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Fabrication of agave tequilana bagasse/PLA composite and preliminary mechanical properties assessment

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ABSTRACT

Bagasse agave tequilana fibres (ATF), an abundant by-product of Mexican tequila production, were characterised, treated and investigated as a reinforcement and filler material for polylactic acid (PLA) green composites.

Two fibre pre-treatments were investigated: alkali (8% NaOH solution) and enzymatic (0.4% Pectate lyase solution). Composites pellets of 20, 40 and 60 % (w/v) of ATF in PLA were manufactured using extrusion moulding. Press moulding was used to fabricate samples composite plates. Tensile, flexural, impact and water absorption properties were investigated on machined samples.

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Assessment of the mechanical properties showed tensile strength of up to 57.1 MPa for 20 % (w/v) of ATF untreated samples. Flexural strength up to 98.8 MPa and impact strength of 6.8 kJ/m^2 for 40% (w/v) of ATF alkali treated samples. These values compare well with those of other new bio-composites. The values of the Young's and flexural moduli are in proximity, if not superior, to those of widely used polymers PLA and GPPS.

The optimal ATF loading was found to be ~ 40 %. Alkali treatment of fibres provided marginally improved mechanical properties; while significantly increasing the samples' water absorption. Microscopy observations confirmed the two pre-treatments enhanced the fibre/matrix adhesion when compared with untreated fibres.

Keywords

Natural fibres-based composites, agave bagasse, PLA, compression moulding.

1 Introduction

Polymers derived from natural sources such as polylactic acid (PLA) exhibit potential as biodegradable replacements for their hydrocarbon counterparts (Stevens, 2002). PLA is also widely acknowledged as one of the most successful degradable polymers to be commercialized on a large-scale. The main advantages of PLA includes similar manufacturing processes to those of thermoplastics as well as the use of renewable fermented agricultural feedstocks, such as maize or sugar beets (Hartmann, 1998; Sawyer, 2003). High strength and low elongation to fracture has also been reported (Garlotta, 2001). Properties of relatively poor impact resistance, slow crystallization rate and low heat deflection temperatures have prevented the adoption of PLA in more demanding applications (e.g. high load bearing components); however, it has been widely used in packaging as well as other commodities (Nagarajan et *al.*, 2015). Moves to adopt more "green" constituents in composite materials have benefitted from the use of PLA.

Environmental concerns as well as reduction of raw material costs have resulted in a considerable growth in the use of natural fibres in composites. These include both purposely grown and harvested fibres, as well as those recovered from agricultural waste. Figure 1 summarises some of the most widely known natural fibres recently investigated (Faruk et *al.*, 2014; Mohanty et *al.*, 2002; Saheb and Jog, 1999).

Natural fibres are often pre-treated before composite fabrication to overcome aggregate forming and moisture resistance, amongst other issues (Bledzki et *al.*, 1996). Their pre-treatment has been shown to enhance natural fibre composites' properties (e.g. tensile strength and biodegradability) (George et *al.*, 2001; John and Anandjiwala, 2008; Li et *al.*, 2007).

PLA green composite fabrication has been demonstrated using a variety of natural fibres such as kenaf (Serizawa et *al.*, 2006), jute (Plackett et *al.*, 2003), hemp (Song et *al.*, 2013) and flax (Shanks et *al.*, 2006). Previous studies have also shown that compounding of natural fibre-based composites can be successfully achieved in a single step using twin-screw extrusion (Gamon et *al.*, 2013; Hietala et *al.*, 2014; Teixeira et *al.*, 2011).

Figure 1

The fibres investigated are a waste by-product of Mexican tequila production, originating from the Agave tequilana plant, see Figure 2 . Agave tequilana is a succulent plant belonging to the Agavaceae family. This plants typically range from 1.2 to 1.8 m in height (Cruz and Alvarez-Jacobs, 1999). Agave tequilana thrives in semi-arid land, requiring low field labour, little watering and little (or no) agrochemicals.

Figure 2

The heart of the Agave plant is harvested for tequila production. Following the removal of leaves, the heart is steam cooked and then milled to extract its juice. From the milling

process, a fibrous by-product known as bagasse is produced. The physical characteristics of the bagasse fibres suggest they can be readily used in composites, without any additional extraction costs (Iñiguez-Covarrubias et *al.*, 2001). Further, the main constituents of ATF (cellulose, hemicellulose and lignin), are reported to be comparable to those of other high cellulose natural fibres used in composites (Iñiguez-Covarrubias et *al.*, 2001).

Annual tequila output has grown steadily despite production methods still employing traditional processes. For example, over 248 million litres of tequila were produced in 2015. As a result, annual estimates of bagasse production have regularly exceeded 300,000 t (CRT, 2016). Although the use of tequila by-products has been explored by a growing number of researchers, it is known that clandestine and landfill disposal still occurs (Crespo González et *al.*, 2013).

Utilisation of the abundant tequila by-products, bagasse and leaf fibres has been investigated. Different degrees of economic adoption and success in reducing environmental impact has also been achieved. For example, Gonzalo Idarraga et *al.*, (1999) explored paper pulping of ATF by chemical and biomechanical processes. They reported that the strength of pulps from the agave fibre was poorer than wood and other agro-based pulps. Iñiguez-Covarrubias et *al.*, (2001) used ATF to substitute corn stubble in livestock feed; whereas Crespo González et *al.*, (2013) studied composting the fibres. ATF has also been used to fabricate carrier bags and containers (Hernandez, 2017). These are reported to be made using 50 % blends of polyethylene/ATF and polypropylene/ATF, respectively. Fabrication costs, which are currently higher than those of polyethylene bags, remains one of the main challenges. Whilst the use of ATF in these products reduces use of both forms of plastic, their disposal requires further consideration (Hernandez, 2017).

Perhaps one the most successful applications of waste ATF so far reported, has been their use as biofuel in the form of pellets and briquettes (Garcia Fuentes, 2012). However, there is

95 still potential to add value to this by-product by extending the range of applications and
96 societal benefits. For example, Langhorst et *al*., (2018) recently assessed ATFs as a
97 reinforcement in polypropylene automotive composites. Other previous polymer studies
98 include that of Tronc et *al*., (2007) who studied ATF as thermoplastic composite
99 reinforcement. The properties of ATF/linear medium density polyethylene composites
100 manufactured by roto-moulding were investigated by López-Bañuelos et al., (2012). Perez-
101 Fonseca et *al*., (2014) combined agave and pine fibres to produce high density polyethylene
102 composites. Moscoso-Sánchez et *al*., (2013) studied the morphology, tensile and impact
103 properties of foamed and un-foamed polypropylene/agave composites. In addition, Cisneros-
104 López et *al*., (2017) investigated the treatment and properties of compression moulded
105 polyethylene/agave composites.

106 Evaluation of PLA/agave composites has been explored by only a limited number of
107 contemporary authors; with composites fabricated using rotational moulding (Cisneros-
108 López et *al*., 2017b) and twin extrusion/injection moulding (Pérez-Fonseca et *al*., 2016).

109 The motivation for the work reported in this paper was to explore further alternatives to oil-
110 based polymers, while adding value to an abundant by-product of Mexico's tequila industry
111 and thus reduce the need for landfill disposal. This paper reports the use of agave bagasse
112 fibres, as reinforcement/filler material in a PLA matrix-based composite. ATF were treated
113 and test samples were manufactured using both twin extrusion and compression moulding at
114 three fibre loading contents (20, 40 and 60%). The effect of surface treatment and fibre
115 loading content on composites manufactured has been studied. An assessment of their
116 tensile, impact, flexural and water absorption properties is presented. Morphology and
117 fractography studies were carried out using optical and environmental scanning electron
118 microscopy (ESEM).

2 Materials and methods

2.1 Raw materials

Raw, unprocessed tequila bagasse (100 % agave) ATF were supplied by the distillery “La Fortaleza” located in Jalisco, Mexico. Additional materials used for preparation of the ATF/PLA composites included: analytical grade NaOH from Acros Organics; Pectate lyase enzyme (Scourzyme®) was provided by Novozymes; and, PLA extrusion grade (Ingeo Biopolymer 2003D) in pellet form was purchased from Natureworks™.

2.2 Characterisation of raw ATF

Raw bagasse fibres were received directly from the distillery. Prior characterisation, fibres were thoroughly pre-washed and oven-dried at 60° C for 24 hrs. to ensure the removal of residual matter present from the tequila production process. Their morphology was investigated using optical and environmental scanning electron microscopy (ESEM). Energy dispersive X-ray (EDX) was used to investigate the constituents of the fibres and their impurities.

The cross-sectional area (CSA) of fibres was measured on vertically epoxy-potted fibre samples (Thomason and Carruthers, 2012). After polishing, the samples were photographed at 50X magnification and then analysed using the open source *ImageJ* software. A total of 350 measurements were taken. Fibre length was measured using the same image processing software on 200 randomly selected fibres. The density of ATF was calculated by measuring the mass of dried specimens and then dividing by their volume. An analytical balance with a 0.00001 g resolution was used with twenty measurements taken. Full details of the fibres characterisation conducted can be found in a separate publication (Huerta-Cardoso, 2018). Table1 shows a summary of the measured properties of ATF prior to treatment.

2.3 Fibre treatments

Following an in-depth review and assessment of treatments (Huerta-Cardoso, 2018), ATF with an average length of 10 mm were pre-washed and then oven-dried at 60° C for 24 h. The dried fibres were immersed in two different treatment solutions: NaOH and the enzyme solution. The alkali-treated fibres were produced by immersing them in 8 % weight per volume (w/v) NaOH solution at 21° C, while the enzyme treated samples were produced by immersion, then in 0.4 % (w/v) of pectate lyase at 55° C. In both cases the exposure time was 180 minutes. The procedure for both treatments ensured the pre-dried fibres were first immersed in the correspondent aqueous solution and continuously stirred. Treated fibres were then drained, and subsequently rinsed with distilled water until acid-free, and to allow the removal of loosely bonded physisorbed compounds joined to the fibre surface. Untreated ATF samples were kept as control. The fibres were oven-dried after treatment (at 60° C for 24 h) and then kept in desiccators to control relative humidity (RH) due to the hygroscopic nature of the fibres.

2.4 Composite processing: extrusion and press moulding

A two-step process was used to manufacture the composite samples (i.e. extrusion and press moulding). Extrusion was carried out using a 21 mm LAB Rondol twin-screw extruder with a 2 mm hole diameter die. In a first step, ATF with a mean length of 10 mm and PLA pellets were separately premixed by continuous shaking before extrusion to assure the uniformity during the material feeding. Residence time was estimated at 3 min for all runs. This kept the melt flowing and prevented degradation. In between processing of the different grades, a purge flow was used to clear residual materials. The screw speed was fixed at 50 rpm for the melt mixing and drive torque at 60 %. The extrusion temperature profile is shown in Figure 3 .

The extrudates were prepared and pelletized by adding the premixed fibres and PLA directly into the hopper. Pristine PLA was also pelletized and press moulded as a control. Pellets were oven-dried at 60° C for 24 h and kept in desiccators at 47 ± 3 % RH before press moulding.

Figure 3

In a second step ATF/PLA composite plates were prepared using preferred ATF combinations of 20, 40 and 60 % (w/v); using alkali (AKF), enzyme (ENF) and untreated (UNF) fibre treatments. Composite plates were prepared in a 40 T hot press by press moulding the pellets in a steel frame, of 298 x 298 mm, at 160° C and 55 MPa for 8 minutes. After pressing, the frame was allowed to cool at ambient temperature and the composite plates demoulded.

2.5 Sample preparation

Samples for tensile, flexural, impact and water uptake tests were cut out from fabricated composite plates to standard dimensions by dynamic water jet cutting (Figure 4). All samples had a nominal thickness of 3.8 mm. Before testing, samples were oven-dried at 50° C for 24 h and kept in desiccators.

Figure 4

2.6 Testing and microscopy procedures

Tensile testing was performed using flat “dog bone” samples in accordance to ASTM D638-10 using a calibrated Instron 5500R EM fitted with a 100 kN load cell. The samples had a gage length of 57 mm and an overall length of 165 mm. The tests were conducted at room temperature, fluctuating between 22 to 23° C. The crosshead displacement was set to 2 mm/min in line to followed standard procedure.

190 The three-point bend flexural test was conducted in accordance to ASTM D790 using a
191 calibrated Instron 5500R EM fitted with a 5 kN load cell. Rectangular samples of 100 x 12.7
192 mm were used. The crosshead displacement speed was of 1 mm/min and the support spam
193 was 63.7 mm wide.

194 Impact testing was carried out in accordance to ASTM D4812. It used un-notched Charpy
195 samples of 64 mm in length and width of 10 mm. A Zwick pendulum impact test machine
196 fitted with a 1 J pendulum was used.

197 The morphology and failure mode of composites were analysed by optical microscopy using
198 a Nikon Eclipse E600 at 5x and 10x magnification. The fractured specimens were observed
199 using a FEI XL30 environmental scanning electron microscope (ESEM).

200 Water uptake properties were measured following ASTM D570. The dimensions of the
201 samples used were 57 x 7.2 mm. The percentage increase was calculated using:

202 $$\boldsymbol{MC = \frac{w_1 - w_0}{w_0} \times 100}$$

203 w_0 is the mass of dry sample and w_1 is the mass after exposure.

204 Fifteen samples for each mechanical test type, combination of ATF content and treatment
205 were used; for a total of 135 test results. Similarly, three samples were used for water uptake
206 tests for each ATF content % and treatment combination; for a total of 27 test results.

207 **2.7 Materials test analysis**

208 The mechanical properties of natural fibre composites are known to be influenced by a
209 number of factors including fibre length, the volume fraction of fibres, fibre aspect ratio, their
210 orientation and interfacial adhesion between the fibre-matrix (Saba et *al.*, 2015). Tensile
211 properties are amongst the most widely reported properties of natural fibre reinforced
212 composites and are a crucial factor for the selection of materials.

Tensile tests reflect the average property through the thickness of the sample (Faruk et *al.*, 2012). The Ultimate Tensile Strength (UTS) measures a material's tensile strength at its breaking point (Guidelines, 2010). UTS can be calculated using:

$$\boldsymbol{UTS = \frac{P_{max}}{A}}$$

P is the maximum load recorded during the test, and A is the specimen cross-sectional area. The specimens' elongation during the tensile test can be calculated using:

$$\boldsymbol{\delta = \frac{L - L_0}{L_0}}$$

δ is the change in gauge length, $\boldsymbol{L_0}$ is the initial gauge length, and $\boldsymbol{L}$ is the final length.

Young's modulus (*E*) is defined as the ratio of stress (σ) to strain (ε) at any point along the elastic portion of a stress/strain (load/deformation) curve.

The flexural properties of fibre reinforced composites are influenced by the surface characteristics of the specimens (Faruk et *al.*, 2012). Calculation of flexural stress at the outer surface in three-point bending can be calculated using (Hodgkinson, 2000):

$$\boldsymbol{\sigma = \frac{3PS}{2bd^2}}$$

P is the applied load, *S* is the support span, with *b* and *d* corresponding to the width and thickness of the tested sample, respectively. The Flexural modulus was calculated using (Hodgkinson, 2000):

$$\boldsymbol{E_f = \frac{S^3 m}{4bd^3}}$$

E_f is the flexural modulus, *S* is the support span length, *m* is the slope of the load/deflection curve, with *b* and d being the width and thickness of the sample, respectively.

The impact strength (*I*) is the maximum force necessary to rupture a composite sample caused by impact (Hodgkinson, 2000). For a rectangular un-notched sample, this can be calculated as follows:

$$I = \frac{E_c}{hb} \times 10^3$$

where E_c is the corrected energy absorbed by the specimen; h and b are the thickness and width of the specimen respectively.

3 Results

Over 20 ATF/PLA composites plates were successfully produced, using the target ATF content combinations of 20, 40 and 60 % w/v; and using either fibre enzymatic or NaOH pre-treatments. Additionally, composites with untreated ATF as well as pristine PLA plates were also manufactured. Over 180 test specimens were machined and prepared from the ATF/PLA plates. Density of composite plates used to manufacture test specimens is given in Figure 5.

Figure 5

Analysis of micrographs taken showed large-scale variations in the fibres' aspect ratios and morphologies in all of the pre-treatment variants and also the fibre loading combinations. An example of this is shown in Figure 6 for AKF treated ATF with 40 % w/v. The composite surface has a rough aspect. Some breakage due to kneading during the extrusion process can be observed. There was no clear evidence of columnar crystalline layer formations at the interface (i.e. transcrystallinity),(Quan et *al.*, 2005) on the examined samples.

Figure 6

3.1 Mechanical properties assessment

Tensile, flexural and impact tests results are summarised in Table 2. The UTS of pre-treated ATF/PLA composites ranged from 46 to 57 MPa, see Figure 7. The test results also showed that the UTS of pre-treated ATF samples increased with an increase of fibre content from 20 to 40 % w/v, for both the AKF and the ENF treatments. The improvement was of approximately 6 and 9 %, respectively. The higher UTS was attributed to effective fibre

reinforcement. However, a further increase in the ATF content, up to 60 % resulted in a pronounced drop in the UTS, independently of the fibre treatment.

At the lower fibre 20 % content, the UTS of untreated ATF/PLA composite samples was 57 MPa. This was higher than that of the pre-treated samples at the same fibre loading. However, similar UTS values for both ENF and AKF pre-treatment samples were observed (57 and 54 MPa) when the fibre content was of 40 %. The ENF samples had an even higher UTS than that of UNF samples at 20 % ATF. Otherwise, the UTS of untreated ATF/PLA composite samples dropped linearly with an increasing ATF loading.

The mean value of the Young's modulus of all samples is shown in Figure 8, together with the samples measured elongation. The nominal values of PLA and GPPS (General Purpose Polystyrene) are also plotted for comparison.

Figure 7

The Young's modulus values of the ATF/PLA composite samples ranged from 2.70 to 3.03 GPa. Increasing the ATF loading resulted in an increase in the Young's modulus measured. In contrast, elongation generally decreased with increased ATF content, ranging from 2.68 to 1.87, reflecting an increase in brittleness.

When compared to nominal GPPS, the Young's modulus of the ATF/PLA composite was always lower by approximately 2.25-12.90 %. However when compared with pure PLA samples (2.79 GPa), all of the ATF/PLA composites, regardless of fibre loading and pre-treatment, produced comparable if not superior Young's modulus values.

Figure 8

Figure 9

283 Regardless of the ATF content and treatment, all tensile samples fractured after yielding
284 without necking, in characteristic brittle failure mode. This behaviour is illustrated in Figure 9
285 for the 40% ATF loading, for all treatments.

286 **Figure 10**

287 Flexural strength ranged from 86 to 99 MPa, while the flexural modulus ranged from 3.51 to
288 3.81GPa, (Figure 10). Generally, the flexural strength of pre-treated ATF samples increased
289 with an increase of fibre content from 20 to 40 % w/v, for the two pre-treatments: AKF and
290 ENF. This improvement was of approximately 12 % for both pre-treatment samples. Again,
291 further increasing of the ATF loading, up to 60 %, resulted in a drop in the flexural properties,
292 independently of the fibre treatment used. The flexural strength of pristine PLA samples was
293 111 MPa and was approximately 11 % higher than that of ATF/PLA. However, ATF/PLA
294 samples observed flexural strength values above 50 % greater, when compared to the
295 nominal flexural strength of GPPS.

296 The flexural modulus increased when the ATF loading increased from 20 to 40 % w/v and
297 appears to not be significantly affected when the solids loading reached 60 % for all
298 treatment conditions.

299 The impact strength of the ATF/PLA composites ranged from 5.51 to 8.40, (Figure 11).
300 Measured and nominal reference values have been added for PLA and GPPS, respectively.

301 **Figure 11**

302 As with the UTS and flexural strength results, the impact strength of the ATF/PLA
303 composites improves when the fibre loading is increased from 20 to 40 %, regardless of the
304 pre-treatment used. On the other hand, the lowest impact strength was observed when the
305 fibre loading increased from 40 to 60 %. Irrespective of the ATF/PLA composites loading and
306 pre-treatment, the impact strength of PLA and GPPS exceeded that of ATF/PLA samples.

3.2 Microscopy analysis

Optical and scanning electron microscopy was used to examine both the composites typical fibre/PLA adherence and the fracture surfaces of the tensile, flexural and impact test specimens. Generally, it was observed that there was a good distribution of fibres within the PLA matrix.

Detailed examinations of cross-section untreated fibres samples showed fewer traces of the PLA matrix, which indicated low fibre wetting and more extensive breakdown. Figure 12 presents typical fracture surfaces of tensile test samples with ATF loading of 40% w/v. Weak adhesion in the UNF/PLA sample was confirmed by the presence of voids left by ATF in the matrix, as opposed to cracks extending into it. Conversely, the analysis of AKF/PLA composites presented fewer or no matrix voids, suggesting stronger ATF/PLA adhesion and fibre wetting. AKF treated fibres appear more robustly adhered to the matrix. Propagating cracks were observed as opposed to ATF pulling. The ENF/PLA samples showed moderated adhesion with residual PLA on the fibres surface (Figure 13). Some fibre pull-out was observed, suggesting slightly less adhesion than that observed in the AKF treated specimens.

Figure 12

Figure 13

3.3 Water absorption properties

All samples from all ATF loading and treatment combinations registered an initial rapid increase in weigth after exposure. Figure 14 shows the results of the 40% w/v loading samples.

Figure 14

330 The highest absortion was registered by composites with AKF treated fibres. These samples
331 experienced an initial rapid absorption rate for the first 12 days. After this, the samples
332 absorption continued to grow at a very slow rate. The samples stabilised around a saturation
333 of 3.47 % after 40 days of exposure until the end of the experiment. Other ATF/PLA samples
334 with different fibre treatment did not exceed 2 % saturation. The lowest absorption was by
335 the PLA control samples at 0.75 %.

336 # 4 Discussion

337 ## 4.1 Effect of fibre loading

338 The effect of ATF volume was investigated using 3 different target loading settings; 20, 40
339 and 60 w/v %. Both, the tensile and flexural strength were observed to increase when the
340 ATF loading increased from 20 to 40 % by at least 6 and 12 % respectively as shown in
341 Figure 7. Further loading, up to 60 % resulted in considerably poorer properties. The results
342 observed are comparable with other PLA-based composite materials using harvested fibres
343 from kenaf (Ibrahim et *al.*, 2010), hemp (Masirek et *al.*, 2007) and flax (Oksman et *al.*, 2003).
344 Impact strength was also observed to exhibit a similar trend; with 40% w/v loading showing
345 enhanced strength values, then dropping at 60% ATF w/v. Other authors have observed
346 similar results (Langhorst et *al.*, 2018). This effect has been attributed to the stiffening of
347 polymer chains due to bonding between the fibres and the matrix (Karmarkar et *al.*, 2007).
348 Suggestion for enhancement of impact strength include surface functionalization of agave
349 fibres, the use of coupling agents and the addition of impact modifiers (Langhorst et *al.*,
350 2018).

351 When directly compared to pristine PLA, the addition of agave fibres to PLA matrices did not
352 result in enhanced mechanical properties. The decline of the mechanical properties
353 observed with fibre additions can be attributed to the brittle nature of the added agave fibres.
354 Other strongly influencing factors include damage or shearing of the fibres, caused by the

manufacturing process during the kneading phase of the extrusion moulding (evidence of this is shown in Figure 6). Detailed studies confirming this phenomenon have been conducted before (Beaugrand and Berzin, 2013). Other authors have found that higher fibre aspect ratios or larger fibre surface availability correlate well with improved mechanical properties of flax/polypropylene composites (El-Sabbagh et *al.*, 2014). Analysis of fracture micrographs indicated debonding mechanisms were present (Mehan and Schadler, 2000); which suggests stronger ATF/PLA bonding would further enhance the composites mechanical properties.

The Young's and flexural moduli of the ATF/PLA composites were less affected by increasing the ATF loading from 40 to 60 % w/v, regardless of the treatment (see Figure 8 and Figure 10). The results of these moduli were comparable, if not superior, with those of the PLA control samples. The addition of ATF led to greater stiffness of the matrix. Improved Young's modulus with the addition of natural fibres has been observed by other researchers too (Graupner et al., 2009).

Water absorption increased with the fibre loading. This was in agreement with other fibre publications (Karaduman and Onal, 2011). Increased voids and cellulose content have been reported as the main causes of this behaviour (Dhakal et al., 2007). Our results are also in agreement with a recent study reporting that water absorption in agave composites is a function of the solids loading (Langhorst et al., 2018).

4.2 Effect of fibre treatment

In general, the differences in the measured mechanical properties of the two treatments studied were only marginal; with the AKF samples typically observing marginal superior results over the ENF samples. This was attributed to improved fibre wetting behaviour inferred from microscopy observations such as those shown in Figure 12 and Figure 13, which show stress transfer cracking.

Untreated UNF fibres at the lowest loading of 20 % w/v yielded improved mechanical properties compared to those of the treated ATF samples at the same loading. It appears that at this lower fibre loading level and absence of treatment, the PLA matrix dominates the overall tensile, flexural and impact behaviour of the composite, with the ATF additions having a much smaller effect.

The water absorption results showed the most obvious effect of the fibre pre-treatments. For example, at 40 % w/v fibre loading, AKF composites registered the highest water uptake at 3.47 %, followed by the ENF at 1.95 % and UNF treated samples at 1.5 %. The difference in water uptake between samples is attributed to fibre delignification after treatment, which made the fibres more hydrophilic. Greater water/moisture absorption in alkali-treated natural fibres has been observed when compared to other treatments. This includes areca fibres using alkali and acetylation treatments (Sampathkumar et *al.*, 2012); and banana fibres using acrylic acid and alkali treatments (Jannah et *al.*, 2009).

4.3 Comparison with contemporary ATF composites research

Due to environmental concerns, the use of waste agave fibres as composites filler and reinforcement material has become more relevant. A number of contemporary attempts have been reported. Table 3 summarises the most recent results by other authors in terms of tensile and flexural properties; and compares them to the present research. PLA properties are presented as baseline for comparison.

As seen in Table 3, the results from recent research that used both a PLA matrix and agave fibres composites (Cisneros-López et *al.*, 2017b; Pérez-Fonseca et *al.*, 2016) are in proximity to the results presented here. When comparing with both rotational and compression processes (Cisneros-López et *al.*, 2017b), our results generally show better performances in terms of the tensile strength, Young's modulus, and flexural strength. There might be a number of factors that account for these differences. For example, although the

biopolymers used have very similar properties and come from the same supplier, Ingeo™ Biopolymer 3251D and Biopolymer 2003D (current work), there are slight variation in their properties. There were also differences in the preparation of the agave fibres too. Cisneros-López et *al.*, 2017b ground and sieved the fibres; while they also pulverised the PLA pellets. The sieving controlled the distribution size of the agave fibres and possibly accounted for the differences in the effect of fibre loading too, when compared with our work. Their optimal fibre loading was found at 10 % w/v as opposed to 40 % of the present work. A similar work (Pérez-Fonseca et *al.*, 2016) appears to confirm this; as they did not sieve the fibres and pulverise the PLA either. However, they found an optimal fibre loading of between 20 and 30 %. They used and annealing treatment to enhance the impact and flexural properties.

Langhorst et *al.*, 2018 used a polypropylene (PP) matrix with varying amounts of agave fibres. Generally, their tensile and flexural results were inferior compared with those of the PLA matrix composites. However, the agave fibre/PP composites yielded up to 300 % higher elongation. These results can be attributed to the polymer matrix used. The comparison is consistent with the observed results of Graupner and Müssig (2017). They reported significantly higher tensile strength, Young's modulus and hardness of PLA and PP composites reinforced with lyocell fibres. The superior performance is attributed to enhanced adhesion of the lyocell fibres onto the PLA. Lyocell/PP showed a more ductile behaviour, which is consistent with the higher elongation of the ATF/PP composites (Langhorst et *al.*, 2018).

Our research demonstrates how an abundant by-product can be included in composites to form usable materials. Potential applications for these materials include non-structural components, such as internal panels used in the automotive industry (Ahmad et *al.*, 2015), (Lee and Flanigan, 2002), and (Koronis et *at.*, 2013), due to the reduced mass resulting from these low-cost fibre additions. Consequently, increased energy efficiency and reduced fuel

consumption would be expected. Such new composite materials may offer more sustainable alternatives to conventional oil-based thermoplastic materials as it has been .already pointed out that the use of natural fibres allows obtaining several environmental advantages in comparison to mineral-inorganic counterparts (Joshi et al., 2004) and (Netravali and Chabba 2003). Furthermore, if polymers coming from renewable resources such as PLA are used, problems related to the everyday production of solid, plastic-derived waste may be reduced (Nampoothiri 2010). However, these aspects have to be investigated through a complete life cycle assessment (LCA) to provide enough evidence (Joshi et al., 2004).

The test results and microscopy analysis suggest an optimal ATF loading can be found around 40 %; although no further refinement was pursued. Both fibre pre-treatments were effective. Microscopy analyses also show that it is likely that the AKF fibre treatment leads to enhanced fibre adhesion, although this treatment resulted in higher water absorption. Further investigations are required to understand its effect on the composites properties. In contrast, enzymatic pre-treatment is considered more energy efficient, less chemical intensive with more environmental friendly effluents (Sharma et *al.*, 1999).

5 Conclusions

Tequila distillery bagasse waste has been successfully used to produce usable agave fibre/PLA composites by extrusion/press moulding. The agave fibre/PLA composites produce consistent and repeatable tensile, flexural, impact and water absorption properties. These lightweight, low-cost composites might find applications such as non-structural automotive components and consumer goods; leading to energy efficient transportation and reduced fuel consumption. However, a complete life cycle assessment (LCA) is required to fully assess the sustainability of the agave fibre/PLA composite.

The additions of agave fibres did not directly enhance the mechanical properties of the PLA matrix. We attribute this to the final aspect ratio of the fibres after compounding and the inherent flaws of the bagasse fibres. The observed tensile, flexural and impact strength of the ATF/PLA composites were up to 57.1 MPa, 98.8 MPa and 6.8 kJ/m^2, respectively. These are comparable, if not superior to those reported in the literature for new composites produced using similar materials and conditions and, in some instances superior to GPPS. The brittle nature of the agave fibres contributed to a higher Young's and flexural moduli, which were observed to be slightly superior to those of PLA.

For tensile, flexural and impact properties the optimal fibre loading was of 40 % w/v. At 60 % the mechanical properties of the composites degraded significantly. It was also observed that increasing the fibre content, resulted in higher water absorption.

Both alkali and enzymatic fibre pre-treatments have shown to substantially enhance the fibres/matrix adhesion when compared with untreated fibre composites; resulting from improved fibre wettability and surface morphology. The observed higher water absorption of the alkali treated fibre samples, up to 170 % higher, was related to fibre delignification making the fibres more hydrophilic. Microscopy showed improved fibre/matrix interfaces when using the alkali treatment.

The use of high aspect ratio fibres and prevention of fibre shearing processes could enable ATF/PLA composites to be produced with enhanced mechanical properties, suitable for more demanding applications.

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709 Figure 1 Natural fibres classification summary. Adapted from (Faruk et al., 2014; Mohanty et
710 al., 2002; Saheb and Jog, 1999).

711 Figure 2 Left: Agave plant. Right: Dry bagasse from tequila production showing natural fibres

712 Figure 3 Extrusion profile temperature settings.

713 Figure 4 Left: ATF/PLA composite plate. Right: ATF/PLA machined composite tests
714 specimens. From top to bottom: tensile, flexural and impact.

715 Figure 5 Density of composite boards according to fibre loading and treatment.

716 Figure 6 Micrographs of 40 % w/v ATF/PLA based composites after press moulding. The ATF
717 pre-treatment used was AKF, showing rough aspect (left) and some breakage (right).

718 Figure 7 Ultimate tensile strength of ATF/PLA composites Vs fibre content.

719 Figure 8 Young modulus and elongation of ATF/PLA composites with varying fibre loading
720 and pre-treatment.

721 Figure 9 Mean stress-strain curve for 40 % w/v ATF with different fibre pre-treatments.
722 Standard error <0.65 for all cases.

723 Figure 10 Flexural strength and modulus of ATF/PLA composites. Error bars represent
724 standard error.

725 Figure 11 Impact strength of ATF/PLA composites, PLA and GPPS

726 Figure 12 Optical micrographs of cross sections of ATF/PLA samples with 40 % (w/v) loading
727 after tensile failure. Left: Untreated ATF. Right AKF Treated ATF.

728 Figure 13 ESEM micrograph of ENF treated composite. ATF loading 40% w/v.

729 Figure 14 Water absorption at saturation for 40% (w/v) ATF/PLA composites. In all cases, the

730 standard error <0.13.

Parameter	No. of measurements	Mean	Median	Mode	Standard deviation	Skewness	Max	Min
CSA (μm^2)	350	75	66.54	30.38	41.41	0.89	214.60	16.62
Length (mm)	200	77	77.41	66.65	18.27	-0.13	125.94	27.02
Density (g/cm^3)	20	1.2	1.15	-	0.11	0.70	1.47	1.01
Aspect ratio* (mm)	100	249.19	-	-	15.25			

731 *Cylindrical approximation

732 Table 1 Summary of the measured properties of ATF

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Treatment	ATF loading (%)	UTS Mean (MPa)	SD	Flexural Modulus Mean (GPa)	SD	Impact strength Mean (kJ/m^2)	SD
Neat PLA	0	62.55	2.92	3.78	0.13	10.11	1.12
UNF	**20**	57.16	0.54	3.70	0.02	8.40	0.20
	40	49.67	2.46	3.77	0.01	7.88	0.29
	60	47.25	1.15	3.78	0.01	7.46	0.53
AKF	**20**	50.79	2.33	3.60	0.01	6.38	0.25
	40	53.97	1.07	3.81	0.02	6.76	0.32
	60	46.68	2.46	3.73	0.01	6.0	0.51
ENF	**20**	52.32	1.04	3.51	0.01	5.95	0.19
	40	57.19	0.90	3.82	0.01	6.55	0.27
	60	46.91	1.07	3.80	0.02	5.50	0.06

745 UTS = Ultimate tensile strength

746 Table 2 Overview of ATF/PLA composites: tensile, flexural and impact test results

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Composite preparation				Tensile properties			Flexural properties		Source
				ASTM D638-10			ASTM D790		
Polymer matrix	**Treatment**	**Fabrication**	**Fibre loading % (w/v)**	**UTS (MPa)**	**E (GPa)**	**Elongation (%)**	**Flexural Strength (MPa)**	**Flexural Modulus(GPa)**	
Pristine PLA	N/A	Extrusion-compression	0	62.6	2.8	2.9	110.9	3.8	Present work
PLA	NaOH	Extrusion-compression	20	50.8	2.9	2.3	88.4	3.6	Present work
			40	54.0	2.8	2.5	98.8	3.81	
			60	46.7	3.0	1.9	86.3	3.7	
PLA	pectate lyase	Extrusion-compression	20	52.3	2.7	2.4	87.7	3.5	Present work
			40	57.2	2.9	2.1	98.5	3.8	
			60	46.9	2.9	2.0	85.8	3.8	
PP	compatibilizer (PPgMA)	Injection moulding	0	29.	1.6	11.1	35.2	1.2.	(Langhorst et *al.*, 2018)
			10		1.8	8.3	37.2	1.3	
				27.1					
			20	25.1	2.0	5.6	39.7	1.6	
			30	23.2	2.2	4.2	39.2	1.8	
PLA	N/A	Rotational moulding	0	59	1.9	3.5	93	3.5	(Cisneros-López et *al.*, 2017b)
			10	45.0	2.0	2.9	67	3.6	
			20	26.0	1.4	2.7	36	2.5	
			40	6.0	0.4	2.6	5	0.4	
PLA	N/A	Compression moulding	0	60	2.0	3.5	92	3.7	(Cisneros-López et *al.*, 2017b)
			10	47.0	2.2	2.3	72	3.8	
			20	42.0	2.3	2.3	56	3.7	
			40	29.0	1.8	1.8	35	3.0	
PLA	N/A	Injection moulding + annealing	0	60	1.2	-	95	2.3	(Pérez-Fonseca et *al.*, 2016)
			10	53	1.5	2.3	71.5	2.9	
			20	55	1.5	2.3	70.2	3.1	
			30	47	1.6	1.8	70.2	3.2	

752 Table 3 summary of recent composites research using agave fibre. Abbreviations: polylactic

753 acid (PLA), polypropylene (PP), ultimate tensile strength (UTS) and Young's modulus (E).



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Fabrication of agave tequilana bagasse/PLA composite and preliminary mechanical properties assessment

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