Mechanical Properties of Polylactide Matrix Composite Reinforced with Long Maize Stalk Fibers

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ABSTRACT

The aim of presented study was to investigate the effect of using long rotation maize stalk fibers as filler on the mechanical and thermomechanical properties of an injection molded biocomposite with a polylactide matrix using different fiber percentages. To evaluate the effect of maize stalk particle reinforcement on the PLA matrix, the mechanical properties of the biocomposites including tensile strength, tensile modulus, relative strain at maximum stress, strain at break, stress at break and Charpy unnotched impact strength were determined. From a mechanical point of view, the best fiber content was at 15% as it caused the least reduction in strain at break, stress at break, and strain at maximum stress relative to the polylactide matrix.

Keywords: polylactide, biocomposites, maize stalk, mechanical properties.

INTRODUCTION

Over the past 70 years, there has been a noticeable dynamic growth in the production of plastics worldwide. When only 1.5 million tons of plastics were produced in 1950, their production has already reached 367 million tons in 2019 [1]. The growing popularity of such materials is due to their low density, low price, ease of processing, high durability, good chemical resistance and good mechanical properties. Plastics have been used in a huge range of products and applications, particularly in the packaging, construction and automotive industries. Nowadays many products made of macromolecular plastics especially food packaging, clothing, household appliances save energy, reduce CO_2 emission, water and food consumption. They play an important role in the development of modern society [2].

In 2015, about 99% of polymer plastics were petroleum-based [3]. Each year, 6% of extracted crude oil is used to produce them. Oil consumption for plastics production is expected to increase to 20% of annual oil production by 2050 [4]. Therefore, the biopolymer industry is growing, using biomass as an alternative raw material to produce plastics. This is a promising solution that does not deplete fossil fuel reserves [5].

The high durability and good chemical resistance of most of the polymeric plastics are advantages of their functional properties. However, this poses a threat to the environment due to their resistance to natural degradation. Therefore, waste management of these materials has become a major challenge in many countries especially in the area of sustainable development. Currently, there are many places on earth where there are large amounts of polymeric plastic waste found in the natural environment especially in oceans and seas [6].

One of the ways to solve the problem of polymer waste is to use biodegradable plastics which are decomposed in the environment by microorganisms such as fungi, algae, bacteria etc. The biological degradation of plastics takes from a few to several weeks. During biodegradation, polymer chains decompose into H_2O, CO_2 and biomass [7].

Degradation time depends on the type of plastic and the interaction of moisture, temperature, pH, salinity, presence or absence of oxygen, UV light and/or enzymes that cause shortening and weakening of polymer chains [8, 9].

Biodegradable polymer plastics are obtained from natural and synthetic sources. From synthetic sources, biodegradable polymers are produced typically from petrochemical feedstocks such as polycaprolactone [10]. Biodegradable polymers made from natural raw materials are referred to as bioplastics. The raw material for their production is usually starch, cellulose, lignin and bioethanol. This group includes poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV), polyhydroxybutyrate (PHB) and polyhydroxyalkanoate (PHA), and polylactide (PLA) [11, 12].

Among the above bioplastics, polylactide is currently the most widely used [13]. The PLA is an aliphatic polyester and is made by ring-opening polymerization of lactide or by polycondensation of lactic acid. In the second method, the polymer has a lower molecular weight but is more commonly used in industrial production. Lactic acid, from which lactide is also derived, is produced from natural polysaccharides by fermentation of starch-rich biomass (potatoes or maize) [14, 15]. The PLA is a thermoplastic material, which can be processed by extrusion including blow molding, thermoforming and injection molding [16]. This polymer is biodegradable over a period of several months to 2 years. It has good tensile and compressive strength but has low ductility of less than 10% elongation at break. It is relatively brittle and has poor impact strength as well as poor gas and moisture barrier properties [17]. The PLA has no reactive side groups, and therefore is chemically inert and hydrophobic in nature [18]. Poor ductility and brittleness preclude PLA in applications where the material is plastically deformed at high stress levels. It is mainly used as a packaging material for products with short shelf life, including food and beverages [19]. It is also used in the biomedical industry in drug delivery systems, implants and tissue scaffolds [20].

The PLA relative to the most commonly used synthetic polymers (PE, PP, PET, PVC) is several times more expensive. In many consumer applications, higher strength and/or lower unit price of material is required. Therefore, for reinforcement purposes, natural fibers from plants commonly grown around the world are often used as fillers for PLA [21]. Plant fibers used in polymer composites can be extracted either from plants grown for textile purposes (jute, sisal, hemp, bamboo) or from by-products of agricultural production or food processing (straw, stalks, cobs, husks, shells, bran, etc.). From by-products, cellulosic fibers have a 90% lower manufacturing cost [22]. Composites consisting of biodegradable polymers and natural plant fibers are referred to as green composites [24]. These materials are entirely decomposed by microorganisms ultimately leaving only carbon dioxide and water as byproducts. The type of plant fiber, amount of fiber in the composite, fiber size, and fiber shape affect the final properties of green composite [25].

The aim of this study was to investigate the effect of using long rotation maize stalk fibers as filler on the mechanical and thermomechanical properties of an injection molded biocomposite with a polylactide matrix using different fiber percentages. To evaluate the effect of maize stalk particle reinforcement on the PLA matrix, the mechanical properties of the biocomposites including tensile strength, tensile modulus, relative strain at maximum stress, strain at break, stress at break and Charpy unnotched impact strength were determined.

The paper presents preliminary studies for fundamental research aimed at developing parameters of shredding process of maize stalks in multi-edge systems from the point of view of obtaining the most favorable selected properties of polymer composite.

MATERIALS

The composite materials were prepared from a polylactide (PLA) matrix reinforced with maize straw (CS) stalk fibers. The polylactide (3251 D) was supplied by NatureWorks (Minnetonka, U.S.). According to the manufacturer, the polylactide with a density of 1.24 g/cm^3, a glass transition temperature of 55 to 65 °C, and a melt mass flow rate (190 °C/2.16 kg) of 35 g/10 min is intended for injection molding applications. The maize stalks were hand harvested (Mogilno, Poland) and were dried for a period of 60 days at 18 °C. After the drying period, they were shredded on a multi-hole, multi-edge system, which was equipped with two discs with different shapes of cutting surfaces. The shape of discs is shown in Figure 1. The movable disc operated at a rotational speed of 2050 rpm.

The different fiber lengths were separated using a Fritsch Anlysette 3 Pro sieve analyzer (Idar-Oberstein, Germany) equipped with a RETSCH DIN-ISO
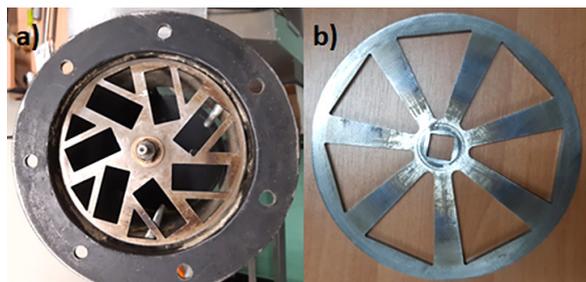
Fig. 1. Shredding system: a) fixed disc, b) movable disc

3310/1 measuring sieve set. The material was sieved for a period of 10 minutes with an amplitude of 2.5 Hz according to DIN 66165. Fiber lengths ranging from 4 mm, 2 mm, 1.6 mm, and 1 mm were used to produce the composite, which were 20.5%, 32%, 18%, and 29.5%, respectively. The fibers had an elongated needle-like shape. The average moisture content of fibers determined using a Radwag MA 110.R scale (Radom, Poland) was 9.5 %.

METHODS

Extrusion

Shredded maize stalks and PLA granules were dried in a Wamed laboratory dryer type SUP-100 (Warsaw, Poland) at 40 °C for 48 hours to remove residual moisture. The materials were then weighed separately using a Radwag WAS 160/X analytical balance (Radom, Poland). Composite formulations with 10, 15 and 20 wt% filler content were prepared and designated PLA/CS10, PLA/CS15 and PLA/CS20, respectively.

The composite formulations were mixed manually and then poured into the hopper of a Metalchem single-screw extruder type W25-30D (Gliwice, Poland). The plastifying system of the extruder has one screw with a diameter of 25 mm (D) and a length to diameter ratio (L/D) of 30. A sample containing pure PLA was prepared under identical conditions as the composite formulations. Temperature in the plastifying system of the extruder was 170 °C in the 1st section, 175 °C in the 2nd section, 175 °C in the 3rd section and 175 °C on the nozzle, respectively, and was established on the basis of previous studies in which, among others, the thermal stability temperatures of natural fibers used were determined [26]. The rotational speed was set at 92 rpm. The extruded strands were cooled with compressed air and cut into granules using a knife granulator.

Injection molding

The granules obtained from extrusion to reduce moisture content were dried at 40 °C for 72 hours and then processed by injection molding in a TRX 80 Eco from Tederic (Shanchai, China). The temperature of the injection molding plastifying system from the feed section to the injection nozzle was 170–170–175–180 °C. The short circuit force was 600 kN. After the material was injected into the mold, the samples were cooled for 60 seconds at 35 °C. The resulting samples had the shape of a dog bone and their nominal dimensions were 80 mm long, 10 mm wide and 4 mm thick.

Mechanical test

For mechanical testing, six samples from each composition were used for each test. The results of mechanical tests are the results obtained from the arithmetic mean of each parameter along with the calculated standard deviation values.

Static tensile tests of PLA and PLA/CS samples were carried out on an Instron model 3367 universal testing machine (Norwood, U.S.). The tensile rate was 50 mm/min. Test was conducted according to PN-EN ISO 527-1:2020-01.

Charpy impact tests were carried out on unnotched samples using a LIANGONG XJ 5Z impact hammer (Shandong, China) according to PN-EN ISO 179-2 using a 2 J hammer.

Thermomechanical test

Thermomechanical properties of PLA and PLA/CSF samples were tested with a dynamic mechanical analyzer type Q800 TA Instruments (New Castle, DE, US) according to PN-EN ISO 6721-1. The test samples were cuboid in shape with dimensions of 70 mm × 10 mm × 4 mm. Thermal sweep was scheduled from 30 °C to 140 °C at a heating rate of 2 °C/min, with a strain frequency of 1 Hz and an amplitude of 15 μm.

RESULTS

Tensile strength of PLA was 66.7 ± 2.3 MPa. It decreased by 29% to 47.2 ± 2.9 MPa in samples containing 10% stalk particles compared to samples with pure PLA, as shown in Figure 2. This is most likely due to the weak interfacial bonding between the CS stalk fiber (hydrophilic) and PLA plastic
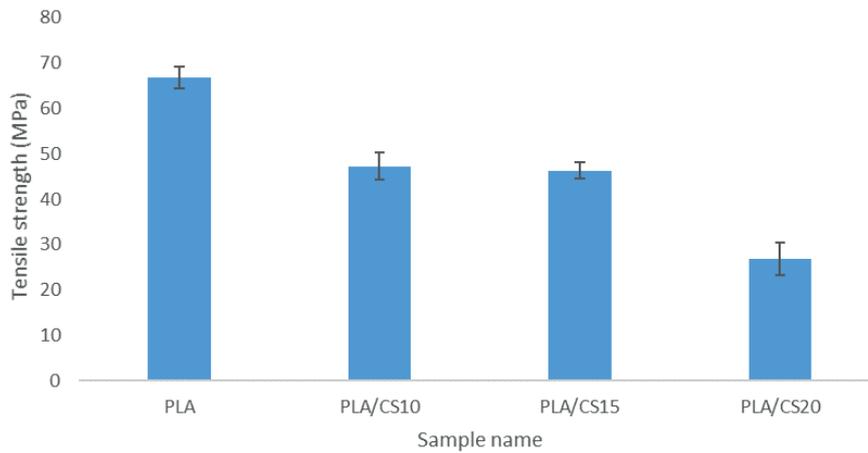
Fig. 2. Tensile strength comparison of PLA and biocomposite samples

(hydrophobic) [27, 28]. It has been observed that increasing the proportion of stalks above 15% causes a drastic decrease in tensile strength. Previous studies also showed similar behavior in decreasing the tensile strength of PLA-based biocomposites reinforced with maize stalk particles with increasing maize straw fiber content [23, 29, 30].

Tensile strain is a measure of the relative strain length of samples caused by the applied tensile force. At the maximum stress, the pure PLA samples had the highest strain as shown in Figure 3. The strain value of PLA samples was 3.42 ± 0.17 %. Reduction in strain of fiber samples is due to poor interfacial adhesion between PLA and fiber [31]. The improvement of interfacial adhesion can be obtained by modifying the fiber surface [27, 28, 32]. The CS can affect the interphase formation and mobility of polymers molecules in the composite. Short fibers cause the formation of aggregates which improve the strain especially at high composite loads [29]. Fiber due to its higher brittleness

than PLA can influence the increase in the amount of brittle phase in composite and decrease in ductile phase by which the result can be decrease in strain [33, 34]. The highest tensile strain at maximum load of samples containing maize stalk fibers was at 15 % fiber proportion [29].

Figure 4 shows the Young's modulus values of tested materials. It can be seen that the modulus of elasticity or stiffness increases with the amount of fiber contribution. The stiffening effect results from the reduced mobility of the polymer chain due to the use of the CS fiber, which shows better mechanical properties compared to the polymer matrix [33] This observation is also noticeable when other fibers with polylactide matrix are used as observed by Nyambo [23, 35] among others. There was an increase in Young's modulus of 17%, 9% and 28% relative to the matrix in the studied composites reinforced with 10, 15 and 20% maize fiber. However, it was much less than in the study of Tarres et al. in which the matrix was HDPE [36].
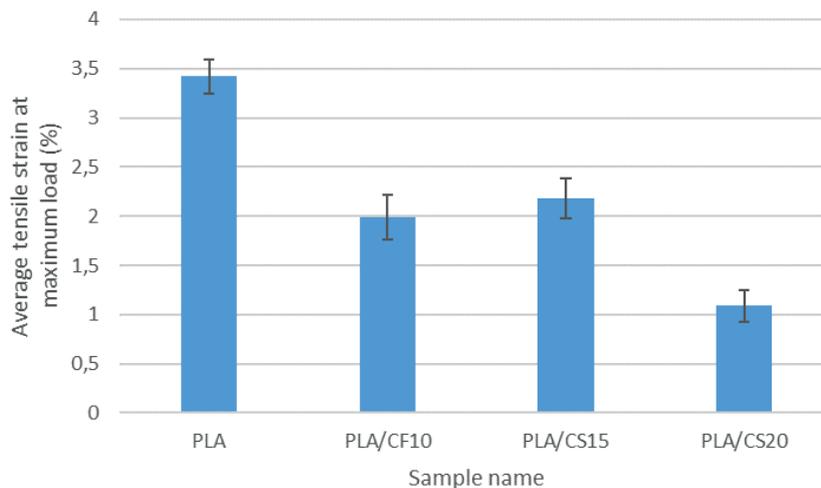

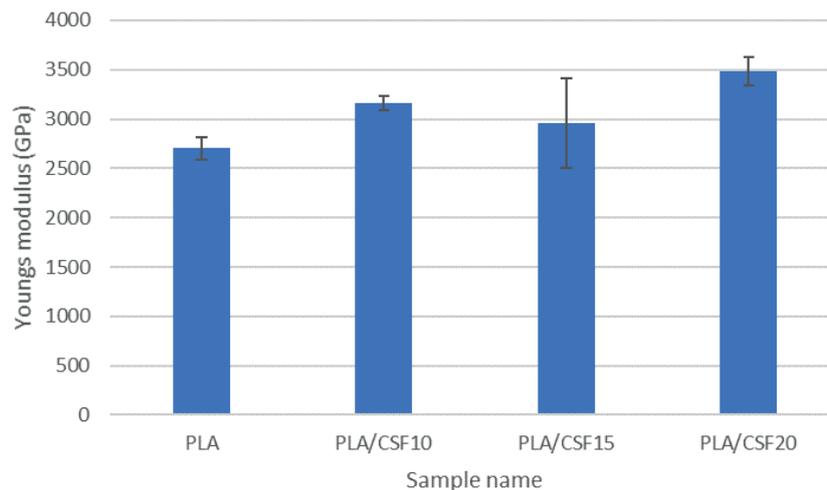**Fig. 3.** Average tensile strain at maximum sample load

Fig. 4. Modulus of elasticity of tested materials

Figure 5 shows the average values of stress at break of the tested samples. The highest value of stress at break was recorded for the samples with pure PLA and its value was 61.59 ± 3.75 MPa. The addition of maize fiber to the polymer matrix decreased the stress at break. The stress values at 10% and 15% fiber content were very similar. This may be due to the PLA/CS15 composite having a higher proportion of fiber fraction less than 340 μm in length than the PLA/CS10 composite, which transmit stress better [37]. On the other hand, above 15% CS in the matrix, there is a noticeable strong decrease in stress related to poor material homogenization. A similar effect is noticed by Zhang et al [33].

Figure 6 shows the average values of strain at break of the tested samples. The samples with pure PLA had the highest strain and their strain value was 4.06 ± 0.53 %. Addition of 10% maize stalk fiber to the PLA matrix reduced the strain at break by 51%. The samples containing 15% had the highest strain among the biocomposites.

The PLA belongs to brittle polymers. It showed an average value of 22.31 ± 1.51 kJ/m^2 when tested for impact strength. The addition of CS to the PLA matrix resulted in a linear decrease in impact strength with increasing fiber amount, from 19.4 ± 2.08 kJ/m^2 at 10% maize stalk fiber content up to 12.99 ± 0.52 kJ/m^2 at 20 % fiber content as shown in Figure 7. Decreased impact in the tested composites may be due to the stiffening effect of the matrix previously observed in the tensile test. Poor interfacial adhesion between the composite components results in poor stress transfer between them [38, 39, 40]. Furthermore, it is possible that an increase in CS content generates fiber clusters in biocomposites, which can function as crack initiation sites as is the case with wood fibers [41].
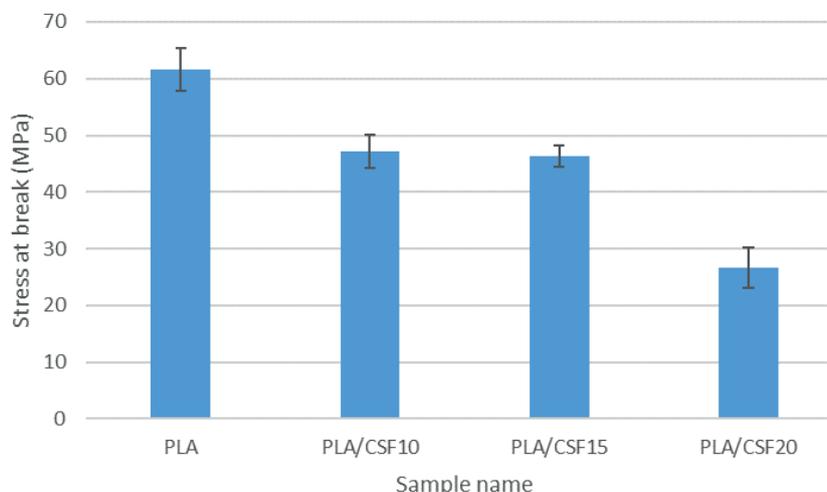

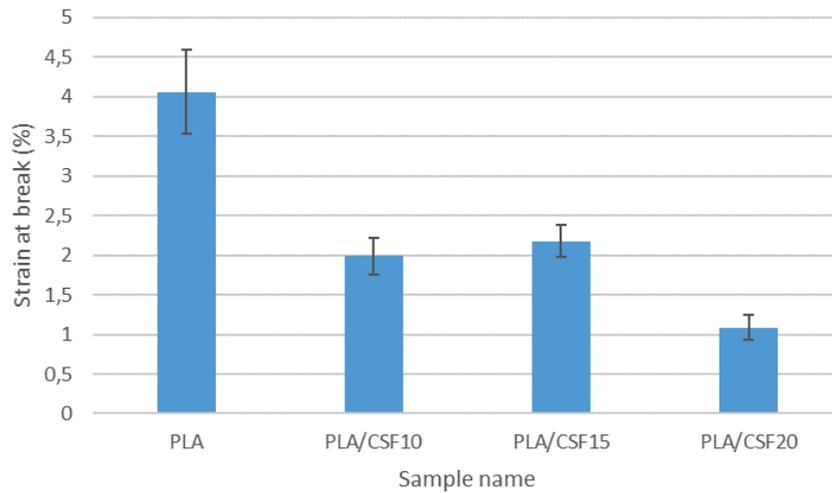**Fig. 5.** Stress at break of tested samples

Fig. 6. Relative strain at break

Thermomechanical properties of tested samples were determined by the variation of damping factor (tanδ) and the storage modulus (E ') as temperature function. Figure 9 shows the DMA thermomechanical curves. In Table 1 shows the values of E' at 30 °C, 50 °C, 75 °C, 130 °C and the maximum value of Tan δ and the temperature at which this value is obtained. It can be seen that all composite samples showed higher E ' values than the pure polylactide sample. Especially at 30 °C, where the storage modulus of pure PLA sample was 3380 MPa, and with the increasing amount of fiber in the matrix this E' increases up to 4111 MPa at 20% CS. The effect of increasing stiffness for polymer composites strengthened with natural filler has already been described elsewhere (42, 43). The effect of corn stalk strengthening is very evident beyond the glass transition area, where the value of modulus retained at 75 °C increased

from 22.13 MPa without CS up to 65.65 MPa with 15% corn fiber content. This effect may be due to the effect that relatively hard fillers have on the polymer matrix. The fillers also affect the change in G' value at 130 °C after cold crystallization. A twofold increase in modulus from 246 to 587.8 MPa at 20% fiber content is evident compared to pure PLA.

Tan δ is the quotient between E ″ and E ' (44). It is a ratio that determines the efficiency of energy loss of the material for molecular regrouping and internal friction (45). It can be seen in the graph that the maximum Tan δ value decreases along with the increase in CS. Which can be related to the increased stiffness imparted by the CS fibers. The addition of fibers hinders the molecular motion associated with damping and may be an advantage in some applications where better resistance to mechanical loading is required. The
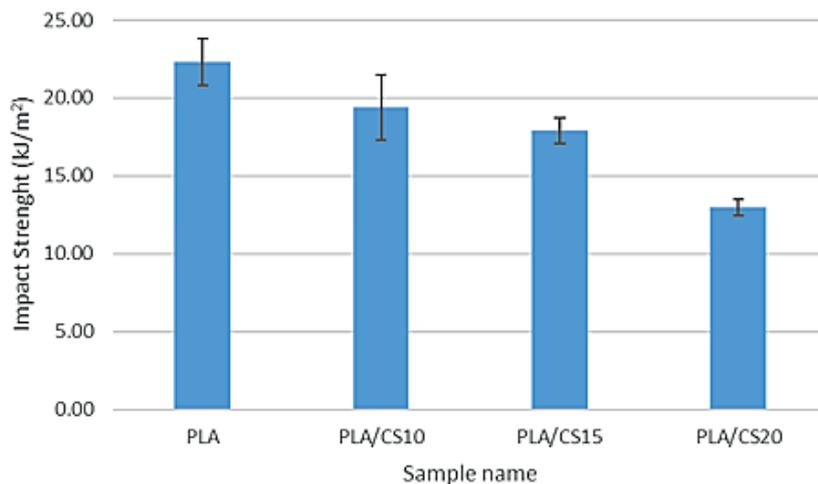

Fig. 7. Impact strength of tested samples

Table 1. Variation of storage modulus E' at 30 °C, 50 °C, 75 °C, 130 °C and Tan delta temperature dependence of the tested samples

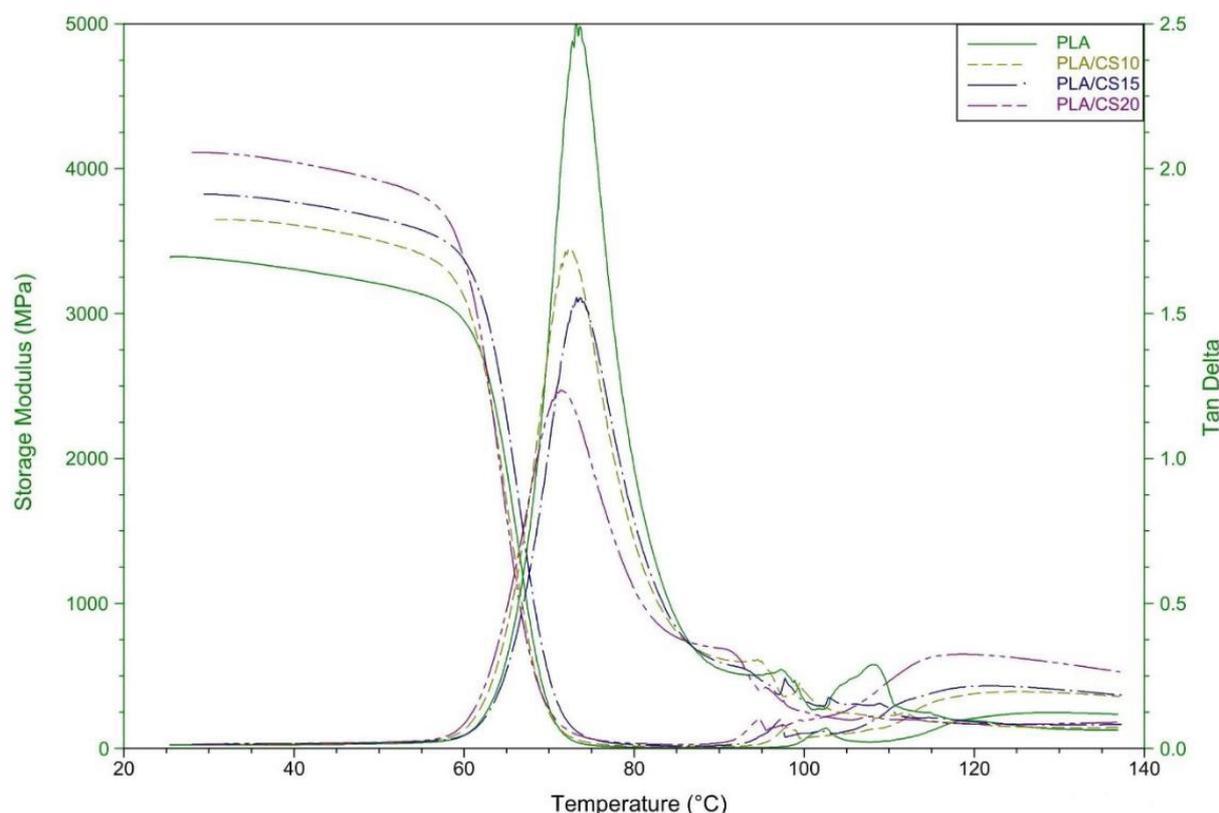| Composition symbol	E'30°C	E'50°C	E'75°C	E'130°C	Tan delta/ temperature
PLA	3380 MPa	3205 MPa	22.13 MPa	246.9 MPa	2.48/73.25°C
PLA/CS10	3648 MPa	3502 MPa	36.92 MPa	3112 MPa	1.72/72.41°C
PLA/CS15	3824 MPa	3659 MPa	64.65 MPa	3372 MPa	1.55/73.24°C
PLA/CS20	4111 MPa	3918 MPa	65.26 MPa	3382 MPa	1.23/71.33°C

Fig. 8. The DMA thermomechanical curves of the tested samples

high temperature obtained for the sample containing 15% CS may indicate a better interfacial interaction between the composite components.

CONCLUSIONS

In the presented study, PLA-CS biocomposites (up to 20% CS by weight) were produced by extrusion followed by injection molding. The aim was to evaluate the effect of using long maize stalk fibers in the polylactide matrix on mechanical and thermomechanical properties. The CS is one of by-products resulting from the cultivation of maize for grain. The poor interfacial interaction between the fiber and the polymer matrix caused the obtained composites to have poorer mechanical properties than pure polylactide. The CS caused a reduction in tensile strength from 29 to 40%, strain at break from 47 to 73%, stress at break from 23 to 57%, and impact strength from 14 to 42%. However, an improvement in modulus of elasticity of 10 to 25% as well as an improvement in retention modulus of 8 to 21% at 30 °C and 9 to 22% at 50 °C are noticeable.

From a mechanical point of view, the best fiber content was at 15% as it caused the least reduction in strain at break, stress at break, and strain at maximum stress relative to the polylactide matrix. The determined maize stalk fibers content in the composite will provide a reference value for further studies on the shredding process of maize fibers in terms of their use as fillers in polymer composites.

This study shows that corn stalk (CS) fibers can be a good alternative to other lignocellulosic additives used in green composites. When used in green composites, corn stalks due to their very high availability and biodegradability allow for some economic and environmental benefits relevant to the closed loop plastics industry.

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