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ORIGINAL RESEARCH

Mechanical performance, thermal stability and morphological analysis of date palm fiber reinforced polypropylene composites toward functional bio-products

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Abstract Sustainable bio-materials are now potential alternatives for synthetic composites to achieve more functional green products. However, the microstructure-performance synergy is critical in such materials. In this work, the mechanical properties, thermal stability, and morphological analysis of the date palm polypropylene composites are investigated. Various reinforcement conditions, chemical treatments, surface topology, thermogravimetric analysis, and its derivative were utilized to explore the relation of the microstructure with the composite performance. Date palm leaflets (DPLs) were treated with sodium hydroxide at various conditions to determine the optimal samples. Morphological analysis was also performed. Results reveal that this treatment improves the tensile strength and modulus of the composites. Moreover, DPL fibers have positive impacts on both tensile and flexural modulus. At 30 wt% of DPL fibers, the fibers scored the highest values. Both TGA and DTG analyses show that DPL fibers can withstand a temperature up to 227 °C. Also, TG-DTG thermograms show that the addition of DPL fibers has enhanced the thermal stability of polypropylene composites. Scanning electron microscope enhanced our understanding of the composite performance trends towards assessing their capabilities for more reliable implementations of more sustainable green products.

Keywords Sustainable materials · Natural fibers · Biomaterials · Bio-products · Thermal stability

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Introduction

Green polymeric composites gained a big concern in many disciplines since they possess characteristics that do not exist in ceramic and metal materials. Such desired characteristics include high specific mechanical properties, lightweight, degradability features, and low cost of processing (AL-Oqla et al. 2018; Alarifi 2021; Rodriguez et al. 2016). Nevertheless, synthetic composites contribute to environmental pollution by releasing a huge amount of carbon dioxide and other toxic substances during their decomposition (Jang et al. 2020; Karan et al. 2019). Consequently, attempts have been made to substitute synthetic polymeric composites with bio-polymeric alternatives, which in turn reduce the adverse outcomes of polymer degradation. Natural fibers based composites have attracted researchers around the world due to the advantages of natural fibers including low cost, lightness, biodegradability, availability, and good thermal and mechanical properties (AL-Oqla et al. 2019; AL-Oqla 2021b; Ates et al. 2020; Fares et al. 2019; Hayajneh et al. 2021; Thakur et al. 2012).

Several lignocellulosic natural fibers, such as kenaf, pineapple leaf, bamboo, banana, cotton, date palm, olive leaves, grape, hay, lemon, reed, rice husk, kenaf, jute, as well as others, are utilized in natural fibers reinforced composites (Asyraf et al. 2021a; Azman et al. 2021). The effects of natural lignocellulosic fibers on mechanical and thermal properties in numerous polymer matrices were investigated in several studies. It was reported that various weight percentages of green olive leaves as fillers were utilized to produce low-density polyethylene (LDPE) composites by using a double-screw mixer machine for mechanical characterization. Both flexural strength and modulus in conjunction with impact rupture were improved up to 40 wt% of the fibers. In addition, (AL-Oqla 2021a) used three weight percentages (20, 30 & 40) of grape fibers to reinforce LDPE. Soaked and dry fibers were employed. The tensile strength and the modulus of elasticity properties were investigated. Both dry and soaked fibers were found capable of improving the tensile strength regardless of the wt% whereas the modulus of elasticity was decreased at 20 wt% and improved at both 30 wt% and 40 wt% of fibers. Furthermore, (AL-Oqla 2021c) utilized various loading (10, 20 & 30 wt%) of lignocellulosic fibers including hay, palm, lemon, and reed to fabricate polypropylene (PP) composites via a single-screw extruder and compression molding. They evaluated tribological and mechanical properties. Their results indicate that the tensile strength values were decreased at all wt% regardless of the type of fibers. However, changes in strength within the same fiber type occurred based on the weight percentage.

Nevertheless, no consistent pattern was observed in the tensile modulus property. A similar trend was noticed in the impact strength property of the composites. Regardless of the type of fibers, the coefficient of friction increased at all weight percent of fibers. On the other hand, Yew et al. (2019) studied the effects of using coir fibers on the thermal properties of the epoxy matrix. The effect of soaking time of 5% sodium hydroxide on thermal stability was also investigated. It was reported that treatment by NaOH can cause better thermal stability and lower resistance of flame retardancy. Additionally, the effects of various wt% of banana fibers on the mechanical and thermal properties of 3 wt% nano-clay-PP were investigated by Biswal et al. (2012) who found that the fibers improve the tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength except at 40 wt% where these properties decrease, but they are still higher than the values of the virgin matrix. In conjunction with mechanical properties, the fibers also improved thermal stability.

Among the aforementioned natural fibers, date palm (*Phoenix dactylifera*) fibers preserve some characteristics that distinguish them from other natural fibers. For example, date palm (if particularly compared with coir, sisal, and hemp) has the highest production rate of waste where its annual pruning process produces 4.2 million tons of waste that makes them widely available and renewable. Further, date palm possesses the lowest density, 0.9–1.2 g/cm^3, and the lowest cost, 0.05 USD/Kg when compared specifically with coir, hemp, and sisal (AL-Oqla and Sapuan 2014). Moreover, date palms can withstand harsh conditions (El-Juhany 2010). Such properties make parts of date palm tree usable in many applications such as baskets, bags, crates, fans, food covers, mats, trays, furniture, livestock food and decoration beads, shades, fishing boats medical treatment, etc. (Ghori et al. 2018). In addition, date palm fibers as well as others were utilized in composites especially for bio-implantable sensors, photovoltaic applications, and automotive industries like bumpers (Fares et al. 2019; Muthalagu et al. 2021). Nonetheless, hydrophilic date palm fibers, like other natural fibers, have a major drawback of poor bonding with hydrophobic polymers that requires some chemical treatment to overcome this issue (AL-Oqla et al. 2021b; Thakur et al. 2013).

Lignocellulosic fibers contain cellulose, hemicellulose, lignin, and pectin. The existence of amorphous components like lignin and hemicellulose on a fiber surface decreases its ability to properly interact with the polymer matrices. The immersion of such fibers in chemical solutions usually removes the hydroxyl group constituents (hemicellulose, lignin, and pectin) along with the impurities. Therefore, better adhesion between the hydrophilic fibers and the hydrophobic polymer is achieved (Asyraf et al. 2021b; Nurazzi et al. 2021). Since the bonding between fillers and matrices is one of the most factors that affect mechanical and thermal properties, many researchers have chemically treated different fibers to improve their bonding with polymeric matrices. For instance, Zanini et al. (2021) immersed Australian Royal palm residue in a sodium hydroxide (NaOH) of 4% concentration for 1 h. It was revealed that NaOH can make the surface rougher. It can also remove the non-cellulosic substances and increase the possibility of interaction between fibers and matrices. Moreover, Al-Otaibi et al. (2020) has decreased the number of pull-outs and enhanced the adhesion at the polymer-fiber interface by soaking the date palm fibers in NaOH of 5% concentration for 24 h. Chihaoui et al. (2020) have improved the mechanical properties of the polypropylene matrix by soaking the date palm waste in 5% NaOH for 2 h. In any case, it was found that treated date palm fibers with higher concentrations of NaOH (more than 9%) and long periods (more than 24 h.) can cause damage for fibers leading to bad performance in the composites (Alsaeed et al. 2013a).

Several parts of the date palm tree have been used as fillers in polymer matrices. For example, Alarifi (2021) used leaves, core–shell, and the branch of the date palm tree in epoxy resin. Al-Otaibi et al. (2020) tried to reinforce the date palm fibers in different types of polypropylene. Moreover, Belgacem et al. (2020) reinforced polypropylene by the waste of the date palm tree (rachis, leaf, and leaflet). Date palm seeds were utilized as a reinforcement for vinyl ester by Nagaraj et al. (2020). Additionally, date palm fronds were exploited as a filler for low-density polyethylene by AlZebdeh et al. (2017). Date palm seeds were also used with epoxy by Tripathy et al. (2016) and Alsaeed et al. (2013a). More details about these studies and their remarks are presented in Table 1.

Polypropylene, on the other hand, can be considered one of the most widely used polymers. It offers many advantages including a low-processing temperature, which enables the use of natural fibers that suffer from low thermal stability. It is also thermally stable and recyclable. PP, in addition, can work under severe conditions (Agarwal et al. 2021). We conclude based on literature data that date palm fibers can improve the mechanical and thermal properties of various polymer matrices. However, the literature lacks any previous work demonstrating the effect of utilizing date palm leaflets of uniform shapes with specific aspect ratios to reinforce polypropylene and to characterize their thermal properties in conjunction with the mechanical ones.

Consequently, the current research is another step aiming to investigate the mechanical properties, thermal stability as well as performing morphological analysis of the date palm leaflets/polypropylene composites. This research was performed by carrying out a series of experimental works to reveal and optimize the effect of various reinforcement and treatment conditions on such composites. The work also aimed to assess the effect of microstructure and surface topology on the mechanical performance trends to develop a better understanding of their capabilities in enhancing the performance and reliability of sustainable green products.

Materials and methods

Materials

Virgin polypropylene matrix in the granules form was obtained from Saudi Arabia (SABIC Company) with a density of 0.9 g/cm^3 and thermal conductivity of 0.26 W/m.k. Date palm fibers were extracted from the leaflets only with a specific aspect ratio (10.4) and a density of 0.90 g/cm^3. Some of the date palm leaflets were chemically treated by sodium hydroxide (purity > 99%) in various concentrations and at different times as detailed later in the next sections.

Methods

Figure 1 displays a flowchart of the current research starting from the procedure of date palm leaflets preparation, passing through the chemical treatment and composites performing, and it ends with mechanical, thermal, and morphological characterizations.

Preparation of date palm leaflet fibers

The natural date palm leaflets (Fig. 2a) were obtained as a filler in the PP matrix. The leaflet fibers were prepared by several steps. Initially, the leaflets were extracted and gathered from a local date palm tree (Fig. 2b). Then, they were washed with water to remove the dust and deposits. After that, the leaflets were sliced manually to small long fibers (Fig. 2c). Then, the sliced

Table 1 Physical properties of different parts of the date palm tree and their effects on the mechanical and thermal properties of composites

Reference	Matrix	Date tree part	Dimensions	wt%	Density	Treatment	Remarks
Alarifi (2021)	Epoxy	Leaves Branches Core shells	1 µm	–	–	–	The branch fiber/epoxy hits the highest tensile strength while the leave fiber composite reaches the highest tensile modulus A similar trend is observed in tensile flexural properties
Al-Otaibi et al. (2020)	HPP–ICP–RPP	–	< 2 mm & 8–12 mm	(5–15) Interval 5%	–	NaOH	The chemical treatment improved the tensile properties Increasing the wt% positively affects the tensile modulus. In contrast, the increase in the wt% led to a reduction in the tensile strength The additional fibers can decrease thermal stability Chemically treated fibers give better results than untreated fibers
Belgacem et al. (2020)	PP	Mixture (rachis, leaflet & leaf)	10 mesh	40	–	Defibration Enzymatic NaOH	All types of treatment enhanced the tensile properties Enzymatic reached the highest tensile strength and modulus followed by the chemical treatment
Nagaraj et al. (2020)	Vinyl ester	Date seeds	30–60 µm	(5–50) Interval 5%	–	–	30 wt% climaxed the tensile, flexural, impact, and hardness properties Thermal stability was highly improved by the addition of 30 wt% as compared to 5 wt% of date palm seeds

Table 1 (continued)

Reference	Matrix	Date tree part	Dimensions	wt%	Density	Treatment	Remarks
Gheith et al. (2019)	Epoxy	–	0.8–1 mm	40–60 Interval 10%	–	–	The incorporation of date palm fibers improved the thermal stability at all wt% The flexural strength and modulus also increased at all wt%
Saba et al. (2019)	Epoxy	–	0.8–1 mm	40–60 Interval 10%	–	–	The addition of date palm fibers improved the flexural strength and modulus at all wt%, but 60 wt% reached the lowest value, which was better than the neat epoxy The same behavior was observed in impact strength
AlZebdeh et al. (2017)	HDPE	Date palm fronds	Continuous in three directions (0,45 & 90°)	20	1.17 g/cm^3	NaOH	Adding date palm fronds to LDPE caused deteriorations in both tensile strength and modulus at all orientation angles. However, 45° orientation touched the highest tensile strength when compared to 0 and 90°
Tripathy et al. (2016)	Epoxy	Date palm stem	15 mm	10	–	NaOH	Tensile and flexural strength were investigated at 10 wt% Treated fibers gave better results
Alsaeed et al. (2013a)	Epoxy	The outer layer of the tree stem	Continuous with the sample length	–	–	NaOH	6 wt% of sodium hydroxide reduced the tensilestrength when compared to 3 wt% of NaOH

Table 1 (continued)

Reference	Matrix	Date tree part	Dimensions	wt%	Density	Treatment	Remarks
Mahmoudi (2013)	PP	Date palm rushes	10 & 0.5 mm	5–30 Interval 5%	1540 $kg.m^3$	NaOH	Coupling the date palm fibers enhanced tensile strength and modulus Better results obtained by treated fibers when compared to untreated fibers

HPP homopolymer polypropylene, *ICP* impact copolymer Polypropylene, *RPP* recycled polypropylene, *HDPE* high-density polyethylene

leaflet fibers were chopped to small lengths (Fig. 2d), in such a way that maintains the aspect ratio at higher than 10 as suggested by Azeredo et al. (2009). Since the dimensions of used fibers affect the properties of the composite (Yaghoobi and Fereidoon 2019), the aspect ratio was ensured by calculating the length and width of DPLs for a selection of 75 leaflet fragments, and their values were 10.4 ± 0.27. This was performed to enhance the overall mechanical performance of the fabricated composites.

Alkaline treatment

To improve the wettability between the fibers and the matrix, an alkali surface treatment was obtained where the chopped fibers were soaked for different periods in different concentrations of sodium hydroxide aqueous solution at a temperature range of (23–27 °C). After the chemical treatment, the fibers were filtered from the aqueous solution, and then they were rinsed many times by distilled water to carry away the remnant of NaOH. Eventually, the treated fibers were dried again for 36 h to reduce the amount of moisture.

To find out the effect of the NaOH concentration and immersion period on the fiber properties, two levels of sodium hydroxide concentrations and soaking periods were examined at DPLs (Table 2). Each combination of the concentration and immersion period was replicated three times. The combination that demonstrated the highest tensile strength and modulus of elasticity was applied for all wt% of DPLs. Some fibers were also left as it is without any chemical processing. The mechanical properties of the treated and untreated fibers were then investigated.

Composite preparation

Three levels of fiber weight percentages (10 wt%, 20 wt%, and 30 wt%) were compounded with a PP matrix (Table 3). The components of the composite were mixed at a temperature of 190 °C by a singular screw extruder and then compressed at room temperature.

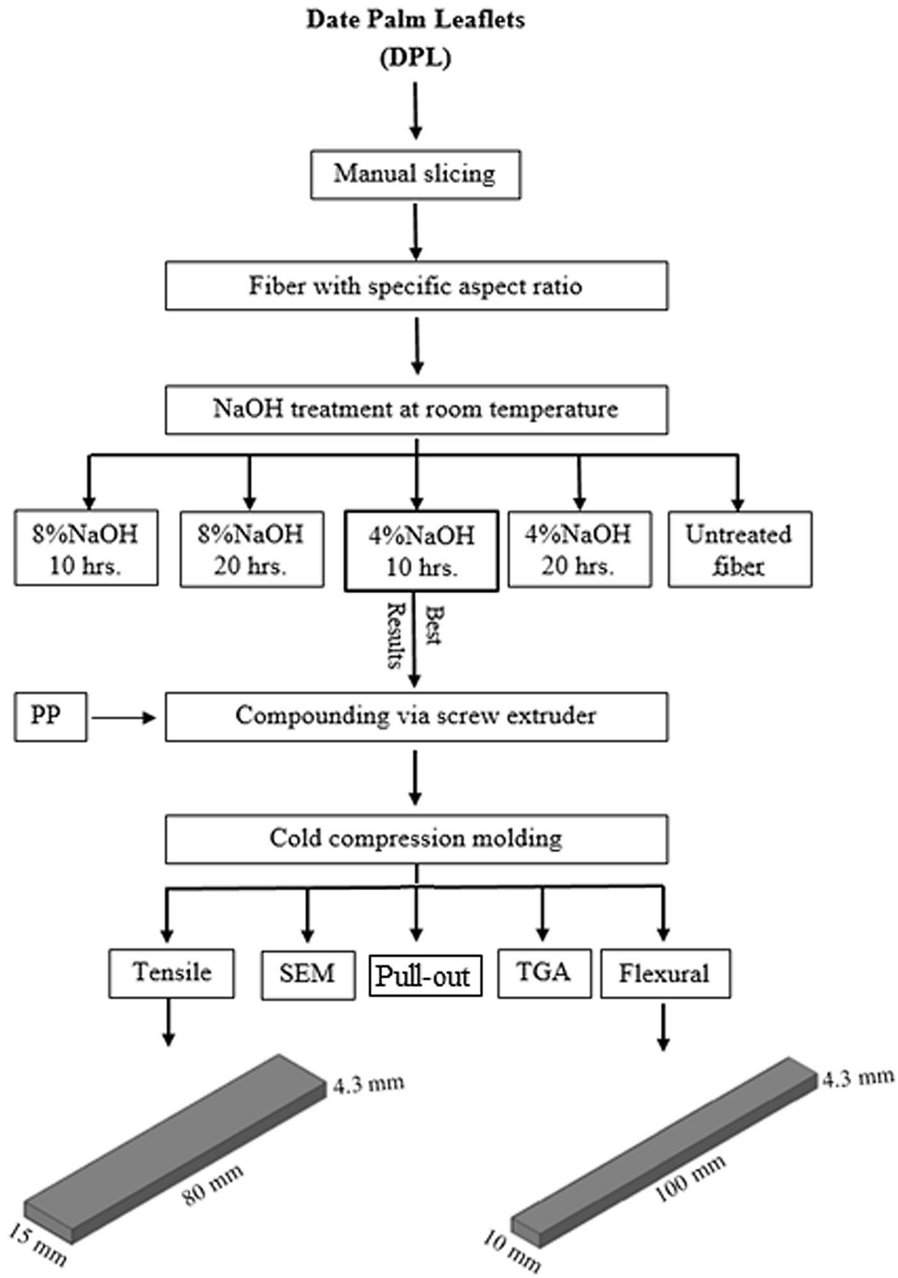

Fig. 1 Flowchart of the current research

Characterization

Tensile test

A Universal Testing Machine UTM (model WDW-20) with 40 KN capacity was utilized to carry out a uniaxial tensile test to evaluate the tensile strength, tensile modulus, and elongation at break. The tests were performed at room temperature according to the ASTM D3039-3039 M standard. The specimen dimensions were set at 80 × 15 × 4.3 mm while the crosshead speed and gauge length were 2 mm/min and 50 mm, respectively.

Fig. 2 DPL fibers preparation: **a** DPLs, **b** extracted leaflets, **c** sliced DPLs, **d** chopped DPL

Table 2 The combination of different concentrations and soaking times

Formula	NaOH concentration (wt%)	Time (h)
UT-DPL	0	0
4NaOH-10Hr	4	10
4NaOH-20Hr	4	20
8NaOH-10Hr	8	10
8NaOH-20Hr	8	20

Table 3 Formulation of PP composites

Sample formula	DPL (wt%)	PP (wt%)
Neat PP	0	100
10DPL-PP	10	90
20DPL-PP	20	80
30DPL-PP	30	70

Flexural test

A three-point bending test was performed on the same Universal Testing Machine that was utilized in the tensile test at room temperature according to ASTM D790 to determine the flexural strength and flexural modulus with a crosshead speed of 5 mm/min and a support span of 70 mm. The specimens were in the form of a rectangle with dimensions of $100 \times 10 \times 4.3$ mm^3 (Fig. 3).

The pull-out test

To ensure the suitability of the date palm fibers with the PP, the interfacial bonding capacity was studied via pull-out technique to determine the maximum applied load that the fibers/polymer adhesive forces can withstand. The pull-out technique can indicate the apparent shear strength at the interface between

Fig. 3 Samples before the flexural test

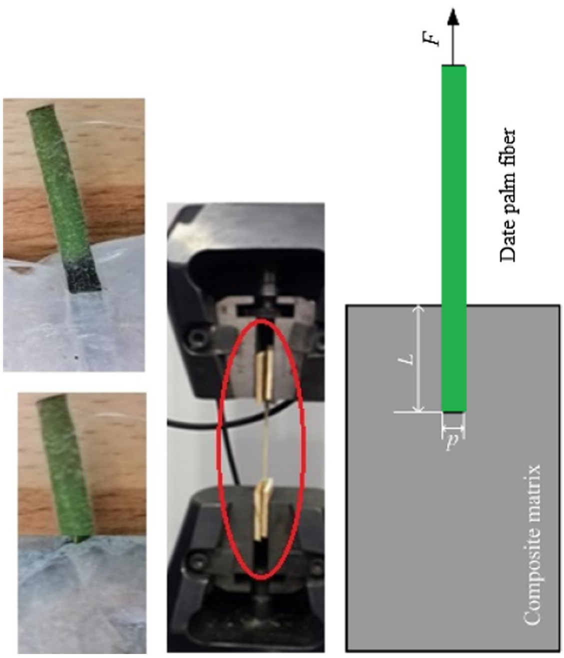

Fig. 4 Schematic representations of the pull-out test as well as fibers under test

the fibers and the polymer. It also represents the compatibility of the agricultural waste fibers with polymer materials. The single fiber pull-out tests were performed using a microcomputer-controlled universal testing machine of maximum applied force 22.5 N. Figure 4 shows the schematic diagram of the single fiber pull-out test for the rectangular prism date palm fibers. The Interfacial Shear Strength (IFSS) were calculated (Eq. 1) (Liu et al. 2019)

$$\tau_{IFSS} = F/A = F/2(t * L + P * L) \quad (1)$$

where F is the maximum force, A is the interfacial area between date palm fibers and polypropylene matrix, t is the thickness of the date palm fibers, P is the width of the date palm fiber, and L is the date palm fiber length embedded in the composite matrix. A crosshead speed of 0.5 mm/min was used. Specimens were prepared with embedded-fiber lengths of 15 mm–20 mm and a free-fiber length of ~ 40 mm.

Thermogravimetric characterization

The low-processing temperature is one of the most prominent drawbacks of natural fibers. Exposing natural fibers to thermal stresses may cause earlier degradation during the processing of polymeric composite and thus deteriorate the mechanical properties (Oktaee et al. 2017) Therefore, thermogravimetric analysis (TGA) was used to investigate the thermal stability of DPL fibers. Also, TGA was used to investigate the influence of coupling 30 wt% of DPL fibers with PP matrix on thermal behavior. Samples of (25 ± 3 mg) in aluminum pans were scanned from 30 to 600 °C at a heating rate of (10 °C/min) under a nitrogen atmosphere of 20 ml/min as a flow rate. The test was performed concerning dynamic TGA using a TG analyzer (NETZSCH TG 209 F1 Iris).

Scanning electron microscope

The surface morphology and the cross-sectional properties of both treated DPL fibers and untreated DPL were analyzed using a scanning electron microscope (SEM) (Quanta™ 450 FEG SEM) with an acceleration voltage of (10–20) KV. Additionally, the interfacial bonding between the fibers and the matrix, the distribution of the fibers through the matrix, and the type of fracture have also been depicted after the tensile test for the composites. To avoid electrostatic charging, a gold layer of 18 nm was sputtered over the samples surfaces.

Results and discussion

Results of alkaline treatment

The effect of alkaline treatment on the DPL/PP composites was investigated under the considered various concentrations and time durations. According to

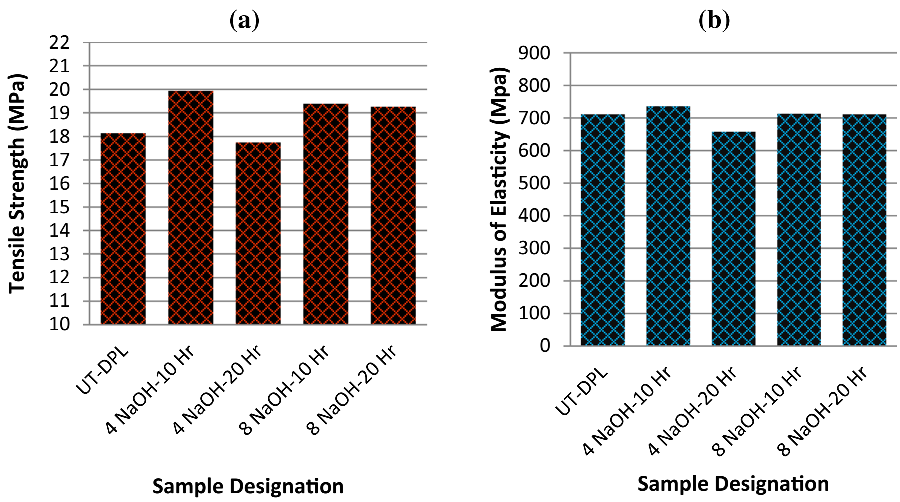

Fig. 5 The effect of NaOH concentration and soaking time on **a** tensile strength and **b** modulus of elasticity

Fig. 5a and b, it can be noticed that sodium hydroxide positively affected both the tensile strength and modulus of elasticity at all combinations except at the combination of (4NaOH-20Hr) where it decreased marginally. Further, it can be seen from Fig. 5a and b that the combination (4NaOH-10Hr) has touched the highest values where the tensile strength increased about 11% and the modulus of elasticity raised from 711 to 735 MPa. Therefore, the combination (4NaOH-10Hr) can be applied to the whole quantity of DPL fibers. Moreover, it can be observed that the long soaking time, 20 h, has negatively affected the tensile properties regardless of the wt% of NaOH solution. This is due to the over-treating time. A similar conclusion was obtained by Asumani et al. (2012) for kenaf fibers. To facilitate interpreting the increment that happened in tensile strength and modulus at the combination of (4NaOH-10Hr), a morphology comparison of two fibers of untreated and treated DPL was revealed by scanning electron microscope analysis.

Tensile test

The impact of DPL loading on the neat PP concerning tensile properties is presented in Fig. 6. Reinforcing the polymer with DPL fibers led to a decrease in tensile strength (Fig. 6a). This reduction is due to the low amount of DPL fibers (10 wt% and 20 wt%). Such a small number of fibers causes stress concentrations in the composite and prevents the fibers from playing their key role in resisting the external load. A similar interpretation was obtained by Asim et al. (2021). At 30 wt% of DPL fibers, the strength slightly increased but it stayed below the neat PP. This marginal increment can be attributed to the fact that at 30 wt%, the fibers began to exercise their main function, which is to bear the external forces. Figure 6b shows that the modulus of elasticity is enhanced by adding all wt% of DPL fibers. For instance, 10 wt% of DPL fibers increased the modulus of elasticity from 654 to 735 MPa. At 20 wt% of DPL fibers decreased from 735 to 712 MPa, but it is still higher than of neat PP. By comparison, 30 wt% of DPL fibers improved the modulus from 654 to 722 MPa. The tensile strength and the modulus of elasticity have the same trend. Concerning the elongation to break (EB), as depicted in Fig. 6c, it is noticeable that increasing the wt% of DPL fibers leads to decreasing the EB where 30 wt% of DPL fibers composite have dropped by 73%. This means that DPL fibers reinforcing has enhanced the brittleness of the PP matrix. Such a trend is expected due to the nature of composites.

Stress–strain curve

Figure 7 shows the tensile stress–strain curve of the 10 wt% DPL-PP. It can be shown that all replicates of the composite experienced a brittle fracture. Such a fracture may refer to the presence of treated brittle fibers. Also, the figure displays some natural differences in the tensile strength, elongation at break but with high constancy in modulus of elasticity.

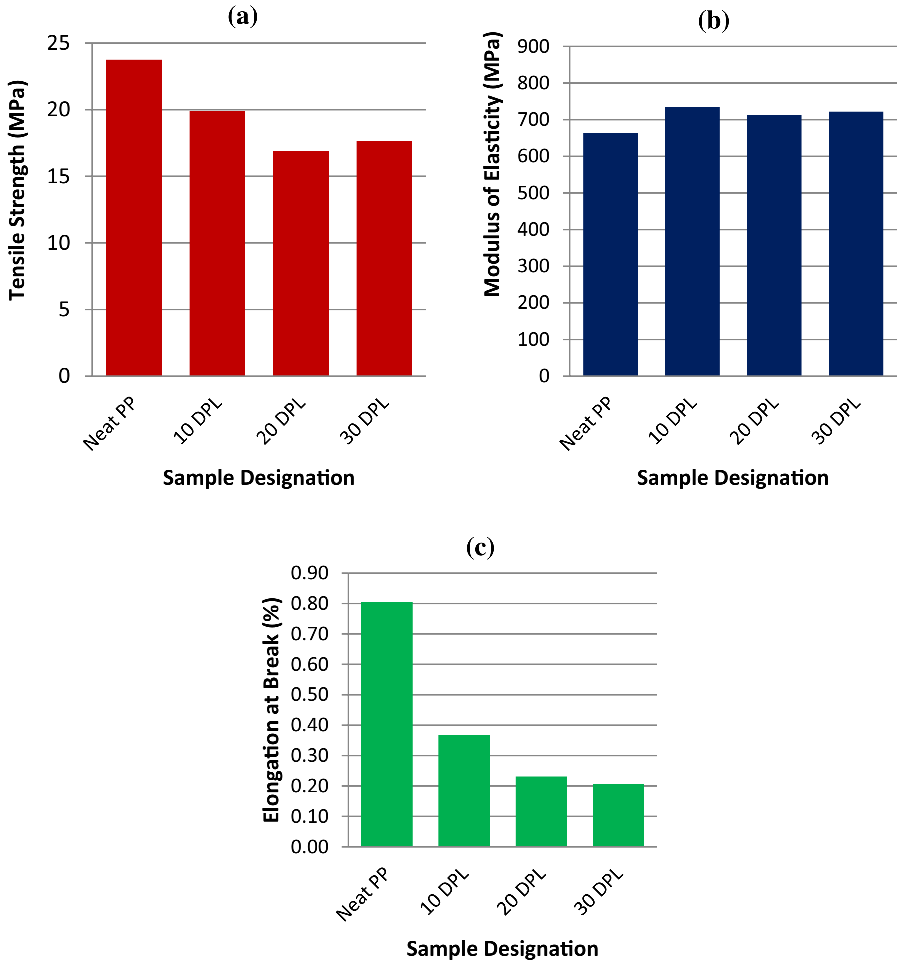

Fig. 6 Tensile properties of DPL-PP composites

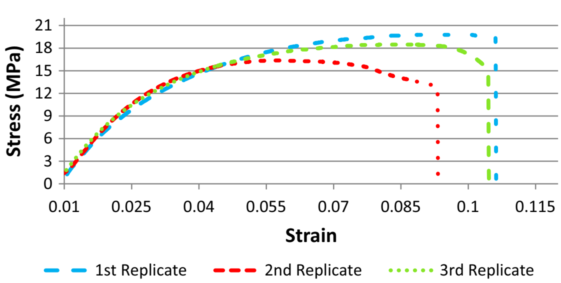

Fig. 7 Stress–Strain Curve of 10DPL-PP

Flexural test

The effects of reinforcing PP matrix with DPL fibers on both flexural strength and flexural modulus are displayed in Fig. 8. It is clear that embedding 10 wt% of DPL fibers reduced the flexural strength by approximately 13%. At 20 wt% of DPL fibers, the flexural strength also reduced by 14%. On the contrary, 30 wt% of DPL fibers made the flexural strength more valuable. However, all wt% of DPL fibers cannot reach the flexural strength of PP. The flexural modulus in Fig. 8b depicts similar behavior, i.e., 10 wt% and 20 wt% of DPL fibers reduced the flexural modulus. However, the 30 wt% case of DPL fibers increased the flexural modulus. The reduction that happened at 10wt% and 20 wt% of DPL fibers is attributed to stress concentrations inside the composite.

Interfacial shear strength

Figure 9 shows the interfacial shear strengths of treated and untreated date palm fibers with polypropylene. It can be demonstrated that the interfacial

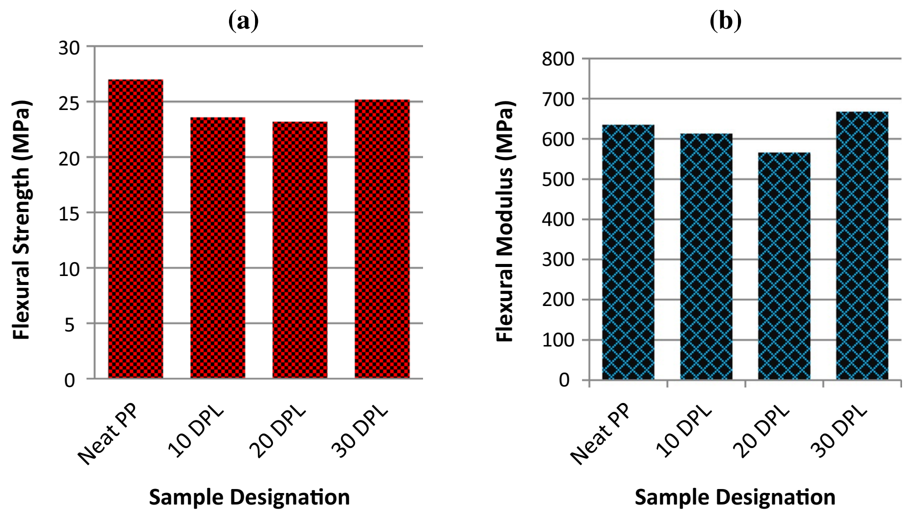

Fig. 8 Flexural properties of DPL-PP composites

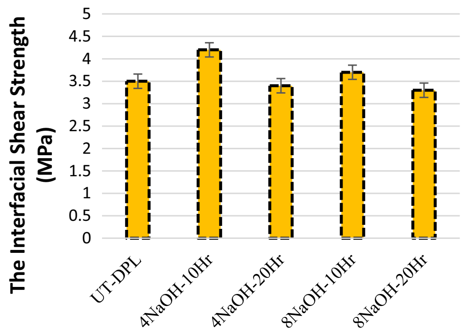

Fig. 9 The interfacial shear strength of the treated and untreated date palm fibers/PP composites

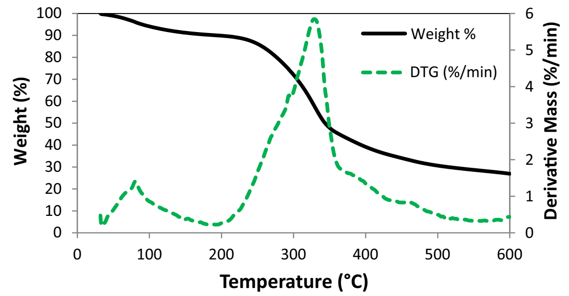

Fig. 10 TG and DTG thermogram of DPL fibers

shear strength between the untreated fibers and PP was reasonably high. It was 3.5 MPa. However, treated fibers with 4% NaOH for 10 h have the highest interfacial shear strength with PP. This is due to the effect of treatment on the date palm leaflet surface. It was capable of enhancing the surface roughness of the fibers and delaminating the lignin layer causing more pores, and it resulted in better mechanical interlocking with the PP matrix. The same treatment for 20 h has a negative effect on the surface resulting in only 3.4 MPa interfacial shear bonding. The interfacial bonding of date palm fibers with PP has demonstrated a good composite mechanical performance like the tensile strength and modulus, which presents promising opportunities for the date pale fibers to be employed in producing green products for sustainable industries.

Thermogravimetric analysis (TGA)

Thermogravimetric analysis of DPL fibers

Figure 10 displays the TGA and its derivative (DTG) curves of DPL fibers. It can be observed from the TGA that initial mass loss (7.5%) occurred in the range from room temperature up to~ 125 °C due to moisture evaporation. The DPL fibers have started decomposing at a temperature (T_{onset}) of 227 °C where the fibers have lost 12% of their mass, which closely matches the findings of Ali et al. (2017) regarding date palm tree surface fibers. When the mass loss has reached (50%), the degradation temperature ($T_{50\%}$) was approximately found to be 342 °C. The degradation ended at a temperature (T_{endset}) of 354 °C. The mass loss, 54%, happened from T_{onset} to T_{endset} referring to the decomposition of hemicellulose and cellulose. The decomposition after

350 was related to the degradation of non-cellulosic substances (Alothman et al. 2021; Dehghani et al. 2013). Based on these results it can be concluded that the DPL fibers were not affected by the processing temperature of PP, i.e. 190 °C, because its initial degradation temperature, 227 °C, is higher than the composite's manufacturing temperature. Also, this test indicates that DPL fibers can resist high temperatures, up to 227 °C. Moreover, the DTG curve presents the maximum degradation temperature (T_{max}) which is defined as the temperature at which the mass loss rate hits the maximum value. In the case of DPL fibers, the T_{max} reaches 326 °C. Furthermore, from DTG, it can be observed that there are three prominent apexes. The first one (P1) represents the moisture evaporation rate, the second one (P2) displays the degradation rate of cellulosic content while the third one (P3) shows the rate of degradation of non-cellulosic materials.

Thermogravimetric analysis of PP and its composite

Figure 11 demonstrates the TGA and DTG for the PP matrix and 30 wt% DPL/PP composite. As observed from the TGA curve (Fig. 11a), the PP matrix has a higher onset temperature (321 °C) than 30 wt% DPL/PP composite (269 °C) and DPL fibers (227 °C). A similar result was obtained for the PP-bagasse composite by Correa-Aguirre et al. (2020). However, PP has no char residue after 550 °C. In contrast, DPL fibers and their composites have char residues of 27 and 5%, respectively. The presence of char residue at the end of the test in DPL fibers and 30 wt% DPL/PP refers to the lignin, which contains a very thermally stable aromatic phenyl group (Ahmad Saffian et al. 2020). Moreover, the TGA curve presents the final degradation temperature and the temperature at a mass loss of 50% of the 30wt. The percentage of DPL/PP composite was higher than that in the PP matrix. Thus, DPL fibers improve the thermal stability because it works as a heat barrier in the PP matrix leading to an increase in the amount of char residue indicating better flame retardancy of PP (Samal et al. 2009).

The neat PP shows single-step degradation whereas the composite, 30wt% DPL/PP displays a two-step degradation as depicted from DTG. The first step represents the degradation of DPL fibers and the second step corresponds to the degradation of PP. A similar pattern was noticed by Gheith et al. (2019) for date palm fibers-epoxy composites. The presence of DPL fibers in virgin PP has shifted the peak of the DTG curve from 410 to 450 °C (Fig. 11b). This means that 30 wt% of DPL fibers has enhanced the thermal stability of PP. The thermal parameters are tabulated in Table 4.

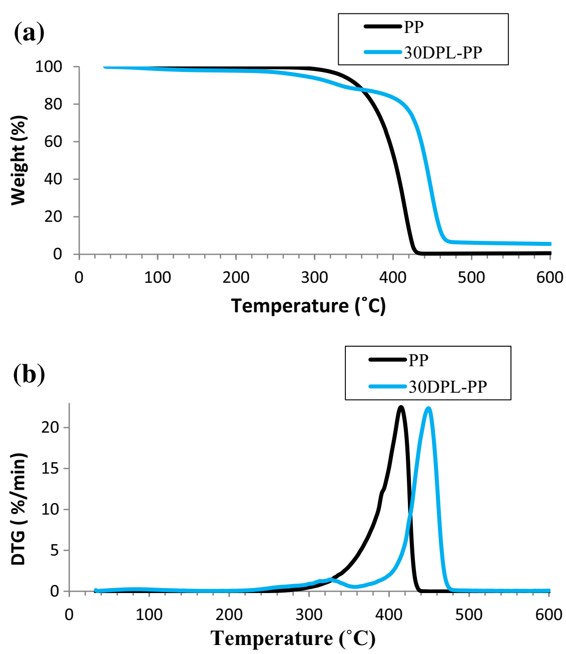

Fig. 11 Thermogravimetric Analysis for the PP and 30 wt% DPL/PP composite **a** TGA and **b** DTG

Table 4 TG and DTG data for DPL fibers, PP matrix, and 30DPL-PP composite

Sample	T_i (°C)	$T_{50\%}$ (°C)	T_f (°C)	T_{max} (°C)	The residue after 550 °C (%)
DPL	227	342	354	326	27.7
PP	321	403	430	410	0
30DPL-PP	269	440	470	450	5

T_i initial degradation temperature, T_f final degradation temperature, T_{max} the temperature at which the mass loss rate hits the maximum value, T_{50} the temperature when the mass loss is 50%

Morphological analysis

The Morphology of treated and untreated DPL fibers

Figure 12 shows a comparison at the same magnification between two DPL fibers before (Fig. 12a) and after (Fig. 12b) the chemical treatment by the combination of (4NaOH-10Hr). It can be observed that the untreated fibers (Fig. 12a) contain a larger number of deposits when compared to treated fibers (Fig. 12b). In addition, the lignin layer is observed before and after the treatment, which means that the combination (4NaOH-10Hr) has an insignificant impact on the lignin layer removal. However, before the treatment, the lignin is visible, and no pores are observed in the micrograph (Fig. 12c). After the treatment, the lignin layer tends to be delaminated, but it is not separated due to little amount of NaOH. In addition, some pits have emerged as a result of chemical treatment (Fig. 12d). This result is in agreement with (Alsaeed et al. 2013b). What happened during the treatment process (i.e., the impurities removal and the presence of some pits) may interpret the small increment of both tensile strength and modulus (Fig. 5). The pits in the lignin layer (due to treatment) make the surface rougher, which enhances the interlocking with PP. Also, these pits expose some parts of cellulose that interact with PP, enhancing mechanical properties (Dehghani et al. 2013).

The SEM micrographs of the cross-sectional view of untreated fibers are presented in Fig. 13. A similar SEM micrograph for DPL fibers was observed by Asim et al. (2020). From Fig. 13a, the lignin layer can be observed easily in untreated fibers as marked by the yellow circle. Moreover, the thickness of the DPL fibers is measured by SEM in the same figure. Also, the structure of DPL fibers is depicted in Fig. 13b. It can be seen that the fibrils have a solid cylindrical shape.

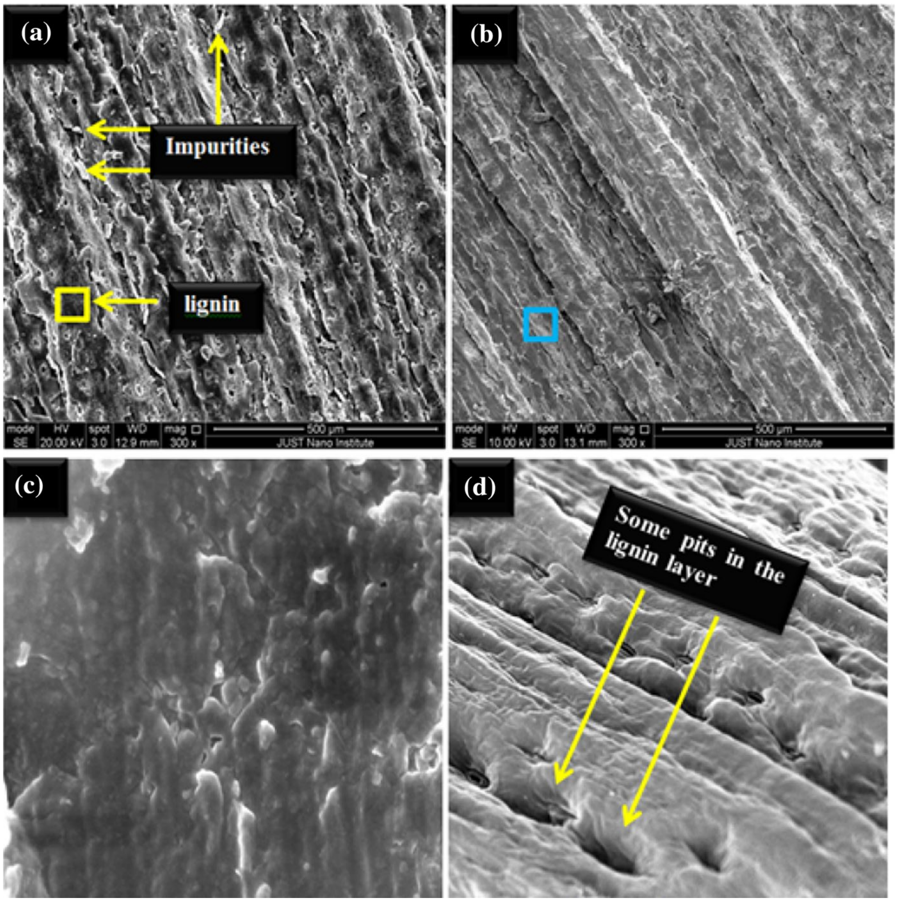

Fig. 12 SEM micrograph of **a** untreated, **b** treated DPL fibers, **c** a magnification for the yellow square in micrograph **a**, **d** a magnification for the blue square in micrograph (**b**)

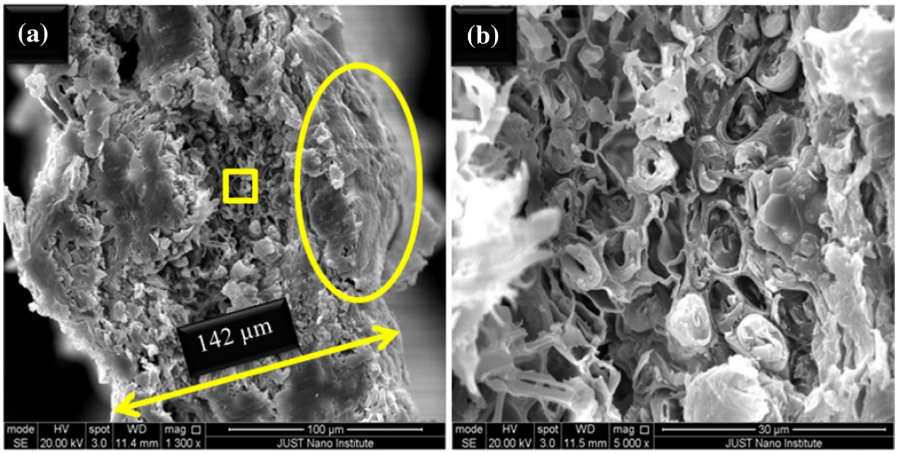

Fig. 13 SEM micrograph of **a** cross-sectional view of untreated DPL fibers, **b** a magnification for the yellow square in micrograph (**a**)

Surface morphology after tensile test

Figure 14 shows the morphology of the top surface of the 30DPL-PP composite after the tensile test. It can be noticed that there is a large number of DPL fibers (covering the surface of the composite) and the matrix is almost hidden (Fig. 14a). All presented fibers in the micrograph are broken (Fig. 14a) but they are not separated from the matrix. This can be inferred by the absence of pores in SEM micrographs. The breakage of the fibers instead of pulling out may refer to the good adhesion between PP and DPL fibers. Nevertheless, the nature of breakage (Fig. 14b) in fibers indicates their brittle fracture. Figure 14c shows ductile fracture of the PP matrix.

Conclusions

This work used a new form of DPL fibers to reinforce virgin PP. The morphological analysis was capable of developing a better understanding of the synergy of microstructure and performance trends of such composites. Embedding of DPL fibers has raised both tensile and flexural modulus of the polypropylene matrix. However, DPL fibers have lowered the tensile and flexural strength, especially at the 20 wt% cases. This reduction was attributed to the deficiency of DPL fibers, causing a high number of stress sites inside the composite. However, the 30 wt% DPL case was capable of improving both tensile and flexural properties since the DPL fibers have begun to resist the external load. The chemical treatment,

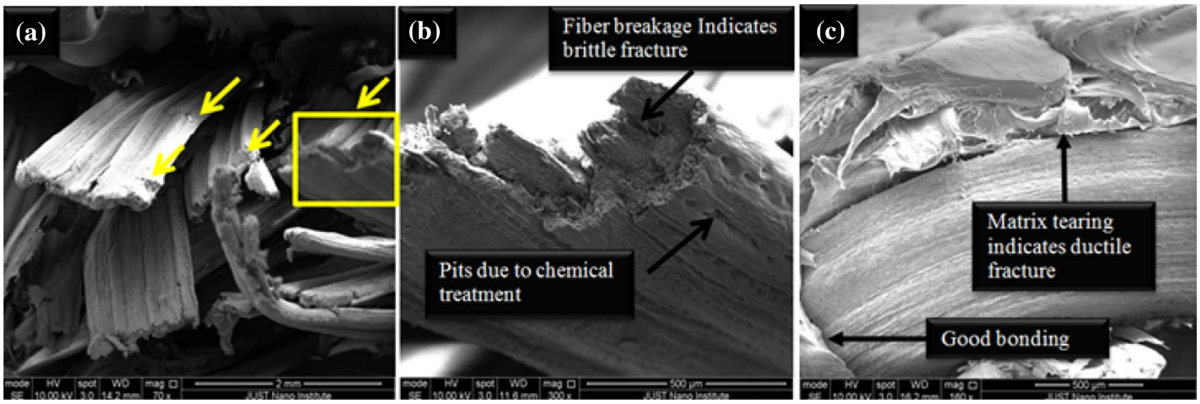

Fig. 14 SEM micrographs of **a** 30 wt% DPL/PP composite, **b** a magnification for the yellow square in micrograph (**b**), while **c** another micrograph for the same composite to illustrate more details

particularly, the 4NaOH-10Hr, enhanced the tensile properties. Aside from mechanical properties, DPL fibers of 30 wt% improved the thermal properties where the mass loss percent decreased, and the maximum degradation temperature increased by 40 °C. Moreover, the mechanical properties, thermal stability, and morphological analysis of the date palm polypropylene composites were assessed and interrelated to the microstructure of the composites. This reveals more reliable capabilities of such composites for better implementations in sustainable green products.

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Declarations

Conflict of interest All authors declare that they have no conflict of interest.

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