High-density polyethylene/date palm tree fiber composites: Effect of chemical treatment and fiber type

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ABSTRACT

Date palm tree fiber (DPTF) at a loading of 20 wt% was incorporated as a natural filler in high-density polyethylene (HDPE) to fabricate various HDPE/DPTF composites. The effect of fiber type and its chemical treatment on the properties of the resultant composites was investigated. Two different parts of the date palm tree, namely: meshes and leaflets were used to obtain the DPTFs, which were chemically treated by polyethylene glycol (PEG). Mechanical, water resistance morphological properties of the composites containing the treated and untreated DPTFs were studied. Generally, composites made with treated DPTFs had better mechanical properties, namely higher impact strength and Shore D hardness than that of neat HDPE and composites made with untreated DPTFs. The highest impact strength and hardness was measured for composites containing treated DPTFs obtained from the meshes. In addition, composites made with treated DPTFs had better water resistance properties (more hydrophobic nature and less water absorption) than that of composites with untreated DPTFs. Contrary, as shown by water vapour transmission rate measurements, the diffusion of water in its vapour state increased when treated DPTFs (obtained from both meshes and leaflets) was used. Microscopic observations indicated the formation of a homogenous fiber particle distribution in composites made with treated DPTFs in comparison to those made with untreated DPTFs. This indicates the presence of a good interfacial adhesion (thus compatibility) between the treated DPTFs and the HDPE matrix, which resulted in the formation of composites with improved mechanical as well as water resistance properties.

Keywords: Chemical treatment; date palm; high-density polyethylene; mechanical properties; water resistance

1 Introduction

In recent years, natural fibers (NFs) have received a great interest by many scientists and polymer engineers to be used as reinforcing materials in polymer composites instead of conventional fillers [1, 2]. This is due to many advantages that are associated with NFs, which include ease of decomposability, environmentally friendly, low cost, low weight and high mechanical properties. Nowadays, NFs-reinforced polymer composites are used in various industrial products and outdoor

applications. For example, NFs-reinforced polymer composites are used in transportation (automobiles, railway coaches, aerospace), military applications, building and construction industries (ceiling panels, partition boards), packaging, and many other consumer products [3-5]. NFs can be classified into three main categories, which are plant fibers, animal fibers, and mineral fibers. However, the most common NFs used to reinforce polymers are plant fibers. These include leaf-type fibers (pineapple, sisal, and abaca), core-type fibers (hemp, jute, and kenaf), grass/reed-type fibers (wheat, corn, and rice), seed-type fibers (cotton, kapok, and coir), bast-type fibers (flax, jute, hemp and ramie), and other types (wood and roots) [5, 6]. Generally speaking, NFs derived from plants are similar and mainly consist cellulose, hemicellulose, lignin, and other substances. It has been reported that a large variety of NFs, which include wood, cotton, bagasse, rice straw, rice husk, wheat straw, flax, hemp, pineapple leaf, coir, oil palm, date palm, doum fruit, ramie, kenaf, bamboo, sisal and jute are used to reinforce polymers [7, 8]. Palm trees are widely grown in Libya and nearby countries (in North Africa and the Middle East), which can be considered as a good source of NFs. They would be a good reinforcing filler for polymers, which can be of a great interest in the area of polymer composites [9, 10]. In fact, palm tree fibers (PTFs) have been successfully utilized as fillers for the reinforcement of thermoplastics and thermoset composites in recent years [11]. PTFs could be extracted from different parts of the palm trees, namely: the midribs, spadix stems, leaflets, and meshes. These PTFs are hydrophilic lightweight fibers that are easy-to-obtain at low-cost and possess good durability that withstand well against deterioration [12]. In addition, it was found that composites containing fibers that obtained from palm trees have moderate tensile and flexural properties compared with composites containing other NFs such as grass reeds, kenaf, ramie, sisal, coir, banana fiber [9]. This might be attributed to the fact that PTFs have the highest cellulosic content (nearly 50%) as compared to other NFs [13]. This might be also due to the low bulk density of PTFs compared to other NFs [14]. Generally, polymer composites that are reinforced with PTFs have been successfully used for many applications including parts and components, which are used in the automotive industry [15, 16]. The main aim of this work is to fabricate and study the effect of different types of date palm tree fibers (DPTFs) and their chemical treatment on the properties of highdensity polyethylene (HDPE) composites. These include: mechanical (impact strength and Shore D hardness), water resistance(water absorption (WA), hydrophobicity, water vapor transmission rate (WVTR)) and morphological properties of the resultant composites. In general, polyethylene is the most commonly used polymer matrix in polymer composites, which are reinforced with NFs because of its relatively low processing temperature and good processability [17]. Therefore, HDPE has been chosen as the matrix material in this study.

2 Experimental work

2.1 Materials

Date palm tree meshes and leaflets were obtained from Al-Grabolli city, Libya. HDPE obtained from Saudi Basic Industries Corporation (SABIC) (HDPE F00952, Melt flow index (MFI) = 0.05 g/10 min (ISO 1133, 190 °C, 2.16 Kg), density = 952 g/cm^3 (ISO 1183)). Polyethylene glycol (PEG) with weight average molecular weight of 400 g/mole (PEG400) in liquid form was purchased from Alfa Aesar, UK.

2.2 Preparation and treatment of DPTFs

Date palm tree meshes and leaflets were first washed with tap water to remove any contaminants, adhering dirt and dust. They were air-dried at room temperature for 48 h and then grinded and sieved to obtain DPTFs with sizes ranging from 38 to 150µm. PEG was used as a treatment agent for the DPTFs obtained from both meshes and leaflets. The dry DPTFs were stirred in PEG solution (30 vol%) at 100 °C for 2h in a reflux system. They were then removed from the solution, washed with deionized water to remove the excess PEG, and then dried in an oven at 80 °C for 24 h.

2.3 Preparation of composites

The treated and untreated DPTFs that obtained from both meshes and leaflets were dried in an oven at 80 °C for about 4 h, after which they were mixed with HDPE using twin screw extruder (Brabender, Germany) (L/D ratio of 48) with screw speed of 70 r.p.m. at 180°C. The obtained composites were subsequently cooled in air for 24 h and then cut to small pieces by scissors. Details of the composites and their codes are reported in Table 1.

Table 1: *Composition of composites and their codes*

Composite code	HDPE (wt%)	DPTFs from untreated meshes	DPTFs from treated meshes	DPTFs from untreated	DPTFs from treated leaflets
Code	(W170)	(wt%)	(wt%)	leaflets (wt%)	(wt%)
HDPE	100	0.0	0.0	0.0	0.0
Comp.1	80	20	0.0	0.0	0.0
Comp.2	80	0.0	20	0.0	0.0
Comp.3	80	0.0	0.0	20	0.0
Comp.4	80	0.0	0.0	0.0	20

2.4 Characterization of DPTFs

2.4.1 Fourier transform infrared spectroscopy (FTIR)

FTIR was employed to monitor the changes in chemical structure of DPTFs obtained from both meshes and leaflets caused by PEG treatment. FTIR analysis was carried out using Tensor II machine (Bruker, Germany) with a wavenumber resolution of 4 cm⁻¹. The samples were mixed with KBr powder, pressed into pellets and scanned in a range of 500 to 4000 cm⁻¹ with an average of 32 scans.

2.4.2 Characterization of composites

2.4.2.1 Impact strength and hardness

Specimens for impact strength test were prepared using injection molding machine (Xplore 12ml, Netherlands). Charpy impact test was carried out using (CEAST Resil-Impactor tester) at room temperature with impact energy of 15 J. The specimens for impact test were prepared and notched according to ASTM (D256-10). A minimum of five specimens were tested and an average value was taken. Hardness was investigated by a Durometer in Shore D scale at room temperature according to ASTM (D2240) (a minimum of 10 measurements were taken for each composite and an average value was recorded).

2.4.2.2 Water absorption (WA)

Water uptake measurements were used to determine the WA behavior of the composites. Composites were pressed at 5 MPa using a small press at 180 °C to obtain thin films (~ 1mm). Films with an area of 2 cm² were cut and used in this test. Before testing, all films have been dried at 80 °C in an air-circulated oven overnight, then placed in a desiccator. Subsequently, films were weighed with a OHAUS analytical digital balance (with a resolution of 0.1 mg). The films were immersed in water and weighed over time for three weeks at room temperature. The excess water on the surface of each film was removed with blotting paper before weighing. This test has been conducted in accordance with the procedure recommended by ASTM D570 [18]. Five specimens from each sample were weighed before and after immersion (over time) and the water uptake (WU) was calculated as follows:

$$WU(\%) = \frac{m_1 - m_0}{m_0} x \, 100 \tag{1}$$

Where, m_1 is the mass of the sample after immersion (g) and m_0 is the mass of the sample before immersion (g).

2.4.2.3 Contact angle measurements

The contact angle measurements were carried determine the out to hydrphilicity/hydrophobicity of the neat HDPE and the obtained composites' surface. The measurements were carried out using Ramè-Hart instrument (model 200-F4) at room temperature. Drops of water (3 µL) were deposited on the surface of the HDPE and all the composites with a micro-syringe. Images of the water drops were acquired through a digital camera positioned on a static contact angle analyzer. The contact angle (θ) was measured automatically from the image setup. The contact angle measurements were repeated five times for each sample and the average value was recorded.

2.4.2.4 Water vapor transmission rate (WVTR) test

The WVTR test was used to determine the amount of water vapor that passes through the composite under 70% relative humidity (RH) at room temperature for 24 h. This specific RH was achieved using saturated NaCl solution following the work of Timusk [19] and Kuishan [20]. Zeolite (~2g) was placed in glass containers, which

were sealed with films made from HDPE and the HDPE/DPTF composites. Films were prepared by pressing samples at 5 MPa using a small press machine at 180 °C, which gave thin films of ~1 mm thickness. Films with diameters of 3 cm were cut and used. The containers were then placed in a dissector filled with the saturated salt solution to maintain a constant RH at room temperature for 24 hours. All WVTR measurements were repeated three times and an average value was recorded. The WVTR was calculated as follows [20]:

$$WVTR = \frac{w_1 - w_0}{A} \tag{2}$$

Where, w_0 is the weight of sample before exposure to water (g), w_1 is weight of sample after exposure to water (g) and A is the exposed area of the film in m^2 .

2.4.2.5 Morphological properties

Microscopic observations of HDPE and its composites were carried out by an optical microscope (XP-501 transmission polarizing microscope, Turkey), equipped with a color digital camera (Moticam 2) and Motic Images Plus 2 software at different magnifications.

3 Results and discussion

3.1 Characterization of DPTFs

3.1.2 FTIR analysis

FTIR was used to identify the functional groups present in the DPTFs obtained from meshes and leaflets and to monitor the changes in their chemical structures caused by PEG treatment. It has been reported that these plant fibers contain a variety of functional groups such as alkenes, phenolic hydroxyl group, aromatic groups, b-glucose linkages and other oxygen-containing groups (ester, ketone and alcohol) [21]. In general, the characteristic bands of these functional groups correspond to the absorption bands of lignin, hemicellulose and cellulose [22]. Figure 1 presents the FTIR spectra of DPTFs obtained from the meshes before and after PEG treatment. Figure 2 shows the FTIR spectra of DPTFs obtained from the leaflets before and after PEG treatment.

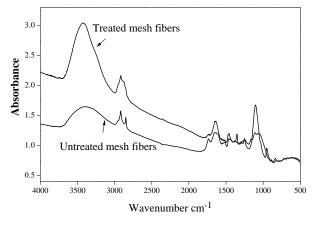


Figure 1: FTIR spectra of DPTFs obtained from treated and untreated meshes.

The broad band at 3750-3200 cm⁻¹ shown in Figure 1 is typical of hydroxyl (-OH) groups (stretching and flexing vibration frequencies of the intra- and intermolecular hydrogen bonds of cellulose) [23-25]. From Figure 1, one can see that the broad absorption band for DPTFs obtained from meshes at 3750-3200 cm⁻¹ of -OH groups was intensified (became narrower and more defined) after PEG treatment, suggesting the occurrence of hydrogen bonding between PEG and the functional groups in the fibers. This could lead to reduce the number of -OH groups from the fiber surface, which results in meshes with a hydrophobic nature [21]. The absorption band at 2990-2905 cm⁻¹ is due to the stretching vibrations of CH₂. The small absorption band at 2890-2805 cm-1 are due to the C-H symmetric stretching of the methylene (CH₂ and CH₃) groups [26]. The latter two bands are generally overlapped after PEG treatment. This means that the proportions of CH₂ and CH₃ were higher in the treated DPTFs than in the untreated one. This is rather expected because PEG contains a greater proportion of these groups, which can be recorded in the FTIR spectra [27]. A small band was observed at 1780-1714 cm⁻¹ due to the carbonyl (C=O) stretching from the ester linkage of hemicellulose and lignin. An increase in the intensity of this band was observed after the treatment with PEG. This could indicate a slight change in hemicellulose and lignin has occurred due to the treatment with PEG. Furthermore, the peak representing the ether groups in treated mesh at 1253 cm⁻¹ had changed noticeably; it became wider compared to the same peak in the untreated mesh fiber water spectrum. This may indicate the removal of some materials during PEG treatment [28]. Overall, it appears that treatment of the DPTFs obtained from meshes with PEG resulted in a significant interaction between the PEG and the mesh fibers via hydrogen bonding.

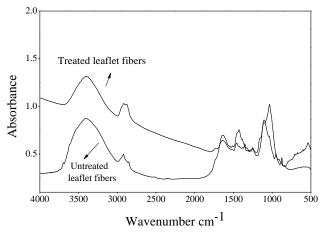


Figure 2: FTIR spectra of DPTFs obtained from treated and untreated leaflets.

From Figure 2, it can be seen that contrary to the DPTFs obtained from the meshes, the absorption band for -OH groups at 3750-3200 cm⁻¹ in the DPTFs obtained from leaflets were similar before and after PEG treatment (no significant change in beak broadening was noticed). Furthermore, treatment of leaflets with PEG did not cause any other significant changes in the FTIR spectra of the DPTFs obtained from the

leaflets. From Figure 1 and 2, one can clearly see that the influence of PEG treatment was much more distinct in the case of DPTFs obtained from the meshes than those obtained from the leaflets.

3.2 Characterization of composites

3.2.1 Impact strength and hardness

It has been shown that NFs can improve the fracture toughness, crack resistance, tension performance, flexural properties, impact strength, and fatigue behavior of composites [29]. The impact strength and Shore D hardness of neat HDPE and composites made with DPTFs obtained from meshes and leaflets are shown in Table 2. Composites with untreated DPTFs (obtained from both meshes and leaflets) had higher impact and lower Shore D hardness than that of pure HDPE. However, composites with treated DPTFs (obtained from both meshes and leaflets) appeared to have higher impact strength and Shore D hardness than that of pure HDPE and composites with untreated ones. This probably indicates that PEG treatment improved the interfacial adhesion between the fibers and the HDPE matrix (better compatibility). This resulted in better fiber particle distribution, which led to the formation of composites with improved mechanical properties [10]. Moreover, as seen in Table 2 the highest impact strength and hardness was obtained by composite made with treated DPTFs obtained from the meshes. This is in agreement with the FTIR results, which indicated better interfacial adhesion between DPTFs obtained from PEG-treated meshes and HDPE. This resulted in even better fiber-matrix adhesion and therefore much better fiber distribution occurred in comparison to the composites made with the DPTFs obtained from the leaflets. Consequently, higher impact strength and hardness were observed for composites made with treated DPTFs obtained from the meshes.

Table 2: The impact strength and Shore D hardness of HDPE and HDPE/DPTF composites

Composite code	Impact strength (KJ/m ²)	Shore D hardness
HDPE	8.19 (0.10)*	57.5 (0.32)*
Comp.1	10.04 (0.76)*	57.3 (1.24)*
Comp.2	11.06 (0.52)*	63.8 (1.42)*
Comp.3	9.53 (0.42)*	57.4 (0.92)*
Comp.4	10.31 (0.36)*	59.3 (1.42)*

^{*} Standard deviation between brackets

3.2.2 Water uptake test

Composites based on plant fibers are sensitive to water. Therefore, it is of great significance to study the water absorption (WA) characteristics of these composites. This is because WA could affect the properties of these composites, resulting in changes of bulk properties such as dimensional stability, as well as mechanical and physical properties [30]. Figure 3 shows the WA behavior for the HDPE/DPTF composites.

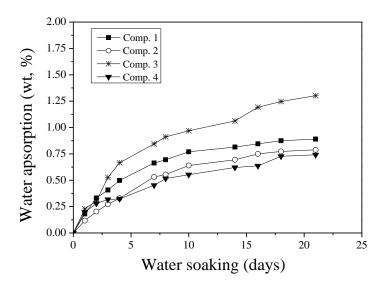


Figure 3: WA behavior for the prepared composites.

From Figure 3, one can see that in general water uptake of the composites increased with soaking time and thereafter it became relatively constant after 16 days except for the composite made with untreated DPTFs obtained from leaflets. It has been shown that cellulose and hemicellulose components in plant fibers are mostly responsible for the WA of NFs, since they contain many easily accessible -OH groups which offer a level of hydrophilic character to these fibres [31, 32]. As illustrated in Figure 3, the incorporation of treated and untreated DPTFs obtained from meshes and leaflets had no significant effect on the water uptake of HDPE. All HDPE/DPTF composites generally showed a relatively very low water uptake, ranging from 0.75-1.25 wt%. From Figure 3, one can also see that treatment of DPTFs with PEG slightly reduced the WA of the composites containing them in comparison to composites with untreated fibers. According to Shu-pin et. al. [33], the improvement of water-related properties can enhance the interfacial bonding between wood fibers and polypropylene matrix by heat treatment with PEG. Also, heat treatment with PEG could result in improvement in the dimensional stability of these composites. Russly and Lugman [34] claim that treatment with PEG could result in a decrease in void volume, which enhance the adhesion between fibers and polymer, which restrict water penetration and storage at the interface. However, it is known that WA of such composites can be affected by the existence of lumens, holes, voids, flaws, poor interfacial adhesion, and microcracks at the interface between the filler and the polymer matrix [35].

3.2.3 Contact angle measurements

The value of the contact angle can vary from 0-180°, 0° representing fully wetted surfaces (very hydrophilic) and 180° representing totally non-wettable surfaces (very hydrophobic). In other words, wettability and hydrophilicity/hydrophobicity are closely related phenomena. It should be mentioned here that hydrophobic surfaces are characterized by a contact angle of 90° or more and hydrophilic surfaces are

characterized by a contact angle of less than 90°. Table 4 shows the effect of PEG treatment on the contact angle of HDPE/DPTF composites made with DPTFs obtained from both mesh and leaflet. Contact angle of neat HDPE is also shown for compassion.

Table 4: Contact angle of HDPE and HDPE/DPTF composites

Composite code	Contact angle (θ°)	
HDPE	81.10 (3.1)*	
Comp.1	77.82 (1.0)*	
Comp.2	78.78 (0.9)*	
Comp.3	74.67 (2.8)*	
Comp.4	78.46 (2.6)*	

^{*} Standard deviations between brackets

As shown in Table 4, the contact angle of neat HDPE and composites were <90°, which indicates that they all had surfaces with a hydrophilic nature. However, the contact angle of composites was lower than that of neat HDPE. This indicates that the presence of treated and untreated fibers resulted in composites with more hydrophilic nature. This is properly due to the presence of excess of polar groups on the surface of the composites, which caused by the addition of mesh and leaflet fibers (treated and untreated). This is because these fibers, which are derived from plants mainly consist of cellulose, hemicellulose, lignin, and extractives, as mentioned previously. It should be mentioned here that the decrease in contact angle can be probably due to increased roughness of the film surface caused by the fibers [36, 37]. Also, contact angle measurements may depend on other factors, such as surface energy, surface chemical structure, viscosity of the liquid and surface cleanliness [38-39]. From Table 4, one can also see that in both cases (composites with mesh and leaflets), lower contact angle was observed when untreated fiber was used. This indicates that using treated fibers resulted in composite surfaces with less hydrophilic nature than that of composites made with untreated fibers. This is probably due formation of hydrogen bonding between -OH groups of PEG and -OH groups of cellulosic materials of mesh and leaflet fibers. Therefore, less polar groups are available in the composite surface which led to the formation of surfaces with less hydrophilic character. This in in good agreement with the WA measurements, which showed that composites made with treated DPTFs had lower WU values (see Figure 3).

3.2.4 Water vapor transmission rate (WVTR)

WVTR is the standard measurement by which polymer films are compared for their ability to resist water vapor transmission through them. Lower WVTR values indicate better water resistance of a polymer material. Table 3 shows the WVTR for the HDPE and its composite films which contain treated and untreated DPTFs obtained from meshes and leaflets.

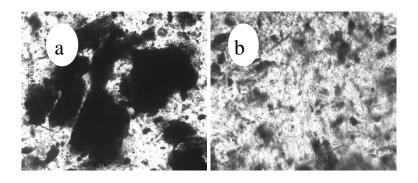
Composite code	WVTR $(g/m^2/24h)$	
HDPE	0.007 (0.001)*	
Comp.1	0.019 (0.006)*	
Comp.2	0.032 (0.299)*	
Comp.3	0.020 (0.003)*	
Comp.4	0.031 (0.001)*	

Table 3: WVTR of neat HDPE and HDPE/DPTF composites

Table 3 illustrates that neat HDPE films displayed very low WVTR, which is 0.007 g/m²/24h. However, all composites exhibited higher WVTR than that of neat HDPE. The highest increase (from 0.007 to 0.031-0.032 g/m²/24h) was obtained by the composites, which contain treated DPTFs obtained from meshes and leaflets. Whereas a lower increase (from 0.007 to 0.019-0.020 g/m²/24h) was obtained by the composites containing untreated DPTFs obtained from meshes and leaflets. This is contrary to the results obtained from the WA test, which indicated that composites made with treated DPTFs had less WA. This can be explained by the fact that the WVTR test depends on diffusion of water vapour through the composite film in addition to the solubility.

3.2.5 Morphological properties

The optical microscopy images in Figure 4 clearly show the agglomeration of fiber particles in the composites made with untreated meshes and leaflets (Figure 4a and 4c) in comparison to composites made with treated meshes and leaflets (Figure 4b and 4d). This heterogeneous particle distribution led to lower interfacial adhesion between the untreated fibersand HDPE matrix. Contrary, composites made with treated fibers exhibited better compatibility between the fiber and HDPE, which was apparent from the absence of fiber aggregation (relatively better fiber distribution). Moreover, no sign of voids and filler pull-outs (retreats) from the HDPE matrices were observed in all composites made with treated fibers.



^{*} Standard deviation between brackets

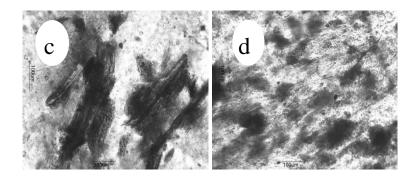


Figure 4: Optical microscopy images of composites made with: a) untreated meshes, b) treated meshes, c) untreated leaflets and d) treated leaflets.

4 Conclusion

The effect of adding different types of date palm tree fibers (DPTFs) and their chemical treatment on the properties of HDPE was investigated. DPTFs were obtained from two different parts in the date palm tree (i.e., meshes and leaflets), which were treated by polyethylene glycol (PEG). Mechanical, water resistance and morphological properties of the obtained composites were investigated. Overall, treating the DPTFs with PEG appeared to have a considerable effect in reinforcing HDPE. Results revealed that the treatment of DPTFs improved the impact strength and Shore D hardness compared to neat HDPE, significantly. Moreover, composites made with DPTFs obtained from treated meshes had better mechanical properties than that made with treated leaflets. In addition, ccomposites made with treated DPTFs (obtained from both meshes and leaflets) had better water resistance properties (less hydrophilic nature and less water absorption) than that of composites made with untreated DPTFs. Contrary, the water vapour transmission rate measurements indicated that the diffusion of water in its vapour state increased when treated DPTF (both mesh and leaflets) was used. Optical microscopy images clearly showed the formation of significantly less fiber aggregation in composites made with treated DPTFs (both meshes and leaflets). Moreover, no sign of voids and filler pull-outs (retreats) from the HDPE matrix were observed in composites made with treated DPTFs. This resulted in better fiber particle distribution, thus composites with improved mechanical as well as water resistance properties were formed.

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