Eco-friendly date-seed nanofillers for polyethylene terephthalate composite reinforcement

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Abstract

Polymer composites have been widely exploited in numerous industries. Micro-particle fillers are typically added as reinforcement materials to improve the characteristics of these composites. In this work, organic nanoparticles of date seeds were added as a filler for polyethylene terephthalate (PET) to produce enhanced polymer nanocomposites. A date-seed nanofiller (DSN) was prepared and examined with x-ray diffraction measurements, and then added to PET by hot compression. The characteristics of the PET-DSN composite were experimentally investigated through tests of mass loss, compressive strength, and Vickers micro-hardness. The PET-DSN microstructure was inspected using a scanning electron microscope and Fourier-transform infrared spectroscopy. The results show chemical stability of the PET-DSN composite. Moreover, key mechanical properties of the composite, namely hardness, compressive strength and wear resistance, were improved and optimized with a DSN reinforcement of 0.75 wt%.

1. Introduction

Nanocomposites represent a new generation of multiphase solid materials in which one of the phases has at least one nanometer-scale dimension [1]. A nanocomposite has a matrix material that can be of the ceramic, metallic, or polymer types [2]. Recently, polymer materials have replaced conventional materials in many applications due to their low density, ease of processing, light weight, low price, corrosion resistance, and thermal insulation [3]. Currently, polymeric nanocomposite materials have numerous applications. For example, such materials have been used in organic coatings [4], self-healing materials [5], self-repairing aeronautic panels [6], automotive applications [7], food packaging [8], and gas barrier applications [9].

Several types of polyethylene are typically used as polymer matrix materials in composites [10]. Polyethylene terephthalate (PET) is one of the commonly used types which is distinguished by good properties of strength, durability and cheapness as well as availability in the surrounding environment [11, 12]. In particular, PET has excellent tensile and impact strength, chemical resistance, clarity, processability, colourability, and reasonable thermal stability [13, 14]. Due to these properties, PET has been used in numerous applications including electronic instruments, automobile products, housewares, lighting products, power tools, material handling equipment, and sporting goods [15]. Moreover, PET is used as the substrate of carbon nanotube (CNT) film heater to fabricate thermochromic displays [16].

Different types of fillers can be dispersed within matrix materials to improve the composite functional, mechanical, and physical properties. In general, fillers could be inorganic or organic. On the one hand, the most common inorganic fillers are carbon nanofibers (CNFs), carbon nanotubes (CNTs), and exfoliated graphite (EG) [17, 18]. On the other hand, several organic fillers are extracted from waste materials such as the tamarind kernel powder [19], rice husk [20], coffee husk [21], Delonix regia (Dr) [22], wood flour [23] and date-seed (DS) particles [24]. These organic fillers are frequently used to enhance the mechanical properties of composites [25, 26].
In fact, date-palm (DP) particle reinforcement has been efficiently used as a filler material in both thermosetting and thermoplastic products [27]. For composites with a thermosetting matrix, date-palm stem fibers (DPF) have been used as fillers in epoxy composites [28]. Also, Saba et al studied DPF/epoxy composites at different loadings, and showed that 50% DPF loading enhances tensile and impact strengths as well as the morphological properties of epoxy [29]. For composites with a thermoplastic matrix, date palms have been exploited as a filler for a polyethylene matrix material. For example, an investigation has been conducted on the natural weathering and thermal stability of composites of polypropylene (PP) and date palm fibers or particles (DPF/DPP) [30]. The results revealed that adding DPF to the PP materials led to improved interfacial adhesion as well as enhanced mechanical and thermal properties.

Moreover, date-seed (DS) particles have been used as fillers. Fikry et al [31] analyzed several physical characteristics of defatted roasted date seed powder which can be used in developing bioactive brew. Ruggiero et al [32] demonstrated enhanced adhesion, cohesion and wear properties of epoxy resin reinforced with waste date seed particles. In particular, Elkhouly et al [33] compared the mechanical characteristics of glass-epoxy composites reinforced with DS fillers, and two inorganic fillers, namely silicon carbide (SiC), and aluminum oxide (Al₂O₃). The results revealed that the DS filler is competitive with inorganic fillers and is still more economical to use. Also, the wear loss performance of DS-reinforced glass-epoxy composites was optimized using the Taguchi method and the flower pollination algorithm. In addition, other recent methods explored the mechanical properties of glass-epoxy materials reinforced with inorganic fillers [34, 35].

Recently, fillers have been produced at the nanoscale to further improve composite characteristics. Nanoscale fillers can be of different shapes and sizes, which can be categorized into three types, namely two-dimensional (2D) layered, one-dimensional (1D) fibrous or zero-dimensional (0D) spherical nanofillers [36]. Nanofillers exhibit ultra-large interfacial areas when bonded to polymer matrices, and are used particularly to improve polymer properties through serving as reinforcing materials and acting as active ingredients [3, 4, 37]. Specifically, the reinforcement of a polymeric matrix with a nanofiller would render the composite with several mechanical, electrical, thermal, and magnetic characteristics [38]. Interestingly, such enhanced composites can be used as sensors for pressure and other environmental changes [39].

More recently, nanoscale date-palm fillers have been used in packaging and dental applications [40]. Adel et al [41] extracted oxidized nanocellulose from date palm sheath fibers, and used it as a packaging additive in chitosan films. Salih et al [42] created maxillary denture bases from composites of Poly(methyl methacrylate) (PMMA) reinforced with nanoscale pressed date (Ajwa) powder.

Evidently, most of the published work investigates DS micro-particles as a filler for polyethylene matrix materials to produce polymer composites. In contrast, the use of date-seed nanofillers (DSN) in nanocomposites has been generally overlooked. These nanofillers are eco-friendly, cost-effective, and have the potential for producing nanocomposites with improved properties. Moreover, as mentioned above, PET has excellent properties that justify its use as a matrix material in polymeric nanocomposites.

In this work, we propose the use of organic date-seed nanofiller (DSN) as a reinforcement material for polyethylene terephthalate (PET) composites. Specifically, a DSN powder has been prepared and added to PET polymers by hot compression. The DSN mechanical characteristics are investigated using tests of mass loss, compressive strength, and Vickers micro-hardness. The surface of the DSN-reinforced composite was inspected by a scanning electron microscope (SEM). Fourier transform infrared (FT-IR) spectral analysis of the SEM images was performed. The results reflect favorable properties of the produced composite.

2. Experimental work

2.1. Materials

In this work, the polyethylene terephthalate (PET) is used as the composite matrix. The PET material was supplied by Sigma-Aldrich, St. Louis, U.S.A. The PET material has a molecular weight of 192 g mol⁻¹, and a density of 1.3–1.4 g cm⁻³. The mechanical and chemical properties of the PET material are illustrated in Table 1 [43].

Date seeds were collected from date palm trees in Beni-Suef, Egypt. The seeds were packed and processed at the Material Research Laboratory, Beni-Suef University, Egypt. Date seeds were added as a reinforcement to the

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**Table 1. Mechanical and chemical characteristics of the PET material [43].**

| Property                  | Breaking strength (MPa) | Melting point (°C) | Weight average MW (g/mol) | Tensile yield strain (%) | Young’s modulus (MPa) | Water absorption (%) |
|---------------------------|-------------------------|-------------------|---------------------------|-------------------------|-----------------------|---------------------|
| Value                     | 50                      | 220               | 30,000–80,000             | 4                       | 1700                  | 0.5                 |
PET material to enhance the mechanical properties of the composite. Typical chemical composition of date seeds includes carbon (C), oxygen (O), aluminum (AL), potassium (K), iron (Fe) and copper (Cu).

2.2. Nanofiller preparation
In this work, the DSN material was prepared in the aforementioned Material Research Lab. First of all, the seeds were cleaned with fresh water, and then dried in air for 24 h. For eliminating moisture, the dried seeds were heated in an oven at 70 °C for 24 h. After that, the seeds were crushed and milled for 140 h using a ball mill until the target nanoscale particle size was achieved and verified by x-ray diffraction.

2.3. Nanofiller structural characterization
The x-ray diffraction technique (with the Bruker XRDML format) was used to identify the crystal structure of the produced DSN powder using Cu-Kα radiation (α = 1.54060 Å) with a scanning angle of 2° (corresponding to a current of 25 mA) at a scanning velocity of 4 m min⁻¹, and a voltage of 40 kV. Measuring the average size of a crystallite (or grain) was based on the Scherrer equation [44]. The produced average particle diameter was ~22 nm.

2.4. Nanocomposite fabrication
The DSN-reinforced PET composites were produced using a warm compression method at a pressure of 0.85 MPa, with DSN weight fractions of 0, 0.25, 0.5, 0.75 and 1 wt%. The matrix and filler materials were mixed and heated in a cylindrical pressing die for 10 min at a temperature of 145 °C. This pressing die was made of M238 hot-working steel with an inner diameter of 6.5 mm. After reaching a temperature of 185°C–195°C, hot pressing of the composites was carried out by a hydraulic pressing system. Specimens were then gradually cooled to room temperature. Each of these cylindrical specimens had a 6.5-mm diameter and an 11-mm length. Mechanical tests were performed on the PET-DSN composite specimens.

3. Experimental tests
Several experimental tests were carried on the PET-DSN composite samples including mechanical tests, worn-surface testing, and Fourier-transform infrared spectroscopy.

3.1. Mechanical characterization
Three mechanical characterization tests of the PET-DSN composites were conducted, namely the micro-hardness test, the wear test, and the ultimate compressive strength test.

3.1.1. Wear testing
Wear characteristics of composites are typically measured by the mass loss. For the PET-DSN composite, mass losses at DSN weight fractions of 0, 0.25, 0.5, 0.75 and 1 wt%, were found using a pin-on-disc setup. In this setup, specimens are loaded, using a cantilever system, against silicon-carbide (SiC) abrasives placed on a hardened steel disk. A pin is then installed in the wear mechanism in a steel holder to keep the pin strongly perpendicular to the flat surface of the rotating counter disk. Using this setup, wear testing was conducted on the cylindrical specimens under normal loads of 10, 20 and 30 N, respectively. The speed of rotation was set to 393 rpm while the sliding distance was set to 95 m. The wear medium was selected as 600-grit SiC abrasive paper. The abrasive wear of the pins was defined during sliding as the incurred weight loss. The wear pin was cleaned in acetone before and after each test, and then weighed on a sensitive microbalance with a 0.0001-g sensitivity.

3.1.2. Micro-hardness testing
In order to investigate the durability of the PET material with and without the DSN reinforcement, micro-hardness testing of the PET-DSN specimens was carried out using the Vickers hardness test. In this test, each specimen is indented with a diamond indenter, which has a right-pyramid shape with a square base and an angle of 136° between each two opposite faces. The indenter is subjected to a load of 5 to 500 kg, where the full load is normally applied for 15 s. After the removal of the load, the lengths of the two diagonals, marked by the indenter on the material surface, are measured using a microscope and the average diagonal is calculated. The Vickers hardness is the quotient obtained by dividing the load (in kg) by the area of indentation (in square mm). In this work, the average of at least five measurements for each sample was used to calculate the sample hardness.
3.1.3. Ultimate compressive strength testing

Based on the ASTM D695 standard, the ultimate compressive strengths of the PET-DSN composites were tested for different DSN reinforcements. The tests were performed at room temperature by a computerized universal testing machine (UH series, Shimadzu Corporation), with a cross-head speed of 6 mm min\(^{-1}\).

3.2. FTIR spectroscopy

The Fourier-transform infrared (FTIR) spectroscopy is an analytical technique used to analyze the composition of organic and inorganic materials. A Vertex 70 FTIR spectroscopy device (Bruker Corporation) was used to analyze the molecular composition of the PET material with and without DSN reinforcement within a wave number range of 4 000 cm\(^{-1}\) to 500 cm\(^{-1}\). The FTIR technique is based on the fact that distinct molecular structures generate distinct spectral patterns.

3.3. Worn surface testing

The PET-DSN composite morphology was explored by a field-emission scanning electron microscope (FE-SEM) fitted with an energy dispersive spectroscopy (EDS) device. In particular, the worn specimen surface was covered with titanium in a vacuum sputter chamber. Then, the JSM-6510-LA SEM (Jeol Ltd) was used to examine and record the fractured surface morphology for the worn specimens.

4. Results and discussion

4.1. DSN chemical and microstructural analysis

The chemical composition of the DSN powder was analyzed using energy-dispersive x-ray diffraction (EDX). The weight percentages for the compound elements are shown in table 2. These weights are generally consistent with reported chemical analysis results for date seeds [24].

The DSN microstructure was examined using a SEM device. Figure 1 shows one of the captured SEM images. While this image demonstrates large variability in the shape and scale of DSN particles, each particle appears to be almost spherical. Similar observations on nanoparticle shapes were reported in [36, 44]. These shape and scale variations give nanofillers the advantage of strengthening the mechanical properties of reinforced materials. In addition, finer particles are better for composite reinforcement because they act as suitable barriers for dislocation and can also be dispersed at inter-particle boundaries uniformly. The DSN average particle diameter was found to be \( \sim 22 \) nm, as estimated from the x-ray diffraction (XRD) patterns of the DSN powder.

Table 2. Chemical composition of the date-seed powder.

| Elements | C | O | Cl | Al | K | Fe | Cu |
|----------|---|---|----|----|---|----|----|
| Weight%  | 46| 45| 0.31| 0.42| 0.6| 0.9| 0.7|

Figure 1. A micrograph of the date-seed nanofiller powder using the FE-SEM technique.
4.2. Wear analysis of PET-DSN composites

The mass loss (ML) of PET-DSN composite specimens was evaluated at different DSN weight fractions (wt%) and normal loads. Figure 2 illustrates the effect of five DSN weight fractions and three applied normal loads on the mass loss. Several observations can be made as follows.

Firstly, we can realize that ML decreases with increasing the DSN weight percentage from 0.25 to 0.75 wt%. This is due to the DSN large surface area which leads to a large interfacial area between the PET and DSN materials. This enlarged interface leads to enhanced mechanical properties of the PET-DSN composites. In particular, the wear resistance is improved since the nanofiller particles transmit friction and hence boost the nanocomposite load carrying capacity [45–47].

Secondly, a PET-DSN composite with a high DSN concentration of 1 wt% causes a relative ML increase compared to a PET matrix reinforced with a DSN of 0.75 wt%. The increased loss associated with the 1 wt% DSN material is most likely due to uneven filler distributions, which might happen because of poor or improper mixing, particle agglomeration, or DSN powder heterogeneity [29, 47]. All these factors can lead to a notable decrease in the adhesion between nanoparticales and matrix materials, as well as poor compressive strength of the resulting nanocomposites. Similar failure patterns have been observed and reported [30, 47].

Thirdly, figure 2 shows that ML decreases as the load is raised from 10 to 30 N. This is because the increase in the normal load increases the contact stresses, deepens the SiC abrasive penetration of the composite surface, and hence aggravates the nanocomposite mass loss [35].

Figure 3 shows the relation between the wear rate (or percentage) relative to the filler-free matrix material and the DSN weight percentage at different normal loads. Compared to the DSN-free PET matrix, we see that the wear (mass loss) rate is reduced to 80%, 15%, 6%, and 11% when the DSN material is added by weight fractions of 0.25, 0.5, 0.75 and 1 wt%, respectively [44, 45].

In this work, the statistical significance of the change in the nanocomposite characteristics due to the DSN reinforcement is assessed using the F-test and ANOVA methods for a level of significance, \( \alpha = 0.05 \) with a 95% confidence level.
The resulting $p$-value shows that the DSN reinforcement has significantly changed the observed nanocomposite characteristics compared to the unreinforced PET composite. Indeed, the test results show significant variance due to the filler wt% factor ($p$-value = 0.00) but lesser variance due to the normal load factor ($p$-value = 0.093) [24, 34]. The statistical analysis was performed using MINITAB 18.

4.3. Micro-Hardness of PET-DSN composites

Micro-hardness of the composite specimens was assessed for different DSN weight fractions, as shown in figure 4. The hardness of the unreinforced PET material (1.2 HV) increased to 14 HV when the DSN weight percentage became 0.75 wt%. The hardness can be improved by up to 75.1%. This improvement in micro-hardness can be attributed to the strong interfacial adhesion between the DSN and PET materials, and the molecular-level dispersion of the DSN material in the matrix [44, 47].

4.4. Compressive strength analysis

For the test specimens, the compressive strength of the PET-DSN composite was evaluated for DSN fractions of 0 to 0.75 wt%. The compressive strength of the pure PET matrix was determined to be 74.6 MPa. This strength increased by 41.7% when the DSN weight fraction reached 0.75 wt% (See figure 5). This increase in strength is due to the DSN high specific surface area that can influence the extent of the interface between the two materials and the filler dispersion. In particular, the nanoparticle distribution in the PET matrix results in a transfer of stress from the PET matrix to the DSN particles through compression, which causes effective load transfer and increased strength. Moreover, the interfacial adhesion of the DSN material to PET helps the composite withstand the stress build-up. Specifically, nanofillers can fill the free space between polymer matrix chains, increase the intermolecular attraction force, and hence make the composite very dense and less permeable [40, 45, 47, 48].
4.5. FTIR analysis

The FTIR spectra for the PET matrix and the PET-DSN composite are shown in figure 6. It can be noted that alkenes (CH$_2$) are formed around a wave number of 2359 cm$^{-1}$ for all DSN weight fractions of figures 6(a)–(c).

Figure 6. The FTIR spectra of PET composites without and with DSN reinforcement.
As shown in figure 6(a), the spectrum of the DSN-free PET shows two unique peaks representing C=C–H and C-O-C stretching vibrations in ethers at wave numbers of 1552 cm$^{-1}$ and 1139 cm$^{-1}$, respectively. Figures 6(b), (c) show that the spectra of the DSN-reinforced PET are notably different from that of the DSN-free PET specimens, where the strong bands at 1700–1300 cm$^{-1}$ are assigned to the C=C weak stretching vibration and C–H stretching vibration of CH$_3$, respectively. It can be noted in figure 6(c) that the bands at wave numbers of 3470 and 2942 cm$^{-1}$ are attributed to CH$_2$ splitting. Moreover, the FTIR analysis demonstrates that the PET-DSN composite with a DSN weight of 0.75 wt% has a higher spectrum [24, 33].

4.6. Worn surface inspection results

A scanning electron microscope (SEM) was used to inspect the worn surfaces of unreinforced PET and PET reinforced with DSN of 0.75 wt%. As shown in figure 7(a), for the surface parallel to the sliding path, there is a continuous groove and a deeper groove. During the wear process, some finer PET grains were scratched off. The worn surfaces of the PET-DSN composite with DSN fractions of 0.5 and 0.75 wt% are shown in figures 7(b), (c), respectively. Shallower and narrower furrows were observed on the worn surfaces compared to the DSN-free PET surface. The role of the interfacial bonding between the PET and DSN materials is clear. As described above, the abrasive wear of the PET-DSN nanocomposites consists of two levels. In the first level, the SiC abrasive particles penetrate and cut into the soft polymer matrix, resulting in excessive removal of PET from the composites. Hence, the SiC abrasives also impact the DS nanoparticles, allowing the SiC abrasives to get blunt. In the second level, the DS nanoparticles are released from the polymer matrix and act as a barrier to the abrasive micro-cutting action, causing a decrease in SiC abrasive cutting ability and the resulting nanocomposite wear rate. Distinct parallel grooves and ridges running in the sliding direction are shown in the micrographs of figures 7(b), (c). [24, 33, 45].

5. Conclusions

In this work, we investigated the effect of date-seed nanofillers (DSN) as a reinforcement material on the characteristics of the polyethylene terephthalate (PET) matrix. Several key conclusions can be made. The DSN
reinforcement can generally produce polyethylene terephthalate (PET) composites with favorable mechanical properties including reduced wear mass loss, improved hardness, and higher compressive strength. Specifically, the PET-DSN composite with a DSN fraction of 0.75 wt% results in a 74.5% lower wear mass loss. Also, the hardness of a DSN-free PET matrix was improved by up to 75% after adding this fraction of DSN reinforcement. Moreover, the compression strength of the DSN-free PET matrix was increased by 41.7% for the same DSN addition. As well, this DSN-enhanced composite showed a higher spectral peak with more bonding molecules. In summary, date seeds, as functional fillers, have effectively demonstrated improved characteristics for polymer-based nanocomposites.

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