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ABSTRACT Polypropylene composites were prepared from sugarcane bagasse fibers by extrusion and injection molding. Wood flour was used as reference filler in the study. The fiber content of the composites changed between 0 and 30 wt% in 5 wt% steps. A maleated polypropylene was used as coupling agent to improve interfacial adhesion.

Mechanical properties were characterized by tensile and fracture **testing**, while **local** deformation processes **were followed by acoustic emission and**

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instrumented impact testing as well as by the analysis of scanning electron micrographs. The results showed that sugarcane bagasse fibers reinforce polypropylene similarly to other natural fibers. They increases stiffness, but decreases tensile yield stress, tensile strength and deformability. Increased interfacial adhesion leads to the considerable improvement of reinforcement. Bagasse fiber and wood flour filled composites have very similar properties. The impact resistance of the composites increased in the presence of both fibers compared to the neat matrix. Debonding is the dominating process in the absence of the coupling agent, while mainly fiber fracture occurs in its presence. Increased plastic deformation after debonding results in slightly improved impact resistance. Keywords: natural fiber reinforcement, mechanical properties, fracture resistance, instrumented impact testing, interfacial adhesion, local deformation

processes 1. INTRODUCTION The use of natural resources increases continuously in all areas of our life. The number of possible raw materials produced by nature is enormous and we use only a small fraction presently. The increase of interest in natural materials is driven by the improving environmental awareness of the public, but also by other factors. Natural fibers are extensively used in the automotive and building industry already now and further increase is expected in the future. Natural fibers have many advantages, such as abundant source, low price, but these fibers are also light, thus they decrease the weight of the vehicles. Decreasing weight results in decreased fuel consumption and smaller emission. The fibers used in the largest quantity are flax, hemp and kenaf, but a number of other fibers like bamboo, ramie, sisal, coir and wood flour are also applied for various purposes such as inner door panel, under body cover, seat backs, ceiling liner, etc. Natural fibers have numerous advantages, but also a few drawbacks like sensitivity to moisture, small transverse strength, variability in properties, etc. The properties of heterogeneous polymers including fiber-reinforced composites are determined by four factors, by component properties, composition, structure and interfacial interactions. In the case of wood flour and other natural fibers, the main factor proved to be the size and aspect ratio of the particles [x]. Larger aspect ratio improves stress transfer and reinforcement, but it makes processing more difficult. The surface energy of natural fibers is small, thus their adhesion to the polymer matrix is weak, especially in polyolefins, which do not contain any polar functional groups. Interfacial interactions may be stronger if the polymer contains groups capable of forming specific interactions, e.g. hydrogen bonds, like in poly(lactic acid) (PLA), for example [x]. In the case of large particles, the transverse strength, i.e. the strength of the fiber vertically to its axis, proved to be small, which led to small tensile strength and impact resistance [x]. Various fibers coming from a variety of sources can have a wide range of characteristics and thus the properties of the composites prepared from them can vary widely [x]. One of the advantages of natural fibers is that they are cheap. Moreover, they can be obtained from local resources very often as waste, which further decreases their price. Using local resources is extremely important in developing societies in which the value added application of cheap natural resources can improve economy considerably. Indonesia is an agricultural country producing considerable amounts of coconut, rubber, coffee, tea, tobacco, but also sugar. The fibers for the sugarcane bagasse form a waste, which is burnt to produce energy for the production of the sugar. With the intensification and modernization of the processes, this energy source becomes insufficient and the bagasse becomes a waste, which must be handled in a different, but environmentally friendly and economically advantageous way. A possible approach for the value added application of fibers from sugarcane bagasse is their use as reinforcing fibers in polymer composites. Accordingly, the goal of the present project was to explore the possibility of using sugarcane bagasse fibers as reinforcement for polypropylene. We prepared composites with various fiber contents in order to determine the effect of composition on composite properties. PP/wood composites were also prepared to use them as reference. In the study, we focused on the analysis of deformation processes and the mechanism of failure in order to find ways to improve properties if necessary. The potentials of the fibers for practical applications are also considered briefly in the final part of the paper. 2. EXPERIMENTAL 2.1. Materials The

polypropylene used as matrix for the composites was the Tipolen H 649
grade homopolymer produced by the MOL Group Ltd., Hungary. The
polymer **has** a nominal density **of 0.9 g/**

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cm³ and a melt flow rate of 2.5 g/10 min at 230 °C and 2.16 kg load. The bagasse fibers were obtained directly from the sugar mill. They were cleaned and dried, cut up and sieved. Two fractions were obtained a

fibrous and a powdery. The fibrous fraction was used in the present experiments. The average particle size of the fibers was determined from scanning electron micrographs. The average length of the fibers was 4560 μm , while their average diameter was 340 μm . The wood flour used as reference was the Filtracell EFC 1000 grade produced by Rettenmaier and Sohne GmbH, Germany. The average size of the particles was about 210 μm and its aspect ratio, i.e. the ratio of its length and diameter, was 6.8. A polypropylene functionalized with maleic anhydride was used as coupling agent. The Scona 2112 grade maleated PP (MAPP) was supplied by Byk-Chemie GmbH, Germany, and it had a melt flow rate of 4-8 at 190 °C and 2.16 kg, and a maleic anhydride content of 0.9-1.2 %. The fiber content of the composites changed from 0 to 30 wt% in 5 wt% steps. The ratio of MAPP/fiber was 0.1 in all composites.

2.2. Sample preparation The fiber and the polymer were homogenized in a twin- screw compounder (Brabender DSK 42/7, Brabender, Germany) at set temperatures of 170-180-185-190 °C and 40 rpm. Extrusion was repeated twice in order to increase homogeneity. The granulated composites were injection molded into standard (ISO 527 1A) tensile bars of 4 mm thickness using a Demag IntElect 50/330-100 machine. Processing parameters were 40-180-180- 185-190 °C set temperatures, 300-700 bar injection pressure, 50 bar back pressure, 50 mm/s injection speed, 25 s holding time, and 30 s cooling time. The specimens were stored at ambient temperature (25 °C, 50 % RH) for a week before further testing.

3.3. Characterization, measurements The

mechanical properties were characterized by tensile and impact **testing.**

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Tensile tests **were**

carried out using an Instron 5566 universal testing machine with a gauge length of 115 mm and 5 mm/min crosshead speed. Modulus, yield properties (yield stress and yield strain), tensile strength and elongation-at-break were derived from recorded stress vs. strain traces. Local deformation processes were followed by acoustic emission testing.

Acoustic emission (AE) signals were recorded with a Sensophone AED 40/4 apparatus. A single A11 resonance detector with the resonance frequency of 150 kHz was attached to the center of the specimen. Pre-amplification was 20 dB and the peak amplitude detected by the equipment was 108 dB μV . Reference voltage was 1 mV. The threshold level of detection was set to 20 dB. Impact resistance **was** characterized **by the**

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notched Charpy impact strength, which was determined according to the ISO 179 standard at 23 °C with 2 mm notch depth. Instrumented impact testing was carried out using a Ceast Resil 5.5 instrument (CEAST spa, Pianezza, Italy) with a 4 J hammer. The particle characteristics of the fibers as well as the appearance of broken surfaces were studied by scanning electron microscopy (Jeol JSM 6380 LA apparatus, Jeol Ltd., Tokyo, Japan).

Micrographs were recorded both **on fracture surfaces created during tensile** and fracture **testing,**

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respectively, or on specimens cooled down to liquid nitrogen temperature and broken. 3. RESULTS AND DISCUSSION Fiber reinforced composites are used as structural materials. Their most important properties are stiffness, strength and impact resistance. Overall mechanical properties of composites are determined by local deformation processes. Accordingly, we discuss tensile properties and reinforcement in the first two sections of the paper. Subsequently we focus on fracture behavior and the analysis of local processes in the next two. Finally, structure-property correlations

are discussed in the last section of the paper with some reference to practical consequences. 3.1. Tensile properties The main

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role of reinforcing fibers is to increase the stiffness and the strength of the polymer used as matrix. The Young's modulus of polypropylene reinforced with the two types of fibers is plotted against fiber content in Fig. 1. Previous experience indicates that stiffness does not depend on fiber characteristics and adhesion very much, but mainly on the amount of fiber added to the matrix. The presence of MAPP does not influence stiffness indeed, but fiber characteristics seem to have an effect; the modulus of composites containing the bagasse fibers is smaller than that prepared with wood flour. Only one plausible explanation exist for this phenomenon, the very large bagasse fibers debond from the matrix already at the very small deformations of the modulus determination, voids form, which decrease modulus. We must also note that the gradient of the modulus vs. fiber content correlations decrease with increasing fiber loadings. This effect indicates the presence of some structural factor, either the fibers touch each other forming associations [x] or their average orientation decreases with increasing fiber content. Such a change in orientation with filler content was observed earlier [Belina, Jani]. Tensile yield stress is plotted against fiber content in Fig. 2. The difference in the reinforcing effect of the two fibers, bagasse and wood, remained the same, but the influence of interfacial adhesion is much larger than in the case of stiffness. Several studies proved earlier that tensile characteristics determined at larger deformations depend much more on interfacial adhesion than stiffness [x]. It is interesting to note, that the effect of different particle characteristics does not appear in yield stress at poor adhesion, possible because of the large size of the two fibers. Although wood is much smaller than the bagasse fiber, but 210 μm is till much larger than the usual size of mineral fillers, which is in the micron range. The structural effect mentioned above affects properties also in this case and the effect can be seen in the composition dependence of yield stress. We much emphasize, however, that both wood and bagasse reinforce PP at good adhesion. The tensile strength of the composites is presented as a function of fiber content in Fig. 3. Quite surprisingly, the differences in particle characteristics is even less visible than in the case of tensile yield stress. On the other hand, the effect of adhesion on reinforcement is significant in this case; strength almost doubles in the composition range studied. One of the major drawback of wood and natural fiber reinforced composites is their limited deformability, which result in very rigid materials with small impact resistance. As shown by Fig. 4, the elongation-at-break values of the composites decrease considerably with increasing fiber content to very small values. Hardly any difference can be made among the composites in this respect, composites containing wood might have slightly larger deformability, and adhesion seems to decreases elongation, but the differences are very small. The results presented above clearly show that the fibers applied in this study can be used in practice only when a coupling agent is added to improve adhesion and that the deformability of the composites is small. 3.2. Reinforcement A general, qualitative idea can be obtained about the reinforcing effect of the two fibers from Figs. 2 and 3 presented above. However, it is very difficult to draw conclusions from those figures about reinforcement and it is impossible to estimate it quantitatively. A model is needed in order to determine the

reinforcing effect of fibers more accurately. Such a model was developed earlier for the description of the tensile and impact characteristics of heterogeneous materials [x]. Eq. 1 describes the composition dependence of tensile strength in the following way $\sigma = \sigma_0 (1 - V_f + 1.5 \exp(B V_f)) (1 + \epsilon/\epsilon_0)^n$ where σ and σ_0 are the true tensile strength of the composite and the matrix, respectively, V_f is the volume fraction of the fiber in the composite, B expresses the load bearing capacity of the fiber and it depends on interfacial adhesion. In the equation true tensile strength ($\sigma = \sigma_0 (1 + \epsilon/\epsilon_0)^n$, $\epsilon = L/L_0$, relative elongation) accounts for the change in specimen cross-section and n for strain hardening. n characterizes the strain hardening tendency of the polymer and can be determined from matrix properties. The rearrangement of the equation leads to reduced tensile strength $\ln(\sigma/\sigma_0) + 2.5 \ln(1 + \epsilon/\epsilon_0) = \ln(1 - V_f + 1.5 \exp(B V_f))$ and if we plot its natural logarithm against composition,

we should obtain straight lines, the slope of which expresses the reinforcing effect of the

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fibers quantitatively. The tensile strength of the four set of composites is plotted in the way suggested by Eq. 2 in Fig. 5. Straight lines are obtained indeed with smaller or larger deviations from the line. Particle characteristics do not seem to influence reinforcement much, the points for the two fillers fall on the same lines, but interfacial adhesion does. The addition of the coupling agent increased the slope of the lines, i.e. reinforcing effect considerably. The parameters of the correlations are collected in Table 1. The conclusions drawn from the direct observation of Fig. 5 are expressed 12 quantitatively by the numbers in the table. Very small differences can be seen in the slope of the lines for the composites prepared with the different fibers, but much larger as an effect of changing adhesion. Fig. 5 calls attention to another fact. The model applied assumes the homogeneous distribution of the heterogeneous phase in a matrix. If structural phenomena occur (changing orientation, aggregation) in the composites, the points deviate from the straight line. Such deviation can be seen in composites with good adhesion, i.e. which contain MAPP, thus confirming our earlier assumption that the development of a special structure (fiber association) or the change of some structural factor (orientation) influences properties at large fiber contents.

3.3. Fracture resistance

Impact strength is often a very important property for materials used in structural applications. Because of the dynamic conditions of testing, its composition dependence often differs considerably from that of tensile properties. The impact resistance of the composites is plotted against fiber content in Fig. 6. The standard deviation of the measurement is quite larger, but some conclusion can be drawn from the data quite unambiguously. Somewhat surprisingly, the impact strength of all composites is larger than that of the matrix. One of the major problems of wood reinforced composites is their limited impact strength, which cannot be improved in any way [x]. The only reasonable explanation for the phenomena is that local deformation processes occurring around the fibers during fracture absorb some energy, which increases impact strength. The other observation, which can be made, is that impact resistance increases slightly in the absence of the coupling agent, i.e. in the case of poor adhesion, while it decreases when the coupling agent is present. Obviously, the deformation process resulting in the absorption of energy is hindered by the presence of the coupling agent, by strong adhesion. It must be emphasized here, though, that although the fibers increased fracture resistance compared to the matrix, the absolute value of impact strength is still small, the largest values are around 3.5 kJ/m². At least 15 kJ/m² impact strength is expected from structural materials used in the automotive industry. Instrumented impact testing was carried out on the composites in the hope to obtain more information about the fracture process. The force vs. time traces of selected composites are presented in Fig. 7. All specimens fail by brittle fracture. The largest difference can be seen in the maximum force, which is related to fracture initiation stress. All composites seem to have similar maximum loads and the addition of MAPP decreases it somewhat. It is very difficult to tell anything about the

area under the traces which is related to fracture energy by the simple visual observation of the fractograms, but a more detailed analysis indicated that it changes similarly to the impact resistance values presented in Fig. 6. Accordingly, the fibers improved the impact resistance of polypropylene both by increasing fracture initiation stress and fracture energy due to a local process or processes, which consume surplus energy during fracture, compared to the neat polymer.

3.4. Local processes, deformation mechanism

The previous sections clearly proved that local processes taking place around the reinforcing particles play a role in the determination of impact resistance and generally properties. The study of wood reinforced composites showed that several local processes can take place during deformation, the shear yielding of the matrix, its cavitation, debonding, i.e. the separation of the interface between the filler or fiber and the matrix, fiber pull-out, or fiber fracture [x]. These processes are competitive and they can take place simultaneously or consecutively. Usually one of the processes dominates during deformation and fracture, but the occurrence of the others cannot be excluded either. Some of the local processes generate elastic waves within the material, which can be detected by piezoelectric sensors thus acoustic emission testing gives information about the local processes. The result of such an acoustic emission measurement is presented in Fig. 8. Small circles indicate individual events occurring within the material. The stress vs. strain trace of the composite is also plotted for reference (left axis). The number of signals is not very large and they can be divided into two groups. Events start to appear above a certain deformation and signals stop to develop at larger elongations; further signals appear again later until the specimen breaks. The evaluation of individual signals is difficult, but their evolution, the cumulative number of signals offers more information about the deformation process. The

cumulative number of signals is also plotted in

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Fig. 8 (right hand axis). The correlation clearly shows the occurrence of two processes during the testing of this composite reinforced with 20 wt% bagasse fibers not containing the coupling agent. Based on previous experience [x] we can assume that these two processes are debonding and the pullout or fracture of the fibers. The addition of the coupling agent, i.e. MAPP

changes both the number of signals and the shape of the cumulative number of signal trace (Fig.

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9). Initiation starts at larger deformation than in the previous case and the number of signals is ten times larger. The cumulative number of signal trace increases rather steeply and only one process can be distinguished in this case. The changing shape of the trace indicates a different deformation mechanism, which earlier was identified as the fracture of the fibers [x]. In previous sections, we saw that the use of the coupling agent had a profound effect on the properties of the composites, and the results presented here indicate that the changes are caused by the modification of local processes. Further analysis of local processes is possible, if we determine the characteristic stress values at which the local processes are initiated. The procedure is demonstrated in Fig. 8. In the first case, two characteristic values can be determined (σ_{AE1} , σ_{AE2}), while only one in the second. The composition dependence of the characteristic values is presented in Fig. 10. The first process is initiated at very small stresses and it does not depend on composition. The process must be the debonding of large particles from the matrix. The second process is more difficult to identify, it can be either the pullout or the fracture of the fibers. In the case of strong

adhesion, the only process occurring is initiated at larger stress. The different mechanism is shown also by the dissimilar composition dependence. Earlier this process was identified as the fracture of the fibers [x]. Although we may have some ideas about the local processes emitting the signals, we need further proof for their unambiguous identification. Further evidence might be supplied by SEM micrographs. Typical micrographs recorded on fracture surfaces generated in the tensile test are presented in Fig. 11. All the composites contained 20 wt% fibers. Fig. 11a recorded on a composite reinforced with bagasse shows the debonding and pullout of a fiber from PP. Some debonding can be still seen in the presence of MAPP (Fig. 11b), but also the fracture of some of the fibers occur. Practically the same picture is conveyed by the micrographs recorded on composites containing the wood fibers. Debonding of large particles is shown in Fig. 11c, but the micrograph offers further information. It shows that particles touch each other, which may explain the deviation from linearity in Fig. 5, in spite the fact that larger deviation were observed in the case of composites containing also MAPP. Moreover, strong plastic deformation is seen around debonded particles, which may account for the increased impact resistance of the composites (see Fig. 6), especially in the case of poor adhesion. A large broken particle can be seen in Fig. 11d confirming our assumption that fiber fracture occurs in the case of good adhesion and that it is the dominating deformation process in composites containing MAPP.

3.5. Discussion

The results presented in the previous sections answered a number of questions emerged in the study. The analysis of local processes confirmed our tentative assumptions about the presence of structural effects, thus offering a reasonable explanation for the phenomenon observed during the evaluation of reinforcement (see Section 3.2). It explained also the larger impact resistance of the composites compared to the matrix by showing that after the debonding of large particles considerable plastic deformation might take place, which consumes energy. It also explained the larger reinforcing effect of the fibers in the presence of the coupling agent. At poor adhesion, the debonding of the particles may result in slightly improved impact resistance, but only fibers adhering strongly to the matrix carry considerable load. Earlier we claimed that local deformation processes are important in the determination of composite properties. The tensile strength of the composites is plotted against the initiation stress of the various local processes in Fig. 12. According to the correlation, the initiation of the local process leads to immediate failure in the case of strong adhesion, i.e. the local process determines strength. Although not immediately, but failure occurs soon after the start of the second process also in the case of poor adhesion. On the other hand, debonding does not necessarily leads to failure; considerable plastic deformation takes place after its initiation. The analysis and especially the control of local processes offer a possibility to improve the properties of the composites. In the case of structural materials, especially those used in the automotive industry, an important question is the balance of stiffness and impact resistance. Fracture resistance is plotted against stiffness in Fig. 13. The matrix has a small value in the lower left corner of the plot. All composites have larger stiffness, but quite surprisingly also larger impact resistance. Increasing adhesion leads to decreasing impact strength, but larger moduli are accompanied by larger impact strength in the case of poor adhesion. We tried to explain the observed changes of properties with the occurrence of various local process, but the unusual correlations need further study and explanation. Nevertheless, the applied combination of particle characteristics and adhesion offers some advantages for practical applications. Nevertheless, it is clear that in spite of the somewhat larger impact resistance compared to the matrix, only composites with proper adhesion can be used in practice.

4. CONCLUSIONS

Sugarcane bagasse fibers reinforce polypropylene similarly to other natural fibers. They increase stiffness, but decrease tensile yield stress, tensile strength and deformability. Increasing interfacial adhesion achieved with the application of a coupling agent results in the considerable improvement of reinforcement. Composite strength increases with increasing fiber content, but deformability decreases even further. Although the very large bagasse fibers debond more easily from polypropylene than the smaller wood fibers used as reference, most properties are very similar in the two types of composites. The analysis of the

composition dependence of strength indicated the presence of some structural effect, either the aggregation of particles or the change of orientation with increasing fiber content. The impact resistance of the composites increased in the presence of both fibers compared to the neat matrix. The analysis of local deformation processes indicated

that debonding is the dominating process in the absence of the

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coupling agent, while mainly fiber factor occurs in its presence. Increased plastic deformation after debonding results in slightly improved impact resistance. Although bagasse fibers can be used for the reinforcement of PP, fiber extraction and handling must be improved to obtain smaller fibers with a better size distribution. A coupling agent must be used in all cases to obtain reasonable properties. 5.

ACKNOWLEDGEMENTS The National Research Fund of Hungary (OTKA K 120039) is greatly acknowledged for the financial support of the research. Indonesian grant. 6. REFERENCES Table 1

Reinforcing effect (parameter B) of the studied fibers calculated from the tensile strength of the composites (see Eqs. 1 and 2) at good and poor adhesion Fiber Matrix strength, σ_0 (MPa) Parameter B R2a Bagasse 15.079 4.8600 0.98205 Wood 14.560 5.0037 0.99744 Bagasse, MAPP 18.392 5.9637 0.99814 Wood, MAPP 16.853 7.2978 0.99943 a) Determination coefficient indicating the goodness of the fit. CAPTIONS

Fig. 1 Composition dependence of the stiffness of PP/natural fiber composites prepared with bagass fibers and wood flour as reference. Symbols: (\circ), bagasse, (?) bagasse, MAPP, (?) wood flour, (?) wood flour, MAPP. Fig. 2 Effect of fiber content on the tensile yield stress of PP/fiber composites. Influence of interfacial adhesion. Symbols are the same as in Fig. 1. Fig. 3 Tensile strength of PP/lignocellulosic fiber composites plotted against fiber content. Symbols are the same as in Fig. 1. Fig. 4 Decreasing elongation-at-break values of PP/natural fiber composites with increasing fiber content. Symbols are the same as in Fig. 1. Fig. 5 Tensile strength of PP composites reinforced with natural fibers plotted against fiber content in the linear representation of Eq. 2. Slopes express the load bearing capacity of the fibers. Symbols are the same as in Fig. 1. Fig. 6 Effect of fiber content on the Charpy notched impact strength of PP/natural fiber composites. Symbols are the same as in Fig. 1. Fig. 7 Instrumented impact traces (force vs. time) recorded on selected PP/natural fiber composites. Fiber content: 20 wt%. Symbols are the same as in Fig. 1. Fig. 8 Fig. 9 Fig. 10 Fig. 11 Fig. 12 Fig. 13 Results of the acoustic emission testing of a PP/bagasse composite. 20 wt% fiber, poor adhesion (no MAPP). Symbols: (\circ) individual signals, ??? cumulative number of signals (right axis) and stress vs. elongation trace (left axis). Increased number of signals during the acoustic emission testing of a PP/bagasse composite containing 20 wt% fiber and MAPP. Good adhesion. Symbols are the same as in Fig. 8. Dependence of characteristic stresses (σ_{AE1} and σ_{AE2}) on fiber content. Different local processes and mechanism. Symbols are the same as in Fig. 1. SEM micrographs recorded on the fracture surface of selected PP/natural fiber composites. The surfaces were created in tensile testing. Fiber content is 20 wt%. a) bagasse, b) bagasse MAPP, c) wood, d) wood, MAPP. Correlation between the tensile strength of the composites and the characteristic stress determined by acoustic emission testing. Symbols are the same as in Fig. 1. Correlation between the impact resistance and stiffness of PP/natural fiber composites. Symbols are the same as in Fig. 1. 2 3 4 5 6 7 8 9 10 11 13 14 15 16 17 18 19 20 21 22 23 24 25