ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 22, No. 3. 2016

Effect of the Ecological Methods on the Surface Modification of the Kenaf Fibers

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crossref http://dx.doi.org/10.5755/j01.ms.22.3.8694

Received 15 November 2014; accepted 11 October 2015

Kenaf is a biodegradable and environmental friendly bast fibre. The most rapidly expanding application field for kenaf fibre is composites as reinforcing material. In this study four different chemical treatments were applied to kenaf fibre by using conventional, ultrasonic and microwave methods. Weight loss, tensile strength, elongation properties, morphological characteristics (SEM) and FTIR analysis of the treated kenaf fibre were carried out. Valuable results were obtained from formic acid and acetic acid treatments of kenaf fibre by ultrasonic and microwave methods. The reasons for the ultrasonic and microwave processes to be successful are the strength achieved by sonication and microwave.
Keywords: bio fibre, surface treatment, kenaf fibre, ultrasonic energy, microwaves.

1. INTRODUCTION

Harms of the synthetic based polymers are discussed in recent years. Using of the natural materials instead of the synthetic based polymers is groving rapidly [1, 2].

Properties of the natural fibers such as biodegradability, sustainability, good mechanical and thermal insulation properties, low density and price are increasing the usage of them [3 – 5]. Some lignocelluloses based fibers, which are used in composites and garment production, are hydrophilic due to their strong polarized hydroxyl groups [6, 7].

Good adhesion should be provided between environmental friendly fibres and matrix for the production of the composites [8]. Therefore, chemical surface treatments are applied to the natural fibers to improve the adhesion with matrix. After surface treatments degradation and swelling occurs on fibers.

Kenaf fibers are annual plant (hibiscus cannabinus L.) and belong to Malveceae family. It grows on warm climate zones of the world. Kenaf grows rapidly and reaches to 3 m. Kenaf is a stem fiber and fibers are obtained from the 34 – 38 % of the stem. Kenaf fibers are processed by extrusion, molding and nonwoven processes due to their excellent tensile and flexural strength properties. Kenaf fibers are widely used in European automotive industry for sound insulation [9 – 11]. Chemical surface treatments are required to increase the usage of the kenaf fibers in the industry. But conventional surface treatments consume too much water, chemicals and energy. Environmental friendly methods exist alternatively to conventional method. Surface treatments performed by using sound waves (ultrasonic) and microwave energy is alternative environmental friendly methods [12 – 17]. Ultrasonic energy is occurred by rising microscopic bubbles known as cavitation [18, 19]. Cavitation has chemical and mechanical effects. For example, decomposing of high molecular mass by dispersion effect, and carries the chemicals to the capillaries of the fibers by degasing effect, these processes are performed by consuming low energy, low substances and in short time [19 – 21].

Another environment friendly method is using of microwave energy. Microwave is a high frequency radio waves located between 30 MHz – 30.000 MHz on infrared spectrum [22]. Microwave energy is used in drying, dyeing and printing processes due to its rapid and energy efficient properties [21]. In order to apply microwave energy to a material, material should have dielectric loss and dipolar electric charges should occur in the material when electromagnetic field applied. Therefore, aqueous structures or structures existing in water are suitable for the microwave heating due to easy dipolar electric charge composing properties of the water molecules.

Microwave energy method is defined as environment friendly method because of its accelerator and time reducing effects on chemical processes. Using the microwave energy in textile wet processes is related with application, research and development studies which enclose application areas of heating up, drying, condensation, dying and printing processes [23 – 26]. Microwave energy method proved its environment friendly effect, especially on polyester dying, coloration of flax and cotton, surface treatments of cellulosic and protein based fibers [27 – 29].

In this study, four different chemical treatments were applied to kenaf fibre by using conventional, ultrasonic and microwave methods. Than weight loss, tensile strength, elongation properties, morphological characteristics (SEM) and FTIR analysis of the treated kenaf fibre were carried out.

2. EXPERIMENTAL

2.1. Materials

Kenaf fibres were collected from South Africa. Their overall lengths were between 30 mm and 60 mm. The

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kenaf fibres were washed with water to remove the adhering dirt (20 °C distilled water for 30 min). They were dried in an oven at 70 °C for 4 hours. After treatments they were conditioned for 48 hours prior to testing under 20 ± 2 °C and 65 ± 2 % RH conditions.

2.2. Fibre treatments

Kenaf fibres were exposed to three processes: Table 1 and Table 2 show the procedures and temperature-time settings of the conventional, ultrasonic and microwave processes. Conventional process was applied in laboratory condition. Branson B2200B E4 (220 V and 205 W) ultrasonic bath was used for the ultrasonic method with 20 kHz frequencies. Microwave method was performed by using Kenwood Mw 440 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 vessel and treated by the microwave energy according to the experimental design.

2.3. Analytical methods

The weight loss was determined on atmospherically conditioned kenaf fibres after different treatment processes. The weight loss percentage (*Wj*) was calculated from the differences in weight using the following equation:

$$W_j = \frac{W_{pre} - W_{after}}{W_{pre}} \cdot 100\,(\%), \qquad (1)$$

where W_{pre} is the weight of the conditioned fibres prior to pre-treatment and W_{after} is the weight after performed pre-treatment.

Table 1. Procedures of the conventional, ultrasonic and microwave processes

Chemicals	Producer-Code	Concentration	Rinsing process (25°C, 10 min, pH 7)	Drying process
Sodium hydroxide	Merck (106462)	5 g/L	Distilled water	At room temperature
Sodium hydroxide and Ethanol	Merck (106462 and 100983)	10 g/L	Ethanol	At room temperature
Formic acid	Merck (100263)	99 %	Distilled water	At room temperature
Acetic acid	Merck (109944)	100 %	Distilled water	At room temperature

Table 2. Temperature and time settings of the conventional, ultrasonic and microwave processes

	Conventional		Ultrasonic		Microwave	
	T, °C	t, min	T, °C	t, min	T, °C	t, min
Sodium hydroxide	23	30	23	30	82	7
Sodium hydroxide and ethanol	78	120	78	60	82	7
Formic acid	20	40	20	20	82	7
Acetic acid	30	40	30	20	82	7

2.4. Testing and characterization of kenaf fibres after chemical treatments

Mechanical properties of the kenaf fibres after treatments were determined according to ASTM D 3822 test method by using Instron 4411 (50 N load, 10 mm/min speed) tensile test instrument. Morphology properties were investigated by using JEOL JSM-5410 LV Scanning Electron Microscope operated at 20 kV.

2.5. FTIR spectroscopy test

The attenuated total reflectance-Fourier transform infrared (FT-IR) characterization of the fabrics has been determined using a Perkin Elmer Spectrum 100 FT-IR model spectrophotometer. The Fourier transform infrared attenuated total reflection (FT-IR) spectra were recorded on a Perkin Elmer Spectrum 100 FTIR model spectrophotometer with accessory diamond. Afterwards, the analysis was performed using a Perkin Elmer Spectrometer (Spectrum 100). The FT-IR spectrum of kenaf fibers were recorded in the range of 4000 – 380 cm^{-1} (resolution: 2 cm^{-1}).

3. RESULTS AND DISCUSSION

3.1. Weight loss of kenaf fibres

Swelling is a physic chemical phenomenon, occuring when natural fibres contact with water and chemicals. Water molecules penetrate into the voids of the fibers open the hydrogen bonds and make the cellulosic fibers adequate for swelling. During the swelling process hydrogen bonds of the cellulose molecules are broken and water molecules migrate among the hydrogen bonds. Thereby, water molecules bonds to the OH – groups of the cellulose fibers by the help of hydrogen bonds and as a result of these phenomenon cellulose fibers begins to swell. The degree of the swelling depends on to the free hydroxyl groups of the fiber [30].

Several factors affect the swelling of the cellulose such as ratio of the crystalline and amorphous zones, amount of the OH groups, concentration and type of the solvent. Also degree of the swelling changes depending on the temperature, pH, acidic groups, concentration of the electrolyte, environmental and chemical factors [31].

Weight loss results of the kenaf fibre after chemical treatments are given in Fig. 1.

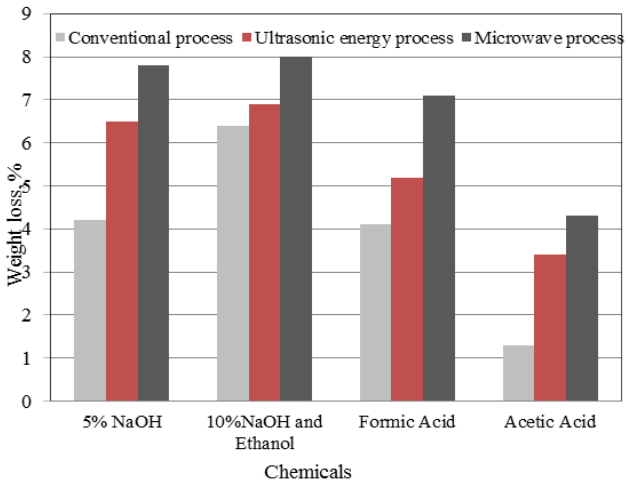

Fig. 1. Weight loss of kenaf fibers for different treatment conditions (at room temperature)

Chemical treatment processes on the decomposition of impurities and natural pigments are generally characterized by weight loss. It is evident from the results that using NaOH, formic acid and acetic acid caused weight lost [48 – 50]. Higher weight losses were obtained by ultrasonic and microwave methods in all processes, by the help of the sonication and microwave energy. By the rapid and short microwave energy method, after acetylation process, chemicals extended the morphological structure of the kenaf fibre. As a result of the measurements; acetic acid caused the fiber to lose less weight than other chemicals.

3.2. Fibre tensile strength and elongation test

Morphological structure of the fiber, crystalline and amorphous zone ratios and orientations, physical and chemical properties affect the mechanical properties of the lignocellulose-based fibers. Moisture absorption capabilities of the lignocellulos fibers change according to structure of the fiber. Treatment of the cellulosic fibers increases the distribution of the fibers in composites. Also treatment processes roughen the surface of the fiber and applications of chemical vapour increase the mechanical properties of the cellulosic fiber reinforced composites [32]. Fig. 2 and Fig. 3 show the tensile strength and elongation properties of the kenaf fibres after conventional, ultrasonic and microwave chemical treatment processes.

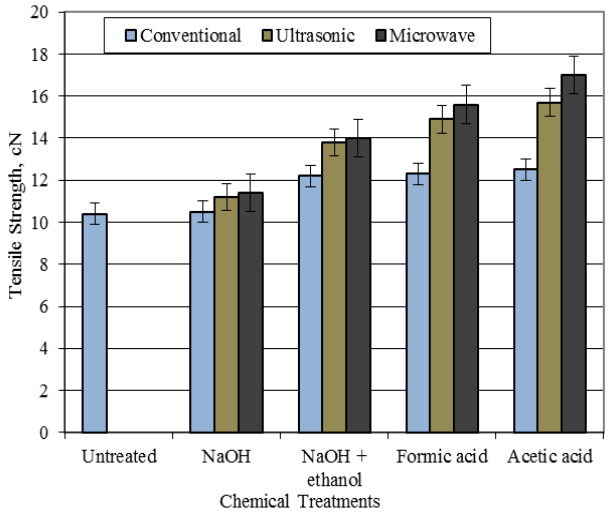

Fig. 2. Tensile strength values of untreated and NaOH, NaOH + ethanol, formic acid and acetic acid treated kenaf fibers

According to the tensile strength results tensile strength of the kenaf fibre increased after all chemical treatments by using all methods. Better results were obtained by using alternative ecological methods (ultrasonic and microwave methods) than conventional method. Higher tensile strength and elongation properties were obtained from NaOH + ethanol, acetic acid and formic acid treatments than NaOH treatment. NaOH treatment is a conventional mercerization process, which removes the part of lignin and hemicellulose and help the fiber getting wet easily [33].

Alkali treatments also affect the cellulose fibrils and decrease the polymerization degree of the cellulose and affect the quantity of the lignin and hemicelluloses removed from the fiber [34]. Therefore, amorphous zones of the fibers increase so elongation properties of the kenaf fibers increase [35, 36]. By the alkali process oil and wax of the cellulose fibers are partially removed and many open cellulose open ends occurred. Therefore, free energy of the fiber surface was increased, fiber surface was roughened and mechanical bonding occurred between fiber and polymer matrix.

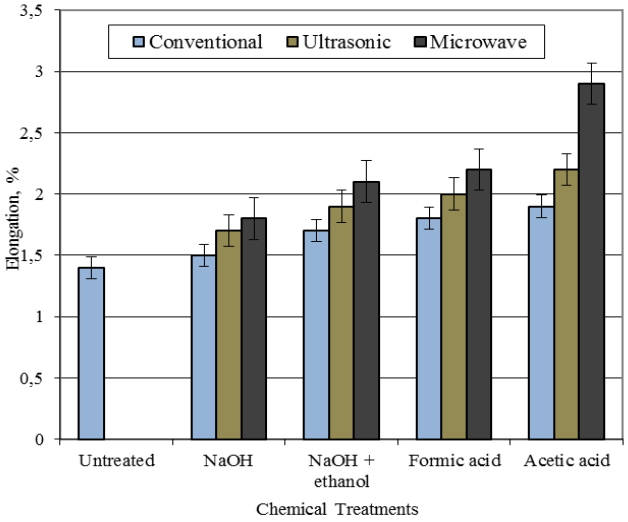

Fig. 3. Elongation values of untreated and NaOH, NaOH + ethanol, formic acid and acetic acid treated kenaf fibers

Tensile strength and elongation properties of the NaOH + ethanol treated kenaf fibers were better than NaOH treated kenaf fibers. The reason of that is kenaf fibers first treated with NaOH and then treated with Ethanol, by the activation of the –OH groups of the ethanol, hydroxyl groups of the cellulose and lignin were affected [37, 38].

Tensile strength and elongation properties of the formic acid treated kenaf fibers were higher than the NaOH and NaOH + ethanol treated kenaf fibers. Baley at al. indicated that formic acid treatments have improved the wettability, surface energy and mechanical properties of the fibers better than the alkali treatments [39]. Formic acid diffused into the fiber better than NaOH and improved the mechanical properties by the help of ultrasonic energy and microwave energy methods. Oils and waxes were removed by the formic acid so kenaf fibers were fibrillated and elongation properties were improved due to the increasing of the amorphous areas, without decreasing the tensile strength properties.

Application of acetic acid (CH_3COOH) to the cellulosic fibers is known as acetylation or esterification method [40, 41]. Tensile strength and elongation properties of the acetylated kenaf fibers were higher than the NaOH, NaOH + ethanol and formic acid treated kenaf fibers. Acetylated fibers interact with hydroxyl groups, which are situated on the cell wall, thus become effective between cross linked lignin and cellulose [42 – 44]. After acetylation, fibrillary structure is increased and also amorphous structure is developped due to the changes occurred on cell wall. So elongation properties of the kenaf fiber are increased. Mechanical properties of the treated kenaf fibers were better when treated by using ecological processes (ultrasonic and microwave). By using ultrasonic

process, micro bubbles caused by ultrasonic energy carry chemicals into the kenaf fibers. Chemicals are carried into the kenaf fibers in the form of wave of the ultrasonic energy. Thus, molecules of the chemical substances are carried in a short time without harming the fiber morphology. Because of that reason, molecular chains of the kenaf fibers are not damaged and tensile strength and elongation properties of the fibers are higher than conventional process. The best mechanical properties were obtained by using microwave energy treatment process. Microwave energy system creates high frequency and dielectric heating system and molecular structures of kenaf fibers move side-by-side because of the electric field created by the microwave energy. During this movement, the friction raises spectacular heat. This heating starts in every point of the fiber not only on the surface and starts at the same time so this heating effect occurs rapid and uniform. Therefore, high speed and effective heating is occupied in a short time. In the liquid, molecules of the chemical substances are diffused into the kenaf fibers efficiently. That's why heating of a material by using microwave energy is more economic than conventional process. Mechanical properties of the fibers are not affected negatively due to the short reaction time. Even, better wrinkle recovery resistance and tensile strength results were obtained by using microwave energy than conventional process on the esterification of the cotton fibers [45 – 48].

3.3. FTIR spectroscopy

The effects of fibre treatments on the kenaf fibre surfaces were also studied by using FTIR (Fig. 4).

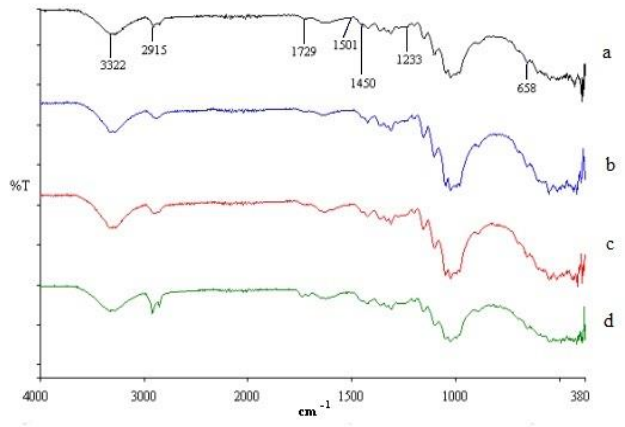

Fig. 4. FTIR spectra: a – untreated; b – conventional treated; c – ultrasonic treated; d – microwave treated kenaf fibre

Peak values of the acetic acid treated kenaf fibers were investigated till 4000 cm^{-1} band. According to the spectra 1500 $cm^{-1}$is lignin, 664 cm^{-1} is COOH and 1450 cm^{-1} is CH_2 peaks. The peak at 2900 cm^{-1} is attributed to the CH stretch of the microwave treated kenaf fibers. Kenaf fibres treated with acetic acid by using conventional, ultrasonic and microwave methods show the absorption band of carbonyl stretching at 1730 cm^{-1}. The peak at 1730 cm^{-1} is attributed to the C = O stretching of the acetyl groups of hemicellulose. The removal of hemicellulose from the fiber surfaces causes this peak to disappear [15, 16]. The peak at 1233 cm^{-1}, which is C = O stretch of acetyl group of lignin is reduced. This was due to the partial removal of lignin from the fiber surface [16, 17].

3.4. Fibre surface morphology

Morphology of the acid treated kenaf fibers by using conventional, ultrasonic and microwave methods were investigated and SEM micrographs are given in Fig. 5.

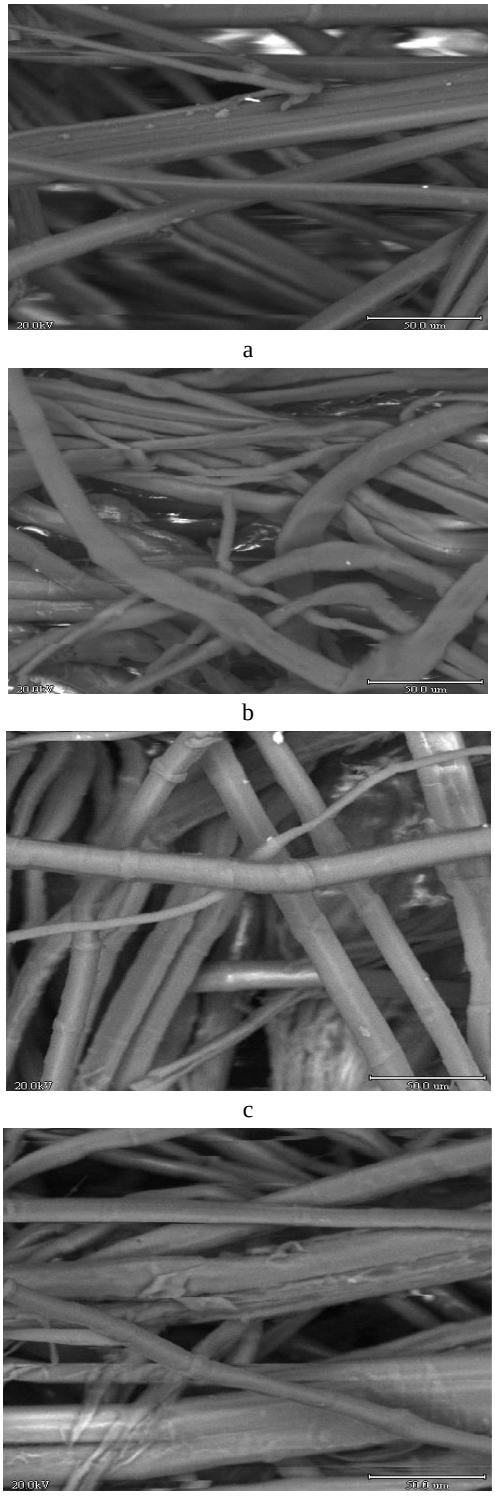

Fig. 5. SEM micrographs: a – untreated kenaf fiber; b – acetic acid treated kenaf fiber by using conventional method; c – acetic acid treated kenaf fiber by using ultrasonic energy method; d – acetic acid treated kenaf fiber by using microwave method

Acetylation reaction of the acetic acid treatment was effective on the SEM surface morphology of the kenaf fibers. Impurities, inorganic substances, oils and waxes, and non-cellulosic substances were removed from the fiber surface. Chemical treatment of the fiber can clean the fiber surface, chemically modify the surface and increase the surface roughness. Chemical treatments with acetic acid by means of microwave and ultrasonic energy, the outer layers of parenchyma cells have been removed to expose the inner fibres. Fig. 5 c and d show that outer layer of kenaf fibres were removed more by using microwave and ultrasonic energy processes. As seen from the SEM micrographs, fibrillation occurred on the surface of the kenaf fibers by using microwave energy treatment in a short time and without damaging the molecular structure and without decreasing the mechanical properties [40].

4. CONCLUSIONS

According to the tensile strength and elongation properties of the untreated and treated kenaf fibers higher tensile strength and elongation properties were obtained by using ultrasonic and microwave processes. Microwave process was more effective than the ultrasonic process. The reason for the ultrasonic and microwave processes to be successful is the strength achieved by sonication and microwave energy. Ultrasonic energy and microwave processes provide both energy and time saving. Also microwave is more effective than ultrasonic energy in chemical reactions. Considering all these results, formic acid and acetic acid methods were found to adequately modify the fibre's surfaces. In this study, %weight loss increase is outstanding with the success of ridding the impurities. Weight loss results can be correlated with mechanical test results, which can be treated as a proof for enhanced interfacial interactions with ultrasonic energy and microwave treatments.

REFERENCES

1. **Wambua, P., Ivens, J., Verpoest, I.** Natural Fibres: Can They Replace Glass in Fibre Reinforced Plastics? *Composite Science and Technology* 63 (9) 2003: pp. 1259 – 1264. http://dx.doi.org/10.1016/S0266-3538(03)00096-4
2. **Tserki, V., Zafeiropoulos, N. E., Simon, F., Panayiotou, C.** A Study of the Effect Acetylation and Propionylation Surface Treatments on Natural Fibres *Composites Part A* 36 (8) 2005: pp. 1110 – 1118.
3. **Li, Y., Mai, Y.W., Ye, L.** Sisal Fibre and Its Composites: A Review of Recent Developments *Composite Science and Technology* 60 (11) 2000: pp. 2037 – 2055.
4. **Wong, S., Shanks, R., Hodzic, A.** Interfacial Improvements in Poly (3-hydroxybutyrate) – Flax Fibre Composites with Hydrogen Bonding Additives *Composite Science and Technology* 64 2004: pp. 1321 – 1330.
5. **Weyenberg, I. V., Ivens, J., Coster, A. D., Kino, B., Baetens, E., Verpoest, I.** Influence of Processing and Chemical Treatment of Flax Fibres on Their Composites *Composite Science and Technology* 63 (9) 2003: pp. 1241 – 1246.
6. **Baiardo, M., Zini, E., Scandola, M.** Flax Fibre – Polyester Composites *Composites Part A* 35 (6) 2004: pp. 703 – 710. http://dx.doi.org/10.1016/j.compositesa.2004.02.004
7. **Gassan, J., Bledzki, A. K.** The Influence of Fiber-Surface Treatment on the Mechanical Properties of Jute-Polypropylene Composites *Composites Part A* 28 (12) 1997: pp. 1001 – 1005. http://dx.doi.org/10.1016/S1359-835X(97)00042-0
8. **Piggott, M. R., Sanadi, A., Chua, P. S., Andison, D.** Mechanical Interactions in the Interfacial Region of Fiber Reinforced Thermosets. In: Ishida H, Koenig JL, Editors. Composites Interfaces. Elsevier Science Publishing, 109 1986.
9. **Chen, Y., Chiparus, O., Sun, L., Negulescu, I., Parikh, D. V., Calamari, T. A.** Natural Fibers for Automotive Nonwoven Composites *Journal of Industrial Textiles* 35 (1) 2005: pp. 47 – 62.
10. **Mohanty, A. K., Misra, M., Hinrichsen, G.** Biofibres, Biodegradable Polymers and Biocomposites: An Overview *Macromolacular Materials and Engineering* 276 (1) 2000: pp. 1 – 24. http://dx.doi.org/10.1002/(sici)1439-2054(20000301)276:1<1::aid-mame1>3.0.co;2-w
11. **Aziz, S. H., Ansell, M. P.** The Effect of Alkalization and Fibre Alignment on the Mechanical and Thermal Properties of Kenaf and Hemp Bast Fibre Composites: Part 1-Polyester Resin Matrix *Composites Science and Technology* 64 (9) 2004: pp. 1219 – 1230. http://dx.doi.org/10.1016/j.compscitech.2003.10.001
12. **Mistik, S. I.** Effect of Ultrasonic Energy on Reductive Cleaning of Dyed Polyester Fabrics *Asian Journal of Chemistry* 24 (8) 2012: pp. 3724 – 3728.
13. **Kocak, D.** Study of the Effect of Different Chemical Compound Applied on Luffa Cylindrical Fibres with the Help of Ultrasonic Energy *Journal of Polymer Engineering* 28 (8) 2008: pp. 501 – 518.
14. **Merdan, N., Inanici, Y.** Using of Ultrasonic Energy in Dyeing of Microfiber *Asian Journal of Chemistry* 24 (3) 2012: pp. 969 – 974**.**
15. **Merdan, N.** Ultrasonic Energy Utilization in Implementation UV Absorbers to Cotton Fabrics *Asian Journal of Chemistry* 23 (3) 2011: pp. 1263 – 1268**.**
16. **Suslick, K. S., Cline, R. E., Hammerton, D. A.** Sonochemical Hot Pot *Journal of American Chemical Soc*iety 108 (18) 1986: pp. 5641 – 5642. http://dx.doi.org/10.1021/ja00278a055
17. **Mason, T. J.** Sonochemistry. Oxford, New York, 1999.
18. **Vajnhandl, S., Le Marechal, A. M.** Ultrasound in Textile Dyeing and the Decolouration/Mineralization of Textile Dyes *Dyes Pigment* 65 (2) 2005: pp. 89 – 101.
19. **Beckham, H. W., Zhan, J., Good, J., Mock, G., McCall, B., Cato, M., Klutz, D., Mills, G.** Fundamental Investigations of Ultrasonic Effects in Textile Wet Processing. Natl Text Cent Ann Rep August: 193, 1995.
20. **Vouters, M., Rumeau, P., Tierce, P., Costes, S.** Ultrasounds: An Industrial Solution to Optimize Costs, Environmental Requests and Quality for Textile Finishing *Ultrasonic Sonochemistry* 11 (1) 2004: pp. 33 – 38. http://dx.doi.org/10.1016/S1350-4177(03)00113-5
21. **Polona, D. D.** "Woven Fabric Engineering", Book Edited by, Chapter 16" The Microwaves – Solution for Improving Woven Fabric, Drago Katovic, ISBN 978-953-307-194-7, 2010.
22. **Haggag, K.** Fixation of Pad-Dyeings on Cotton Using Microwave Heating *American Dyestuff Reporter* 26 (8) 1990: pp. 26 – 30.

23. **Ozerdam, A., Tarakcıoglu, I., Ozguney, A.** The Use of Microwave Energy for the Fixation of Reactive Printed Cotton Fabrics *Tekstil ve Konfeksiyon* 4 2008: pp. 289 – 296.
24. **Keshwani, D. R., Cheng, J. J., Burns, J. C., Li, L., Chiang, V.** Microwave Pre-Treatment of Switch Grass to Enhance Enzymatic Hydrolysis. In: Proceedings of the ASABE Annual International Meeting, No. 077127, 2007.
25. **Nanda, R., Patel, G.** Microwave Oven: A Tool for Guide Response in Shade Translation in Reactive Dyeing *Colourage* 49 (12) 2002: pp. 83 – 88.
26. **Sun, H., Lin, L., Jiang, X., Bai, X.** The Improvement of Dyeability of Flax Fibre by Microwave Treatment *Pigment Resin Technol*ogy 34 (4) 2005: pp. 190 – 196.
27. **Merdan, N., Sancak, E., Kocak, D.** Effects of Applied Different Surface Modification Processes with Cellulose Enzyme on Properties of Luffa Fibres *Asian Journal of Chemistry* 24 **(**3) 2012: pp. 975 – 980.
28. **Luukko, K., Maloney, T. C.** Swelling of Mechanical Pulp Fibres *Cellulose* 6 (2) 1999: pp. 123 – 136.
29. **Lindström, T., Carlsson, G.** The Effect of Chemical Environment on Fibre Swelling *Svensk Papperstidning* 85 (3) 1982: pp. 14 – 20.
30. **Bledzki, A. K., Gassan, J.** Composites Reinforced with Cellulose Based Fibres *Progress in Polymer Science* 24 (2) 1999: pp. 221 – 274.
31. **Gassan, J., Bledzki, A. K.** Alkali Treatment of Jute Fibres: Relationship between Structure and Mechanical Properties *Journal of Applied Polymer Science* 71 1999: pp. 623 – 629.
32. **Li, X., Tabil, L.G., Panigrahi, S.** Chemical Treatments of Natural Fibre for Use in Natural-Fibre Reinforced Composites: A Review *Journal of Polymer Environment* 15 2007: pp. 25 – 33.
33. **Joseph, K., Mattoso, L.H.C., Toledo, R.D., Thomas, S., de Carvalho, L.H, Pothen, L., Kala, S., James, B.** ''Natural Fiber Reinforced Thermoplastic Composites,'' in Natural Polymers and Agrofibers Composites, E. Frollini, A.L. Lea˜o, and L.H.C. Mattoso, Eds., Sa˜n Carlos, Brazil, Embrapa, USP-IQSC, UNESP, 2000: pp. 159.
34. **Mishra, S., Misra, M., Tripathy, S. S., Nayak, S. K., Mohanty, A. K.** The Influence of Chemical Surface Modification on the Performance of Sisal-Polyester Biocomposites *Polymer Composites* 23 (2) 2002: pp. 164 – 170. http://dx.doi.org/10.1002/pc.10422
35. **Doan, T. T. L.** Investigation on Jute Fibres and their Composites Based on Polypropylene and Epoxy Matrices doctoral thesis, Der Fakultat Maschinenwesen DerTechnischen Universitat Dresden, 2006.
36. **Wang, B.** Pre-treatment of Flax Fibers for Use in Rotationally Molded Biocomposites, M.Sc. Thesis, University of Saskatchewan, 2004.
37. **Baley, C., Busnel, F., Grohens, Y., Sire, O.** Influence of Chemical Treatments on Surface Properties and Adhesion of Flax Fibre-Polyester Resin *Composites: Part A* 37 (10) 2006: pp. 1626 – 1637. http://dx.doi.org/10.1016/j.compositesa.2005.10.014
38. **Andersson, M., Tillman, A. M**. Acetylation of Jute: Effects on Strength, Rot Resistance and Hydrophobicity *Journal of Applied Polymer Science* 37 1989: pp. 3437 – 3447. http://dx.doi.org/10.1002/app.1989.070371214
39. **Flemming, M., Ziegmann, G., Roth, S.** Faserverbund bauweisen: Fasern und Matrices, Springer, Berlin, 1995: pp. 1 – 5. http://dx.doi.org/10.1007/978-3-642-57776-5_1
40. **Rong, M. Z., Zhang, M. Q., Liu, Y., Yang, G. C., Zeng, H. M.** The Effect of Fibre Treatment on the Mechanical Properties of Unidirectional Sisal Reinforced Epoxy Composites *Composite Science and Technology* 61 (10) 2001: pp. 1437 – 1447.
41. **Hill, A. S. C., Abdul Khalil, H. P. S., Hale, M. D.** A Study of the Potential of Acetylation to Improve the Properties of Plant Fibres *Industrial Crops and Products* 8 (1) 1998: pp. 53 – 63.
42. **Sreekala, M. S., Thomas, S.** Effect of Fibre Surface Modification on Water Sorption Characteristics of Oil Plam Fibres *Composite Science and Technology* 63 (6) 2003: pp. 861 – 869.
43. **Satge, C., Verneuil, B., Branland, P., Granet, R., Krausz, P., Rozier, J., Petit, C.** Rapid Homogeneous Esterification of Cellulose Induced by Microwave Irradiation *Carbohydrate Polymers* 49 (3) 2002: pp. 373 – 376.
44. **Katovic, D., Bischof, V. S.** Application of Microwaves in Textile Finishing Processes *Tekstil* 54 (7) 2005: pp. 313 – 318.
45. **Thostenson, E. T., Chou, T. W.** Microwave Processing: Fundamentals and Applications *Composites: Part A Applied Science and Manufacturing* 30 (9) 1999: pp. 1055 – 1071.
46. **Jones, D. A., Lelyveld, T. P., Mavrofidis, S. D., Kingman, S. W., Miles, N. J.** Microwave Heating Applications in Environmental Engineering: A Review *Resources, Conservation and Recycling* 34 (2) 2002: pp. 75 – 90. http://dx.doi.org/10.1016/S0921-3449(01)00088-X
47. **Ibrahim, N. A., Hadithon, K. A., Abdan, K.** Effect of Fibre Treatment on Mechanical Properties of Kenaf Fibre – Ecoflex Composites *Journal of Reinforced Plastics and Composites* 29 (14) 2010: pp. 2192 – 2198. http://dx.doi.org/10.1177/0731684409347592
48. **Liu, W., Mohanty, A. K., Askeland, P., Drzal, L. T., Misra, M.** Effect of Alkali Treatment on the Structure Morphology and Thermal Properties of Native Grass Fibres as Reinforcement for Polymer Matrix Composites *Journal of Material Science* 39 2004: pp. 1051 – 1054.