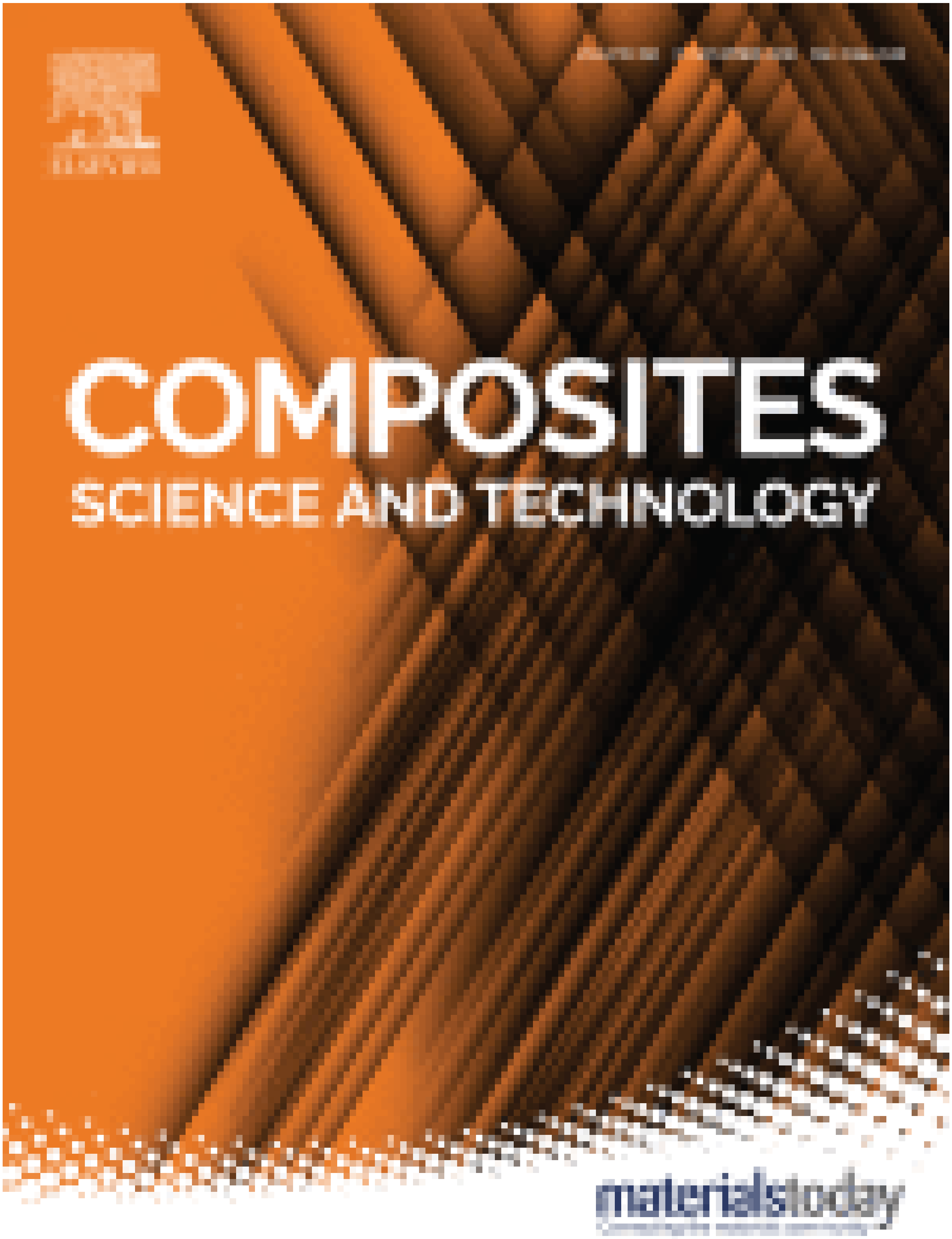
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Composites Science and Technology 200 (2020) 108428

Contents lists available at ScienceDirect

Composites Science and Technology

journal homepage: http://www.elsevier.com/locate/compscitech

Reinforcement of polypropylene with alkali-treated sugarcane bagasse fibers: Mechanism and consequences

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A R T I C L E I N F O

Keywords:
A. Natural fibre composites
B. Impact behavior
B. Mechanical properties
D. Acoustic emission
E. Injection moulding

A B S T R A C T

Polypropylene composites were prepared from neat and alkali-treated sugarcane bagasse fibers. The results showed that alkali treatment leads to an increase in composite stiffness and strength. A maximum is achieved in these properties at around 5 wt% NaOH content of the treating solution. The increase in properties was assigned to the improvement in inherent fiber characteristics. Acoustic emission testing and electron microscopy showed that the two main local deformation processes related to the fibers are their fracture and debonding; the latter is accompanied by the shear yielding of the matrix. Increased inherent strength of the fibers results in an increase in the fracture initiation stress and fracture energy of the composites. Interfacial adhesion has a slight effect on stiffness, but more significant on strength and impact resistance. Changing adhesion modifies the relative importance of local deformation processes, the number of debonding events decreases, while fiber fracture increases with increasing adhesion. Increased interfacial adhesion improves stress transfer and the load bearing capacity of the fibers as well, but suppresses matrix yielding. Alkali treatment increases inherent fiber strength, which can be directly correlated with composite strength.

1. Introduction

Polypropylene (PP) is a commodity polymer with one of the best price/performance ratios among all structural materials. Fiber modification increases its stiffness [1], but often also its strength [2] further. Traditionally, glass [1,2] (GF) and occasionally carbon fibers (CF) [3,4] are used as reinforcing materials, achieving a stiffness as large as 13 GPa at least with carbon fibers [4]. Recently, traditional fibers have often been replaced with natural fibers or wood flour [5–8]. These fibers have various advantages including their natural origin, beneficial effect on carbon footprint, they are light and cheap, and have reasonable stiffness and strength as well [5,6]. On the other hand, natural fibers have several drawbacks like the dependence of properties on the source of the fiber, the year of the harvest and climatic conditions. They are also sensitive to water and heat, have poor adhesion to most polymer matrices especially to polyolefins (PE, PP), and small transverse strength [5,6].

Because of the benefits of natural fibers, many attempts are made to compensate for their weaknesses in one way or another. The diameter of the fibers or the size of wood particles is large and weak adhesion leads to the easy debonding of the matrix and the fiber under the effect of external load leading to small tensile strengths [9]. The easiest way to compensate for weak interfacial adhesion is the surface modification of the fibers [10–13]. Many approaches are used for surface treatment, the simplest is the coating of the fiber with a surfactant, often stearic acid [10,12]. However, this treatment decreases the surface energy of the fiber leading to a further decrease in composite strength. Coupling is often achieved with the use of organosilanes [14], isocyanates [14,15] or other reactive compounds [16]. In polyolefins, efficient coupling can be achieved with the use of functionalized polymers, mainly with maleated PE or PP, which increases interfacial adhesion and tensile strength [10,17–19]. Coupling suppresses debonding to a large extent, but frequently the fracture of the fibers or wood particles becomes the

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https://doi.org/10.1016/j.compscitech.2020.108428
Received 18 June 2020; Received in revised form 17 August 2020; Accepted 24 August 2020
Available online 27 August 2020

dominating failure mechanism as a result [9,20,21].

In the case of good adhesion, the poor transverse strength of fibers or wood particles leads to the failure of the composites, and it is much more difficult to compensate for this weakness. Inherent fiber strength was improved by the impregnation of wood particles with a phenolic resin in one case; a slight improvement in strength was obtained in poly(lactic acid)/wood composites as the result [22]. Very few other examples are found for the increase of fiber strength apart from the alkali treatment of lignonellulosic fibers, which is an approach applied quite often for the improvement of fiber and thus composite strength [23–27]. A wide range of NaOH contents (0.03–40 wt%) and treatment times (2 min–48 h) have been used in this procedure and various extents of improvement was observed in composite properties, mostly in strength [23,28]. The explanations are also diverse and sometimes controversial relating the improvement of properties to changing crystal modification [29], crystallinity [24–26], microfibrillar angle [25,26], surface quality [11,23], etc. Besides being controversial, the explanations are rarely supported with convincing experimental evidence.

In a previous project, we subjected sugarcane bagasse fibers to alkali treatment and determined the composition and structure of the fibers [30]. NaOH content changed from 1 to 40 wt% and treatment time was 1 h. A maximum was found in the stiffness and a more pronounced one in the strength of the fibers and the analysis of the results showed that contrary to the explanations cited above [11,23–26,29], the changes in properties can be related less to the modification of structure than to changing chemical composition. Although fiber strength increased considerably, we could not be certain that this increase is transferred also to the composites. Consequently, the goal of this study was to prepare composites with fibers treated with NaOH and determine their properties. The effect of the concentration of the treating NaOH solution on the properties of composites containing 20 wt% fiber was determined in preliminary experiments and then a more detailed study was carried out to define the influence of fiber content on composite properties. Untreated fibers were used as reference and composites were prepared with and without a coupling agent. Special attention was paid to local deformation processes and the mechanism of failure. The change in the inherent strength of the fibers was estimated by the study of local deformation processes, which has never been done before. Consequences of the results for practice are also briefly mentioned at the end the paper.

2. Experimental

2.1. Materials

A polypropylene (PP) homopolymer, the Tipplen H649 FH (MFR: 2.5 g/10 min, 230 °C, 2.16 kg, density: 0.9 g/cm^3) grade produced by the Mol Group Ltd., Hungary was used as matrix in this study. Sugarcane bagasse fibers were used as reinforcing material, which were obtained directly from the Candi Baru Sugar Factory, Sidoarjo, Indonesia. They were washed with ethanol, dried, cut up and sieved. The average length of the fraction used during the work was 4560 ± 1870 μm and its average diameter was 340 ± 156 μm. A maleic anhydride grafted polypropylene (MAPP) was used as coupling agent to enhance the adhesion between the matrix and the lignocellulosic fibers. The grade used was the Scona TPPP 2112 FA produced by Byk-Chemie GmbH. The MFR of the coupling agent was 3.5 g/10 min (190 °C, 2.16 kg) and its maleic anhydride content was 0.9–1.2 wt%. MAPP was added to the composites at 10 wt% calculated for the amount of the sugarcane bagasse fibers and the same amount was used also in the preliminary experiments (see Section 3.1). The NaOH flakes and the acetic acid solution of 96 wt% concentration used in the alkali treatment procedure were purchased from Molar Chemicals, Hungary.

2.2. Sample preparation

Before treatment, sugarcane bagasse fibers were dried at 105 °C for 24 h and then they were sieved to separate the material to fiber-like and powdery fractions. 300 g of sieved fibers were placed into a container and 5 L sodium hydroxide solution of 5 wt% concentration was poured on them. Another container contained 5 L acetic acid solution of 10 wt% concentration diluted from the concentrated solution, and another container was filled with 10 L of distilled water. The suspension was stirred in every 5–10 min during the 1 h of the treatment. Subsequently the fibers were separated and placed into the acetic acid solution and let them soak for 10 min. The fibers were washed several times with tap water after neutralization and then they were placed into the container containing the distilled water. Fibers were prepared in a similar way for the preliminary study, but with NaOH solutions of different concentrations and in smaller amounts.

Right before extrusion, the fibers were dried again (4 h, 105 °C) to evaporate the water traces absorbed in the lab during storage. A Brabender DSK 42/7 (Brabender, Germany) twin-screw compounder was used for homogenization with the temperature profile of 170-180-185-190 °C and screw speed of 40 min^{-1}. Extrusion was repeated once to improve homogeneity. ISO 527 1A type standard dog bone specimens (thickness 4 mm; width 10 mm) were injection molded from the granules using a Demag IntElect 50/330-100 injection molding machine. The temperature profile was set to 170-180-185-190 °C, the temperature at the hopper was 40 °C and the temperature of the mold was adjusted to 40 °C as well. Injection speed was 50 mm/s and, depending on the composition, the injection pressure was 300–700 bar, while back pressure was 50 bar. Holding pressure was 2/3 of the injection pressure and holding time was set to 25 s. Cooling time was 30 s. The injection-molded specimens were stored at 23 °C and 50% relative humidity for a week before characterization.

2.3. Characterization

Mechanical properties (Young's modulus, yield stress and strain as well as tensile strength and elongation-at-break) were determined using an Instron 5566 universal testing machine. The distance between the grips was 115 mm and the rate of testing was set to 5 mm/min. Acoustic emission (AE) testing was carried out simultaneously with tensile testing in order to follow local deformation processes. The measurements were done by using a Sensophone AED 404 device. The AE signals were recorded with the help of an a11 type resonance detector (resonance frequency 150 kHz). The detector was clipped to the middle of the specimen and silicon grease was used to promote the transfer of ultrasonic vibrations between the specimen and the detector. The threshold level was set to 23 dB in order to filter out noises. A Ceast Resil 5.5 impact tester was used to determine the impact resistance of the samples. The specimens were prepared according to the ISO 179 standard (Charpy impact test with a notch depth of 2 mm). A hammer with 4 J capacity equipped with a piezoelectric sensor was used for instrumented impact testing. A strip of silicone rubber was glued onto the hammer in order to reduce dynamic resonances. The morphology of fracture surfaces (after both tensile and impact testing) was studied by scanning electron microscopy using a Jeol JSM 6380 LA apparatus (Jeol Ltd., Tokyo, Japan). Before recording the micrographs, fracture surfaces were sputtered with gold for 35 s using a Jeol Fine Coater apparatus.

3. Results

The results are presented in several sections. The conclusions of the preliminary experiments are reported first and then the composition dependence of properties are shown in the next section. Deformation and failure mechanisms are discussed subsequently followed by the presentation of general correlations and practical consequences in the last section of the paper.

3.1. Effect of alkali concentration

Our previous study on the effect of alkali treatment on fiber characteristics indicated a maximum in fiber stiffness and strength at around 5 wt% NaOH content of the treating solution [30]. We could not assume a priori that the optimum in composite properties will be reached at the same extent of treatment, at the same alkali concentration. Consequently, the goal of the preliminary study was to identify the extremum in properties, if it exists at all, and its location on the NaOH concentration scale. Fibers were treated with solutions of different alkali concentration and then composites were prepared at 20 wt% fiber content from them. The tensile strength of the composites is plotted against the concentration of the treating solution in Fig. 1. A maximum is detected at around 5 wt% NaOH content indeed. Apparently, the improvement in the stiffness and strength of neat fibers as an effect of alkali treatment is transferred to the composites as well. The extent of increase is smaller than in the case of the fibers, strength increased from around 400 to 600 MPa in the longitudinal direction there, but a maximum clearly exists, the strength of composites containing the treated fibers is definitely larger than that of the materials prepared with the neat fiber.

The increase of composite stiffness and strength as the result of the alkali treatment of natural fibers has been shown before [25,26,30] and it was more or less expected also in the case of bagasse fibers. On the other hand, impact resistance also increased considerably, from around 2.5 kJ/m^2 to a value around 4 kJ/m^2 (Fig. 2), which is rather surprising, since larger stiffness and strength are usually accompanied by smaller impact resistance. We must call the attention here to the fact that the preliminary experiments were done in the presence of a coupling agent resulting in good adhesion [17,31,32], which decreases debonding and usually increases the number of fiber fractures [9,21]. One might assume that the improved impact resistance results from the modification of the inherent strength of the fibers, which hinders the fracture of the fibers and thus fracture initiation and propagation in the composites. However, this tentative explanation had to be checked and it was proved indeed in the further course of the study (see Section 3.3).

3.2. Composition dependence of properties

The effect of fiber treatment on composite properties was studied at various fiber contents with and without the coupling agent. Based on our previous study showing a maximum in fiber modulus and strength [30], which was confirmed by the results of the preliminary study (see Section 3.1), 5 wt% was selected as the concentration of the NaOH solution used for the treatment. The Young's modulus of composites containing the treated and untreated fibers is plotted against fiber content in Fig. 3. Composites containing the alkali treated fibers are clearly stiffer than those prepared with the neat fibers. Adhesion has practically no effect on stiffness in the case of the treated fibers, which is in agreement with previous experience [9,10,20]. On the other hand, coupling resulted in the decrease of stiffness for the neat fibers that is difficult to explain. The premature failure of fibers with large diameter parallel to their axis was one possible explanation, which, however, needed further confirmation

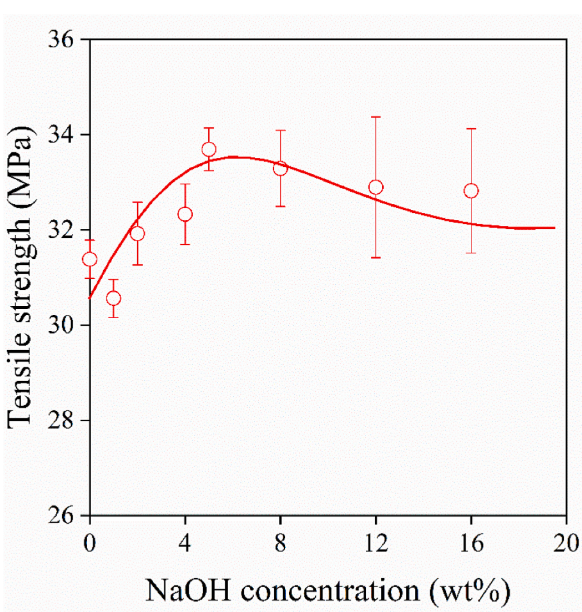

Fig. 1. Correlation between the concentration of the NaOH solutions used for the alkali treatment of sugarcane bagasse fibers and the tensile strength of their PP composites. Treatment time: 1 h, fiber content: 20 wt%.

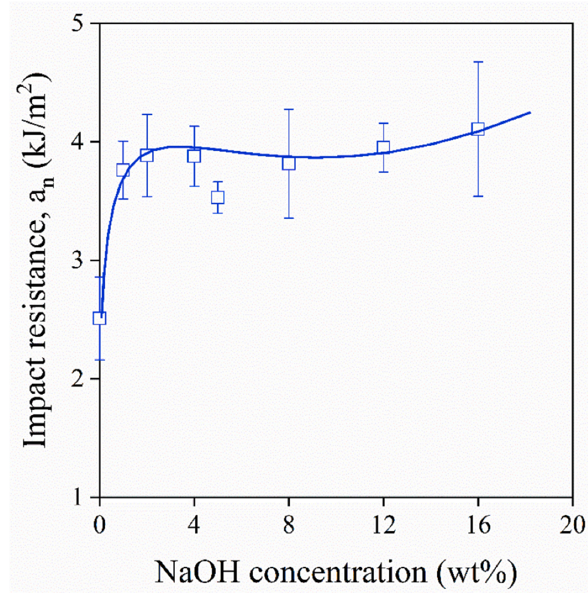

Fig. 2. Effect of the concentration of the treating solution on the impact resistance of PP/sugarcane bagasse fiber composites at 20 wt% fiber content.

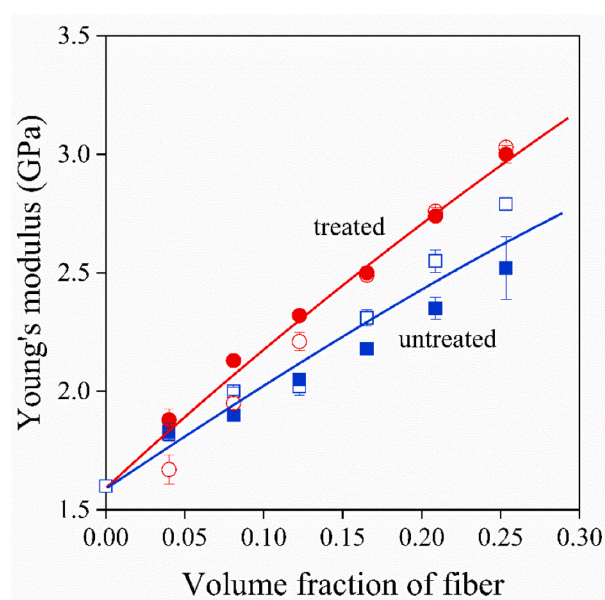

Fig. 3. The stiffness of PP/sugarcane bagasse fiber composites plotted against fiber content. Effect of alkali treatment (5 wt% NaOH) and coupling. Symbols: (□) untreated, (■) untreated, MAPP, (○) alkali treated, (●) alkali treated, MAPP.

by the results of other experiments like acoustic emission testing and microscopy.

Alkali treatment has a similar effect on the tensile strength of the composites as on stiffness; composites containing the treated fibers are stronger than materials prepared with the neat fibers (Fig. 4). The difference is not large, but the increase is unambiguous. As expected, coupling has a much larger influence on strength than on stiffness. Composite strength increases considerably with fiber content in the presence of the coupling agent, i.e. at good adhesion [17,31,32], while it remains practically constant in the absence of the coupling agent. The phenomenon was observed before and was explained with changing local deformation processes occurring around the fibers, i.e. debonding, matrix yielding and the fracture of the fibers [17,20,21].

The preliminary experiments indicated an interesting increase in the impact resistance of composites prepared with the treated fibers. However, composites contained the fibers at a single composition, at 20 wt% fiber content in those experiments. The effect of fiber content on impact resistance is presented in Fig. 5 for the four series of composites studied. Treatment improves impact resistance indeed and the effect depends also on the presence or absence of the coupling agent. The largest impact resistances were measured for the composites containing the treated fibers at poor adhesion, while the smallest for the untreated fiber at good adhesion. The treated fibers with coupling, as well as the untreated fibers without the coupling agent gave more or less similar impact resistance values somewhere between the two extremes. Obviously, the combined effect of fiber treatment and interfacial adhesion determines impact resistance, which changes in a range somewhere between 2 and 4.5 kJ/m^2. The change is influenced by inherent fiber properties and local deformation processes discussed in the next section. We must emphasize here, though, that the impact resistances measured are moderate, and much larger values are required in certain applications.

3.3. Deformation and failure mechanism

Experience shows that in heterogeneous polymeric materials macroscopic properties are determined by local deformation processes taking place during deformation [16,33]. These processes are related to the matrix or to the heterogeneities. Certain processes can be followed by acoustic emission testing, by the measurement of elastic waves

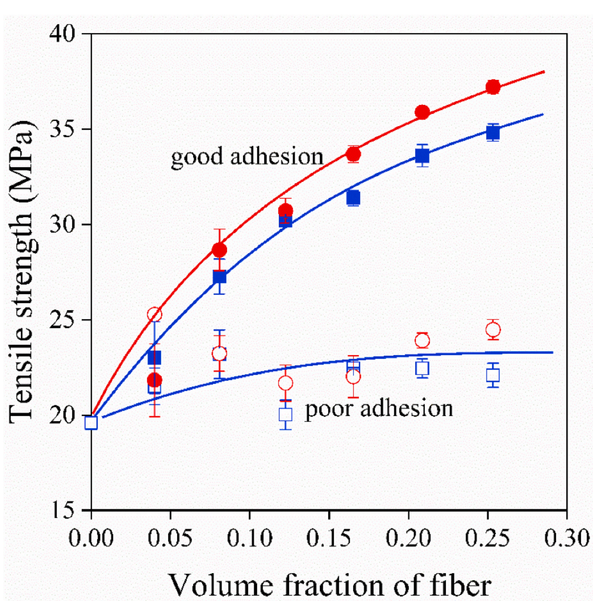

Fig. 4. Correlation between the tensile strength of PP/sugarcane bagasse fiber composites and fiber content. Symbols: (□) untreated, (■) untreated, MAPP, (○) alkali treated, (●) alkali treated, MAPP.

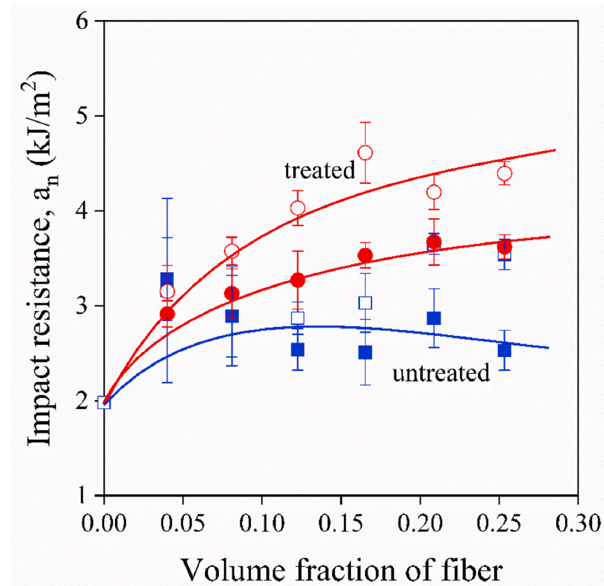

Fig. 5. Impact resistance of PP composites reinforced with sugarcane bagasse fibers plotted as a function of fiber content. Symbols: (□) untreated, (■) untreated, MAPP, (○) alkali treated, (●) alkali treated, MAPP.

resulting from a local process. These waves can be recorded by piezoelectric sensors placed on the specimen during tensile testing. Such processes can be the debonding of the matrix from the fiber, matrix cracking or fiber fracture. The result of such a test is presented in Fig. 6 (left). The measurement was done on the composite containing 20 wt% of the treated fiber in the absence of the coupling agent, i.e. at poor adhesion. The small circles in the figure are individual events or signals indicating the occurrence of at least one local process. The signals can be divided into two main groups. The first group is located at small deformations, below 2%. The amplitude of these signals (vertical position) is small compared to the group detected at larger deformations. The distribution of the signals indicates the occurrence of two local events and according to previous experience [9,20,21] these can be assigned to the debonding of the fibers and to fiber fracture or pullout. The construction of the cumulative number of signals trace (continuous correlation, right axis) also shows the two stages and thus the two processes. The number of events is smaller in the first step (debonding), while more signals are detected in the second process (fiber fracture). The other continuous line (left axis) shows the corresponding stress vs. elongation trace as reference. Adhesion changes the correlations considerably (see Fig. 6(right)). Stronger interfacial adhesion led to the development of larger stresses and also to a larger number of events with slightly larger amplitudes. The first group of events disappeared; only one local process takes place during deformation. Changing interfacial adhesion obviously changed local deformation processes and the modification of these latter led to the changes in properties. Acoustic emission testing yielded very similar results for the untreated fiber, differences can be observed mainly in the distribution, as well as in the number and amplitude of the events recorded.

Acoustic emission testing showed that two local processes, debonding and fiber fracture, take place in the studied composites during deformation, the relative number of which depends on adhesion. However, the unambiguous identification of the two processes is difficult or even impossible based on these experiments. Scanning electron microscopy offers further information and can confirm the occurrence of the processes mentioned. Two typical micrographs are presented in Fig. 7, again for the treated fiber with and without the coupling agent. The debonding of large particles can be seen in Fig. 7(left) confirming that mainly this process takes place in the absence of the coupling agent. At good adhesion, mostly the fracture of the fibers occurs as shown by

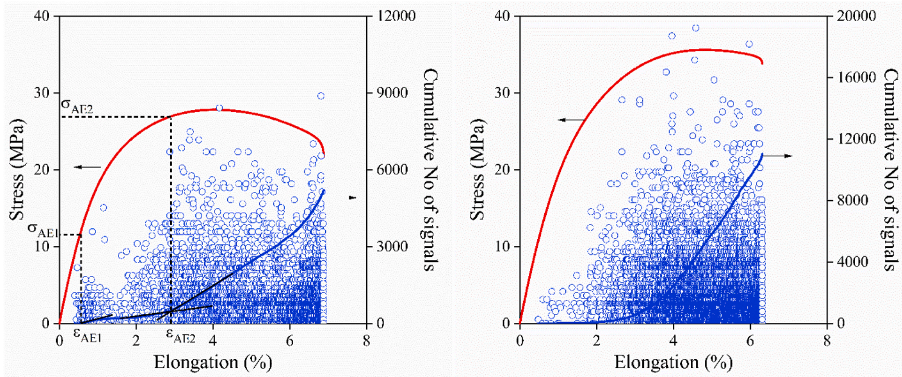

Fig. 6. The result of the acoustic emission testing of PP composites reinforced with alkali treated sugarcane bagasse fiber. Fiber content: 20 wt%. Symbols: (◯) individual acoustic signals, solid lines: cumulative number of signal trace (right axis), stress vs. elongation correlation (left axis); left figure: poor adhesion (no MAPP), right figure: good adhesion (MAPP).

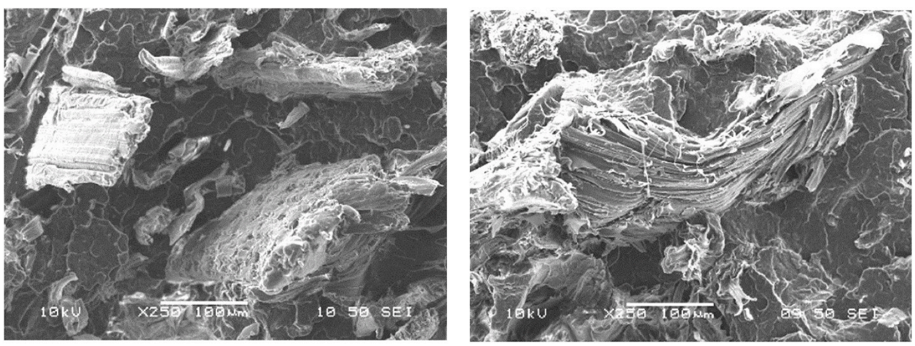

Fig. 7. SEM micrographs recorded on the fracture surface of PP composites reinforced with 20 wt% alkali-treated sugarcane bagasse fiber; left: no MAPP, right: with MAPP.

Fig. 7(right). The aspect ratio of the fibers is small and they even twist during processing thus we conclude that less pullout takes place, but the fracture of the fiber is the dominating process in the presence of the coupling agent.

Acoustic emission testing and the SEM study proved that in the presence of the coupling agent the dominating local process is fiber fracture, which thus determines both the strength and impact resistance of the composites. The results unambiguously prove that treated fibers are stronger and this can be explained only with an increase in the inherent strength of the fibers. Previous experience showed that acoustic emission testing allows the estimation of inherent fiber strength. The extrapolation of the characteristic stress determined from the cumulative number of signal vs. elongation traces in the way indicated in Fig. 6 (left) to volume fraction 1 gives fiber strength [34]. An earlier study showed that fiber strength extrapolates to the same value in different polymer matrices [34] thus validating the procedure. In Fig. 8 characteristic stresses are plotted against the volume fraction of the fiber in the way suggested above. The following exponential function was fitted to the characteristic stresses derived from AE testing

$$\sigma_{AE} = \sigma_{AE0} + a \exp(b\, \phi_f) \tag{1}$$

where σ_{AE} and σ_{AE0} are the characteristic stress determined by acoustic emission testing and its extrapolated value not having any physical meaning, respectively, ϕ_f is the volume fraction of the fiber in the composites, while a and b are fitting constants. The results of the fitting procedure clearly show that treated and untreated fibers extrapolate to different values, to 45.0 and 38.7 MPa, respectively. The determination coefficient indicating the goodness of the fit was 0.9947 and 0.9977, respectively, in the two cases. The difference of about 6 MPa does not seem to be large, but sufficient to bring about the moderate changes

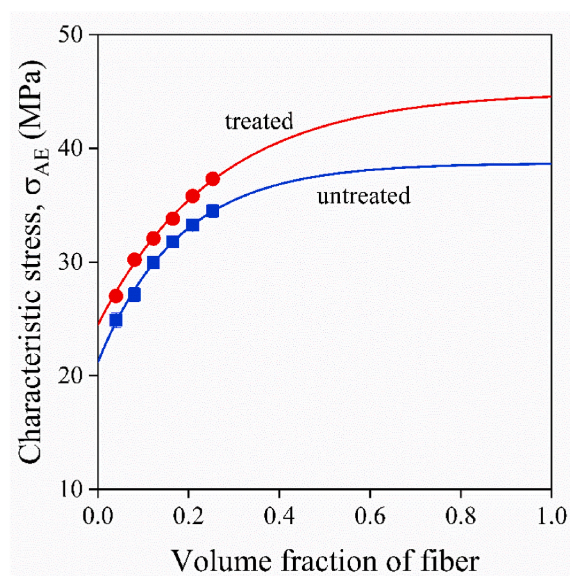

Fig. 8. Estimation of the inherent strength of sugarcane bagasse fibers used for the reinforcement of PP. Characteristic stresses plotted against fiber content. Good adhesion (MAPP). Symbols: (■) untreated, (●) alkali-treated (5 wt%). The solid lines are exponential functions (see Eq. (1)) fitted to the measured data to determine inherent fiber strength.

observed in the strength and impact resistance of the composites as the result of the alkali treatment of the fibers.

3.4. Correlations, consequences

The alkali treatment of sugarcane bagasse fibers resulted in the increase of their stiffness and strength; a maximum was observed in these characteristics as a function of the NaOH concentration of the treating solution. A similar maximum was observed in the stiffness, strength and an increase in the impact resistance of the composites indicating that inherent fiber characteristics influence composite properties strongly and the effect of treatment was transferred from the fibers to the composites. In order to check the relationship, the tensile strength of the composites at good adhesion was plotted against the strength of the fibers in Fig. 9. Although the standard deviation of fiber strengths is quite large, the correlation is unambiguous, composite strength increases with increasing fiber strength.

In practice, often the combination of large stiffness and impact resistance is required for structural materials. The two quantities are plotted against each other in Fig. 10. Increased modulus is accompanied by increased impact strength in three of the series.

The debonding and the fracture of the fibers proved to be the main local deformation processes related to the fibers. Debonding facilitates the shear yielding of the matrix, which consumes considerable amount of energy during fracture. However, increased adhesion hinders the shear yielding of the polymer. The increased inherent strength of the fibers leads to increased fracture resistance in the case of the treated fibers, but the effect was reduced somewhat by good adhesion. Large extent of debonding of the untreated fibers resulted also in reasonable impact resistance, but coupling decreased shear yielding and the smaller strength of the untreated fibers led to decreased fracture strength. Although modulus and impact resistance could be increased simultaneously and the alkali treatment of the fibers was beneficial for composite properties, the impact resistance values obtained are moderate at most. The composites prepared in this study can be used in structural applications in which large stiffness and strength are required and a smaller fracture resistance is acceptable.

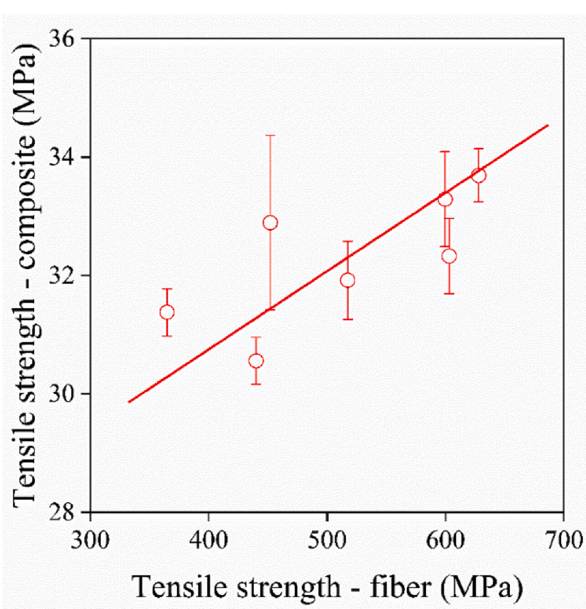

Fig. 9. Correlation between the strength of sugarcane bagasse fibers treated with alkaline solutions of different concentrations and that of the PP composites prepared from them. Fiber content: 20 wt%.

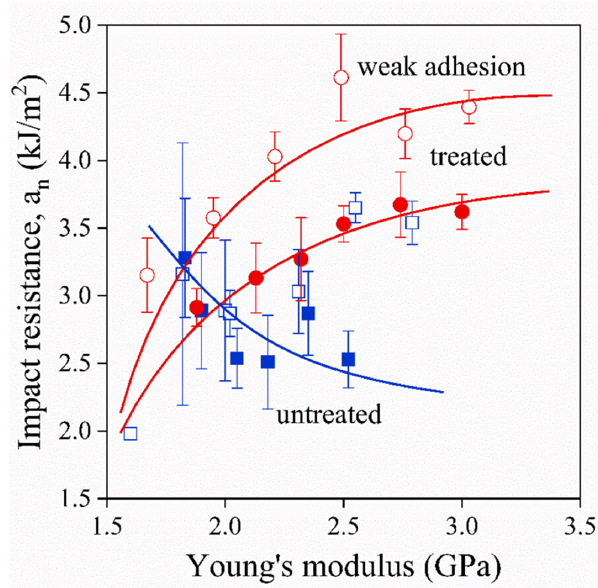

Fig. 10. Correlation between the stiffness and impact resistance of PP composites reinforced with sugarcane bagasse fibers. Effect of alkali treatment and interfacial adhesion. Symbols: (□) untreated, (■) untreated, MAPP, (○) alkali treated, (●) alkali treated, MAPP.

4. Conclusions

The study of PP composites reinforced with sugarcane bagasse fibers treated with NaOH showed that alkali treatment results in increased composite stiffness and strength compared to materials prepared with the untreated fibers. A maximum is achieved in these properties at around 5 wt% NaOH content of the treating solution. The increase is moderate, but clear. The reason for the increase in properties could be identified as the result of an improvement in inherent fiber properties. Acoustic emission testing and electron microscopy showed that the two main local deformation processes related to the fibers are their fracture and debonding, the latter accompanied by the shear yielding of the matrix. The increased inherent strength of the fibers results in an increase in the fracture initiation stress and fracture energy of the composites. Interfacial adhesion has a slight effect on stiffness, but much more significant on strength and impact resistance. Changing adhesion modifies the relative importance of local processes; the number of debonding events decreases, while fiber fracture increases with increasing adhesion. Increased interfacial adhesion improves stress transfer and the load bearing capacity of the fibers, but suppresses matrix yielding. Alkali treatment increased inherent fiber strength, which could be directly correlated with composite strength. Using the sugarcane bagasse fibers results in a simultaneous increase of stiffness and impact strength in PP composites, but the combination of properties, especially impact resistance, is not exceptional. Although alkali treatment is beneficial for composite properties as claimed in the literature, its application needs serious considerations, the slight improvement achieved might not be always worth the effort.

Author contribution

András Bartos: Methodology, Investigation, Data Curation, Writing - Original Draft, Writing - Review & Editing; Visualization; Benny Putra Utomo: Investigation; Barnabás Kanyar: Investigation, Juliana Anggono: Conceptualization, Writing - Review & Editing; Felycia Edi Soetaredjo: Investigation, Resources, János Móczó: Conceptualization, Methodology, Data Curation, Writing - Original Draft; Writing - Review & Editing; Béla Pukánszky: Conceptualization, Methodology, Writing - Original Draft, Supervision.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as they form part of an ongoing study.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

5. Acknowledgements

The authors thank Judit Kócs for her help in the sieving and handling of the fibers. The National Research, Development and Innovation Fund of Hungary (OTKA K 120039 and FK 129270) is greatly acknowledged for the financial support of the research. We are also grateful to the Ministry of Research, Technology and Higher Education of the Republic of Indonesia for the research grant 002/SP2H/LT/K7/KM/2017. The Candi Baru Sugar Factory, Indonesia is acknowledged for providing the sugarcane bagasse fibers.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.compscitech.2020.108428.

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