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Green composites based on *Atriplex halimus* fibers and PLA matrix

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Abstract: This work focuses on the potential use of cellulose fibers extracted from Mediterranean saltbush (*Atriplex halimus*) as a filler in polymeric matrix. The fully biodegradable composites were prepared from PLA as matrix and micro-cellulose fibers ranging from 0 to 15 wt%. The influence of the fiber content on the structure, mechanical, thermal and water absorption properties was evaluated. Mechanical results indicated that fibers acted effectively as reinforcement, increasing the tensile strength and the Young's modulus of PLA by 25% and 45 %, respectively. This is due to the good stress transfer between fibers and matrix through the strong interactions that have been evidenced by FTIR spectroscopy. The thermogravimetric analysis showed that PLA composites have a slightly lower degradation temperatures than the pure PLA but they still have favourable thermal stability. Water absorption measurements and biodegradability tests showed that the addition addition of fibers accelerates degradation kinetics and confirm that the prepared composites are an environmentally safe material suited for different applications.

Keywords: biodegradability; cellulose fibers; mechanical properties; PLA polymer; thermal properties.

1 Introduction

The uncertainty of petroleum supply and environmental pollutions caused by non-degradable plastics have attracted world concerns over developing environmentally friendly materials from natural and renewable resources [1].

Poly(lactic acid) is one of the most studied biodegradable polymers mainly because of its relatively high melting point, crystallinity and stiffness compared to other biopolymers. Currently, the majority of this linear aliphatic thermoplastic polyester is produced by polymerization of lactide, a cyclic dimer derived from lactic acid, which in turn is obtained by the fermentation of agricultural products such as corn, potato, cane sugar, and rice. In spite of its high biocompatibility and biodegradability, which make it a very good candidate for biomedical and packaging uses, a large number of its applications is still limited by its high production cost and brittleness [2],[3].

However, to achieve the performance needed for many engineering applications, the pure PLA is generally reinforced by different type of fibers or fillers. Recently growing interest in the use of natural/bio-fibers as reinforcements or fillers for biodegradable polymers is noticed, thus creating a new class of materials known as fully "biobased composites" [4].

These natural fibers present many advantages compared to synthetic ones, such as natural availability, low cost, low density, good mechanical properties, eco-friendly and bio-degradability characteristics [5],[6]. They have even the functional capability to substitute or to compete glass fibers by having a higher specific strength and a similar specific modulus [5],[7]. The most common natural plants used in applications are bast fibers, such as hemp, jute, flax, kenaf, and sisal or agricultural crop residues such as oil palm, pineapple leaf, banana, and sugar palm. They can be obtained in abundance, low cost, and they are also renewable sources of biomass [5],[8].

Several research papers deal with the optimization of natural and man-made cellulose fibre-reinforced PLA composites. Some investigate biodegradability, others look for ways to improve temperature resistance or the composites' mechanical characteristics [9]. In this study we have investigated the development of an environmental-friendly composite material prepared from polylactic acid (PLA) and cellulose fibers extracted from Mediterranean saltbush (*Atriplex halimus*) plant. This plant, commonly used as feed for animals, grows in the dry regions of North Africa and belongs to the Chenopodiaceae family [10]. The structure, mechanical and thermal properties of the prepared composites were determined by, FTIR spectroscopy, mechanical tensile tests, and thermogravimetric analysis. Biodegradability and permeability were also investigated.

2 Materials and methods

2.1 Materials

2.1.1 Matrix

Poly (lactic acid) (PLA), film grade was supplied by UMET laboratory (University of Lille 1, France). This biopolymer has a number-average molecular weight (M_n) of 94700 g/mol, weight-average molecular weight (M_w) of 179600 g/mol (determined by SEC analysis) and a density of 1.24 g/cm³.

2.2.1 Reinforcement

Cellulose microfibers were extracted from a saltbush (*Atriplex halimus*) plant (Figure 1), the details of the process have been reported earlier by us [10]. We obtained two sizes of cellulose fibers: short fibers (SF: length 100 μ m, diameter 80 μ m, aspect ratio 1.25) and long fibers (LF: length 1938 μ m, diameter 430 μ m, aspect ratio 4.5). The average length of the fibers was evaluated by microscopic observation. The chemical composition of the fibers was also estimated after various stages of chemical treatment using standard test methods (ASTM D1103-60 and ASTM D1104-56).

2.2.2 Chemicals

Dichloromethane (CH₂Cl₂) has been used to dissolve PLA and all reagents were supplied by Sigma Aldrich (France).

2.2 Composites processing

Solution-cast film samples of the biodegradable materials with 0, 5, 10 and 15 wt% fiber contents were prepared with a dry film thickness of around 100 μ m and using dichloromethane as a solvent referring to many researchers [11]. 2 g of PLA was dissolved in 80 ml of dichloromethane CH₂Cl₂ and stirred without heating for 30 min until complete dissolution of PLA, then proper amounts of the cellulose fibers were added and stirred for a further 3 min. Subsequently, the mixture was cast onto Petri dishes to generate films after solvent evaporation at room temperature conditions.

The following terms (PLA) and (PLA-SF5,10,15),(PLA-LF5,10,15) are used to define the non-reinforced PLA film, and PLA composites containing short fibers (SF) and long fibers (LF) 5,10,15 % wt/wt of fibers to matrix.

2.3 Characterization^[u2]

In this work, an Olympus BX41 optical light microscope with a capture camera (Micro Publisher 3.3 RTV) was used to examine the distribution of fibers in the matrix for both sides and to define the dimensions of the cellulose fibers.

Tensile tests were performed on a VERSA Test AFG100N tensile testing machine at a cross-head speed of 14mm/min. The test bars had a rectangular cross-section with dimensions of 6 mm×24 mm prepared by using a mold and press machine. The data were averaged over five to eight specimens.

Thermogravimetric analysis (TGA) was performed using an ATG PYRIS 1 Perkin Elmer with specimen size of 5 to 10 mg. The isothermal tests were carried from 40 to 600°C at a heating rate of 20°C /min in the presence of nitrogen.

FT-IR measurements for different films were performed using a Perkin Elmer spectrometer ranging from 4000 to 400 cm⁻¹ to identify different types of chemical bonds existing. The powdered samples and KBr (2 mg sample per 150 mg KBr) were pressed into a disk for FT-IR measurement.

Biodegradability of neat PLA and PLA/cellulose composites was evaluated by burying the samples in soil and measuring the weight loss, the soil media used was a mixture of organic hummus and sand. The samples were cut into 30x 30 mm squares, oven dried at 80°C to constant weight, weighted for the first time and each sample was placed in vessel containing soil. Exposure to the soil environment continued for 10 months, at predetermined time intervals, samples were taken out from soil, carefully washed with water and dried for weighing and returned again to the soil.

Water absorption of the PLA composites was also determined by placing different samples in an aqueous media. The rate (%) of absorbed water was calculated by:

$$\text{Water absorption (\%)} = \frac{m_f - m_i}{m_i} \times 100$$

where W_i is the weight of dried sample before immersion and W_f represents the weight of dried material after water immersion.

3 Results and discussion

3.1 Morphology

The aim of microscopic examination was essentially to investigate the influence of fiber microstructure on mechanical properties. The micrographs show that generally a

relatively good dispersion was achieved in composites with low fiber contents even fibers were randomly incorporated.

Figure 2A representing the PLA matrix shows a transparent film with a smooth surface at the exception of the presence of some air bubbles formed after solvent evaporation. The addition of fibers don't do not really affect optical properties of composites such as transparency which remained virtually unmodified compared to the neat PLA [11].

Figure 2B and C shows clear images of the cellulose fibers embedded in the biodegradable matrix, especially short fibers (SF) which were highly more homogeneous than the long fibers (LF). However, a phenomenon of agglomeration of fibers is observed in the formulations containing 15% of long fibers. This is probably due to the limited amount of polymer which is not sufficient to cover the full surface of the fibers [11],[12].

3.2. Mechanical analysis[u3]

Typical stress–strain curves of neat PLA and PLA composites with different fiber contents are shown in Figure 3. A sensitive growing on the tensile properties of composites was noticed with the progressive increments of cellulose fibers.

The modulus of the PLA was increased from 2.4 GPa to 3.1 GPa and 3.5 GPa with the addition of 15 wt% of short fibers and long fibers respectively, which corresponds to a 26% and 45% increase. Similarly for tensile strength, an increase of 40% and 50% was observed for PLA-LF15 and PLA-SF15 composites compared to pure PLA. In contrast, a sensitive decrease in the elongation at break from 15% to 10% for SF and to 6% for LF was noticed . These results correspond with many studies where generally we notice an improvement in mechanical properties of composites reinforced by cellulosic fibers or microcrystalline cellulose [13],[14] , [15],[16].

Higher tensile elastic modulus refers to the rigid property of material that requires more stress to produce the elastic deformation when tensile force is applied [17]. The increase of elastic modulus could be attributed to strong intermolecular force between cellulose fibers and PLA molecules, consequently cellulose fibers particles restricted mobility of PLA polymer chain and resulted in enhancing the stiffness of the composite material [18],[19].

The positive impact of the cellulose fibers essentially in the composite strength showed that the *Atriplex halimus* fibers have good mechanical properties and that there are strong interactions between the PLA and the cellulose fibers [12]. However, the large standard deviations reported in Table 1 on the modulus and elongation at break values reported in Table 1 indicated that the fibers, especially long ones, were not homogeneously distributed.

Remarkably, the mechanical response of composites reinforced with SF is markedly superior than to composites with LF essentially due to their small size. This can confirm the proposal of Huda [4], Osswald [20] who suggested that there is a critical fiber length[u4]; if this length is not required the matrix cannot effectively grip the fiber to take the strain, and the fibers will slip and be pulled out, instead of being broken under tension.

Moreover, Kamonwan [17] indicated that the filler size could have affected the stress distribution between the matrix and filler thus the better mixing or dispersion process of PLA/fibers composites needs to accomplish.

3.3. Thermal properties

TGA characterization allows to understand changes in the physical and chemical properties of composites measured as a function of increasing temperature through weight-loss. The results of this test can help to qualify composites for thermally sensitive applications.

The thermal stability of pure PLA and fiber-reinforced PLA composites was investigated with TGA. Thermogravimetric curves of *Atriplex halimus* fibers, neat PLA and PLA / fibers composites are shown in Figures 6 and 7, respectively.

It is reported that PLA decomposes between 285 and 368°C, releasing cyclic oligomers, lactide molecules, CO and CO₂ [14],[16]. The TG curve In our case of neat PLA shows a single step thermal degradation at 392°C.

In case of *Atriplex halimus* fibers, three-stage weight losses were observed; a first weight loss between 50°C and 130°C that can be ascribed to moisture loss, Second stage with a small hump in the temperature range from 250 to 300°C is characteristic of low molecular weight components, such as hemicelluloses. An other weight loss stage appearing at 358°C, is corresponded to the thermal degradation of cellulose and Final weight loss stage above 400°C is probably due to lignin decomposition suggested by other researchers at 450°C [16],[21].

For PLA/ fiber composites, the incorporation of *Atriplex halimus* fibers in PLA matrix has affected the thermal degradation temperature. The different composites showed a lower degradation temperature (less than 45°C) than that of PLA. Two-stage loss of mass was mainly observed for all composites; in the range from of 300°C to 400°C due to PLA depolymerization and a second at 350°C corresponding to the degradation of cellulose fibers. The thermal stability of composites decreased, which indicates that the distribution of fibers into PLA matrix is not regular with some agglomerations.

We can say that whatever the rate or the type of fiber, the maximum degradation temperatures of the composites is still around 365°C. Compared to the temperatures recorded by other studies varying between 320 à and 340°C, these values are very acceptable.

These lower degradation temperatures may be attributed to the decrease of molecular weight of PLA and sometimes to the low of affinity between the fibers/matrix [22],[23],[24].

3.4. FT-IR analysis

Typical FT-IR spectra of the PLA and natural fiber reinforced PLA composites are presented in Figure 8. The spectrum of *Atriplex halimus* fibers is also represented for comparison.

The principal absorbance peaks obtained for the PLA are as follows: the peak around 3512 cm^{-1} was attributed to the hydroxyl (OH) stretching, the strong peaks at 2996 and 2943 were assigned to the asymmetric and symmetric mode of C-H stretching, respectively. The C=O stretching (from ester linkage) was observed at 1760 cm^{-1} , the peak at 1457 cm^{-1} was characterised by the CH_3 band, the C-H deformation appeared at 1383 cm^{-1} . the O-C asymmetric mode of the ester groups was observed at 1088 cm^{-1} , and the peaks appeared at 957 and 913 cm^{-1} were attributed to the rocking mode of CH_3 . Also the peaks at 864 and 753 cm^{-1} , can be referred to the amorphous and crystalline phases of the PLA, respectively [2],[24].

The spectral bands for *Atriplex halimus* fibers are at 3403 cm^{-1} (-OH intermolecular hydrogen bonds) related to the presence of cellulose and hemicellulose, 2929 cm^{-1} (C-H stretching) present in hemicellulose component, 1639 cm^{-1} (frequency of deformation vibration of CH_2 and CH groups are located), 1371 cm^{-1} (C-H band), 1318 cm^{-1} (CH bending mode), 1281 cm^{-1} (C-O stretching mode of ester group) stretch of the acetyl group in lignins, 1110 cm^{-1} (C-O-C ether band), and 1051 cm^{-1} (C-O-C stretching vibration). Previous studies have confirmed these trends the spectral of cellulosic materials [14],[16],[17].

The spectra of the fiber reinforced PLA composites have similar features to those of Pure PLA except for some changes due to the interaction with cellulose fibers; the OH stretching in the PLA/Fibers composite that appeared at 3437 cm^{-1} , was found to be different to that of the fibres (3496 cm^{-1}) and the PLA (3512 cm^{-1}). This indicates that in the PLA/Fibers composites, the OH groups (from fibres) formed hydrogen bonds with the carbonyl groups (C=O) of PLA as shown in Figure 8. A similar observation has also been reported for the PLA / rice starch composites by other researchers [25],[26]. In addition, it was also evident that the intensity of C=O (1756 cm^{-1}) stretching in the PLA / Fibers composites was significantly higher than that of the PLA.

This was due to the esterification between OH groups of *Atriplex* fibres and terminal carboxylic acid groups (COOH) of PLA. This agrees with the work reported by Semba et al [27] who showed that the peak intensity of the C=O group increased due to the chemical reaction between the dicumyl peroxide and the PLA in a blend of PLA/polycaprolactone.

3.5. Water absorption

Figure 9 shows the percentage moisture absorption of the pure PLA and PLA/ fibers composites as a function of time of immersion at room temperature. The curve indicates that the water uptake behaviour of PLA composites obeys Fick's law [28], where a rapid absorption was observed within the first days of immersion then slows down to reach the equilibrium after 35 days of immersion.

Water absorption is generally attributed to several factors; firstly, to the presence of some microgaps or defects between the polymer chains. ; sSecondly, to the capillary

processes through the gaps and flaws at the interface between cellulosic fibers and the PLA matrix. ; Thirdly thirdly to micro-cracks in the matrix formed during processing [16].

In our case this rapid water absorption [u5] at the beginning as water molecules penetrate into the fibers structure, mainly into capillaries as well as spaces between the fibers particles and the matrix.

The addition of long and short cellulose fibers increased the amount of absorbed water; from 0.9% for neat PLA and 1.6% for composites containing 15 wt% of LF. This can be attributed to the hydrophilic nature of the cellulose fibers and to the multiple hydroxyl groups which are available for interaction with the water molecules [25], [29].

3.6 Biodegradability

The degradation of PLA is generally made by hydrolysis process which depends essentially on the permeability, the composition of the polymer and the microbial activity of soil burial. As shown in Figure 9 the samples exhibited a slow weight loss in the first 20 days of soil burial, this period represents the time required for the hydrolysis step and which is confirmed by the water absorption results.

After 3 months of incubation many changes in color and shape were observed, then the sample became fragile and brittle at the end of six months. After 9 months they were already in pieces such that only a few parts could be handled. We can say that the necessary period for total degradation of PLA by soil burial is estimated at 10 months [24], [39].

PLA/fibers showed a higher weight loss than pure PLA with a rate of 92% and 90% for composites containing 15 wt% of LF and SF due to the fact the cellulose fibers are more hydrophilic than the PLA, which accelerates the hydrolysis process. Similar results were reported for PVA/coconut-shell-powder composites [30].

4 Conclusions

The incorporation of cellulose fibers in a polymeric matrix is an interesting alternative to develop new materials with good mechanical properties and weak environmental impact. In this context we have investigated the use of new cellulose fibers extracted from *Atriplex halimus* plant as filler for PLA matrix.

The visual and morphological study of PLA composites showed that a relatively good dispersion was achieved except for some aggregates in high content of long fibers. That is why the short fibers were more effective than the long ones by giving better mechanical properties; with a fiber content of 15 % wt of short fibers the tensile strength and the young Young's modulus were raised by 25 % and 45 %, respectively, while the elongation at break was sensibly reduced.

FTIR spectroscopy evidenced the changes in the structure of PLA by adding cellulose fibers and indicated the existence of interaction between (OH) groups of fibers and carboxylic group (COOH) present in PLA.

Furthermore, the addition of the fibers has weakened the thermal stability and the water resistance of pure PLA by the fact that the cellulose fibers are more hydrophilic.

Biodegradation tests showed that the incorporation of fibers accelerate the kinetics of degradation, but the most suitable method for PLA composites at the end of their cycle of life is composting.

Finally, the prepared composites have potential for considerable growth with a green concept. Primary applications for these biocomposites include, for example, toys for children, furniture, hardware for electronic products, or one-way disposable products, and so on.

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((captions))

Figure 1: *Atriplex halimus* plant and optic micrographs of long fibers (LF)/short fibers (SF).

Figure 2: Optic micrographs of: (A) pure PLA, (B) PLA-SF10% biocomposite, (c) PLA-LF10% biocomposites.

Figure 3: The stress-strain curves for PLA composites (A) containing short fibers and (B) long fibers.

Figure 4: The effect of long fibers content on the mechanical properties of PLA composites.

Figure 5: The effect of the short fibers content on the mechanical properties of PLA composites.

Figure 6 TGA (a) and DTG (b) scans for PLA composites containing 0, 5, 10, 15 % wt of short fibers.

Figure 7: TGA (a) and DTG (b) scans for PLA composites containing 0, 5, 10, 15 % wt of short fibers.

Figure 8: (a) FT-IR spectra for cellulose fibers, non-reinforced PLA and composites, (b): possible interactions between PLA and cellulose fibers.

Figure 9: Weight loss curves of pure PLA, and PLA composites containing 15 % wt of short and long fibers and water absorption as function of storage time, type and the content of fiber.

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