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Mechanical characterization of PHA reinforced with Alfa fibers: influence of fiber content and strain rate

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Abstract

Biocomposites made from renewable resources have a lower environmental impact than inorganic composites. However, these new biocomposites must have the same mechanical properties as traditional composites to be able to replace them. The main objective of this study is to analyse the influence of strain rate and fiber content on the mechanical behaviour of a new biocomposite composed of a biodegradable PolyHydroxyAlkanoate (PHA) matrix reinforced by Alfa fibers. These biocomposites were elaborated after an alkaline treatment and fiber bleaching procedure. SEM and chemical analysis showed that these treatments remove the non-cellulosic materials such as pectin, hemicellulose and lignin, and improve the cellulose content. Three fiber contents of 5, 10 and 15%wt and three strain rates were investigated to analyse their dependence on uniaxial tensile tests. By increasing the amount of fiber and the strain rate, the mechanical response was improved with higher Young's modulus and tensile strength but lower elongation at break. Poisson's ratio have also shown dependence on strain rate and fiber contents. These results underline the good mechanical properties of this PHA/Alfa biocomposite which could be an interesting alternative in the development of products, such as automotive interior parts or some packaging applications.

Keywords

Biocomposite, Alfa fibers, PolyHydroxyAlkanoates (PHA), Alkaline Bleaching treatments, Fiber contents, Mechanical characterization

Introduction

Polymer matrix composites are composed of organic polymers into which short or continuous fibers are introduced. Their light weight and high resistance to abrasion and corrosion as well as high stiffness and strength for reinforcement make them widely used in all industrial sectors [1]. Aramid, glass and carbon fibers have long been used as reinforcement due to their low cost and excellent mechanical properties [2]. However, producing these composites requires a significant amount of energy, and their lack of recyclability negatively impacts the environment [3].

In response to the environmental problems associated with these composites, one solution is to use vegetable fibers extracted from renewable resources as reinforcement. These natural fibers also have the advantage of being biodegradable. Flax, jute, hemp, sisal, pineapple, and coir are commonly used as sources of natural fibers in manufacturing ecological composites [4, 5, 6, 7]. To be considered a completely biocomposite, the matrix with such fibers must be a renewable polymer. The literature shows that several biodegradable plastic matrices have been investigated and classified into two groups: natural polymers obtained from natural resources and synthetic polymers produced from oil [8].

Among the possible biodegradable matrices [9], PolyHydroxyAlkanoates (PHAs) are a promising family of biopolymers for use as a biodegradable matrix in biocomposites. PHAs are intracellular biopolymers synthesized by many bacteria using renewable biomass from agricultural activities [10, 11]. Fermentation processes can produce these

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biopolymers from natural carbon resources, which exhibit valuable biodegradability and biocompatibility properties [12]. This biodegradable matrix has attracted much attention due to its similar material properties to conventional plastics such as non-degradable polyethylene and polypropylene. PHAs can be either homopolymers, such as polyhydroxybutyrate (poly(3HB)) or copolymers, such as polyhydroxybutyrate-co-hydroxyvalerate (poly(3HB-co-3HV) also named PHBV [13].

Natural fibers, such as Alfa fibers (or Esparto), have been investigated as reinforcement in composites by many researchers in North Africa [14, 15, 16]. The Alfa plant, an endemic plant of the western Mediterranean regions, grows in semi-arid areas [17]. Currently, this plant is widespread in a large geographical area mainly in Algeria, Morocco and Tunisia totalling about 6 millions hectares of natural steppes [18]. While in Spain and Portugal, Alfa grass cover a surface area of approximately 400,000 hectares [17]. Most research on incorporating Alfa fibers into composites has used polyester resin [19] or polypropylene as a matrix [20, 21, 22].

A few studies have analyzed the mechanical behaviour of composites incorporating Alfa fibers into a biopolymer matrix. Belhassen et al. [15] investigated the effect of Alfa fibers on the mechanical properties of a starch-based biopolymer, a commercial matrix named Biopar-S®. The authors showed that the incorporation of lignocellulosic fibers of Alfa in this type of biopolymer causes a high increase in the elastic modulus and the maximum tensile and flexural strength as a function of the fiber content (from 10 to 30%) with a decrease of elongation at break. Arabeche et al. [23] explored the crystallization behaviour and rheological properties of poly(butylene succinate) (PBS) composites reinforced with Alfa fibers. They observed an increase in viscosity with an increase in the elastic melt complex modulus (G' and G''). The uniform dispersion of Alfa fibers within the PBS matrix and strong interfacial interactions between the two components are responsible for the improved performance of the composites.

On the other hand, Gunning et al. [24] tested PHAs reinforced with natural fibers (hemp, jute, and lyocell) and showed a reduction in mechanical properties with too high a fiber content. They thus obtained a reduction in the mechanical performances of composites containing more than 20% of jute. Indeed, according to Jahangir [25], a high fiber content, leads to the formation of more voids at the fiber/matrix interface, making

the stress transfer to the fibers inefficient and causing the formation of microcracks when the material is subjected to stress and thus a reduction in tensile strength.

However, according to the literature, using natural fibers (Alfa fibers) in conjunction with a PHA polymer matrix is rare and has only been investigated in a study conducted by Hammiche et al. [14]. The authors examined the impact of incorporating Alfa fibers into PHBV based composites by testing various fiber treatments and fiber content. These authors have also examined the biodegradation behaviour of these biocomposites in aqueous environments. Additionally, Werchefani et al. [26] demonstrated that the thermal properties of polylactic acid (PLA) composites containing Alfa fibers could be improved by using a combination of mechanical, chemical, and enzyme treatments.

Chemical treatments of fibers are very important for increasing the mechanical response of composites, as the adhesion between the matrix and the fibers improves considerably thanks to these chemical modifications [27]. Indeed, adhesion is an important factor in the transmission of stresses from the matrix to the fibers and contributes to the properties of composites. Poor surface adhesion caused by insufficient wetting is the main reason for the formation of a poor or inefficient interface between the fiber and the matrix. Several studies [28, 29, 30] have also indicated that the main factor limiting the mechanical properties of natural fiber reinforced thermoplastic composites is the chemical incompatibility between the hydrophilic lignocellulosic molecules of the natural fiber and the hydrophobic thermoplastic molecules. Usually, an alkaline treatment sodium hydroxide (NaOH) is the most commonly used treatment. As explained before, this alkaline treatment aims to improve the adhesive interface between fibers and matrix by making the fibers more hydrophilic. This treatment also enables the elimination of some amorphous materials such as wax, pectin and hemicellulose [31, 32]. El-Abbassi et al. [21] have shown that the percentage of cellulose in fibers varies depending on the extraction and harvesting season, and the alkaline treatment can also enhance the fiber-matrix interface of polymer composites exposed to water-aging. Another chemical treatment with sodium hypochlorite (NaClO) solution, known as bleaching process, is also often used to increase the absorbency, flexibility and resistance of the fibers in the matrix polymer [33, 34]. The bleaching treatment can also

remove some contaminants that may negatively affect fiber performance [35]. Thus, one of the objectives of this article is to investigate the influence of alkaline and bleaching treatments on the cellulose content and its role in adhesion with the polymer matrix. The Alfa fibers obtained after these two treatments are referred to as Alfa Fiber Alkaline Bleaching (AFAB) in this article.

The other main objective of the present study is to determine the mechanical properties of PHA/AFAB biocomposites as a function of the fiber content but also the influence of the strain rate on the behaviour of these materials. Conventionally, polymers and composites are tested at a single slow strain rate and the strain rate variation on these materials is rarely explored. However, polymer materials are highly dependant on loading speed (e.g [36, 37, 38]). In this context, the mechanical properties of PHA/AFAB biocomposites, with three fiber contents of 5, 10 and 15%wt were determined by tensile testing at three strain rates, respectively 10^{-5} , 10^{-4} and 10^{-3} s^{-1} . The effects of the fiber ratio and strain rate on Young's modulus, tensile strength and elongation at break were investigated. We also studied these effects on the Poisson's ratio which is not classic but very important for the numerical simulations. By SEM analysis, the strain rate effects on fracture surfaces of biocomposites were finally analysed. Thus, this study aimed to quantify the strain rate effects on the overall behaviour in terms of elastic properties, damage and ultimate characteristics of this new PHA/AFAB biocomposites. The longer-term objective being to establish the field of application for this composite in the automotive interior parts or some packaging industries.

It is also important to have an idea of the orientation of the fibers in the matrix after injection. For this, we carried out a tomographic analysis which allowed us to observe this phenomenon. This X-ray tomography analysis allowed us to verify the proportion of fibers incorporated within the matrix. Thus, it was possible to examine the uniform distribution and alignment of the fibers in the tensile specimen depending on the injection direction.

An ANOVA analysis (analysis of variance) was also carried out to enhance this study, two factors were tested to evaluate their impact: strain rate and fiber content. Using this method, the impact of these two factors on the results was assessed by comparing variations between and within groups. ANOVA was used to determine whether the means were

significantly affected by these factors, providing essential information for understanding the effect of these variables on the tensile test results obtained.

Materials and Methods

Matrix PHA

In this study, we used the grade of PHA called PHI002 as polymer matrix. This PHA, provided by NaturePlast, is a biosourced thermoplastic resin derived from over 90% annually renewable plant resources, as specified in ASTM D6866. The biobased nature gives PHAs particularly good biodegradability and biocompatibility properties [12]. This material is characterized by a density of 1.25 g cm^{-3} , a melt index (190 °C, 2.16 kg), and a melting temperature of between 170 and 176 °C. This biodegradable matrix was mixed with different percentages of natural fibers extracted from Alfa plant stems to elaborate PHA/Alfa fiber biocomposites.

Alfa fibers

As explained in introduction, Alfa plants (also named Esparto (*Stipa tenacissima*)) grow in semi-arid regions along the North African Mediterranean coast. These plants are mainly used to produce high-quality paper such as cigarette paper, decorative paper, and dielectric applications for capacitors. The Alfa plants used in this study were collected in the region of Kasserine in west-central Tunisia. Alfa plants were harvested by hand using special knives and after the seeds matured in July-August.

Due to their hydrophilic character related to the presence of hydroxyl groups in the fibers' structure [39] that also affect their moisture resistance [40], the fiber/matrix adhesion is reduced, which results in a reduction in the mechanical properties of the composite [31]. To overcome this drawback, two treatment methods (alkaline and bleaching) have been tested to enhance the surface properties of the Alfa fibers [20, 40, 41].

Alkaline and bleaching treatments of Alfa fibers

In this section, the extraction methods of Alfa Fibers (AF) from Alfa Stems (AS) (see Figure 1-a) are explained, and the two steps of the alkaline treatment and bleaching method to obtain the Alfa Fiber Alkaline Bleaching (AFAB) (see Figure 1-b) are detailed. Alkaline treatment and

bleaching methods equivalent to those employed by Labidi et al. [34] are used, in this study, to obtain the AFAB.

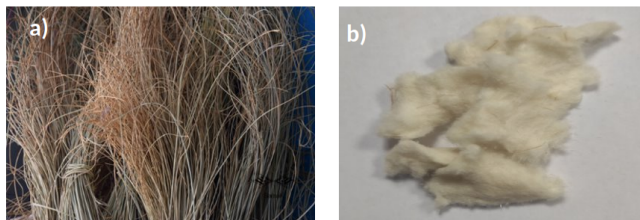


Figure 1. a) Initial Alfa stems (AS) before alkaline and bleaching treatment and b) Alfa fiber alkaline bleaching (AFAB) obtained at the end of the treatment.

Figure 2 shows the different steps used in this study to obtain AFAB. The first step was to manually remove foreign bodies to obtain clean and usable AS. The stems were then cut into 3 to 4 mm pieces. Next, a water retting treatment step was added whereby these fibers were immersed in salt water for six days and subsequently dried in an oven until they reached constant mass. In the second step, the fibers were subjected to the alkaline treatment, which involved immersing them in a 3.25 M NaOH solution at 100 °C for 2h30 min, using a fixed ratio of 1 g of stem mass to 20 mL of solution volume. Finally, the fibers were thoroughly rinsed in distilled water until their pH became neutral to eliminate excess NaOH.

After this initial alkaline treatment, the fibers were subjected to a manual post-treatment with a mortar and pestle for approximately 5 min to further disintegrate the fibers. The fibers were then dried at around 80 °C until a constant mass was obtained.

A bleaching process was finally used with a 1.7% NaClO sodium hypochlorite solution to further increase the cellulose content and reduce the lignin content. The pH of the solution was adjusted to 7 by adding acetic acid. Next, the fibers were subjected to a reflux step for 2 h at 60 °C. This bleaching process was repeated twice. After bleaching, the fibers were carefully rinsed with distilled water down to pH=7 to remove excess NaOH.

It is important to note that these alkaline and bleaching treatments use many chemicals which harm the eco-responsibility of these biocomposite materials and that it would be desirable to find equivalent treatments but more chemically neutral [42].

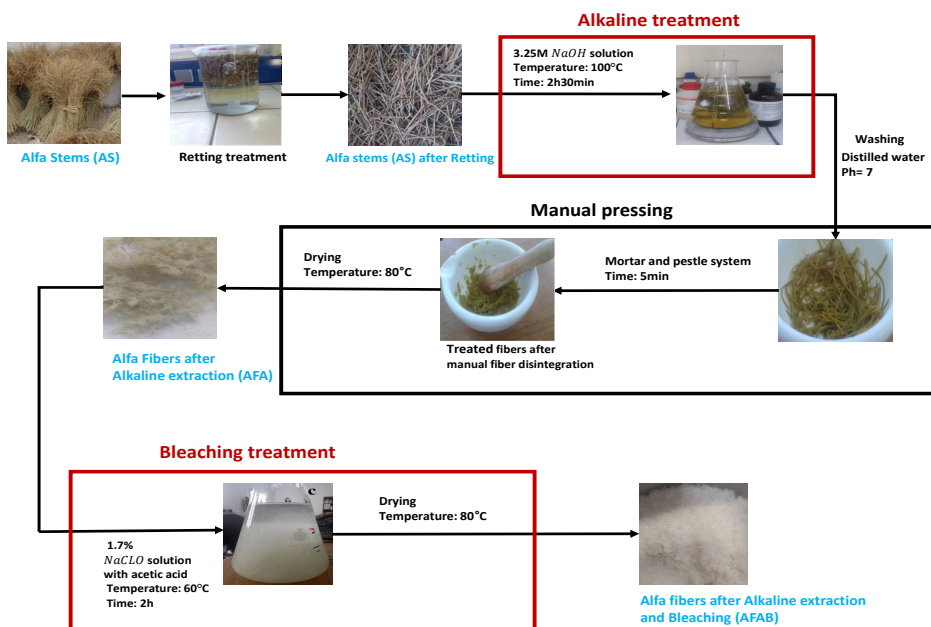


Figure 2. Experimental protocol detailing the alkaline and bleaching treatments to obtain AFAB.

Fabrication process of PHA/Alfa biocomposites

The protocol presented in Figure 3 details the various steps of the preparation of PHA/AFAB biocomposites and the tensile specimens. Three types of Dumbbell specimens with fiber contents of 5, 10 and 15%wt were produced.

First, to ensure homogeneity in humidity level and better adhesion between the AFAB and the PHA matrix, a drying process for the AFAB was carried out at 60 °C under vacuum for 24 h before any further processing. The AFAB and the PHA matrix were then extruded together on a single-screw extruder at a temperature of 180 °C and a rotational speed of 30 rpm to produce a composite material suitable for injection molding. The extrusion process disperses the AFAB in the PHA matrix and improves adhesion between the matrix and the fibers. After the extrusion process, the biocomposite materials were granulated. Finally, the biocomposite materials were injected using a Battenfeld HM 80 – 210

S Unilog B6 plastic press in a dedicated mold at a temperature of 180 °C and a pressure of 1000 bars.

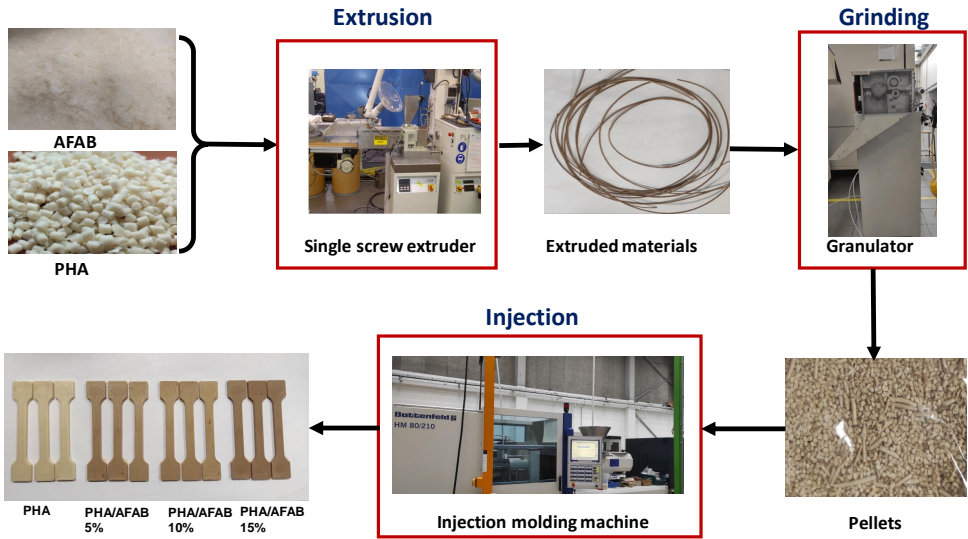


Figure 3. Different steps of PHA/Alfa fibers (AFAB) biocomposites elaboration.

The dimensions of the Dumbbell-shaped tensile specimens, according to ISO-527 standard, obtained after injection, are shown in Figure 4.

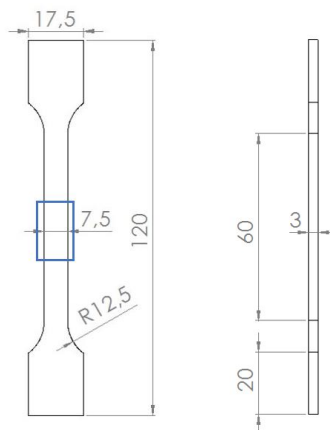


Figure 4. Dimensions (in mm) of Dumbbell-shaped tensile specimens according to ISO-527 standard and area in blue for the X-ray tomography and DIC analyses.

To ensure sample homogeneity and encourage recrystallization, a heat treatment step was systematically applied to all specimens prior to each experimental mechanical campaign. Samples were first annealed in a humidity chamber (SECASI Technologies SLH 100/70 VRT) at a temperature of 120 °C for a period of 2 h.

Next, a cooling step was conducted progressively, decreasing the temperature by 10 °C every 2 h. This heat treatment is necessary to guarantee the mechanical properties of biocomposites over time, as the recrystallization of these materials leads to a change in characteristics over time [43].

Characterization of PHA/Alfa biocomposites

In this section, methods to determine chemical compositions of both treated and untreated fibers to analyze the influence of the alkaline and bleaching treatments on the cellulose content of the fibers will be presented. SEM and Tomography analysis will also be explained. Next, we present the methods used to perform tensile tests.

Chemical composition of Alfa fibers before and after treatment

A chemical analysis was performed on Alfa Stems (AS), Alfa Fibers after the Alkaline treatment (AFA), and Alfa Fibers after the Alkaline Bleaching treatment (AFAB) to determine their composition. The main constituents of these fibers, namely cellulose, hemicellulose, lignin and extractable material (pectins, waxes and minerals) were extracted using the following protocol described by several authors [44, 45]. The same extraction protocol was applied to determine the extractable matter, hemicellulose and lignin content of the AS, AFA, and AFAB to ensure consistency in the analysis.

First, to determine the extractable matter content, the three types of fibers were dried in a vacuum oven for 24 h at 80 °C until constant mass. Next, they were pretreated in a toluene/ethanol solution at 60 °C and held under reflux for 6 h to remove grease and wax. The amount of extractable matter was calculated using the difference in weight between the initial sample and the sample after extraction.

Second, to determine the hemicellulose content, a sample of 1 g of extractable amount was mixed with a liquid NaOH solution of 17.5%. The solid fraction was then filtered, cleaned, neutralized, dried and weighed.

The amount of hemicellulose was calculated using the difference in weight between the initial sample and the recovered solid fraction.

The lignin content was determined using a 1 g sample of an extractable amount treated with a 72% concentrated sulfuric acid. The solid lignin fraction was recovered by filtration, drying, cooling and weighing. After determining the contents of extractable matter, hemicellulose and lignin in the three types of fibers, the cellulose content was calculated directly using the following formula:

$$100 - \%(Extractable + Hemicellulose + Lignin) = \%Cellulose \quad (1)$$

This equation is based on the fact that cellulose is the major component of fibers, accounting for most of their weight. Hence, the remaining percentage can be attributed to cellulose by subtracting extracts, hemicellulose and lignin by 100%.

SEM and X-ray tomography analysis

A JEOL JSM-IT500 HR Scanning Electron Microscope was used to analyze the surface and size of the Alfa fibers before and after the alkaline and bleaching treatments and the morphology of the biocomposites PHA/Alfa. Tomographic analyses was also conducted to determine the proportion of fibers in each composition and the orientation of the fibers along the specimen axis.

Tomography is an imaging technique using X-rays to analyze the microstructure of materials without being intrusive or destructive. This technique provides three-dimensional images via a reconstruction calculation from a series of X-rays from different angles. It gives data on the internal characteristics of the material, including density, porosity, element distribution, angle, size and shape of defects, or inclusions. The first objective of the X-ray tomography analysis performed on PHA/AFAB composite tensile specimens was to verify the percentage of fibers incorporated into the matrix. The second objective was to analyze the homogeneity of the distribution and orientation of the fibers in the sample as a function of the injection direction. To obtain the most representative results, this tomography analysis was conducted on the central position of the specimens (see the zone in blue of the sample shown in Figure 4). The MyGVL software was used to visualise 3D objects (in vector, point

and mesh format), analyse and take measurements on these objects, and display the results.

Experimental traction campaign

Tensile tests were performed at room temperature using an Instron 5969 universal testing machine equipped with a 5 kN load cell. The local strain was measured using an axial Instron extensometer with a length of 12.5 mm. Three strain rates of 10^{-5} , 10^{-4} and 10^{-3} s^{-1} were tested to analyze the effect of strain rate in the stress-strain curves. Two methods were tested to impose the strain rate during the tensile test. The first method, called "machine displacement control", involves using the tensile machine to impose the strain rate on the grips. The second, called "extensometer control", aims to impose the strain rate using an extensometer.

The evolution of deformation during experiments is captured using Digital Image Correlation (DIC) (see Figure 5). To prepare specimens for DIC, the sample surface is cleaned and spray-painted with a stochastic pattern in white and black using acrylic paint from a spray can. Two high-speed cameras equipped with 50 mm lenses are employed to capture images for DIC. The main advantage of the DIC system is that it can be used to measure the local strain field during the test in all the directions corresponding to those of the specimen gauge. With the axial (Major strain) and transverse (Minor strain) strains measured using the DIC system, the Poisson's ratio can then be determined.

Figure 5-a presents the evolution of the axial strain rate as a function of the axial strain measured by the DIC method in the zone between the extensometer knives (see Figures 4 and 5-b), using the two methods of control of the strain rate during a tensile test at 10^{-4} s^{-1} on a PHA specimen. This figure shows clearly that the strain rate is not constant during the test when machine displacement control is used with a non-linear increasing strain rate of slope $27 \times 10^{-4} \text{ s}^{-1}$, whereas with the extensometer control, a constant strain rate of 10^{-4} s^{-1} is obtained in the central zone of the specimen. Hence, the measurements obtained using the extensometer show strain stability, indicating that the control method effectively maintained a constant strain rate value throughout the test. This result can be attributed to the ability of the extensometer to accurately detect dimensional changes in the specimen, enabling the applied force

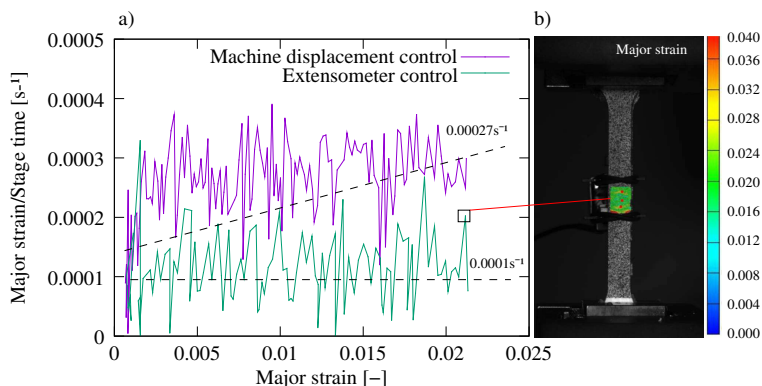


Figure 5. Strain rate control during tensile test of PHA with strain rate of 10^{-4} s^{-1} : (a) major strain/stage time-major strain curves with the two methods to control the strain rate and (b) visualization of major strain in the area between the extensometer's knives during extensometer control method.

to be precisely adjusted to maintain the desired strain. In the following section, all results are presented with this extensometer control method. At each test, a check was made to ensure that the strain rate was constant during the test.

Results and discussions

Effect of treatment on the microstructure and chemical composition of fibers

In order to understand the impact of the treatments on the microstructure and chemical composition of the fibers, Table 1 presents the chemical composition (cellulose, hemicellulose, lignin, and extractable material) of AS after the steps of the alkaline treatment (AFA) and the bleaching treatment (AFAB).

Table 1. Percentage in %wt of cellulose, hemicellulose, lignin and extractable material for the AS, AFA and AFAB.

	Cellulose	Hemicellulose	Lignin	Extractable material
AS	44.40	22.70	23.60	09.30
AFA	82.40	04.50	06.80	06.10
AFAB	90.50	02.30	02.50	04.70

Alkaline and bleaching treatments significantly increase cellulose content while reducing the presence of hemicellulose and lignin in the fibers. At the end of these two treatments, the fibers showed poor levels of hemicellulose (2.30%wt) and lignin (2.50%wt). This observation suggests that the bleaching process reduced the lignin and hemicellulose content, but it was the alkaline treatment that increased the cellulose content the most. These results are consistent with previous studies [34, 46] showing that alkaline bleaching treatments of Alfa fibers greatly increase the cellulose content in contrast to untreated fibers. A percentage of 90%wt cellulose in the AFAB at the end of the treatment is a notable rate to improve product quality.

SEM images of fiber surfaces are obtained before and after the treatments. Figure 6-a shows the AS before both treatments. At this stage, the stems have a diameter of 1.3 mm. Figure 6-b indicates the presence of coated impurities on the AS surface. In contrast, Figure 6-c shows the Alfa fibers divided into fiber bundles and several elementary fibers after the alkaline and bleaching treatments according to their respective dimensions. The fiber bundles are composed of several elementary fibers. Figure 6-d shows the rough surface of the AFAB, with ridges indicating the presence of cellulose microfibrils wrapped in a mixture of hemicellulose, lignin and pectin.

The size of the AFAB was a critical factor in determining the mechanical properties of the composite, as demonstrated in previous studies of wood flour particle sizes in polypropylene composites [47]. AFAB with a length of 1 mm after the alkaline and bleaching treatments were used; this was achieved by grinding the fibers using a 1 mm grid before injecting them into the polymer matrix.

After this grinding, the dimensions of the AFAB fibers were measured by SEM to calculate the aspect ratio. To this end, several AFAB SEM micrographs were taken, and several dimensional measurements were carried out from these photos. Figure 7 shows SEM micrography representing lengths and diameters of several AFAB.

AFAB SEM micrographics were analyzed using ImageJ software to obtain statistical distributions of fiber diameters and lengths. Histograms of length and diameter distributions, shown in Figure 8, revealed that the diameters ranged from 6 to 15 μm , while the lengths ranged from 300 to 1500 μm . The observed variation in fiber sizes is attributed

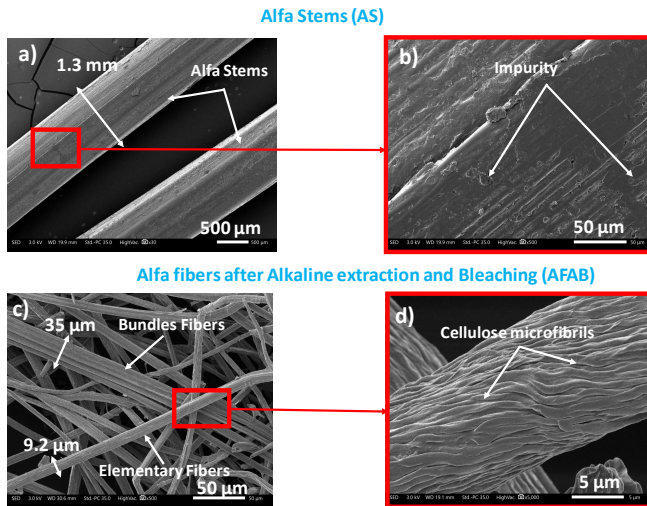


Figure 6. SEM images of AS (a,b) and AFAB (c,d) with dimensions of fibers, the presence of impurities on AS and rough cellulose microfibrils on AFAB.

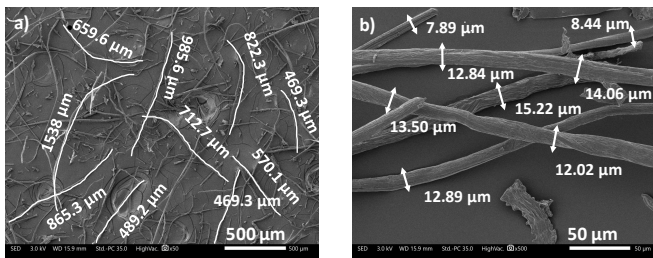


Figure 7. SEM analysis of elementary fibers to determine (a) fiber lengths and (b) fiber diameters

to the removal of non-cellulosic materials that bind the fiber bundles, causing their breakage and the appearance of elementary fibers. The aspect ratio, defined as the ratio between the length of the fibers and their diameter $L_D = \frac{L}{D}$, is an important factor in determining the final mechanical properties of natural fiber-based composites. The aspect ratio strongly influences the mechanical properties of hybrid composites, as a high aspect ratio facilitates efficient stress transfer to the matrix [48]. A higher ratio generally indicates greater strength but lower elongation.

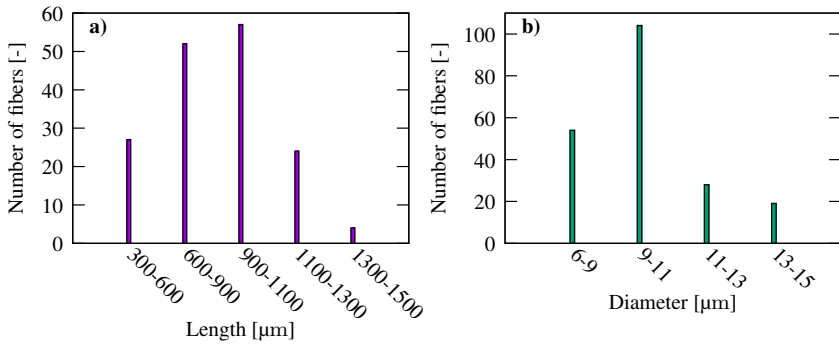


Figure 8. Distribution of the AFAB dimensions according to the number of fibers: (a) Length distribution and (b) Diameter distribution.

Conventionally, a ratio $L_D > 10$ improves the mechanical properties [15]. Based on the fiber length and diameter distributions shown in Figure 8, the average diameter of AFAB is about $D = 10.9 \mu\text{m}$, and the average length is about $L = 914 \mu\text{m}$, which leads to a ratio of $L_D \approx 84$. This aspect ratio is quite acceptable and shows that the alkaline and bleaching treatments improve the mechanical properties of AFAB, which should enable the beneficial reinforcement of composites based on these natural fibers.

Percentage of fibers and their orientation in the specimen

X-ray tomography analysis provides a detailed insight into the distribution and orientation of the fibers within the biocomposites studied. Based on the data collected, Figure 9 presents the percentage of AFAB within the PHA matrix along the axial direction of the sample center for each type of biocomposite.

The PHA/AFAB biocomposite with 5%wt fiber content complies with the 5.29%wt target value. In the case of the PHA/AFAB biocomposite with a proportion of 10%wt, a fiber percentage of 9.33%wt is actually obtained in the central zone. For the PHA/AFAB biocomposite with a ratio of 15%wt, the analysis shows a rate of 15.03%wt. The results also showed that the percentage of the fibers in the biocomposites was correct and confirmed that the fibers were uniformly dispersed throughout the matrix.

Figure 10 shows the histograms of the distribution of fiber angles in the matrix relative to the transverse axis of the specimens for the different biocomposite fiber ratios. The majority of the fiber angles are at 90° to the

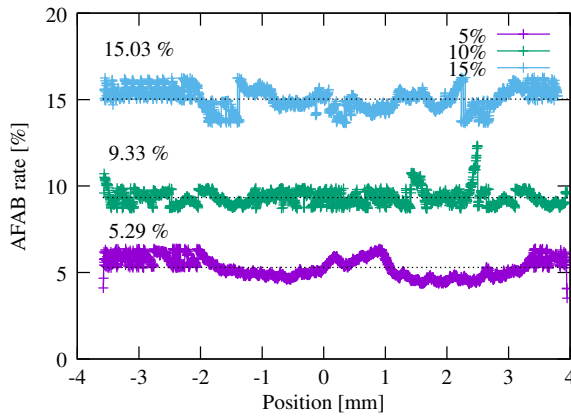


Figure 9. X-ray tomography: percentage distribution of fibers in 5, 10, and 15%wt PHA/AFAB biocomposites along the transverse direction of the specimen.

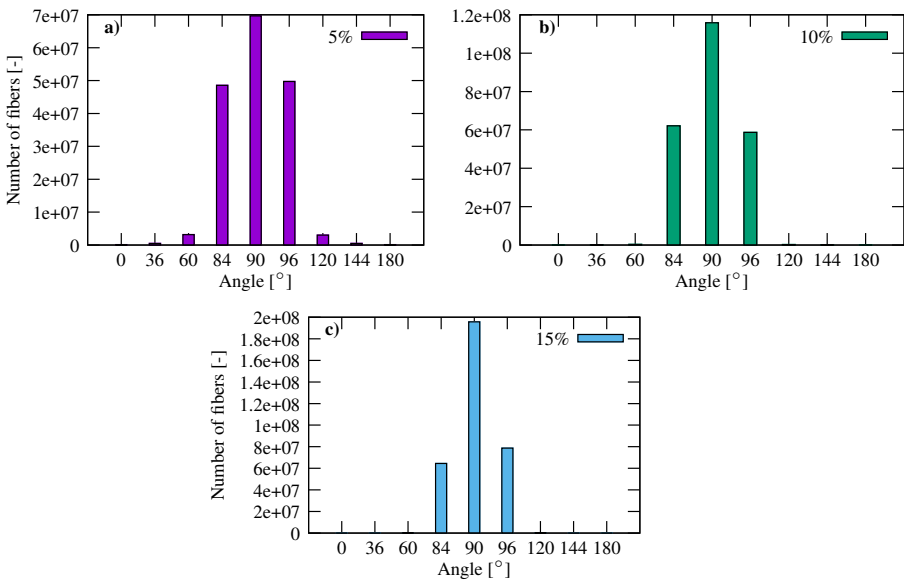


Figure 10. X-ray tomography: fiber orientation distribution in the matrix in 5, 10, and 15%wt PHA/AFAB biocomposites.

transverse direction of the specimen, showing that the fibers are oriented in the direction of specimen injection.

Effect of fiber content and strain rate on mechanical properties

To evaluate the impact of fiber content and strain rate on mechanical properties, nominal stress-strain curves for PHA and biocomposites are presented in Figure 11. These curves show the nominal stress-strain curves for PHA and biocomposites at different fiber contents of 5, 10, and 15%wt as a function of the three different strain rates of 10^{-5} , 10^{-4} and 10^{-3} s^{-1} . To check the reproducibility of the tests, three specimens of each material were tested until failure at the three strain rates. For each condition, the mean curve was calculated, represented by a continuous line. An uncertainty area was drawn around each curve to highlight the error and repeatability of the tests. All the results show very good reproducibility of the tests.

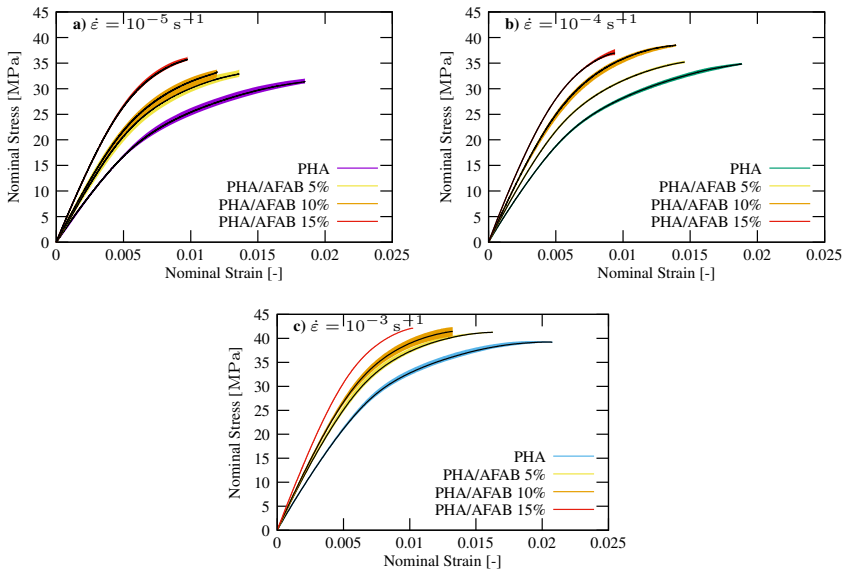


Figure 11. Nominal stress-strain curves of PHA and biocomposites with the different fiber contents of 5, 10, and 15%wt in the case of the three different strain rates of a) 10^{-5} , b) 10^{-4} and c) 10^{-3} s^{-1} (envelope curve showing test reproducibility).

These results highlight first the impact of strain rates on the PHA and biocomposites. The PHA shows a relative low ductility and elongation at break. This biodegradable material is brittle and cannot undergo any large deformation. However, the higher the strain rate, the greater its rigidity and strength. Second, with the addition of Alfa fibers, the result is the same:

adding AFAB to the matrix of PHA increases the rigidity and strength of biocomposites.

Young's modulus E , tensile strength R_m , and elongation at break A were determined to confirm these results for all conditions. Young's modulus was determined by fitting the linear part of the nominal stress-strain curves for strain ranging from 0 to 0.002 for all specimens. Table 2 and Figure 12 summarize these mechanical properties of each material tested in the case of the three strain rates.

Table 2. Evolution of Young's modulus E , tensile strength R_m , and elongation at break A for the different PHA/AFAB compositions and the three strain rates.

	PHA	5%wt	10%wt	15%wt
$\dot{\epsilon}=10^{-5} \text{ s}^{-1}$				
E [MPa]	3710±26	4325±55	4946±39	6241±102
R_m [MPa]	32±0.4	33±0.5	34±0.6	36±0.3
A %	2±0.1	1.6±0.1	1.2±0.1	0.95±0.1
$\dot{\epsilon}=10^{-4} \text{ s}^{-1}$				
E [MPa]	4155±6	4907±76	5544±60	6503±10
R_m [MPa]	35±0.1	36±0.5	37±0.5	38±0.2
A %	2±0.1	1.7±0.1	1.4±0.1	1±0.1
$\dot{\epsilon}=10^{-3} \text{ s}^{-1}$				
E [MPa]	4622±26	5397±33	5911±83	6921±17
R_m [MPa]	39±0.4	40±0.1	41±0.1	42±0.4
A %	2.2±0.1	1.75±0.1	1.5±0.1	1±0.1

These results confirm the increase in Young's modulus and mechanical strength, and the decrease in elongation at break, with increasing fiber content. In contrast, increasing the strain rate leads to an increase in Young's modulus and mechanical strength but a reduction in elongation at break. The increase in Young's modulus with fiber content (see Figure 12-a) can be attributed to the reinforcement provided by the fibers, which facilitates stress transfer from the matrix to the fibers. This result is consistent with the findings of previous studies [49, 50, 51].

The tensile strength (see Figure 12-b) of biocomposites is also greatly influenced by the adhesion between the fibers and the polymer [52]. The PHA/AFAB biocomposites exhibited higher tensile strengths, with a maximum value of 42 MPa recorded for the PHA reinforced with 15%wt AFAB.

Figure 12-c shows that the polymer PHA is very brittle and that adding fiber to PHA reduces elongation at break. In conclusion, the impact of

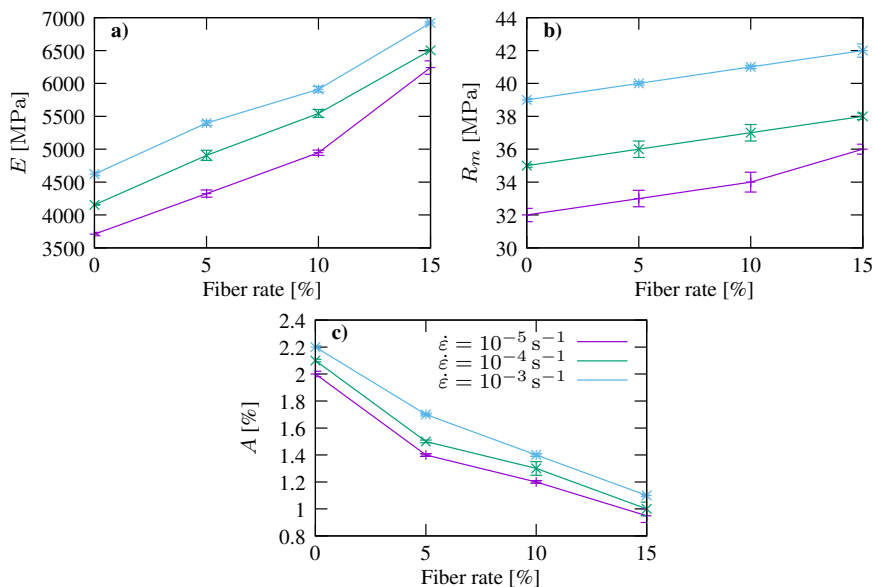


Figure 12. Evolution of the mechanical properties of PHA/AFAB biocomposite as a function of the fiber rate (in %wt) and strain rates: a) Young modulus E , b) tensile strength R_m , and c) elongation at break A .

the strain rate on the mechanical properties of biocomposites is non-negligible for all mechanical properties: the faster the strain rate, the more the mechanical properties are reduced.

Variation of Poisson's ratio in function of strain rate and fiber content

Before underlining the importance of Poisson's ratio in mechanics, it is important to provide a precise definition of this parameter. Poisson's ratio is simply the negative value of the ratio of transverse strain to axial strain in an elastic material subjected to uniaxial stress. In the literature, the Poisson's ratio for polymers like PHA is often established at 0.4. However, for biocomposites, these data are not always available and yet they are important for numerical modeling of the behaviour of these materials. That's why it is interested to determine this factor for PHA to confirm its value compared to the literature and subsequently for the biocomposites. The aim of this measurement is also to show the evolution of this ratio as a function of the strain applied during the test, and to analyze its dependence on strain rate and fiber content.

The minor-major strain curves and Poisson's ratio are presented in Figures 13 and 14 for the strain rate and fiber content, respectively. Poisson's ratio was obtained by a linear regression after 0.1% of strain due to the difficulty of the DIC measurement for small strain.

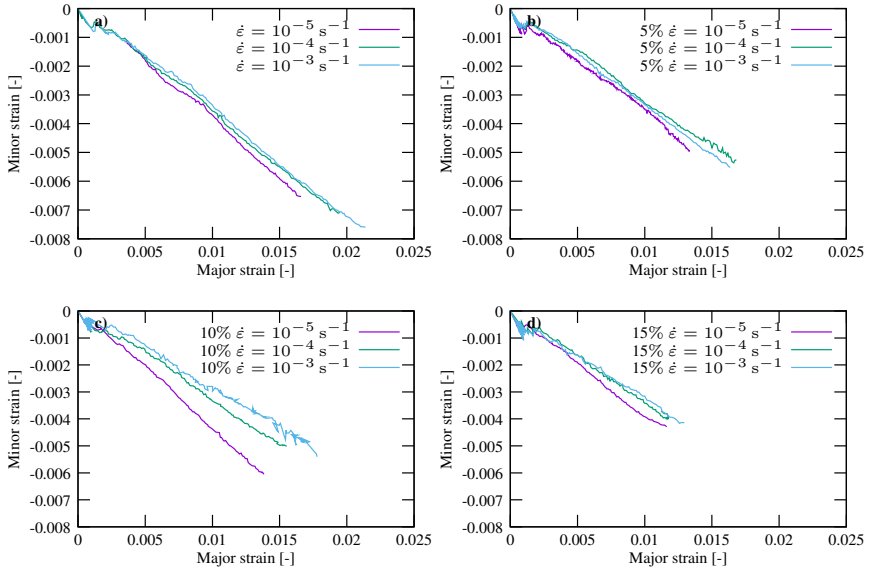


Figure 13. Influence of the strain rate in the minor-major strain curves in the case of PHA (a) and biocomposites with different fiber rates of 5, 10 and 15%wt (b,c,d).

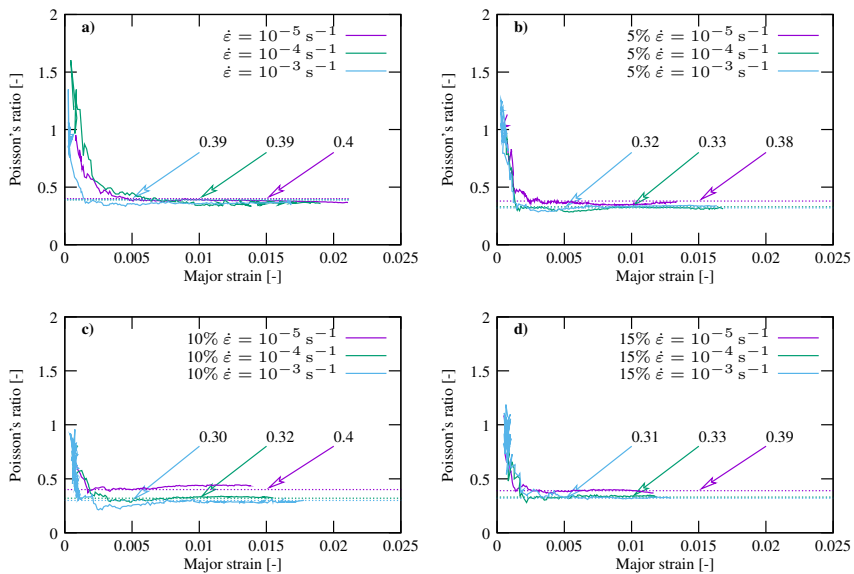


Figure 14. Influence of the strain rate in the variation of the Poisson's ratio as a function of the major strain in the case of PHA (a) and biocomposites with different fiber rates of 5, 10 and 15%wt (b,c,d).

Table 3 shows Poisson's ratio for each material and strain rates. The ratio is highest for the fiber-free material (around 0.39).

Table 3. Poisson's ratio of PHA and biocomposites according to the strain rate and fiber content.

$\dot{\epsilon}$ [s^{-1}]	PHA	5%wt	10%wt	15%wt
10^{-5}	0.40±0.1	0.38±0.1	0.40±0.1	0.39±0.1
10^{-4}	0.39±0.1	0.33±0.1	0.32±0.1	0.33±0.1
10^{-3}	0.39±0.1	0.32±0.1	0.30±0.1	0.31±0.1

At the slowest speed of $10^{-5} s^{-1}$, it is relatively constant (between 0.4 and 0.38) but decreases with faster speeds, tending towards a value of 0.31 with fiber contents between 5 and 15%wt. These results show that this material parameter depends on the fiber content and strain rates used and that these composites are nearly incompressible.

Analysis of variance of strain rate and fiber contents

ANOVA (Analysis of Variance) analysis was finally made with these results, and the statistical power of each factor was clarified. In this study, two main factors are examined: the strain rate (noted S) (10^{-5} , 10^{-4} and $10^{-3} s^{-1}$) and the Alfa fibers content (noted M) (PHA, PHA/Alfa 5%wt, PHA/Alfa 10%wt and PHA/Alfa 15%wt). These parameters are crucial for studying the effects of strain rate and Alfa fibers content on the experimental results. Tables 4 to 6 show the ANOVA results for Young's modulus, tensile strength and elongation at break.

Table 4. ANOVA table of Young's modulus.

Source	DoF	SSTR	MSTR	F statistic	P-value
Alfa fibers content M	3	9215518	3071839	337.34	0.000
Strain rate S	2	1647081	823541	90.44	0.000
Residual error	6	54636	9106		
Total	11	10917236			

Table 5. ANOVA table of tensile strength.

Source	DoF	SSTR	MSTR	F statistic	P-value
Alfa fibers content M	3	18.250	6.0833	73.00	0.000
Strain rate S	2	92.167	46.0833	553.00	0.000
Residual error	6	0.500	0.0833		
Total	11	110.917			

Table 6. ANOVA table of elongation at break.

Source	DoF	SSTR	MSTR	F statistic	P-value
Alfa fibers content M	3	1.91083	0.636944	150.36	0.000
Strain rate S	2	0.06125	0.030625	7.23	0.025
Residual error	6	0.02542	0.004236		
Total	11	1.99750			

These tables give the P-values which indicate the significance levels of each factor. Lower P-values indicate that the factor values have higher probability of falling within the ranges which impact the outcome of the experiment. Additional crucial indicators in ANOVA include degrees of freedom (DoF), treatment sum of squares (SSTR), treatment mean squares (MSTR), and F statistics values. These metrics collectively provide insights into the significance and impact of different factors on the experimental results, helping to determine the most influential variables on mechanical strength.

From the Tables 4, 5 and 6, P-value is less than the specified significance level of 0.05 [53]. That's why H_0 hypothesis is rejected. The results show that both factors, fiber content and strain rate, are statistically significant.

The F-value indicates the factor which has a more significant effect [54]. If the F-value of a factor is bigger than the other one, this factor's effect is more significant [54]. The results shows that fiber content is significant for Young's modulus and elongation at break, with the highest F-value giving the most significant effect on mechanical properties. However, with regard to tensile strength, the F-value shows that the strain rate is more significant than the fiber content.

SEM analysis of fracture surfaces

SEM images of fracture surfaces of the PHA/Alfa 10%wt biocomposite in the case of the three different strain rates of 10^{-5} , 10^{-4} and 10^{-3} s^{-1} are shown in Figure 15. The same observations were made for all fiber percentages in the matrix, and only the images with 10%wt of AFAB are presented.

Microscopic images show average adhesion between the PHA matrix and the AFAB. The investigation of the fracture facies shows that several fibers are fractured (see Figure 15-d,f). However, some fibers detach from the matrix rather than fracture (see Figure 15-a,b,c,e). To improve adhesion, the use of coupling agents, as the silane, is recommended to

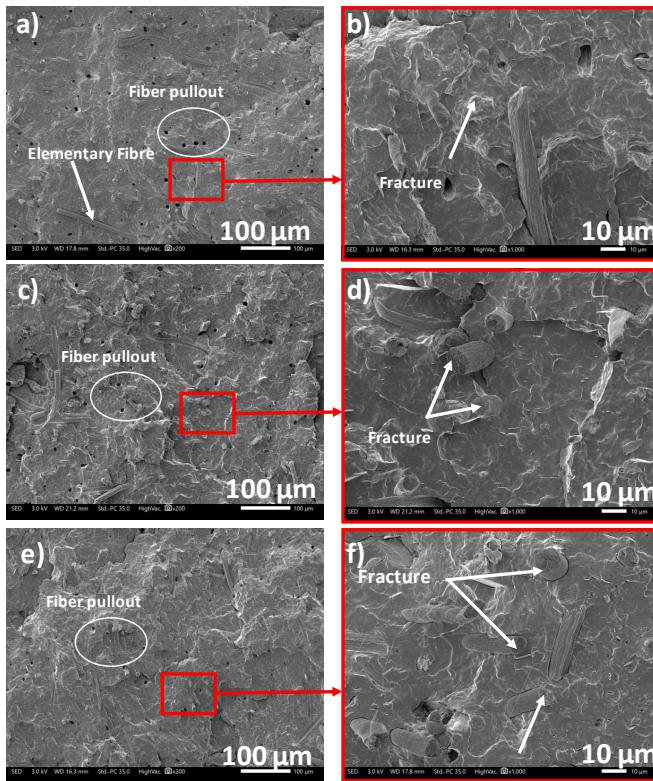


Figure 15. SEM analysis of the fracture surfaces of a PHA/AFAB 10%wt biocomposites at a strain rate of 10^{-5} (a,b), 10^{-4} (c,d) and 10^{-3} s^{-1} (e,f).

avoid this phenomenon and it would have been interesting to try this type of treatment to see its influence [55]. However, it is worth noting that in the case of the strain rate of 10^{-5} s^{-1} (see Figure 15-a,b), delamination was greater than in the case of the strain rate of 10^{-4} and 10^{-3} s^{-1} .

Conclusions

In this study, biocomposite materials based on Alfa cellulosic fibers as reinforcing element and PHA biopolymer matrix were prepared by mixing the fibers and the matrix under melted condition and mechanical shearing, followed by injection molding. Alfa fibers were extracted from stems using an alkaline method followed by a bleaching treatment. Mechanical tensile tests were performed with three strain rates to analyse the influence

of this parameter on the behaviour of this biocomposite. The mechanical response shows that the PHA is very brittle and the adjonction of fibers reduce also the elongation but improve the mechanical strength, which indicate a substantial reinforcement potential. This biocomposite is of interest for applications such as the automotive industry or packaging but which will have to undergo low elongations.

The main conclusion of the obtained results is summarized below:

1. The chemical and bleaching treatments have a significant influence on the surface of the fibers, making them rougher after these two processes, as well as a high cellulose content.
2. Increasing the fiber content increases Young's modulus and mechanical strength, but reduces ductility.
3. Increasing the strain rate increases strength and Young's modulus, reduces elongation at break.
4. Reduction in Poisson's ratio with the incorporation of Alfa fibers, particularly at high strain rates.
5. The analysis of variance (ANOVA) shows that both factors, fiber content and strain rate, are statistically significant.

It is therefore essential to take strain rate into account when testing biocomposites to verify their mechanical strength under conditions representative of their service life. To get a complete picture of the behaviour of this new material, it would have been interesting to test it at even faster loading speeds as carried out in the study of Zrida et al. [22]. Considering the pronounced sensitivity to moisture uptake of the biocomposites, further investigations should also needed to deduce guidelines for component design and performance for applications under typical climatic conditions as discussed in the article of Sobczak el al. [56].

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Research Data availability

Data will be made available on request.

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