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# Processing and properties of flax fibers reinforced PLA/PBS biocomposites

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## Abstract

Reinforced plastic materials are widely used in high sophisticated applications. Fiber length has a strong impact on the mechanical properties of composite materials. Being able to reliably and accurately evaluate the residual fiber length distribution of a sample is the key to understand the mechanisms of fiber breakage through experimental investigations, permitting the prediction of properties for flax fiber reinforced components. To optimize the properties of the composites, the greatest average length is sought. The objective is therefore to investigate the effect of the mixing process (single and twin screw extrusion) and the injection shaping process on flax fibers reinforced composites.

However, the subsequent image analysis is frequently performed automatically using the fiber length measurement system "FASEP" that can be used to analyze fiber length distributions automatically. After separating the fibers out of the composite material without any damage, and preparing them, statistical analysis can be done. After image processing and analysis with mathematical methods, a complete investigation of the fiber length distribution could be determined. The main results show that a greater concentration of shorter flax fibers was found in the twin screw extruded composite with more than 16 % of particles are smaller than 180  $\mu\text{m}$  when compared against the single screw extruded material. However, volume average results of flax fibers are higher for composite manufactured with single screw extrusion. The control of fiber breakage and final morphology is studied by Scanning Electron Microscopy. As a result of the injection step, the lengths of the fibers have been reduced, compared to the dimensions after extrusion. No reliable correlation between fiber length distribution and mechanical properties, measured with tensile test was found. Also, there is no significant effect of the melt processing types on the tensile properties considering the experimental errors.

**Keywords:** *Biocomposites; PLA/PBS blends; Flax fibers; Fiber length distribution; Mechanical properties.*

## 1. Introduction

Combining polymeric matrices and fibrous materials has been an established process for several decades and has expanded the use of thermoplastics into various high grade applications such as the automotive, aerospace, and wind industries. By integrating reinforcing fibers into a thermoplastic it is possible to obtain an enhanced and new composite which can be tailored to its application. This has led to high production demands of fiber reinforced plastic composite materials for its substitution of a number of traditional materials [1,2].

Vegetable fibers can be used as a reinforcement for polymers such as PLA, PVC, PE or PP in substitution of synthetic fibers (glass, kevlar, carbon...) in some construction materials. The incorporation of vegetable fibers (wood, flax, hemp) in thermoplastic or thermosetting materials as a replacement for glass fibers is a concept already commercialized [3,4].

The flow generated during mixing and the way the composite is shaped also play a prominent role and yet are less studied. Mixing operations determine the dispersion and distribution of the fibers in the matrix. The fibers can be mixed with the thermoplastic polymer using different processes such as single or double screw extrusion. This first mixing operation is called compounding and is usually followed by shaping operations of the finished part (injection molding).

During compounding, the flow generates high stresses and tends to separate the assembled fibers into bundles, which is a good thing. But it also tends to break the fibers lengthwise, causing them to lose their reinforcing action. At the same time, a good distributive flow is essential to obtain homogeneous final properties [5]. A compromise must therefore be found via the mixing processes and the fibers length distribution to dissociate the fibers without breaking them too much.

This is the key point on which mixing will have a major impact: the final size of the reinforcement in the composite. It is on this parameter that the coming work will focus. The control of fiber breakage is less studied in the literature, especially for cellulosic fibers. The emergence of thermoplastic-cellulosic fiber composites is relatively recent. However, the minimization of fiber breakage remains one of the last means of action to improve the final properties of the composites. This aspect remains important to consider.

The main objective of this work is to understand how the manufacturing process directly affects the length of the flax fibers reinforced poly-(lactic acid) (PLA) and poly-(butylene succinate) (PBS) with 80PLA/20PBS formulation. Blend based on poly (lactic acid) (PLA) and poly (butylene succinate) (PBS) matrix as a fully biodegradable polymers were retained in this work. It was found that the PBS content in the PLA/PBS blend should not exceed 20% (w/w) [6]. So, one can conclude that up to the suitable ratio 20% (w/w), these two biodegradable polymers are miscible and the PBS phase can be well-dispersed in the PLA matrix phase. Based on the results of some authors, after a mechanical [7], morphological [8], and thermal [9] characterization of PLA/PBS mixtures with different PBS contents: 10, 15 and 20% by weight with respect to PLA, 80% PLA mixed with 20% PBS appears to be the best formulation [10,11] that guarantees a valid compromise to maintain and modulate the thermal and mechanical properties of PLA/PBS mixture as well as its topography [12]. It is also necessary to point out that the transfer of stress from the matrix to the fibers, becomes less efficient with a fiber content higher than 20% [5]. In addition, we could not operate with mixtures in fiber content higher than 20% on the extruder because beyond this value there is a risk of not obtaining a homogeneous and good quality extruded material. This experimental investigation will attempt to evaluate the influence of the single and twin screw extrusion and of an injection molding on fiber length distribution. This is in order to broaden their field of applications and/or to bring out new applications.

Currently, there is a lack of knowledge on the behavior of cellulosic fibers during the compounding and injection phases with thermoplastic matrices. Moreover, the influence of mixing/forming processes on the properties of composites is not always studied in a precise and systematic way. This is the main motivation of our current work which is devoted to the study and comparison of the behavior and breakage of flax plant fibers during their compounding with an 80PLA/20PBS matrix by single screw extrusion, twin screw extrusion and after its injection molding. To better examine the dispersion of the fibers in the Poly (Lactic Acid) / Poly (Butylene Succinate) blend for the various processes carried out, the morphology of the reinforcements was studied after each stage of the processing using Scanning Electron Microscopy. The idea is then to link the state of the fibers in the composite to the mechanical properties as a secondary objective. Another originality of this work results in the developing of the technique of the fiber length measurement system "FASEP" that can be used to analyze fiber length distributions automatically in cellulosic flax fibers which make it possible to observe in direct the lengths of the fibers after each elaboration process.

## **2. Materials and Experimental techniques**

### *2.1. Materials*

Flax fibers provided by Apply Carbon (Languidic, France), are used as reinforcements. Two biodegradable thermoplastics commonly studied in the literature [10-16] are chosen for this study: PLA (PLA7001D provided by NatureWorks) and PBS (BioPBSTM FZ71PM provided by Mitsubishi Chemical Europe GmbH). Initial fibers of the same size (4 mm), were used in this study. Before the elaboration of the films, the polymers and the flax fibers are steamed under a vacuum at a temperature of 40°C during 48h.

### *2.2. Composite processing*

#### *2.2.1. Single screw extrusion*

The polymers are extruded with a Labstation Plasticorder Brabender single screw extruder to obtain the film. The processing parameters of the films are summarized in Table 1 [10].

Table 1. Film processing parameters [10].

Sample	Speed	Temperature	Belt speed (degree)
PLA	25 rpm	180°C	5
PBS	9 rpm	180°C	4
80 PLA / 20 PBS	25 rpm	180°C	6
PLA/PBS/Flax	25 rpm	180°C	10

### 2.2.2. Twin screw extrusion

Compounding of the composites was performed in a Brabender TECHNOLOGIE twin-screw extruder. Twin-screw extrusion is one of the most widely used processes for the preparation of thermoplastic composites. It is a continuous process that melts the material and transforms the materials through two parallel screws rotating in a barrel. The various extrusion parameters identified for implementation in this study are: Screw speed (30 rpm), Temperature profile of the sleeve (180°C) and Flow rate (2 kg/h) [10]. The round profile is cut into granules by a Meccanoplastica granulator.

### 2.2.3. Injection molding of specimens

The pellets previously obtained by extrusion are used to produce the required specimen samples in a BATTENFELD injection press. The process used to shape composites for tensile testing consists in injecting tensile specimens (according to ISO 527-2 1BA, Fig.1) [10]. Injection requires control of various parameters, such as Mussel temperature (25°C), Holding time (9.6 s), Injection pressure (1600 bar), Material temperature: (180°C), Closing force of the mold (800 kN), Injection time (0.25 s), Cycle time (54 s).



Figure 1. Healthy specimens obtained for the characterization of the studied material.

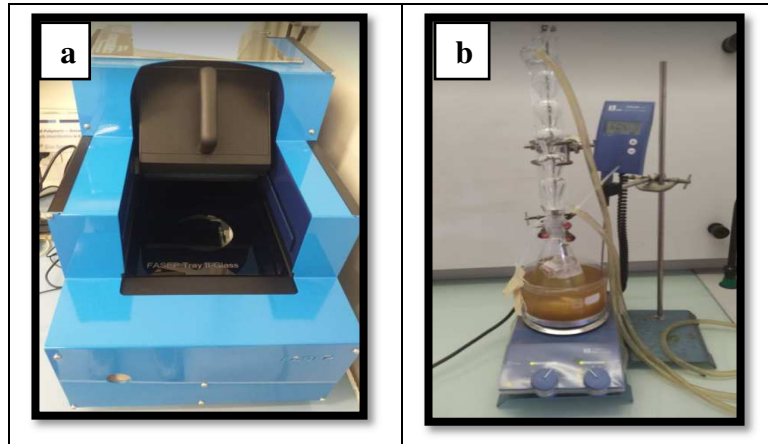
## 2.3. Tensile test and Fracture surfaces

The samples were tested on an MTS Synergy tensile machine 1000 RT. The samples were tensile loaded at a rate of 10 mm/min and the forces were measured with a 10N load cell. The deformation is measured by an MTS extensometer with distance between clamps of 25 mm. Thus, the stresses, strains and average Young's modulus of different specimens could be determined on a population of 5 specimens. The formulation has a dumbbell geometry according to ISO 527. The Young's modulus is calculated between 0.05% and 0.5% strain.

### 2.4. Fiber Length Measurement: Study of fiber length distribution

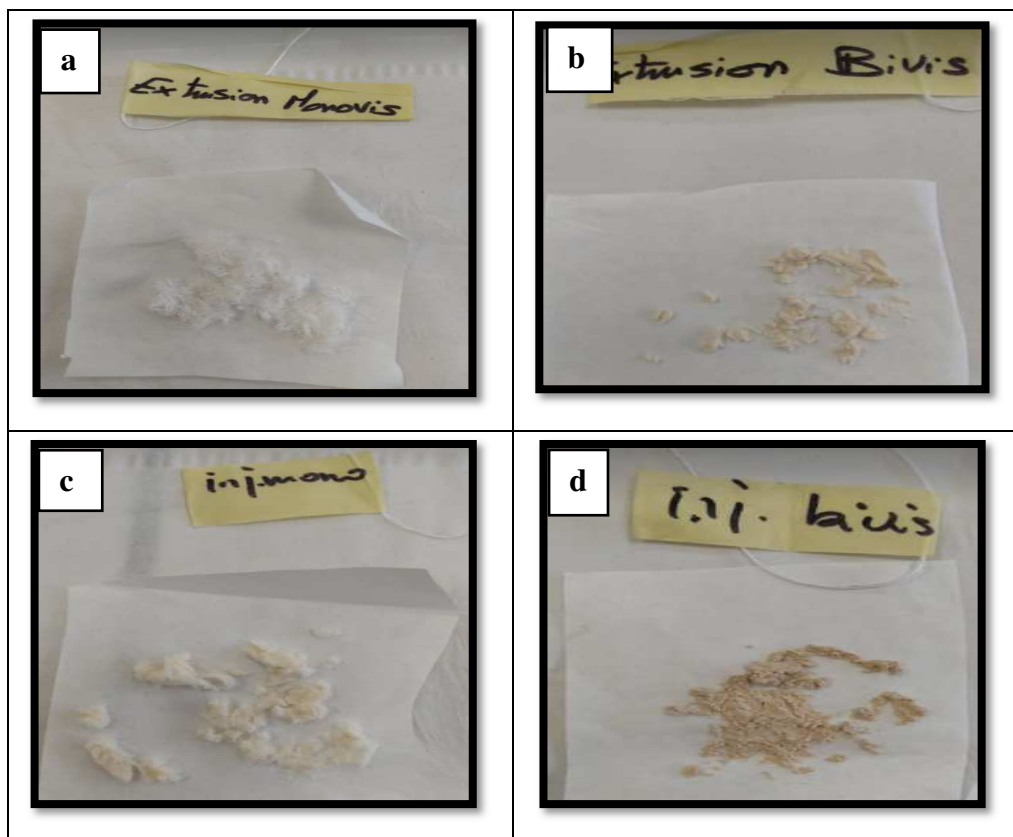
#### 2.4.1. Matrix Removal: Fiber extraction after compounding and injection

In the following, we present the analysis of the fiber size distribution after incorporation of flax fibers in the 80PLA/20PBS matrix by single-screw / twin-screw extrusion and after injection of the materials presented at the beginning of this section. In order to obtain fiber length measurements, flax fibers have to be separated from the polymer matrix they were encased in. The analyses were performed by dissolving the PLA and PBS matrix with chloroform at 66°C under magnetic stirring in an oil bath for 72 hours (Fig.2).



**Figure 2.** Real photos of (a) FASEP scanner and (b) polymer dissolution protocol.

The specimens are then left in a furnace at 50°C for 24 hours enabling the complete removal of the polymer matrix, leaving behind an entangled mass of short and long fibers which are ready to be scanned (**Fig.2**). The observations of the fibers were obtained by a FASEP scanner (**Fig.3**), the professional system for fiber length analysis works according to ISO 22314. The 'FASEP' fiber length measurement system can be used to analyze fiber length distributions automatically. The system is equipped with a high resolution scanner, a dark-field illumination box and an image analysis software. The image analysis makes it possible to measure straight, curved and crossing fibers over a range of fiber lengths.



**Fig. 3.** Extracted fibers: (a) after single screw extrusion (b) after twin screw extrusion (c) after single screw extrusion and injection (d) after twin screw extrusion and injection.

#### 2.4.2. Analysis of Data

From the obtained record of measurements, the data can be exported into Microsoft Excel and manipulated for statistical analysis which can be represented as a fiber length distributional plot. The output data is presented as minimum fiber length, maximum fiber length, number of recorded measurements, number average fiber length ( $L_n$ ) and volume average fiber length ( $L_p$ ). The equations for ( $L_n$ ) and ( $L_p$ ) are shown below where  $L_i$  is the actual measured length of the fiber and  $n$  is the number of fiber length measurements.

$L_n = \text{Total sum of fiber length} / \text{Total count of fibers}$

$$L_n = \frac{\sum L_i}{n} \quad \text{Equation 1}$$

$L_p = \text{Total sum of squares of length of the fibers} / \text{Total sum of fiber length}$

$$L_p = \frac{\sum L_i^2}{L_i} \quad \text{Equation 2}$$

#### 2.5. Study of morphology by Scanning Electron Microscopy (SEM)

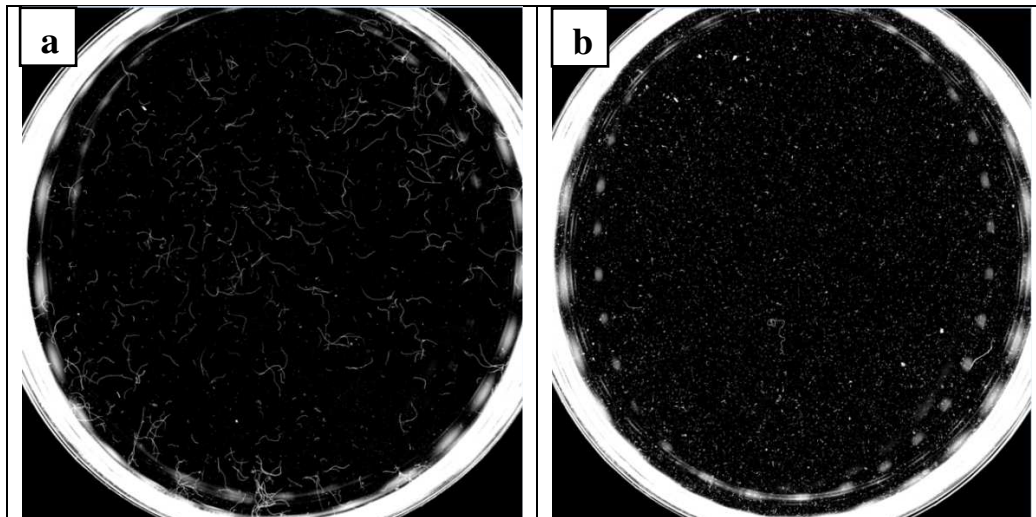
Scanning electron microscopy is a technique based on electron-matter interactions. The residual fibers after the removal of the matrix were observed. For this study, a Joel JSM- IT500HR SEM microscope was used. This microscopic characterization will allow us to understand the morphology of the fibers, their structure at the microscopic scale, as well as to see the effect of each extrusion or injection process on the flax fibers distribution and dispersion.

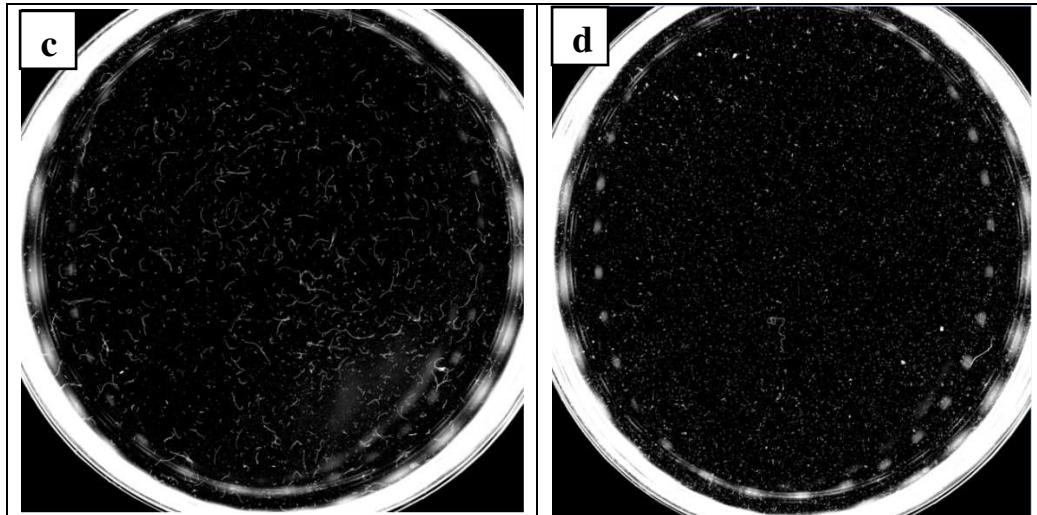
### 3. Results and Discussion

#### 3.1. Fiber length distribution

To optimize the properties of the composites, the greatest average length is sought. The objective is therefore to find a compromise in the mixing process (single or double screw) to promote the decohesion of the fiber bundles, but limit their fragmentation.

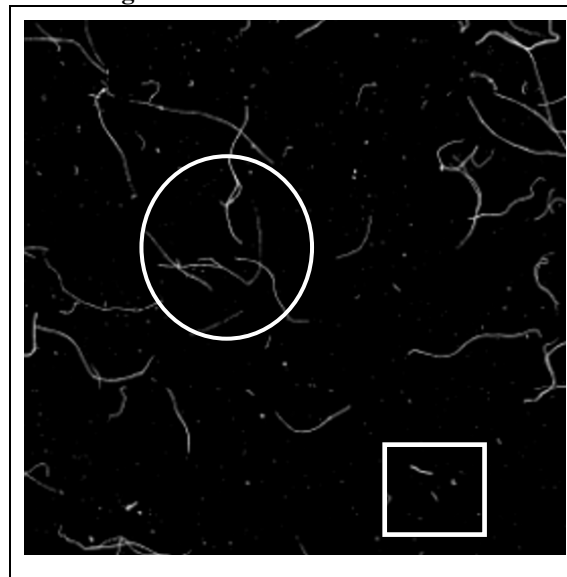
Clear images with a black background and white fibers are obtained (**Fig. 4**). Natural fibers are known to be birefringent under polarized light due to their high semi-crystalline cellulose content.





**Figure 4.** Cartographies of flax fibers lengths distribution (a) Fibers dispersed using Single screw extrusion (b) Fibers dispersed using Twin screw extrusion. (c) Fibers dispersed using Injection molding after single screw extrusion (d) Fibers dispersed using Injection molding after twin screw extrusion

The fiber mappings extracted from the composites are shown in **Fig. 4**. The extraction conditions are chosen so that the fibers are well defined and dispersed to facilitate automatic image analysis. The composites loaded with 4 mm flax fibers show rare clusters, and a size distribution could be made without considering them. The results show that the fibers undergo a lot of damage (breaking) during mixing under the action of elongation and shear forces. If we look at the cartographies, we realize that at the millimeter scale, the state of the condition of the fibers is very different from one sample to another: in some places, the fibers have been largely cut by the process (white square), while in other areas, they remained entangled (white circle), which protected them in some way from breakage as shown in **Fig. 5**.



**Figure 5.** Zoom in the cartography of flax fibers lengths distribution

Below in Table 2 is a summary of numerical results taken from 4 different process of manufacturing.

Table 2: Summary of comparison results between process

Sample	Ln: mean Fiber Length ( $\mu\text{m}$ )	Lp: Volume Mean Fiber Length ( $\mu\text{m}$ )	Maximum Fiber Length Measured ( $\mu\text{m}$ )	Minimum Fiber Length Measured ( $\mu\text{m}$ )
Single screw extruded material (E1)	1190	3308	8285	100
Twin screw extruded material (E2)	187	327	4165	30
Single screw extruded then injected material (EI 1)	655	2031	8152	100
Twin screw extruded then injected material (EI 2)	156	269	2761	39

To investigate the fiber length reduction after each process, the volume mean frequencies of fiber length was calculated and compared with the initial length of flax fibers (Fig.6).

This study showed a reduction in the average length (in number) of the fibers from 4 mm ( $L_{p\text{initial}}=3986\mu\text{m}$ ) to 269  $\mu\text{m}$  after the injection of the pellets obtained in twin-screw extrusion (Figure 8). This effect was mainly associated with the compounding operation and not with the injection operation. This result is in agreement with Keller (2003) [17] who studied the size evolution of hemp fibers in PEA and PHBV matrices.

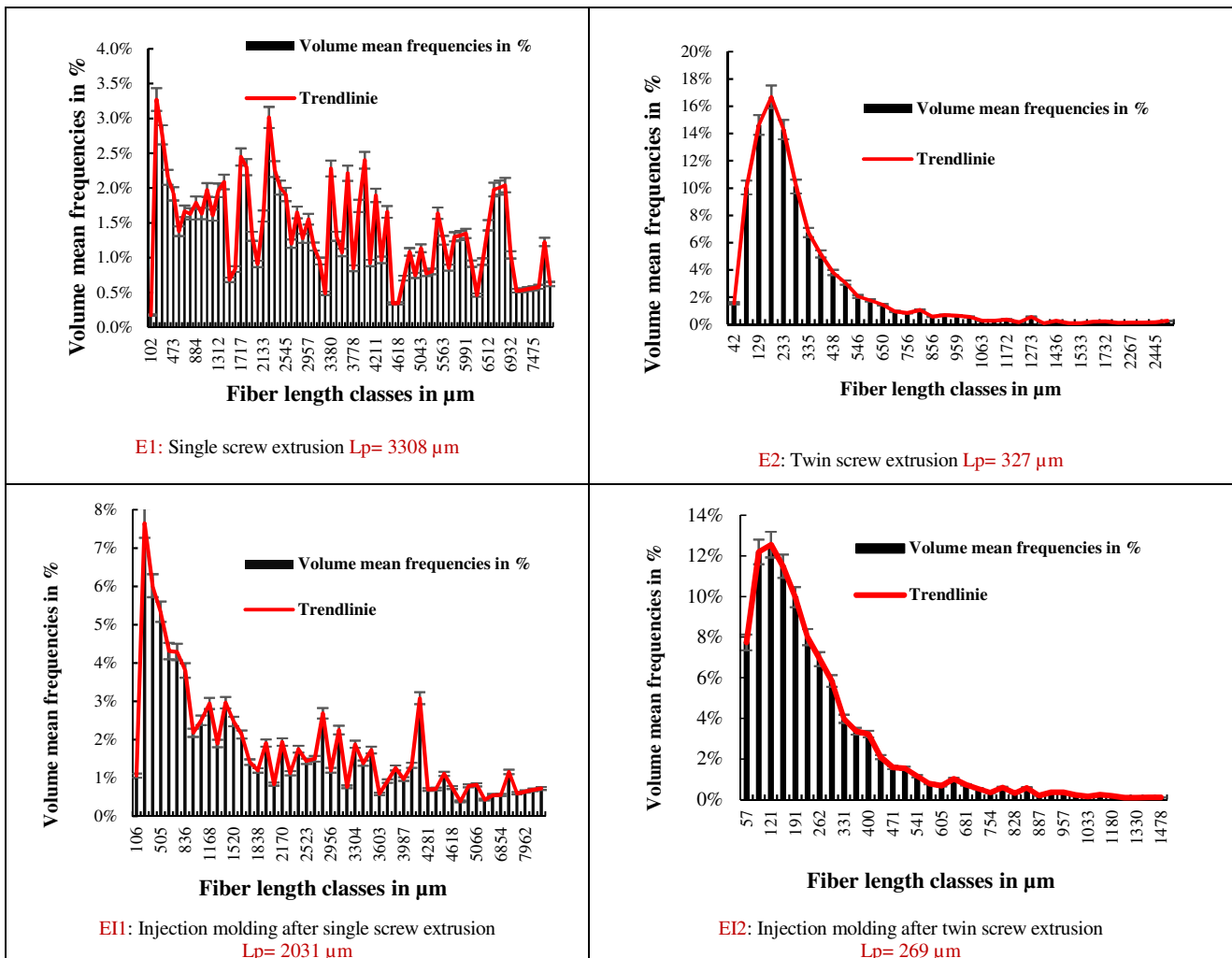


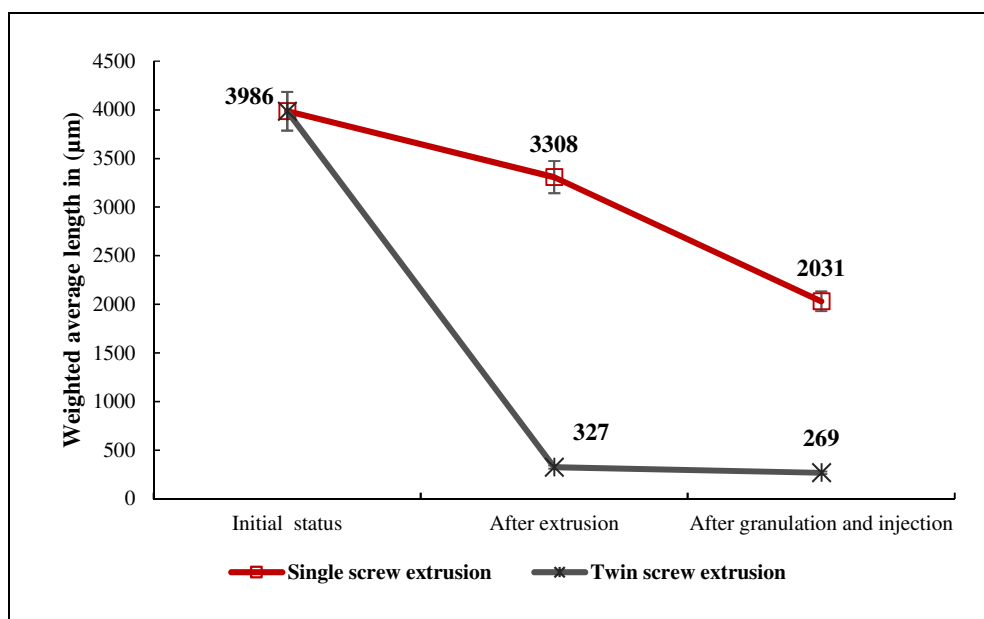
Figure 6. Flax fibers distribution for 80PLA/20PBS/20% Flax composite.

**Fig.6** shows the fiber length distribution plot for 80PLA/20PBS/20% Flax processed with single/twin screw extrusion and also after their molding injection. It is initially clear from the results that a greater concentration of shorter fibers was found in the twin screw extruded composite with more than 16 % of particles are smaller than 180  $\mu\text{m}$  when compared against the single screw extruded material. These particles probably do not mechanically reinforce the composites, but they can play on their rheological behavior. However, volume average results are higher for composite manufactured with single screw extrusion. This could suggest that at decreased dispersion of the flax fibers showed in single screw extrusion compared to twin screw extrusion, there is an increase of fiber-fiber interactions leading to clusters of fibers being forced which are able to protect each other from the high shear forces, preserving their length [18]. Figure 6 shows also the fiber length distribution plot. Although the volume average results are higher for the two extruded composites compared to their injected samples. As mentioned in **Fig.7**, the results tend to show that the injection process had also an effect on the flax fibers which underwent a decrease in their length.

	Initial status	After extrusion	After injection
Single screw cycle	3986 $\mu\text{m}$	3308 $\mu\text{m}$ (-17%)	2031 $\mu\text{m}$ (-38,6%)
Twin screw cycle	3986 $\mu\text{m}$	327 $\mu\text{m}$ (-91,8%)	269 $\mu\text{m}$ (-17,7%)

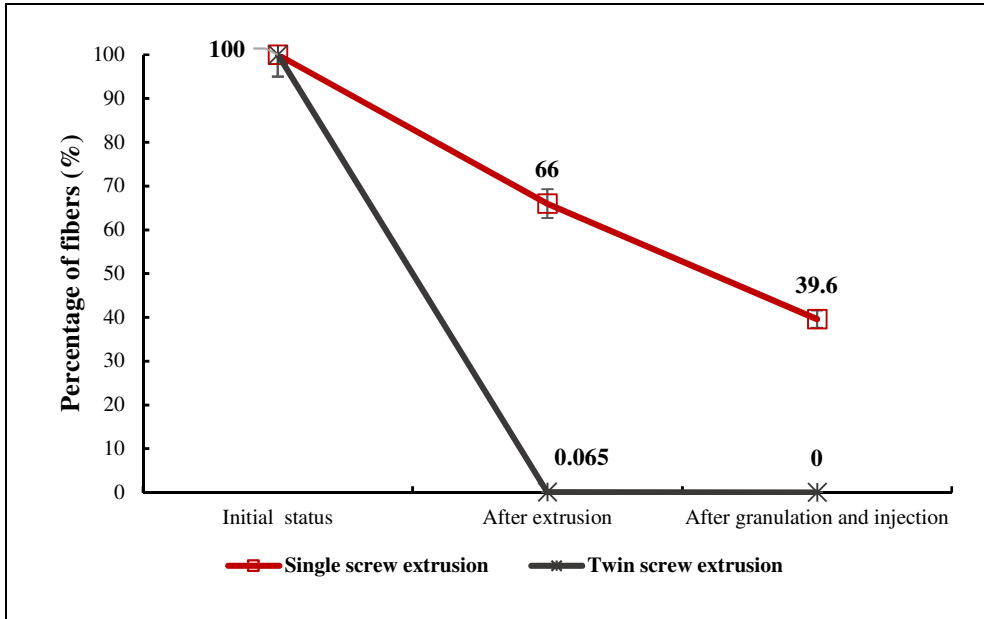
**Figure 7:** Effect of the extrusion and injection processes on flax fibers lengths distribution

The volume averages are marginally larger for Sample E1 at 3308  $\mu\text{m}$  and for E2 at 327  $\mu\text{m}$  compared to 2031  $\mu\text{m}$  for E11 and 269  $\mu\text{m}$  for E12 which are seemingly subsequently broken along the injection molding process (**Fig.8**) giving a rise to a higher distribution of shorter fibers less than 2 mm.



**Figure 8.** Evolution of the volume-weighted average length of flax fibers after compounding and injection

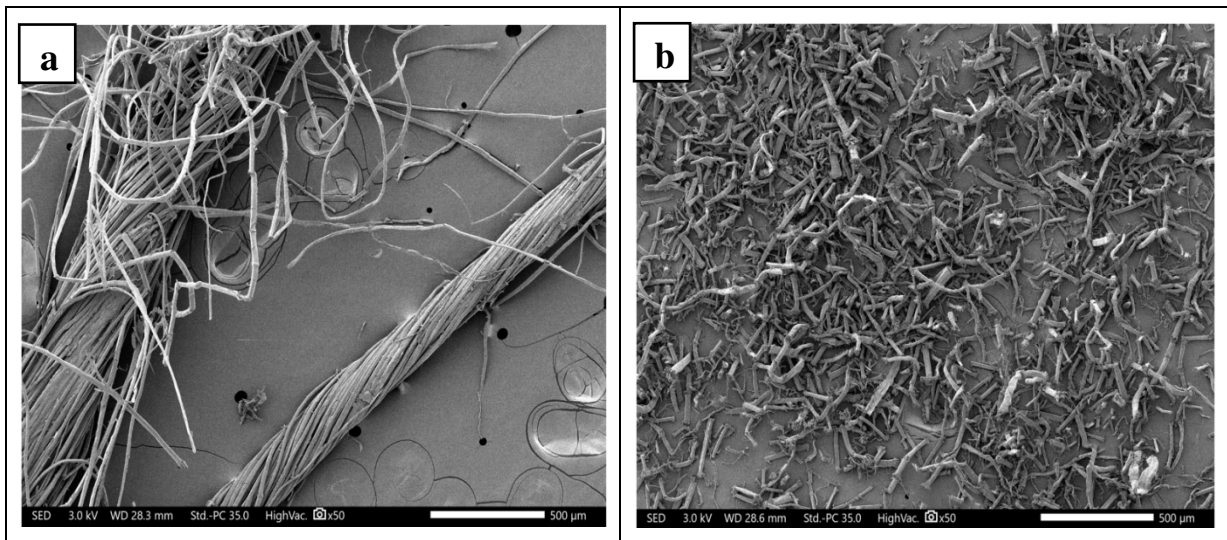
It is to note that 66 % of particles bigger than 2 mm are present after single screw extrusion then the percentage decrease to 39.6 % after the compound injection. For the twin screw extrusion, almost all the fibers are less than 2 mm (99,9%) in length with more than 10% of particles are smaller than 77  $\mu\text{m}$ , a number average length of 327  $\mu\text{m}$  and 269  $\mu\text{m}$  before and after the compound injection respectively (**Fig.9**). Le Moigne et al. (2011) [19] also observed a very high proportion of particles smaller than 40  $\mu\text{m}$  for flax and sisal fibers in a PP matrix. These particles likely originate from frictional phenomena that cause erosion and decohesion of fibers in a bundle.



**Figure 9.** Evolution of the percentage of flax fibers over 2mm weighted in volume after compounding and injection

*3.2. Evolution of fiber morphology after each stage of the processing of composite materials.*

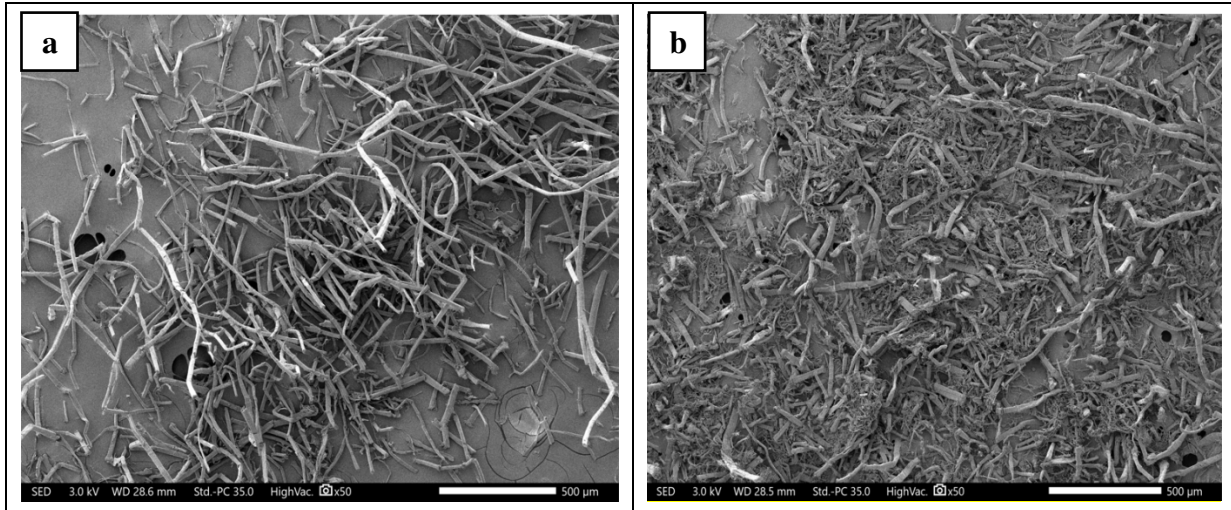
To better examine the dispersion of the fibers in the PLA / PBS blend for the various processes carried out, the morphology of the reinforcements was studied after each stage of the processing. The phenomena of fiber breakage are described as arising from three main factors: fiber-fiber interactions, Fiber-fiber interactions with the mixing equipment and fiber-matrix interactions: these include interactions between the fibers and the still solid pellets and interactions with the flowing molten polymer. The micrographs of representative's fiber after matrix removal were visualized by Scanning Electron Microscope (SEM) The diameters and lengths of the raw fibers were observed after extrusion (single-screw/twin-screw) and after injection, the reinforcements were extracted from the composites by chemical means.



**Figure 10.** extruded flax fibers observed by SEM.  
 Single screw extrusion: (a) Fiber bundles with longer diameters of still present macro-fibrils  
 Twin screw extrusion: (b) Fibrillation with more broken elementary fibers.

After compounding, a decrease in fiber length is observed, compared to the original dimensions of the raw fibers (**Fig.10**). During extrusion, the fiber bundles are divided and shortened by shearing. The bundles are separated by the breakage of the middling lamellae [20,21]. The average lengths are lower for twin-screw extruded fibers. These lower values could be explained by the greater ease of individualizing the fiber bundles into individual

fibers. During twin screw compounding, the flow generates high stresses and tends to separate the assembled fibers, which it tends to break the fibers lengthwise, causing them to lose their reinforcing action. The greater the fineness of the sample (high defibration rate), the tighter the distribution becomes in the low values. The histograms in Figure 8 illustrate this phenomenon very well. This decrease in length was already observed by Kessler et al [22]. The greater presence of long fiber bundles is observed after single screw extrusion. Some bundles are already split while others are very compact. This shows that single screw extrusion is not sufficient to fully isolate the single fibers.



**Figure 11.** Micrographs of fibers extracted by dissolving the composites after injection visualized by Scanning Electron Microscope (SEM). Injection after single screw extrusion: (a) The flax bundles are all dissociated and switch to micro-fibrils: bundles are no longer visible when no intact fiber units are still visible, the overall is highly destructured, Injection after twin screw extrusion: (b) lengths and diameters of the fibers have been reduced compared to the dimensions after extrusion

As a result of the injection step, the lengths of the fibers have been reduced, compared to the dimensions after extrusion. The injection step accentuates the splitting of the fiber bundles. The results are in agreement with previous work published in the literature for injected Lin/PP materials [23].

**Fig.11** shows that most of the reinforcements are in the form of single fibers, with a few bundles. This reveals a strong separation of the fiber bundles. It can be said that the extrusion process has the major effect on the reduction of the length of the fibers without affecting the diameters too much, on the other hand, the injection process has a remarkable influence on the residual fiber diameters. It is reported in the literature that the fiber bundles do not systematically separate into single fibers during mixing with the matrix. In a study by **Le Duigou et al [24]**, flax fibers mixed with PLA by single-screw extrusion at 20 rpm retained a diameter of 150 µm (initial diameter of 182 µm), with an aspect ratio around 20. Then the compounds are subjected to injection cycles to study the effects of potential recycling. After one injection, the aspect ratio of the fibers drops to 9 for a diameter of 50 µm. In contrast, two other studies reveal that the flax bundles separate completely into elementary fibers upon compaction with PP, unlike sisal bundles [25,26].

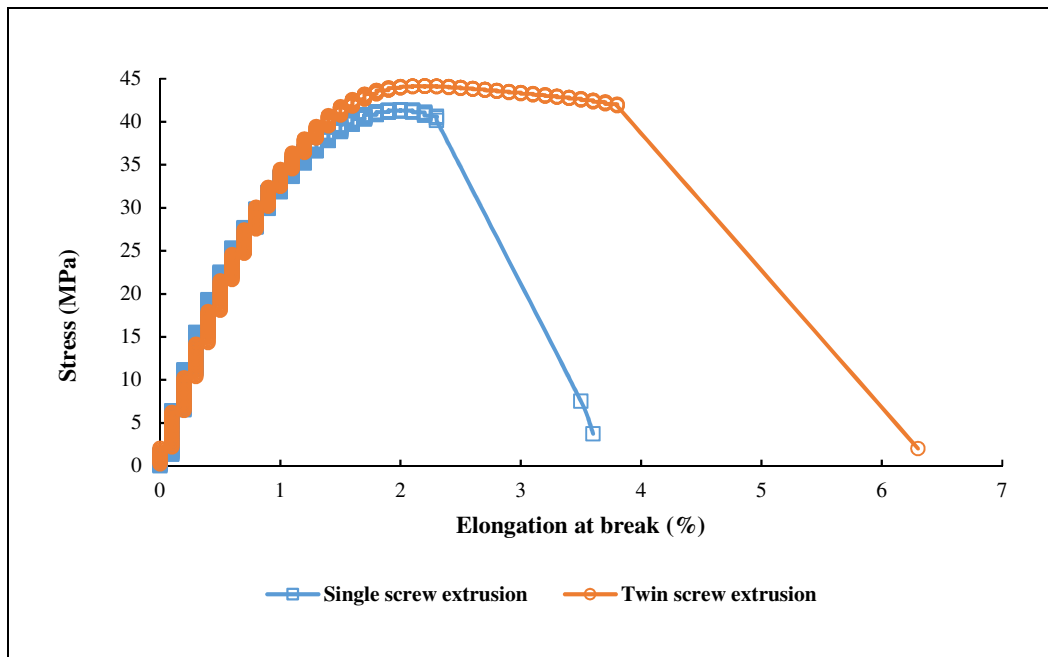
Flax fibers show more or less marked defects on their surface and mainly in the transverse direction. Visible surface defects are often heterogeneously distributed along the fibers and typically extend across the entire width of the fiber. Also, one can conclude that the optimization of morphological characteristics consists in a compromise between fineness and desired length. Work remains to be done in order to try to improve the homogeneity of the morphological properties of the fibers, in particular their length. However, the results presented on the characterization of the lengths are to be taken as relative and not absolute results.

### 3.3. Mechanical properties

Given the lack of precision in the dosage of the fibers in the extruder, it is difficult to evaluate precisely the impact of the compounding process on the mechanical properties. If we compare the best mechanical properties of the flax fiber composites obtained by single screw and twin screw extrusion, we realize that they are close. Table 3 summarizes the results of the tensile mechanical behaviors of the 80PLA/20PBS blend with 20 wt% flax fibers for both extrusion processes prior to specimen injection.

**Table 3:** Tensile mechanical properties of 20% flax fiber reinforced composites

	Specimens with single screw extrusion	Specimens with twin screw extrusion
	Average value	Average value
Young's modulus (MPa)	4353 ±176	4155± 115
Stress at break (MPa)	41.5 ± 0.80	44 ± 0.37
Elongation at break (%)	2.4 ± 0.15	3.8 ± 0.11

**Figure 12.** Tensile curves of 80PLA/20PBS blended with 20 wt% flax fibers

Regarding the tensile data (**Fig.12**), there is no significant effect of the melt processing types on the tensile properties considering the experimental errors. So, no reliable correlation can be established between the fiber length and the processing types in terms of mechanical properties. The stress at break of the composites is only slightly influenced by the change of the extrusion process. It is a little bit higher for twin-screw extruded composites with 20% fibers. It can be extrapolated that single-screw extruded composites have less resistance to stress than twin-screw prepared composites. This is due to the good dispersion [24] of the flax fibers in the matrix caused by the twin screw extrusion compared to single screw extrusion. Since the elongation at break showed few differences generally visible between composites prepared by the two extrusion and injection processes (2.4 % et 3.8 %). It can be deduced that for the same concentration of flax fibers, twin-screw extruded composites would result in slightly more ductile materials. The results obtained here in twin screw extrusion can be related to those of Bos et al (2006) [18] that showed an influence of the process type on the tensile properties of PP/flax composites. The work conducted by Ausias et al. (2013) [27] which concern thermoplastic composites reinforced with flax or hemp fibers and implemented by single-screw, twin-screw, or mixer extrusion showed that the Young's modulus is not a priori influenced by the load transfer phenomena at the interface, nor by the final aspect ratio of the reinforcement. However, the obtained results in relation with the characterization of the mechanical properties are to be taken as relative and not absolute results.

### Conclusion:

This research work consisted in deepening the knowledge on the rupture of flax fibers during their compounding with 80PLA/20PBS polymer matrix. The objective was to provide a systematic study on the relationship between the melt blending process and the length distribution of the flax fibers. The composites were elaborated

by single and twin screw extrusion and then passed to injection molding. The following conclusions can be drawn:

- The size distributions of the fibers were automatically analyzed with the fiber length measurement system “FASEP”.
- After single-screw extrusion, it appears that the flax fibers break up less in length than when they pass through a twin-screw extruder. Thus, they keep acceptable final lengths and the longitudinal breakage rate remains very low.
- To better examine the dispersion of the fibers in the PLA/PBS blend for the various processes carried out, the morphology of the reinforcements was studied after each stage of the processing. The Flax fibers show more or less marked defects on their surface and mainly in the transverse direction. The results show that the average lengths are lower for twin-screw extruded fibers compared to the single screw extrusion. As a result of the injection step, the lengths of the fibers have been reduced, compared to the dimensions after extrusion process.
- A correlation between fiber length distribution and tensile properties was found. It would seem that the single screw extrusion process leads to slightly stiffer composites. It can be extrapolated that single-screw extruded composites have less resistance to stress than twin-screw prepared composites. This is due to the good dispersion of the flax fibers in the matrix. In term of elongation at break, twin-screw extruded composites would result in slightly more ductile materials.

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