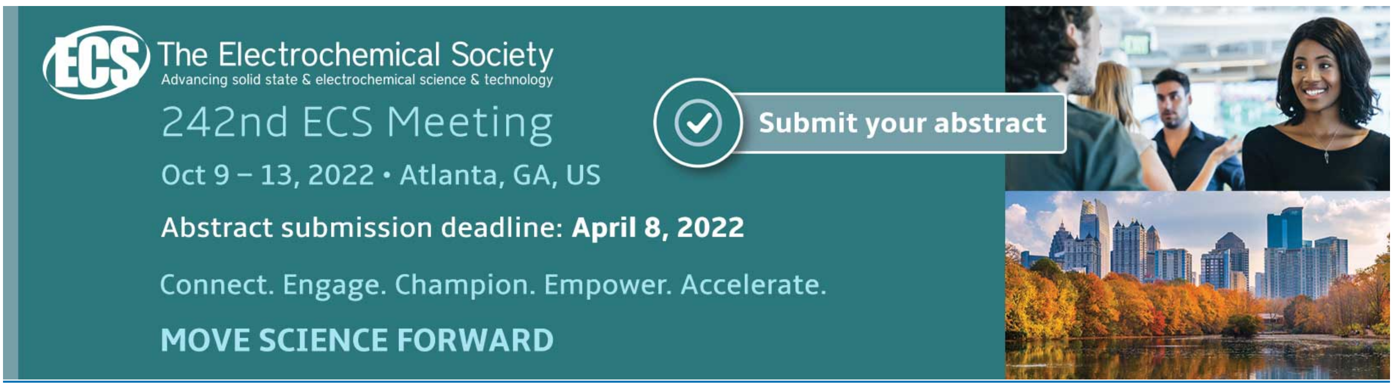
IOP Conference Series: Materials Science and Engineering

PAPER • OPEN ACCESS

Microwave Assisted Surface Treatment of Okra Fibers

To cite this article: A.A. ZG *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **460** 012042



IOP Conf. Series: Materials Science and Engineering 460 (2018) 012042 doi:10.1088/1757-899X/460/1/012042

Microwave Assisted Surface Treatment of Okra Fibers

A.A. İZGİ[1], E.D.KOÇAK[2], B.YILMAZ ŞAHİNBAŞKAN[2]

[1]*Marmara University, Institute of Pure and Applied Sciences, Department of Polimer Science and Technology, Goztepe/Istanbul, TURKEY*

[2]*Marmara University, Faculty of Technology, Department of Textile Enegineering, Goztepe/ Istanbul, TURKEY*

Corresponding author: aybenizgi@marun.edu.tr

Abstract-All over the world, the importance of the sustainable and biodegradable materials is increased very fast. The use of agricultural stem waste as a technical textile material can be made a huge contribution for save the environment. The aim of this research was to pre-treat the okra fibers with microwave energy. The fibers were obtained from Marmara Region agricultural waste by use a specially designed machine. They were microwave-assisted surface treated with 7 % sodium bisulphate and 10 % acetic acid. The results of tensile strength, fiber diameter, SEM and X-Ray analyses were compared to the conventionally treated fiber samples. Finally, microwave-assisted treatments were quite good. Bisulphate treatment was more effective than treatments with acetic acid. When the SEM images was investigated, a lot of porosity was observed on the surface of acetic acid treated fiber and the tensile strength values increased bisulphate treatment.

***Key words*- agriculture waste, microwave energy, okra fibers, surface modification**

I. INTRODUCTION

In the strengthening of new generation composites and nonwoven surfaces, natural fibers are increasingly regarded as an environmentally friendly alternative to synthetic fibers. These fibers are considered in the aerospace industry, construction and automotive sectors instead of glass and carbon fibers which are harmful to the environment and expensive [1].The properties of the natural fibers are listed in Table 1.

Table 1. Natural fibers properties[2].

NATURAL FIBERS PROPERTIES	Characteristic	Properties
	Physical	Low density, Surface topology, Texture, Form and geometry, good thermal insulation performance, specific heat, electrical conductivity, high sound absorption coefficient.
	Chemical and Biological	Chemical composition (cellulose, lignin, etc.), Batch quality, availability, planting limitless, odder emission etc..
	Mechanical	Good Mechanical Properies (Elastic modulus, shear modulus, Poisson's ratio, Yield strenght, elongation to break, specific modulus of elasticity, tensile strenght, spesific shear modulus etc..)
	Technical	Processing knowledge, friendly processing, processing energy consumption, processing cost, transferring cost, raw fiber cost,cost of energy input.
	Environmental	Eco-firendly, goverment support, bio- degradability, social positive view.

Okra (Layd's finger) plant is a plant with lignocellulosic content, known as Hibiscus esculentus L. and Okra (*Abelmoschus esculentus* (L.) Moench), a member of the family Mallow (Malvaceae). Okra plant is considered as one of the abundant sources of natural fibers [3].

Generally, when the okra plant is grown as a vegetable, the plant body is left on the fields after the plants are collected. After collecting the fruits of the okra plant, the remaining amount of agricultural waste is 2.11 tons / da [4]. The okra remaining as agricultural wastes are left in the soil as organic matter. In parallel with the population increase in the world, the demands of the

human beings are increased. However, with the increasing demand, the amount of agricultural wastes also increases. For the environment, there is a need to effectively recycle and re-use these wastes and to reduce environmental pollution with new resources.

Chemical modifications of natural fibers are intended to improve adhesion to the polymer matrix and increase the fibrillization to hold the fibers together. Approaches such as alkaline treatment, acetylation, silane treatment, benzolization, peroxide treatment, permanganate treatment, enzyme treatment and isocyanate treatment are applied by researchers [1].

The main factor in the interfacial adhesion strength is the linkage between functional groups on the fiber surface and in the nonwoven or composites matrix. Fiber surfaces must be modified to increase the number of functional groups on the surface of inert textiles [5].

Microwave energy is an alternative heating method widely used for pre- treatment, dyeing, finishing, drying, fixing, sterilization, surface modification and grafting of textile materials. Compared to conventional heating methods, it provides fast, effective, uniform, and energy efficient heating due to absorption in textiles [6]. Using of microwave technology in textile industry is related with heating up, pretreatment, drying, condensation, dyeing and printing [7].

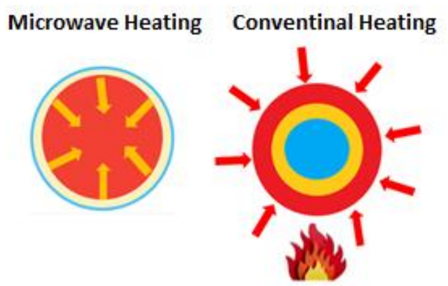


Figure 1.Microwave heating versus conventional heating.

In this study, acetic acid and sodium bisulphate was applied on okra fibers with two different kinds of processes which are conventional and microwave energy methods. The results of the okra fibers have been evaluated in terms of their mechanical and morphological properties.

II. EXPERIMENTAL

A. Fibers

Okra fibres were collected from Marmara region. Their overall lengths were 120 cm. These fibers were separated and cut according to their diameter and length. Okra body waste fibers were obtained by separating from plant extracts with the aid of a special machine designed to obtain fiber from lignocellulosic plants (Figure 2). It was waited in the water-filled container to remove residue and adhering dirt (20 days). The fibers were shaken under running water until cleaned and they were dried in an oven at 100 °C for 2 hours. After dried, fiber were conditioned for 24 hours prior to testing under 20 ±2 °C and 65 ±2 %RH conditions.

Figure 2. Removal of fibers from okra stem.

B. Surface Modification

Okra fibers have been treated into two methods in which are given as conventional method and microwave method in Table 2. Conventional process was applied in laboratory condition. Kerman' Laboratory sample heater (220 Volt and 3000 Watt) was used for conventional method. Microwave method was carried out with a Kenwood Mw440 at a frequency of 2.45 GHz. The microwave oven was set to Medium-Low (M-L) power of 350 W. The samples were placed in a sealed glass holder and treated by the microwave energy according to the experimental design.Treated okra waste fibers are shown in Figure 3.

Table 2. Process conditions.

Method	Time (min)	Temperature (°C)	Concentration (%)	Rinsing Process	Drying Condition	Chemicals
Conventional	10,20,30,40	60	7	25 °C, 10 min, pH 7 with distilled	At room temperature	Sodium bisulphate
		60	10			Acetic acid
Microwave	3,5,7,10	60	7			Sodium bisulphate
		60	10			Acetic acid

Figure 3. Treated okra waste fibers.

C. *Testing and Characterization of Okra Fibers After Chemical Treatments*

Tensile strength (N) values of the untreated/treated okra fibres were determined according to ASTM D 3822 test method by using Instron 4411 (50 N Load, 10 mm/min speed) tensile test instrument. Fiber diameters have been measured with Projectina CH-9495 microscope in Physical Test Laboratory of Marmara University Faculty of Technology, Department of Textile Engineering. Treated and untreated fibers morphological views have been taken with JEOL JSM-T330 electron microscope.Before and after surface treatment of okra body waste fibers, crystallinity was measured at 40 kV and 40 mA by D8 Bruker aXS Advance Germany brand X-ray diffraction (XRD). Diffraction density was measured at 0^o - 50^o at a scan rate of 2^o / sec. The Crystallinity index in the results of the measurements was carried out according to equation 1.

$$Crystallinity\ index\ (\%) = ((I002 - Iam)\ /\ I002)\ x100 \quad (1)$$

III. RESULTS AND DISCUSSIONS

A. *Results of Mechanical Testing*

The values of the tensile strengths of the fibers treated with sodium bisulphate and acetic acid are shown in Figure 4 and 5, respectively.

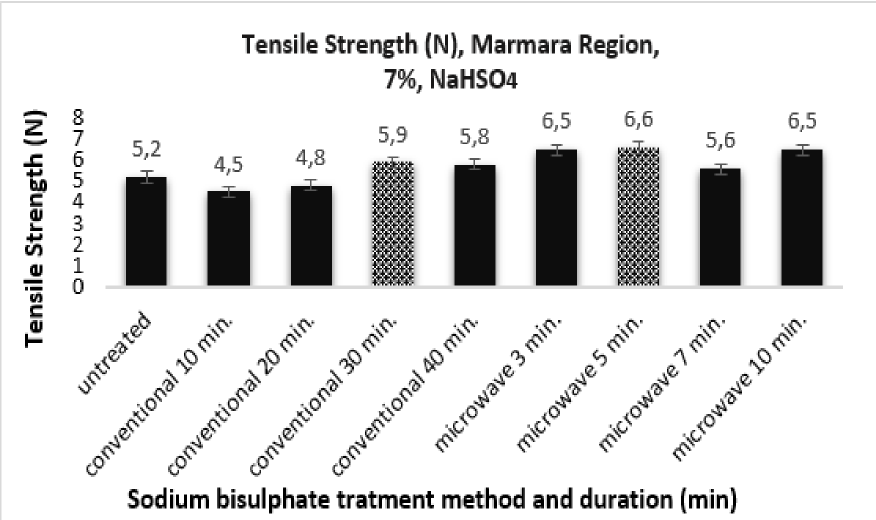


Figure 4. The tensile strength values of sodium bisulphate treated fibers with conventional and microwave energy methods.

The highest strength value of the okra fibers after surface modification compared to the untreated fiber was obtained at 7% concentration and 30 minutes of operation according to the conventional method with sodium bisulphate.

The strength was increased after surface modification with 7% sodium bisulphate for 5 minutes by the microwave method according to the raw fiber.

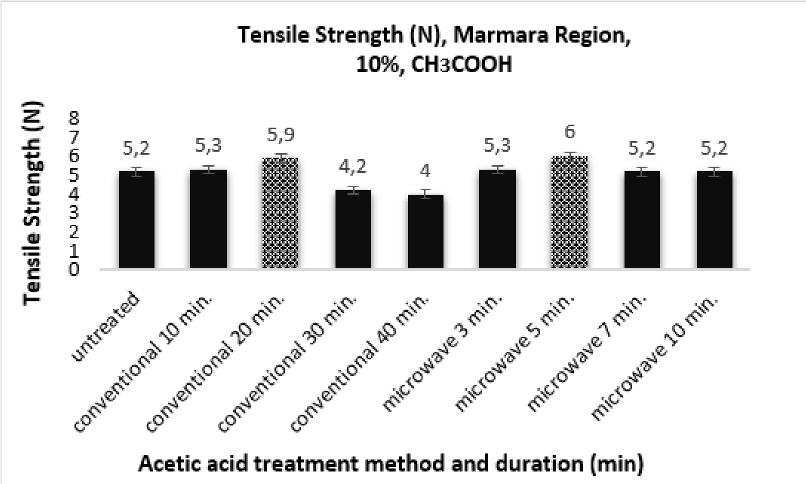


Figure 5. The tensile strength values of acetic acid treated fibers with conventional and microwave energy methods.

In the surface modification of the okra fibers with acetic acid by conventional method, generally the strength reduction is seen relative to the untreated fiber. Decrease in strength was determined depending on the increase in the processing time. The highest strength value was obtained at 10% concentration and 20 minutes of operation.

Improvement in strength is generally seen in surface modification of okra fibers with acetic acid by microwave method compared to untreated fiber. At 7 and 10 minutes of operation, the tensile strength values are at the same level as the untreated fiber. The highest strength was obtained at 10% concentration and 5 minutes of operation.

When the strength values were examined, it was determined that the highest strength values for both chemicals were obtained by the modification made by the microwave method.

The treated fibers which have highest strenght values were selected and their SEM, X-Ray analysis and diameter images were given in the following results.

B. Fibers Diameter

The physical diameters (μm) and longitudinal images of okra body waste fibers obtained from the Marmara Region were obtained by using light microscopes. The diameter changes images of untreated and treated fibers with different chemicals/process parameters are shown in Figure 6-8.

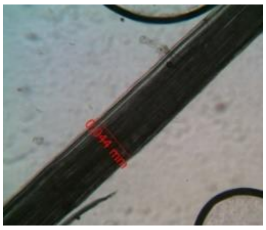
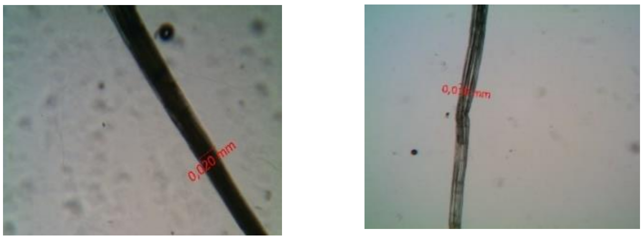


Figure 6. Untreated okra waste fiber.



Conventional method 30 min **Microwave method 5 min**

Figure 7. Longitudinal images of Marmara region okra waste fibers surface modified with 7 % $NaHSO_4$ by different methods.

Conventional method 20 min **Microwave method 5 min**

Figure 8. Longitudinal images of Marmara region okra waste fibers surface modified with 10 % CH_3COOH by different methods.

When the fiber diameters investigated, it was found that the microwave method applied samples gave the best results for both chemicals. Microwave-treated fibers contain impurity removal and high amounts of fiber fibrillation. Thus, the fibers treated with the microwave method are more thin than the fibers treated with the conventional method.

C. Crystallinity of Okra Fibers (XRD)

Crystallinity index values obtained from surface modification of okra fibers with different chemical and both methods are given in Figures 9-13. The results were compared with the untreated sample.

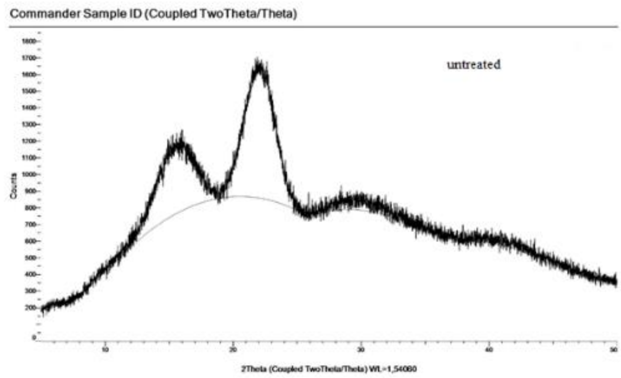


Figure 9. X-ray diffractometer results of untreated okra fibers.

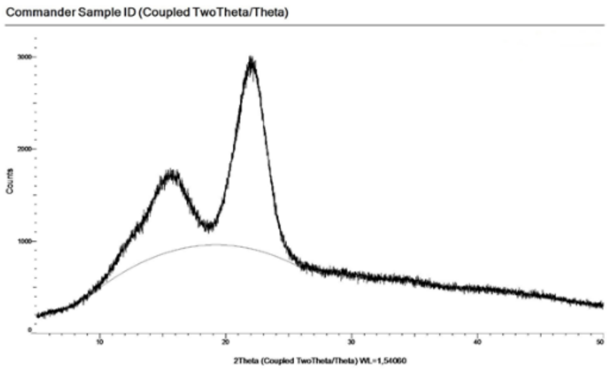


Figure 10. X-ray diffractometer results of conventionally treated fibers with 7% $NaHSO_4$ for 30 min.

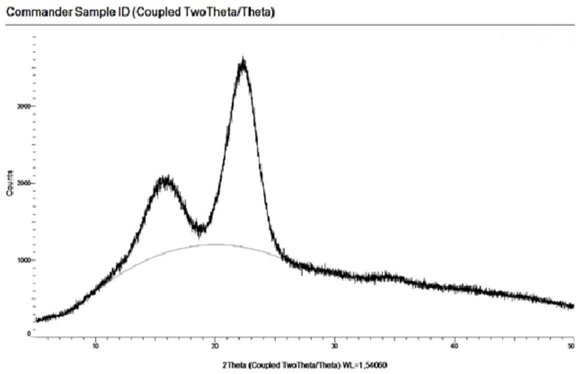


Figure 11. X-ray diffractometer results of treated fibers by microwave method with 7 % $NaHSO_4$ for 5 min.

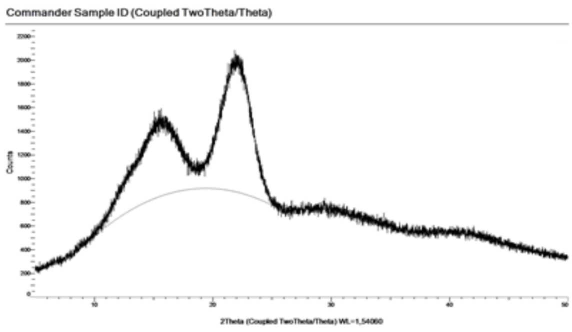


Figure 12. X-ray diffractometer results of conventionally treated fibers with 10% CH_3COOH method, 20 min.

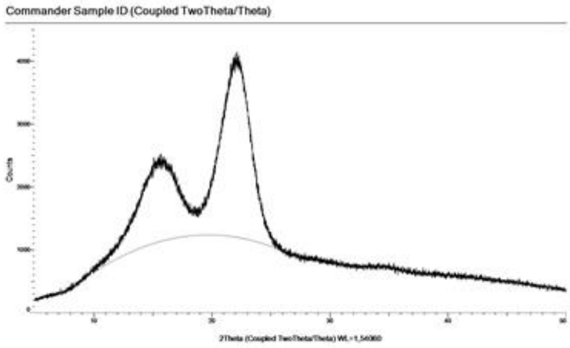


Figure 13. X-ray diffractometer results of treated fibers by microwave method with 10% CH_3COOH for 5 min.

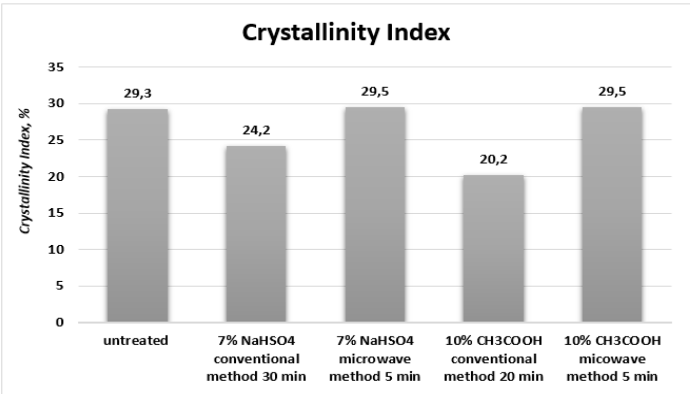


Figure 14. The crystallinity index (%) values of the fibers treated with sodium bisulphate and acetic acid.

The crystallinity index (%) values of okra stem fibers treated with 7% sodium bisulphate and 10% acetic acid at different times are seen in the Figure 14 . Accordingly, microwave treated fibers with sodium bisulphate and acetic acid have the highest crystallinity index value.It has been found that microwave treated okra stem fibers are shown higher crystallinity than the conventionally treated okra fibers. Innovative method have more effective results by the microwave method in a shorter time.

D. SEM Images

The SEM images of untreated / treated fibers are shown in Figure 15-19.

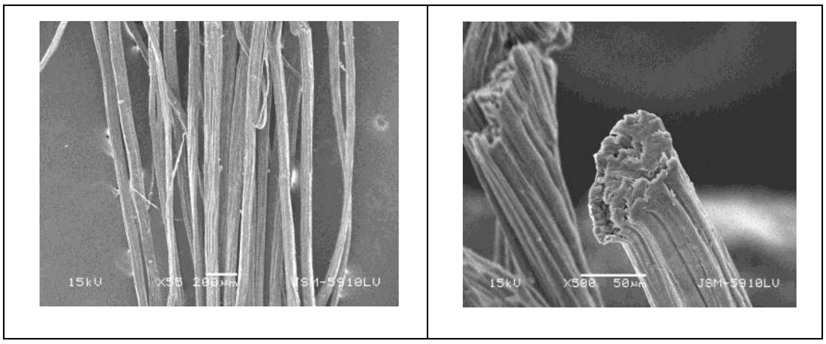


Figure 15. Longitudinal and cross-sectional SEM images of the untreated okra fiber.

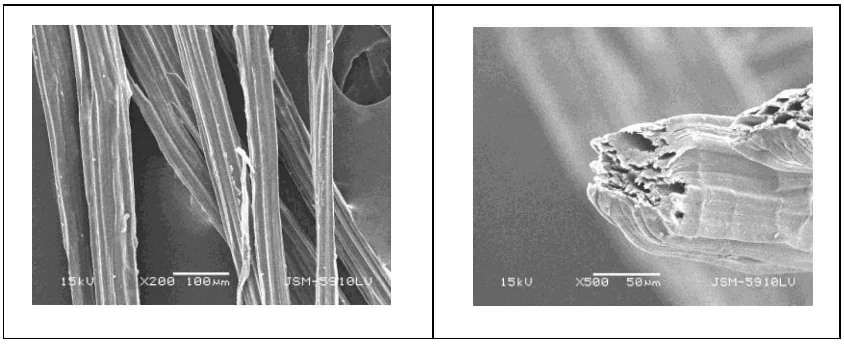


Figure 16. Longitudinal and cross-sectional SEM images of the okra body fibers treated with sodium bisulphate by conventional method.

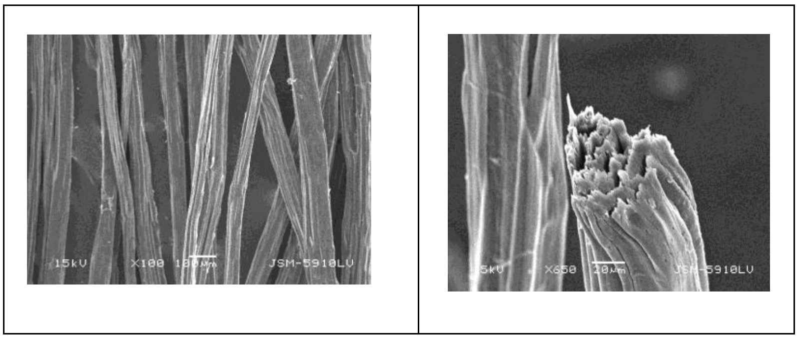


Figure 17. Longitudinal and cross-sectional SEM images of the okra fibers treated with sodium bisulphate and microwave energy by method.

.

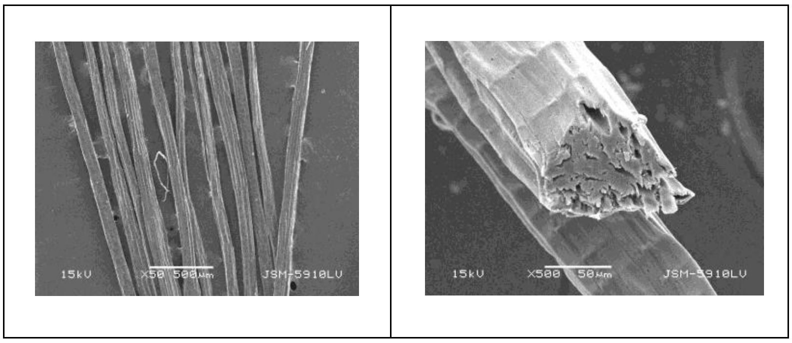


Figure 18. Longitudinal and cross-sectional SEM images of the okra fibers treated with acetic acid by conventional method.

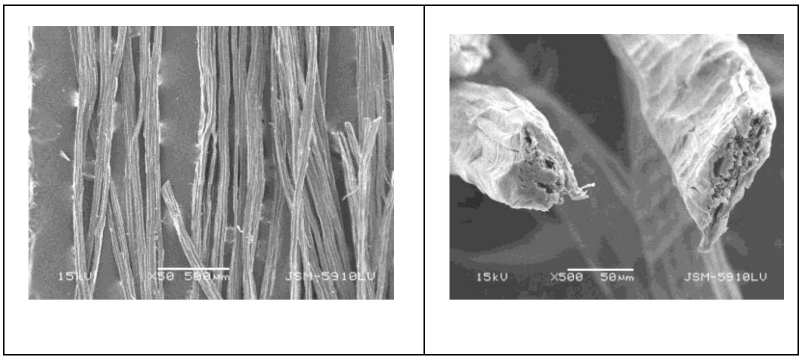


Figure 19. Longitudinal and cross-sectional SEM images of the okra fibers treated with acetic acid by microwave energy method.

SEM images of okra fibers after conventionally and microwave assisted treatments with both chemicals are shown in Figure. 15-19. High amount of porosity and fibrillation are observed in okra fibers treated with acetic acid by microwave method.

Following the treatment with sodium bisulphate and acetic acid by means of microwave, the outer layers of parenchyma cells have been removed to expose the inner fibres[8]. Impurities and waxes were effectively removed from the fiber surface with both metdos and chemicals compared to untreated fibers.

Conclusions

Two different processes carried out with acetic acid and sodium bisulphate chemicals were proved to be successful. The modification of fiber surface with microwave energy method had better results than conventionally surface modifications. The fibre strength were increased by microwave method.

According to the tensile strength, fiber diameter, SEM and X-Ray analysis results of untreated and treated okra fibers, higher tensile strength properties were obtained after treatments by microwave processes. Microwave process was more effective than the conventional process for surface modification of okra fibers and provided energy and time saving. Also microwave energy is more effective than conventional treatment in chemical reactions.

Considering all these results, surface treatments with sodium bisulphate and acetic acid by microwave energy have a positive effect on mechanical and morphological properties of okra fibers which were obtained from Marmara Region agricultural waste.

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] Asumani O.M.L, Reid R.G, Paskaramoorthy R, The effects of alkali–silane treatment on the tensile and flexural properties of short fibre non-woven kenaf reinforced polypropylene composites, Composites: Part A 43, 1431–1440,(2012).

[2] Al-oqla F.M, Sapuan, S.M, Natural fiber reinforced polymer composites in industrial applications: feasibility of date palm fibers for sustainable automotive industry, Journal of Cleaner Production, 66, 347-354, (2014).

[3] Koçak, E.D, Mıstık, S.İ, Merdan, N, Nayci Duman, M, Nonwoven Production from Agricultural Okra Wastes and İnvestigation of Their Thermal Conductivities, IOP Conference Series: Materials Science and Engineering, 254,(2017).

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

[5] Li X, Tabil L.G, Panigrahi S, Chemical Treatments of Natural Fiber for Use in Natural Fiber-Reinforced Composites: A Review, Journal of Polymers and the Environment, 15,25–33, (2007).

[6] Yılmaz Şahinbaşkan B, Koçak E.D, Merdan N,Akalın Mehmet, Dyeing of Polypropylene Blends by Using Microwave Energy. Journal of Engineered Fibers and Fabrics, 12, 20-27,(2017)

[7] Kocak D, Merdan N, Yüksek M. and Sancak E, Effects of Chemical Modifications on Mechanical Properties of Luffa cylindrica, Asian Journal of Chemistry, Vol.25, No.2 637-641,(2013),

[8] Merdan N, Sancak E, Kocak D. and Yuksek M, Effect of Applied Different Surface Modification Processes with Cellulose Enzyme on Properties of Luffa Fibres, Asian Journal of Chemistry, Vol.24, No.3,975-980,(2012).