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STUDY OF THE MECHANICAL PROPERTIES OF POLYLACTIDE COMPOSITES WITH JUTE REINFORCEMENTS

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Abstract

The concern that has arisen in recent years over the excessive use of oil-based materials has made the development of new materials with low environmental impact imminent, in this context. In this study, environmentally friendly composites were obtained with a thermoplastic polylactide matrix (PLA), and jute fibers (fabrics and non-woven mats) as reinforcement. PLA/jute bio-composites were manufactured by thermocompression. The effect of the amount of jute fibers reinforcement (in the 30-50 wt.% range) on the tensile and flexural properties of these composites was analyzed, and the fiber-matrix interaction was assessed by scanning electron microscopy (SEM). The results show that thermocompression moulding is a simple technique to obtain high environmental efficiency bio-composites with high reinforcement loading (up to 50 wt.%). As expected, the tensile properties are directly related to the amount of fiber loading, as well as the directionality these fibers have in the composite. Mechanical performance is also highly dependent on fiber-matrix interactions. These bio-composites could be attractive as lightweight interior panels in automotive industry, case/covers in electric-electronics applications, shovels' components in the wind energy industry, among others, due to their balanced mechanical properties, and the rather complex shapes that could be obtained by thermocompression.

Keywords

Biopolymers; mechanical properties; biofibers

1 INTRODUCTION

New high mechanical performance materials to replace metallic materials and their alloys have emerged in the recent years. In this sense, composite materials have been extensively studied due to the wide range of final properties that can be obtained, making them invaluable in industrial applications such as the automotive industry or the transport industry [1, 2]. Fiber-reinforced polymeric materials are among the most common, as materials with unique properties can be tailored by correctly defining the type of matrix and reinforcing fibers. These materials generally provide high strength, good stiffness, and low weight [3]. Thermoset matrices such as epoxy resins, polyurethanes, unsaturated polyesters, among others [4-7], and thermoplastic matrices such as polyethylene (PE), polypropylene (PP), and polyvinylchloride (PVC), among others [8-10], have been some of the most widely used materials in composites. This has resulted in a huge production of polymers reaching 368 million tonnes worldwide by 2019 [11] leading to an increase in the CO_2 emissions to the environment during their production. This phenomenon, together with the extremely low biodegradation (actually, disintegration in controlled compost soil) rate petroleum-derived polymers show, has promoted the accumulation of these materials over long periods, thus leading to a growing environmental impact [12].

Polymer composites combine the excellent reinforcing properties of fibers, with the exceptional processability of polymers, thus leading to materials with unique properties. The use of thermosetting matrices such as epoxies (EP), and unsaturated polyester (UP) has been generalized sue to the exceptional properties with glass fibers (GF) and carbon fiber (CF) [13-15]. Those fibers offer an unique mechanical properties (strength and stiffness) that, together with a remarkably low density, allow their use in high-performance applications such as aerospace, aeronautics, automotive parts, and so on [16-19]. Nevertheless, manufacturing of CF requires complex processes at high temperatures which contribute to increase the overall carbon footprint. This has led to an increased in the environment awareness due to the negative effects associated with the indiscriminate consumption of petroleum-derived materials, and high energy consumption of many manufacturing processes. For this reason, the research and development of new materials with a lower environmental impact has emerged in the last decade [20].

The choice of natural plant fibers is a solution to conventional fibers. The most used natural fibers are flax, sisal, hemp, jute, and many others, which have different physical, chemical, and mechanical properties depending on their composition [21-24]. Jute fiber offers interesting balanced properties in terms of insulating capacity, hygroscopicity, biodegradability, and rather good mechanical properties. Nevertheless, as with other natural fibers, the final properties are influenced by various parameters such as the fiber length, composition, manufacturing process, crop conditions, plant part, and so on. Biswas, Ahsan [25], reported and in-depth study of the properties of jute, bamboo and coir natural fibers. They reported tensile strength values between 200 and 600 MPa with an elongation at break from 1.25% up to 1.9% for a fiber span length of 25 mm. With regard to the Young's modulus, values ranging from 10 to 27 GPa, depending on the fiber span length, were reported. In addition natural fibers are cost-effective ($0.5 per kilo) compared to synthetic fibers such as glass fiber (GF) [26].

Environmentally friendly materials can be related to two stages in their life-cycle. The first concern is directly related to the origin, which can be petroleum or a renewable resource. The second topic considers the end life, thus sorting materials in two main groups, namely biodegradable (actually, disintegrable or compostable under certain conditions), or non-biodegradable [27, 28]. Despite some remarkable advances have been done in the field of bio-based thermosetting materials such as epoxies (EP) from epoxidized vegetable oils [7, 29-31], unsaturated polyesters (UP) with fully or partial bio-based content [32, 33], phenolics (PF) [34, 35], and so on, the most important advances in the field of biopolymers has been observed in the thermoplastic industry. Additionally the use of thermoplastic materials allows recycling the composite by the remelting of the polymer matrix [36].

Thermoplastic biopolymers include both bio-based and biodegradable polymers. Some aliphatic/aromatic petroleum-derived polyesters such as poly(ε-caprolactone) (PCL) have gained interest as can disintegrate under controlled composting conditions. Despite these polymers are interesting, biobased and biodegradable polymers offer the best environmental efficiency. Among these, polylactide PLA and polyhydroxyalkanoates and PHAs are being intensively studied [37-40]. Polylactide (PLA) is, with difference, one of the most widely used biobased and biodegradable thermoplastic polymer. PLA is an aliphatic polyester obtained by the fermentation of natural resources such as starch-rich materials [41]. Outstanding properties such as rapid degradability under composting conditions, biocompatibility with the human body, high tensile strength, transparency, have positioned PLA as a suitable material for sectors as biomedicine or food packaging [42-44]. In addition, PLA has driven the development of new composite materials using renewable raw materials in the form of reinforcing fibers or fillers [27, 45, 46]. The final properties of these new materials will depend on both the nature of the reinforcement and the amount of filler [47-49].

The use of thermoplastic matrix allows to manufacture composites in a different way than with thermosetting matrices. In particular, composites with interesting technical properties can be manufactured by conventional thermoplastic processing techniques such as injection moulding, extrusion, roto-moulding and thermocompression. It is worthy to note the interesting characteristics of thermocompression moulding since it offers several advances due to a lower cost, minimal waste, simplicity and better mechanical properties [50].

This has led several authors to focus their efforts on the study of PLA/natural fiber composite materials. An important parameter to consider during the manufacture is the processing temperature since most lignocellulosic fibers (including flax, hemp, jute and so on) can undergo degradation above 195 ºC and as a result the composite performance could be reduced [51]. Du, Peng [52] manufactured bio-composites based on jute fabrics pre-treated with an alkaline solution in a PLA matrix by hot compression molding. The pretreatment resulted in better bonding between the fibers and the matrix, as well as improved thermal stability. Silanization and alkali treatments have also been successfully used to improve fiber-polymer interactions in PLA-flax fiber composites [53]. Khan, Terano [54] investigated the influence of fiber orientation on the mechanical properties of bio-composites made from non-woven jute fabrics and woven jute fabrics in PLA matrices. Woven materials offer superior mechanical properties compared to non-woven materials, giving better warp than weft directions. Jute has been proposed as reinforcement in environmentally friendly composite laminates with different thermosetting matrices such as unsaturated polyester (UP) and epoxy (EP) resins, with balanced mechanical properties and a clear advantage from an environmental standpoint, compared with their counterparts with conventional glass fibers [55-57]. Despite jute fiber has been widely used as reinforcement into thermosetting resins, an interesting challenge is manufacturing of jute-based composites with thermoplastic polymers matrices by using conventional manufacturing techniques of polymers.

The main objective of this study is the development and characterization of high environmentally friendly green composites based on polylactide (PLA) matrix and different jute reinforcement contents in both woven and nonwoven structures. The novelty of this work relies on the use of a cost-effective thermocompression moulding process to obtain high jute loading PLA composites up to 50 wt.%. The influence of the weight content of the jute fiber on the mechanical properties of the biocomposites in terms of flexural and tensile properties is shown. Furthermore, the quality of the interface between the jute fiber and the PLA matrix is assessed by scanning electron microscopy (SEM).

2 EXPERIMENTAL

2.1 Materials

PLA grade Ingeo^{TM} 6201D supplied by NatureWorks LLC (Minnetonka, MN, USA) was used as the thermoplastic matrix. It has a glass transition temperature (T_g), around 60 ºC and a melting peak temperature close to 170 ºC; this grade possesses a melt flow index (MFI) between 15-30 g/10 min measured at 210 ºC. It also has a relative density of 1.24 g/cm^3. Two types of jute textiles were used as reinforcement fibers. The first one was a biaxial plain wave fabric with a surface density of 380 g/cm^2. The second one, consisted of a non-woven fabric with a surface density of 270 g/cm^2, both of them were kindly supplied by Hilaturas Ferre S.A. (Alicante, Spain).

2.2 MANUFACTURING OF THE PLA/JUTE BIOCOMPOSITES.

Manufacturing of PLA/jute biocomposites was carried out by thermocompression moulding in a Hoytom M.N 1417 hot-press from Robima S.A. (Bilbao, Spain) equipped with a temperature control from Dupra S.A. (Castalla, Spain). Prior to the processing of the biocomposites, PLA pellets were dried at 60 °C for 24 hours to remove moisture and prevent PLA from degradation by hydrolysis [58]. A schematic plot of the thermocompression process can be seen in **Figure 1**. Despite there have been developed many processes which combine different stacking sequences of polymer film/sheet and fabrics, this work focuses on the potential of a still more easy processing technology. This consists of depositing interleaved layers of PLA pellets and jute fiber in an aluminum mold (**Figure 1a & Figure 1b**) designed to produce squared plates of 200x200 mm and different thickness. Different weight proportions of jute fibers/PLA were selected for this work, ranging from 30 to 50 wt.% as proposed in **Table 1**.

TABLE 1 Composition and code designation of the different PLA/jute bio-composites.

Jute Fiber (wt%)	**Type fiber**					
	Woven			Non-woven		
	Code	**Jute layers**	**PLA layers**	**Code**	**Jute layers**	**PLA layers**
0	PLA_0	-	1	PLA_0	-	1
30	PLA_WJF_{30}	3	4	PLA_NWJF_{30}	5	6
40	PLA_WJF_{40}	4	5	PLA_NWJF_{40}	6	7
50	PLA_WJF_{50}	5	6	PLA_NWJF_{50}	8	9

The corresponding amounts of PLA pellets were weighed and distributed homogeneously between the jute fabric layers to give the above-mentioned wt.% jute as seen in **Table 1**. To enhance full embedment of jute fabrics with PLA, both the top and the bottom layers consisted of PLA pellets (**Figure 1c**). Since the melt peak temperature of PLA is around 165-170 ºC, the mould was closed and subjected to a temperature of 190 ºC with a constant load of 2 Tn for a total time of 20 min. These conditions allow PLA to flow and embed the jute reinforcements. After the heating program, the aluminium mould was cooled down to 70 ºC in a water circulating bath. Finally, the obtained green composite plates were released from the mould for further characterization.

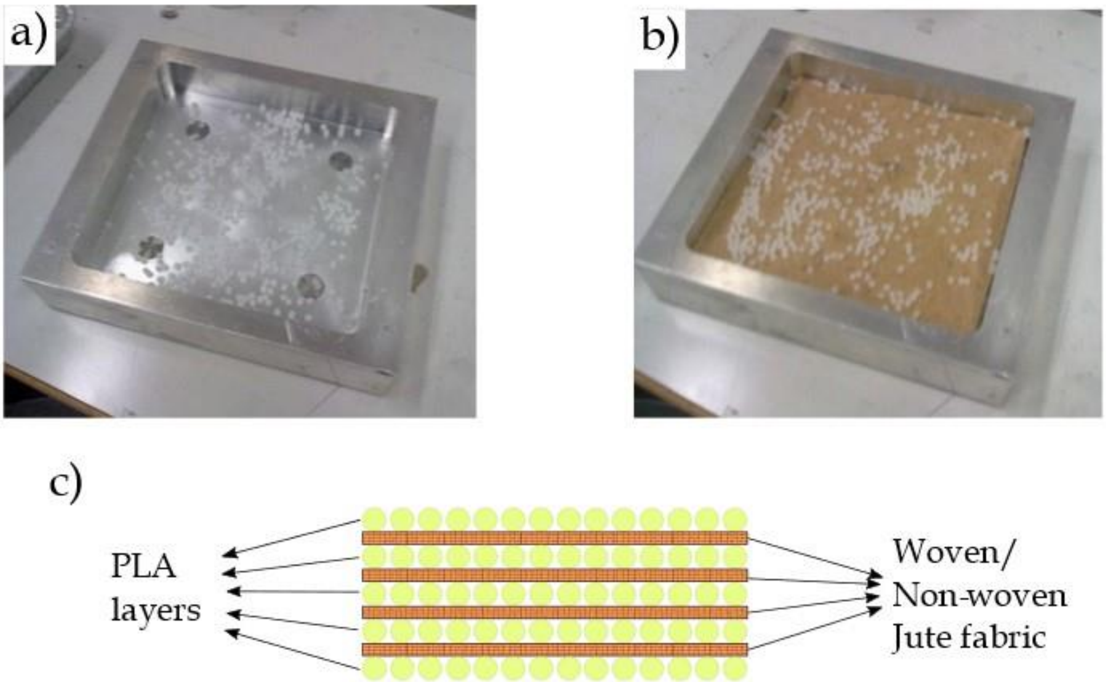

FIGURE 1 Manufacturing process of PLA/jute biocomposites by thermocompression moulding. a) placing the first PLA layer on the aluminum mould; b) placing the last PLA layer on top of the jute fiber layer; c) schematic representation of the stacking sequence of PLA pellets and jute fibers.

Figure 2 shows the appearance of woven (**Figure 2a**) and non-woven (**Figure 2b**) jute fiber fabrics supplied by the manufacturer, where the distribution and orientation of the fibers in the different fabrics can be seen. **Figure 2c** and **Figure 2d,** show the surface appearance obtained after thermocompression moulding, where it was possible to observe the good compaction that jute fabrics have with the PLA matrix since the matrix was able to completely embed the jute fabrics. This resulted in highly homogeneous composites.

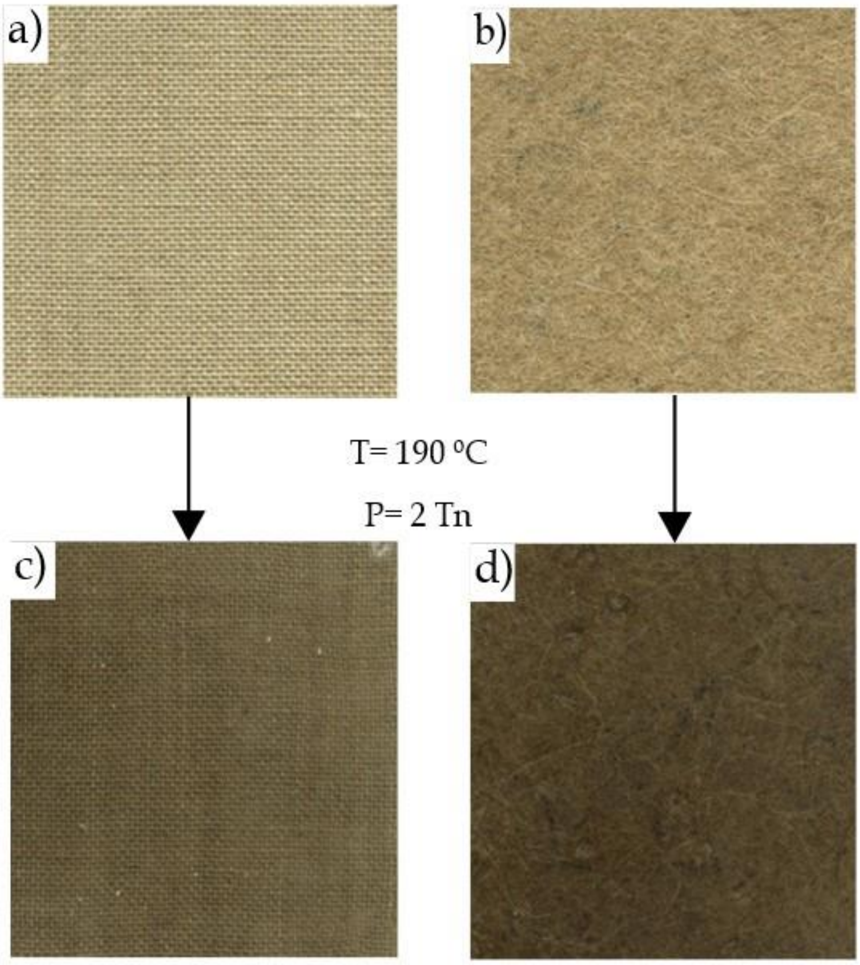

FIGURE 2 Images of the woven jute fabric (left column) and the non-woven jute fabric (right column). a)- b) before thermocompression moulding and c)-d) after thermocompression moulding.

2.3 MECHANICAL PROPERTIES OF THE PLA/JUTE BIOCOMPOSITES

The mechanical properties of the PLA/jute biocomposites were evaluated in tensile and flexural conditions. Both tests were performed in a universal testing machine Instron 3340 (Norwood, MA, USA) at room temperature (~25 ºC). The machine was equipped with a load cell of 5 kN. Tensile tests were carried out following ASTM D3039/D3039M. Tensile test specimens had these dimensions: 250 mm length, 25 mm width, and 2.5-3.5 mm thick. Tests were carried out at a crosshead rate of 10 mm/min. By means of this test it was possible to obtain the tensile strength at break (σ_b), the Young's modulus (E_t), and the elongation at break ($\%\varepsilon_b$). Flexural test was performed according to ASTM D790 standard. The specimens for flexural tests had the following dimensions: 100 mm length, 10 mm width, and 2.5-3.5 mm thick. The crosshead rate for flexural tests was set to 5 mm/min and the corresponding flexural strength (σ_f), and the flexural modulus (E_f) were obtained. To obtain reliable data, at least five different specimens were tested for each composite and the corresponding mechanical parameters were averaged.

2.4 MORPHOLOGY ANALYSIS OF THE PLA/JUTE INTERFACE

The interaction between the jute fibers (woven and non-woven fibers) and the PLA matrix was studied by scanning electron microscopy (SEM) in the fractured cross-sections of the specimens after tensile tests. A Phenom scanning electron microscope from FEI Company (Eindhoven, The Netherlands) operated at 10 kV was used. Prior to the surface characterization, all samples were sputtered with a thin layer of gold/palladium alloy in an EMITECH Sputter Coater model SC7620 from Quorum Technologies (East Sussex, UK). Furthermore, a stereomicroscope system model SZX7 supplied by Olympus (Tokyo, Japan) was used to study the morphology of the cross-section of the different biocomposites based on woven and non-woven jute fibers.

3 RESULTS AND DISCUSSION

3.1 MECHANICAL PROPERTIES OF THE PLA/JUTE BIOCOMPOSITES

The mechanical characterization of neat PLA and the PLA/jute biocomposites with jute fibers are gathered in **Table 2**. It is possible to see that neat PLA has a high Young's modulus (E_t), and a high tensile strength (σ_b) with values of 3430.3 MPa and 56.7 MPa, respectively. These values are in accordance with the literature since PLA is a stiff material that also presents a brittle behaviour, which is evidenced by its extremely low elongation at break of about 1.7 % [58, 59]. PLA/jute biocomposites containing 30 wt.% of woven jute fiber show a slight decrease in the stiffness, which can be seen by a decrease of 7.61% and 12% of its Young's modulus and tensile strength, respectively, as well as a noticeable increase of around 53% in its elongation at break (2.6%). These results suggest that this amount of fiber is not sufficient to reinforce the PLA matrix and hence, in this case would be acting as a filler. Similar situation was reported by Burrola-Núñez, Herrera-Franco [60]. They observed that the incorporation of low amounts of short jute fibers (5 wt.%) was not enough to reinforce the PLA matrix. Furthermore, this effect of decreasing Young’s modulus and tensile strength was also attributed to the low adhesion of the fibers to the matrix.

TABLE 2 Tensile and flexural properties of the PLA/Jute bio-composites.

Code	**Tensile Properties**			**Flexural Properties**	
	E_t (MPa)	**σ_b (MPa)**	**ε_b (%)**	**E_f (MPa)**	**σ_f (MPa)**
PLA_0	3430.3±60.3	56.7±2.8	1.7±0.06	3338.8±102.5	39.8±2.1
PLA_WJF_{30}	3169.2±128.4	49.8±1.9	2.6±0.09	3740.4±152.2	86.0±3.4
PLA_WJF_{40}	3325.7±166.3	53.2±3.1	2.6±0.1	3621.8±144.2	94.0±3.8
PLA_WJF_{50}	3779.0±151.1	44.9±2.8	1.5±0.1	3993.6±115.7	71.7±2.9
PLA_NWJF_{30}	1111.7±65.1	14.7±0.6	2.5±0.02	3489.3±137.2	39.4±2.5
PLA_NWJF_{40}	1078.0±58.7	29.1±1.3	2.6±0.03	3597.1±146.7	42.8±2.6
PLA_NWJF_{50}	1345.3±69.2	34.5±2.1	2.6±0.06	3679.8±128.4	62.2±3.1

In addition, these results also suggest stresses are not transmitted properly between the matrix and the reinforcing fiber [61]. This is due to the different structure of the polymer matrix and the reinforcing fibers. Jute fibers are highly hydrophylic, as other lignocellulosic fibers, while PLA matrix is hydrophobic due to very low polarity, compared to the fibers [62, 63]. It is worth bearing in mind that the 40 wt.% PLA/jute biocomposite with woven jute fibers presents a slight increase in the Young's modulus and tensile strength with values of 3325.7 MPa and 53.2 MPa, respectively, these values being very close to those of neat PLA. The PLA/jute biocomposite containing 40 wt.% of jute fiber has a higher elongation at break, which makes the properties in general very attractive, since the integration of natural fibers allows to reduce the amount of PLA resulting in a material with balanced mechanical properties and reduced costs. These results are in agreement with those presented by Singh, Singh [64]. They reported a noticeable improvement in tensile strength (64 MPa by increasing the jute fiber loading in the composites. The best results were obtained in composites with 30 wt.% jute fibers. By increasing the percentage of jute fibers up to 50 wt.%, a clear increase in the material's stiffness is observed with a Young's modulus of 3779 MPa. Consequently, the brittleness also increases. Hence, the composite with 50 wt.% shows a clear decrease in the elongation at break and the tensile strength. This phenomenon is probably related with a deficient embedding of the fiber into the PLA matrix due to the reduction of the polymer proportion in this composition. This effect was also reported by Gunti et al. in PLA composites reinforced with short jute fibers previously subjected to alkali treatment [65].

The tensile properties of PLA/jute biocomposites reinforced with woven jute fibers are directly related to the direction of fibers (weft and warp). Therefore, if the applied load is aligned with one of these directions the material will have the best mechanical properties. Furthermore, the mechanical performance of PLA/jute biocomposites depends to a large extent on the quality of the interface between the reinforcing fiber and the surrounding matrix [66]. As expected, due to the random arrangement of the fibers in the nonwoven jute bio-composites, the resulting tensile properties are lower compared to biocomposites with woven jute fiber. Taking this into account, it is remarkable that both Young's modulus and tensile strength show a considerable reduction, reaching values of 1111.7 MPa and 14.7 MPa, respectively, when the composite is reinforced with 30 wt.% non-woven jute fiber. Despite increasing the percentage of jute fiber up to 40 or 50 wt.%, the reinforcing effect is not noticeable to any great extent as the modulus remains almost invariable with values around 1300 MPa. This may be because both the fibers and the matrix act as separate phases, *i.e.* poor load transfer between the matrix and the fiber. This effect can be attributed to several phenomena. On one hand, to the low affinity between the jute fibers and the PLA matrix due to their hydrophilic and hydrophobic nature [67], respectively. On the other hand, Khan, Shaikh [68] claims that composites with high reinforcement loading, do not allow full fiber embedding into the polymer matrix, resulting in a lack of bonding between the fibers and the matrix, which causes a decrease in the stiffness of the material due to poor stress distribution. Despite this, it may be observed that the tensile strength is positively influenced by the increase in fiber loading.

Regarding PLA/jute biocomposites with a fiber content of 50 wt.%, the σ_b value is 34.5 MPa, 134% higher than the PLA/jute biocomposite with a 30 wt.% non-woven jute fiber. It should be noted that the amount of non-woven fiber loading does not affect elongation at break, remaining at about 2.6%. As observed in the morphology analysis, the PLA/jute biocomposites manufactured with non-woven jute presented higher gap between the fiber and the surrounding PLA matrix, thus providing a reduction in the mechanical properties. As a result, an increase in fiber content does not provide an improvement in the elongation at break. Despite having relatively low tensile properties, PLA/jute biocomposites with non-woven jute mats exhibit mechanical properties similar to those of some commodity thermoplastics such as polypropylene (PP) [69], or high-density polyethylene (HDPE) [70], with the advantage that having natural fibers makes the resulting materials more attractive from an environmental point of view and production costs.

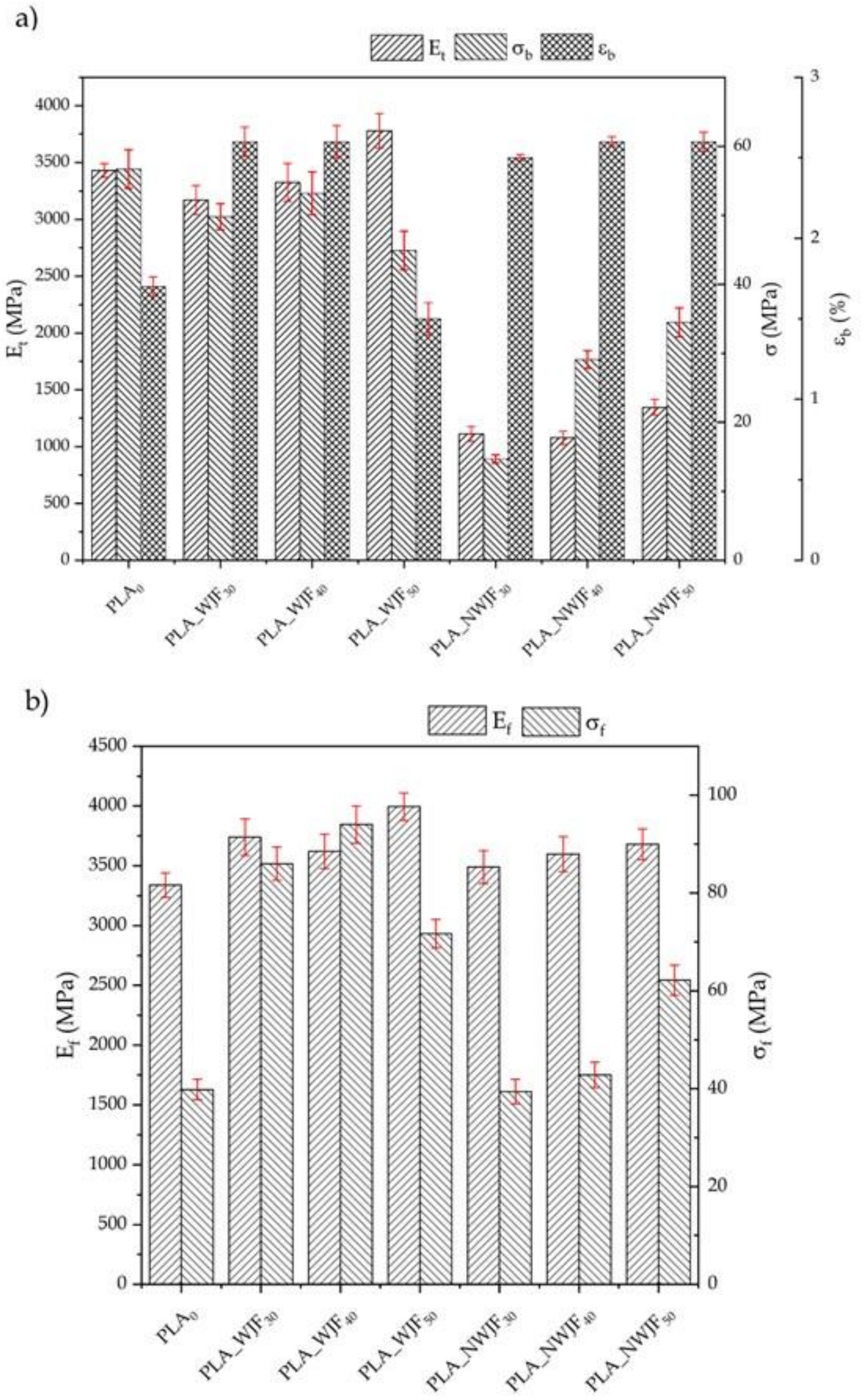

FIGURE 3. Mechanical properties of the PLA/Jute bio-composites a) tensile test properties: Young modulus (E_t), tensile strength (σ_b), elongation at break (ε_b) and b) flexural properties: flexural modulus (E_f), flexural strength (σ_f).

Regarding the flexural properties of the PLA/jute biocomposites reinforced with jute fabrics, it is possible to see that neat PLA has a flexural modulus and flexural strength of 3338.8 and 39.8 MPa, respectively, which are close to those reported by Dong, Ghataura [71] in PLA composites reinforced with coir fibers. They reported a flexural strength and flexural modulus values of 57 MPa and 2.80 GPa, respectively, in PLA composites with 20 wt.% short coir fibers. It can be clearly seen that both the flexural modulus and the flexural strength of PLA/jute biocomposites increase with the increase in the wt.% of jute fiber [72]. With regard to PLA/jute biocomposites with jute fabrics, the flexural strength and stiffness are generally provided by the external layers. In flexural conditions, the upper face is subjected to compressive stress, while the bottom face is mainly subjected to stretching (tension). Therefore, the flexural behaviour of the PLA/jute biocomposites depends on the strength of their external layers [73]. Biocomposites containing 30 wt.% of woven jute fiber, show an increase of 12% and 116% of the flexural modulus and flexural strength, respectively. These results show flexural conditions are less aggressive in terms of stress concentration phenomena as tensile conditions. By increasing the woven jute fiber load up to 50 wt.%, the flexural modulus rises to 3993.6 MPa, which corresponds to an improvement of about 20% compared to neat PLA, thus showing a clear improvement in flexural stiffness. Similarly, there is an increase in flexural strength, reaching values of 71.7 MPa. The configuration of the jute fibers on biocomposites, *i.e.* the fiber orientation, makes them capable of withstanding greater loads [74]. A maximum flexural strength of 94.0 MPa was obtained for PLA/jute biocomposite with 40 wt.% woven jute fiber, as reported previously with regard to tensile properties. Higher fiber content as that for PLA/jute biocomposites with 50 wt.% woven jute fiber, show a decrease in flexural properties due to poor fiber embedding.

As with woven fiber-reinforced composites, the flexural properties of PLA/jute biocomposites with non-woven jute mats are related to the amount of fiber loading [75], resulting in increased flexural properties by increasing the wt.% of fiber in PLA/jute biocomposites. It can be seen that biocomposites containing 50 wt.% non-woven jute mats, show flexural modulus and flexural strength values of 3679.8 and 62.2 MPa, respectively. The increase in flexural properties achieved by non-woven jute fibers is slightly lower than that observed for the woven jute fibers. This may be due to several factors, including the low interaction between the jute fibers and the PLA matrix [76], and the distribution of the fibers in the matrix [68]. Jawaid, Khalil [77] suggested that the random distribution of non-woven fibers in a polymer matrix causes a counterproductive effect on the flexural properties since having a smaller continuous surface makes the composite to have a lower load capacity. This effect that can be clearly seen in composites containing woven jute fibers, which are composed of long, perfectly aligned and continuous fibers, and this, results in better load capacity. Singh, Singh [64], have recently reported interesting results in jute/PLA bio-composites by using different processing temperatures ranging from 160 ºC to 180 ºC. The most relevant results that they provide indicate an increasing trend in mechanical properties up to 30 v/v.% jute fiber and above this, mechanical properties decrease. They attribute this to the weak fiber-matrix interface interactions as the PLA matrix content is reduced.

3.2 FIBER-MATRIX INTERACTION AND MORPHOLOGY ANALYSIS OF THE PLA/JUTE BIO-COMPOSITES

There are several factors with a direct influence of mechanical performance of composite materials. Among these, it is worthy to note the fiber distribution, fiber-matrix interaction, and directionality of the reinforcement fibers in the matrix. In general, the reinforcement fiber is stiffer than the polymer matrix and if there is good fiber-matrix interaction (the so-called isodeformation conditions), the stress transfer from the low stiffness component (the polymer matrix) to the high stiffness component (the reinforcing fiber) is allowed. In contrast, if the polymer-fiber interactions are weak, the applied stresses could not be appropriately transmitted from the matrix to the fiber, causing the premature failure of the material due to the lack of cohesion between the polymer matrix and the reinforcing fiber [78].

The fiber distribution and directionality also play a critical role in the mechanical performance of composite materials. If the load is applied in the fiber direction, an improvement in the strength and stiffness is generally obtained [74, 79]. **Figure 4** shows stereomicroscopy optical images of the cross-section of PLA/jute biocomposites with woven jute fibers (**Figure 4a, Figure 4c, and Figure 4e**), and PLA/jute biocomposites with non-woven jute fibers (**Figure 4b,**

Figure 4d, and Figure 4f). The alignment of the jute fibers in PLA/jute biocomposites with woven jute bio-composites is clearly observed with the typical wave shape. Unlike PLA/jute biocomposites reinforced with woven jute fibers, PLA/jute biocomposites with non-woven fibers show a non-uniform distribution of the fibers, which is characteristic of this type of material (random). Despite this, a rather good and homogeneous embedding can be observed in both cases between the different jute fibers and the PLA matrix.

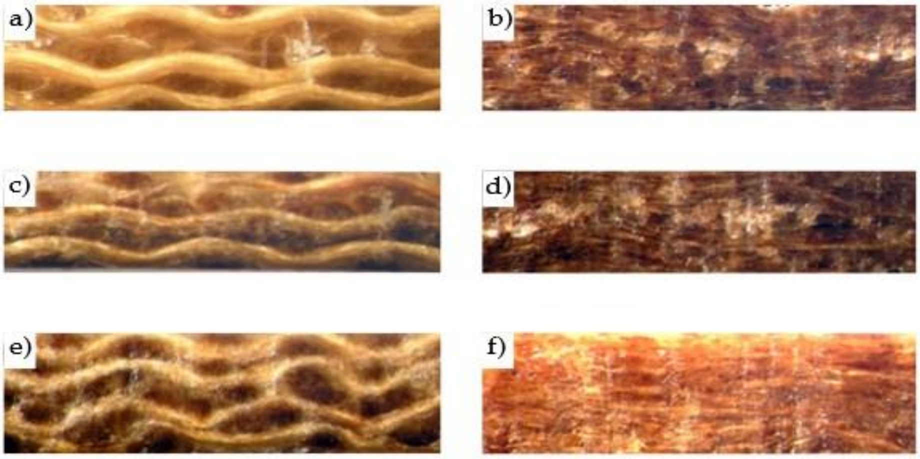

FIGURE 4 Stereomicroscopy optical images at magnification of 12.5× corresponding to the cross-section of PLA/jute biocomposites reinforced with woven jute fibers (left column) and PLA/jute biocomposites reinforced with non-woven jute fiber s (right column) of a)-b) 30 wt.%, c)-d) 40 wt.%, and e)-f) 50 wt.%.

Jute fiber is composed of aligned tubular cells which are responsible for its moderate mechanical properties and excellent insulation properties according to Fidelis, Pereira [80]. **Figure 5** shows typical scanning electron microscopy (SEM) images of the jute fiber morphology.

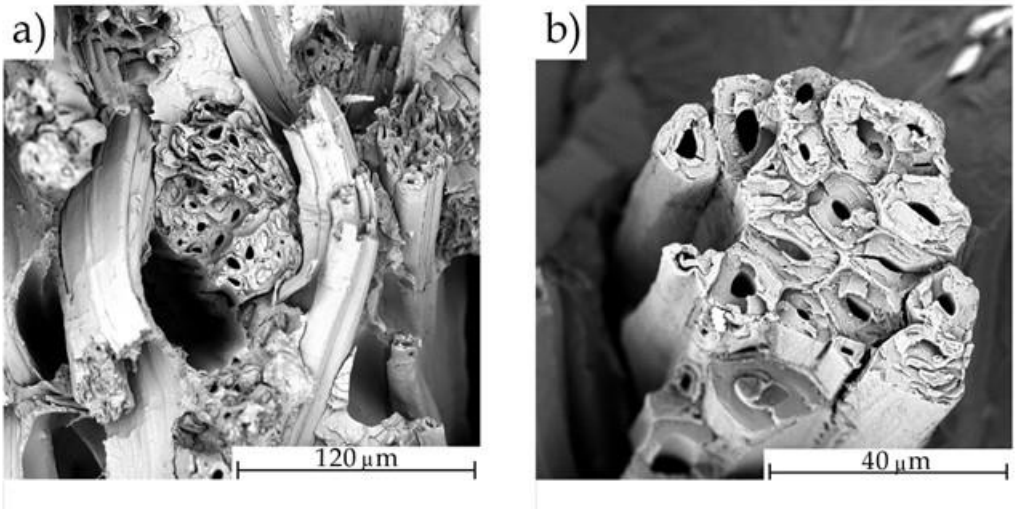

FIGURE 5 Scanning electron microscopy (SEM) images of the jute fiber at different magnifications, a) 1000×, (scale bar of 120 µm), and b) 3000× (scale bar of 40 µm).

To better understand the phenomenon of the interaction between the jute reinforcement fibers and the matrix, the fractured surfaces subjected to tensile tests were studied by scanning electron microscopy. As already mentioned, the good synergy between the polymer matrix and the reinforcing fiber, has a direct effect on mechanical performance. Strong polymer-fiber interactions positively contribute to a good load transfer, resulting in the desired reinforcing effect by fully embedding the fibers into the structure of biocomposites [65]. **Figures 6a** and **6c** refer to the PLA/jute biocomposites reinforced with woven jute fibers (40 wt.% and 50 wt.%). In these images, the directionality of the fibers can be clearly seen, as well as the presence of small gaps between the jute fibers and the surrounding PLA matrix. This is because the wettability of the matrix with the fibers is not good enough, resulting in weak polymer-fiber interactions. This is evidenced by some pulled-out fibers from the matrix [81]. No tear marks are visible on the fiber breakage surface, but a clean break of the fibers. This may be due to the low extensibility of the jute fibers to tensile stress, as suggested by Jawaid, Khalil [77]. The presence of a small gap between the fiber and the surrounding PLA matrix might be responsible for poor load transfer and, subsequently, non-optimum mechanical properties of PLA/jute biocomposites. The pull-out phenomenon can be clearly observed in **Figure 6a** and **Figure 6c** since the matrix shows a homogeneous surface that copies the shape of the pulled-out fiber. Under these conditions with a poor interaction, the failure mechanism for the composites is the debonding of the fibers from the matrix providing the morphology mentioned above as proposed by [23].

Figure 6b and **Figure 6d** show the fractured surface of PLA/jute biocomposites reinforced with non-woven jute fibers. In this type of biocomposites, the random fiber distribution is remarkable. The random alignment of the fibers can act as stress concentrators, causing premature separation of the fibers from the matrix and causing propagation of matrix microcracks, which leads to a decrease in the tensile properties. This may be one of the reasons why the gap between the fibers and the matrix is larger compared to woven jute fiber-reinforced biocomposites as suggested by Khan, Terano [54]. This morphology is in accordance with the low values obtained in tensile tests on PLA/jute biocomposites reinforced with non-woven jute fibers. Similar results were reported by Alavudeen, Rajini [81], in polyester composites reinforced with banana/kenaf fibers. They concluded that the tensile performance of the plain-woven composites was superior to that of the randomly oriented bio-composites for the same fiber content because of the discontinuity of the matrix caused inadequate stress transmission.

The low affinity of the reinforcement fibers and the PLA matrix is largely due to the nature of the PLA matrix and the natural fibers, as jute fibers that are mostly composed of cellulose and lignin. As a result, fibers have a marked hydrophilic character due to the hydroxyl group, and polyesters such as PLA have a strong hydrophobic nature. This difference is responsible for the low PLA-jute interactions. [52, 82]. **Figure 7** shows an example of the fracture surface of the samples. PLA matrix shows a smooth and homogeneous fracture surface, typical of a brittle fracture [44]. In addition, it was possible to observe the effects of pulled-out fibers due to the little affinity that these two components have.

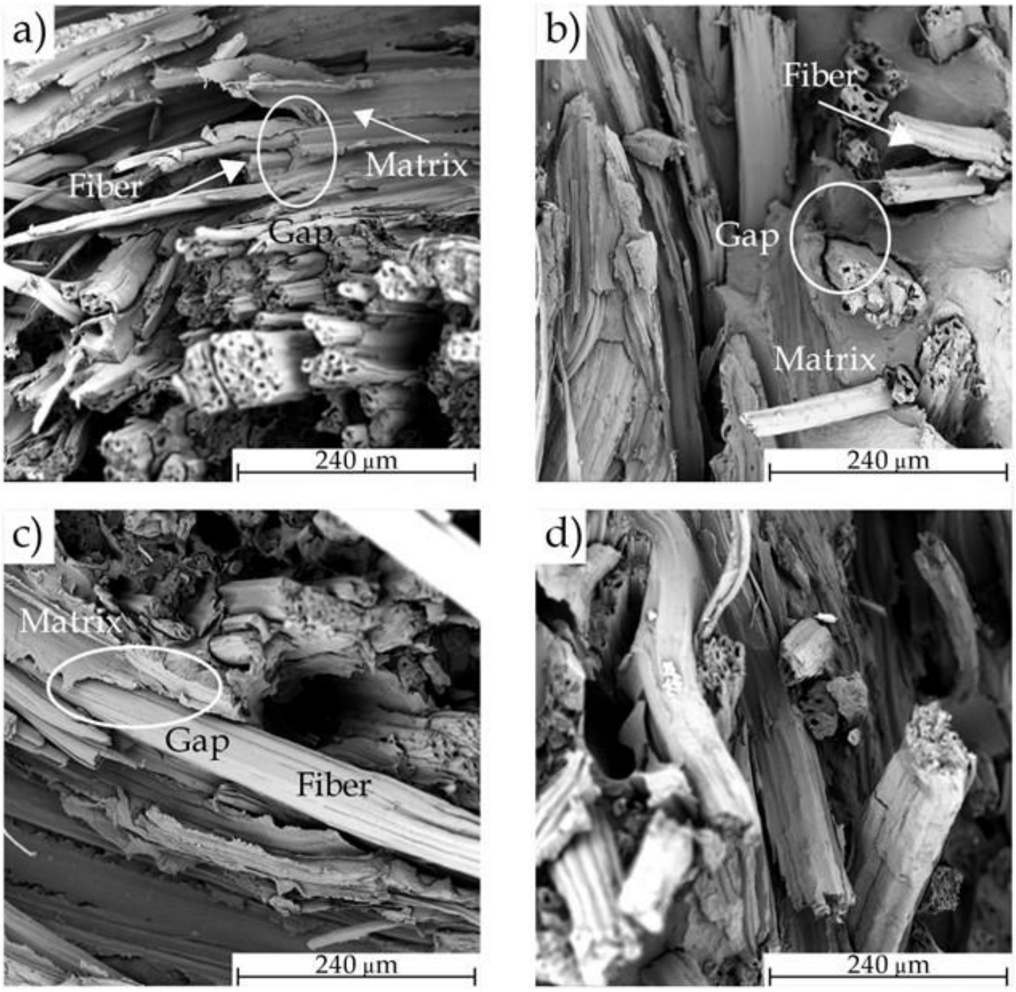

FIGURE 6 Scanning emission microscopy (SEM) images of the fracture surface of PLA/jute biocomposites with different percentages of woven jute fiber (left column) and non-woven jute fiber (right column): a-b) 40 wt.%; c-d) 50 wt.%. The images were taken at 500× (scale bar of 240 µm).

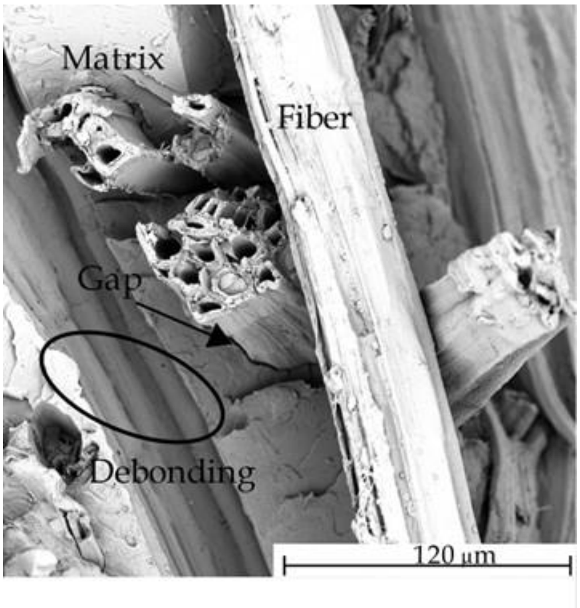

FIGURE 7 Scanning electron microscopy (SEM) image of the fracture surface of PLA/jute biocomposite containing 50 wt.% woven jute fiber. The image was taken at 1000× (scale bar of 120 µm).

4 CONCLUSIONS

1. The present work has confirmed the feasibility of manufacturing high environmentally friendly biocomposites of polylactide (PLA) and jute fiber reinforcements.

2.These bio-composites can be easily manufactured by thermocompression molding by directly stacking fabrics and PLA pellets. This stacking system provided enough wettability of the fibers with the matrix during the thermocompression process at 190 ºC, resulting in biocomposites with a high percentage of reinforcing fiber (up to 50 wt.%).

3. The morphology analysis by scanning electron microscopy (SEM) showed that the interaction between the jute fibers and the PLA matrix was relatively low. In spite of this, the interaction of the PLA matrix with the woven fibers was better, since the PLA-jute fiber gap was smaller than the one obtained in PLA/jute biocomposites with non-woven jute fibers.

4. With respect to the mechanical tensile properties under tensile conditions, better properties are obtained in PLA/jute composites with woven jute fibers, mainly due to the directionality of their fibers, this effect is more noticeable in the high Young's modulus obtained.

5. With regard to the flexural properties of PLA/jute biocomposites, these are not critically affected by the directionality of the reinforcement fibers, tending to increase with increasing fiber content.

6. These biocomposites fully obtained from renewable sources, offer balanced mechanical performance comparable to many conventional petroleum-based polymeric materials. Therefore, the partial or total use of these PLA/jute biocomposites in the different areas such as construction, automotive, wind energy, and furniture, among others would help to a sustainable development. In addition, the use of natural fibers allows a cost reduction, due to the lower percentage of polymer used.

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FIGURE CAPTIONS

FIGURE 1 Manufacturing process of PLA/jute bio- composites by hot compression moulding. a) placing the first PLA layer on the aluminum mould; b) placing the last PLA layer on top of the jute fiber layer; c) schematic representation of the stacking sequence of PLA pellets and jute fibers.

FIGURE 2 Images of the woven jute fabric (left column) and the non-woven jute fabric (right column). a)- b) before hot compression moulding and c)-d) after hot compression moulding.

FIGURE 3 Mechanical properties of the PLA/Jute bio-composites a) tensile test properties: Young modulus (E_t), tensile strength (σ_b), elongation at break (ε_b) and b) flexural properties: flexural modulus (E_f), flexural strength (σ_f).

FIGURE 4 Stereomicroscopy images at magnification of 12.5× corresponding to the cross section of the woven jute fiber green composite (left column) and non-woven jute fiber green composite (right column) of a)-b) 30 wt.%, c)-d) 40 wt.%, and e)-f) 50 wt.%.

FIGURE 5 SEM images of the jute fiber used: (a) 1000×, with a marked scale of 120 µm. (b) 3000×, with a marked scale of 40 µm.

FIGURE 6 Scanning emission microscopy (SEM) images of the fracture surface of PLA-based bio-composites with different percentages of woven jute fiber (left column) and non-woven jute fiber (right column): (a) and (b) 40 wt%; (c) and (d) 50 wt%. The images were taken at 500× with a marked scale of 240 µm.

FIGURE 7 Scanning emission microscopy (SEM) image of the fracture surface of PLA-based bio-composites (i.e. 50 wt% woven jute fiber). The image was taken at 1000× with a marked scale of 120 µm.

TABLE CAPTIONS

TABLE 1 Composition and code designation of the different PLA/jute bio- composites.

TABLE 2 Tensile and flexural properties of the PLA/Jute bio-composites.