheruzzaman, M., Rahman, M. H., Abdur Razzaque, S. M., Sakinul Islam, M., and Shamsul Alam, M., 2009, *"Surface Modification of Okra Bast Fiber and Its Physico-Chemical Characteristics,"* Fibers Polym., **10**(1), pp. 65–70.

[8] Rosa, I. De, Kenny, J., Maniruzzaman, M., Monti, M., Puglia, D., and Sarasini, F., 2011, *"Effect of Chemical Treatments on the Mechanical and Thermal Behaviour of Okra (Abelmoschus Esculentus) Fibres,"* Compos. Sci. Technol., **71**(2), pp. 246–254.

[9] Yılmaz, N. D., Konak, S., Yılmaz, K., Kartal, A. A., and Kayahan, E., 2016, *"Characterization, Modification and Use of Biomass: Okra Fibers,"* Bioinspired, Biomim. Nanobiomaterials, **5**(3), pp. 85– 95.

[10] Moniruzzaman, M., Maniruzzaman, M., Gafur, M. A., and Santulli, C., 2009, *"Lady's Finger Fibres for Possible Use as a Reinforcement in Composite Materials,"* J. Biobased Mater. Bioenergy, **3**, pp. 1–5.

[11] Kishimoto, A., Koumoto, K., Yanagida, H., and Nameki, M., 1991, *"Microstructure Dependence of Mechanical and Dielectric Strengths-I. Porosity,"* Eng. Fract. Mech., **40**(4–5), pp. 927–930.

[12] Srinivasababu, N., 2015, *"An Overview of Okra Fibre Reinforced Polymer Composites,"* Mater. Sci. Eng., **83**(1), p. 12003.

[13] Onyedum, O., Aduloju, S. C., Sheidu, S. O., Metu, C. S., and Owolabi, O. B., 2015, *"Comparative Mechanical Analysis of Okra Fiber and Banana Fiber Composite Used in Manufacturing Automotive Car Bumpers,"* Am. J. Eng. Technol. Soc., **2**(6), pp. 193–199.

[14] Kumar, D. S., Tony, D. E., Kumar, a P., Kumar, K. A., Rao, D. B. S., and Nadendla, R., 2013, *"A Review on : Abelmoschus Esculentus (Okra),"* Int. Res. J. Pharm. appied Sci., **3**(4), pp. 129–132.

[15] Fortunati, E., Puglia, D., Monti, M., Santulli, C., Maniruzzaman, M., and Kenny, J. M., 2013,

IOP Conf. Series: Materials Science and Engineering **254** (2017) 192007 doi:10.1088/1757-899X/254/19/192007

"Cellulose Nanocrystals Extracted from Okra Fibers in PVA Nanocomposites," J. Appl. Polym. Sci, **128**, pp. 3220–3230.

[16] Arifuzzaman Khan, G., Ahsanul Haque, M., and Shamsul Alam, M., 2014, *"Studies on Okra Bast Fibre- Reinforced Phenol Formaldehyde Resin Composites," Biomass and Bioenergy*, Springer International Publishing, pp. 157–174.

[17] Islam, M. S., and Pickering, K. L., 2014, *"An Empirical Equation for Predicting Mechanical Property of Chemically Treated Natural Fibre Using a Statistically Designed Experiment,"* Fibers Polym., **15**(2), pp. 355–363.

[18] Mwaikambo, L. Y., and Ansell, M. P., 2002, *"Chemical Modification of Hemp, Sisal, Jute, and Kapok Fibers by Alkalization,"* J. Appl. Polym. Sci., **84**(12), pp. 2222–2234.

[19] Hamideh Hajiha, Mohini Sain, and Lucia H. Mei, 2014, *"Modification and Characterization of Hemp and Sisal Fibers,"* J. Nat. Fibers, **11**(2), pp. 144–168.

[20] Rong, M. Z., Zhang, M. Q., Liu, Y., Yang, G. C., and Zeng, H. M., 2001, *"The Effect of Fiber Treatment on the Mechanical Properties of Unidirectional Sisal-Reinforced Epoxy Composites,"* Compos. Sci. Technol., **61**, pp. 1437–1447.

[21] Bledzki, A. K., and Gassan, J., 1999, *"Composites Reinforced with Cellulose Based Fibres,"* Prog. Polym. Sci, **24**, pp. 221–274.

[22] Gassan, J., and Bledzki, A. K., 1998, *"Alkali Treatment of Jute Fibers : Relationship Between Structure and Mechanical Properties,"* J. Appl. Polym. Sci., pp. 623–629.

[23] Li, X., Tabil, L. G., and Panigrahi, S., 2007, *"Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review,"* J. Polym. Environ., **15**(1), pp. 25–33.