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The potential of using date palm fibres as reinforcement for polymeric composites

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ABSTRACT

Interfacial adhesion of natural fibres as reinforcement for fibre polymeric composites is the key parameter in designing composites. In the current study, interfacial adhesion of date palm fibre with epoxy matrix is experimentally investigated using single fibre pull out technique. The influence of NaOH treatment concentrations (0–9%), fibre embedded length and fibre diameter on the interfacial adhesion property was considered in this study. Scanning Electron Microscopy (SEM) was used to observe the surface morphology and damage feature on the fibre and bonding area before and after conducting the experiments. The results revealed that 6% concentration of NaOH is the optimum solution for treating the date palm fibre to maintain high interfacial adhesion and strength with epoxy matrix. The embedded length of the fibre controlled the interfacial adhesion property, where 10 mm embedded length was the optimum fibre length.

1. Introduction

In recent years, natural fibres have drawn considerable attention as substitutes to the synthetic fibres such as glass and carbon fibres. Natural fibres applications have been growing in many sectors such as automobiles, furniture, packing and construction industries parts where a high load carrying capacity is not required [1–6]. The attractive features of natural fibres over the traditional counterparts include relatively low cost, low weight, high specific modulus, less hazards, abundant and renewable resources [1,2,6–9]. In [5], it has been reported that low weight (20–30 wt.%) and high volume of natural fibres compared to synthetic fibres in polymeric composites have improved the fuel efficiency and reduced emissions in the automobile industries. Natural fibres can be disposed at the end of their life cycle by composting or by recovery of their calorific value in a furnace, which is not possible with synthetic fibres [1,5,10]. In contrast, the natural fibres also exhibit some undesirable characteristics which are the high moisture absorption and highly anisotropic properties [10–12]. Many studies on mechanical properties of natural fibres and how these incorporate with various thermosets (epoxy, polyester, and phenol formaldehyde) and thermoplastics (PE, PS and PEEK) have been attempted [1,4,8,9,13–15]. All these studies emphasized that the properties of composites depend on the natural fibres' interfacial bonding with the synthetic matrices. In other words, the adhesion between the reinforcing fibres and the matrix plays an important role in the final mechanical properties of the materials since the stress transfer between matrix and fibres determines reinforcement efficiency. However, because of cellulose, hemicelluloses, pectins and lignin richness in natural fibres, natural fibres tend to be strong polar and hydrophilic materials whilst polymer materials are a polar and exhibit significant hydrophobicity [2,4,8,10,16]. In other words, there are significant problems of compatibility between the fibre and the matrix due to weak interface. In general, poor interfacial interaction leads to internal strains, porosity, environmental degradation, moisture absorption, poor mechanical properties of composite parts, and de-bonding over time. Therefore, surface modification of the natural fibres by means treatment is one of the largest areas of current research to improve compatibility and interfacial bond strength. It is worth to mention that the chemical treatment of the fibres can either increase or decrease the strength of the fibres, and hence good understanding of what occurs structurally is required [1–4,6–10,16].

Chemical treatments as bleaching, acetylation and alkali treatment aim to improve the fibre/matrix adhesion by increasing the surface roughness of the natural fibres through of clean the fibre surface from impurities and disrupting the moisture absorption process through of coat of OH groups in fibre [1,3,6,8–10,16]. Alawar et al. [2] investigated the effects of chemical treatments on surface morphology, and mechanical properties of date palm fibre (DPF) considering two kind of treatments in deferent concentrations (NaOH 0.5–5% and HCl 0.3–1.6 N). The results of that work revealed that HCl had high reduction in the tensile and huge distortions on surface due to the acid attack on the surface of the fibre. However, NaOH treatment enhanced the surface morphology of the fibre and increase the number of grooves on the surface. In [16], the effect of alkali treatment (NaOH) under conditions (ambient temperatures, elevated temperatures and high pressure steaming treatment) on the tensile strength of jute fibres has been

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studied. The treatment resulted in rough jute fibre surface and better separation by removing the surface impurities, non-cellulosic materials, inorganic substance and wax. Also the range representing O–H stretching of hydrogen bond became less intense upon alkali treatment. The optimum enhancing of tensile strength and elongation at break were 65% and 38%, respectively for alkali-steam treatment compared to the untreated fibres. In very recent work [6], the influence of alkaline treatment on the flexural properties of polyester matrix composite reinforced with Alfa fibres (random orientation and 40 wt.%) has been reported. The alkali treatment, NaOH, at 1%, 5%, and 10% concentrations for various socking periods of 0, 24 and 48 h has been conducted. The flexural strength and flexural modulus improved by 60% and 62% respectively at 10% NaOH consideration for a period of 24 h .The longer treatment time of Alfa fibre (48 h with 5% NaOH) decreased the flexural strength and flexural modulus due excess delignification natural fibre that led to weakening of the fibre strength and the damage occurred on the fibre.

The date palm tree, a member of the palm tree family (*Phoenix dactylifera*), is normally found in the Middle East, Northern Africa, the Canary Islands, Pakistan, India, and in the United States (California). The palm tree stem is covered with a mesh made of single fibres. Usually, these fibres create a natural woven mat of crossed fibres of different diameters. The possibility of finding use for date palm fibres (DPF) in fibre composite will open a new market for what normally considered waste or used in low value products. The current study aims to investigate the effect of alkali treatment of date palm fibre on its interfacial adhesion with epoxy matrix.

2. Preparation of the materials

The used resin in this study was epoxy resin (R246TX) which was supplied by Australian calibrating services PTY. Ltd., Australia. Date palm fibres were obtained from a date palm tree in a farm in Kuwait. All fibres were obtained from the outer layer of the tree stem. Only fibres from the same bunch were used to ensure the consistency. The fibres were carefully extracted from the stem manually. Assuming that these fibres are of uniform cross-sectional geometry, The diameters and the shape of the fibres were selected using a Motic stereomicroscope (SMZ168 series). Three measurements were taken on each fibre.

2.1. Alkali treatment

In the current work, selected date palm fibres were washed with tap water to remove all of the dirt and then left to dry at room temperature for 24 h. The cleaned fibres were cut into an average length of 100 mm. Three different NaOH concentrations were prepared as 3, 6, and 9 wt.%. The selected fibres were immersed in NaOH aqueous solution for 24 h at room temperature. After treatment, the fibres were washed with tap water and then dried for 24 h at room temperature.

2.2. Samples preparation

To fabricate the single fibre pull out (SFPO) specimens, both ends of the selected fibres (treated with 3, 6, and 9 wt.% NaOH) were adhered to two pieces of rubber to hold the ends of the fibres and also to prevent the resin from leaking during curing process. All the specimens were fixed to a gage length of 10 mm. Gage length was set to be short because longer fibres tend to have a higher possibility of flaws and hence affects the consistency of results. Fig. 1a shows the SFPO sample preparation before pouring the resin mixture into the metal mould (90 mm $\times$ 10 mm $\times$ 10 mm). Before proceeding to the specimens' fabrication, a non-stick paper was placed into the mould to prevent the mixture from sticking and to easily remove it after curing process. The epoxy resin was mixed with hardener in a ratio of 1:4. The mixture were left for about 5–10 min and then poured carefully into the mould to avoid generating the bubbles in the sample. After that, the mixture was poured into the mould and left to be cured at room temperature for 24 h. Later, the sample was removed from the mould as seen in Fig. 1b. Finally, the composite was cut to the desired fibres embedment lengths of 5, 10, 15, and 20 mm using a hand saw.

2.3. Experimental procedure

For the single fibre pull out tests, the specimen was clamped tightly on Hounsfield tensometer (250–2500 N) system. The crosshead speed was set to 1 mm/min. Three test replicates were performed for each concentration of treatment (3–9 wt.% NaOH) and the average values were determined. The single fibre pull out tests were performed based on the ASTM STP 452 [17]. The morphology of the fibres and matrix surfaces was studied under scanning electron microscope. All specimens were gold sputtered before the examination.

3. Results and discussion

3.1. Effect of NaOH concentration on the surface characteristics of the fibres

The micrographs of the prepared fibres are shown in Fig. 2a–d. Fig. 2a represents the micrograph of the untreated date palm fibre. From the figure, one can see that there is a layer covered the fibres (waxy layer). This layer may worsen the interfacial adhesion of the fibre with the matrix. Therefore, it is recommended to remove it via chemical treatment such as NaOH. Since there are different findings regarding the effect of NaOH treatments on the surface characteristics of the natural fibres, the fibres were treated with 3–9% concentrations of NaOH. From Fig. 2b and c, it is clear that the NaOH treatment removes some of the waxy layer depending on the NaOH concentration. In Fig. 2b (3% NaOH), a slight removal of the outer layer and appearance of the inner fibre can be noticed. At higher NaOH concentration of 6% NaOH, complete appearance of the inner fibres and exposure of the holes are evidence. A rough surface of the fibre is appeared which could assist in enhancing the interfacial adhesion of the fibre with the matrix. At very high NaOH concentration of 9% (Fig. 2d), complete removal of the outer layer and appearance of the inner fibres can be noticed. From the above, it can be said that increase the NaOH concentration boosts the removal of the waxy layer covered the natural fibre.

The effect of the NaOH treatment result is in agreement with the reported works which have been published on hemp [18,19], jute [20], oil palm [21], coir [22], betalnut [23] and kenaf [24]. It has been found that NaOH was the most effective method in treating the hemp fibres and enhancing the interfacial adhesion properties. The NaOH treatment acts on the middle lamella and on the primary cell wall resulting in removing the hemicelluloses and dissolving pectins. On the other hand, bleaching (pure, and followed by acetylation or alkalisation), pure acetylation, sodium sulphite and sulphuric acid with potassium permanganate have been used to treated Okar fibres [25]. In that work, all the treatments tends to expose the lumens and cleaning process occurred during the treatment. Acetylated and bleached smoothened the surface of the fibre due to acetylation. Permanganate treated fibres showed small holes around the mid section of the fibrils. Acid solution attacked the fibre and destroyed the main structure of the fibre. Similar result of acid solution treatment have been reported on hemp in Hemp [19].

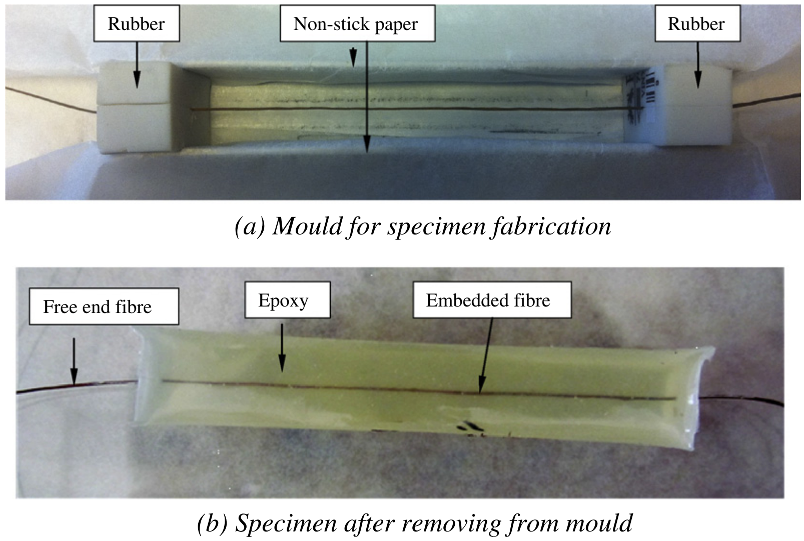

Fig. 1. Images showing the procedure of fixing the single fibre in the mold and then sample after preparation.

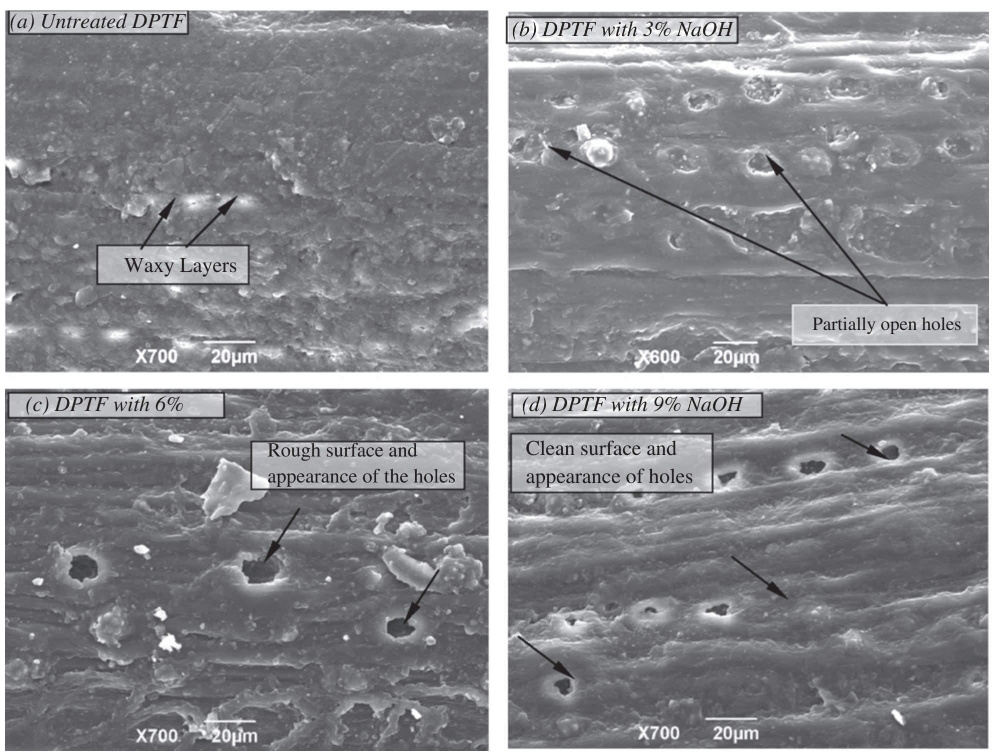

Fig. 2. Micrographs of the untreated and treated date palm fibres surface with different NaOH concentrations.

3.2. Tensile strength of single fibre

The tensile testing data of the date palm fibre treated with different NaOH concentrations (3, 6, and 9 wt.%) are presented in Fig. 3a–c. Each test was repeated three times and the results are displayed as strength against strain. In general, the tensile behaviour of the fibres exhibit ductile behaviour for the all treated fibres, i.e. elastic and elastic regions can be noticed in all the curves presented. However, the higher concentration of NaOH (9%) reduces the strain by about 4% and 15% compared to the 3% and the 6% NaOH. The highest tensile strength (400 MPa) is exhibited when the fibre was treated with low NaOH concentration (3%), Fig. 3a. The lowest tensile strength, of about 110 MPa, is evidence with the treated fibre with high concentration of NaOH (9%), Fig. 3c. In summary, one can say that the increase in NaOH concentration worsens the tensile properties of the date palm fibre. On the other hand, it was noticed that in Fig. 2 the higher concentration of NaOH enhances the surface characteristics of the fibre by removing the waxy layer from the surface. Therefore, there is an optimum NaOH concentration in which good interfacial adhesion can be proposed. For the current study, it can propose that 6% NaOH is the optimum concentration which provides acceptable fibre strength and surface characteristics. This can be further discussed with the single fibre pull out test in the following section.

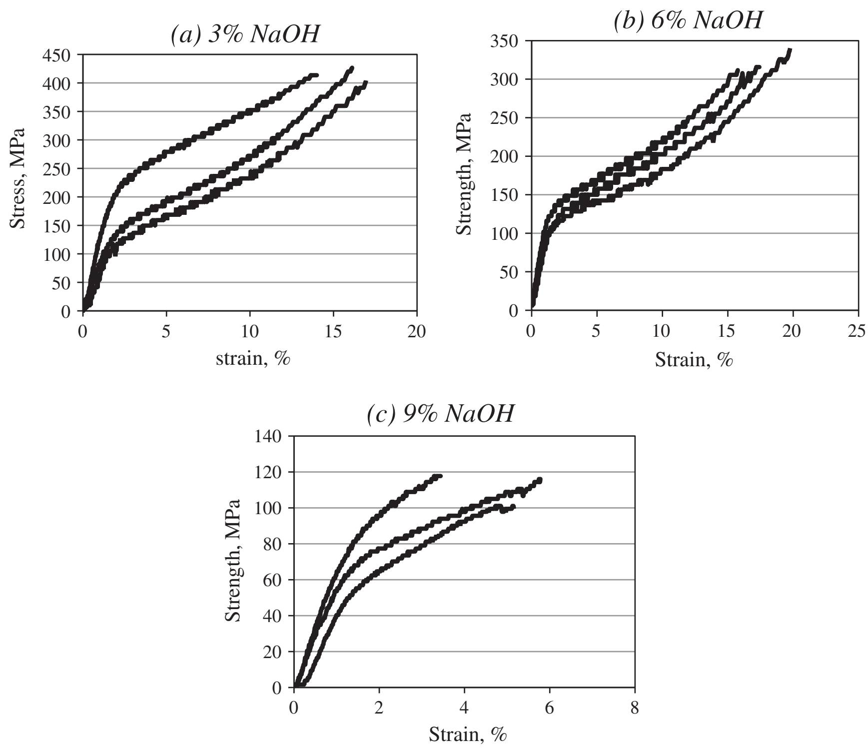

Fig. 3. Tensile testing data of single fibre treated with different NaOH concentrations.

The single fibre pull out data are displayed as maximum stress and shear against NaOH concentrations of 3%, 6% and 9% with four different impeded lengths of 5 mm, 10 mm, 15 mm and 20 mm as shown in Fig. 4a and b. From Fig. 4a, one can see that NaOH concentrations and the fibre embedded length influence the stress subjected on the fibre. There is an increase in the stress when the embedded length increases up to 15 mm especially at NaOH concentrations of 3% and 6%. The longer imbedded length (20 mm) shows reduction in the stress at all NaOH concentrations. This is due to the differences in the tensile strength of the fibre and/or the interfacial adhesion property of the fibre with the matrix, i.e. the ability of the fibre to carry the load and transfer it to the matrix. In term of the fibre strength, it was mentioned in the previous section that the higher NaOH concentration (9%) worsened the tensile strength of the fibre. It seems that the low stress subjected on the fibre during the pull out test is due to the breakage of the fibres rather than pull out. For the low stress generated on the fibre treated with 3% NaOH treatment, it seems the interfacial adhesion is the most pronounced factor that affects the value of the stress during the pull out testing.

The interfacial adhesion of the fibre with matrix can be determined by measuring the shear stress generated in the bonding area (between the fibre and the matrix). Fig. 4b displays the maximum shear stress in the interfacial area of the fibre with the matrix at different embedded lengths and NaOH concentrations. At low NaOH concentration value of 3%, the shear stress in the bonding area is relatively low compared to other concentrations. This could be due to the outer waxy layer surrounded the fibres which lower the interaction between the asperities of both epoxy and fibre surfaces. This could lead to pull out process during the pull out testing. Further explanation will be given in the SEM observation section. On the other hand, Fig. 4b shows that the shear in the interfacial area is high when the 6% NaOH concentration was used in the fibre treatment. In other words, there is high resistance to the pull out process during the loading in spite of the high tensile strength of the fibre at this concentration (Fig. 3b). At high NaOH concentration of 9%, it seems

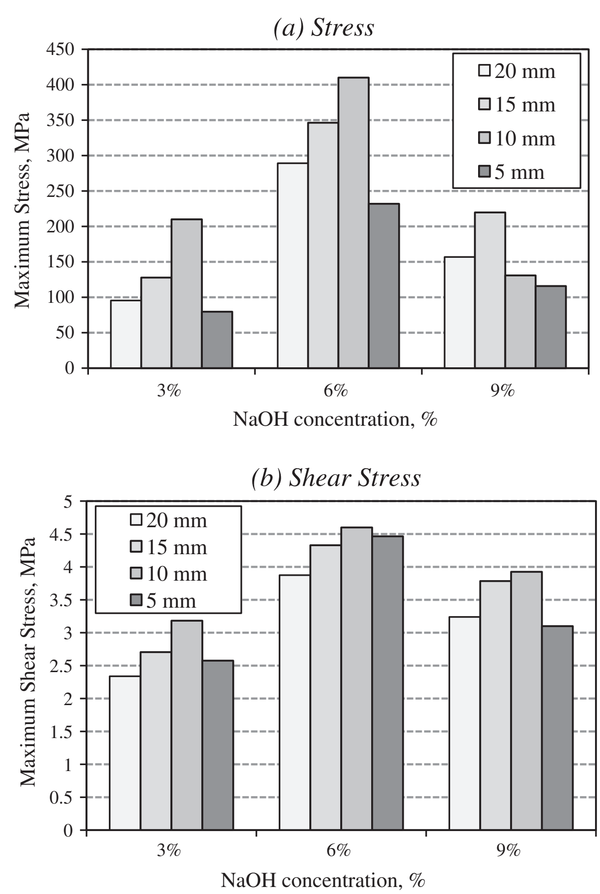

Fig. 4. Single fibre pull out data showing the maximum stress and shear stress of the treated fibre with different NaOH concentrations.

there is a reduction in the value of the shear in the bonding area. This is not due to the poor interfacial adhesion of the fibre but it is due to the weakness of the fibres which occurred due to the high concentration of the NaOH as was evidence in Fig. 3c.

To clarify the above argue, schematic drawing showing the three treated fibre behaviours under pull out loading conditions is displayed in Fig. 5a–c. In the case of the treated fibre with 3% NaOH, there is an outer layer covers the fibres. When the load applies, the outer layer adhered onto the epoxy and detachment between the inner fibres and the outer layer took place. At this stage, pull out process occurs, Fig. 5a. On the other hand, the outer layer is removed when the fibre was treated with 6% NaOH which in turn leads to high interaction and adhesion of the inner fibres with the epoxy. Moreover, during the curing process, some of the epoxy resin can inter inside the fibre which generates interlocking region between the fibre surface and the epoxy matrix. Such behaviour prevents pull out of the fibres which agrees with the high stress and shear results given in Fig. 4 in the case of 6% NaOH concentration, Fig. 5b. In the case of 9% NaOH treatment, the outer layer was removed, however, the fibre was weakened due to the high concentration of the NaOH in the treatment process. It is suggested that there will be no pull out process, but early breakage to the fibre will occur, Fig. 5c.

3.3. SEM observation and discussion of the experimental results

To support the findings and the proposed mechanism, the tested samples were scanned using scanning electron microscopy. The micrographs of the tested samples are given in Fig. 6 for different NaOH concentrations. For the 3% NaOH treatment, the micrographs shown in Fig. 6a and b obviously indicate pull out mechanism took place during the testing and the outer layer of the fibre seems to be adhered onto the epoxy (c.f. Fig. 6b). This supports the thought given in Fig. 5a and in high agreement with the experimental results introduced in Fig. 4a, i.e. low shear and stress of fibres at low NaOH concentration of 3%.

When the fibre was treated with the 6% NaOH, there is no evidence of fibre pull out mechanism. However, tearing and breakage of fibres can be noticed indicating the high interfacial adhesion of the fibre with the matrix at this treatment condition, Fig. 6c. From the magnified micrograph (Fig. 6d), one can say that the bonding between the treated date palm fibre with the 6% NaOH and the epoxy region is strong.

For the 9% NaOH treatment, it was not easy to examine the surface of the samples due to the deep allocation of the fibre breakage. Therefore, the sample was broken to observe the embedded part of the fibre. Fig. 6e shows the micrograph of the embedded fibre (treated with 9% NaOH) indicating that the bonding of the fibre with the matrix is very strong. However, there is a clear damage to the fibres, i.e. weakening process took place on the fibre due to the high concentration of NaOH during the treatment process. Those micrographs support the experimental results (Fig. 4) and the thoughts in Fig. 5.To correlate the experimental finding with the theoretical ones, the critical length of fibres is determined based on

$$L_c = \frac{\sigma d}{\tau_c} \quad (1)$$

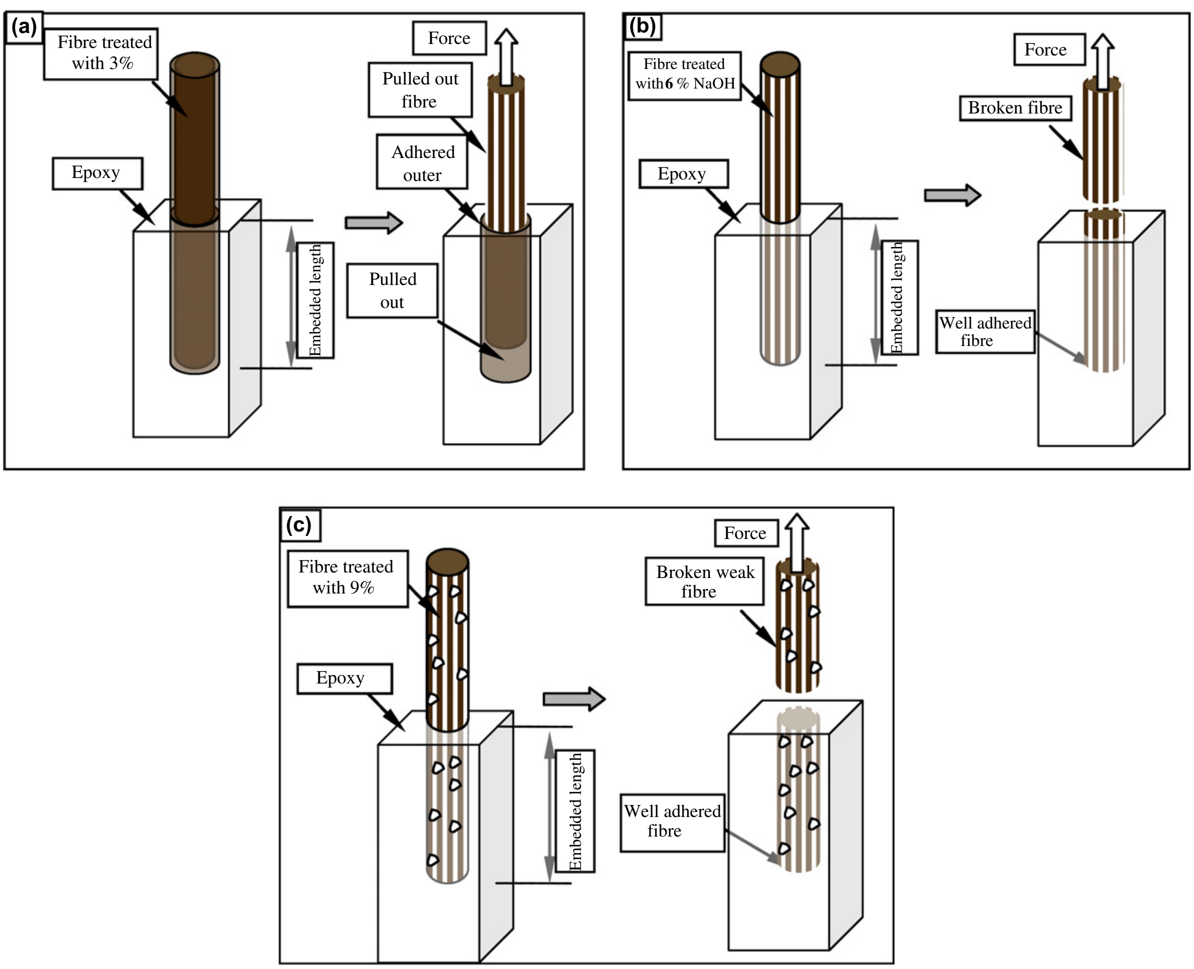

Fig. 5. Schematic drawing showing the fibre behaviour under pull out testing for treated date palm fibre with different NaOH concentration as (a) 3% NaOH, (b) 6% NaOH, and (c) 9% NaOH.

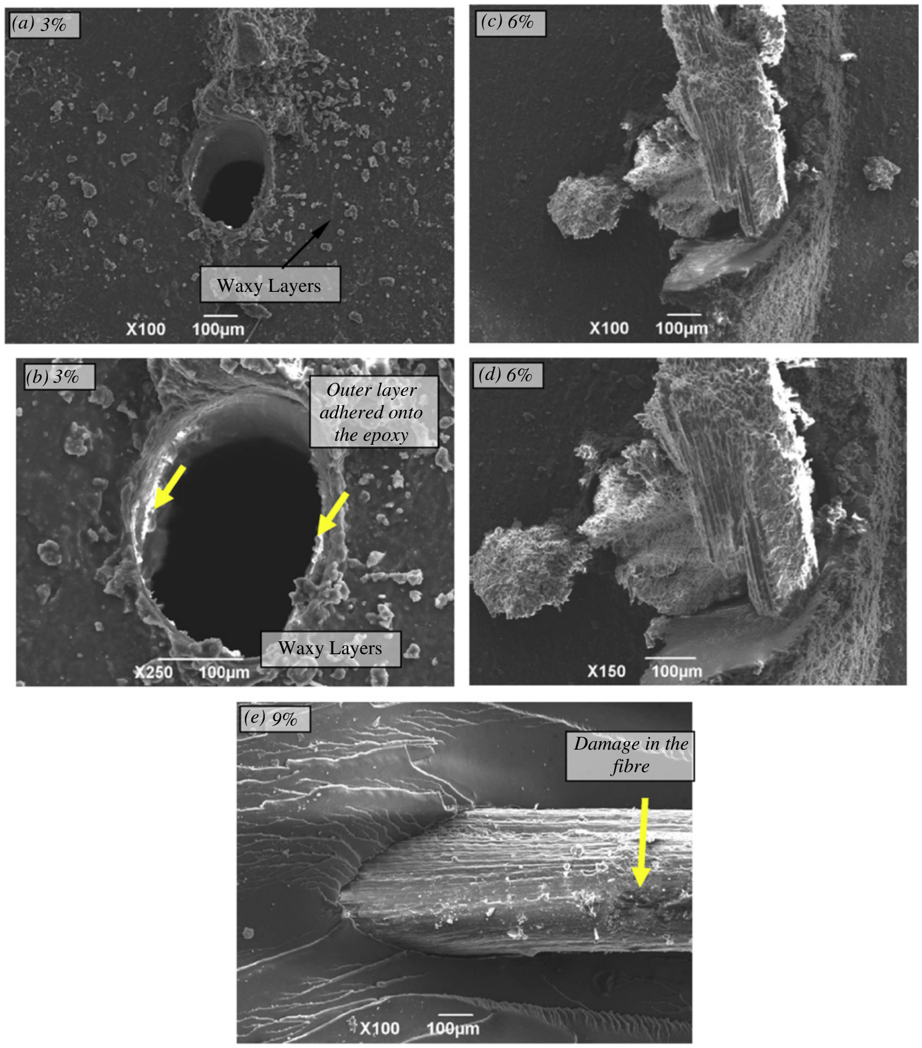

Fig. 6. Micrographs of samples prepared with different NaOH percentage treatment fibre after single fibre pull out experiments.

where, L_c = critical length, σ = tensile strength of the fibre, d = diameter of the fibre, τ_c = shear strength of the bond between the matrix and the fibre. Simply, the critical length, L_c, is the minimum length that the fibres must have to strengthen a material to their maximum load. The experimental results obtained for the tensile and pull out tests are used to calculate the critical length of each fibre at its condition. Based on this, Fig. 7 represents the relation of the critical length with the diameter of the fibre at each NaOH concentration (3–9%). The figure clearly indicates that increase the diameter of the fibre requires longer fibre length for all the NaOH concentrations. Furthermore, the low and high NaOH concentrations (3% and 9%) require longer fibre length at all fibre diameters. This is mainly due to the poor interfacial adhesion of the fibre at low NaOH concentration and weakness of the fibre at high NaOH concentration. Therefore, 6% NaOH is the most suitable percentage for date palm fibre treatment. It is recommended that this finding should be adopted with other types of fibres, i.e. it is not necessarily that the higher the NaOH concentration is the better the fibre's characteristics.

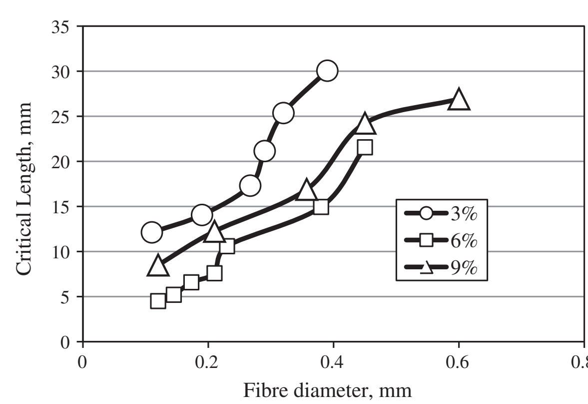

Fig. 7. Critical length relation with the fibre diameter and NaOH concentration.

Table 1 summarises the some of the most recent works studying the influence of interfacial adhesion of natural fibres with various matrices on the fibre and/or fibre/polymer composites properties in term of mechanical [18,19,26,27], physical and chemical [26] and tribological [21–24]. All those works showed significant influence of all the chemical treatment on the fibre and/or the composites properties. However, it is not true that all types of chemical treatment and concentration improve the fibre and/or composites characteristics. For instance, ethylene diamine tetraacetic acid (1.35 × 10.3 mol/L) (EDTA) [18], polyethylene imine (2000 g/mol) (PEI) [28], HCl [29], and Maleic anhydride [19] deteriorated the surface and the structure of the fibres. Despite of the enhancement on the interfacial adhesion of the fibre with the matrix, the fibre

Table 1
Summary of the previous works on interfacial adhesion of natural fibres.

Fibre	Matrix	Treatment	Remarks
Hemp [18]	Silica colloid	Ethylene diamine tetraacetic acid (1.35×10.3 mol/L) (EDTA)	EDTA decreased adhesion force by 33%
			PEI decreased adhesion force by 26%
		Polyethylene imine (2000 g/mol) (PEI)	NaOH increased adhesion force by 130%
		NaOH (1.6 mol/L)	
Coir [31]	Polyethylene	Silane (0.5 wt.%)	Silane increased adhesion force by 12%
		NaOH (1.6 mol/L)	
		Dodecane bromide (C12) (3 mL)	NaOH decreased adhesion by 4%
			C12 increased adhesion force by 4%
Betel nut [29]	Polyester Epoxy	HCl 4%	6% NaOH with polyester increased adhesion force by 130%
		HCl 6%	
		NaOH 4%	
		NaOH 6%	6% NaOH with epoxy increased adhesion force by 112%
Banana [26]	Polyurethane	NaOH 10%	Interfacial adhesion increased by 93%
Hemp [19]	Polyactide (PLA)	NaOH 5%	5% NaOH increased adhesion force by 100%
		Silane 0.5%	
		Maleic anhydride 5%	0.5% silane increased adhesion force by 45%
		Acetic anhydride	Acetic anhydride increased adhesion force by 9%
			5% Maleic anhydride had no effect on adhesion force
Oil Palm [32]	Polyester	6%	Highly enhanced the interfacial adhesion compared to the untreated fibres
Kenaf [24]	Epoxy	6%	Moderate improvement to the interfacial adhesion compared to the untreated fibres

strength dramatically worsened after treatment. In other words, there was negative effect in addition the treated fibre in the polymer matrix, i.e. neat polymer showed better mechanical properties compared to the treated fibre/polymer composites. This is highly in agreement with the current work, where high NaOH concentration (9%) enhanced the interfacial adhesion of the date palm fibre but worsened the tensile strength of the fibre itself. However, from the table, it is clearly that the NaOH chemical treatment is the most pronounced technique in enhancing the interfacial adhesion of natural fibres and maintain good tensile strength of fibre considering the optimum concentration of NaOH treatment. On the other hand, the optimum percentage of NaOH varies for each type of fibre which is dependent on the amount of OH group in the natural fibre. For example, 10% NaOH concentration is needed to remove the waxy and tissues from the banana fibres, [26]. This amount of NaOH is not desired with other types of natural fibres, e.g. date palm, kenaf [24], coir [22], betal nut [27,30] etc.

4. Conclusion

From the results obtained and observation made on the surface morphology, few points can be concluded as follows:

1. The NaOH concentration in the treatment solution affects the strength and the interfacial adhesion of the fibre with the matrix. The low concentration of the NaOH exhibited less influence on the strength of the fibre and surface interfacial adhesion. On the other hand, the higher concentration (9%) weakened and damaged the fibre. 6% NaOH concentration is the optimum concentration in which there were less damage to the strength of the fibre and high enhancement to the interfacial adhesion of the fibre with the matrix.
2. The embedded length of the fibre controlled the interfacial adhesion property of the fibre. 10 mm embedded length was the optimum fibre length which can transfer the load from the matrix to the fibre. In term of critical length, it was highly controlled by the diameter and the treatment of the fibre (concentration of NaOH). The lowest fibre critical length can be obtained at lower fibre diameter and 6% NaOH treatment.
3. The SEM observation indicated pull out mechanism at low NaOH concentration treatment of 3%. At 6% NaOH, there was a breakage of fibres indicating the high interfacial adhesion of the fibre with the matrix. Early breakage to the fibre was observed at high level of NaOH concentration (9%) which was due to the weakening process that occurred on the fibre during the treatment process.

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