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A study of mechanical and morphological properties of PLA based biocomposites prepared with EJO vegetable oil based plasticiser and kenaf fibres

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Abstract. A novel environmental friendly plasticized PLA kenaf biocomposites were fabricated by melt blending technique through a combination of epoxidized jatropha oil, one such non-edible vegetable oil material and renewable plasticizer. The mechanical and morphological properties of the composites were evaluated. The addition of EJO with 40 % kenaf fibre loading on PLA had improved the mechanical properties of the polymer composites. SEM micrographs clearly revealed the good physical interlocking for PLA and kenaf fibre at low loading levels (40 wt.%) while higher content (50 wt.%) of kenaf contributed to poor filler-matrix compatibility attributes that results in voids. Alkalisation of kenaf fibre provides good interaction bonding between kenaf and PLA matrix which resulted on increased value of mechanical properties of treated biocomposites compared with the untreated biocomposites.

1. Introduction
Recently, the growing environmental awareness together with the new rules and regulations have forced the industries to seek an alternative for environmentally and ecologically friendly materials for their products. The environmental impact caused by exploitation of plastics represent the largest proportion of domestic waste and contribute to the polymer pollution. Therefore, the focus on these day is developing environmentally friendly materials which are naturally degradable when disposed in the environment [1].

Biodegradable polymers have received much attention as they offer environmental benefits such as biodegradability, greenhouse gas emissions and renewability of the base material [2]. Poly(lactic) acid (PLA), a corn-based polymer is the most promising biodegradable polymer and have been introduced commercially in the market as an alternative to conventional synthetic polymers [3-4]. There are number of promising products in the market for this polymer, such as automotive parts from PLA since PLA based automotive parts emit less CO₂ compared to the other petroleum based-thermoplastics [5]. Other than that, PLA has high strength, thermal plasticity and biocompatibility [6]. This polymer provides good strength and easy processability in most equipment, however it is considered expensive and the physical properties such as brittleness limit the PLA polymer applications [7]. Hence, a way to improve the mechanical and thermal properties of PLA as well as to make it more cost effective material is by the addition of fibre or filler materials to the system [8].

Natural fibres such as flax [9-10], hemp [11], jute [12] or kenaf [13] have recently gained more attention in replacing synthetic fibres and been used for energy or biomaterials production for many years.
in order to replace or conserve fossil fuels. Moreover, the potential advantages of these natural fibres have been well documented such as low cost, good thermal properties, low density and non-abrasive processing [14]. Thus, it is crucial to utilize these natural fibres and incorporate them into biodegradable polymer such as PLA in order to improve the properties of PLA as well as effectively reduce the production cost by partially replacing the high price of PLA by low cost natural fibres without destroying the biodegradability of the polymer matrix.

Over the past few years, natural fibres reinforced polymer composites have attracted increasing attention and have been considered for various applications due to their lower cost, good mechanical properties and lightweight. The use of natural fibres offers the advantage of weight reduction (over 10-30%), good mechanical and manufacturing properties, high stability, no emission of toxic substances and overall reduction in cost. In the field of technical utilisation of natural fibres, kenaf reinforced composites are considered as one of the most important areas [2].

In the present study, kenaf fibre with 40 wt.% fibre content reinforced PLA composites were processed by melt blending and followed by hot and cold press method. However, the inferior fibre-matrix adhesion arises from the hydrophilic nature of the kenaf fibres have limited compatibility with the hydrophobic nature of PLA thermoplastic. The degree of bonding or adhesion between the fibre and the matrix at interface is extremely important as the modulus, strength and toughness of a composite relies on the chemical interactions or mechanical interlocking between the fibre and the matrix. Over the last decades, several approaches have been introduced towards enhancing interfacial adhesion including the use of MAPP and silane coupling agents [15], chemical modifications such as acid [16] and alkali [17]. Due to the nature of kenaf fibre, NaOH treatment was used to improve the interaction compatibility between PLA and kenaf fibre. The purpose of chemical modification of natural fibre is to improve interaction of fibre with polymer matrix. Several findings have revealed that the chemical treatments improve the properties of composites. By improving the fibre/matrix adhesion in this manner, the mechanical properties of the composite will be improved at the expense of a reduction in impact behavior [18].

Plasticiser produced from natural resources, are currently being employed in a biodegradable polymer, as a replacement for traditionally used phthalates in the plastic industry, which are characterised by their high toxicity [19]. Recent years, modified vegetable oils have gained a lot of interest as a renewable source of plasticiser for polymers (Robertson et al., 2010). Numerous research works have been reported on the use of epoxidized vegetable oil (EVO) as a plasticiser for the PLA composites [20-21]. There are few advantages on the use of epoxidized vegetable oil in polymer industry: biodegradable, environmentally friendly, renewable and possibly cheaper [22]. Furthermore, it has potential to be used as an additive for polymer industry that require properties, such as good lubricity, low vitality, high index viscosity and good solvency [23].

Research on composite materials based on polymers from vegetable oils has been developed and be used for industrial applications as well. The study and research in the literature dealing with the use of vegetable oil based resins in natural fibre reinforced composites are emerging from time to time [24-25]. Jatropha oil with higher content of monounsaturated oleic acid (C18:1) and polyunsaturated linoleic acid (C18:2) compared to palm oil and soybean oil which gives higher percentage of oxirane oxygen content (OOC) upon epoxidation process had been widely studied as plasticiser in PLA biocomposites. At least, to our knowledge, there is presently no published work on using EJO as plasticizer for PLA/natural fibre system. Hence, with the present work, it was aimed to introduce and propose the potential of EJO as a reactive plasticiser together with a combination of kenaf as natural fibre for a reinforcement to the PLA composite system.

2. Experimental

2.1 Material
Poly(lactic acid) (PLA) polymer resins of grade 2003 D was obtained from the Nature Works LLC Company from USA. Meanwhile, crude jatropha oil which is a non-food grade material was supplied by the Bionas Sdn Bhd, Kuala Lumpur, Malaysia and was used as received. Kenaf fibre in the size of 40 micrometer mesh was obtained from the National Kenaf Tobacco Board, Kelantan. Reagent grade hydrogen peroxide 30% and magnesium sulphate were supplied by Merck, Germany. Sodium hydroxide reagent grade and formic acid were supplied by Systerm.

2.2 Epoxidation of Jatropha Oil
The epoxidation of jatropha oil was carried out by “in-situ” epoxidation method as described by Goud et al., (2007) with a slight modification in the molar ratio of 1:0.6:1.7– organic acid: double bonds: hydrogen peroxide at temperature of 60 °C for 5 hours reaction time.

2.3 Fibre Treatment
Fibres were treated with alkali soaked in hot distilled water with continuous stirring at 60°C for 4 hours to remove excess wax and other impurities. Then, the treated fibre was washed with distilled water until reaching pH7 and dried at temperature 60 °C for 24 hours in the oven.

2.4 Preparation of PLA/Kenaf Treated/EJO (PLA/TK/EJO) Biocomposites
The PLA/KFT/EJO blends were prepared by the melt blending technique of PLA with 3 wt.% epoxidized jatropha oil and 40 wt.% ratio of treated kenaf fibre. The melt blending was carried out using a Brabender Internal Mixer (Germany) at 170 °C for 10 min and 50 rpm motor speed. After melt blending, the composites were removed and cooled at room temperature. The composite samples were then placed in the hot and cold press for a compression moulding with a pressure of 110 kg/cm at 170 °C for 11 min in order to produce biocomposite sheets of uniform 1mm and 3mm thickness. The biocomposites were then being analysed for their physical, chemical, mechanical and morphological properties characterizations. Prior to mixing process, PLA pellets and kenaf fibre were dried in oven at 60 °C for 24 hours to remove moisture content.

2.5 Characterisations
Fibres were treated with alkali soaked in hot distilled water with continuous stirring at 60°C for 4 hours to remove excess wax and other impurities. Then, the treated fibre was washed with distilled water until reaching pH7 and dried at temperature 60 °C for 24 hours in the oven.

2.5.1 FTIR Analysis. The structural changes of untreated and treated kenaf fibre were analysed using (Perkin-Elmer: Model 1000 Series) instrument equipped with a universal attenuated total reflectance (UATR) accessory. The spectra were recorded between 4000 cm⁻¹ to 280 cm⁻¹ wavenumber range.

2.5.2 Fibre Image Analysis. The untreated and treated fibres were mounted for longitudinal view onto a microscope slide. The diameter of the fibres were measured using an optical microscope model Olympus SZX12.

2.5.3 Tensile Properties. The tensile properties: tensile strength and tensile modulus were determined by Toyoseiki Universal Testing Machine (Model Strograph R1), with a grip attachment distance of 45 mm. Load of 1 kN was applied at constant cross head speed of 5 mm/min. The neat PLA and composites sheets were cut into dumbbell shapes prior to use in accordance to ASTM D638-10 standard. The width and thickness of the specimens were measured at three different points and the average was calculated. The resultant value for tensile properties of neat PLA and composites were taken from an average of seven samples. Error bar represents one standard deviation for all presented data.

2.5.4 Surface Morphology Test. The surface morphology of untreated and treated kenaf fibre, PLA/KF, PLA/KFT and PLA/KFT/EJO were examined using Scanning electron microscopy (SEM JEOL model JSM-6300F) with an accelerating voltage of 20 kV. Possible fibre pull out, fibre splitting, debonding, matrix cracking and fibre-matrix adhesion were analysed. The specimens were coated with gold prior to each test to avoid electrostatic charging during electron irradiation.

3. Results and Discussion

3.1 FTIR and morphology analysis of kenaf fibre and PLA/TK/EJO biocomposites
FTIR spectroscopy is a useful technique to study the functional groups of both untreated and treated kenaf fibre and to monitor the effect of fibre treatment and chemical changes of the lignocellulosic fibre before and after the treatment. Figure 1 shows the IR spectra for untreated and treated kenaf fibre. A broad peak can be observed at region around 3300 cm⁻¹ indicating the presence of hydroxyl (OH) group in both but the intensity of the peak is lower for treated fibre due to the removal of hemicellulose and lignin. Alkali treatment reduces the hydrogen bonding due to the removal of the hydroxyl groups (-OH) by reacting with sodium hydroxide. The hydroxyl groups are also involved in hydrogen bonding with the carboxyl groups, perhaps also with the fatty acids that are available on the fibre surface of kenaf fibres. Thus, this was indicated by the reduction and broadening of the peaks around 3200 cm⁻¹. The absorbance peaks around 2900-2800 cm⁻¹ were attributed
to stretching of C-H group. In addition, the vibration peak of C-H stretches at 2900 cm\(^{-1}\) in cellulose and hemicellulose presents in the untreated fibre becomes weak, indicating the part of the hemicellulose was removed [26].

A peak at 1729.83 cm\(^{-1}\) that present for untreated fibre disappeared for treated fibre. According to [27] and [28], the peak is corresponding to the C=O stretching of the acetyl group in hemicellulose. [29] assigned the peak as the ester linkage of carboxylic group in the ferulic and p-coumeric acid of lignin and/or hemicellulose. The disappearance of this peak for treated fibre confirmed the removal of lignin and hemicellulose during mercerization process. Mercerization removes the waxy layer, adhesive pectins and hemicelluloses that bind fibre bundles to each other and to the pectin and hemicellulose rich sheaths of the core [30].

The intensity for peak at 1506.13 cm\(^{-1}\) for untreated fibre is decreasing after alkaline treatment process. The peak is associated to the C=C aromatic symmetrical stretching for lignin [31]. The absorbance between 1380-1320 cm\(^{-1}\) for both untreated and treated fibre were related to the bending vibration of C-H and C-O groups of the aromatic ring in polysaccharides. A peak at absorbance 1230.36 cm\(^{-1}\) for untreated fibre was found to be shifted to 1319.07 cm\(^{-1}\) after treatment process and the intensity of the peak also decreasing. According to [32], the peak is attributed to the aryl group in lignin, thus the change observed due to the removal of lignin after fibre treatment. Finally, the intense peaks around 1070-1010 cm\(^{-1}\) region were due to stretching of C-O and O-H groups [33].

![Figure 1. FTIR spectra of untreated and treated kenaf fibre.](image-url)
Figure 2 displays the FTIR spectrum of PLA, PLA/Kenaf (Untreated), PLA/Kenaf (Treated) and PLA/Kenaf (Treated)/EJO samples. Biocomposite sample is assumed to have a combination of PLA and fibre absorbance peak. However as for all three samples besides PLA spectrum, they had approximately similar wavelength as compared to PLA’s except the difference in the peak intensity in the C=O stretching and C-O stretching for esters. Those two peaks has shifted towards lower temperature, from 1748 cm\(^{-1}\) (neat PLA) to 1745 cm\(^{-1}\) in C=O stretching region and from 1184 cm\(^{-1}\) (neat PLA) to 1180 cm\(^{-1}\) and from 1079 cm\(^{-1}\) (neat PLA) to 1076 cm\(^{-1}\) in C-O stretching region indicated the miscibility and interaction between PLA, Kenaf and EJO took place. Furthermore, the shift may possibly due to an interaction between the hydroxyl group of PLA and epoxy group of EJO through hydrogen bonding (H-bonding) interaction, resulting in enhanced morphological properties which can be verified through SEM analysis in following Figure (Figure 4). A proposed possible site for interaction between PLA and EJO is shown in Figure 3.

In order to study the failure mechanism and to examine the possible fibre-matrix mechanism, the fracture surface of composites was studied using SEM [34]. Figure 4 shows the fractured surface micrographs of tensile specimen of neat PLA, PLA/KFUT, PLA/KFT and PLA/KFT/EJO samples respectively. It can be seen that the neat PLA presented an irregular and coarseness fracture surface, due to its highly brittle nature and failure occurs by micro-cracking where the cracks spread easily and freely. Based on the observation, a visible fibre
pull-out can be seen for all three biocomposites. The micrograph of PLA/KFUT displays a notable gaps between a fibre and a matrix. In addition, presence of aggregation and fibre breakage is visible. It is reported by [35], the gaps probably due to the debonding during mechanical testing or poor dispersion during composites preparations which indicates a weak fibre-matrix adhesion. Fibre-matrix interfacial bonding is primarily dependent on the degree of adhesion between the fibre and the matrix. An important aspect of the ultimate performance of the final composite is influenced by the act of means stress transfer from the matrix to the fibre. Thus, it is important to understand these affecting factors and ultimately improving the properties of the composite. Therefore, one way to modify and improving the fibre–matrix interfacial bonding is through the fibre surface modification. In order to make the fibres become more compatible with the matrix, various methods of treatments on the fibre such as bleaching, alkali, silane, benzoylation, acrylation and acetylene have been applied by many researchers [36-38]. As for matrix modification, addition of coupling agents is necessary to enhance fibre-matrix adhesion. Among all of these possible modification methods available, the most common and simply implemented method is alkaline treatment of the fibres using sodium hydroxide (NaOH) solution. Chemical modifications may introduce new moieties that can effectively interlock with the matrix. Mercerization of plant fibres have achieved various levels of success in improving fibre strength, compatibility and fibre matrix adhesion in natural fibre reinforced composites [39]. Therefore, it is considered that the alkalisation treatment applied in this study can provide a better properties to the PLA/KF/EO biocomposites with ultimate desired performance.

In Figure 4, while PLA/KFUT had many exposed fibres pulled out from the matrix and filler related failure, PLA/KFT and PLA/KF/EO had more embedded fibres and significantly voids had been reduced. As it can be observed from the SEM micrograph of tensile fracture surface of PLA/KFT in Figure 4, the filler related failures were significantly reduced. It is possible to see the wax and impurities had been disappeared and removed after alkaline treatment. Surface treatment facilitates good adhesion between kenaf and PLA matrix through increased surface roughness. Furthermore, kenaf fibres are well trapped by the PLA matrix. Better stress transfer from the PLA matrix to kenaf fibre is expected through occurrence of the thin layer by the matrix linking the fibre surface to the matrix. Thus, improved adhesion leads to a significant increase in a tensile properties.

As for PLA/KF/EO, good interfacial adhesion and better compatibility between the interfaces was observed as there was better wetting out of the PLA/EO onto kenaf fibres. Changes of surface topography and good fibre-matrix adhesion is required for effective stress transfer from matrix to the fibre [40]. It is worth noting that the enhancement in fibre-matrix adhesion explains the improvement in mechanical properties of PLA/KFT and PLA/KF/EO compared to the untreated one.
3.2 Mean Diameter of Kenaf fibre

Figure 5 displays the image analysis diameter of untreated and treated kenaf fibres. As can be seen, the diameter of untreated kenaf fibres are larger than those of treated fibres. The measurement diameter of untreated kenaf fibres are between 0.12 to 0.22 μm while for the treated fibres, the diameter average values are between 0.081 to 0.14 mm. The results indicated that the diameter of kenaf fibres had been reduced after treated with alkali concentration.

This might be due to the phenomenon of fibre degradation and fibre delignification that occurred in kenaf fibre during mercerization treatment process. Kenaf fibre became rougher and finer due to the leaching of non-cellulosic materials like pectin, hemicellulose and lignin [41]. In addition, it could be explained by the process of alkalisation treatment which removed a great amount of lignin from the fibres. The diameter of the fibre had been reduced due to the removal of lignin. The alkali treatment removes certain hemicellulose and wax covering the external surface of the fibre and therefore it decreases the aspect ratio (length/diameter) values of fibres. The results are in agreement with another work done by [42] whom reported that 30 minutes alkali treatment with 1 % alkali solution at 90 °C contributed to fibre diameter reduction at 32 %. Similar findings have been observed by [43] whom emphasized the degradation of pectin and removal of lignin from the fibre cell wall that causing separation of the ultimate fibre or fibrillation which lead to the fibre diameter decreased by using 0 to 0.03 % alkali concentration.

Figure 4. Surface morphology of (a) PLA (b) PLA/KFUT (c) PLA/KFT and (d) PLA/KFT/EJO

Figure 5. Diameter measurement of (a) Untreated and Treated (b) kenaf fibre.
3.3 Tensile properties of PLA, PLA/KFUT, PLA/KFT and PLA/KFT/EJO biocomposites

The results of tensile strength (TS) and modulus (TM) of neat PLA, PLA/KFUT, PLA/KFT and PLA/KFT/EJO are depicted in Figure 6. From the figure, it can be seen that the value of tensile strength for all three biocomposites are much lower as compared to the neat PLA. This decrement could be attributed to the weak interfacial interaction between PLA and kenaf fibre that leads to the inefficiency of stress transfer from PLA to kenaf fibre. Other than that, according to [44], a types of short fibre might have difficulties to support stress transfer from PLA and therefore lead to the strength reduction as fibre was added to PLA. Furthermore, it is also supported by the research statements from [45] and [46] whom claimed that this decrease is attributed due to the inability of the fibre, irregular shape to support stress transfer from the polymer matrix and poor interfacial bonding generate spaces between fibre and polymer matrix that leads to the weak structure in between them.

![Figure 6](image)

**Figure 6.** Tensile properties of PLA, PLA/KFUT, PLA/KFT and PLA/KFT/EJO biocomposites.

In this study, the tensile strength of composites reinforced with alkali treated fibres improves over that of composites reinforced with untreated fibres. It is clear that the alkalisation treatment of kenaf fibres results in a significant increase in strength over those of composites with untreated one. It is found that by adding treated kenaf fibre into PLA matrix, the tensile strength of biocomposites increased to 40 MPa for 40 wt.% kenaf fibre loading as compared with 36.83 MPa for the untreated fibres. This behaviour can be explained by considering the SEM micrographs presented in Figure 4. The image shows good fibre-matrix interfacial bonding by increasing the interfacial area of contact between the matrix and fibre. The PLA matrix can be seen adhering to the kenaf fibres subsequent to matrix failure. This evident indicate that alkali treatment produces strong bonds between the PLA matrix and kenaf fibre, with a free deposited surface fibres. This increase can be attributed to the removal of lignin and hemicellulose, which facilitates the rearrangement of fibrils along the direction of tensile deformation hence resulting in higher tensile strength [47-48].

EJO was used in this work in order to plasticise PLA and PLA/KF thus modifying the brittleness of neat PLA. The use of plasticiser had previously been reported for plasticising PLA which brings good result in its properties [49]. As the plasticiser introduced to the system, PLA became softer, more flexible, resulting in reduced strength and modulus. Thus, the value of tensile strength for PLA/KFT/EJO found to be lower than the value of PLA/KFT. However, the results for tensile strength did not differ significantly between PLA (36.83 MPa) and PLA/KFT/EJO (36.18 MPa). Introduction of EJO as a plasticiser into a PLA/KFT involves the addition of more free volume and so, increasing the flexibility of polymer chains, which in turns lowers the tensile strength and tensile modulus of the polymer [50].

On the other hand, as can be seen in Figure 6, addition of kenaf fibre to PLA regardless whether the fibre is treated or untreated resulted in a higher increase of composite modulus. The tensile modulus of biocomposite increased from 846.11 MPa for neat PLA to 958.21 MPa for PLA/KF/UT. The increment in tensile modulus with the addition of natural fibres was expected since stiffer behaviour of natural fibres is introduced into the polymer matrix [51-52]. A higher tensile modulus reflecting a rigid material, thus resists deformation for fibre composites.

As shown clearly, there is a significant increased in value of tensile modulus for PLA/KFT composite. Based on the result, PLA/KFT has higher tensile modulus compared to the untreated fibre composites due to the uniform
fibre dispersion in PLA. Additionally, the improvement of modulus may indicate an improvement in effectiveness of oriented cellulose fibre due to the removal of the hemicellulose and lignin content which leads to better packing of cellulose chains. As a result, the fibres become relatively ductile after the removal of some hemicellulose and lignin content resulted in the improvement of stiffness of the fibre. According to [53], the alcalisation treatment have a lasting effect on the mechanical behaviour of natural fibres, especially on fibre stiffness. NaOH solution has ability to remove natural fats, waxes and impurities from the fibre surfaces [54]. Therefore, the alcalisation treatment resulting in opening of more hydroxyl groups and other reactive functional groups on the surface of the fibre [55]. The increase in tensile modulus for PLA/KFT compared to untreated fibre composites was likely attributed to the enhanced surface wettability between fibre and matrix causing from removal of impurities and waxy substances from the fibre surface and the creation of a rougher surface after the modification.

4. Conclusions

PLA matrix, kenaf natural fibres and non edible types of plasticiser namely epoxidized jatropha oil (EJO) were utilised to prepare biodegradable biocomposites and their physical, mechanical and morphological properties were studied and compared. From FTIR spectroscopy, it was found that the disappearance of C=O stretching of the acetyl group in hemicellulose and the decreasing in intensity of the peak after alkaline treatment process confirming the removal of hemicellulose and lignin for treated fibres. The shifted peak towards lower temperature in carbonyl stretching region of PLA/KFT/EJO indicated the miscibility and interaction between PLA, Kenaf and EJO had took place. By SEM observation, it is clear that impurities on untreated kenaf fibres had weakened the bonding strength between PLA and kenaf fibres by evidence of larger voids and notable gaps between fibre and matrix. The alkali treated fibres show a rougher surface topography resulted in better compatibility and good adhesion in both PLA/KFT and PLA/KFT/EJO biocomposites as compared with untreated fibre composites. For kenaf fibre diameter, it was observed that the surface treated fibre composites had lower diameter than the untreated fibre composites which concluded the fibre degradation and delignification had happened during alcalisation treatment. In terms of mechanical properties, kenaf core particles act as rigid filler and by introducing it into PLA matrix, the tensile modulus of PLA/Kenaf composites for both PLA/KFUT and PLA/KFT were significantly higher than those of the PLA matrix itself. The improvement in tensile modulus was much notable for treated kenaf fibre composites due to the improved interfacial interaction resulting in higher tensile stiffness. The promising properties of possible combination of PLA/KFT/EJO biocomposites intern of morphological and mechnical properties suggested that they could be used as a substitite and alternative to synthetic fibre composites. Further works are recommended in order to improvise PLA/KFT/EJO biocomposites to be further developed as an ecologically friendly alternative to the petroleum-based-polymer that could be potentially functions in various research applications.

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