

# **Mechanical properties of leaf sheath date palm fibre biomass waste reinforced polycaprolactone (PCL) biocomposites**

Hom Dhakal<sup>a,\*</sup>, Alain Bourmaud<sup>b</sup>, Francoise Berzin<sup>d</sup>, Fahad Almansour<sup>a</sup>, Zhongyi Zhang<sup>a</sup>, Darshil U. Shah<sup>c</sup>, Johnny Beaugrand<sup>d, e</sup>

<sup>a</sup> Advanced Materials and Manufacturing (AMM) Research Group, School of Engineering, University of Portsmouth, Portsmouth, Hampshire PO1 3DJ, United Kingdom

<sup>b</sup> Université de Bretagne Sud, IRDL, UMR CNRS 6027, Lorient, France

<sup>c</sup> Centre for Natural Material Innovation, Dept. of Architecture, University of Cambridge, Cambridge CB2 1PX, United Kingdom

<sup>d</sup> Fractionnement des Agro Ressources et Environnement (FARE), URCA INRA, 2 esplanade Roland Garros, F-51100 Reims, France

<sup>e</sup> Biopolymères Interactions Assemblages (BIA), INRA, rue de la Géraudière, F-44316 Nantes, France

## **ABSTRACT**

*Date palm fibres are one of the most available natural fibres in North Africa and the Middle East. A significant amount of date palm fibres biomass is wasted annually and only limited amounts are used in low value products. In this study, tensile and low-velocity impact response of biodegradable lignocellulosic biomass reinforced polycaprolactone (PCL) biocomposites are reported. Two different types of laminates reinforced with date palm fibre obtained from agriculture waste were manufactured by an extrusion process. The influence of processing parameters, such as screw rotation speed on the tensile and low velocity impact damage characteristics have been investigated. The tensile strength increased for neat PCL from 19 MPa to 25 MPa with 28 wt.% reinforcement of date palm fibres. Similarly, the tensile modulus for neat PCL was increased from 140 MPa to 282 MPa upon reinforcement. The screw rotation speed showed a moderate effect on palm fibre morphologies, and slight effect on tensile properties of the biocomposites. Specimens with lower incident energy of 25 J achieved better impact resistance compared to that of 50 J. The impact damage of biocomposites analysed through electron microscopy on the fractured surface showed various modes of damage. The biocomposites developed in this work can be used as an economically and environmentally attractive alternative materials for lightweight applications in automotive and marine sectors.*

**Keywords:** *Date Palm Fibres; Low-velocity impact, Biocomposites; Delamination.*

---

\* Corresponding author. Tel: + 44 (0) 23 9284 2582; fax: + 44 (0) 23 9284 2351.

E-mail: [hom.dhakal@port.ac.uk](mailto:hom.dhakal@port.ac.uk)

## 1. Introduction

The total costs of production (energy/power) and maintenance of conventional (synthetic) fiber-reinforced polymer (FRP) composites such as glass and carbon FRP composites are relatively higher when compared with that of natural FRP composites such as date palm, hemp or flax biocomposites. In addition, the problems of unstable interfacial adhesion of the fiber and matrix often results in fiber-pull out, and matrix de-bonding as well as inter-ply delamination are more rampant with synthetic FRP composites than the natural counterparts. Importantly, date palm fiber/polycaprolactone biocomposites are completely biodegradable, renewable, sustainable and recyclable materials, unlike the synthetic FRP composites, ~~hence~~. Hence, date palm/PCL have a very low level of toxicity which makes them environmentally friendly and relatively safe for human health during processing and life cycle. Moreover, their light weight leads to attractive specific properties such as high strength-to-weight ratios in comparison to their non-renewable, high energy-intensive reinforcing synthetic counterparts such as glass and carbon fibres. These positive attributes of natural fibre reinforcements are particularly attractive to the automotive original equipment manufacturers (OEMs) as they are facing the challenge of producing light parts at low cost. In this context, biocomposites reinforced with low density fibres such as date palm and biodegradable matrix such as polycaprolactone (PCL) can offer alternative solutions to the petroleum based polymers and non-renewable synthetic reinforcements (Faruk et al., 2012; Paul et al., 2015).

There are several research works reported in the past focusing on the development and applications of natural plant fibre reinforced thermoplastic and thermoset

59 composites in automotive, marine, and construction industries (Dhakal et al., 2007;  
60 Mohanty et al., 2000). Due to their main constituents being cellulose, hemicellulose  
61 and lignin, plant fibres are also known as lignocellulosic fibres. Investigations on  
62 mechanical properties of these fibres suggest that the specific tensile stiffness<sup>2</sup> of  
63 some commonly used natural fibres such as hemp, flax and sisal are comparable to  
64 those exhibited by synthetic reinforcements (Joshi et al., 2004; Summerscales et al.,  
65 2010). However, these reported works also point out that despite their attractive  
66 mechanical and environmental attributes, these plant fibre reinforcements suffer with  
67 non-uniform properties leading to variable mechanical performances, poor fibre-  
68 matrix compatibility, and concerns relating to long-term durability (Charlet et al.,  
69 2007). Moreover, a plant fibre bundle for example consists of several cells composed  
70 of cellulose, hemicellulose and lignin. The ratio of these constituents vary from one  
71 fibre to the other. This structural complexity and dimensional inhomogeneity often  
72 contribute to varied and distinct properties of plant fibres which influence the final  
73 properties of the composites (Stamboulis et al., 2001).

74 In the last several years, significant interest has emerged in the development of  
75 composite materials from waste precursors. This new approach will not only tackle  
76 waste disposal problems, but also provide an adequate route for resource utilisation,  
77 hence supporting the sustainable development agenda by balancing social,  
78 environmental and economic considerations. The utilisation of waste date palm fibres  
79 in composite reinforcements can be a step forward in contributing resource  
80 maximisation. Currently, the bulk of date palm fibres are used in low value products.  
81 Therefore, the use of agricultural residues such as date palm fibres through sensible  
82 and innovative ways of utilisation is of potential interest to researchers and industry  
83 (Bledzki and Gassan, 1999; Nasser et al., 2016).

84 Date palm fibres are derived from the date palm tree. The date palm tree (*Phoenix*  
85 *dactylifera* L.), a member of the palm tree family (*Arecaceae*), is one of the most  
86 cultivated palms in North Africa and the Middle East, including in countries such as  
87 Tunisia, Algeria, United Arab Emirates, Saudi Arabia, Egypt, Iraq and Iran. After date  
88 fruit harvesting, date palm rachis waste is accumulated on agricultural land every year  
89 in these countries (Chao and Krueger, 2007). The major constituent of date palm fibre  
90 is cellulose (46%), hemicelluloses (18%), lignin (20%) and ash (10.54%). Date palm  
91 trees produce a large quantity of agriculture waste. For example, each tree produces  
92 20 kg of dry leaves annually. This waste is burned in the farms, causing serious  
93 environmental pollution as well as death of important soil microorganisms (Alawar et  
94 al., 2009).

95 The part of the date palm tree which is often used as fibres is the sheath. The  
96 sheath is the part of the tree which surrounds the trunk of the plant attached to its  
97 lateral edges near the top of the trunk as shown in Fig. 1. Sheath is also known under  
98 the name of leaf and is often torn lose when pruning the leaves. Seeking to use them  
99 as useful material resources instead would bring economic growth. If these  
100 agricultural residues are utilised in building materials (Alsaeed et al., 2013) or for  
101 example as composite reinforcements, a tremendous value-added by-product can be  
102 realised. The residue generated from the palm tree is currently used in a variety of  
103 applications such as particle board, low and medium density fibre board, pulp and  
104 paper. But these wastes have not been fully-utilised as an economic alternative  
105 (Abdelaziz et al., 2016; Nasser et al., 2016).

106 Despite several benefits of date palm fibres outlined, to the authors' best  
107 knowledge, there are limited reported studying biocomposites developed from waste  
108 date palm fibre residue and investigating their low velocity impact behaviour. The

109 main objective of this study is to evaluate the suitability of date palm fibre as  
110 alternative reinforcements in composite materials. The reinforcing effects of date  
111 palm fibres obtained from palm leaf sheath on the resulting extruded and injected  
112 PCL biocomposites are investigated by categorising the important parameters  
113 influencing on the low velocity impact and tensile properties. In addition, the  
114 influence of screw rotation speed on palm fibre defibrillation (Alawar et al., 2009)  
115 and flow conditions along the twin screw extruder (TSE) are analysed in order to  
116 calculate the specific mechanical energy (SME) provided to the fibres. Furthermore,  
117 the influence of extruder screw rotation speed and palm fibre reinforcements on the  
118 tensile and impact properties including load bearing capability, energy absorption,  
119 impact damage characteristics from the falling weight impact are analysed and  
120 discussed.

## 121 **2. Materials and methods**

### 122 **2.1 *Materials***

123 Date palm fibres (*Phoenix dactylifera L.*) were obtained from a farm in Al-Ahsa,  
124 located in the Eastern Province of Saudi Arabia, as decametric bundles (Fig 1, a-b).  
125 Prior to Twin Screw Extrusion (TSE), the palm bundles were chopped into a master  
126 batch with an average length of approximately 1 cm (Fig 1, c, average length  $L_w$   
127 equal to 10400  $\mu\text{m}$ ) with a plant shredder equipped with a 2 mm sieve (Retsch, Haan  
128 Germany). The chopped fibre bundles were then stored at 20°C C and 50% relative  
129 humidity in a climatic chamber prior TSE. The SEM images of date palm fibre **are**  
130 depicted in Fig. 2.

131 The box plot give the upper Quartil (Q1), the quartil 3 (Q3), the median length  
132 ( $L_{med}$ ) and the mean Length ( $L_w$ ) with values incrustated in the graphic.

133

Fig. 1.

134 The thermoplastic matrix used was polycaprolactone (PCL) of low melting  
135 temperature (60 °C). PCL (Capa© 6800) was provided by Perstorp, United Kingdom,  
136 having a molecular weight of 80,000 g/mol and a melt flow index (MFI) of 3 g/10  
137 min (160 °C, 2.16 kg).

138 The longitudinal morphology of the date palm fibre examined by SEM is shown in  
139 Fig. 2. The tensile strength and stiffness of the composites is presented in the form of  
140 an Ashby's materials selection chart in Fig. 3 (Shah, 2014), comparing the  
141 performance with other natural bast fibre composites. The comparative physical and  
142 mechanical properties of date palm fibre with other commonly used bast fibre are  
143 presented in Table 1 (Dhakal et al., 2007; Mohanty et al., 2000; Pickering et al.,  
144 2016).

145

Table 1

146

Fig. 2.

147

Fig. 3.

## 148 2.2 *Composite manufacturing*

149 The compounds were prepared using chopped palm fibre (Fig 1, c) mixed with a  
150 PCL matrix at a concentration of 20 wt.% with a laboratory-scale twin-screw co-  
151 rotating extruder TSE ZSE 27 MAXX (Leistritz, Germany). Its main characteristics  
152 are as follows: centerline distance 22.7 mm, screw diameter 28.3 mm, length/diameter  
153 ratio 36. In addition to screw conveying elements, it comprises a left-handed element  
154 in zone 3 to ensure the melting of the polymer matrix and two mixing blocks in zones  
155 6 and 8, to disperse the fibres. The first one is constituted of a block of kneading  
156 discs, staggered at 90°, and the second one of a block of kneading discs, but staggered  
157 at -60°. The matrix is introduced in zone 1, melted in the left-handed element (zone 3)  
158 and the fibres are then added in zone 4. During all experiments, the barrel temperature

159 was kept constant at 120 °C, the feed rate retained was 3.5 kg/h, and screw speed (100  
160 and 400 rpm) was varied.

161 In order to estimate values of parameters that cannot be measured experimentally  
162 during the compounding process, we have used the flow simulation software  
163 Ludovic© dedicated to twin screw extrusion (Vergnes et al., 1998). Specifically,  
164 Ludovic© was used to calculate the specific mechanical energy (SME) transmitted to  
165 the composite all along the extruder.

166 The SME is calculated from equation (1):

167 
$$EMS = \frac{1}{Q} \int_0^x \eta(x) \dot{\gamma}^2(x) V(x) dx \quad (1)$$

168 where, Q is the mass flow rate,  $\eta$  the viscosity,  $\dot{\gamma}$  the shear rate and V the design  
169 volume considered. Fig. 4 represents the change in SME along the screws for two  
170 different screw speeds.

171 Fig. 4.

172 After extrusion, the composite strands were cooled down at room temperature and  
173 the compounds were granulated (approx. 8 mm in length).

174 Standard dumbbell specimens were injected using a bench scale DSM Xplore  
175 (Geleen, The Netherlands) according to the procedure of Haag et al. (2017). Ten  
176 specimens were tested for each condition.

177 To analyse the morphometry of the compounded samples, the PCL dissolution was  
178 done as described by Beaugrand and Berzin (2013) (Fig 1, d-e). Then, the  
179 morphometric description was done according to the method of Di Giuseppe et al.  
180 (2016) using a 2D high-resolution image scanning.

181 Laminates of 200x200 mm length, 5 mm thick were manufactured by press  
182 moulding as described in Ismail et al. (2016). The press is a two columns automatic  
183 laboratory hydraulic press (Carver, Wabash, IN) equipped with heating platens.

### 184 **2.3 Tensile testing**

185 Tensile testing was carried out on a universal testing machine (Testwell, Saint  
186 Ouen, France) equipped with a mechanical extensometer. The tensile testing was  
187 carried out according to the ISO 291:2008 standard. The loading rate used was 1  
188 mm/min.

### 189 **2.4 Low velocity impact test**

190 Zwick/Roell HIT230F instrumented falling weight drop impactor was used for  
191 instrumented falling weight impact testing. The composite specimens were cut from  
192 the Palm/PCL composite laminates using a band saw to a square dimension of 60 x 60  
193 mm with 5.7 mm thickness at two energy levels: 25J (with an impact velocity 1.47  
194 m/s) and 50J (with an impact velocity 2.08 m/s) at room temperature. The diameter of  
195 the hemispherical steel tup was 19.8 mm. Impact parameters such as peak force,  
196 absorbed energy, deformation were continuously recorded for every specimen at each  
197 impact event performed using a load cell and a strain–gauge striker fitted on the  
198 system. A catcher mechanism was activated to avoid the multiple damage on the  
199 specimens. A schematic of drop weight impact mass, hemispherical impact tup and  
200 clamping systems are shown in Fig. 5. Similar impact test parameters were used to  
201 impact hemp/PCL composites in order to compare the impact damage behaviour of  
202 palm/PCL biocomposites.

203 Fig. 5.



## 204        **2.5 Fractured surface characterisation by SEM**

205        Impact failure surfaces of different biocomposites were pre-coated with a thin gold  
206        film and observed in a Phillips XL30CP scanning electron microscope (SEM) at 15  
207        kV acceleration voltages.

## 208        **3. Results and discussion**

### 209        **3.1. Tensile properties (strength and modulus)**

210        The tensile properties of different reinforcement configurations are summarised in  
211        Table 2. The tensile results show that there is a clear influence of date palm  
212        reinforcement on the tensile strength, modulus and strain at break. There was  
213        improvement in tensile strength for example from 19 MPa for neat PCL to 24 MPa for  
214        20wt.% Palm/PCL. Moreover, there is a significant improvement in tensile modulus  
215        from 140 MPa for neat PCL to 284 MPa for 20 wt.% Palm/PCL biocomposites. This  
216        increase in strength and modulus is attributed to the fibre reinforcement effects as  
217        well as employed extruder parameters. The elongation at break is significantly higher  
218        for neat PCL compared to palm fibre/PCL biocomposites and this is expected as the  
219        PCL is a ductile matrix and the strain at break is matrix dominated property in this  
220        case. The tensile strength properties obtained in this study are in close agreement with  
221        the reported work by Mahdavi et al. (2010) where they have presented tensile strength  
222        of date palm fibre reinforced polyethylene composites. In comparison, the results  
223        obtained (24 MPa) in this work were found to be higher than their reported results  
224        using rachis 20 wt.% from date palm as reinforcement in polyethylene matrix. In our  
225        case, the use of a PCL matrix, having more interfacial compatibility with plant  
226        components, is a significant advantage

227 Furthermore, the effect of the screw rotation speed (100 and 400 rpm) was tested  
228 during compounding. There is a slight variation in terms of measured tensile Young's  
229 modulus and tensile strength (Table 2). The rotation speed also seems to affect the  
230 palm fibre morphologies (Fig. 1) with Lw of 985 and 765 when extruded at 100 and  
231 400 rpm, respectively. Because some extrusion parameters, including screw speed, are  
232 known to impact lignocellulosic fibre morphologies, one could have expected  
233 pronounced differences in composite tensile properties between those two. Indeed, the  
234 feed rate, (Beaugrand and Berzin, 2013; Berzin et al., 2014), the recycling (Bourmaud  
235 et al., 2016), could impact on the fibre morphology which in turn impacts mechanical  
236 properties (Beaugrand and Berzin, 2013) or processing alteration (Ismail et al., 2016).  
237 Because screw rotation speed is known to modulate the specific mechanical energy  
238 (SME), the commercial software Ludovic© was used to calculate SME transmitted to  
239 the composite all along the extruder. This commercialised software is based on a one-  
240 dimensional (1D) approach. It allows the calculation of the main thermomechanical  
241 parameters of the process, from the hopper to the die exit, including solid conveying,  
242 melting and melt conveying. It has been shown that the fibre fragmentation depended  
243 on the SME provided to the fibres (Beaugrand and Berzin, 2013; Berzin et al., 2014,  
244 2017). As an example, Fig. 4 shows the evolution along the screw profile of the global  
245 SME that we can compare to experimental values calculated from the measured  
246 torque. It is observed that the SME increases in the left handed element, in the blocks  
247 of kneading discs and in the filled section prior to the die. Marked differences  
248 between 100 rpm and 400 rpm are then obtained.

249 However, there is no pronounced difference in tensile properties between samples  
250 made at 100 and 400 rpm. A close look at the length population distribution (Fig. 1  
251 box plots) sheds a new light. Indeed, the overall length distribution is first quite

disperse, and additionally the trend is not so different, despite the distinct Lw. This illustrates that only one average or median value is generally not enough to describe lignocellulosic fibre morphology (Di Giuseppe et al., 2016; Hamdi et al., 2015)

Table 2

The materials selection chart in Fig. 3 enables comparison of the tensile strength and stiffness of the injection moulded date palm fibre composites with other bast fibre composites. We find that the date palm fibre composites have comparable strength to other injection moulded bast fibre composites and neat resins, however the stiffness is much lower. This is explained by the substantially lower stiffness of date palm fibres in comparison to bast fibres like flax, jute and hemp (Table 1). Anecdotal observations from tensile properties of compression moulded, unidirectional date palm fibre composites reveal substantially lower strength and stiffness in comparison to compression moulded, unidirectional bast fibre composites. Rather the former have similar performance to compression moulded, non-woven (2D random fibre orientation) bast fibre composites.

### ***3.2. Low velocity impact characteristics***

#### ***3.2.1 Force-displacement analysis***

To evaluate impact damage characteristics, criteria such as maximum force, energy absorbed, force displacement and visual observation were used. Force versus displacement traces obtained from the low velocity impact testing for palm fibre/PCL specimens impacted at two different energy levels are depicted in Fig. 6 (a). Contact force is generally defined as reaction force exerted by the specimen to the impactor. As shown in Fig. 6 (a), the maximum contact force has slightly increased with the increase in incident energy level. From these curves, it can be seen that the increase in

energy level from 25 to 50 J has not caused a major difference in peak load. The maximum load reached for 25 J impacted specimen is approximately 2425 N, with a displacement of approximately 8 mm at peak force.

The peak force for specimens impacted at energy level of 50 J has been recorded at approximately 2535 N and unlike 25 J energy level, the curve for this specimens has reached zero which is an indication of samples fully perforated.

Fig. 6 (a-c).

### **3.2.2 Force-time comparisons**

The contact force-time traces corresponding to impact event for each specimen subjected to incident energy of 25 and 50 J are illustrated in Fig. 6 (b). The time taken to complete the impact event is different for each specimen. It is evident that at the lower energy level, the palm fibre/PCL biocomposite shows more impact resistance, thereby taking longer time to complete the impact event compared to higher energy impacted samples.

### **3.2.3 Energy-time comparisons**

The impact strength of composites is defined by its ability to withstand fracture or the amount of energy required to initiate damage. Absorbed energy versus time plots for different specimens are shown in Fig. 6 (c). It is evident from the figure that the absorbed impact energy for both samples are almost identical. However, the visual inspection of impacted samples at 50 J incident energy level shows a big difference than that of incident energy of 25 J. On the samples being impacted at the incident energy of 50 J, the material was fully penetrated. This is due to the high incident energy allowing the impactor to penetrate.

Fig. 7 (a) –(c) show the impact parameters (peak load and absorbed energy) of hemp/PLC biocomposites used to compare the results obtained for palm/PCL biocomposite specimens. It is evidenced from the Figure 7 (a) that hemp/PCL specimens have lower peak load and displacement compared to palm/PCL specimens. Similarly, the hemp/PCL specimens absorbed low impact energy (lower dissipated energy) as depicted in Fig. 7 (c) compared to palm/PCL specimens. This results are evidences that the palm/PCL biocomposites can be promising sustainable materials when improved impact performance is critical.

Fig. 7

### 3.3 *Impact damage characterisation*

The visual inspection of palm/PCL impacted at 25 J (Fig. 8) clearly shows that the specimens have not been fully penetrated but there is a clear indication of radial cracking and perforation.

Fig. 8.

For samples impacted at higher energy level (50 J), there is evidence of matrix cracking around the impacted hole, as energy was not distributed throughout the specimen and it was penetrated through (Fig. 9). The higher velocity of impact event allowed the impactor to puncture the samples. The damage area and size for this sample was larger than the samples impacted at 25 J incident impact energy.

Fig. 9.

The rear faces of all specimens show similar damage propagation. However, larger penetration hole and fibre shear out are visible for specimen impacted at higher energy level (Fig. 9). The extent of damage is larger for samples with 50 J incident energy level.

### 3.4 *Fractured surface of impacted samples*

Representative SEM images of impact fractured surfaces of these two samples are shown in Figs. 10 and 11. From SEM images of the impact at incident energy level of 25 J specimens (Fig. 10), it is observed that matrix bending and fibre delamination are apparent. The SEM images suggest that there was poor fibre matrix interface leading to better energy dissipation which has resulted in better impact resistance behaviour but lower tensile strength and modulus. For higher incident energy level, the fractured surfaces reveal that the fibres are exposed, unravelled and also pulled out. The PCL is a ductile matrix while the SEM images (Fig. 11, b) show no residue of matrix on the pulled out fibre which is an indication of poor fibre-matrix adhesion. This behaviour normally leads to better toughness properties and lower tensile strength and modulus. It can be observed from SEM images that there are some gaps between matrix and fibre which is an indication of insufficient consolidation of fibres during the manufacturing and caused by incompatibility between fibre and the matrix.

Fig. 10.

Fig. 11.

## 4. **Conclusions**

Using date palm fibres from waste as reinforcements in PCL based biocomposites was found to be effective approach for obtaining an acceptable tensile and impact resistance behaviour. It was evident from the experimental results that samples with higher extrusion screw speed have average fibre length shorter than those extruded with lower screw speed (400 vs 100 rpm) but the specimen composites do not withstand more than a slightly higher tensile stress than that of 100 rpm, arguably due to a very disperse fibre morphology population. However, the tensile test results

347 showed that date palm fibre reinforced PCL biocomposite laminates have acceptable  
348 tensile strength and modulus with respect to currently available literature data.

349 With regards to low velocity impact resistance behaviour, both samples showed  
350 similar behaviour apart from 25 J incident energy level showing resistance to  
351 penetration in comparison to 50 J of incident energy.

352 Investigating the suitability of date palm fibres biomass waste as reinforcement in  
353 light weight composite materials shows a tremendous opportunity of utilising this  
354 material to develop a low cost composites. Hence, it can contribute to reduce the  
355 overall environmental damage, better way of utilisation of resources and reduction of  
356 total cost for the manufacturing of composites, which is a clear benefit in many  
357 applications. Furthermore, the findings of this study has a great potential of benefitting  
358 composite manufacturing industries, research institutes and academia, as they work or  
359 study/research to improve the properties of natural fiber-reinforced biocomposite materials.

## 360 **Acknowledgements**

361 Johnny Beaugrand acknowledges the financial support from the MATRICE ‘CPER  
362 France-Champagne-Ardenne’ program and Justine Padovani (INRA) is acknowledged  
363 for her critical discussion. The authors thank Francois Gaudard from INRA for his  
364 technical assistance in polymer extraction and the authors also express their thanks to  
365 Miguel Pernes and Alain Lemaitre (both from INRA) for their support during twin  
366 screw extrusion. Hom Dhakal would like to thank Mr Raphael Teles for his support in  
367 carrying out impact testing and SEM during his placement period here at the APC  
368 Research Group, UoP.

369 **Funding:** This research did not receive any specific grant from funding agencies in  
370 the public, commercial, or not-for-profit sectors.

## 371 **References:**

372 Abdelaziz, S., Guessasma, S., Bouaziz, A., Hamzaoui, R., Beaugrand, J., Soud, A.A.,  
373 2016. Date palm spikelet in mortar: Testing and modelling to reveal the  
374 mechanical performance. *Constr. Build. Mater.* 124, 228–236.

375 Alawar, A., Hamed, A.M., Al-Kaabi, K., 2009. Characterization of treated date palm  
376 tree fiber as composite reinforcement. *Compos. Part B Eng.* 40, 601–606.

377 Alsaeed, T., Yousif, B.F., Ku, H., 2013. The potential of using date palm fibres as  
378 reinforcement for polymeric composites. *Mater. Des.* 43, 177–184.

379 Beaugrand, J., Berzin, F., 2013. Lignocellulosic fiber reinforced composites:  
380 Influence of compounding conditions on defibrization and mechanical  
381 properties. *J. Appl. Polym. Sci.* 128, 1227–1238.

382 Berzin, F., Vergnes, B., Beaugrand, J., 2014. Evolution of lignocellulosic fibre  
383 lengths along the screw profile during twin screw compounding with  
384 polycaprolactone. *Compos. Part A Appl. Sci. Manuf.* 59, 30–36.

385 Berzin, F., Beaugrand, J., Dobosz, S., Budtova, T., Vergnes, B., 2017. Lignocellulosic  
386 fiber breakage in a molten polymer. Part 3. Modelling of the dimensional  
387 evolution of the fibers during compounding by twin screw extrusion, *Compos.*  
388 *Part A*, 101, 422–431.

389 Bledzki, A.K., Gassan, J., 1999. Composites reinforced with cellulose based fibres.  
390 *Prog. Polym. Sci.* 24, 221–274.

391 Bourmaud, A., Åkesson, D., Beaugrand, J., Le Duigou, A., Skrifvars, M., Baley, C.,  
392 2016. Recycling of L-Poly-(lactide)-Poly-(butylene-succinate)-flax  
393 biocomposite. *Polym. Degrad. Stab.* 128, 77–88.

394 Chao, C.C.T., Krueger, R.R., 2007. The date palm (*Phoenix dactylifera* L.): Overview  
395 of biology, uses, and cultivation. *HortScience* 42, 1077–1082.

396 Charlet, K., Baley, C., Morvan, C., Jernot, J.P., Gomina, M., Bréard, J., 2007.  
397 Characteristics of Hermès flax fibres as a function of their location in the stem  
398 and properties of the derived unidirectional composites. *Compos. Part A Appl.*  
399 *Sci. Manuf.* 38, 1912–1921.

400 Dhakal, H.N., Zhang, Z.Y., Richardson, M.O.W., 2007. Effect of water absorption on  
401 the mechanical properties of hemp fibre reinforced unsaturated polyester  
402 composites. *Compos. Sci. Technol.* 67, 1674–1683.

403 Di Giuseppe, E., Castellani, R., Dobosz, S., Malvestio, J., Berzin, F., Beaugrand, J.,  
404 Delisée, C., Vergnes, B., Budtova, T., 2016. Reliability evaluation of automated  
405 analysis, 2D scanner, and micro-tomography methods for measuring fiber  
406 dimensions in polymer-lignocellulosic fiber composites. *Compos. Part A Appl.*  
407 *Sci. Manuf.* 90, 320–329.

408 Faruk, O., Bledzki, A.K., Fink, H.-P., Sain, M., 2012. Biocomposites reinforced with  
409 natural fibers: 2000–2010. *Prog. Polym. Sci.* 37, 1552–1596.

410 Haag, K., Padovani, J., Fita, S., Trouvé, J.P., Pineau, C., Hawkins, S., De Jong, H.,  
411 Deyholos, M.K., Chabbert, B., Müssig, J., Beaugrand, J., 2017. Influence of flax  
412 fibre variety and year-to-year variability on composite properties. *Ind. Crops*  
413 *Prod.* 98, 1–9.

414 Hamdi, S.E., Delisée, C., Malvestio, J., Da Silva, N., Le Duc, A., Beaugrand, J., 2015.  
415 X-ray computed microtomography and 2D image analysis for morphological  
416 characterization of short lignocellulosic fibers raw materials: A benchmark  
417 survey. *Compos. Part A Appl. Sci. Manuf.* 76, 1–9.

418 Ismail, S.O., Dhakal, H.N., Dimla, E., Beaugrand, J., Popov, I., 2016. Effects of  
419 drilling parameters and aspect ratios on delamination and surface roughness of  
420 lignocellulosic HFRP composite laminates. *J. Appl. Polym. Sci.* 133, 1–8.

421 Joshi, S. V., Drzal, L.T., Mohanty, A.K., Arora, S., 2004. Are natural fiber composites



environmentally superior to glass fiber reinforced composites? *Compos. Part A Appl. Sci. Manuf.* 35, 371–376.

Mohanty, A.K., Misra, M., Hinrichsen, G., 2000. Biofibres, biodegradable polymers and biocomposites: An overview. *Macromol. Mater. Eng.* 276–277, 1–24.

Nasser, R., Salem, M., Hiziroglu, S., Al-Mefarrej, H., Mohareb, A., Alam, M., Aref, I., 2016. Chemical Analysis of Different Parts of Date Palm (*Phoenix dactylifera* L.) Using Ultimate, Proximate and Thermo-Gravimetric Techniques for Energy Production. *Energies* 9, 374.

Paul, V., Kanny, K., Redhi, G.G., 2015. Mechanical, thermal and morphological properties of a bio-based composite derived from banana plant source. *Compos. Part A Appl. Sci. Manuf.* 68, 90–100.

Pickering, K.L., Efendy, M.G.A., Le, T.M., 2016. A review of recent developments in natural fibre composites and their mechanical performance. *Compos. Part A Appl. Sci. Manuf.* 83, 98–112.

Shah, D.U., 2014. Natural fibre composites: Comprehensive Ashby-type materials selection charts. *Mater. Des.* 62, 21–31.

Stamboulis, A., Baillie, C.A., Peijs, T., 2001. Effects of environmental conditions on mechanical and physical properties of flax fibers. *Compos. Part A Appl. Sci. Manuf.* 32, 1105–1115.

Summerscales, J., Dissanayake, N.P.J., Virk, A.S., Hall, W., 2010. A review of bast fibres and their composites. Part 1 – Fibres as reinforcements. *Compos. Part A Appl. Sci. Manuf.* 41, 1329–1335.

Vergnes, B., Della Valle, G., Delamare, L., 1998. A global computer software for polymer flows in corotating twin screw extruders, *Polym. Eng. Sci.*, 38, 1781–1792.

#### Figure captions:

Fig. 1. Schematic representation of sequence and processing steps of Date Palm Tree grown in Kingdom of Saudi Arabia and fibre extracted from leaf sheath a) Date Palm Tree grown in Kingdom of Saudi Arabia and b) fibre (fibrillum) extracted from leaf sheath. c) palm fibers lot chopped prior to twin screw extrusion; d) palm fibres from c) extruded at 100 rpm after extraction of the PCL matrix, e) palm fibres from c) extruded at 400 rpm after extraction of the PCL matrix. The scale bar = 1 cm. Lower part: box plots corresponding to the three images.

Fig. 2. SEM images of date palm fibre used in this study showing pores morphology: (a) longitudinal section at lower magnification x 150; (b) at higher magnification x 500

Fig. 3. Ashby chart presenting strength versus modulus of our date palm composites (red) in an Ashby chart, comparing with other bast fibre composites. Adapted from (Shah, 2014). Acronyms are: IM injection moulded, CM compression moulded, RTM resin transfer moulded, UD unidirectional, TP thermoplastic, TS thermoset

463 Fig. 4. Evolution of SME at 3.5 kg/h (○ : 100 rpm ; ● : 400 rpm) along the screw  
464 profile and comparison with experimental SME measured at the die exit (restrictive  
465 zones are in grey)

466 Fig. 5. Schematic of low velocity impact test set up

467 Fig. 6. (a) Force versus displacement, (b) Load versus time and (c) Work versus  
468 time traces of palm/PCL biocomposites.

469 Fig. 7. (a) Force versus displacement, (b) Load versus time and (c) Work versus  
470 time traces of hemp PCL for comparison purpose.

471 Fig. 8. Damage pattern of Palm/PCL specimens impacted at 25 Joules (a) front  
472 face damage and (b) rear face damage.

473 Fig. 9. Damage pattern of Palm/PCL specimens impacted at 50 Joules (a) front  
474 face damage and (b) rear face damage

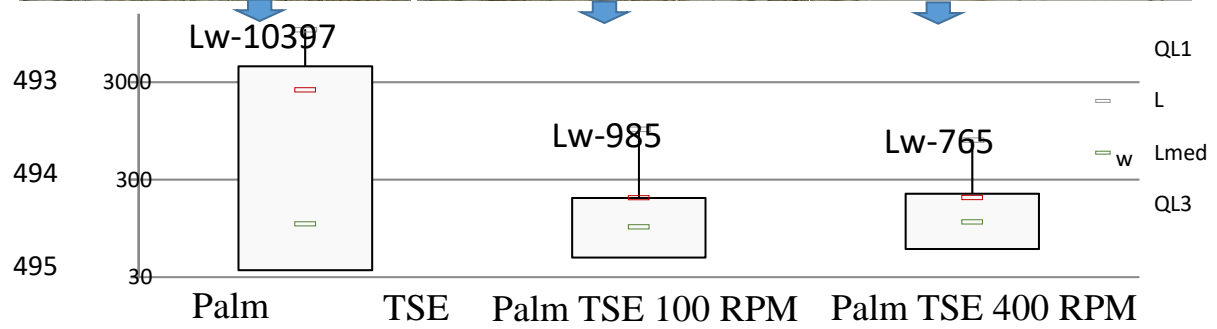
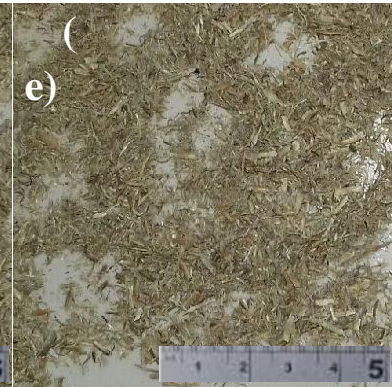
475 Fig. 10. SEM images of Palm/PCL after impact test at 25J, 1: delamination and  
476 debonding, 2: matrix bending, 3: fibres breakage, 4: fibre pull out, 5: pull out fibre  
477 side

478

479 Fig. 11. SEM images of Palm/PCL after impact test at 50J, 1: delamination, 2:  
480 matrix breaking, 3: fibres breakage

481

482



503 Fig. 1. Schematic representation of sequence and processing steps of Date Palm  
 504 Tree grown in Kingdom of Saudi Arabia and fibre extracted from leaf sheath a) Date  
 505 Palm Tree grown in Kingdom of Saudi Arabia and b) fibre (fibrillum) extracted from  
 506 leaf sheath. c) palm fibers lot chopped prior to twin screw extrusion; d) palm fibres  
 507 from c) extruded at 100 rpm after extraction of the PCL matrix, e) palm fibres from c)  
 508 extruded at 400 rpm after extraction of the PCL matrix. The scale bar = 1 cm. Lower  
 509 part: box plots corresponding to the three images.

510

511

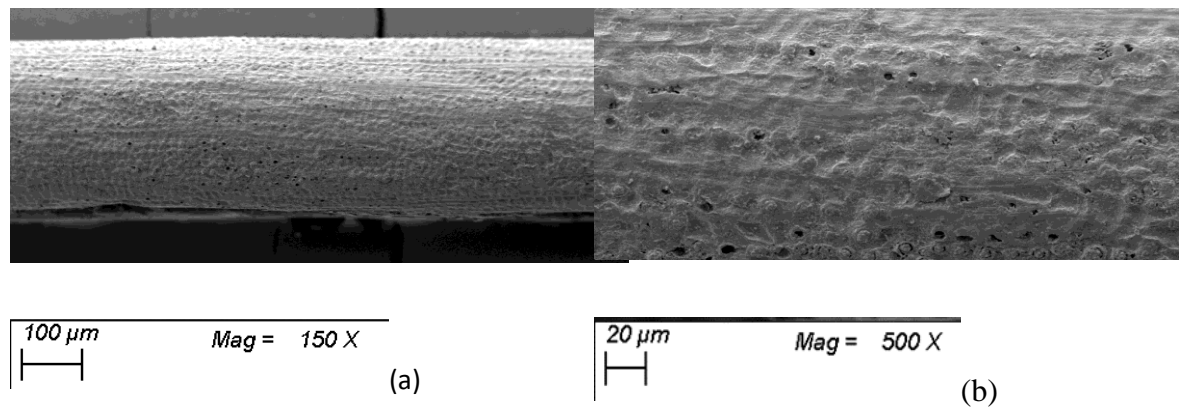


Fig. 2. SEM images of date palm fibre used in this study showing pores morphology: (a) longitudinal section at lower magnification x 150; (b) at higher magnification x 500.

512

513

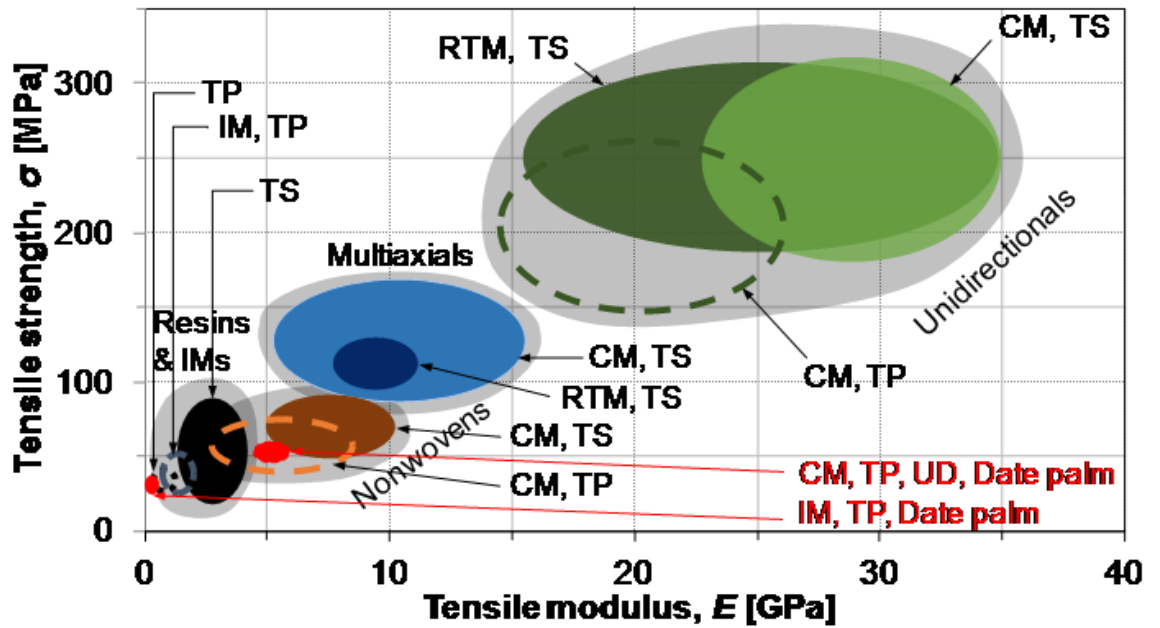
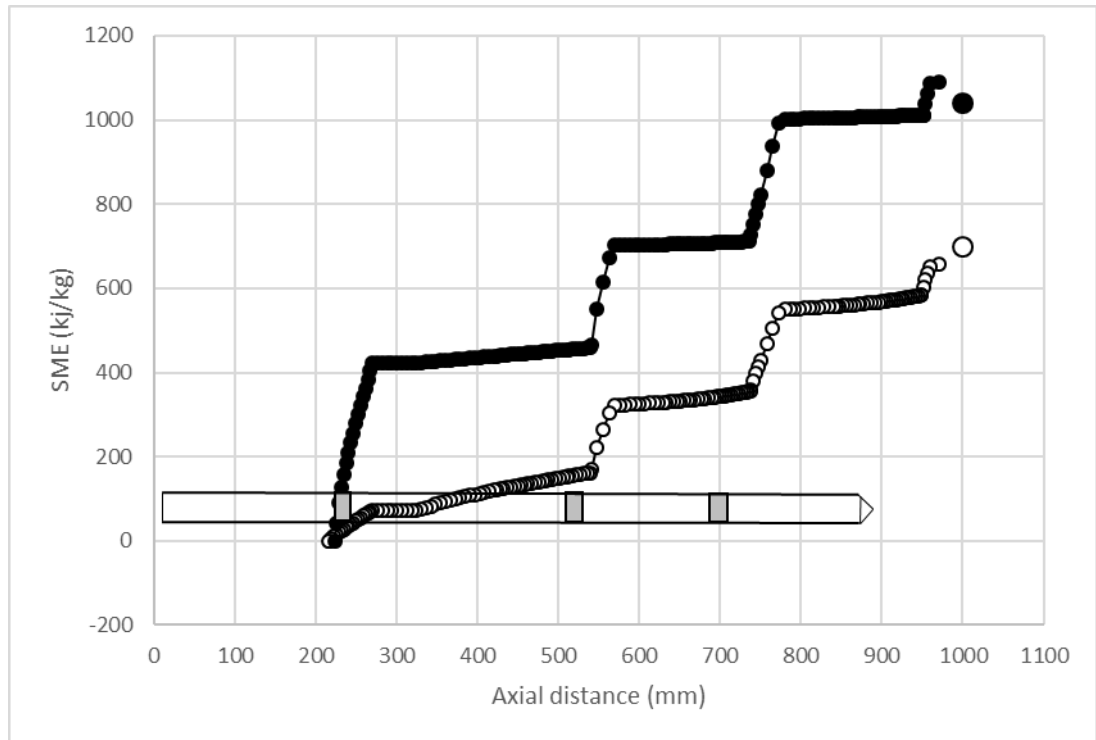


Fig. 3. Ashby's chart presenting strength versus modulus of our date palm composites (red) in an Ashby chart, comparing with other bast fibre composites. The strength versus modulus in an Ashby's chart (adapted from REF: Shah, 2014). Acronyms are: IM injection moulded, CM compression moulded, RTM resin transfer moulded, UD unidirectional, TP thermoplastic, TS thermoset.

514

515

516



517

518 Fig. 4. Evolution of SME at 3.5 kg/h (○ : 100 rpm ; ● : 400 rpm) along the screw  
 519 profile and comparison with experimental SME measured at the die exit (restrictive  
 520 zones are in grey).

521

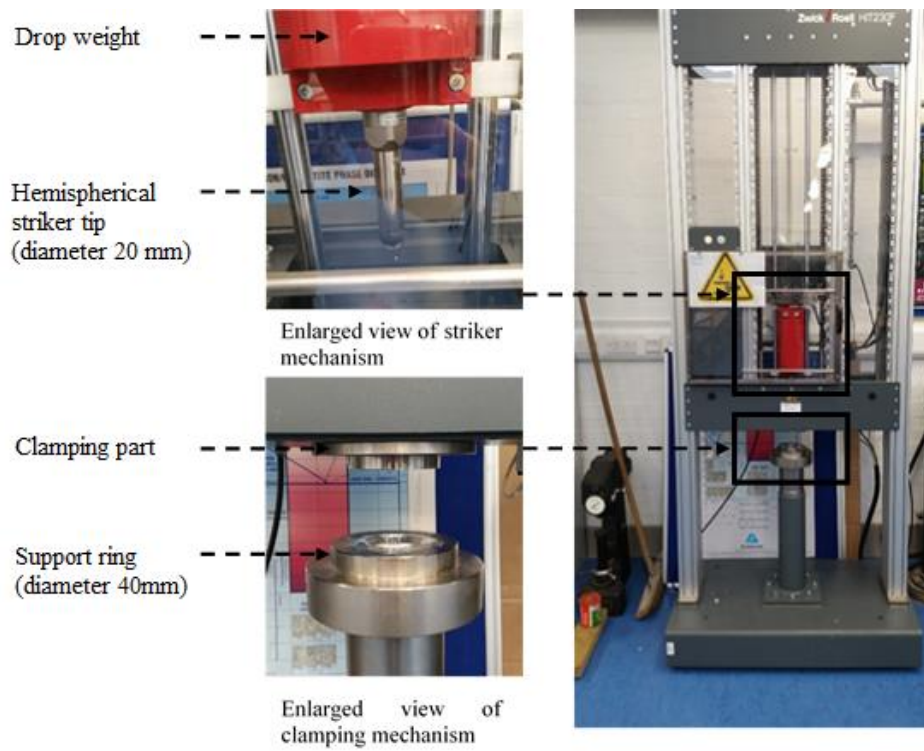
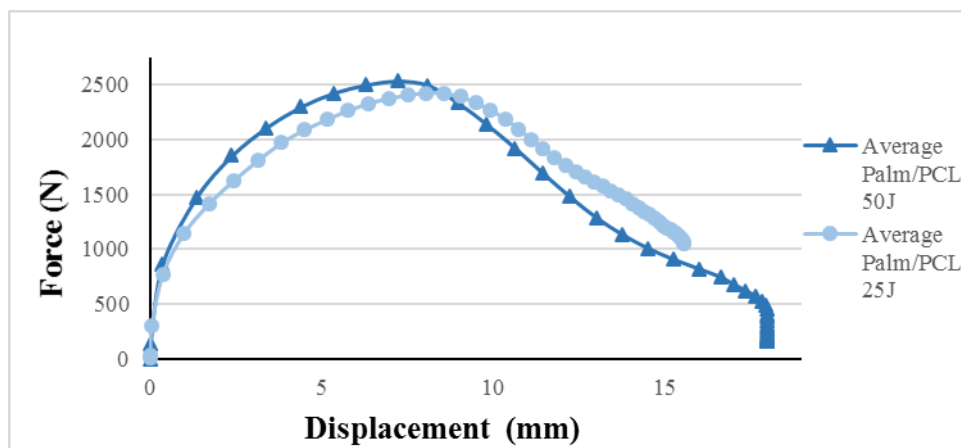
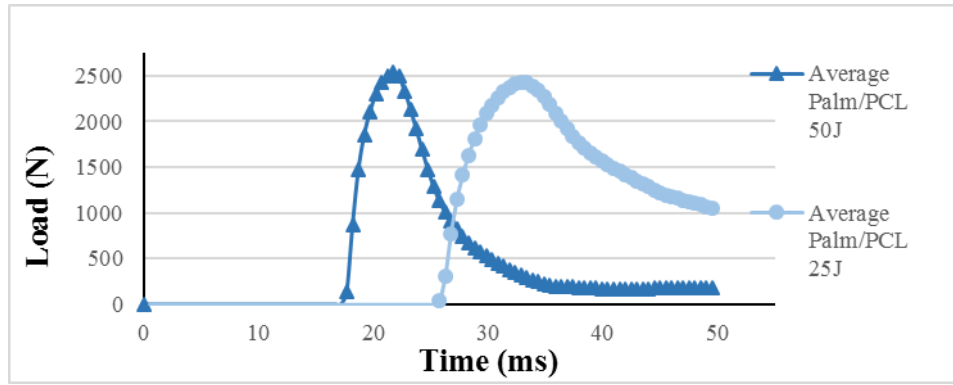


Fig. 5. Schematic of low velocity impact test set up.

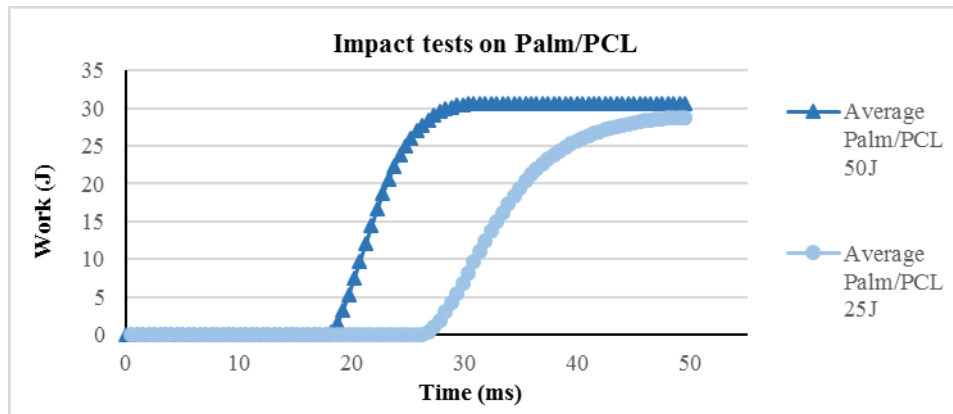
522



(a)

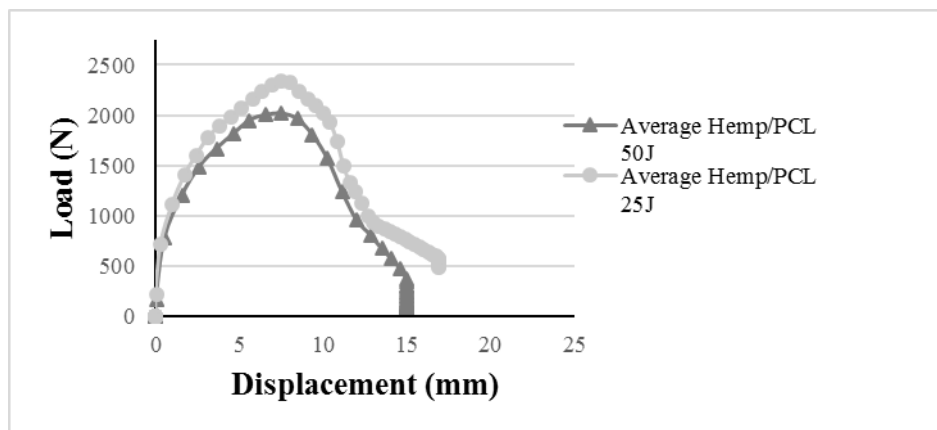


(b)



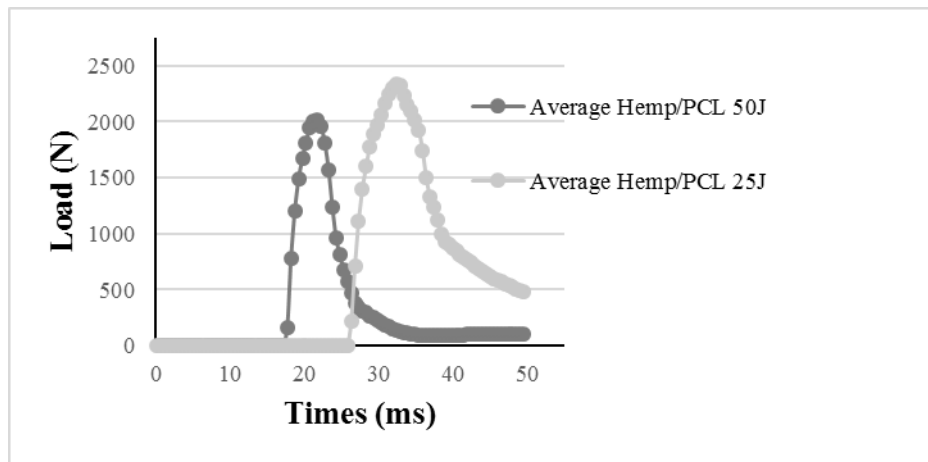
(c)

Fig. 6. (a) Force versus displacement, (b) Load versus time and (c) Work versus time traces of palm/PCL biocomposites.

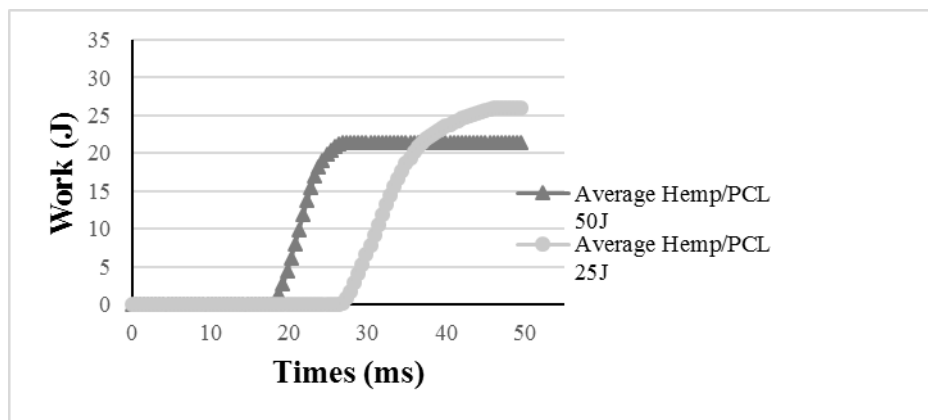


(a)





(b)



(c)

Fig. 7. (a) Force versus displacement, (b) Load versus time and (c) Work versus time traces of hemp PCL for comparison purpose.

523

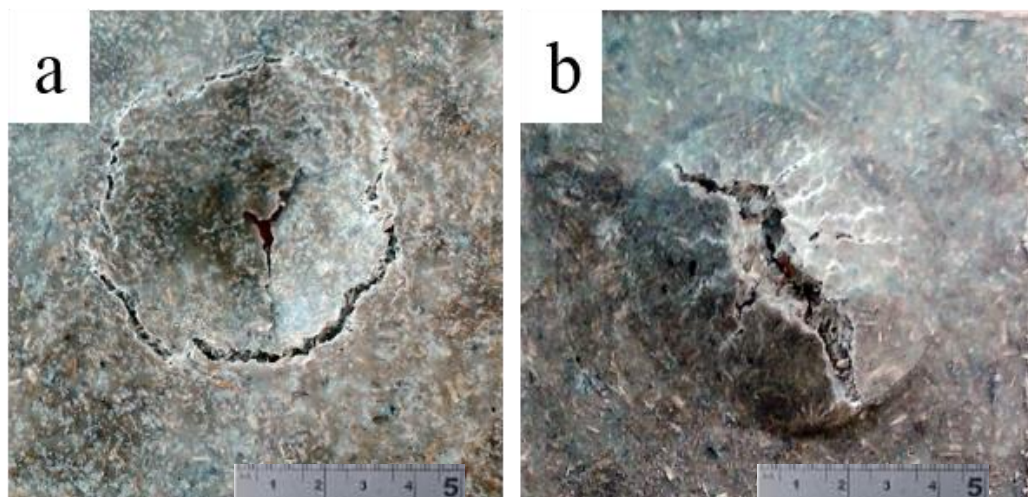


Fig. 8. Damage pattern of Palm/PCL specimens impacted at 25 Joules (a) front face damage and (b) rear face damage.

524

525

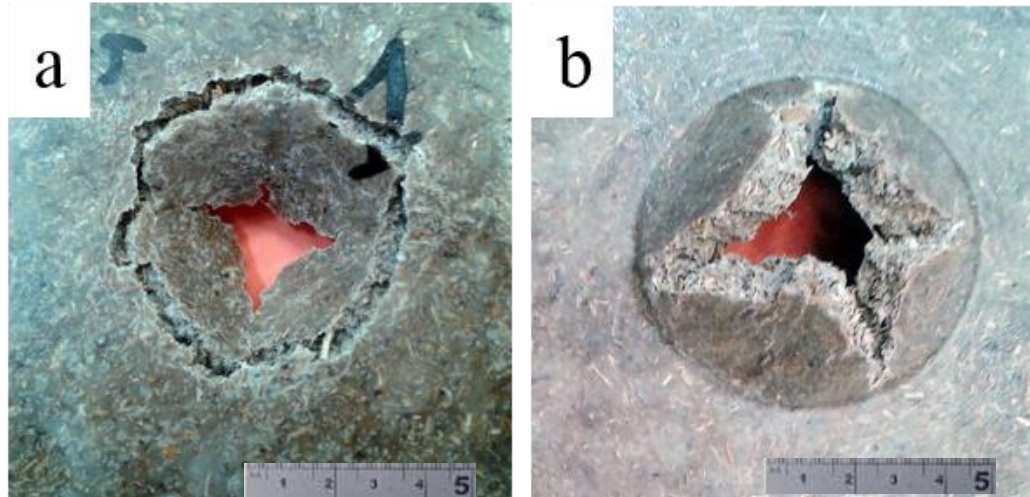


Fig. 9. Damage pattern of Palm/PCL specimens impacted at 50 Joules (a) front face damage and (b) rear face damage.

526

527

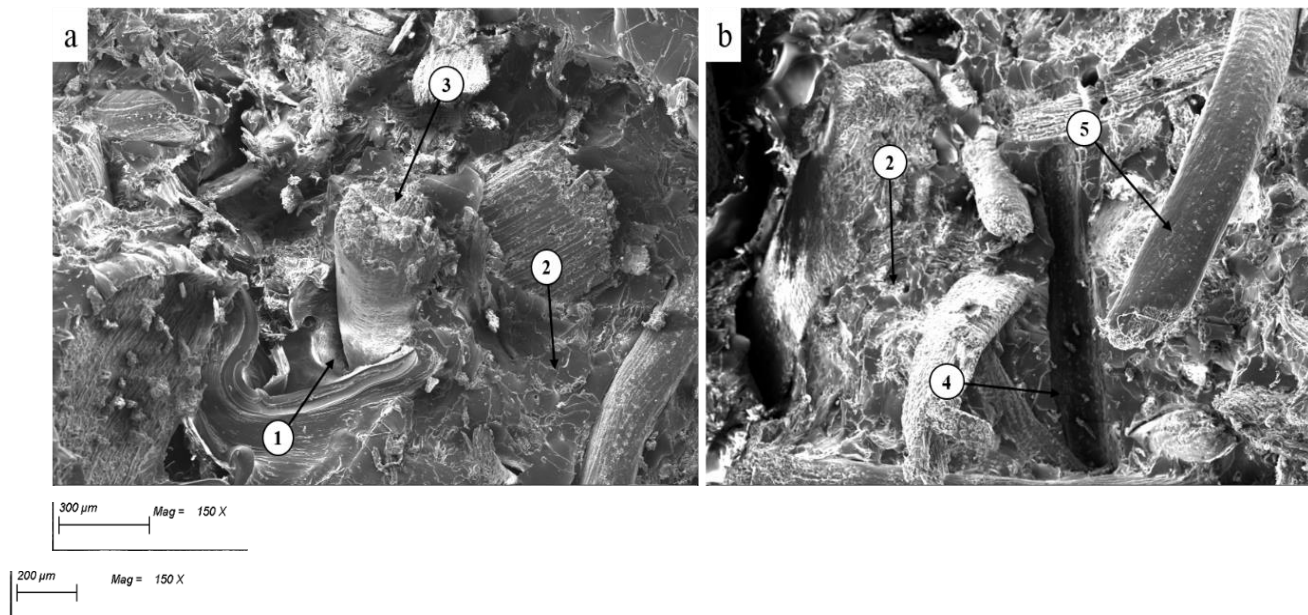


Fig. 10. SEM images of Palm/PCL after impact test at 25J, 1: delamination and debonding, 2: matrix bending, 3: fibres breakage, 4: fibre pull out, 5: pull out fibre side.

528

529

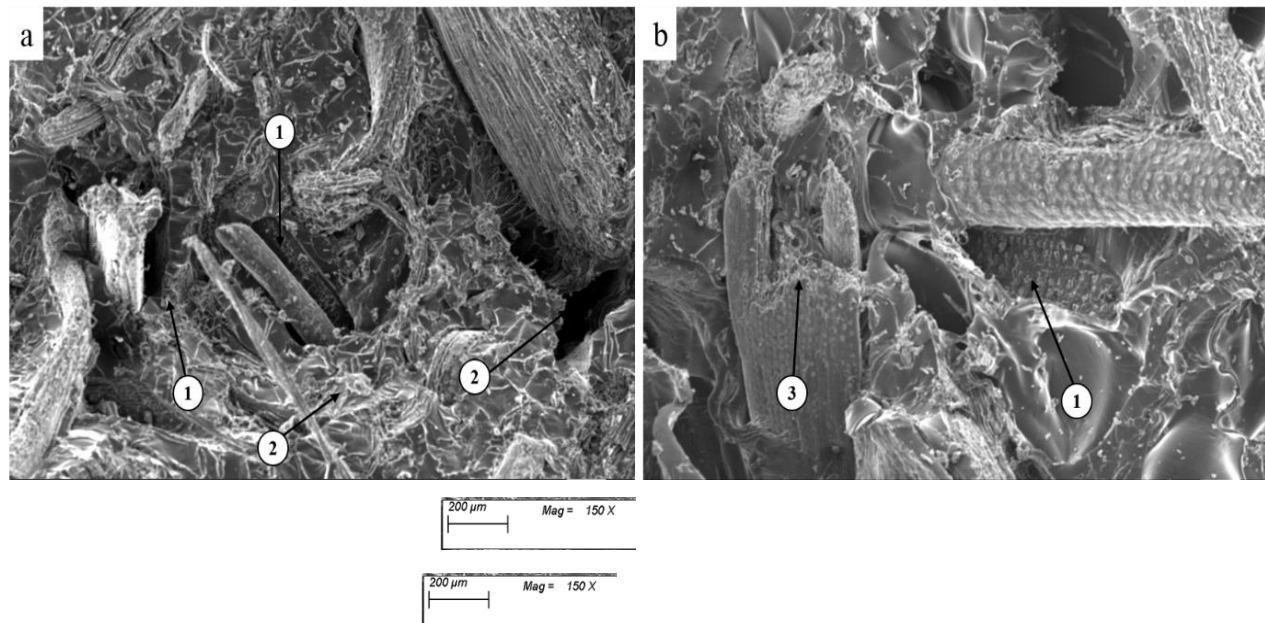


Fig. 11. SEM pictures of Palm/PCL after impact test at 50J, 1: delamination, 2: matrix breaking, 3: fibres breakage.

530

531 Table captions:

532 Table 1: Comparative physical and mechanical properties of date palm fibre  
 533 (bundles) with commonly used bast fibres (Dhakal et al., 2007; Mohanty et al., 2000;  
 534 Pickering et al., 2016).

Fibre types	Density (g/cm <sup>3</sup> )	Tensile strength at break (MPa)	Tensile modulus (GPa)	Elongation at break (%)
Date palm	0.92	170-275	5-12	5-10
Hemp	1.14	550-1110	58-70	1.6
Flax	1.50	345-1100	27.6	2.7-3.2
Jute	1.3-1.45	393-773	13-26.5	1.16-1.5

535

536

537 Table 2: Effects of palm fibre reinforcement on tensile properties of different  
 538 palm/PCL biocomposites.

Specimen types	Tensile strength at break (MPa)	Tensile modulus (MPa)	Tensile strain (%)
PCL neat	19.00 (±1.70)	140.00 (±24.0)	418.00 (±60.0)
PCL Palm 20 wt.% 100 RPM	24.00 (±2.90)	284.00 (±15.20)	21.00 (±4.0)
PCL Palm 20 wt.% 400 RPM	25.00 (±0.70)	279.50 (±14.50)	24.00 (±3.0)

539

540

541 **Highlights:**

- 542       • The focus of the study was valorisation of waste agriculture biomass leaf sheath date
- 543       palm fibre through the development of composite laminates.
- 544       • Fully biodegradable date palm/PCL based biocomposites developed and their
- 545       damage mechanisms under LVI investigated.
- 546       • Important mechanical properties were investigated and compared against
- 547       hemp PCL biocomposites.
- 548       • The developed biocomposites have high potential to be utilized for lightweight
- 549       engineering applications.

550

551