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Research article

Mechanical and thermal properties of corn cob and lavender stem reinforced poly(lactic-acid)-based composites

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Abstract. Biocomposites have recently received more attention because of raising environmental awareness and the drive toward sustainable technologies. The most common biodegradable polymer is poly(lactic acid) (PLA), which has an excellent balance of physical and rheological properties, but there is some limit to its usage. PLA properties can be improved by adding different types of fibers or fillers that come from agricultural waste. In this study, corn cob and lavender stem were used to reinforce PLA without any coupling agent, and the properties of the composites were investigated. The melt flow rate (MFR) values decreased with the corn cob content and increased with the addition of lavender stem. Mechanical tests showed that the tensile and flexural modulus of the composites increased and the strengths decreased with the reinforcement material content. The rigidness of PLA slightly decreased with the addition of fillers. There was no significant effect on the thermal properties. The unremarkable improvement of the reinforcement was due to the lack of appropriate adhesion of the two phases. The structure of the compounds was found to be homogenous on the scanning electron microscopy (SEM) micrographs. The incorporation of corn cob and lavender stem can reduce the production cost of materials.

Keywords: *natural fillers, polylactic acid, injection molding, mechanical properties, thermal properties, rheology*

1. Introduction

Biodegradable polymers have raised increasing interest over the past two decades in the industry. These polymers have been considered as potential replacements for fossil-based polymers. Numerous efforts have been made to reduce the environmental impact of plastic including the development of biopolymers. Among the biodegradable polymers poly (lactic acid) (PLA) has recently gained an increasing interest because of its relatively cheap cost. In addition, PLA is the easiest to process using traditional plastic technologies such as extrusion, injection molding, *etc* [1]. PLA is a biodegradable and biobased aliphatic polyester made from natural sources such as potatoes, corn, sugar beet, sugar cane, and other plants. It is a semi-crystalline polymer with a glass transition temperature of around 58 °C and a melting point between 160–180 °C [2]. PLA has been used for applications such as filaments, textiles, service ware, packaging containers, environmental remediation films, medical implants, surgical sutures, and medical devices because of its biocompatibility. Although PLA possesses good mechanical and rheological properties and is relatively easy to process, it also has some limitations, for example, low toughness, low elongation at break, slow crystallization rate, brittleness, and moisture sensitivity [3].

Many additives, for example, plasticizers, pigments, and nucleating agents have been combined with PLA to improve the inadequate properties and optimize the material for specific applications [4, 5]. Synthetic fibers such as carbon or glass, are also commonly used to reinforce PLA to improve its properties. Several studies have shown the effect of glass [6, 7]

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and carbon fiber [8, 9], which was a promising route to prepare high-performance PLA.

However, the sustainability of the PLA polymer is reduced by the synthetic additives, because these materials may be non-renewable, non-biodegradable, or produced by the pollution-emitting process, so research efforts are centered on obtaining PLA products with particular desired properties by compounding PLA with biodegradable, natural fillers or fibers [10]. In addition, there are several other reasons why natural fibers or fillers are used in composites by the industry or research. Natural fibers and fillers have the benefit of often being non-toxic, biodegradable, easily available, and cheap. The end product's price is also significantly reduced by these reinforcements. Several studies have shown that different types of natural fillers can be added to poly (lactic acid). Cellulose is one of the most commonly used fibers [11–18].

Huda *et al.* [19] investigated the properties of PLA/recycled cellulose composites. Ejaz *et al*. [20] prepared jute/flax-reinforced PLA composites. Nishino *et al.* [21] prepared PLA/kenaf composites with good mechanical properties, thanks to the orientation imparted to the fibers. Agaliotis *et al.* [22] have studied henequen-reinforced PLA composites. Further studies prepared bamboo and kenaf fiber hybrid PLA composites [23] and other fillers, such as wood fibers [24] and hemp [25], have also been investigated.

The incorporation of the fibers with the polymer matrix highly increases the complexity of the design process. The performance and properties of the composites are strongly linked to the properties and content of the constituent materials, the size and shape of the filler or fiber, the particle size distribution, and the adhesion between the discontinuous and continuous phases. The interfacial compatibility of the filler/fiber is the most important factor in obtaining maximum benefits. In the literature, various surface modification methods were incorporated to improve the adhesion. It is also common to use a coupling agent to enhance the interfacial adhesion of the additive to the matrix polymer.

Olonisakin *et al.* [26] investigated the impact of epoxidized soya bean oil as a compatibilizer on the PLA/PLA/polybutylene adipate terephthalate (PBAT) bamboo fiber composites. Chun and Husseinsyah [27] used a new organic coupling agent called coconut oil for corn/PLA composite.

Natural fillers and fibers have another important benefit: The amount of organic waste can be reduced by using them. Gamiz-Conde *et al*. [28] used untreated and low-added-value coffee by-products to reinforce PLA. Corn cob waste is considered to be one of the biggest environmental problems in Egypt. Grinding corn cobs is the first step to solving this problem, to be used as raw materials in several industries [29]. A potential way to use it as a filler in biocomposites. Another agricultural waste is the lavender stem after harvesting. The flower and the leaf of the lavender are used in numerous areas, for example, the cosmetic industry, food industry, *etc*. However, the stem is considered agricultural waste.

This study aims to investigate the thermal and mechanical properties of PLA/corn cob and lavender stem composite prepared by extrusion and injection molding. For the extrusion, different ratios of corn cob, lavender stem, and pure PLA were used in 2.5/97.5, 5/95, 7.5/92.5, and 10/90 w/w% ratios in order to investigate the change in the properties.

2. Experiments

2.1. Materials

To investigate the effect of natural additives on the properties of PLA, IngeoTM Biopolymer PLA (Grade 3025 D) was sourced from NatureWorks Ltd., USA. This material has a density of 1.24 g/cm^3, a tensile strength of 62 MPa, and a deformation at break of 3.5%. The melt flow rate (MFR) is 14 g/10 min under conditions of 210 °C and a 2.16 kg load. As natural additives granulated corn cob and lavender stem were used. The granulated corn cob (CC) (type Feeds 30/100) originated from Cobex Hungária Ltd., Hungary. Feed products are produced from the inner spongy part and the parts on the sides. These products are less dense and milder, with very high hygroscopic attributes. Feeds 30/100 has a density of 200–350 kg/m^3 and has a particle size of 180 µm. The lavender stem (LS) was used in granulated form, originating from a local producer of lavender in Budapest, Hungary.

2.2. Material preparation and processing

The PLA and the reinforcement materials were dried in a drying cabinet (Taisite FCO-230L, China) at 100 °C for at least 10 h. Immediately after drying, both CC and LS were compounded with PLA via extrusion using a Labtech Scientific LTE26-48/15/01

Table 1. Parameters during the extrusion.

Processing parameters		Values for PLA
Barrel temperature	[°C]	175–185
Speed of screw	[rpm]	155
Melt pressure	[bar]	51

Table 2. Parameters during the injection molding.

Processing parameters		Values for PLA
Dosing volume	[cm^3]	84
Decompression	[cm^3]	10
Injection speed	[cm^3/s]	100–60 (2 phase)
Injection pressure	[bar]	1200
Holding pressure	[bar]	1000
Holding pressure time	[s]	16
Back pressure	[bar]	80
Mold temperature	[°C]	25

(Labtech Engineering Co. Ltd, Thailand) twin screw extruder. Both additives were added in 4 different weight ratios: 2.5, 5, 7.5, and 10 w/w%. During the increasing reinforcement ratio, the extrusion parameters stayed the same, as shown in Table 1.

From the granulates standard samples were produced via injection molding using an Arburg 570 S hydraulic injection molding machine (Arburg GmbH, Germany). The mold, equipped with 6 cavities, is designed for producing MSZ EN ISO 527 'Type 1A' test specimens. During the process, most of the injection molding parameters were constant as shown in Table 2.

However, due to the changing of the reinforcement content some of the parameters needed to be altered to manufacture correct specimens. These were barrel temperatures, which were different in the case of CC and LS compounds. In addition, the cooling time was increased from 45 to 60 s.

2.3. Methods

To evaluate the mechanical properties of the produced compounds, a variety of mechanical tests were conducted. Tensile tests, performed according to the MSZ EN ISO 527-1 standard, were carried out using Instron 8850 and Instron 8874 (Instron, USA) mechanical testing machines, each equipped with 5 kN load cells and operated at a crosshead speed of 1 mm/min. Both tensile and bending tests were performed to determine the tensile and flexural modulus and strength. The Charpy impact strength was assessed using a Ceast Resil Impactor Junior (Instron, USA) with a 2 J hammer.

The thermal properties and crystallinity of the samples were analyzed using a DSC 250 differential scanning calorimeter (TA Instruments, USA). Before measurement, the test specimens were dried at 40 °C for a minimum of 12 h. The specimens underwent an initial heating from 20 to 200 °C, followed by cooling back to 20 °C, and a subsequent reheating to 200 °C, all at a rate of 10 °C/min. A tempering period of 1 min was employed between the cooling and heating phases. All measurements were conducted under a dynamic nitrogen atmosphere.

The rheological behavior of the materials was characterized through melt flow rate (MFR) measurements using an Instron MF20 (Instron, USA) machine. The tests were conducted in accordance with the MSZ ISO 1133 standard, at a temperature of 210 °C and under a load of 2.16 kg.

Scanning electron microscopy (SEM) pictures of granulates and the surface of the fracture were taken with the Tescan Vega SEM device (Tescan Group, Czech Republic) to investigate the interfacial adhesion and distribution of CC and LS.

The heat deflection temperature (HDT) was determined using a Ceast HV3 6911.000 (Instron, USA) machine. The measurements were conducted under a stress of 0.45 MPa and a heating rate of 120 °C/h.

3. Results and discussion

3.1. Morphology

Compounds with corn cob

The SEM photograph of the granulates of the PLA/CC compound is shown in Figure 1. In the pictures of compounds containing 10 w/w% CC, the

Figure 1. SEM micrograph of compounds containing 10 w/w% CC.

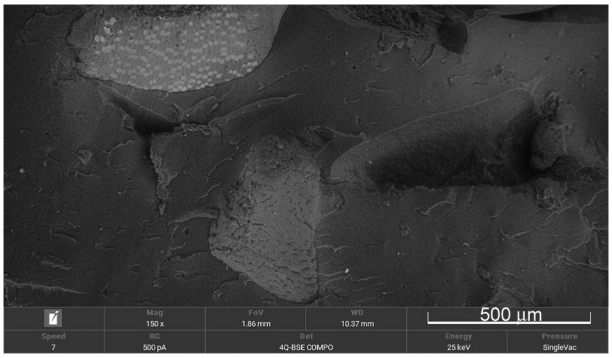

Figure 2. SEM micrographs of fractured surface of compounds containing 5 w/w% CC.

distribution of CC is homogeneous without any agglomerates. This reinforcement has a spherical geometry, which can be useful to improve the inadequate thermal properties of the PLA and also reduce the price of the final product. In addition, the properties of the composites depend on the size and the particle size distribution of the filler. Granulated corn cob has a particle size of 180 µm according to the manufacturer. In the pictures, a larger size of particles can be seen. However, it also shows a uniform particle size distribution.

The SEM photograph of a PLA/CC test specimen, which was broken via the Charpy method is shown in Figure 2. In the pictures of compounds containing 5 w/w% CC, it was also observed that the CC filler comes out during the Charpy breaking because of poor interfacial adhesion.

Compounds with lavender stem

The SEM photograph of the PLA/LS compound containing 10 w/w% LS is shown in Figure 3. The

Figure 3. SEM micrographs of compounds containing 10 w/w% LS.

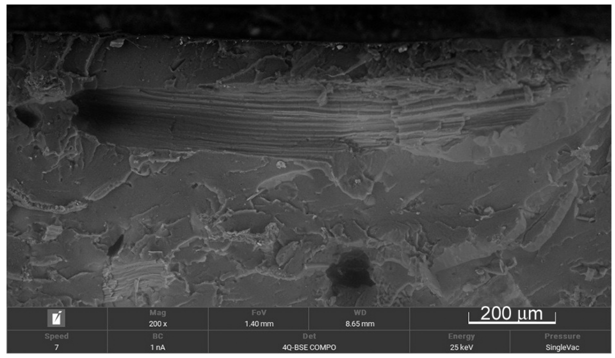

Figure 4. SEM photograph of a fractured surface of compounds containing 5 w/w% LS.

geometry of the reinforcement, which is fiber type, was observed in Figure 3. In addition, the picture shows uniform particle size distribution. A granulated lavender stem, which has a particle size of 300 µm, is shown in the photograph. These micrographs illustrate the individual separation and dispersion of the LS fibers, which indicates that the LS fibers have been separated during the extrusion process and are well dispersed in the PLA matrix.

The fractured surface of the PLA/LS compound containing 5 w/w% LS is shown in Figure 4. The mechanical properties of the compounds, which contain fiber-type reinforcement strongly depend on the adhesion between the fiber and matrix polymer. The poor interfacial adhesion between the two phases is observed in the SEM photograph (Figure 4).

3.2. Thermal properties

Table 3 shows the DSC results for samples recorded during the heating scan at a rate of 10 °C/min. To investigate the changes in the thermal properties depending on the ratio of the CC and LS, several DSC measurements were conducted. The names of the samples contain the short name and ratio of the reinforcement. Crystallinity was calculated from melt enthalpies and the equilibrium melting enthalpy of PLA (93,1 J/g) reported by Saeidlou *et al*. [30]. The results show that there is no significant difference in the glass transition temperature values between neat PLA and reinforced PLA compounds.

The DSC curves show that all samples exhibited cold crystallization during heating (Figure 5). On the curves, two melting peaks were visible because of the recrystallization of the matrix polymer. Considering the possible crystalline forms of PLA, the two peaks probably belong to the α′ and α form [31].

Table 3. Thermal properties of the compounds.

Sample name	Glass transition temperature [°C]	Crystallization temperature [°C]	Crystallization enthalpy [J/g]	Melting enthalpy [J/g]	Crystallinity [%]	Crystallinity after cold crystallization [%]
PLA	59.5	122.2	18	22	4	21
PLA_CC_2.5	60.0	110.2	23	27	5	25
PLA_CC_5	60.4	112.0	25	26	1	24
PLA_CC_7.5	59.0	108.4	23	25	2	23
PLA_CC_10	58.4	110.1	19	26	5	24
PLA_LS_2.5	60.7	109.6	22	31	6	29
PLA_LS_5	59.3	110.2	24	30	7	28
PLA_LS_7.5	58.6	109.5	23	29	7	27
PLA_LS_10	59.3	108.6	25	29	5	27

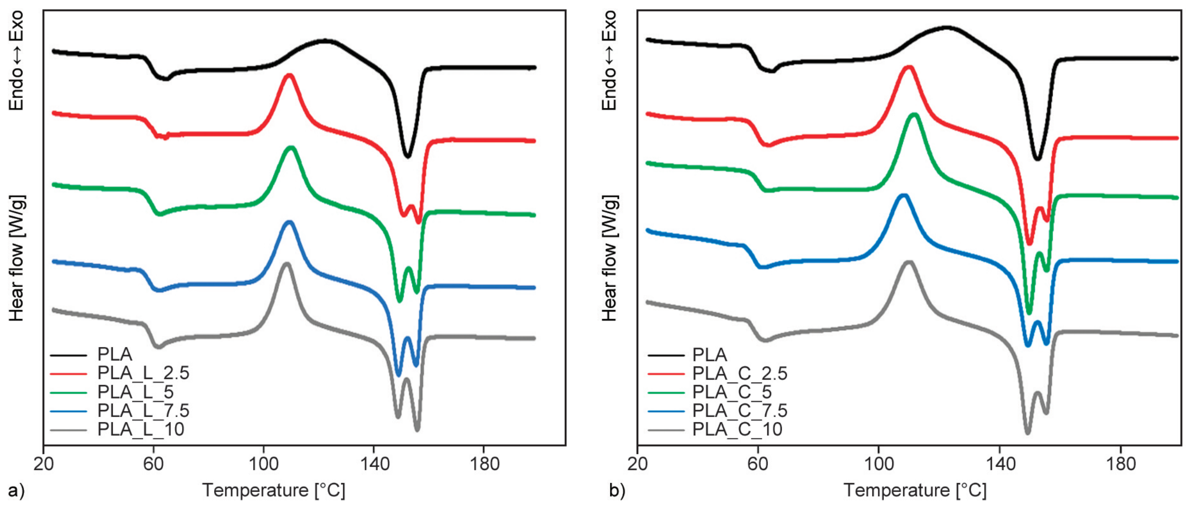

Figure 5. DSC curves of PLA/ CC (a) and PLA/LS (b) at 10 °C/min heating rate.

3.3. Heat distortion temperature

Heat distortion temperature (HDT) represents the upper stability limit of the material in service without significant changes in physical properties under load. Figure 6 shows the values of heat distortion temperatures for the PLA and PLA/CC composites. The HDT showed a slight decrease from about 58,5 to 56 °C with the addition of CC. This is attributed to the lower thermal stability of CC, and the deflection temperature is reduced by the increasing ratio of the filler content.

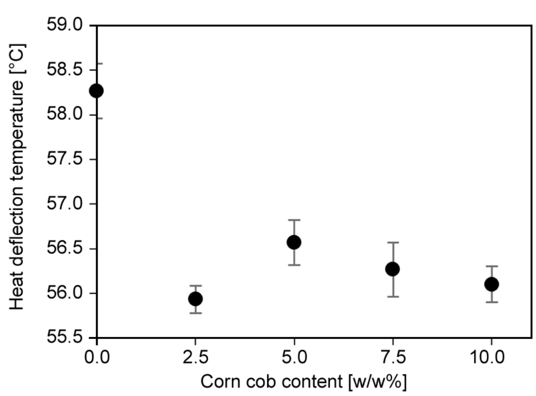

Figure 6. Heat deflection temperature as a function of CC content.

3.4. Rheology

Generally, the MFR value decreases with the filler content, meaning that the viscosity increases, due to the interaction between the two phases. The results show that the MFR values are slightly smaller than those of the neat PLA for PLA/CC composites, because of the demobilized polymer chains in the interphase (Figure 7a). A different tendency was observed for the PLA/LS compounds. The increase in MFR was caused by the poor adhesion between the two phases. In addition, the results of the dramatic increase of the MFR values in the PLA/LS compounds (Figure 7b) could also be attributed to the alignment of fiber turns with the flow direction.

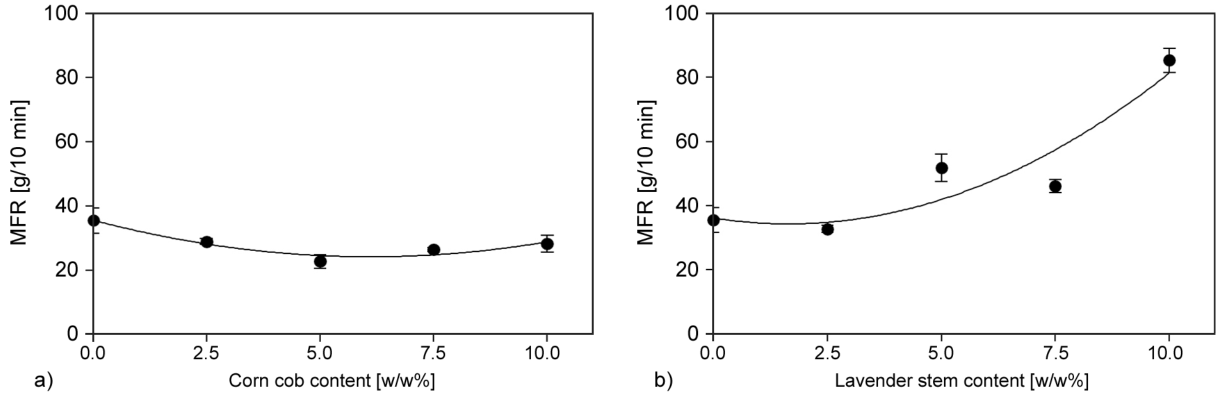

Figure 7. MFR values of PLA composites a) CC content, b) LS content.

3.5. Mechanical properties

Figure 8 and Figure 9 show that both tensile and flexural modulus have increased with the reinforcement content in both compounds. The changes are 10 and 7% compared to the tensile modulus of 3.36 GPa of the neat PLA in both cases. The modulus indicates that the stress transfers from the PLA matrix to the stiffer fiber [32]. In the case of the CC filler, a slight effect is observed, but in the case of the LS filler, a significant increase in the modulus is noted due to the fiber geometry.

While stiffness increased, the tensile strength and deformation at the break- both properties that are related to toughness - decreased, as shown in Figure 10–13. Tensile strength was 51,7 MPa for neat PLA and 45,1 MPa for PLA with 10 w/w% CC content. This could be the result of less than adequate adhesion between the fillers and the PLA matrix. Generally, the addition of high filler content increases the probability of filler agglomeration, which creates regions of stress concentrations that require less energy to elongate crack propagation [33]. Similar tendencies can

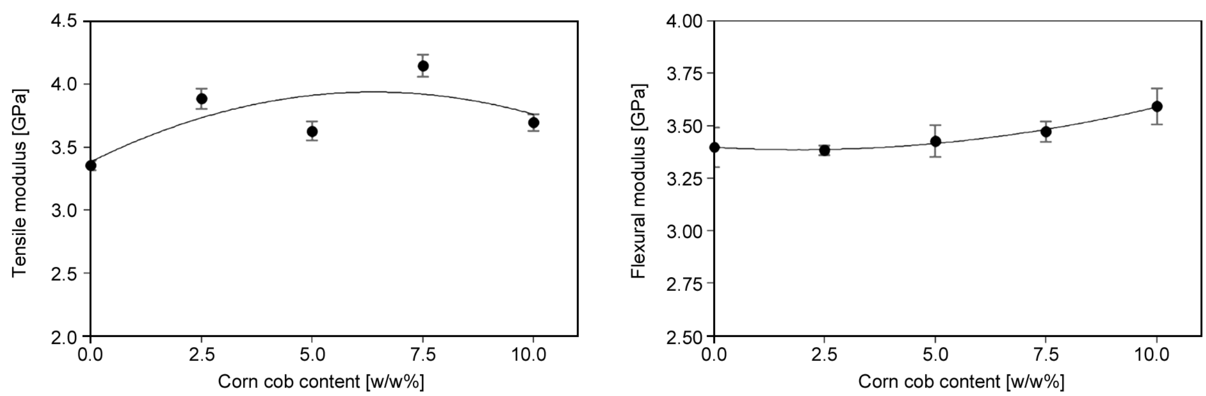

Figure 8. Tensile (a) and flexural (b) modulus of the PLA/CC compounds.

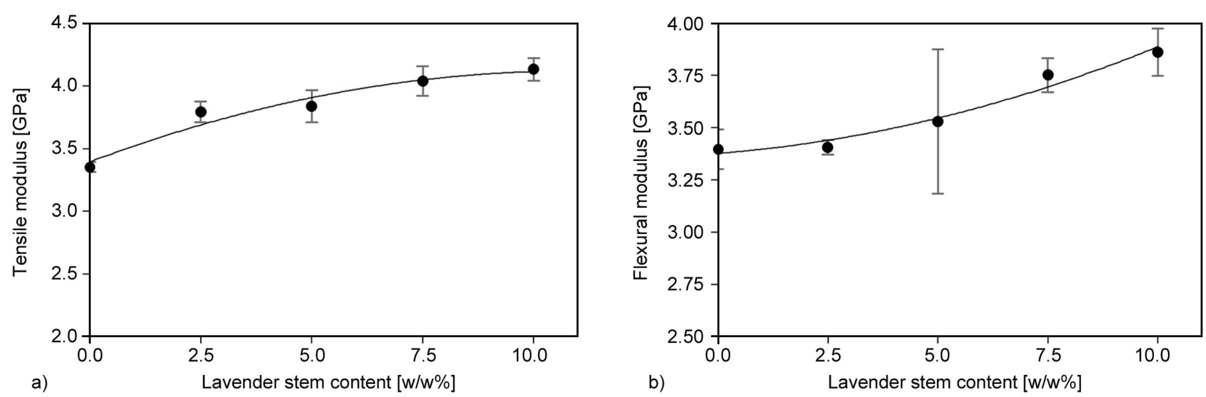

Figure 9. Tensile (a) and flexural (b) modulus of the PLA/LS compounds.

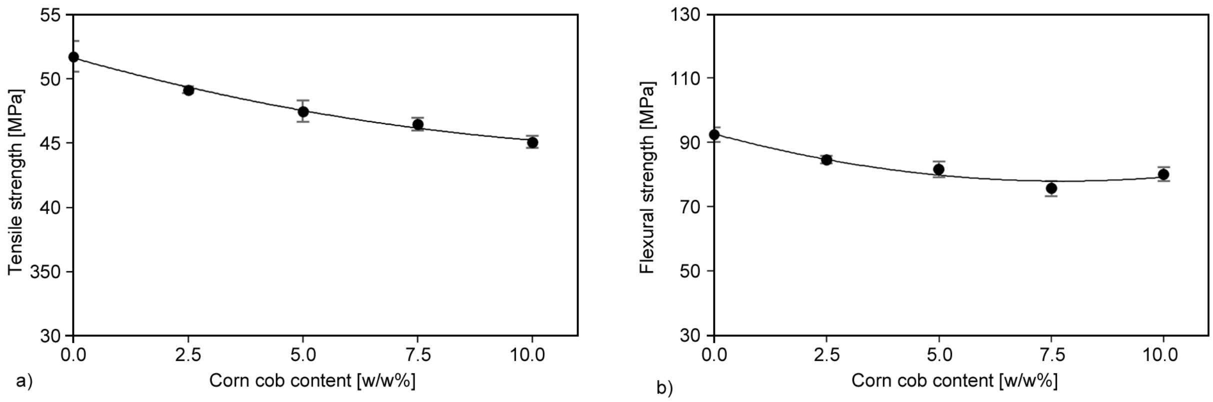

Figure 10. Tensile (a) and flexural (b) strength of the PLA/CC compounds.

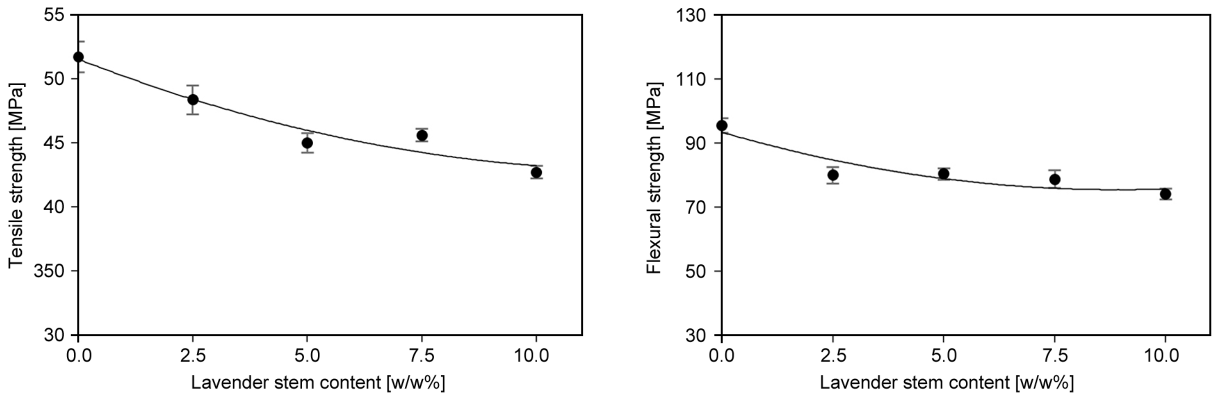

Figure 11. Tensile (a) and flexural (b) strength of the PLA/LS compounds.

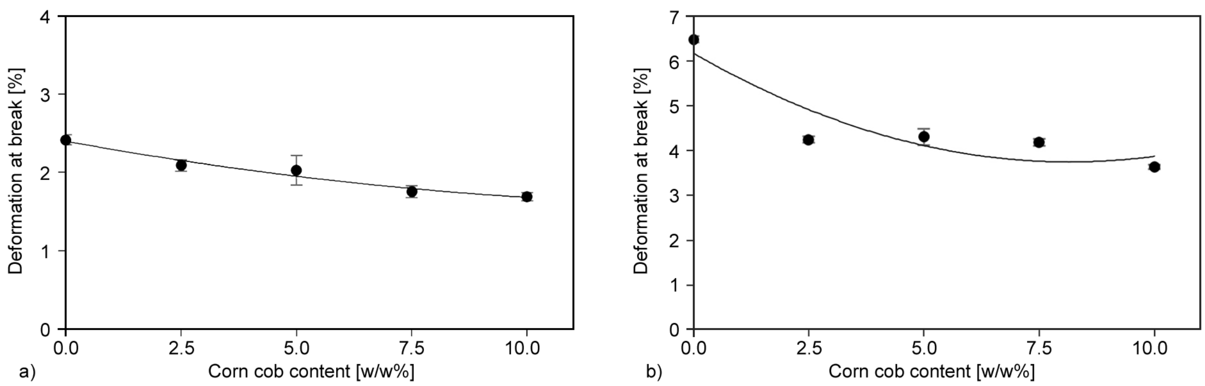

Figure 12. Deformation properties of the PLA/CC compounds. a) tensile; b) flexural.

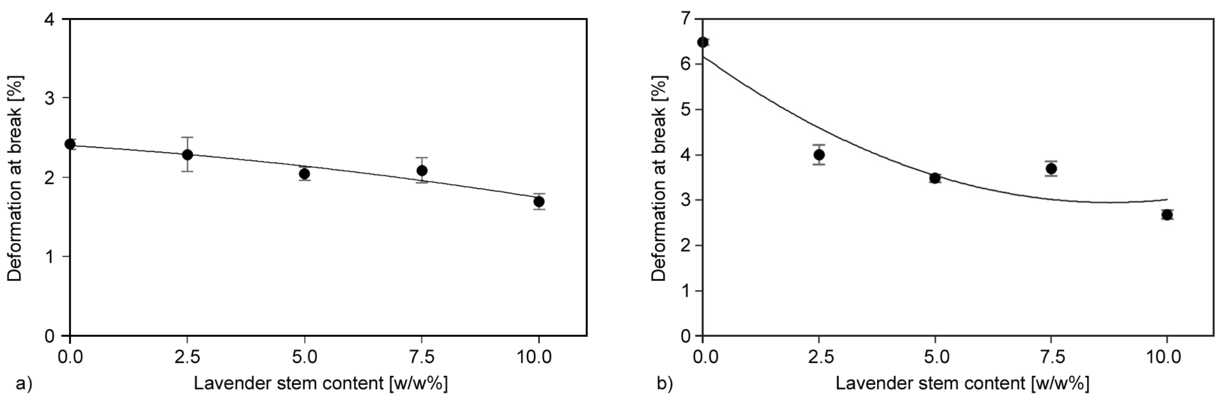

Figure 13. Deformation properties of the PLA/LS compounds. a) tensile; b) flexural.

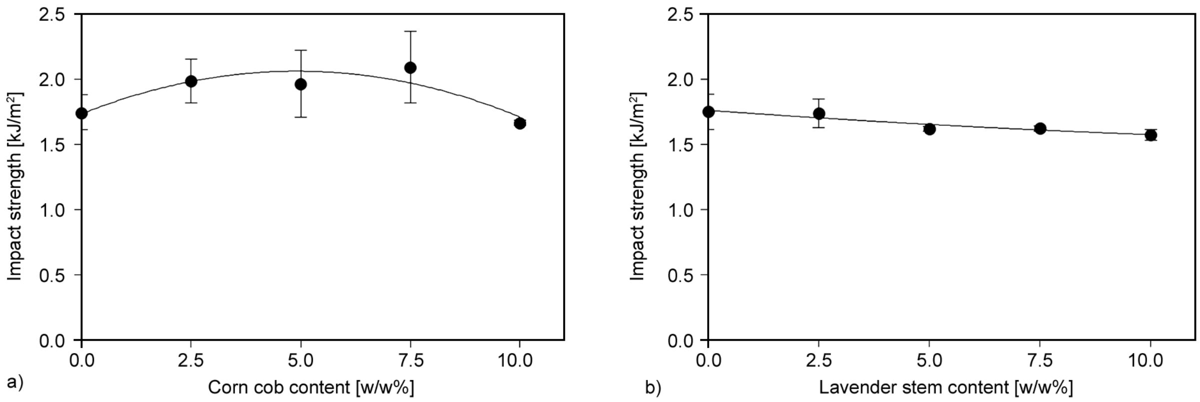

Figure 14. Impact strength of the PLA/CC (a) and PLA/LS (b) compounds.

be seen in Figure 12, where deformation at break decreased from 2.4 to 1.7%. Figure 13 shows almost the same. During the bending test, similar results were observed. The filler and fiber content significantly affected the material's mechanical properties, making the samples stiffer and more brittle.

Figure 14 shows the impact strengths of composites. They are slightly decreased with increased filler content. This is consistent with the tendency of decreased strain, indicating that the rigidness of PLA is slightly improved by the addition of fillers. The slight increase observed in the PLA/CC compounds with smaller CC content may be attributed to an error caused by data scatter.

4. Conclusions

When the results of the study were evaluated and summarized, the shape of the reinforcing material, the structure of the compounds, and the interfacial adhesion between the discontinuous and continuous phases were identified as the most important factors. An effect on the mechanical, thermal and rheological properties is exerted by them. The SEM micrographs show the homogenous structure of the compounds and the shape of the reinforcing material. CC is classified as a filler type of reinforcement, while LS is categorized as a fiber type of reinforcement. It is shown by the micrographs of the fractured surfaces that the adhesion is inadequate to have a significant effect on the properties of the compounds. This corroborated the results of the thermal and mechanical analysis, which determined that poor adhesion between the fiber and the matrix slightly decreases the HDT and the strength of the composite. The adhesion could be improved by using coupling agents from natural sources to preserve the biodegradability of the compounds.

Our experiments show that PLA/CC and PLA/LS compounds are easy to produce. The price of an end product can be reduced by using PLA/CC compounds, as no significant negative effect on the properties is observed. The lavender stem is a suitable additive to reduce the viscosity. In addition, the price of the end product can be reduced by using these materials, since they are agricultural waste. Moreover, because of the innate biodegradability of the corn cob and the lavender stem, this type of reinforced PLA is more environmentally friendly, than the composites reinforced with synthetic fiber.

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