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Tensile and Farcture Properties of Hybrid Date Palm Fibre Composite Structures Embedded with Chopped Rubber

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ABSTRACT
Composites reinforced with natural fibres have been booming in recent years due to their health benefits and environmental friendliness. Today, date palm is particularly attractive in the Middle East region because it is widely available, cheap, and abundant. Date palm has been used in many simple industries since ancient times, for example, in the manufacture of rope. In the present study, date palms were used to reinforce epoxy resin with embedded rubber segments or crushed to use them for industrial applications. First, date palms are chemically treated in three different ways. The fibres are immersed in three different types of chemical solutions (CH_3COOH, HCl and alkaline NaOH with three different concentrations of 10%, 20% and 50%) at boiling temperature for 1 and 2 hours. These fibres are then mechanically crushed into small fibre crisps. These fibres are mixed with epoxy resin in which 5% by weight of the rubber segments are embedded. The effects of the chemical treatment of the date palm fibres are analysed using electro-scan microstructure examination (SEM). The tensile test is performed on the standard tensile test specimens of this composite to investigate the effectiveness of the reinforcement by the epoxy resin. The fracture strength and crack propagation are investigated by measuring the surface release energy of the individual composite specimens of date palm fibre reinforced epoxy (DPTFRE). Fracture properties are measured using standard compact tensile specimens at room temperature. A drop-weight impact test was also performed. The maximum and minimum values of tensile strength and crack resistance are measured. The results show that HCl treatment leads to good compatibility with date palm fibres. It was found that the rubber segments increase the damage tolerance of such material under impact loading.
Keywords:fracture toughness, date palm tree, impact strength, damage

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I. INTRODUCTION

Composites composed of two or more materials contain two phases: the reinforced phase (fibers, flakes or particles) and the matrix phase (polymer, ceramic or metal) [1]. Natural fibers have been used in several works because they are biodegradable and environmentally friendly [2-5]. Compared to common glass and carbon fibers, natural fibers have a competitive advantage and are given special consideration. In [6-8], the use of date palm fiber (DPTF) reinforced polymer as a composite material was investigated. Abdellah, Alawar et al [9] studied the effects of different treatment techniques on DPTF. Pretreatments included sodium hydroxide in an alkaline solution and treatment with hydrochloric acid (HCl) at various concentrations at boiling temperatures. Mechanical, chemical, and surface morphological effects of the various treatments were determined. The results showed that NaOH gave good and optimum results, while HCl deteriorated the mechanical properties.

Khanam and AlMaadeed [10] investigated how date palm fibers were produced and prepared using mixed recycled polymer components. The palm was not chemically treated before its use. A compatibility device with different percentages was used to determine the adhesion between the fibers and the matrix. It was observed and analyzed the influence of the content of conformity devices on the mechanical, thermal and morphological properties. This improves the tensile strength and stiffness of the material. In addition, thermal stability and water absorption were improved.

The effects of alkaline NaOH treatment on date palm fibers were studied by Oushabi et al [11]. (DPTF). To improve the interfacial bonding with a polymer, 5 wt% NaOH was used for 1 h with different doses of silane. The formation of a silane layer on the alkaline surface of the fiber was demonstrated by a SEM study. For polyurethane and epoxy, the treatment improved the peel strength of the DPTF.
Elbadry [12] studied the effects of two different types of pretreatments on the behavior of DPF. The first step was to wipe the surface with a soft sand cloth. The second was a heat treatment at 100°C for 1.5 hours. The final treatment was a one-hour immersion in a NaOH chemical solution at 100°C. The results showed that all three treatment options improved the results, while their variability was lower than that of the crude DPTF.
Ahmadi et al [13] used date palm stem fibers (DPT) as a composite material to reinforce epoxy resin. The DPT had to be cut into three different lengths and percentages by volume. The composite material was produced by hand molding process. The results showed that increasing the fiber length had no effect on the tensile or flexural properties. Increasing the fiber volume fraction had no effect on increasing the tensile strength, but it improved the flexural strength.

Alsaeed et al [14] measured the interfacial adhesion of DPTF with epoxy resin using the fiber pullout test technique. The DPTFs were treated with an alkaline NaOH solution at concentrations ranging from 0% to 9%. Surface morphology and fiber degradation were investigated using SEM. According to the results, the best DPTF treatment was at 6% concentration and the best embedding length was at 10 mm. Asadzadeh et al [15] used DPTF to reinforce different types of polymers to study their effect on flexural strength. They used five different fiber volume fractions combined with a coupling agent to improve the debondability and interfacial adhesion between the polymers. The results showed that the flexural properties improved while the elongation at break decreased. Al-Suliman et al [16] investigated the mechanical properties, water absorption and machinability of DPTF reinforced polymer composite laminates. They used three different manufacturing processes to produce the composite laminates. The polymer resins used were phenol-formaldehyde resin and a two-component bisphenol resin. They found that both tensile and flexural strength needed to be improved, with the bisphenol resin giving better results. The fatigue behavior of bisphenolic resin needed to be improved. Bisphenolic resin has better water absorption than phenol-formaldehyde resin. The machinability of all composites was improved.

Many research papers [17-19] addressed the fracture toughness of reinforced composite polymers, and most of them studied reinforced synthetic fibers and few natural fibers. Betelie et al [20] studied the fracture toughness of natural fiber reinforced epoxy. They fabricated a typical compact stress specimen by using a composite plate and the hand-forming method. The results showed that the fracture toughness improved with increasing fiber volume fraction. In many studies [21-26], it was found that the use of natural fibers as a reinforcing phase with a polymer matrix is attractive for developing environmentally friendly products.

Objective of the present study
The main objectives of the study are the following:
(a) To study how the addition of rubber to palms treated with various chemical treatments, such as acitic and alkali solutions, affects tensile properties, fracture toughness.
(b) The surface morphology of rubber blended with date palm fibers is studied using SEM microscopy under different chemical solutions and during two immersion periods.
The methodology of the work is as follows: The first section outlines and explains the manufacturing process using the molding technique, and the second section summarizes the standard tensile and fracture tests,the SEM micro examination is displayed in the third section, and the main results are related in the fourth section. Finally, the conclusion that was advised is mentioned.

II. MATERIALS AND METHODS

Date palm tree chemical treatment

The date palm fibers (Figure 1) enclosing the stems were obtained from the city of Qena in Upper Egypt and have the physical and mechanical parameters shown in Tables 1 and 2. First, the fibers were cleaned from dust in a water bath and then dried at room temperature. They were physically separated into new fiber bundles, then wiped and dried at room temperature for 24 hours as shown in Figure (2- a,b). Electrical mixing is used to chop the fibers into small pieces for 15 minutes (Figure 2-c, d).

Then, three different types of solutions are used to chemically treat the chopped fibers. The chemical treatment is used to improve the surface of the natural fibers so that they can be more easily detached from the polymer matrix. The mechanical properties of natural fibers are mainly determined by the interfacial bond between the fiber and the polymer. The fiber is immersed in three concentrations of acetic acid solution HCl, CH_3COOH and alkaline solution of NaOH at boiling temperature (100 oC) for 1 hour and 2 hours, respectively. For all samples, the resulting sheet had an average thickness of about 3.5 mm.

Table 1 Physical properties of the date palm fibers with other natural types [1]

fibre types	Coir	Date palm	Hemp	Sisal
Density (g/cm3)	1.15–1.46	0.9–1.2	1.4–1.5	1.33–1.5
Length (mm)	20–150	20–250	5–55	900
Diameter (µm)	10–460	100–1,000	25–500	8–200
Specific modulus (approx..)	4	7	40	17
Annual world production (103)	100	4,200	214	378
Cost per weight (USD/Kg)	0.3	0.02	1.2	1
Thermal conductivity (W/mK)	0.047	0.083	0.115	0.07

Table 2 Mechanical Properties of the date palm and other natural fibers [1]

Properties	Diameter (mm)	Tensile strength (MPa)	Young's Modulus (GPa)	Elongation at break (%)
Jute	25–200	393–773	13–26.5	1.16–1.5
Flax	10–40	600–2,000	12–85	1–4
Sisal	50–200	468–640	9.4–22.0	3–7
Coir	100–450	131–175	4–6	15–40
Raw date palm fiber	100–1,000	58–203	2–7.5	5–10

Figure 1. Photograph of a) date palm tree b) stems surrounding by fiber c) fibers [1, 2].

Figure 2 Renifircement phase a) Fiber bundles; b) Chopped Fibers; c) Rubber from car tires; d) Cut rubber

Specimen compaction molding

The composite is produced by a method known as compaction pressing. First, the date palm fiber powder is combined with epoxy resin and hardener in a ratio of 1:2%. A thin aluminum foil is placed over the bottom base and wall of the mold frame as a release agent; the steel mold frame is 300 mm long, 20 mm wide and 10 mm deep (see Figure 3-a). The combination paste is then gradually fed and spread until the required thickness is achieved, and the mold is filled with the material. In the second phase, the top of the sample paste is covered with aluminum foil and the mold is closed with a 1.5 mm thick steel block with a very small gap to prevent the mold from leaking. The aluminum foil is to prevent the paste mixture from sticking to the surface. To prevent the epoxy from leaking, a leather cover was placed under the aluminum foil to enclose the entire mold. The combined material mixture is then pressed by a manual hydraulic press with a maximum capacity of 5 tons under a load of 3 tons. After several attempts, the optimum manufacturing conditions were established. The specimen is held under the press for 48 hours to consolidate and cure at room temperature. The specimens eventually take shape and are ready to be cut open for testing. The 279 mm x 18 mm x 3 mm plate that was produced (see Figure 3-b).

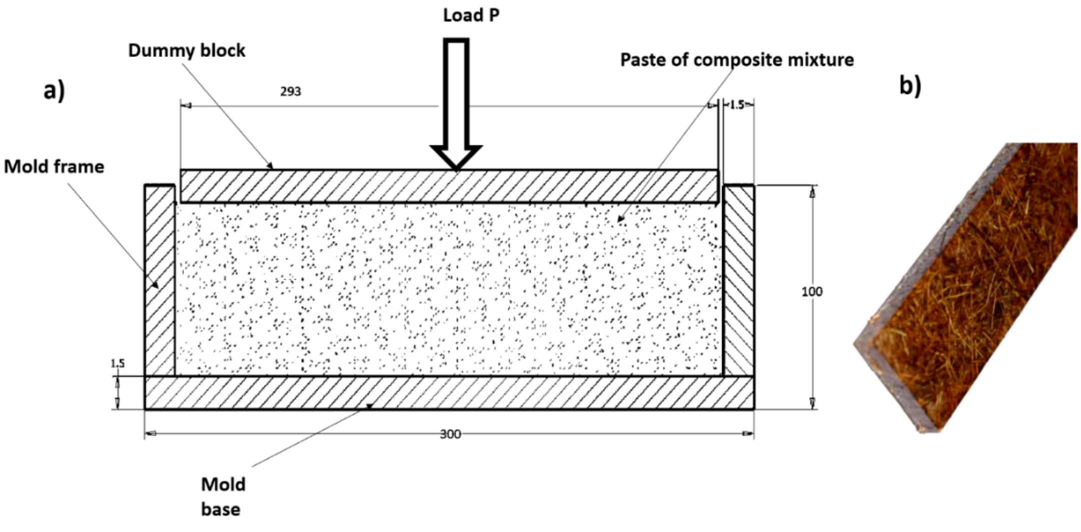


Figure 3 manufacturing set up; a) Compaction molding, b) Date palm tree product

Tensile teste

Standard dog bone tensile specimens (see Figure4) are cut from the previously prepared specimen and processed with a water jet machine. The tensile test is performed using a universal tester according to ASTM D638-14 [28] with a maximum capacity of 20 kN and a crosshead speed of 2 mm/min. When the specimen is tested, the force and strain are measured under computer control. The test is performed until the specimens are completely broken. During the test, the surface damage of the specimen is observed to record any changes. Five specimens are used for each status.

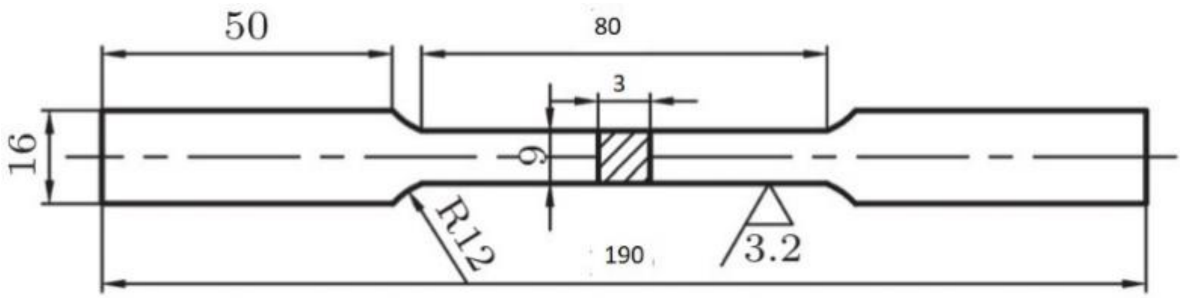


Figure 4 schematic drawing of the tensile test specimen

Compact tension test

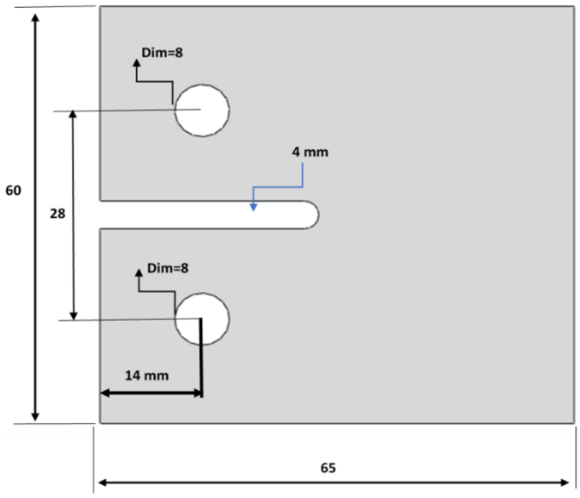
The fracture toughness of DPTF-reinforced epoxy (DPTFRE) is an important factor in material properties. According to ASTM standard E399 [29], fracture toughness test specimens are used. The fracture toughness of DPTFRE is measured with compact tensile specimens as shown in (Figure 5). In order to stop the onset of deterioration, the fracture toughness must be evaluated. Therefore, a compact tensile test is performed to obtain satisfactory fracture toughness data. As shown in Figure 5, the compact tensile from DPTFRE is processed according to the dimensions given in ASTM E399 [29]. An incipient crack must be created at the root of the notch by tapping or sawing with a thin razor blade, and the notch of the initial part must be machined with a cutter or diamond saw [17-19, 30-32]. Loading fixtures are used to load the precracked fracture specimen. The mode I fracture toughness (K_{IC}) values (Mpa.m$^{1/2}$) are calculated using the fracture loads (PQ) obtained from testing five specimens. The critical stress intensity factor for a failure load (PQ) is calculated according to ASTM standard E399 [29] as follows:

$$\mathbf{K_{IC}} = \frac{\mathbf{p_Q}}{\mathbf{h\sqrt{w}}}\mathbf{f(a/w)} \quad (1)$$

$$\mathbf{G_{IC}} = \frac{\mathbf{K_{IC}}^2}{\mathbf{E}} \quad (2)$$

where (h) is the specimen thickness, (w) is the distance from the load line to the specimen's right-hand edge, as shown in Fig. 5, and (a) is the crack length, whose starting value (ao) is likewise shown in Fig. 5. and the shape correction factor is f(aw).

Figure 5 Schematic drawing showing compact tension test geometry



Micrographically study

Microscopic examination was performed using a scanning electron microscope (SEM-EDX Philips). The examination was performed on DPTF before and after chemical treatment. In addition, the cross section of the samples was analysed after tensile and fracture tests to study the fracture and the bond between the fibre and the matrix.

III. RESULTS AND DISCUSSION

SEM micrography of treatment of DPTF

Figure (6-a) is an image taken with an electro-scanning microscope (SEM), showing that the untreated fibers were covered by a thick layer of impurities and a rough surface that weakened the bondability and adhesion with the resin. This layer of impurities, containing dust in the branches of the fibers, plays a dominant role in the fracture of the material, acting like defects in the neutral fiber [27]. These impurities reduce the bonding ability between fibers and polymers. These impurities reduce the compatibility between the fibers and the polymer matrix, which leads to a reduction in the stress transferred between the fiber and the matrix [11]. Chemical treatment of the fibers cleans the fiber surface and removes the wax and dust. It causes the composite fiber bundles to transform into smaller fiber lengths. The smooth surface of the untreated fibers is the reason that the fibers can be easily pulled out from the matrix [15, 22], whereas the rough surface caused by the chemical treatment increases the bond ability with the polymer matrix (see Figures 6 b and d), and this behavior is enhanced in the case of NaOH treatment. The rough surface of the fibers increases with the duration of boiling in the chemical solution for 1 hour. (See Figure 7) NaOH treatment leads to cracks in the fiber, as can be seen in Figure (6-C) and Figure (7-C).

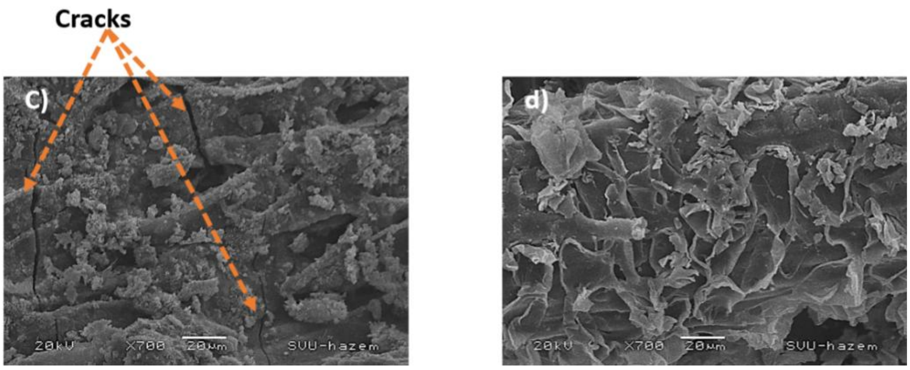


Figure 6 SEM photograph at 1 hr of a) Date palm tree (DPT) without treatment b) With HCl c) with NaOH3 d) with CH_3COOH at 10 %

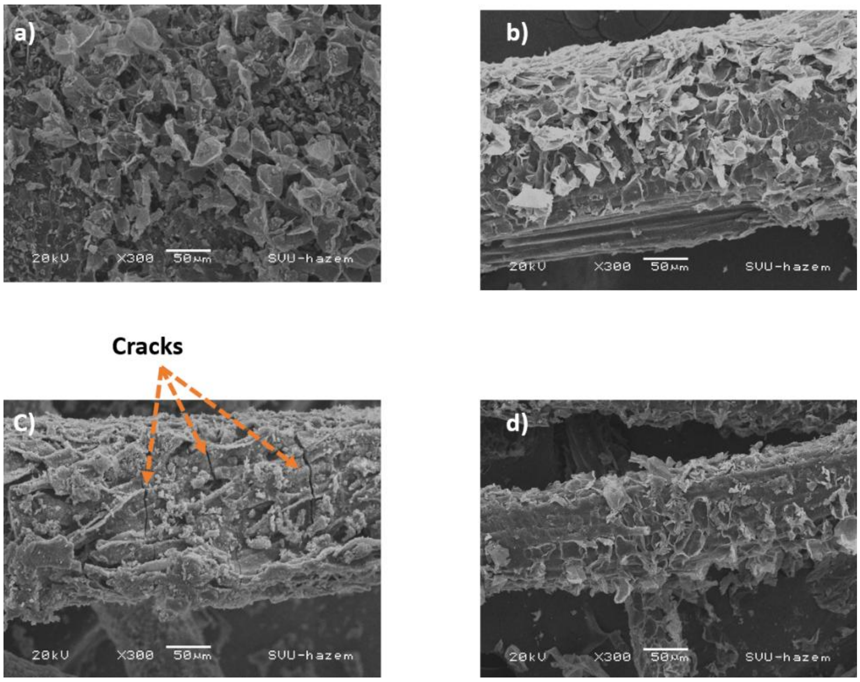


Figure 7 SEM photograph at 2 hr of a) Date palm tree (DPT) without treatment b) With HCl c) with NaOH3 d) with CH_3COOH at 10 %

Tensile test:

It can be observed (Figures8to10) that all treatments performed on date palm improved with crushed rubber treated with acidic media such as HCl and CH_3COOH lead to an increase in strength and elastic modulus with increasing treatment time, while with Na OH the strength decreases, which is due to the fact that with NaOH the fiber breakage and damage occur (Figure 12). As can be seen in Figure (11-a,b), the strength increases linearly with increasing percentage of concentration for HCl, when boiling is done in 2 hours, while the trend under 20% concentration HCl is almost similar after boiling in 1 hour and then decreases. This is due to the fact that increasing the treatment times improves the surface of the fiber, making it more pliable and spongy (see Figure 6,7-b). In Figure 12, it can be seen that when treated with NaOH at a lower concentration of 10% for 1 hour and 2 hours, there are no cracks in the matrix or fiber breaks, while at a higher concentration of 20% for 1 hour and 2 hours, fiber breaks and fiber deformation are observed (Figure 15 c, d). This improvement increases the bonding ability with the resin, therefore, it is observed in SEM micrograph of HCl concentration boiling for 1 hour (Figure 13), the fiber cracking, tearing and breaking can be seen, which means that there is a good adhesion with the matrix, while at 50% HCl the fibers break out of the matrix. The fibers crack, bridge, and break, resulting in good adhesion and good removability. No cracks are observed in the matrix. The acetic acid gives the highest strength at 10% for 1 hour, then it decreases sharply. This could be due to the acid attack on the fiber surface, since CH_3COOH normally increases the surface distortion [27, 36] and pulls out the fibers, damaging the matrix and breaking the rubber (see Figure 14). This is also attributed to the fact that the fibers tend to be densely packed due to the removal of hemicellulose by the acetic acid treatment [34] (see Figure 6,7-d). In addition, at the higher concentration of 50% of CH_3COOH, damage to the matrix with fiber breakage and deformation can be seen (see Figure 15 e, f). The natural fiber is highly polar to NaOH [22, 33], this type of treatment increases the surface roughness, it allows the natural fiber to remove lignin, wax and oils (see Figure15). The NaOH solution attacks the main components of the fiber and more grooves are formed on the surface of the fiber. This further weakens the fiber strength, so that the tensile strength starts to decrease (see Figure 7,8-c and Figure15). All tensile test data with their standard deviation are listed in Table 3. The failure modes are net stress (see Figure15), damage to the matrix and fracture near the fracture surface, fiber breakage, and deformation.

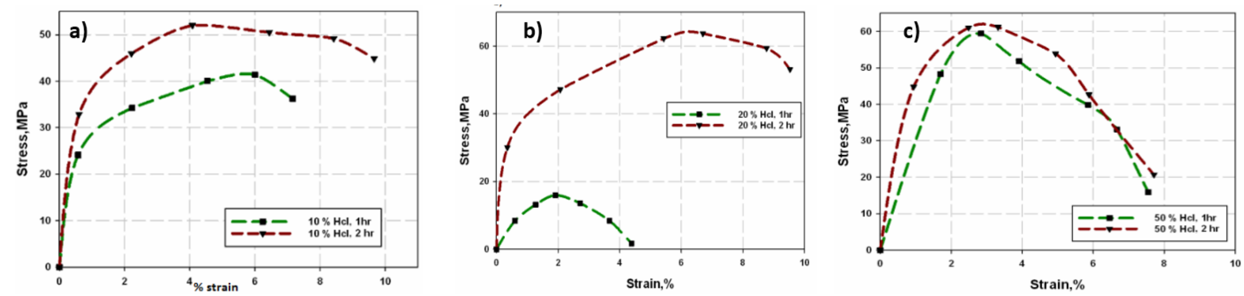


Figure 8 Average Stress strain relation of DPTRE with rubber at HCl treatment in temperature 100Cº for 1hr,2hr at concentration a) 10 % ,b) 20 % ,c) 50 %

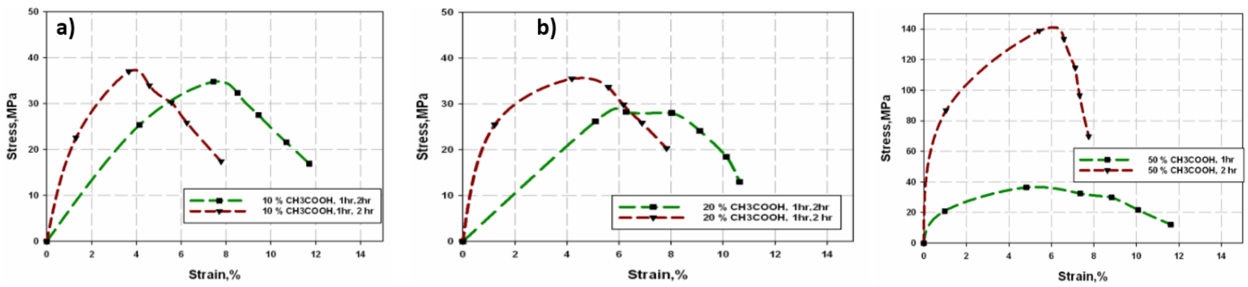


Figure 9 Average Stress strain relation of DPTRE at CH_3COOH treatment in temperature 100Cº for 1hr,2hr at concentration a) 10% ,b) 20 % ,c) 50 %

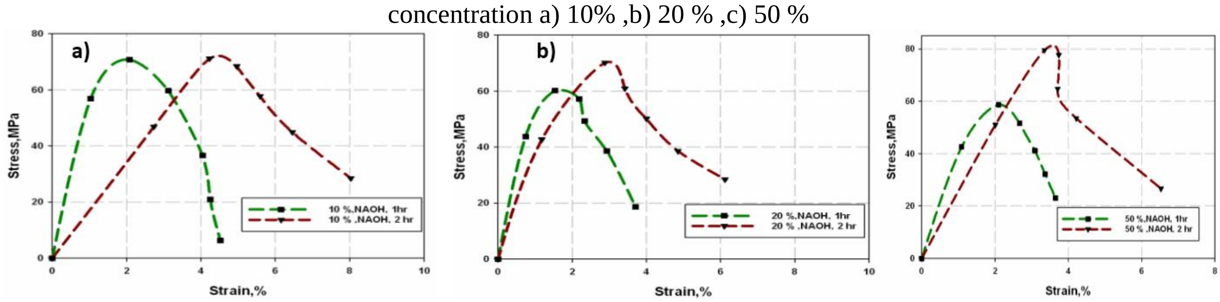


Figure 10 Average Stress strain relation of DPTRE with rubber at NaOH treatment in temperature 100Cº for 1hr,2hr at concentration a) 10% ,b) 20 % , c) 50 %

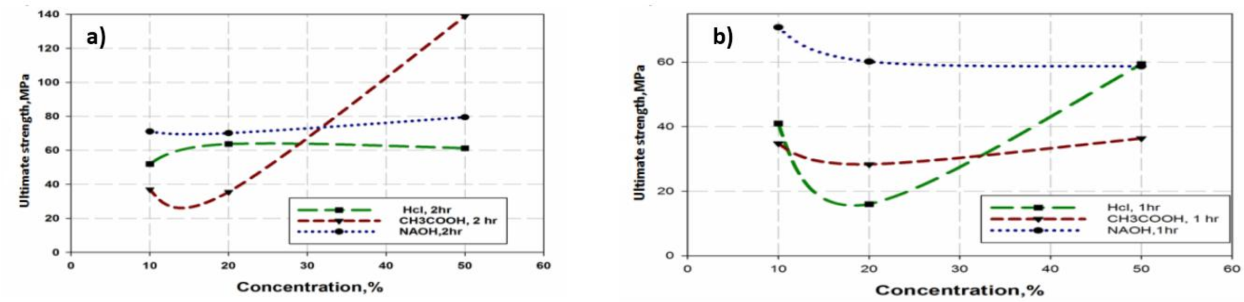


Figure 11 Tensile strength variation with rubber for a) temperature100Cº for1hr, b) temperature100Cº for 2hr .

Table.3. Strength of Date Palm tree fiber reinforced epoxy for specimen with rubber and stander deviation, Elastic Modulus

Specimens with treatments	Average tensile strength (MPa)	S. D	Elastic Modulus,E(GPa)
HCl 10% 1hr	15	3.75	1.6
HCl 10% 2hr	25	6.17	2.4
HCl 20% 1hr	40	9.8	8.35
HCl 20% 2hr	49	11.99	3.075
HCl 50% 1hr	30	7.39	0.8
HCl50% 2hr	119	29.1	12.1

CH_3COOH10% 1hr	202	49.42	31.4
CH_3COOH 10% 2hr	25	6.17	3.9
CH_3COOH 20% 1hr	45	11.01	2.5
CH_3COOH 20% 2hr	120	29.34	9.01
CH_3COOH 50% 1hr	35	8.58	5.7
CH_3COOH 50% 2hr	39	9.55	1.7
Na OH 10% 1hr	126	30.81	16
Na OH 10% 2hr	49	11.99	1.5
Na OH 20% 1hr	110	26.9	10.7
Na OH 20% 2hr	79	19.31	5.17
Na OH 50%1 hr	78.5	19.19	15.6
Na OH 50%2 hr	74.9	18.31	6.34

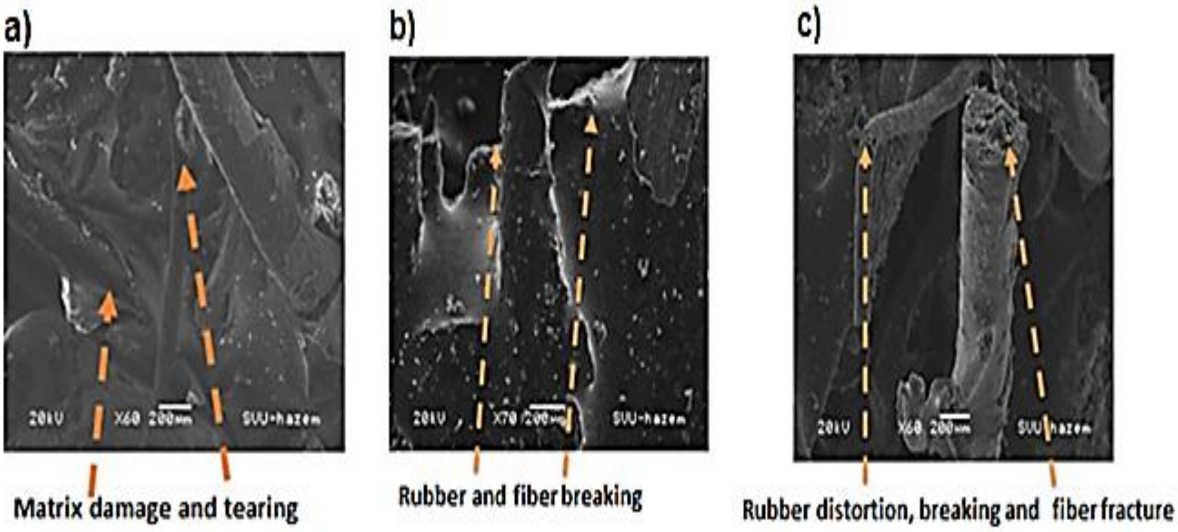


Figure 12 SEM photograph date palm tree with rubber and treatment at 100Cº with a) NaOH,10% .b)with NaOH,20%,at 1hr. c) with NaOH,50%,at 1hr.

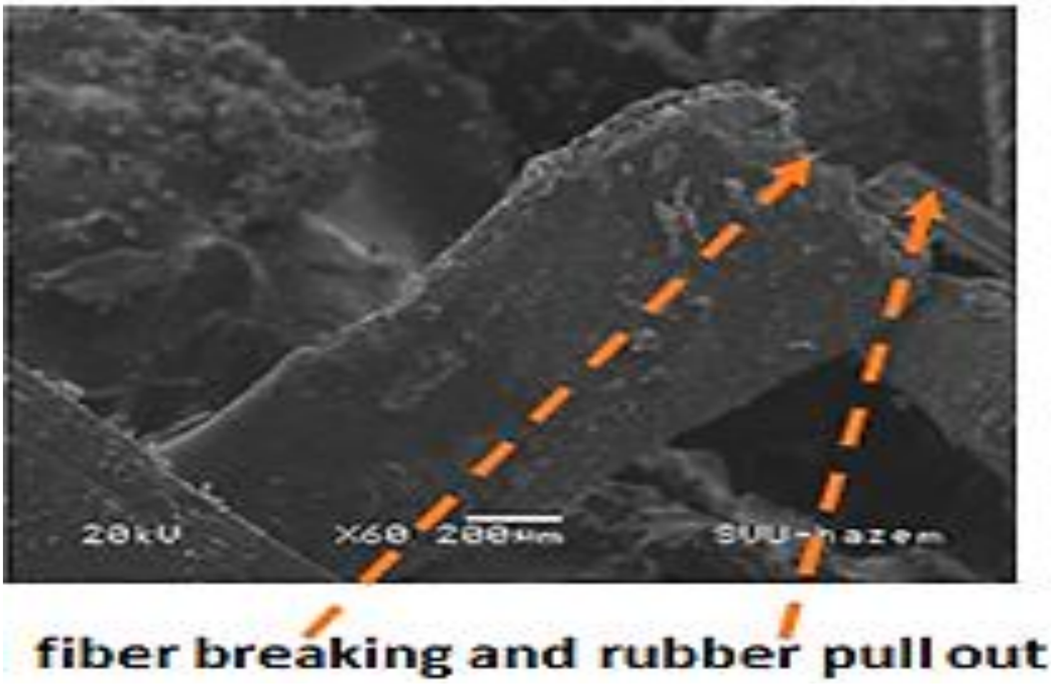


Figure 13 SEM photograph date palm tree with rubber and treatment at 100Cº with HCl,50%,for2hr.

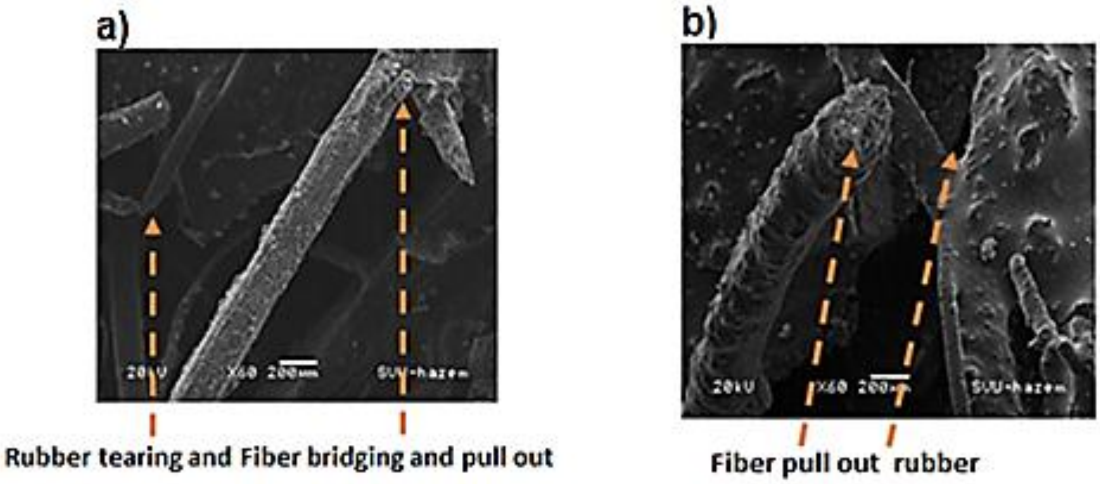


Figure14 SEM photograph date palm tree with rubber and treatment at 100Cº with a) CH_3COOH,10%for1hr. b) CH_3COOH,20%,for 2hr.

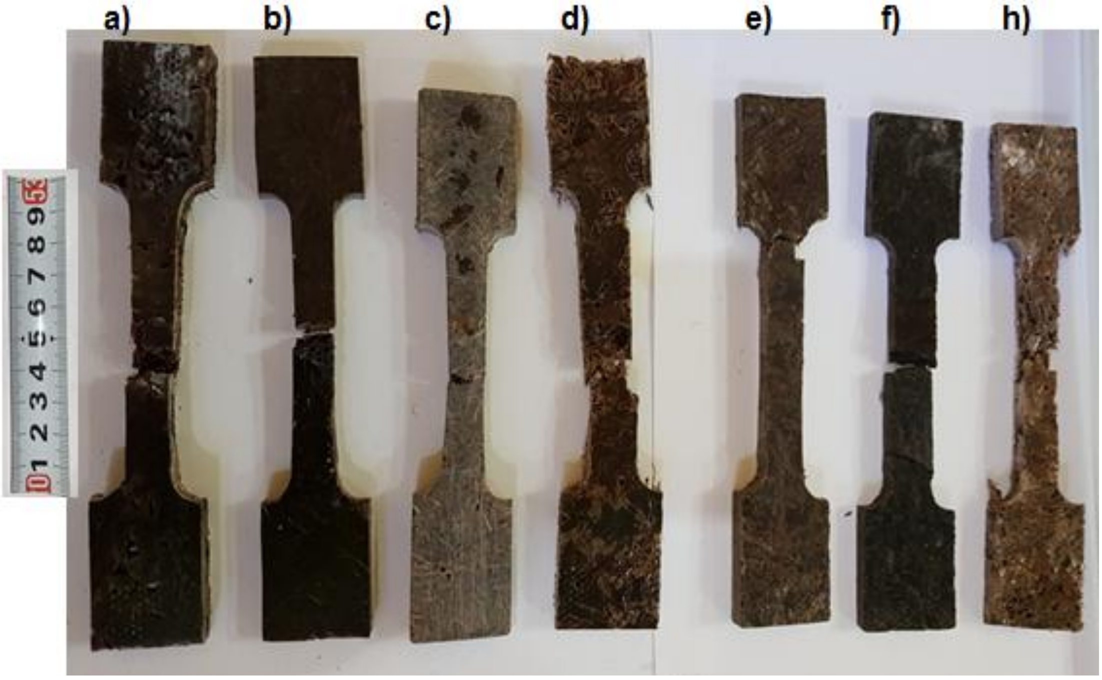


Figure 15 Failure Modes of some samples of DPT with rubber of tensile specimens (a,b) HCl,(c,d,e) NaOH, (f,h) CH_3COOH

Fracture toughness:

Figures 16, 17 and 18 show the load-displacement curve for treatable DPT compact tensile specimens. It is clear that the chemical treatment improved the crack resistance and ductility of the DPT composites. The yield curve shows an increase in softening with nearly constant stability, which could be due to the increase in bridging fibres at the crack front (see Figure5). The two-period treatment gives almost identical results, but the failure length changes approximately as listed in Table 4. The 5% truncation of the slope gives the value of the maximum load PQ at which the crack begins to propagate. This value is then used in Equation 1, with reference to the failure or critical failure length listed in Table 4. The mode I fracture toughness K_{IC} is measured, and by applying Equation 2, the surface release energy is measured and listed in Table 4. Robert O. Ritchie [35] concluded that for ductile (strain-controlled) fracture, e.g., due to coalescence of micropores (as in the present case), the simple models indicate that the mode I fracture toughness K_{IC} scales with the square root of the yield strength multiplied by the elastic modulus, ductility, and a microstructural length scale (e.g., a multiple of the particle spacing).

Table .4 Fracture surface release energy with different treatment for specimen with rubber

Specimens with treatments	Average surface release energy G_{IC}, (kJ/m²)	S. D
HCl 10% 1hr	4.051	1.141
HCl 10% 2hr	9.762	1.389
HCl 20% 1hr	4.253	1.548
HCl 20% 2hr	2.862	1.518
HCl 50% 1hr	6.047	2.578
HCl 50% 2hr	1.864	2.356
CH3COOH 10% 1hr	5.902	3.585
CH3COOH 10% 2hr	9.731	2.692
CH3COOH 20% 1hr	2.316	3.198
CH3COOH 20% 2hr	2.342	4.345
CH3COOH 50% 1hr	9.463	4.425
CH3COOH 50% 2hr	3.351	2.086
NaOH 10% 1hr	4.514	2.142
NaOH 10% 2hr	1.985	2.967
NaOH 20% 1hr	3.189	1.056
NaOH 20% 2hr	2.063	2.354
NaOH 50%1 hr	1.974	2.804
NaOH 50%2 hr	2.347	1.098

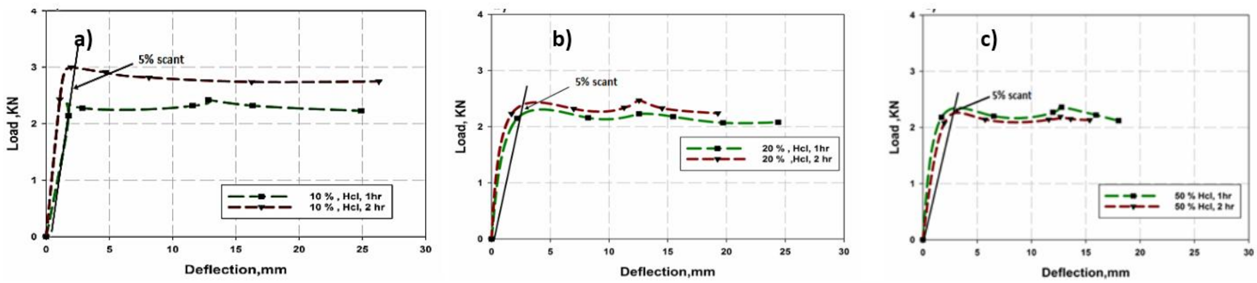


Figure 16. Load- displacement relation in compact tension test of DPTRE with rubber at HCl treatment in temperature 100Cºfor 1hr,2hr at concentration a) 10 %, b) 20 %, c)50%.

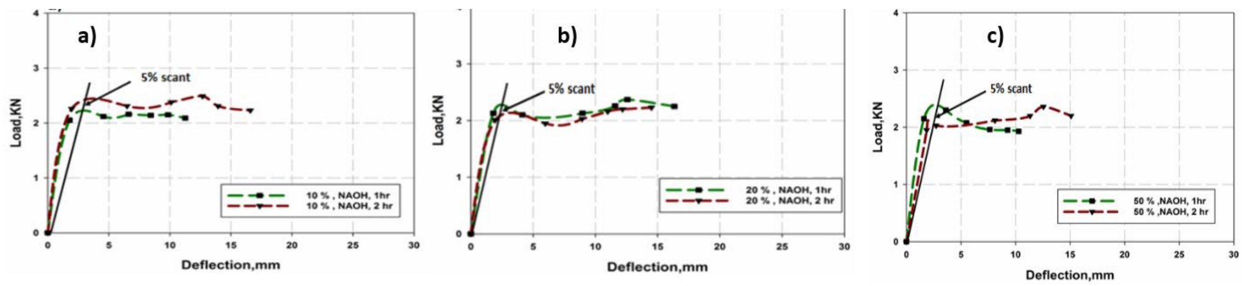


Figure17. Load- displacement relation in compact tension test of DPTRE with rubber at NaOH treatment in temperature 100Cºfor 1hr,2hr at concentration a) 10 %, b) 20 %, c)50%.

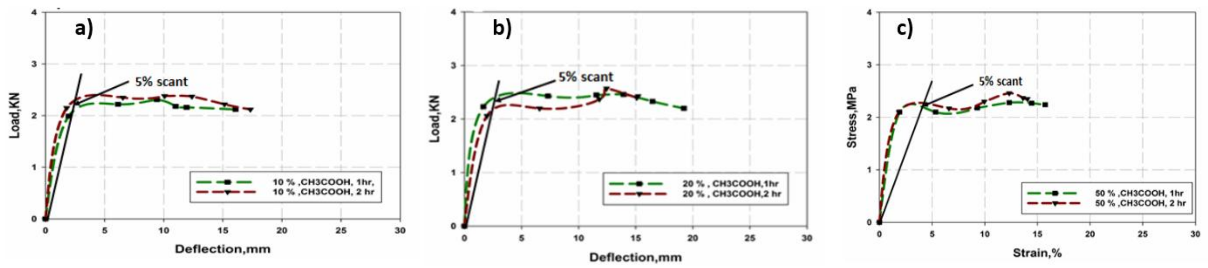


Figure 18. Load- displacement relation in compact tension test of DPTRE with rubber at CH_3COOH treatment in temperature 100Cºfor 1hr,2hr at concentration; a) 10 %, b) 20 %, c)50%.

IV. CONCLUSIONS

Date palm fibres (DPTF) are used as a natural reinforcing material in the composite industry and are therefore attractive. The chemical treatment programme presented in the present work leads to a good improvement of epoxy resins reinforced with date palm fibres by increasing their removability with the epoxy resin. The tensile properties of a composite reinforced with DPTFs chemically treated with HCl show better results and are more compatible with the polymer matrix at all concentrations and time periods, although they have quite low strength, while with other NaOH and CH_3COOH the fibres break and therefore the strength decreases sharply. The same results were obtained for the fracture toughness, which is related to the tensile strength of the composite. The rubber segments increase the damage tolerance of the composite under impact loads.

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