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SUPPLEMENTARY MATERIAL TO
**Effect of chemical treatments on the chemical composition and
properties of flax fibers**

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FLAX FIBERS AND THEIR APPLICATION

From the beginning of human history, flax (*Linum ussitatissimum*) has been cultivated for production of fibers and seeds. Currently, flax fibers are very important raw textile materials for textile^{1–5} and industrial applications,^{6–10} such as pulp and composite materials.

As for the textile applications, flax fibers are increasingly used due to their excellent properties, *i.e.*, very high strength, specific luster and handle, high absorbency and hygroscopicity, good thermal and electrostatic properties, protection against UV radiation, lack of any allergenic effect, optimum conditions for the skin, possibility of use in blends with cotton and chemical fibers, as well as many different possibilities for fiber modification in order to impart special or modify existing fiber properties, such as hydrophilicity, hydrophobicity, sorption, antimicrobial, and other properties.^{1,11–18}

Today, there is an increased interest in the use of plant fibers by different industrial sectors, such as automotive, shipbuilding, architecture, *etc.*, mainly to replace glass fibers. Flax fibers together with other plant fibers have been one of the most attractive fillers for different types of polymers including rubbers, as well as for ceramic matrices due to some of their unique characteristics unparalleled with any other reinforcing/filler materials.^{6,7–10,19} They include renewability, biodegradability, good availability, low cost and density, limited damage to the processing equipment, reduced health hazard and reasonable strength and

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stiffness. Furthermore, there has been a clear trend toward the study and use of bioderived and bioinspired materials for many different biomedical applications, such as surgical meshes¹⁷ and wound dressing materials.²⁰

Non-cellulosic components of flax fibers

Pectin, the main component of pectinous gums, acts mainly as the bonding agents between the elementary fibers in technical flax fibers obtained from flax plants. Pectin is located in the middle lamella and the primary wall, while lignin, located in the middle lamella and the secondary wall, is responsible for the rigidity of the cell wall. The pectins of the middle lamella are encrusted with lignin, which makes it even more difficult for their removal and to split the bundles into fibers. Hemicelluloses are deposited as the amorphous and unoriented cell-wall constituent, which occupy spaces between the fibrils in both the primary and secondary walls.²¹

MATERIALS

Domestic traditionally water-retted flax fibers were used in this investigation. The flax fibers samples were obtained from the flax Venica variety (Czech Republic) grown at the experimental grounds in the vicinity of Banja Luka (the Republic of Srpska, Bosnia and Herzegovina). Flax stems were pulled after 100-day vegetative period and cold water-retted for 15 days. The flax fibers were separated by breaking, scutching and hackling procedures. The investigations were performed on technical (multi-cellular) long flax fibers. The chemical composition of the used fibers, determined according the procedure described later, was water solubles, 1.81 %; fats and waxes, 1.73 %; pectin, 6.97 %; α -cellulose, 75.81 %; hemicelluloses, 7.84 %; lignin, 4.03 % and ash, 0.83 %. All used chemicals were of p.a. grade.

THE CHEMICAL TREATMENT SCHEME AND LIST OF SAMPLES

TABLE S-I. The chemical treatment scheme and list of samples

Sample code	Modification condition		
	Concentration	Temperature	Time, min
FC		Unmodified sample – control	
FH5R30	5 % NaOH	Room temperature	30
FH5R60			60
FH5R120			120
FH7R30	7 % NaOH	Room temperature	30
FH7R60			60
FH7R120			120
FH10R30	10 % NaOH	Room temperature	30
FH10R60			60
FH10R120			120

TABLE S-I. Continued

Sample code	Modification condition		
	Concentration	Temperature	Time, min
FH18R30	18 % NaOH	Room temperature	30
FH18R60			60
FH18R120			120
FH7B30	7 % NaOH	Boiling temperature	30
FH7B60			60
FH7B120			120
FH10B30	10 % NaOH	Boiling temperature	30
FH10B60			60
FH10B120			120
FC5B30	5 g L ⁻¹ NaClO ₂	Boiling temperature	30
FC5B45			45
FC5B60			60
FC10B30	10 g L ⁻¹ NaClO ₂	Boiling temperature	30
FC10B45			45
FC10B60			60
FC15B30	15 g L ⁻¹ NaClO ₂	Boiling temperature	30
FC15B45			45
FC15B60			60
FC20B30	20 g L ⁻¹ NaClO ₂	Boiling temperature	30
FC20B45			45
FC20B60			60

TABLE S-II. The chemical composition and copper number of unmodified and modified flax fibers

Sample code	α -cellulose content, %	Hemicelluloses content, %	Kappa number	Copper number
FC	75.81	7.84	27.53	1.37
FH5R30	86.88	9.15	8.55	0.97
FH5R60	86.43	8.96	8.53	0.94
FH5R120	87.27	8.79	8.42	0.96
FH7R30	87.97	7.29	8.49	0.72
FH7R60	88.58	7.21	8.42	0.68
FH7R120	87.48	6.75	7.98	0.63
FH10R30	91.57	4.34	8.32	0.65
FH10R60	91.98	4.04	8.38	0.73
FH10R120	91.62	4.33	7.68	0.70
FH18R30	92.78	3.89	8.01	0.63
FH18R60	92.33	3.44	7.62	0.58
FH18R120	91.84	2.93	6.74	0.54
FH5B30	95.76	3.11	7.96	0.53
FH5B60	96.30	2.57	7.12	0.43
FH5B120	96.73	2.40	6.46	0.40

TABLE S-II. Continued

Sample code	α -cellulose content, %	Hemicelluloses content, %	Kappa number	Copper number
FH7B30	95.78	2.70	7.27	0.36
FH7B60	96.16	2.49	6.66	0.27
FH7B120	96.39	2.38	5.94	0.21
FH10B30	95.68	2.71	6.35	0.31
FH10B60	95.36	2.46	6.11	0.23
FH10B120	95.78	2.22	5.57	0.15
FC5B30	85.00	10.40	2.85	0.96
FC5B45	85.30	10.55	2.71	0.95
FC5B60	85.23	10.68	2.43	0.97
FC10B30	85.71	10.60	2.56	1.00
FC10B45	85.14	10.68	2.48	0.98
FC10B60	85.10	10.98	2.02	0.99
FC15B30	85.03	10.56	2.42	1.09
FC15B45	85.12	10.69	2.00	1.10
FC15B60	86.35	9.78	1.74	1.12
FC20B30	86.75	9.77	2.01	1.11
FC20B45	85.97	10.86	1.56	1.14
FC20B60	86.27	10.06	1.07	1.19

IODINE SORPTION VALUE (*ISV*) AND CRYSTALLINITY INDEX (*CrI*) OF
UNTREATED AND TREATED FLAX FIBERS

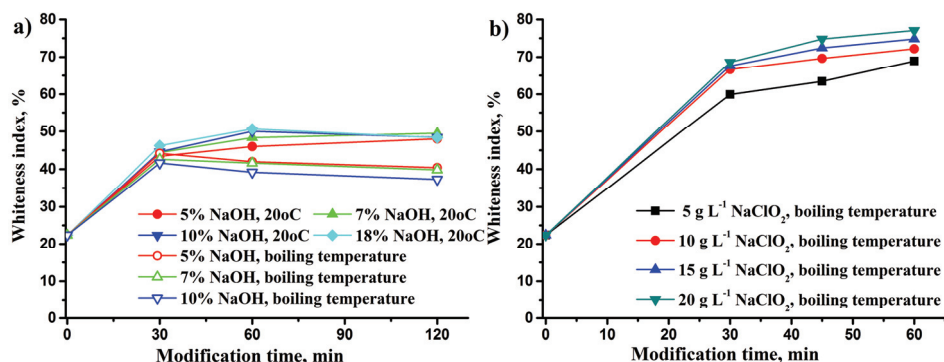
TABLE S-III. Iodine sorption value (*ISV*) and crystallinity index (*CrI*) of untreated and treated flax fibers

Sample code	<i>ISV</i> / mg I ₂ g ⁻¹ cellulose	<i>CrI</i> / %
FC	118.1	71.3
FH5R30	112.5	72.7
FH5R60	107.4	73.9
FH5R120	99.9	75.7
FH10R30	148.0	64.1
FH10R60	149.9	63.6
FH10R120	154.6	62.5
FH18R30	149.4	63.7
FH18R60	145.1	64.8
FH18R120	149.2	63.8
FH5B30	74.3	82.0
FH5B60	90.0	78.2
FH5B120	98.4	76.1
FH10B30	129.3	68.6
FH10B60	128.5	68.8
FH10B120	133.8	67.5

TABLE S-III. Continued

Sample code	ISV / mg I ₂ g ⁻¹ cellulose	CrI / %
FC5B30	97.2	76.4
FC5B45	96.7	76.5
FC5B60	93.9	77.2
FC20B30	88.2	78.6
FC20B45	88.5	78.5
FC20B60	89.2	78.4

WHITENESS INDEX OF THE UNMODIFIED AND MODIFIED FLAX FIBERS

Fig. S-1. Whiteness index of the unmodified flax fibers and flax fibers modified with NaOH and NaClO₂.

REFERENCES

1. E. S. Abdel-Halima, *Carbohydr. Polym.* **88** (2012) 1201
2. N. A. Ibrahim, M. El-Hossamy, M. M. Hashem, R. Refa, B. M. Eid, *Carbohydr. Polym.* **74** (2008) 880
3. L. Jun, F. Jiahao, Z. Hua, Z. Jianchun, *Fibres Text. East. Eur.* **18** (2010) 81
4. A. Lefeuvre, A. Bourmaud, L. Lebrun, C. Morvan, C. Baley, *Ind. Crop. Prod.* **50** (2013) 400
5. H. M. Wang, R. Postle, *Text. Res. J.* **74** (2004) 781
6. D. G. Hepworth, D. M. Bruc, J. F. V. Vincent, G. Jeronimidis, *J. Mater. Sci.* **35** (2000) 293
7. B. Bax, J. Müssig, *Compos. Sci. Technol.* **68** (2008) 1601
8. S. Goutianos, T. Peijs, B. Nystrom, M. Skrifvars, *Appl. Compos. Mater.* **13** (2006) 199
9. D. Puglia, J. Biagiotti, J. M. Kenny, *J. Nat. Fibers* **1** (2004) 23
10. H. Zhang, R. Ming, G. Yang, Y. Li, Q. Li, H. Shao, *Polym. Eng. Sci.* **55** (2015) 2553
11. E. S. Abdel-Halim, W. Konczewicz, M. Zimmiewska, S. S. Al-Deyab, M. H. El-Newehy, *Carbohydr. Polym.* **82** (2010) 195
12. M. Zimmiewska, R. Kozlowski, M. Rawluk, *J. Nat. Fibers* **1** (2004) 69
13. A. J. Jhala, L. M. Hall, *Aust. J. Basic Appl. Sci.* **4** (2010) 4304
14. F. Paladini, R. A. Picca, M. C. Sportelli, N. Cioffi, A. Sannino, M. Pollini, *Mater. Sci. Eng., C* **52** (2015) 1
15. M. N. Belgacem, A. Gandini, *Compos. Interfaces* **12** (2005) 41

16. R. Surina, M. Andrassy, *Text. Res. J.* **83** (2013) 66
17. S. A. A. X. Michel, R. R. M. Vogels, N. D. Bouvy, M. L. W. Knetsch, N. M. S. Akker, M. J. J. Gijbels, C. Marel, J. Vermeersch, D. G. M. Molin, L. H. Koole, *J. Biomed. Mater. Res., B* **102** (2014) 477
18. S. Alix, E. Philippe, A. Bessadok, L. Lebrun, C. Morvan, S. Marais, *Biores. Technol.* **100** (2009) 4742
19. P. Muensri, T. Kunanopparat, P. Menut, S. Siriwattanayotin, *Composites* **42** (2011) 173
20. K. Skorkowska-Telichowska, M. Żuk, A. Kulma, A. Bugajska-Prusak, K. Ratajczak, K. Gasiorowski, K. Kostyn, J. Szopa, *Wound Repair Regen.* **18**,(2010) 168
21. M. Kostic, B. Pejic, P. Skundric, *Bioresource Technol.* **99** (2008) 94.