BUDAPEST UNIVERSITY OF TECHNOLOGY AND ECONOMICS

Laboratory of Plastics and Rubber Technology

Budapest, 05. August, 2020

Dear Editor,

Please find enclosed the manuscript **"Biobased PLA/sugarcane bagasse fiber composites: effect of particle characteristics and interfacial adhesion on properties"** by András Bartos, Kristóf Nagy, Juliana Anggono, Antoni, Hariyati Purwaningsih, János Móczó and Béla Pukánszky for your consideration and to be published in the Composites Part A: Applied Science and Manufacturing. The corresponding author of the manuscript is András Bartos. The paper deals with biodegradable PLA composites reinforced with a natural fiber, sugarcane bagasse. The effect of particle characteristics and a coupling agent (maleic-anhydride grafted PLA) on the mechanical properties of the composites are discussed in the paper.

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 also in the plastics industry. Many products are prepared with natural reinforcement. Such composites compete successfully with traditional glass or carbon fiber reinforced commodity polymers and they are used more and more extensively in automotive and building industry. The ever-increasing drive of the industry towards materials with better properties always allows room for further improvement.

In our study we determined the particle characteristics of the reinforcement before and after processing. The results showed that considerable attrition took place during melt processing in both the length and diameter of the fibers. The particles are fiber like with small aspect ratio and the originally different batches of short and long fibers have similar particle characteristics after processing. Regarding the interfacial interactions between the components, our findings contradict the results of other research groups, which claim poor adhesion between PLA and natural fibers. Based on the analysis of local deformation processes and SEM micrographs we concluded that the adhesion between the components is acceptable due to the formation of hydrogen bonds. Further evidence to support our view is that coupling, i.e. the use of maleated PLA does not have practically any effect properties. Local deformation processes occurring during the deformation of the material depend on the particle characteristics and the orientation rather than on interfacial interactions. The modification of PLA with sugarcane bagasse fibers results in considerably increased stiffness, almost constant tensile strength and slightly increased impact resistance, i.e. a material with reasonable combination of properties for structural applications.

Hereby we also confirm that the manuscript or its contents in some other form, has not been published previously by any of the authors, it is not under consideration for publication in another journal at the time of submission, and all the authors have seen and approved the submission of the manuscript.

I thank you for your care and patience in advance, and send you my best regards.

Yours sincerely
András Bartos

Bartos András

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Küldve: szombat 2020. december 12 14:11
Címzett: András Bartos
Tárgy: Your Submission

Ms. Ref. No.: JCOMA-20-1847
Title: Biobased PLA/sugarcane bagasse fiber composites: Effect of particle characteristics and interfacial adhesion on properties Composites Part A

Dear Mr. András Bartos,

The referees have now completed reviewing your paper and their comments are given below. The paper is recommended to be accepted only after mandatory revisions. Before we can proceed, please let me have a point-by-point response to the issues raised in the reviews together with a revised version of the paper. A final decision will be made after receiving the revised version and your response.

Please clearly indicate where changes have been made, for example by using a different colour. If you are using MS Word, you can highlight changes using the "Track Changes" feature. In 'Tools', choose 'Track Changes', 'Highlight Changes' and tick 'Track changes while editing'.

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With many thanks,

Kristiina Oksman, Ph.D.
Biocomposites editor
Composites Part A

****

Editor:

I have seen your paper and I think there are things to improve before it can be acceptable.

Experimental
Materials: Add information of used materials, supplier name, city and country, see that you are doing that for all used materials and chemicals, including bagasse, both PLA polymers and maleic anhydride, add if any other chemical are used. I also suggest to add a short description and not just a ref. for the preparation of the MA-PLA.
Processing: add supplier name, city and country for all equipment's used. Explain how did you control the fiber content? Not clear for me is this is a batch process or continuos extrusion? I think you need to improve the processing part.
Characterization: same here be sure that you have all information on equipments.
Optical micropcopy is missing, explain how did you characterize the fiber size and length, and how many measurements made and how did you made the samples, removed the matrix?
The mechanical testing add information of the testing, load cell, standard used and how many samples were tested.
For me it is not clear in the description of acoustic emission was used to measure the strain and wondering if that was used in the calculation of the E-modulus or how did you measure the strain?
Can you be clearer on descriptions? Also electron microscopy study, add information there, including if the samples were coated and voltage used.
Impact properties, add number of samples tested. Actually this information is missing in all tests made.

Results
Here you are using particle characterization, are they particles or fibers use only one term.
The fiber length before and after, the standard deviation is very large, is there differences? I suggest that you make annova statistical analysis on these and also show how these looks in your optical microscopy study. Did you use image analysis, how did you measure the length and diameter, this is not easy but you are not discussing that. How did you remove the matrix to be able to make length measurements?
Longer fibers are very difficult to be fed into the extrusion, that need to be discussed, how did you made that.

Mechanical properties, how did you calculate Vol% bagasse, explain that in experimental section.

Regarding the strength, you are writing "Strength does not change much with composition that is beneficial for the application of these composites as structural material."
not need to be improved? It is also not clear how much compatibilizer is added.
The addition don't improve the strength indicating poor adhesion, and that the MAPLA you have made do not show any effect. I think you have to show if you have managed to graft the MA into the PLA.

I don't know if I can agree that impact strength is improved with increased fiber content. You have very large deviation here, wondering how many samples were tested. You might need to increase the number of samples to see clearer results.
Wondering why you have different color for Figure 1 etc in the text? Remove this are write the whole name Figure 1. Etc.

Regarding SEM images, I don't understand what you mean with "considerable fiber fracture takes place is confirmed by the SEM micrographs, but it is further confirmed by Fig. 7. " for me it looks more as a common fracture surface without any adhesion with the matrix- How do you think that fibers are fractured? You might need to study this in another way?

Wondering also how were these surfaces created?

I also suggest that if you submit the figures one by one as you have done now add the caption in the description field or that you are actually adding the figures in the same file as the text either in the text or after with figures captions included for each figure. It is very difficult to see what is was as there are no colors in the figure text.

Write the author contribution following Credit.

Reviewer #1: Review of the article: Biobased PLA/sugarcane bagasse fiber composites: effect of particle characteristics and interfacial adhesion on properties.

Regarding the complete text: The text is very well written and coherent. However, only standard tests were carried out and hardly any new results were obtained.

Table 1: How many fibers were examined for the optical measurements of fiber lengths or fiber diameters?
Mechanical properties: For the sake of completeness, the elongations at break of the different volume fractions would also have to be indicated in a diagram. Furthermore, the y-axes of the graphics (mechanical properties) are scaled too roughly.
Figure 6: In the SEM images it should be clearly marked which locations are described and which locations are used to infer debonding, fracture and shear yielding.

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Answer

to the comments of the Editor and the Referee on the manuscript "Biobased PLA/sugarcane bagasses fiber composites: Effect of fiber characteristics and interfacial adhesion on properties" by Bartos, A., Nagy, K., Anggono, J., Antoni, Purwaningsih, H., Móczó, J. and Pukánszky, B. submitted to Composites Part A (JCOMA-20-1847)

We appreciate the comments of the Editor and the Referee as well as their suggestions to improve the quality of the paper. We took practically all of them into account during revision. The modifications carried out and the answers to the questions are listed below. Questions are printed by normal letters, while answers are written in italic. Modifications are indicated by color-coding in the revised manuscript.

Editorial comments

We are grateful for the Editor for taking the time and reading our manuscript very thoroughly. We also apologize that especially the format was rather sloppy. We considered all questions and suggestions very thoroughly and did our best to improve the manuscript in every department.

1. Experimental. Materials: Add information of used materials, supplier name, city and country, see that you are doing that for all used materials and chemicals, including bagasse, both PLA polymers and maleic anhydride, add if any other chemical are used.

   The Editor is completely right, we were not sufficiently thorough and did not include all this information or only a part of it. We corrected the deficiency and supplied the necessary information for all materials and chemicals, including the solvents used.
2. I also suggest to add a short description and not just a ref. for the preparation of the MA-PLA.

   Besides the literature reference the text was extended with a short description of the preparation process for MAPLA. However, the reader must look up the reference still for the complete description of chemical analysis proving that the grafting was successful and PLA was modified with 2.5 MA unit/polymer chain.
3. Processing: add supplier name, city and country for all equipment's used. Explain how did you control the fiber content? Not clear for me is this is a batch process or continuous extrusion? I think you need to improve the processing part.

   Supplier name, city and country were added for all equipment used. Additional information was supplied about the control of fiber content. In fact the fibers and the granules were mixed in a plastic bag and fed into the hopper. No segregation was observed, the homogeneity was sufficient. The confusion about continuous or batch process must have been caused by the word "compounder", but this is the official name of the equipment used. The compounder is a twin-screw extruder having short screws with large diameter. There are cuts in the flights that increase the mixing volume and the efficiency of homogenization. Shear stresses are not very large, but homogenization efficiency is acceptable. Accordingly, homogenization was a continuous process.
4. Characterization: same here be sure that you have all information on equipments. Optical microscopy is missing, explain how did you characterize the fiber size and length, and how many measurements made and how did you made the samples, removed the matrix?

All necessary information is supplied throughout the entire experimental section. Several sentences are included to describe the type and origin of the digital optical microscope used and the procedure of determining fiber dimensions. In order to characterize fiber dimensions after processing, PLA was dissolved with THF from the composites and then the fibers were filtered and dried. 6-700 fibers were measured to obtain the distribution of both their length and diameter.

5. The mechanical testing add information of the testing, load cell, standard used and how many samples were tested.

   The requested information was included into the revised text. A 10 kN load cell was used, the measurements were done according to the ISO 527 1A standard and tensile characteristics were determined in five parallel measurements.

6. For me it is not clear in the description of acoustic emission was used to measure the strain and wondering if that was used in the calculation of the E-modulus or how did you measure the strain?

   Acoustic emission does not allow the determination of strain, but the sensor picks up elastic waves emitted by local, burst like processes like debonding or fiber fracture. Strain was measured by a strain gauge and modulus was calculated from the measured force and strain. The experimental part was amended to include the measurement of strain and the calculation of the modulus. The acoustic emission technique is described in some detail in Section 3.3 focusing on deformation and failure processes.

7. Can you be clearer on descriptions? Also electron microscopy study, add information there, including if the samples were coated and voltage used.

   We did our best to improve and extend descriptions to make them clearer. Details on electron microscopy are given in the revised text. The specimens were sputtered with gold for 35 s and the micrographs were recorded at 15 kV accelerating voltage.

8. Impact properties, add number of samples tested. Actually this information is missing in all tests made.

   Seven specimens were tested for all composites. The information is included into the experimental section and everywhere else where necessary.

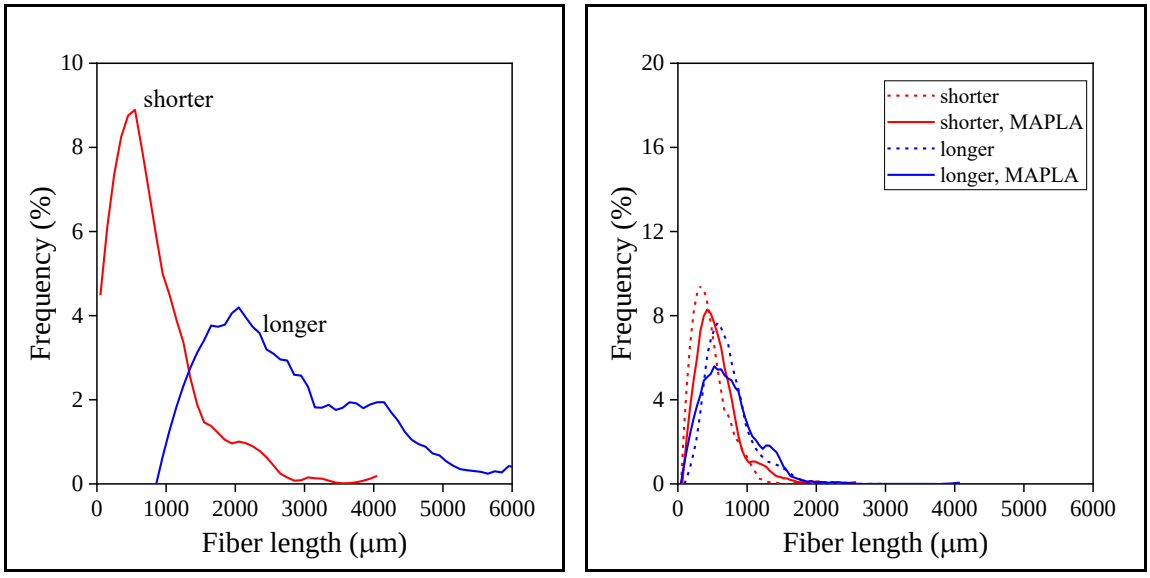
9. Results. Here you are using particle characterization, are they particles or fibers use only one term.

   The word "particles" was changed to fibers everywhere throughout the text. Only one term is used now.

10. The fiber length before and after, the standard deviation is very large, is there differences? I suggest that you make annova statistical analysis on these and also show how these looks in your optical microscopy study. Did you use image analysis, how did you measure the length and diameter, this is not easy but you are not discussing that. How did you remove the matrix to be able to make length measurements?

    In this case the standard deviations show the width of the size distribution. These are natural fibers produced by grinding and sieving and they have a wide size distribution. The length distribution of the fibers before and after processing are shown in Figure A1. The differences, but also the similarities are clear and Anova statistical analysis would not change that. The two batches of fibers are clearly different before processing and they are very similar after, but we acknowledge and state these facts in the text at several places. The difference in the length of the fibers is clearly shown also by the photos of Figure A2. We measured the fibers manually using the ImagePro 10 software and then determined the distribution of length and diameter. You are right, the task is not easy, but it can be made, if you have a good student. We did not include the photos, the micrographs and the distributions, because of the length constraint of the journal and we though that Table 1 is sufficient to show the differences and the similarities, as mentioned above. The matrix was

dissolved from the composites by THF as mentioned in Answer 4 above and the procedure is described in the experimental part in the revised manuscript.



a) b)

Figure A1 *Distribution of the length of the bagasse fibers before and after extrusions; a) neat fibers, b) fiber length distribution after processing.*

a) b)

Figure A2 *Appearance of the two batches of fibers before processing; a) long, b) short.*

11. Longer fibers are very difficult to be fed into the extrusion that need to be discussed, how did you made that.

The Editor is generally right, long fibers are usually difficult to feed into the extruder. However, we did not have any problems in this case, unlike with flax or sugar palm fibers. A possible reason is the stiffness of the fibers that made feeding relatively easy, but led to the large extent of fiber attrition discussed in the paper. We did not think that feeding needs a more detailed explanation and discussion and did not modify the text on this account.

12. Mechanical properties, how did you calculate Vol% bagasse, explain that in experimental section.

    Volume fractions were calculated from weight fractions using the density of the components. This is explained in the experimental section in the revised paper.

13. Regarding the strength, you are writing ”Strength does not change much with composition that is beneficial for the application of these composites as structural material.” not need to be improved? It is also not clear how much compatibilizer is added.

    The Editor is completely right, it is much better if strength improves. However, composite strength depends on the load bearing capacity of the components and composition. If adhesion is poor, like in PP/lignocellulosic fiber composites, strength decreases with increasing fiber content. In PLA, interfacial adhesion is inherently good because the OH groups of the fibers form hydrogen bonds with the polymer. Strong interaction is counterbalanced by the large size of the particles which facilitates debonding. The net effect in our case is practically constant with composition, which is beneficial compared to the large decrease of strength in the case of polyolefin composites. MAPLA content was always 10 % calculated for the amount of the reinforcement used and MAPLA was added on the expense of the PLA matrix. The information is included in the revised paper now.

14. The addition don’t improve the strength indicating poor adhesion, and that the MAPLA you have made do not show any effect. I think you have to show if you have managed to graft the MA into the PLA.

    The Editor is right again that the addition of MAPLA does not improve strength. On the other hand, we do not agree with her statement that the lack of effect indicates poor adhesion. On the contrary, we think that it indicates strong adhesion. According to a large number of literature references the adhesion between PLA and lignocellulosic fibers is poor. However, in a previous study [A1] we proved that this is not true and the adhesion between these two components is strong, as mentioned also in Answer 13. The reason for the lack of effect is that MAPLA is effective only in the case of large particles which debond from the matrix under the effect of external load. The coupling agent prevents debonding thus slightly increasing strength [A2], but this effect is weak since most of the fibers do not debond, but fracture during deformation. We did check the occurrence and efficiency of grafting in a previous study, proved that it takes place, and we characterized the product in detail [AE2]. Accordingly, the reason for the lack of increased reinforcing effect is not poor, but strong adhesion of PLA and the bagasse fibers.

15. I don’t know if I can agree that impact strength is improved with increased fiber content. You have very large deviation here, wondering how many samples were tested. You might need to increase the number of samples to see clearer results.

    Because of the remark of the Editor, we repeated the measurements with a hammer of smaller energy, which led to more accurate measurements and much smaller standard deviation. This latter was always smaller than 10 % that is acceptable in impact testing. Seven specimens were tested for each composition. The results obtained were exactly the same at larger fiber contents as before and deviated only slightly at 5 and 10 wt% fiber loading (Figure A3). However, the fact remained that the impact resistance of the composites is practically always larger than that of the neat polymer. We included the new results into the revised paper to show results with smaller standard deviation.

16. Wondering why you have different color for Figure 1 etc in the text? Remove this are write the whole name Figure 1. Etc.

    The color was removed and the full name of the figures was written throughout the entire paper.

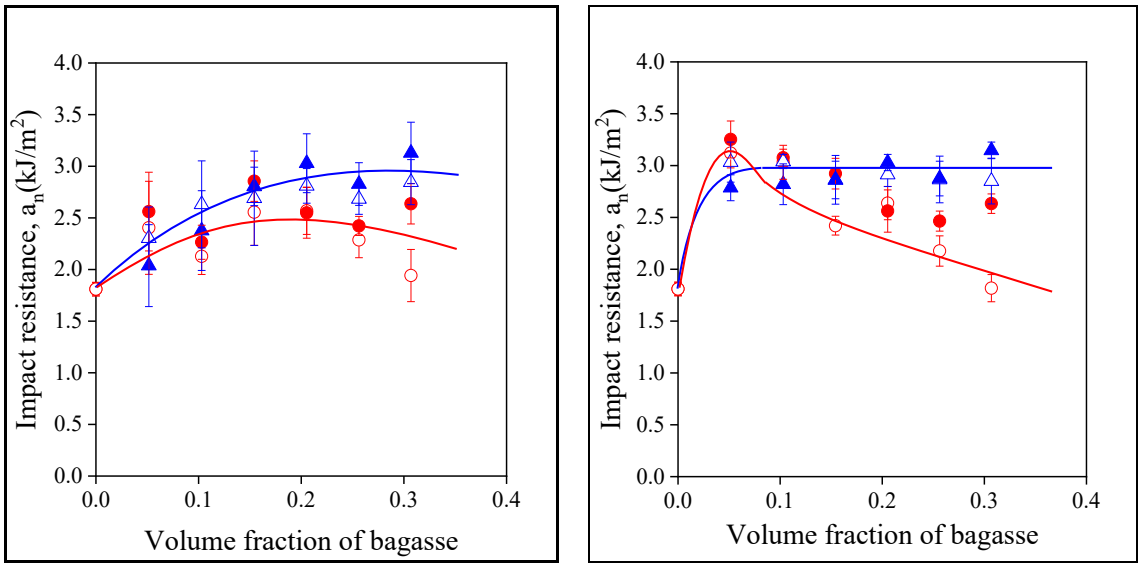


Figure A3 *Results of the impact testing of PLA/bagasse fiber composites; a) original results, b) repeated measurements*

17. Regarding SEM images, I don't understand what you mean with "considerable fiber fracture takes place is confirmed by the SEM micrographs, but it is further confirmed by Fig. 7. " for me it looks more as a common fracture surface without any adhesion with the matrix- How do you think that fibers are fractured? You might need to study this in another way?

    We do not agree completely with the Editor on the interpretation of the SEM micrographs, but admit that it is difficult and often subjective. That is why we never rely solely on the viewing of micrographs when we make our statements. Our conclusion about fiber fracture is based on at least three facts. The first is naturally the micrograph itself. One might wonder if large fibers in the micrographs debonded or fractured. The latter process is clearly indicated by the rough fracture surface and the lack of voids at the edges of the fibers broken. Voids are clearly visible around debonded fibers. Fracture proceeds along the axis of the fibers. The second evidence is supplied by acoustic emission (Figures 4 and 5). Two stages can be clearly distinguished in Figure 4 and the corresponding characteristic values differ considerably as Figure 5 shows. According to our experience the process with the smaller characteristic stress is debonding, while that with the larger values is fiber fracture. The third proof is supplied by Fig. 7. In the case of weak adhesion characteristic stresses decrease with increasing fiber content, at least in PP/wood composites [A3] (see Figure A4). One could say that the same is valid for the PLA/bagasse fiber composites. However, in this case the strength of PLA is larger than the inherent strength of the fibers in the transverse direction, i.e. along their longitudinal axis. The fact that characteristic stresses extrapolate to the same value, to the inherent strength of the fibers, in two matrix polymers, PP and PLA, clearly proves that fiber fracture occurs and it is the dominating local process in these composites. Micrographs were changed in the manuscript to show local processes even better and these latter are indicated by arrows in the revised paper.

18. Wondering also how were these surfaces created?

    The surfaces were created in tensile testing, the broken halves of the tensile specimens were studied at 20 wt% fiber content. The information is included into the experimental section now.

19. I also suggest that if you submit the figures one by one as you have done now add the

caption in the description field or that you are actually adding the figures in the same file as the text either in the text or after with figures captions included for each figure. It is very difficult to see what is was as there are no colors in the figure text.

The Editor is completely right about the captions, we will pay more attention to this issue in the future. We thank her for the remark.

20. Write the author contribution following Credit.

We uploaded credit as well as the declaration of competing interest as separate files according to the instructions on the website of the journal. Now we included them into the revised manuscript as well.

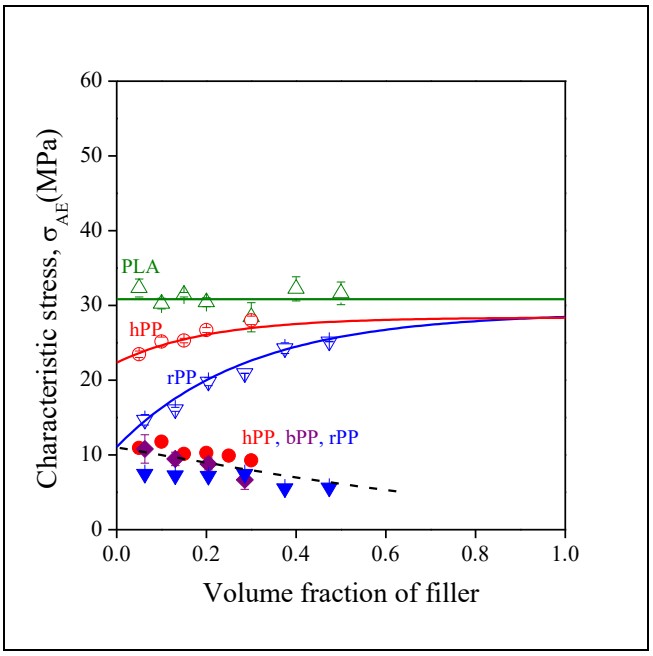


Figure A4 *Composition dependence of characteristic stress determined in polymer/lignocellulosic fiber composites. Determination of the inherent strength of the fibers.*

Reviewer #1

We are very glad that this Reviewer found that "Regarding the complete text: The text is very well written and coherent". He or she had a few questions or suggestions most of which we took into account during revision. The questions and the answers given to them are listed below, the latter in italics.

1. However, only standard tests were carried out and hardly any new results were obtained.

The Reviewer is partly right that mostly standard test were carried out. However, we can hardly regard acoustic emission standard, only a very few groups use the technique. Moreover, not the test, but evaluation, interpretation and conclusions give novelty and not the test. New results and information can be obtained even with standard tests. We do not think that the Referee can find many papers which give detailed analysis of local deformation processes and on their effect on macroscopic composite properties. Similarly, the effect of coupling and interfacial adhesion is difficult to assess and many contradictory

statement are published in the literature about it. We hope that we could shed more light onto these questions in our paper and could offer valuable additional knowledge for the reader about them.

2. Table 1: How many fibers were examined for the optical measurements of fiber lengths or fiber diameters?

   The dimensions of a large number, 6-700, of fibers were measured during the analysis. The information is given in the experimental part now.

3. Mechanical properties: For the sake of completeness, the elongations at break of the different volume fractions would also have to be indicated in a diagram. Furthermore, the y-axes of the graphics (mechanical properties) are scaled too roughly.

   We did not include the elongation-at-break values because they do not offer additional information compared to that written in the text (Figure A5) and because of the length constraint of the journal. We can add it as supplementary information if the Reviewer and/or the Editor wish. We changed the scales of graphics showing mechanical properties.

4. Figure 6: In the SEM images it should be clearly marked which locations are described and which locations are used to infer debonding, fracture and shear yielding.

   The SEM micrographs were changed to show local processes more clearly, these latter are indicated by arrows and they are also labelled according to the corresponding mechanism.

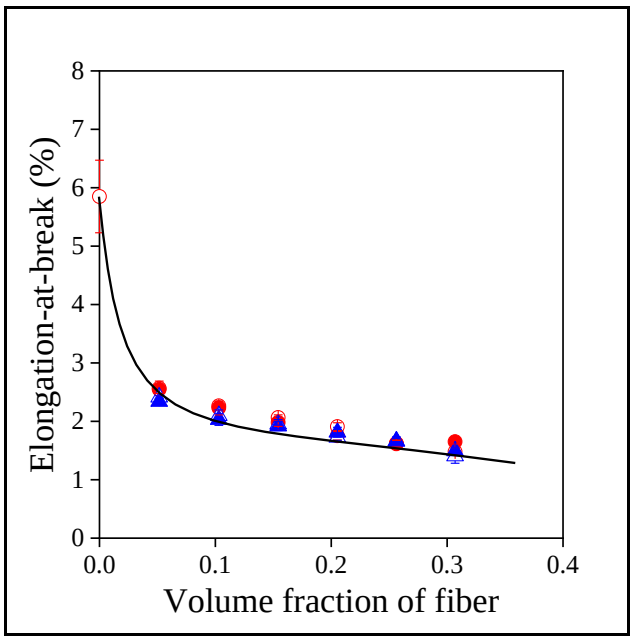


Figure A5 *Composition dependence of the deformability of PLA/bagasse composites. Lack of difference among the materials.*

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A1. Faludi, G., Dora, G., Balázs, I., Renner, K., Móczó, J., Pukánszky, B.: PLA/lignocellulosic fiber composites: Particle characteristics, interfacial adhesion and failure mechanism, *J. Appl. Polym. Sci.* **131**(4), Art. No. 39902 (2014)

A2. Csikós, Á., Faludi, G., Domján, A., Renner, K., Móczó, J., Pukánszky, B.: Modification of interfacial adhesion with a functionalized polymer in PLA/wood composites, *Eur. Polym. J.* **68**, 592-600 (2015)

A3. Faludi, G., Link, Z., Renner, K., Móczó, J., Pukánszky, B.: Factors determining the performance of thermoplastic polymer/wood composites; the limiting role of fiber fracture, *Mater. Design* 61, 203-210 (2014)

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