Alkali treated bagasse for journal

by Juliana Anggono

ALKALI TREATMENT OF LIGNOCELLULOSIC FIBERS EXTRACTED FROM SUGARCANE BAGASSE: COMPOSITION, STRUCTURE, PROPERTIES

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ABSTRACT

Lignocellulosic fibers extracted from sugarcane bagasse were treated with NaOH solutions of different concentration (0-40 wt%) to study the effect of alkali treatment on the composition, structure and properties of the fibers. Composition was determined by the van Soest method, structure was characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM), while mechanical properties by tensile testing. Hemicellulose and lignin content decrease, while cellulose content goes through a maximum as a function of alkali concentration. Crystallinity changes only slightly and microfibril angle (MFA) remains constant thus structural effects and especially MFA are not the primary reasons for changing properties. The stiffness of the fibers shows a slight maximum at around 2-4 wt% NaOH content, while strength goes through a much more pronounced one at around 5-8 wt%. Direct correlation between structure and mechanical properties was not found indicating that composition is more important in the determination of properties than structure. Regression analysis proved that the combination of several compositional variables determines mechanical properties in a non-linear manner. The improvement in fiber properties was explained with the dissolution of weak amorphous fractions and the relative increase of cellulose content.

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1. INTRODUCTION

Because of the continuous search for new materials and the increasing environmental awareness of the industry as well as the public, the interest in materials from renewable resources increases continuously [1]. Biopolymers are synthesized from natural raw materials [2,3] and starch [3,4], cellulose [3,5] and lignin [6,7] are used in increasing quantities in all areas of life. Plastics are often reinforced with fibers [8-10] in order to increase their stiffness and strength, and traditional fibers are replaced with wood or natural fibers in larger and larger extent. Many products are prepared with natural reinforcement including wood plastic composites (WPC) or various automotive parts [11-13]. However, besides their advantages, natural reinforcements have several drawbacks like sensitivity to heat during processing, limited dimensional stability due to water adsorption, poor adhesion between the matrix polymer and the reinforcement, as well as small transverse strength [14]. In order to overcome these deficiencies, attempts are made to improve properties by surface modification, coupling or the treatment of the fibers [15].

One of the approaches to improve the inherent properties of natural reinforcements and thus those of their composites is the alkali treatment of the fibers. Mercerization is a commercial technology developed a long time ago which consists of the treatment of the fibers with a sodium hydroxide solution of 20-27 wt% under tension [16]. The treatment results in increased strength, improved sheen and easier dyeing. Today, all kinds of alkali treatment are called mercerization [17]. The improvement of strength is the main reason and goal of using this approach for the treatment of fibers applied as reinforcements in plastics.

The effect of alkali treatment on the structure and properties of fibers was studied by numerous groups and the increase of stiffness and strength was observed quite often.

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However, the explanation for the improvement of mechanical properties is rather controversial in many cases. The concentration of sodium hydroxide and the time of treatment varies in a very wide range from 0.03 wt% to 40 % and from a few minutes to 48 hours [18-25]. It is generally accepted that the composition of the fibers changes as the result of the treatment; the amorphous parts are dissolved thus the hemicellulose, lignin and wax content of the fibers decrease, although Taha et al. [23] claimed that lignin content remains constant. The groups agree much less about the reasons of the improvement in strength. Although structural changes are thought to result in these changes in most cases, the opinions about the main factors differ widely. Crystallinity is one of these factors, but it was shown to increase [19,26], decrease [27] or go through a maximum [21,24,28,29] as a function of NaOH concentration and time. Mechanical properties are often plotted against crystallinity but the correlations are rarely convincing. Another factor might be a change in the microfibril angle [17,19,30,31] or crystal modification [30], i.e. the transformation from the cellulose I to the cellulose II form as the result of the treatment [28,32]. Alkali treatment removes waxes and amorphous components from the surface of the fibers thus changing surface roughness [26,33-35], which was claimed to improve interfacial adhesion [28]. On the other hand, Gassan and Bledzki [18] think that changing surface quality does not modify composite properties. Unfortunately, the statements are not very often supported with sufficient experimental evidence and the controversies are difficult to resolve.

Nevertheless, it is a fact that under certain conditions the stiffness and strength of natural fibers are improved by alkali treatment, which results in an increase of composite modulus and/or strength, as a consequence. Occasionally, an improvement or maximum is observed in the strength of composites when a polymer is reinforced with treated fibers. Van de Weyenberg et al. [22] reinforced epoxy resin with fibers modified by alkali treatment

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and found considerable increase in the transverse strength of the composites. They explained the improvement with a change in interfacial adhesion, which was contradicted by Gassan and Bledzki [18].

In spite of the contradictions published, the beneficial effect of alkali treatment for fiber characteristics is clear and it often results in better composite properties as well. Considering the contradictions mentioned above, the goal of our study was to treat sugarcane bagasse fibers with sodium hydroxide and then thoroughly characterize their composition, structure and properties including strength and stiffness. Sugarcane bagasse was selected, because it is a cheap, natural raw material, which can be obtained from local sources in Indonesia. Bagasse fiber form a waste and its value added application would be beneficial for the country. In a part of the study, flax fibers were used as reference material in order to extend the validity of our conclusions. An attempt was made to correlate the measured variables and find a plausible explanation for the increase of fiber strength with alkali treatment. The consequence for practice is also mentioned in the final section of the paper.

2. EXPERIMENTAL

2.1. Materials

The bagasse fibers were obtained directly from the sugar mill. They were washed with ethanol, dried, cut up and sieved. Before cutting, longer individual fibers were separated for mechanical characterization, tensile testing. A solution containing 40 wt% sodium hydroxide was prepared from NaOH flakes (Molar Chemicals, Hungary). The solution was diluted to obtain solutions with 1, 2, 4, 8, 15, 20 and 40 wt% alkali content. After treatment the fibers were neutralized with an acetic acid (Molar Chemicals,

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Hungary) solution of 10 wt% concentration prepared from concentrated acetic acid (96 wt%).

2.2. Fiber treatment and sample preparation

Sieved fibers were placed into a beaker and sodium hydroxide solutions of various concentration were poured on them. The suspension was stirred occasionally during the 1 hour of the treatment. Subsequently the fibers were neutralized with acetic acid and then they were washed several times until the pH of the washing water was 7. The fibers were dried at 105 °C in an air circulation oven for 48 hours. Fibers were prepared in a similar way for tensile testing with the only difference that fibers of 15-20 cm length were separated first and then dried under tension to avoid curling. All treatments were done at ambient temperature.

Fibers had to be milled for X-ray diffraction and FTIR measurements. Two grams of the fibers were placed into a Retsch MM 400 ball mill (Retsch GmbH., Germany) and the fibers were ground for 2.5 min at 30 s^{-1} frequency.

3.3. Characterization, measurements

The chemical composition of the fibers was determined by the van Soest method. The detailed description of the method can be found in the paper of van Soest [36]. According to the method, hemicellulose content is determined after treatment with an acidic detergent solution, the amount of cellulose by treating the fibers with sulfuric acid of 72 wt% concentration and lignin by burning the sample in an oven. Composition was analyzed also by Fourier transform infrared spectroscopy (FTIR). Spectra were recorded on KBr pastilles using a Bruker Tensor 27A (Bruker, Massachusetts, US) apparatus. Two

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mg fiber was mixed with 248 mg KBr for the preparation of the samples. Spectra were recorded from 4000 to 400 cm^{-1} at 2 cm^{-1} resolution with 32 scans. Absorbances appearing at 1428 and 1372 cm^{-1} were assigned to cellulose, those detected at 1730, 1249 and 1040 cm^{-1} to hemicellulose and the one observed at 1514 cm^{-1} to lignin. The crystalline structure of the fibers was characterized by X-ray diffraction (XRD). The traces were recorded on powder samples using a Philips PW 1830/PW 1050 (Philips, Netherlands) apparatus with CuK$_\alpha$ radiation at 40 kV and 35 mA anode excitation in the 2θ range between 4 and 40° with 0.04° steps. Crystallinity and microfibril angle (MFA) were determined from the reflection of the cellulose detected at 22.8° according to the method described by Yamamoto [37].

The mechanical properties of the fibers were characterized by tensile testing. Fibers were fixed onto paper frames for the measurements. An Instron 5566 tensile testing machine (Instron, Massachusetts, US) was used for the tests at the gauge length of 20 mm and crosshead speed of 0.5 mm/min. Ten parallel measurements were carried out for each sample. Scanning electron microscopy (SEM) was used for the characterization of the morphology of the neat and treated fibers. The equipment used was a Jeol JSM 6380 LA (Jeol, Japan) apparatus. Before the recording of the images, the samples were sputtered with gold.

3. RESULTS AND DISCUSSION

The results of the experiments are presented in several sections. First, the effect of chemical treatment on the composition of the fibers is discussed, followed by the presentation of changes in their structure. Properties are analyzed next and then correlations between composition and properties are shown in the last section together

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with relevance for practice.

3.1. Composition

The analysis of results published in the literature clearly showed that one of the consequences of the alkali treatment of fibers is a change in their composition. Amorphous parts of the fibers, mainly hemicellulose, lignin and waxes, are dissolved in different extents. At larger concentrations of NaOH and at longer times, also cellulose crystals disintegrate and dissolve. The change of chemical composition inevitably leads to the modification of properties as well. Accordingly, the effect of treatment on composition was followed by two methods, chemical analysis and FTIR spectroscopy.

The cellulose and lignin content of the fibers are plotted against the concentration of the treating solution in **Figure 1**. The cellulose content is large, exceeds 50-60 wt%, while the amount of lignin is below 20 wt%, as expected for such fibers. In accordance with most literature references, lignin content decreases continuously with increasing NaOH concentration. On the other hand, cellulose content shows a maximum. The maximum results from the fact that the dissolution of amorphous components is considerably faster than that of the cellulose crystals, but the latter also degrades at very large alkali concentrations. The observed increase in cellulose content may result in the improvement of mechanical properties as reported many times in the literature [21,29]. Hemicellulose content also decreases with increasing NaOH concentration, while the amount of ash remains constant [19,26,28,34,35,38,39].

As mentioned above, compositional changes were followed also by FTIR spectroscopy. The spectra of the fibers after selected NaOH contents are presented in **Figure 2**. The quantitative analysis of peaks assigned to the various components of the

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fiber yielded the same results as chemical analysis. The inset of **Figure 2** shows the decrease of hemicellulose (1730 cm^{-1}) and lignin (1514 cm^{-1}) content with increasing NaOH concentration. Both chemical analysis and FTIR spectroscopy confirmed the expectations that the chemical composition of the fibers changes during treatment and the changes must result in modified structure and properties.

The chemical composition of the fibers changes because some of the components are dissolved during treatment. The rate of dissolution is different for the various components, but all lead to the decrease of the weight of the sample. Weight loss is plotted against the NaOH concentration of the treating solution in **Figure 3**. According to the figure, the decrease of weight is very substantial at large NaOH contents, the fibers start to lose their integrity. Considering the large change in weight, we may conclude that treatments with solutions above 15 wt% NaOH deteriorate the fibers, at least in the time frame (1 hour) used in these experiments.

3.2. Structure

Cellulose has various crystal forms. In plants, it crystallizes in the cellulose I form, which may transform into the cellulose II modification as the result of alkali treatment [28,32]. Treatment loosens up structure and the sodium ions as well as the water present make possible the transformation. Interesting to note that such transformation was rarely reported during the alkali treatment of fibers used as reinforcements in plastics. Parallel to the change in crystal modification, crystallinity may be also modified during treatment. XRD traces recorded on fibers treated with NaOH solutions of different concentration are plotted in **Figure 4**. According to the traces, crystal form does not change in our case even at the largest alkali concentration. On the other hand, crystallinity and order change

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quite considerably as NaOH concentration increases. Cellulose crystals retain their integrity up to 8 wt% NaOH content, but the regularity of the crystals decreases considerably above that concentration. The quantitative analysis of the results shows that crystallinity increases from about 58 % to 63 % at 5 wt% NaOH content of the solution, but decreases steeply above this alkali concentration down to around 40 %, confirming the conclusions drawn by the visual observation of the traces presented in **Figure 4**.

The improvement of the mechanical properties of the fibers is often explained with the change of the microfibril angle (MFA) during alkali treatment [30]. Decreasing MFA results in better alignment of the fibrils to the direction of the load and thus larger stiffness and strength [22]. MFA can be determined from XRD traces using the approach proposed by Yamamoto [37]. MFA values are plotted against the NaOH content of the treating solution in **Figure 5**. MFA did not change at all for the sugarcane bagasse fibers used in this study, and we detected only very slight changes for flax fibers used as reference. According to these results the change in microfibril angle cannot explain the improvement or changes in the mechanical properties of the fibers, if there is any.

For polymers reinforced with natural fibers, the changes in properties are often assigned to the modification of the surface of the fibers. The removal of waxes and increased surface roughness are claimed to improve interfacial adhesion and thus stiffness and strength [26,33-35] of composites reinforced with natural fibers. The surface morphology of the fibers is demonstrated by the SEM micrographs presented in **Figure 6**. The surface of the neat fiber is relatively smooth and even (**Figure 6a**), but sharper contours appear already after the treatment with 4 wt% NaOH solution (**Figure 6b**). Deep groves (**Figure 6c**) and even larger holes (**Figure 6d**) appear after the treatment with solutions of larger concentrations, and the fibers start to lose their integrity as indicated also by the XRD

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traces. Several authors claim that changing surface morphology improves interfacial adhesion and fiber or composite properties [20,39-41], but Gassan and Bledzki [18] question this claim and relate property changes to the shrinkage of the fibers.

3.3. Properties

The effect of alkali treatment on the stiffness of the fibers is presented in **Figure 7a**. Flax is used as reference in the figure. The modulus of flax is considerably larger than that of the bagasse fibers. The difference might be caused by the dissimilar microfibril angle, which is around 25° for the bagasse and 18° for the flax fibers. In both cases a maximum appears in stiffness at around 3-5 wt% NaOH content of the treating solution. The maximum is more pronounced for flax, but it can be clearly observed also in the case of bagasse. The location of the maximum on the NaOH concentration axis differs from the one determined for cellulose content, which raises some doubt about the exclusive role of this latter factor in the changes of mechanical properties. On the other hand, the maximum determined in crystallinity is much closer to this value.

The tensile strength of the fibers is plotted against NaOH content in **Figure 7b**. The strength of bagasse fibers is larger than that of the flax fibers indicating that instead of microfibril angle other factors determine fiber strength. A maximum is observed in this case too, it appears at around 5-8 wt% for bagasse and closer to 5 wt% for the flax fibers. The maximum is clear, fiber strength increases considerably as an effect of the treatment, and then it decreases at larger alkali concentrations. The increase in fiber strength justifies the increase observed in the strength of composites prepared with various fibers treated with NaOH [22,40,42]. The decrease in strength, to very small values in the case of flax, at larger NaOH content is in accordance with earlier conclusions drawn from the XRD and

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SEM studies. We might mention here also the deformability of the fibers and the effect of alkali treatment on them. The elongation-at-break of the bagasse fibers is larger, it is around 5 %, while that of the flax fibers is approximately 3 % in the average, and it does not change much upon treatment. Although we confirmed the beneficial effect of alkali treatment on the properties of natural fibers and proved that it results from the change of composition and structure, we do not have an unambiguous explanation for the improvement and do not know the determining factor either.

3.4. Correlations

The results presented above indicate clearly that composition and structure determines properties as well as that alkali treatment modifies both factors. In publications, mechanical properties are very often related to the crystallinity of the fibers [21,29]. The Young's modulus and strength of the fibers are plotted against crystallinity in **Figure 8**. Strength apparently increases with increasing crystallinity, while a maximum seems to exist for modulus. We must call the attention here, though, that the standard deviation of the measurements is large and the correlations are rather loose. Crystallinity must influence mechanical properties, but it is not the determining factor, others must play a role as well.

The unambiguous determination of the dominating factor is very difficult, because probably more than one influence properties and their weight might be similar. Moreover, various factors are related to each other, they either change simultaneously or even depend on each other, thus the identification of the dominating one is very difficult or even impossible. Since the correlation between mechanical properties and crystallinity is weak, we decided to analyze the combined effect of various compositional factors on

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properties. We carried out various multiple regression analyses and identified the significant factors. We arrived to the conclusion that a non-linear model taking into account first-order compositional variables and their interactions describes property change quite well. The following model:

$$\sigma = -36.7[C] + 39.1[HC] - 340.5[L] + 8.3[C][L] \tag{1}$$

where σ is the tensile strength of the fiber, while $[C]$, $[HC]$ and $[L]$ is its cellulose, hemicellulose and lignin content, respectively, was obtained in the analysis. The model takes into account the fact that the sum of the components must be 100 %. The two dimensional surface plot of the model is presented in **Figure 9**. The ash content of the fibers was fixed at 2 %. Measured values are also shown in the figure as red symbols together with the corresponding value. Although deviations can be observed between the predicted and measured values, the agreement is reasonable, the determination coefficient, i.e. the goodness of the fit is 0.9110.

Figure 9 proves that the combination of several compositional variables determine the mechanical properties of the fibers. The overall effect is not linear and the interaction of the variables (see the factor $[C][L]$) further complicates evaluation. In order to see the predictive power and validity of the approach, we plotted the calculated fiber strength against the measured values. The correlation is presented in **Figure 10**. Considering the uncertainty of the measurement and the large standard deviation of the measured values (see **Figure 7b**), the correlation is excellent, thus we can state that the changes in composition during alkali treatment unambiguously determine mechanical properties.

Although the correlation shown in **Figure 10** is unambiguous, one might object that the structure of the fibers must determine properties. This might be true, but MFA

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did not change at all and the relationship between mechanical properties and crystallinity was very weak (**Figure 8**). On the other hand, if we consider that the cellulose crystals are much stiffer and stronger than the amorphous components of the fiber, one can easily accept that the dissolution of a part of the amorphous phase results in an improvement of mechanical properties. The maximum in strength was observed at around 5 wt% NaOH content of the treating solution and both hemicellulose and even the lignin content are still considerable even after the treatment. Obviously, the weakest fraction is dissolved during treatment resulting in the improvements of properties. We can expect, as a result, that fibers treated with NaOH solution of 5 wt% concentration will reinforce polymers more than neat fibers and composite properties will improve in accordance with published results [28,43,44].

4. CONCLUSIONS

The results on the alkali treatment of sugarcane bagasse fibers proved that the treatment modifies the composition, structure and properties of the fibers. Hemicellulose and lignin content decrease, while cellulose content goes through a maximum as a function of the alkali content of the treating solution. Crystallinity changes only slightly and microfibril angle remains constant, does not change with NaOH content thus structural effects and especially MFA are not the primary reasons for changing properties, contrary to many reports published in the literature. The stiffness of the fibers shows a slight maximum at around 2-4 wt% NaOH content, while strength a much more pronounced one at around 5-8 wt%. The increase of fiber strength is quite considerable. Direct correlation between structure and mechanical properties were not found indicating that composition is more important in the determination of properties than structure.

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Regression analysis proved that the combination of several compositional variables determines mechanical properties in a non-linear manner. The improvement in fiber properties was explained with the dissolution of a weak amorphous fraction and with the increase of cellulose content. The optimum concentration of the treating solution is around 5 wt% NaOH content if the time of treatment is fixed at 1 hour. The increase of fiber strength is expected to result in the improvement of composite properties.

6. REFERENCES

1. Gandini, A.; Belgacem, M. N. In Monomers, polymers and composites from renewable resources; Belgacem, M. N.; Gandini, A., Eds.; Elsevier: Amsterdam, **2008**, pp 1-16.

2. Jiang, L.; Zhang, J. In Handbook of biopolymers and biodegradable plastics; Ebnesajjad, S., Ed.; William Andrew Publishing: Boston, **2013**, pp 109-128.

3. Endres, H.-J.; Siebert-Raths, A. In Engineering biopolymers; Endres, H.-J.; Siebert-Raths, A., Eds.; Hanser: München, **2011**, pp 71-148.

15

4.   Carvalho, A. J. F. In Handbook of biopolymers and biodegradable plastics; Ebnesajjad, S., Ed.; William Andrew Publishing: Boston, **2013**, pp 129-152.

5.   Dufresne, A. In Handbook of biopolymers and biodegradable plastics; Ebnesajjad, S., Ed.; William Andrew Publishing: Boston, **2013**, pp153-169.

6.   Gandini, A.; Belgacem, M. N. In Monomers, polymers and composites from renewable resources; Belgacem, M. N.; Gandini, A., Eds.; Elsevier: Amsterdam, **2008**, pp 243-271.

7.   Hatakeyama, H.; Hatakeyama, T. In Biopolymers: lignin, proteins, bioactive nanocomposites; Abe, A.; Dusek, K.; Kobayashi, S., Eds.; Springer: Berlin, **2010**, pp 1-63.

8.   Mieck, K. P.; Reußmann, T.; Nechwatal, A. In Handbook of engineering biopolymers; Fakirov, S.; Bhattacharyya, D., Eds.; Carl Hanser Verlag GmbH & Co. KG: München, **2007**, pp 237-266.

9.   Sreekumar, P. A.; Thomas, S. In Properties and performance of natural-fibre composites; Pickering, K. L., Ed.; Woodhead Publishing: Boca Raton, **2008**, pp 67-126.

10.   Faruk, O.; Bledzki, A. K.; Fink, H.-P.; Sain, M., *Prog. Polym. Sci.* **2012**, 37, 1552-1596.

11.   Huda, M. S.; Drzal, L. T.; Ray, D.; Mohanty, A. K.; Mishra, M. In Properties and performance of natural-fibre composites; Pickering, K. L., Ed.; Woodhead Publishing: Boca Raton, **2008**, pp 221-267.

12.   Rusu, D.; Boyer, S. A. E.; Lacrampe, M. F.; Krawczak, P. In Handbook of bioplastics and biocomposites engineering applications,; Pilla, S., Ed.; Scrivener Publishing LLC.: Beverly, **2011**, pp 397-449.

13. Keledi, G.; Sudár, A.; Burgstaller, C.; Renner, K.; Móczó, J.; Pukánszky, B., *Express Polym. Lett.* **2012**, 6, 224-236.

14. Bismarck, A.; Mishra, S.; Lampke, T. In Natural fibers, biopolymers, and biocomposites; Mohanty, A. K.; Misra, M.; Drzal, L. T., Eds.: Boca Raton, **2005**, pp 37-108.

15. Dányádi, L.; Móczó, J.; Pukánszky, B., *Compos. Part A Appl. Sci. Manuf.* **2010**, 41, 199-206.

16. Rippon, J. A.; Evans, D. J. In Handbook of Natural Fibres; Kozłowski, R. M., Ed.; Woodhead Publishing: Boca Raton, **2012**, pp 63-140.

17. Bledzki, A. K.; Gassan, J., *Prog. Polym. Sci.* **1999**, 24, 221-274.

18. Gassan, J.; Bledzki, A. K., *Compos. Sci. Technol.* **1999**, 59, 1303-1309.

19. Sawpan, M. A.; Pickering, K. L.; Fernyhough, A., *Compos. Part A Appl. Sci. Manuf.* **2011**, 42, 888-895.

20. Mwaikambo, L. Y.; Ansell, M. P., *J. Appl. Polym. Sci.* **2002**, 84, 2222-2234.

21. Mwaikambo, L. Y.; Ansell, M. P., *J. Mater. Sci.* **2006**, 41, 2483-2496.

22. Van de Weyenberg, I.; Chi Truong, T.; Vangrimde, B.; Verpoest, I., *Compos. Part A. Appl. Sci. Manuf.* **2006**, 37, 1368-1376.

23. Taha, I.; Steuernagel, L.; Ziegmann, G., *Compos. Interface.* **2007**, 14, 669-684.

24. Das, M.; Chakraborty, D., *J. Appl. Polym. Sci.* **2006**, 102, 5050-5056.

25. Liu, X. Y.; Dai, G. C., *Express Polym. Lett.* **2007**, 1, 299-307.

26. Gassan, J.; Bledzki, A. K., *J. Appl. Polym. Sci.* **1999**, 71, 623-629.

27. Borysiak, S.; Garbarczyk, J., *Fibr. Text. East. Eur.* **2003**, 11, 104-106.

28. Oushabi, A.; Sair, S.; Oudrhiri Hassani, F.; Abboud, Y.; Tanane, O.; El Bouari, A., *S. Afr. J. Chem. Eng.* **2017**, 23, 116-123.

17

29. Mwaikambo, L. Y.; Ansell, M. P., *J. Mater. Sci.* **2006**, 41, 2497-2508.

30. Gassan, J.; Mildner, I.; Bledzki, A. K., *Mech. Compos. Mater.* **1999**, 35, 435-440.

31. Courchene, C. E.; Peter, G. F.; Litvay, J., *Wood Fiber Sci.* **2006**, 38, 112-120.

32. Gupta, P. K.; Uniyal, V.; Naithani, S., *Carbohydr. Polym.* **2013**, 94, 843-849.

33. Kumar Sinha, A.; Narang, H. K.; Bhattacharya, S., *Mater. Today. Proc.* **2017**, 4, 8993-8996.

34. Mukherjee, A.; Ganguly, P. K.; Sur, D., *J. Text. Ind.* **1993**, 84, 348-353.

35. Vijay, R.; Lenin Singaravelu, D.; Vinod, A.; Sanjay, M. R.; Siengchin, S.; Jawaid, M.; Khan, A.; Parameswaranpillai, J., *Int. J. Biol. Macromol.* **2019**, 125, 99-108.

36. Van Soest, P. J., *J. Assoc. Offic. Agricult. Chem.* **1963**, 46, 829-835.

37. Yamamoto, H.; Okuyama, T.; Yoshida, M., *J. Jap. Wood. Res. Soc.* **1993**, 39, 375-381.

38. Shanmugasundaram, N., .; Rajendran, I.; Ramkumar, T., *Carbohydr. Polym.* **2018**, 195, 566-575.

39. Balaji, A. N.; Nagarajan, K. J., *Carbohydr. Polym.* **2017**, 174, 200-208.

40. Mohanty, A. K.; Khan, M. A.; Hinrichsen, G., *Compos. Part A Appl. Sci. Manuf.* **2000**, 31, 143-150.

41. Bisanda, E. T. N.; Ansell, M. P., *Compos. Sci. Technol.* **1991**, 41, 165-178.

42. Valadez-Gonzalez, A.; Cervantes-Uc, J. M.; Olayo, R.; Herrera-Franco, P. J., *Compos. Part B Eng.* **1999**, 30, 309-320.

43. Barreto, A. C. H.; Esmeraldo, M. A.; Rosa, D. S.; Fechine, P. B. A.; Mazzetto, S. E., *Polym. Compos.* **2010**, 31, 1928-1937.

18

44. Cai, M.; Takagi, H.; Nakagaito, A. N.; Katoh, M.; Ueki, T.; Waterhouse, G. I. N.; Li, Y., *Ind. Crop. Prod.* **2015**, 65, 27-35.

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Figure legends

Fig. 1 Effect of the NaOH concentration of the treating solution on the composition of sugarcane bagasse fibers. Treatment time: 1 h. Symbols: (○) cellulose, (□) lignin content.

Fig. 2 FTIR spectra of neat bagasse fibers and of those treated with NaOH solutions of various concentrations. The inset shows the decrease of hemicellulose (1730 cm-1) and lignin (1514 cm-1) content with increasing alkali concentration.

Fig. 3 Weight loss during the alkali treatment of sugarcane bagasse fibers. Treatment time: 1 h.

Fig. 4 XRD traces of the neat bagasse fiber and of those treated with alkali solutions of various concentration. Changes in the crystalline structure of the fibers.

Fig. 5 Independence of the microfibril angle (MFA) of bagasse fibers of the NaOH concentration of the treating solution.

Fig. 6 SEM micrographs recorded on the neat fibers and on fibers treated with NaOH solutions of various concentration; a) neat fiber, b) 4 wt%, c) 8 wt%, d) 30 wt% NaOH content.

Fig. 7 Effect of the concentration of the NaOH solution used for treatment on the stiffness (a) and tensile strength (b) of sugarcane bagasse fibers. Flax is used as reference. Symbols: (○) bagasse, (□) flax.

Fig. 8 Weak correlation between the mechanical properties of bagasse fibers and crystallinity. Symbols: (○) Young's modulus, (□) tensile strength.

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Fig. 9 Simultaneous effect of cellulose and lignin content on the tensile strength of sugarcane bagasse fibers. The solid lines were determined by regression analysis. The red symbols indicate measured values.

Fig. 10 Correlation between the measured strength of sugarcane bagasse fibers and values calculated by regression analysis from compositional variables using Eq. 1.

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