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Up-cycling of agave tequilana bagasse-fibres: A study on the effect of fibre-surface treatments on interfacial bonding and mechanical properties

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ABSTRACT

The aim of this study was to assess the feasibility of upcycling fibre residues from the harvesting and production of tequila to green composites. Specifically, four different surface-modified natural fibres were assessed as raw material for green composite production. Before any surface treatment, the morphology and tensile properties of agave bagasse fibres from the tequila production batches were determined by optical and environmental scanning electron microscopy (ESEM) and single fibre tensile test, respectively. Further to this, agave fibres were exposed by immersion to four surface treatments including alkali, acetylation, enzymatic and silane treatments, in order to improve their morphology and compatibility with polylactic acid (PLA). The effects of these treatments on fibres' morphology, mechanical properties (i.e. Youngs modulus and ultimate tensile strength), interfacial shear strength (IFSS), and water absorption were assessed. Overall, surface treatments showed improvements in agave bagasse fibre properties with the best results for alkali treated fibres with an ultimate tensile strength of 119.10 MPa, Young modulus of 3.05 GPa, and an IFSS of up to ~60% higher (5.21 MPa) to that performed by untreated samples. These tests allowed to identify alkali treatment as the most suitable for agave bagasse fibres. These results shed light on the interfacial interaction between agave bagasse fibres and PLA and the potential to up-cycle these residue agave fibres to manufacture PLA-based green composites.

1. Introduction

Green composites from renewable resources such as natural fibres can be considered as more efficient, sustainable and biocompatible solution to the environmental and economic concerns arising from the large-scale production, and consumption of oil-based materials [1–4]. The use of natural fibres in composites has been the subject of intensive research over the past decades [5]. Moreover, the application of materials considered as waste or industrial and agricultural by-products as feed-stocks for composites has been a natural choice to make composites 'greener' [5].

Every year more than 300 000 tonnes of waste fibres are produced during the production of tequila [6]. However, the lack of financial resources hamper the tequila industry to tackling the environmental and financial burden posed by their vinasses and bagasse [7,8]. However, these residues streams could be reused to produce green composites as they have physical and mechanical properties similar to those shown by other high cellulose content fibres widely used in natural fibre based-composites [9,10]. Bagasse fibres are mainly composed of ~70% cellulose, and ~20% lignin and waxes [9]. Fibres chemical composition is rich in hydroxyl, carboxyl, and other functional groups; leading to a positive response to chemical modifications such as alkali treatments or acetylation [11–14]. Properties from agave tequilana fibres (ATF) are influenced by their cellulose content and structure. For example, higher cellulose content and a lower microfibrillar angle result in fibres with higher modulus and tensile strength with low elongation [15,16]. In contrast, fibres with higher lignin content, lower length/diameter (l/d) ratio and a higher microfibrillar angle may present lower strength and modulus with higher elongation. ATF are readily available as raw material source but currently have limited or no value. Therefore, the application of ATF as a filler or reinforcement may offer several advantages for green composite applications, such as low cost, low density, high toughness, and biodegradability.

ATF are non-abrasive to processing equipment, are CO_2 neutral when

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incinerated, have a low bulk density and perform well as acoustic and thermal insulators [17]. However, due to their lignocellulosic nature, ATF have poor interfacial bonding with polymer matrices and tend to form aggregates during compounding. They also have low resistance to moisture. These can cause decreased mechanical properties on the resulting composite material, and therefore limiting their wide spread use [18]. Other aspects to consider are the feedstock types, age, harvesting conditions, thermal and mechanical stresses during tequila extraction, and degradation during storage and handling conditions, which all influenced ATF properties.

Tensile properties of natural fibres (i.e. tensile strength and Young's modulus) have been used to determine fibres suitability for composites. ATF present a lower ultimate tensile strength and Young's modulus values compared with data reported for other agaves species, e.g. sisal and henequen, which are widely used as reinforcement for composites in the automotive industry and with a joint global production estimated at around 300 000 tonnes with a value of $75 million [19].

ATF are mainly composed of cellulose, hemicellulose, lignin, pectin, fat, waxes and water-soluble substances [20,21]; they require low production energy, are non-abrasive to processing equipment, are CO_2 neutral when incinerated, have a low bulk density and perform well as acoustic and thermal insulators [17]. Due to their ligno-cellulosic nature, natural fibres present poor interfacial bonding with polymer matrices, tend to form aggregates during compounding, and have low resistance to moisture. These can cause decreased mechanical properties on the resulting composite material, and therefore limiting their wide spread use [18]. However, to overcome these drawbacks, fibre compatibility through surface treatments is often used to improve the interfacial bonding, ease the compounding and improve the resulting properties for natural fibre-based composites [22,23].

Composites based on surface-modified natural fibres, in general, display better mechanical and physical properties due to improved adhesion and enhancement of polar interactions at the fibre/matrix interface that improve the reinforcing effect [10,24–27]. The degree of adhesion and the level of the interfacial shear strength between fibre and matrix are critical parameters that lead to superior quality composites. Several methods such as microbond test, fibre pull-out, three-fibre method and fibre push-out are used to determine the interfacial shear strength [28–30].

Dewaxing [13,31–37], coupling agents [36,38–41], alkali [11,12,31,36,38–47], and enzymatic treatments [48–54] have been the most commonly used methods reported in the literature for other natural fibres. The effect of these treatments has been explored in natural fibres such as kenaf, sisal, hemp, jute and coir [18,23,55]. However, to the best of the authors knowledge, the effect of surface modification treatments on ATF from tequila waste streams and their influence on interfacial adhesion with PLA has not been reported [56]. Thus, a better understanding of the physical properties and surface adhesive bonding of ATF may render this material as a feasible raw material source for developing green composites.

For example, alkali treatment is based on the principles of mercerisation [57]. Alkali treatment removes lignin, wax and other materials covering the external surface of the fibre cell wall positively affecting the properties of the composite material [11,12]. Therefore, this method can be used to increase adhesion of ATF with the PLA matrix by enhancing an effective surface area of the fibres. Furthermore, the process is widely recognised as a simple and cost-effective method [58].

Acetylation is another of the most studied reactions for natural fibres. This process has been used to address fibre compatibility issues and the swelling of the fibres in composite systems used in humid conditions [59]. The acetylation process substitutes the cell wall hydroxyl groups on the natural fibres with acetyl and propionyl groups, rendering the fibres surface more hydrophobic, and thus, more suitable for resin reinforcement. Acetylation has been used on oil palm and coir fibres [60], and on flax fibres [13,14] to improve the fibre dispersion and interface of the final composite.

Another effective and low-cost method is the enzymatic treatment [61] which is also referred to as bio-scouring method, to achieve the delignification or removal of non-cellulosic compounds from the fibres [48,53,54,62]. ATF and other natural fibres also contain large amounts of pectin which can adsorb moisture in both crystalline and amorphous regions [9,63]. The selective removal of pectin from ATF can prevent any future problems that may arise from the pectin without disrupting the cellulose structure as this contributes to fibre strength.

The aim of this research is to determine the potential of agave bagasse as a raw material source for natural fibres and green composites. In particular, this study evaluates the properties of ATF and the effect of four treatments including acetylation, silane, alkali and enzymatic surface treatments on the fibre's properties in terms of surface morphology, water absorption, tensile properties and interfacial adhesion between ATF and PLA through pull-out test.

2. Materials and methods

2.1. Agave fibre sampling and materials

10 kg of dry bagasse was provided by "La Fortaleza" distillery (Mexico) after being steam cooked (for 24 h at 60 °C), and milled to extract the juice to produce tequila. Dry ATF were randomly selected from the bagasse provided and divided into five different batches of 200 g each. Every ATF batch was repeatedly washed with tap water to ensure the removal of the residual matter from the tequila extraction process. ATF batches were finally oven dried for 24 h at 60 °C and kept in desiccators at ~21 °C and 47 ± 3% relative humidity (RH).

For fibre treatments, all chemical materials were analytical grade and supplied by Acros organics (UK). Pectate lyase enzyme was provided by Novozymes (Denmark) and polylactic acid (PLA) was provided by Mitsui & Co Europe PLC (Table 1 shows typical PLA properties).

Before any treatment, a 200 g batch of ATF was taken apart for further characterisation, and kept as a control sample. Hereinafter this is referred as untreated fibres (UNF). The remaining four ATF batches were exposed to alkali, acetylation, enzymatic and silane treatments by immersion. All treatments were carried out in triplicate using 50 g of fibres. Treatment conditions used for the different treatments are summarised in Table 2. The treatments protocols were based on a screening study and previously published works on different natural fibres [54,65,66]. Different sets of treated specimens are referred hereinafter as alkali-treated fibres (AKF), acetylated fibres (ACF), enzyme treated fibres (ENF), and silane-treated fibres (SIF).

2.2. Structure and morphology

Structure and morphology of treated and UNF samples was

Table 1
Typical properties of PLA [64].

Property	Value
Density	1.24 g/cm^3
Tensile strength MD	110.1 MPa
TD	144.5 MPa
Tensile Modulus MD	3302 MPa
TD	3852 MPa
Elongation at break MD	160%
TD	100%
Elmendor tear MD	15 g/mL
TD	13 g/mL
Spencer impact	2.5 J
Haze	2.1
Gloss, 20°	90
Glass transition	52–58 °C
Melting point	150 °C
Molecular weight	50 kDa

MD = Machine direction, TD = transverse direction.

Table 2
Agave tequilana fibres (ATF) surface treatments.

Sample	Solution	Conc. (%)	Treatment Temp (°C) and time (min)	Drying Temp (°C) and time (hour)
UNF	Untreated fibres	–	–	–
AKF	(w/v) NaOH water solution	3%	21 °C/60 min	60 °C/ 24 h
		8%	21 °C/180 min	
ACF	(w/w) Toluene + Acetic anhydride + 0.026% perchloric acid (70%)	70:30	60 °C/60 min	60 °C/ 24 h
		50:50	60 °C/120 min	
ENF	(weight percentage compared to the fibre) pectate lyase + 0.5 mL non-ionic wetting agent in a buffer solution pH 8.3	0.4%	55 °C/30 min	60 °C/ 24 h
		2%	55 °C/60 min	
SIF	3-aminopropyltriethoxysilane (weight percentage compared to the fibre) hydrolysed in a water-ethanol solution (40:60 w/w) with a pH 5.5, 1 h stirred previous treatment.	3%	25 °C/60 min	60 °C/ 24 h
		10%	25 °C/240 min	

AKF: alkali-treated fibres; ACF: acetylated fibres; ENF: enzyme treated fibres; and SIF: silane-treated fibres.

characterised using optical microscopy (Leika, Germany) and environmental scanning electron microscopy (ESEM) (FEI XL30). Energy dispersive X-ray (EDX) was performed to determine the elemental composition of deposited materials on the fibre surface.

To determine changes in morphology, five dry ATF were randomly selected from each of the untreated and treated batches. Fibres were mounted with carbon tape on aluminium stubs for analysis at room temperature and under vacuum, using an ESEM (FEI XL30) and according to a method reported by Sgriccia et al. [40].

Prior treatments and in order to determine the fibre length, 200 randomly selected UNF were selected from the 200 g of UNF batch, and were mounted onto a thick paperboard and scanned at high-resolution; images obtained were then processed and measured using ImageJ software [67].

Due the non-homogeneity of fibre length and the scatter in diameter across the fibres observed by planimetering [68,69], cross sectional area (CSA) was determined using the following method [70]: individual fibres were held together in groups of 10 and embedded vertically in epoxy. Samples were then grounded and polished; cross sections were photographed at magnification 50X. The process was repeated at every 2 mm steps along the 20 mm length of each sample to provide 7 different micrographs for every sample. Each micrograph was then processed and analysed using ImageJ software to find the CSA. 350 measurements were taken to determine CSA.

2.3. Fibre treatments

Firstly, fibres were immersed in the correspondent aqueous solution for each treatment. Then, the fibres were drained and rinsed with distilled water until acid-free. This also allowed the removal of physisorbed compounds from the fibre surface. Finally, all samples were kept for 24 h in an oven at 60 °C to remove moisture, and, in the case of SIF, to complete the chemical reaction (as polysiloxanes are mainly prepared by a multistep "hydrolytic polycondensation" consisting of elementary hydrolysis and condensation reactions). Samples were finally kept in desiccators at ~21 °C and 47 ± 3% RH.

2.4. Mechanical properties

Single fibre tensile tests were performed on the five different ATF batches using an INSTRON 5500R EM with a 100 N load cell and strain rate of 0.5 mm/min according to ASTM D3379-75 [69]. Single fibres were individually mounted using a thick paper tab with a slot of 30 mm length equal to the gage length cut out in the middle of the tab. Samples were randomly chosen and pasted at both ends of the slot in the paper tab using fast glue. The precise gauge size of the fibre was measured using a Vernier calliper to the nearest 0.1 mm. Seven samples for each treatment presented in Table 2 were tested with the ultimate tensile strength calculated using Equation (1).

$$T = \frac{F}{A} \quad (1)$$

where T is tensile strength, F is the maximum load recorded during test, and A is the mean CSA determined using a method previously reported [70]. From the data obtained for every specimen tested, the strain was also calculated using the following equation (2):

$$\varepsilon = \frac{\Delta L}{L_0} = \frac{L - L_0}{L_0} \quad (2)$$

where ΔL is the change in gauge length, L_0 is the initial gauge length, and L is the final length. Young's Modulus (E) was calculated by dividing the stress (σ), by the strain (ε), in the elastic portion of the stress-strain curve. The obtained stress-strain data from 60 samples was processed using OriginPro software to plot behaviour presented by ATF during tensile stress [71].

2.5. Density

To determine ATF volume density, 20 samples for each batch of treated and UNF were oven dried until constant weight, and linear density was calculated by measurement of the mass and length using an analytical balance with a precision of $1{\cdot}10^{-5}$ g and an optical microscope at magnification 5X, respectively [72]. The average linear density and diameter were used to calculate the volume density of the fibre.

2.6. Water absorption at saturation

Water absorption was determined by immersion. Samples composed of bundles of every treated and untreated fibres were dried at 60 °C for 24 h, weighed in an analytical balance with a precision of $1{\cdot}10^{-4}$ g, and placed inside a beaker containing distilled water at room temperature (25 °C). The weight difference was measured at intervals of every hour until saturation and moisture content (MC) was calculated using:

$$MC = \frac{w_1 - w_0}{w_0} \times 100$$

where w_0 is the mass of dry sample and w_1 is the mass after immersion. Measurements were done by removing the fibres from the beaker, wiping them dry and weighing them with a precision of $1{\cdot}10^{-4}$ g. Three replicates for every treated and untreated set of fibres were completed.

2.7. Interfacial shear strength: pull-out test

For the preparation of pull-out test coupons, 4 mm diameter cylindrical shaped moulds were filled with PLA micro-pellets, and placed inside an oven heated at 175 °C for 10 min. Single randomly selected fibres from each treated and UNF batch were then embedded vertically into the cylinder-shaped PLA matrix at 175 °C, then allowed to cool in air at room temperature (Fig. 1). Further details on sample preparation are cited by Morlin and Czigany [73]. The fibres' CSA and embedded length were measured using an optical microscope at magnification 5X. Samples were aligned with the axis of the cross-head on an INSTRON 5500R EM with a 100 N load cell at room temperature. The load was applied by gripping the resin cylinder with a steel holder fixed to the lower machine clamp

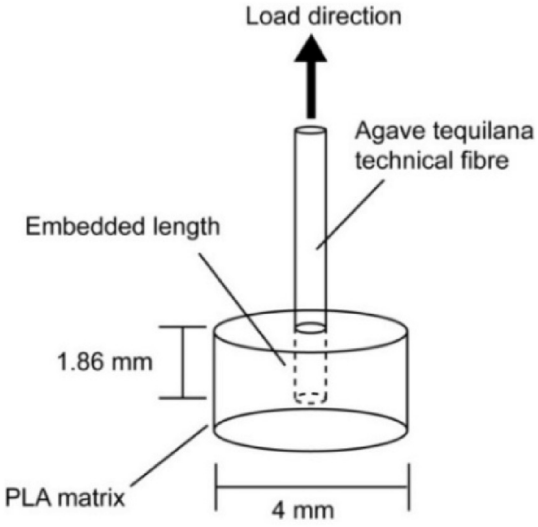


Fig. 1. Schematic view of the pull-out specimen.

and pulling from the free end of the fibre at a strain rate of 0.35 mm/min. The load and displacements were recorded until the fibre pull-out completed. The procedure was repeated for every set of treated and untreated fibres, and 15 samples from every group were tested. From the data recorded, the debonding force was converted into interfacial shear strength (IFSS) (τ) using:

$$\tau = \frac{L}{D \quad le}$$

Where L is the maximum load recorded during debonding, le is the embedded length and D is the fibre diameter obtained directly from CSA measurements of each sample.

3. Results and discussion

3.1. Properties of untreated ATF

The tequila extraction process leaves the agave bagasse nearly ready for its use as source of fibres and with promising properties similar to other natural fibres used in composites [35,72,74]. UNF obtained from this source were still arranged in bundles covered by non-cellulosic materials. Table 3 summarises the properties of the untreated ATF. Mean ATF length was found to be 77.11 mm with a mean CSA of 74.99 μm. Mean ATF density was found to be 1.19 g/cm^3.

In terms of mechanical properties, ATF exhibit significant variability in tensile strengths and serration effects during tensile tests. Ultimate tensile strength (UTS) for UNF was 79.35 ± 9.71 MPa at a 95% confidence level (CL). This is a common characteristic of natural fibres [75,76]. It can be explained by the structure and number of defects present within the fibres. Under stress, cracks propagate through weak bonding between cells, causing intercellular fracture without the removal of microfibrils causing fibre failure. The long elongation ATF presented was primary a result of microfibrils orientation and angle. Mean stress-strain plot of ATF (Fig. 2) shows ductile failure (see Fig. 3).

UTS and Young's modulus (E) values from ATF (see Table 3) were found to be higher (~36–90%) than those previously reported for the same agave species within the waste stream (i.e. agave tequilana 41.5–58.1 MPa) [9], while the strain was in agreement (12–15%). This may be due to the CSA (affected by harvesting method and fibre extraction conditions) since there is a significant relationship between CSA and UTS ($p < 0.05$). However, E and UTS values were lower in comparison to data reported for another agave species (i.e. agave sisalana 385–577 MPa) [87] widely used and produced specifically as reinforcement for composites. The lower mechanical properties of ATF used in this study, in comparison with other non-waste natural fibres [18,74,77,85,86] is likely to be the result of mechanical and thermal stresses during tequila production. These processes might have partially removed cementing materials that kept the cohesion between fibre cells. Nevertheless, ATF fibres presented mechanical properties in similar ranges when compared to other agricultural wastes used as fibre source. Therefore, it is possible to consider the use of ATF as reinforcement filler for polymeric materials in low-performance applications, as it can be appreciated when compared to other natural fibre properties used in composites (Table 3).

3.2. Effect of surface treatments on the morphology of ATF

Fibre morphology is important to predict fibre interaction with the polymeric matrix. UNF showed non-uniform geometrical characteristics. Fibres were still assembled into bundles. Each fibre consisted of two cell walls arranged as concentric tubes with a middle lumen (Fig. 4a). Fibre cells were united by the middle lamellae, which consisted of cellulose, lignin, pectins and hemicelluloses [88]. Therefore, UNF surface area is reduced and still not fully accessible to PLA. Furthermore, the presence of other minor components, such as waxes, inorganic salts and nitrogenous substances [63] could result in an increase of adhesion in a fibre-polymeric matrix interface in the production of composites. Additionally, the longitudinal section of UNF reveals a non-constant transverse dimension and a large number of flaws from its natural origin and extraction process that could cause early fibre failure if put under tensile stress (Fig. 4a).

ESEM micrographs showed differences in the surface morphology of fibres depending on the treatment applied: AKF (Fig. 4b) and ENF showed relatively detached individual cells after treatment. Bundles presented a cleaner surface and appeared more separated due the elimination of the interfibrillar material, hemicellulose and lignin in comparison with ACF and SIF. Therefore, an increase of surface area and roughness on fibres occurred after fibres were treated with NaOH and

Table 3
Summary of properties of untreated agave tequilana fibres (ATF) in comparison with other natural fibres.

	Fibre type	Density (g/cm3)	Length (mm)	Diameter (μm)	UTS (MPa)	E (GPa)	Elongation (%)	Ref.
	ATF	1.19	77.11	74.99*	79.35	3.29	13.09	Current study
common fibres for composites	Bamboo	0.6–1.1	1.5–4	25–40	140–800	11–32	2.5–3.7	[77]
	Flax	1.4	10–65	5–38	800–1500	60–80	1.2–1.6	[18,74]
	Hemp	1.48	5–55	10–51	550–900	70	1.6	[18,74]
	Jute	1.46	0.8–6	5–25	400–800	10–30	1–1.8	[18,74]
	Sisal	1.33	0.8–8	7–47	600–700	38	2–3	[18,74]
by-products/waste	Sugarcane bagasse	1.2	0.8–2.8	10–34	20–290	19.7–27.1	1.1	[74]
	Banana	1.35	0.17	13.16	355	33.8	5.3	[78–80]
	Coir	1.15–1.46	20–150	10–460	95–230	2.8–6	15–51.4	[81]
	Rice straw	–	0.4–3.4	4–16	–	–	–	[82,83]
	Wheat straw	–	0.4–3.2	8–34	–	–	–	[84–86]

*CSA = cross-sectional area; SD - standard deviation; E – Young's modulus; UTS – Ultimate tensile strength.

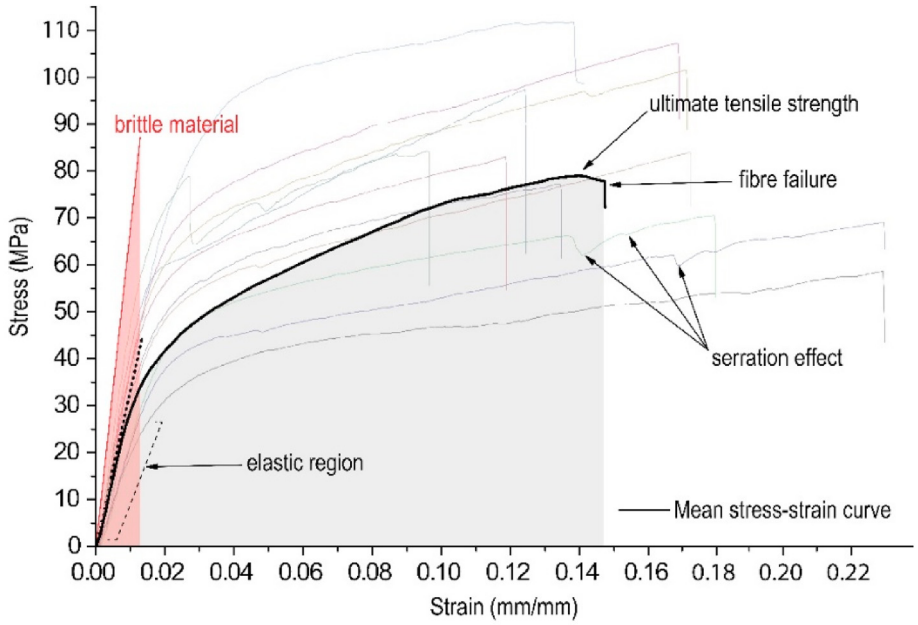


Fig. 2. Agave tequilana fibres tensile behaviour. Mean stress-strain curve from experimental data (60 specimens tested).

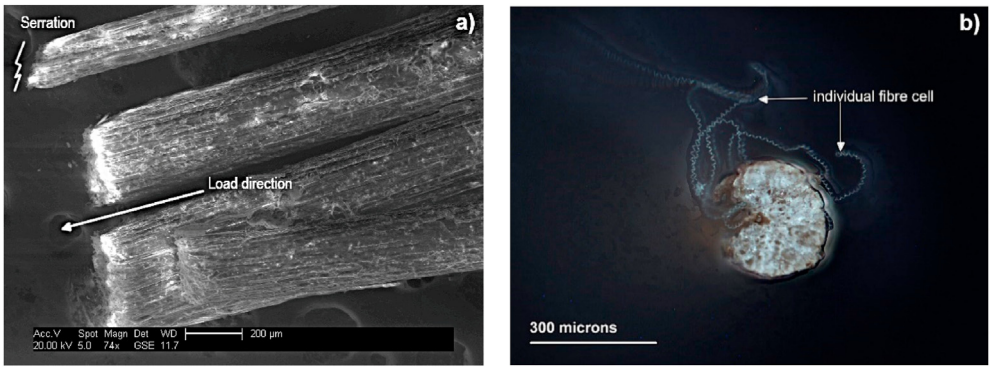


Fig. 3. (ESEM) and 3 b (optical microscopy) show the fracture mode of ATF and the separation between fibre-cells. This individual fibre breakage at different planes might account for non-linear region observed prior to fibre failure.Agave tequilana fibre tensile failure: a) ESEM image of ATF showing failure mode after tensile test (magnification 74X); b) Micrograph of CSA of ATF after fibre failure.

pectate lyase enzyme. Consequently, an increase of adhesion in a fibre-polymeric matrix interface could be expected as these two main changes can promote a better mechanical interlocking [89].

ESEM images further showed a clear improvement in fibre surface morphology with a more uniform and smoother surface for acetylated fibres (ACF) (Fig. 4d). This indicates that fibres were successfully coated, and plasticized by the replacement of the hydroxyl groups from the cell wall with new acetyl groups, making the fibres hydrophobic.

For ENF (Fig. 4c), partial fibrillation was observed due to the breaking down of the fibre bundle into smaller fibres. This resulted in the exposing of helical fibrils, by the action of pectate lyase enzyme leaving a considerable surface roughness, and increasing the effective surface area available for contact with the matrix. However, the fibre structure still had non-cellulosic materials attached to the surface. This may be attributed to the incomplete activity of the enzyme to break the pectin molecules in partial areas of the fibre [53]. Roughness was not greater than that exhibited by alkali treated samples (Fig. 4b). An extended enzymatic treatment time, and constant pH monitoring, may be required to further eliminate undesirable cementing materials and thus ensure optimal enzyme activity [54].

The forming of a polysiloxane coating bonded to the surface of ATW fibre is shown in Figure 4e (SIF). The R-groups from this layer are likely to react in further composite preparation with functional groups present in PLA, forming a stable covalent bond working as a bridge between ATF and PLA. Energy dispersive X-ray microanalysis (EDX) was conducted to confirm the presence of a 3.75% of silicon (Si) content bonded to the cellulose fibre surface for silane-treated samples. ATF morphology can be positively modified to improve fibre-polymeric matrix interaction through chemical (i.e. alkali treatment) and enzymatic treatments (i.e. bioscouring). These processes delivered an active surface by the introduction of reactive groups or by the partial removal of lignin, waxes, and hemicellulose.

3.3. *Effect of surface treatments on fibre strength*

All surface treatments increased to some extent the strength of ATF. Ultimate tensile strength (UTS) of treated samples was in general higher than UNF, and in particular AKF samples presented the best results. Table 4 shows a summary of the surface treatments effect on the mechanical properties of ATF in comparison with untreated fibres. UTS of treated samples was observed to increase up to a certain maximum value with the increase of time of treatment as shown in Fig. 5. From this sweet spot onwards, the increase of treatment time reduces the UTS of the fibres. All treated fibres continued to display brittle failure within the

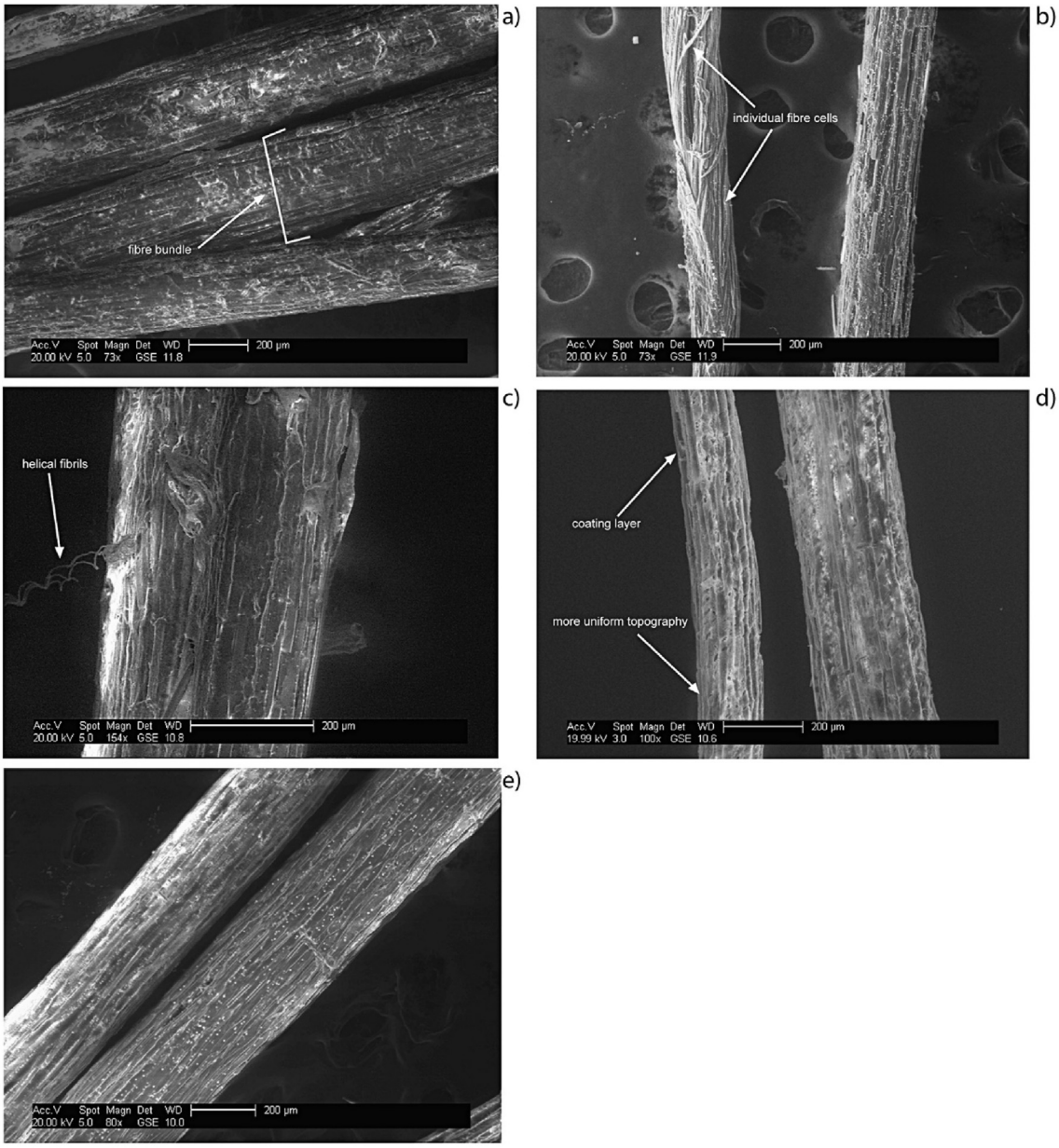


Fig. 4. ESEM of untreated and treated agave tequilana fibres: a) UNF presenting a closely packed fibre bundle; b) AKF (NaOH 8% - 180 min) showing loose fibre bundles; c) ENF (pectate lyase 4% - 60 min) showing partially loose fibre bundles; d) ACF ($(CH_3CO)_2O$ 50% - 120 min) showing a more uniform and coated fibre bundle; e) SIF (APTES 10% - 60 min) presenting a uniform fibre bundle.

Table 4
Summary of Agave tequilana fibres properties after surface treatments.

Sample	Density (g/cm^3)	CSA (μm^2)	UTS (MPa)	*E* (GPa)	Strain (%)
UNF	1.13	74.99	79.35	3.29	13.09
AKF (8% - 120 min)	0.88	58.61	119.11	3.06	24.82
ACF (50% - 60 min)	1.11	73.59	98.76	3.35	12.98
SIF (10% - 240 min)	0.86	57.16	86.79	2.89	16.69
ENF (0.4% - 180 min)	0.99	65.99	93.46	2.95	17.97

maximum applied stress as in the case of untreated fibres.

AKF samples treated with 8% NaOH for 120 min present the best outcome in terms of mechanical properties, with UTS of 119.10 MPa, *E* of 3.05 GPa, and strain at breakage of almost 25% (i.e. 47% higher than the value of UNF). Alkali treatment is likely to allow moisture to penetrate into fibres creating porosity and voids that promote the growth of cracks during tensile tests. The good results may be attributed to a change in the chemical structure of cellulose within the fibres. Cellulose molecular chains in the microfibrils lose their crystalline structure as a result of the alkali treatment, and fibrils become more capable for rearranging themselves along the direction of tensile deformation. Hence, alkali treatment results in a higher stress development on ATF due to better load sharing among fibres [25]. Other studies have reported increase in mechanical properties (tensile modulus and tensile strength) of Agave fibres treated in alkali solutions [90]. ACF showed the second-best results with UTS of 98.76 MPa and *E* of 3.34 GPa, related to the forming of an additional coating layer to the fibre surface.

Single fibre tensile test results of natural fibres are difficult to analyse due to widely scattered values [91]. Hence, experimental data from treated and untreated ATF was analysed using a two-parameter Weibull

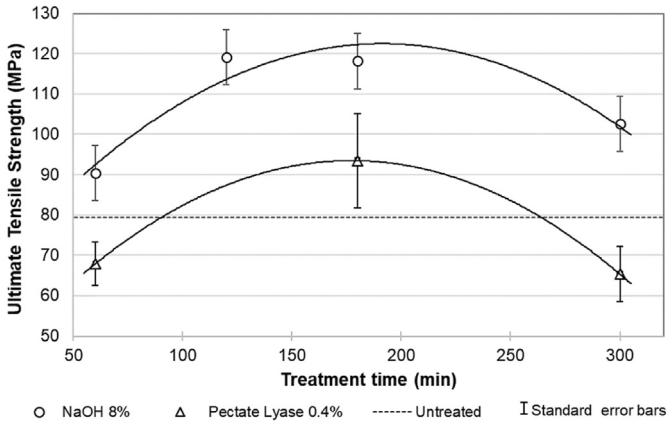


Fig. 5. Effect of treatment time on ultimate tensile strength for treated agave tequilana fibres: AKF samples treated with 8% NaOH solution and ENF samples treated with 0.4% pectate lyase (7 samples tested per each treatment).

distribution [92]. Results presented a good fit with Weibull distribution at 95% confidence level, having an $R^2 = 0.94$ for UNF as seen in Fig. 6.

3.4. Effect of surface treatments on water absorption at saturation

In general, ATF are hygroscopic by nature. ATF samples absorbed water very rapidly during the first stages of the test (0–60 min), reaching an equilibrium point, where no more water was absorbed and the content of water in the samples remained stable (Fig. 7). As it was expected, UNF showed the highest percentage of water absorption (up to 47%). The hydrophilic character of cellulose is responsible for the water absorption in ATF, and therefore the higher cellulose content led to the higher amount of water being absorbed. Every treatment showed improvements in water absorption. ACF shows the best results with 29% of water absorption after 6 h of exposure. This reduction in water absorption was expected for ACF as acetylation has been reported to create an additional hydrophobic layer on the surface of other natural fibres [31,32,60]. Fibres treated with pectate lyase, silane, and alkali treatments had a water absorption between 35% and 37% after 6 h of exposure.

This reduction in absorption properties was attributed in the case of ENF and AKF to the partial removal of lignin and hemicellulose, and to the change of fibre's hydrophilic nature to a more hydrophobic one, as both treatments promote the activation of hydroxyl groups in cellulose by breaking the hydrogen bonds.

3.5. Effect of surface treatments on interfacial shear strength (IFSS) between ATF and PLA

Load versus displacement curve for the pull-out tests of PLA and ATF samples is presented in Fig. 8. During the tests, the debonding process started just after the elastic deformation with crack propagation. Once reached the maximum stress, the fibre was consistently withdrawn from the matrix. Finally, there was a progressive reduction of stress with an extension trace (stage IV).

The non-linear behaviour from the traces (Fig. 9) shows the difference in the trend of the curves dropping after the load reached its maximum value. As the UNF gradually reached the maximum load (average = 4.85 N) there was a generally smooth transition (dynamic sliding), followed by a linear decrease with occasional stick-slip activity (stage IV) until the fibre was finally pulled-out from the matrix. This is a consistent behaviour from a weak interphase that has been also observed in other natural fibres where friction of fibre-matrix occurs due to uneven and bump surfaces of the natural fibres [93]. Consequently, resulting in the low compatibility between ATF and PLA. In contrast, for AKF and ENF, this behaviour differed as a result of the higher roughness on the fibres surface, being the mechanical interlocking the main reason for the variability presented for these samples as shown in Fig. 9.

For silane-treated and acetylated fibres, the load-displacement trace shows a weak bonding in comparison to AKF treated samples. Consequently, limiting the use of these treatments for load bearing applications as this reduced interfacial compatibility with PLA may represent an earlier crack propagation and composite failure due to an inappropriate transfer of load. The main factor improving the bonding between phases is the increment in effective surface area due to the removal of non-cellulosic materials [89]. Notably, after interface failure, the curve decreased in an almost linear fashion, which is more evident for acetylated fibres, until the fibre was pulled-out leaving small traces of friction.

Every treatment had a positive effect on the IFSS. AKF exhibited the best results with a maximum increase in IFSS of up to ~60% when compared with untreated fibres as it is shown in Fig. 10 and Fig. 11. This increased IFSS evidences the improvement in bonding within the PLA matrix. The highest IFSS, with a mean value of 5.21 MPa, was obtained for the samples treated with 8% NaOH for 60 min. This can be attributed to an increase of available hydroxyl (–OH) groups from the alkali treatment, the removal of the non-cellulosic materials, and to the increased roughness in the surface area of the fibre (Fig. 4a). These results are in agreement with other studies. For instance, Mylsamy and Rajendra [12] studied the reinforcement of epoxy resin with untreated and alkali treated short agave fibres. They found that alkali treated fibres give better mechanical results than untreated fibres, as they consider that the alkali treatment facilitates more sites for fibre–resin interface. Geethikaa and Rao [94] also used alkali treated agave fibres in their study. They stated that the alkali treatment removed the cementing material in the fibres (lignin and hemicelluloses). This resulted in an increase of the fibres' surface area and, as a consequence, the adhesion between fibre and matrix derived in improved tensile properties [65].

ENF samples presented a similar surface roughness from the enzymatic treatment (Fig. 4c), although to a lesser extent with minor variations when compared to the UNF samples. ENF at 0.4% concentration for 60 min presented the second best IFSS with a mean value of 4.03 MPa. Thus, a better mechanical interlocking promoted by alkali and bio-scouring treatments within a time range of 60–160 min can be considered as the main factor improving the bonding between ATF and PLA (Fig. 10).

ATF exhibited variability in tensile properties as well as in IFSS, which is quite common to natural fibres. Therefore, the values obtained by pull-out test were statistically analysed using a Weibull distribution. Fig. 11 shows that there is a reasonably good agreement between experimental IFSS data and the two-parameter Weibull distribution.

The effects of all the treatments on the properties of ATF are summarised in Table 5. Although there is not a single treatment that improves all the properties of ATF, the alkali treatment (AKF) is the one with better balanced effects. This surface treatment increases UTS and strain at break, and presents the higher value of IFSS as a result of mechanical interlocking between treated fibres and matrix. On the other hand, the water absorption is still considerable with this treatment. Depending on the application of the ATF and polymer and final fate of the material, water absorption could be desirable or not. In the case of biodegradable materials, water absorption could promote the degradation, thus it would be a desirable property.

4. Conclusions

This study showed that ATF residues from the tequila production, can be used as reinforcement fibres for PLA-based green composites. This offers several advantages for PLA alongside other green composite

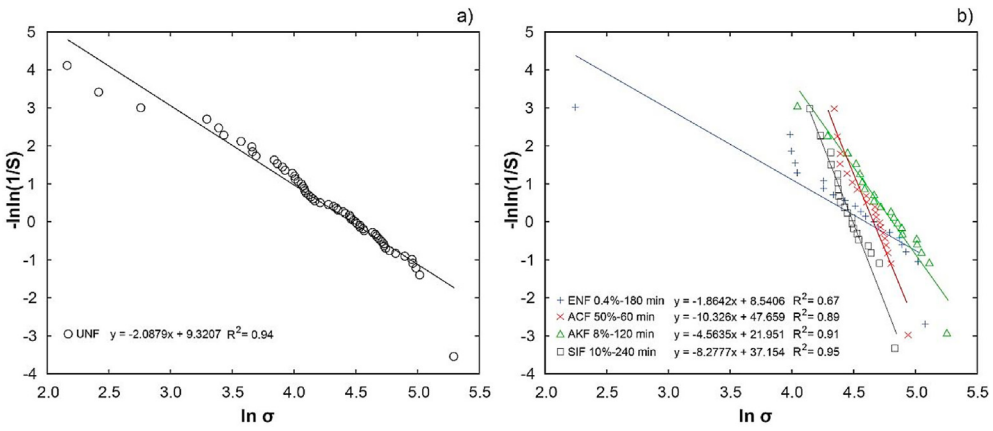


Fig. 6. Weibull distribution for ultimate tensile strength for Agave tequilana fibres. a) Plot of –ln ln (1/S) vs ln σ for the tensile strength for UNF; b) Plot of –ln ln (1/S) vs ln σ for the tensile strength for all treated ATF.

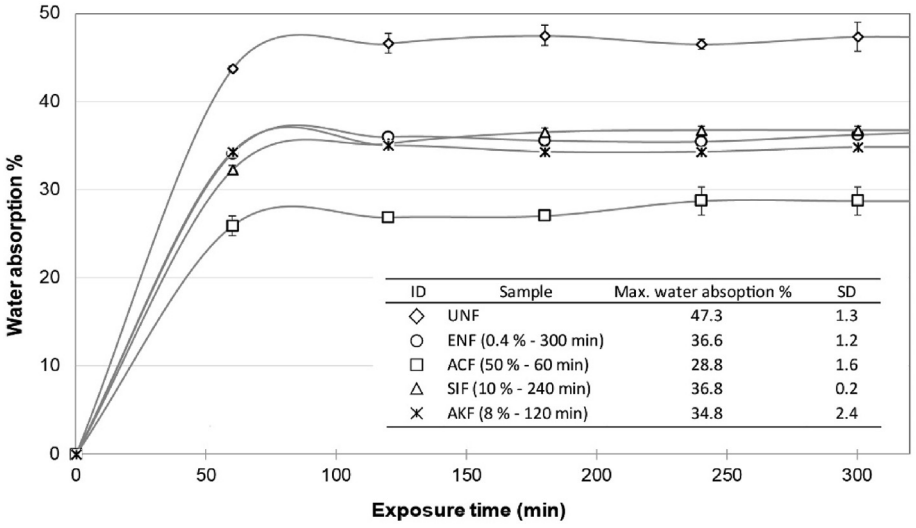


Fig. 7. Water absorption at saturation for agave tequilana fibres after surface treatments. Standard deviation marks are smaller than the markers in some cases.

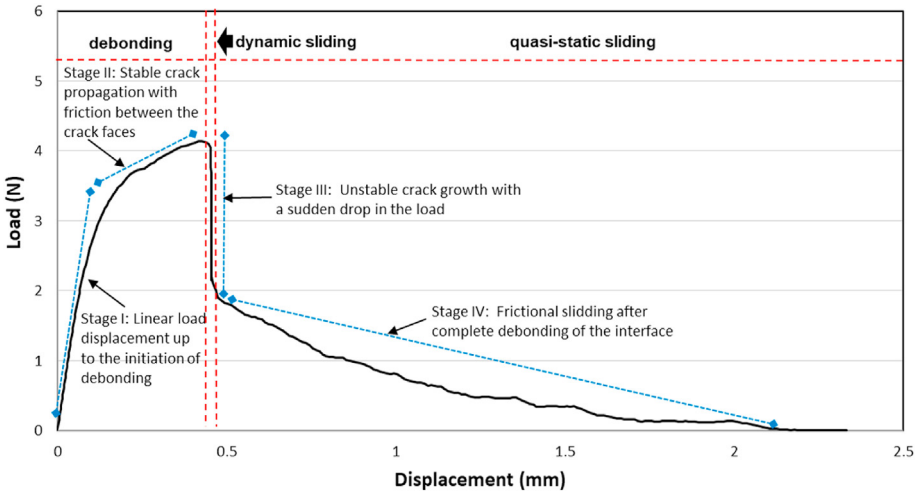


Fig. 8. Experimental load versus displacement curve for a fibre pull-out test for PLA and untreated ATF.

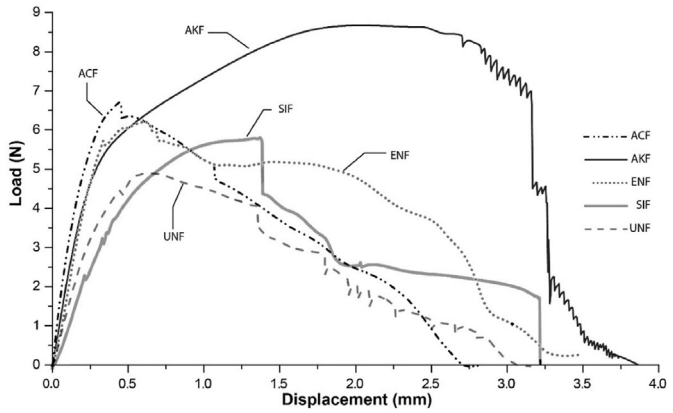


Fig. 9. Typical Load-displacement traces from untreated and treated agave tequilana fibres during pull-out test. Load-displacement traces were obtained from the average of at least 18 samples tested.

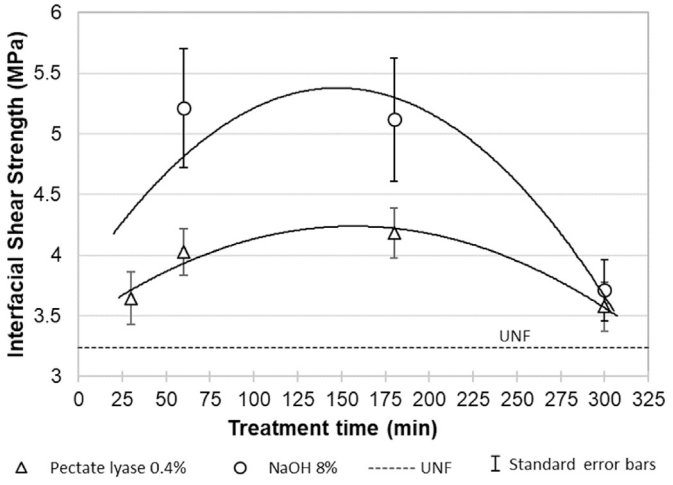


Fig. 10. Effect of treatment time on interfacial shear strength for agave tequilana fibres treated with 8% NaOH and 0.4% Pectate Lyase.

Table 5
Summary of the effect of fibre surface treatment on mechanical properties, water absorption and IFSS between ATF and PLA.

Fibre Treatment	Mechanical properties			Water absorption	Interfacial shear strength
	UTS (MPa)	*E* (GPa)	Strain (%)	(at saturation)	(IFSS) (MPa)
UNF	79.35	3.29	13.1	47%	3.24
AKF (8% - 120 min)	119.11	3.06	24.8	35%	3.07
AKF (8% - 60 min)	90.38	3.11	21.4	47.2%	5.21
ACF (50% - 60 min)	98.76	3.35	13.0	29%	1.54
SIF (10% - 240 min)	86.79	2.89	16.7	37%	2.19
ENF (0.4% - 180 min)	93.46	2.95	18.0	37%	4.03

applications, such as; low cost, low density, moderate toughness, acceptable specific strength properties, and biodegradability.

The bonding between the PLA matrix and ATF is key for an optimal performance of any future ATF-based green composites. This can be improved by surface treatments. ATF tensile properties, interfacial bonding between PLA and ATF, and absorption properties are improved by surface modification methods. AKF demonstrated the best results for ultimate tensile strength and interfacial shear strength in comparison with the UNF samples with an increment of approximately 50% and 60%, respectively. Therefore, a higher fibre load content could be achieved through the use of treated ATF. Increased roughness in the surface area of the fibre and the removal of the non-cellulosic materials demonstrated by alkali and enzyme treated fibres are considered to be the main factors improving interfacial bonding. In terms of water absorption, ACF showed the best results with only a reduction of water intake of 18% compared to untreated fibres. The absorption of water could lead to the degradation of the fibre, thus compromising the mechanical properties and the application of ATF in fibre reinforced composites. However, this could be beneficial when considering biodegradable composites. In summary, it is possible to envision a green composite material based on treated ATF and PLA capable of performing under low stress conditions without compromising the integrity of the final composite product or the

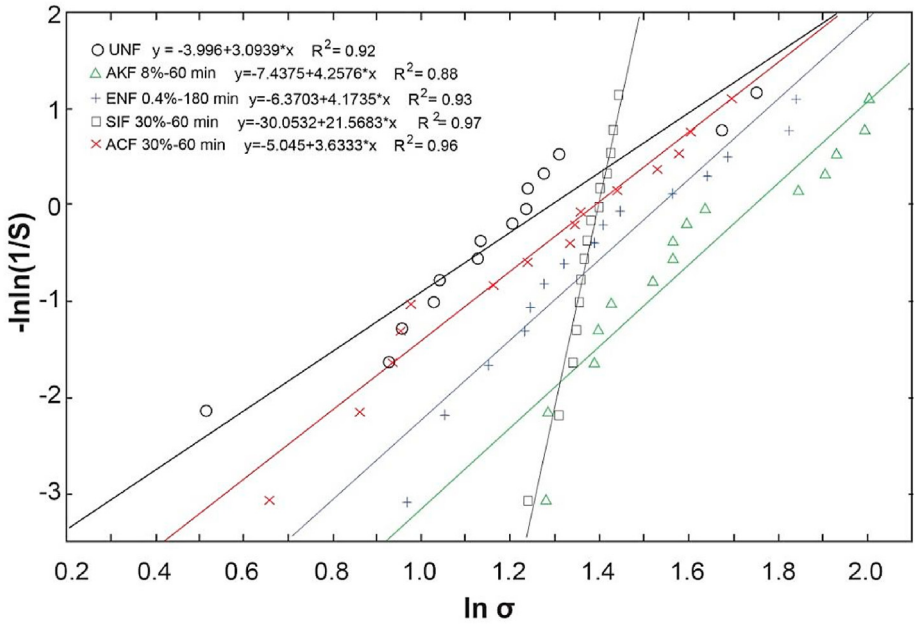


Fig. 11. Pull-out test results in Weibull coordinates for all treated samples in comparison with untreated ATF.

environment.

Authors credit statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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