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Effect of fiber treatment on the mechanical properties of date palm fiber reinforced PP/EPDM composites

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In this work, the effect of date palm fiber (DPF) incorporation and its surface treatment with sodium hydroxide on the mechanical properties like tensile, bending, and impact of polymer composites based on polypropylene (PP)/ethylene–propylene–diene monomer (EPDM) is investigated. Maleic anhydride grafted polypropylene (MAPP) is used as a compatibilizer to improve the compatibility of (DPF) filled PP/EPDM composites. Results have shown that the incorporation of fibers increased the tensile and bending strength and decreased the impact strength. Treated fiber reinforced composites yield better mechanical properties as compared to the raw fiber reinforced composites. Scanning electron microscopy of the fracture surfaces of the composite specimens indicates that the MAPP and the treated fibers improved the interfacial interaction between the fiber and the matrix.

Keywords: date palm fiber; tensile; bending; impact; fiber treatment

1. Introduction

Over the last decade, polymer composites reinforced with natural fibers have attracted attention of scientists and various industries. The rapid growth in the consumption of plastic products, persistence of plastics in the environment, the shortage of landfill space, the depletion of petroleum resources, and entrapment by the ingestion of packaging plastic by marine and land animals have spurred efforts to look for better alternatives. The advantages of using natural fibers compared to synthetic fibers include low weight, recyclability, biodegradability, and renewability. It has relatively high strength and stiffness and has no skin irritations effects.[1–3] Enhanced properties have been obtained by using natural cellulosic fibers such as jute,[4] coir,[5] sisal,[6] straw,[5] jute,[6] flax,[7] hemp,[8] etc.

The properties of composites depend on the matrix, fibers, and on their interfacial bonding. The adhesion between the reinforcing fibers and the matrix in composite materials plays an important role in the final mechanical properties of the material, since the stress transfer between matrix and fibers determines the reinforcement efficiency. The surface of synthetic fibers is usually modified using various types of processes in order to improve the fiber surface wettability with the matrix and to create a strong bond at the fiber–matrix interface, which, in turn, provides an effective stress transfer between the matrix and the reinforcement fibers.[9] When natural fibers are used as reinforcement in composite materials, many problems occur at the interface due

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to incompatibility. All plant-derived cellulose fibers are polar and hydrophilic in nature, mainly as a consequence of their chemical structure. Polyolefins such as polypropylene (PP), on the other hand, are largely non-polar and hydrophobic. The incompatibility of the polar cellulose fibers and non-polar thermoplastic matrix leads to poor adhesion, which then results in a composite material with poor mechanical properties.[10] Therefore, surface modification of the natural fibers by means of treatment is one of the largest areas of current research to improve compatibility and interfacial bond strength.[11] It is worth to mention that the chemical treatment of the fibers can either increase or decrease the strength of the fibers, and hence, good understanding of what occurs structurally is required.[12–14]

Various fiber surface treatments are reported, such as silane,[15] acetylating,[16] coupling agents,[17,18] and monomer grafting under UV radiation.[19] Alkali treatment processes are cost-effective and have been widely used to improve natural fiber surface properties and thus composite properties.[20–22] The alkalization process affects natural fibers and has particular importance for fiber matrix adhesion by the creation of a high fiber surface area, which is required for the optimization of fiber resin reinforcement.[23] The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The type of alkali (KOH, LiOH, and NaOH) and its concentration influence the degree of swelling. Consequently, sodium hydroxide (NaOH) treatment results in a higher amount of swelling.[24] The adhesion between the fibers and the matrix can be improved by modifying the matrix with a coupling agent that adheres well to both fibers and matrix. Maleic anhydride grafted polypropylene (MAPP) has been shown to be one of the most suitable coupling agents available for the use in natural fiber reinforced PP composites.[25,26] PP as a thermoplastic is widely used in many fields, such as building materials, furniture, automobile, marine, and various important industries. However, the poor impact resistance, especially at low temperature, considerably limits its application. Therefore, PP is usually modified with elastomers, such as ethylene–propylene–diene monomer (EPDM) to improve its impact strength.[27–29]

The date palm tree, a member of the palm tree family (Phoenix dactylifera), is naturally found in the Middle East, Northern Africa, the Canary Islands, Pakistan, India, Iran, and in the USA (California). There are more than 100 Million date palm trees in the world, and each tree can grow for more than 100 years.[30] The palm tree stem is covered with a mesh as made of single fibers (see Figure 1). These fibers create a natural woven mat of crossed fibers of different diameters. Traditionally, the mat is removed from the trees and cleaned to make ropes and baskets in many parts of the world. However, these applications are accounted for a small percentage of the total potential world production.[30] The use of fibers surrounding the stem of date palm trees as reinforcement in polymeric materials has been reported in few studies.[31] The mechanical properties (flexural properties and impact strength) of DPF/polyester composites were found to be influenced by fiber content and fiber treatment method.[30]

The main objectives of the present work are as follows:

(1) To obtain various mechanical properties of the PP/EPDM/date palm fiber at different fibers weight fractions (0–30%).
(2) Comparison between the properties of samples made with raw fibers and treated fibers.
(3) To determine the optimum fibers weight fractions for best mechanical properties.

Figure 1. Date palm tree stem covered with mesh made of fibers.

Therefore, composite specimens made of palm fibers and PP/EPDM were prepared and according to the standards; the mechanical testing included tensile test, bending test, and impact test is conducted on the specimens to characterize the mechanical behavior of date fiber/PP composites and also to study the effect of treatment of date fibers on these mechanical properties.

2. Experimental

2.1. Materials

The PP/EPDM granules with 80:20 weight ratios with PP: EPC40R and EPDM: KELTAN 8340A of Kimia Forooz Inc. Co., Iran, were used for polymer matrix. MAPP was bought from Sigma Aldrich Chemical Co., USA. Sodium hydroxide (NaOH) was obtained from Merck Co., Germany, and the fibers were collected from amplexicaul, the sheathing leaf base, which surrounds the date palm tree stems.

2.2. Treatment of date palm fibers

Fibers were collected from date palm trees in Iran. After that, the fibers were washed by distilled water to eliminate dirt and were dried in an oven at 70 °C for 24 h. These fibers are termed as raw fibers. Then, DPF immersed in a solution of 1 wt.% NaOH for 1 h at 100 °C. The treated fibers were washed out in distilled water for several times to remove the excess of NaOH on the fibers, at the end of treatment process; the fibers are cooled to room temperatures. Then, the DPF was washed thoroughly with distilled water and dried in an oven at 70 °C for 24 h; these fibers are called as treated fibers. Result shows that the treatment of conditions of 1% NaOH for 1 h at 100 °C is the optimum treatment that gives the maximum tensile strength and the better surface morphology of the single DPF.[31] In the next step, the treated and raw fibers were cut to the length of 1–3 mm pieces using an industrial granulator.

2.3. Sample preparation

Both treated and raw fibers, at four levels of fiber weight fractions, namely 5, 10, 20, and 30 wt.%., and the amount of 2% by wt. *of* MAPP as compatibilizer are mixed with granules PP/EPDM. PP/EPDM–date fiber composite was compounded using a Twin screw extruder COLLIN (Ebersberg, D-8017, Germany) at 6 Zone Temperature of 160–200 °C with 60 rpm rotor speed and gave out the material from both the spirals and then immersed in cold water for 10 min to prevent the burning possibility of fibers. Then, after being crushed and turned into granules by grinder machine (WIESER-A8992, Germany), the material was dried in an oven at 70 °C for 24 h for further steps in injection molding device; the granules of PP/EPDM/DPF are shown in Figure 2. After that by means of injection molding machine (EM 80, Iran), at the temperature of 165–180 °C and back pressure of 80% and conservator pressure of 50% and injection speed of 80%, the production was done and specimens were made ready for mechanical tests. The specimens were designed and manufactured based on ASTM standards. These standards are usually employed for the determination of properties of reinforced plastics.

The specimens after fabrication were coded. The code is designated as X-YZ, X is the kind of test which is performed on the specimen, i.e. I for impact notched test, T for tensile test, and B for bending test .Y stands for the kind of fibers used as reinforcement, R stands for raw fibers and T is for treated fibers, and Z is for percentage of weight fraction of fibers used in the specimens. Values for Z are 0, 5, 10, 20, and 30. Typical test specimens are shown in Figure 3. X-0 means the specimen made by PP/EPDM.

2.4. Testing procedures and equipments

Tensile, bending, and Charpy impact tests were carried out according to ASTM standards. For each test and the type of composites, five specimens were tested and the average values are reported. Tensile and bending tests were conducted according to the

Figure 2. Granules of PP/EPDM/DPF.

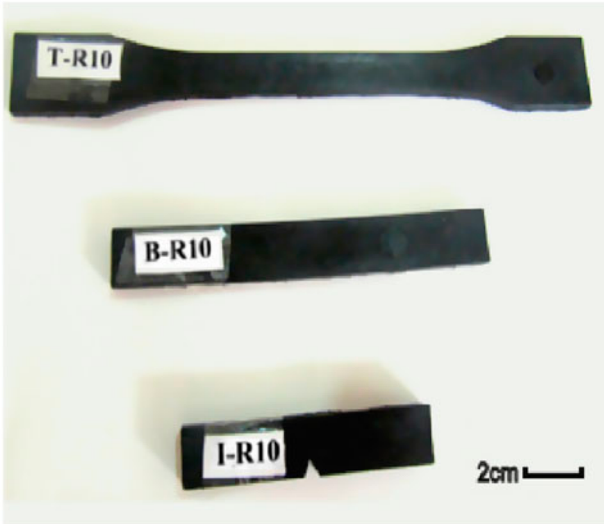

Figure 3. Tensile, bending, and impact specimens (respectively from top to bottom) for raw fiber reinforced composite with 10 wt.% fraction of fibers, according to ASTM standards.

ASTM D638 with a specimen dimension of 165 × 13 × 3 mm, dumbbell shape. The specimen gauge length was 10 cm and ASTM D790 with sample dimension 127 × 12.7 × 3.2 mm, respectively, on universal testing machine (*Hounsfield, H25 K-S, UK*) with a load cell 2.5 kN at a crosshead speed of 5 mm/min and standard notched specimens with 63 × 12.7 × 10 mm were fabricated for Charpy impact testing, according to the ASTM D256 by using pendulum impact instrument, with the capacity of 15 J (TESTER MT220, TERCO). The tests were carried out at room temperature 23 ± 2 °C and relative humidity 40 ± 5%. Fractured surfaces of composite specimens were studied using a scanning electron microscope (SEM) (Vega**,** MV 2300**,** Tescan, Czech Republic), to investigate the morphology and interface between the fibers and the matrix in the composites.

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3. Results and discussion

3.1. Impact test

The impact performance of fiber reinforced composites depends on many factors including the nature of the constituent, fiber/matrix interface, the construction and geometry of the composite and test condition.[32] The values of impact strength of PP/EPDM matrix with different weight fractions of raw and treated fibers for notched specimens are shown in Table 1. It has been found that with blending both kinds of raw and treated fibers to PP/EPDM in notched specimens, impact strength is reduced about 63% by increasing the fiber content from 0 to 30%. These results are obtained when comparing the PP/EPDM with PP/EPDM/30%DPF composite for specimens with raw fibers, in which the impact strength decreased from 42.85 to 15.76 kJ/m^2. Likewise, the same specimens for the treated fibers impact test showed that the impact strength decreases from 42.85 to 17.31 kJ/m^2. On the other hand, the impact strength of samples without DPF is higher than that for corresponding samples with DPF addition, which implies that the incorporation of DPF in order to improve matrix stiffness is always at the expense of loss of ductility. Comparison between the samples made with treated and raw fibers shows that all the samples made with treated fibers have higher impact strength. The variation of the impact strength for different weight fractions of fibers for treated and raw fibers is shown in Figure 4. These results confirm the creation of a good interface between fiber and polymeric matrix in the case of treated fibers.

Table 1. Charpy impact test results for PP/EPDM/DPF composites.

	Sample code								
Properties	T-0	TR-5	TT-5	TR-10	TT-10	TR-20	TT-20	TR-30	TT-30
Impact strength (IS) (kJ/m^2)	42.85	32.28	32.71	25	27.37	19.28	21.32	15.76	17.31
Increased IS with respect to PP/EPDM (T-0) (%)	–	−0.1	5.7	8	20	8.3	15	−7	−9

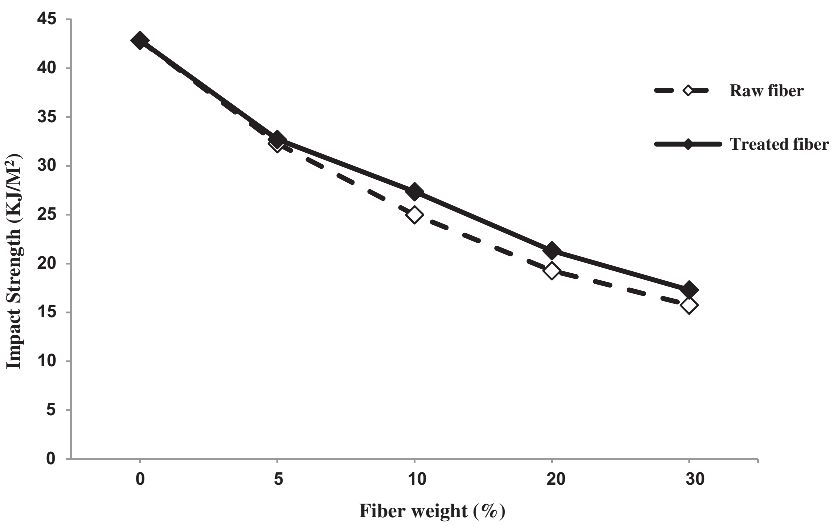

Figure 4. Variation of impact strength at different fiber weight fractions.

Table 2. Tensile test results for PP/EPDM/Date palm fiber composite.

	Sample code								
Properties	T-0	TR-5	TT-5	TR-10	TT-10	TR-20	TT-20	TR-30	TT-30
Tensile strength (TS) (MPa)	15.78	18.65	19.44	19.42	21.20	22.88	25.62	22.94	23.49
Tensile modulus (TM) (GPa)	750.07	894.16	955.50	1059.8	1121.8	1170.3	1356.1	1283.3	1445.9
Elongation at break (%)	26	11	11.5	9	10	6.5	7	5	5.5
Density (g/cm^3)	1.05	1.038	1.064	1.025	1.075	1.013	1.1	1	1.12
Increased TS with respect to PP/EPDM (T-0) (%)	–	18	23	22	34	44	62	45	48
Increased TM with respect to PP/EPDM (T-0) (%)	–	19	27	41	50	56	80	71	92

3.2. Tensile test

Tensile strength, Young's modulus, and elongation at break of composites PP/EPDM/DPF at various weight fractions for both fibers are shown in Table 2. Also, the stress–strain curves for various specimens are shown in Figure 5. Generally, the expected reinforcing effect depends on the interfacial adhesion between both components, i.e. matrix and fiber, which allows an efficient stress transfer from the matrix to the filler.[33] It can be seen in Table 2 that the tensile strength and tensile modulus increased with fiber weight fraction. The fibers, which have higher stiffness than the polymeric matrix, increase the modulus of the composites. This shows that date palm fibers have effectively played its role as reinforcement and rendered its good mechanical properties to the matrix materials.[30]

As shown in Table 3, the tensile strength is increased by 62% due to the effect of adding 20% treated fibers and by 45% due to the effect 30% raw fibers. But, the effect of adding DPF on tensile modulus is higher. Adding 30% treated and raw fibers to PP/EPDM resulted in 92 and 71% increase in the values of tensile modulus, respectively. Again, the tensile strength and Young's modulus of the treated fiber reinforced composites were higher than those of the raw fiber reinforced composites. Treated fiber addition with compatibilizer resulted in favorable tensile strength as depicted in Figure 6. The improve tensile strength for samples with treated fibers can attributed to several factors; better filler dispersion creation of some bonds between PP/EPDM and DPF and by greater wettability of DPF by the PP/EPDM matrix, which improves dispersion and orientation of DPF in the polymer.[34] However, there was a decrement from 20 to 30% fiber volume fraction for treated DPF, which results in inefficient fiber–matrix bonding and a lower stress transfer between the fibers and the matrix.[30] The results of elongation at break show that adding DPF decreases the elongation approximately by 80% when compared to PP/EPDM. Though at similar fiber loading, composites with untreated fiber show lower elongation at break than composites with treated fiber. The incorporation of fillers in polymeric matrix generally leads to a decrease in the elongation at break due to the rigidity of the fibers.[35]

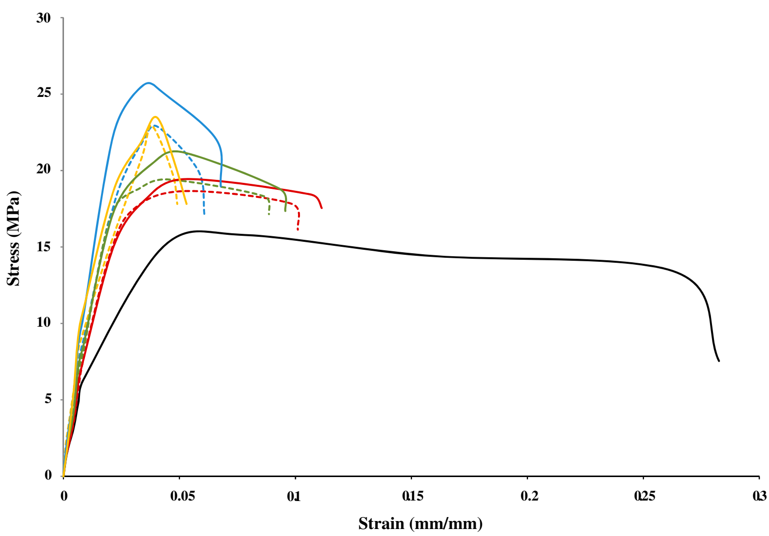

Figure 5. Stress–strain curves for PP/EPDM/DPF.

Table 3. Bending test results for PP/EPDM/Date palm fiber composite.

	Sample code								
Properties	T-0	TR-5	TT-5	TR-10	TT-10	TR-20	TT-20	TR-30	TT-30
Bending strength (BS) (MP)	30	29.74	31.71	32.4	36.25	32.5	34.68	27.87	27.18
Bending modulus (BM) (MPa)	925	1156.2	1387.5	1535.5	2081.2	1618.7	1850	1572	1572.5
Strain at break (%)	Unlimited	21.6	20	14.4	16	12.8	13.6	12.4	12
Increased BS with respect to PP/EPDM (T-0) (%)	–	−0.1*	5.7	8	20	8.3	15	−7	−9
Increased BM with respect to PP/EPDM (T-0) (%)	–	25	50	66	125	75	100	69	70

*Negative sign shows decrease.

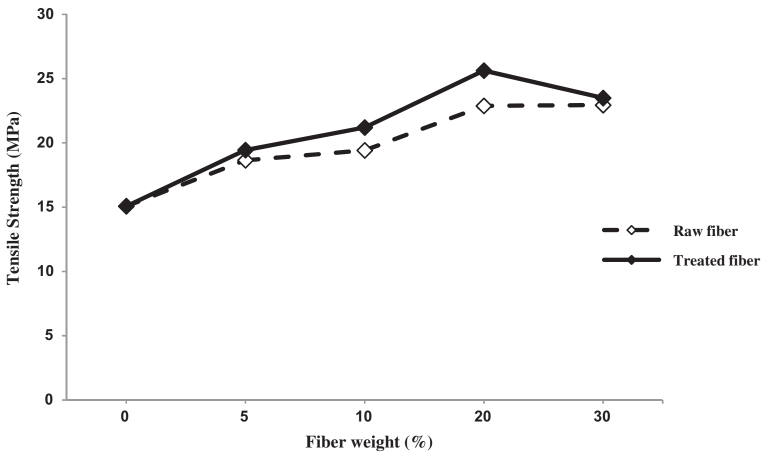

Figure 6. Variation of tensile strength at different fiber weight fractions.

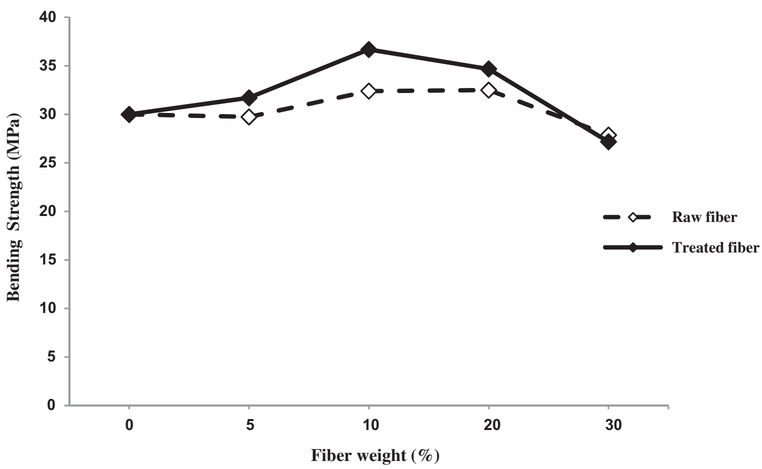

Figure 7. Variation of bending strength at different fiber weight fractions.

3.3. Bending test

The bending strength of PP/EPDM/DPF at various weight fractions of both fibers is shown in Table 3. The bending strength increased with increased in fiber weight fraction, which is related to the effectiveness of the reinforcement as well as is due to the good bonding between matrix and fiber. By adding DPF into the PP/EPDM, the strength values increased from 30 MPa for PP/EPDM composite to 36.25 and 32.4 MPa for PP/EPDM/10% treated and raw fiber composites, respectively. There is a decrement, from 10 to 20% fiber fractions for specimens with treated fibers and from 20 to 30% for specimens with raw fibers. Again, treated fiber reinforced composites had higher bending strength values compared with the raw fiber composites, as shown in Figure 7. Furthermore, the bending modulus with fiber weight fractions increased. The date palm fibers are high modulus material, and higher fiber concentration demands higher stress

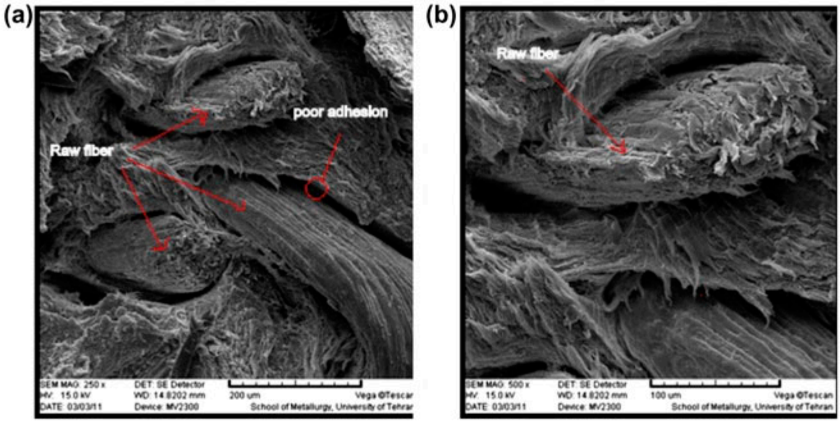

Figure 8. SEM micrographs of fracture surface of PP/EPDM with raw fibers.

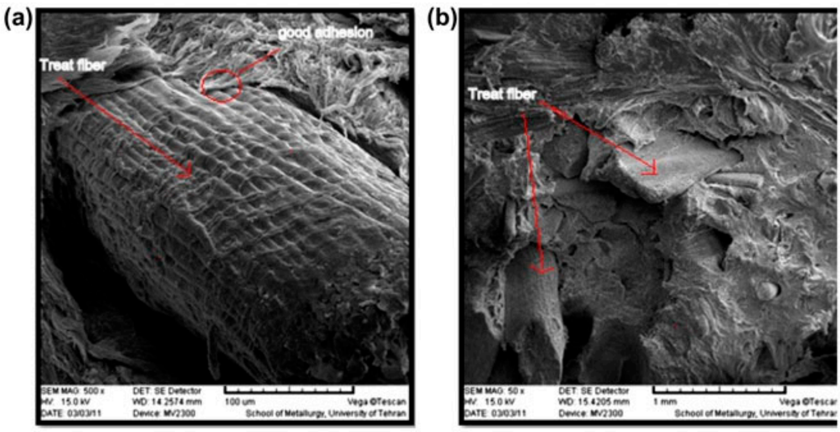

Figure 9. SEM micrographs of fracture surface of PP/EPDM with treated fibers.

for the same deformation. Increased fiber/matrix adhesion provides increased stress transfer between them. However, the increase in DPF addition up the 10 wt.% decreased the fiber/matrix adhesion, resulting in a lower bending strength values for 30% fiber composites.[14,36] But, strain at break has decreased with fiber loading this reduction in strain at break with increasing fiber loading might be due to the decreased deformability of a rigid interface between the fiber and PP/EPDM matrix. The strain at break at B-0 is unlimited (Table 3) because the specimen did not reach to point of break.

3.4. Morphological structures

Fractured surfaces of the specimens were analyzed using SEM. The micrographs of the fractured surfaces of specimens with raw fiber are shown in Figure 8(a) and (b), the surface containing a large number of uncompleted grown fibers and artificial impurities, which may result in poor adhesion between the raw fibers and the matrix. The micrographs of the fracture surfaces of the specimens with treated fibers are shown as Figure 9(a) and (b). This treatment results in an improvement of the fiber surface by removing artificial impurities such as dust and sand, which can contribute to reduced moisture absorption. Treated fiber composites have better adhesion between the two phases and a formation of a certain interface. Certainly, there is evidence of improvement in the interfacial bonding between the fiber and PP/EPDM using MAPP as coupling agent.

4. Conclusions

(1) The results of the present study shown that composite with good strength properties could be developed using date palm fiber.
(2) Raw fibers were chemically treated with sodium hydroxide to increase their compatibility with the PP/EPDM matrix.
(3) Impact properties decrease by adding fibers to PP/EPDM matrix. Again, the same trend can be observed for impact strength where the composites with treated fiber always possess higher strength than the composite with untreated (raw) DPF.
(4) The tensile strength and tensile modulus increased and elongation at break decreased with increasing fiber loading irrespective of fiber type. However, tensile properties of the treated fiber reinforced composites were higher than those of the raw fiber reinforced composites.
(5) Bending tests, by adding DPF to PP/EPDM matrix, the strength, and modulus are increased and % strain at break is decreased. The same trend can be observed for bending strength where the PP/EPDM composites with treated always possess higher strength than the composites with raw DPF.
(6) The SEM images of the DPF reinforce PP/EPDM composites indicate agglomeration of raw and treated fibers in the PP/EPDM matrix. This feature suggests weak interfacial bonding between the raw fiber and matrix. On the other hand, alkali treated DPF/PP/EPDM composites show better dispersion of the fiber into the matrix. This resulted in a better interfacial bonding between the treated fiber and matrix.

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