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Durability of date palm fibres and their use as reinforcement in hot dry climates

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Abstract

This paper concerns the durability of male date palm surface fibres (MDPSF) immersed in three alkaline solutions: calcium hydroxide, sodium hydroxide and Lawrence solution with a pH ranging between 12.5 and 12.95. In addition the durability of MDPSF as reinforcement in small scale concrete beams has been studied. The concrete specimens were cured for six months in two different ways: 28 days in water followed by curing in wet chamber; and 14 days in water followed by a hot dry environment. The durability of MDPSF is evaluated by the loss of tensile strength and elongation, occurred over time, of fibres submitted in alkaline solutions using the tension test. The durability of the fibre-reinforced concrete is estimated by the variation of the first cracking strength, maximum post-cracking load and the maximum load ratio obtained from bending test. Some scanning electron microscopy (SEM) images of fibres extracted from alkaline solutions, as well as from the matrix at the fibre-matrix interface, have been examined to characterize the microstructure of the fibres. The results of the investigation have shown that MDPSF are easily attacked by alkaline solution under both curing conditions. Hence the durability of MDPSF-reinforced concrete is poor.

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Keywords: Date palm fibres; Vegetable fibres; Fibre concrete; Curing; Hot dry environment; Strength; Elongation; Durability; SEM; Toughness

1. Introduction

The importance of vegetable fibres is in their availability in many countries with agriculturally based economies. Several researchers [1–12] confirm that the presence of fibres in concretes improves their cracking strengths, their toughness and post-cracking performance. However, the use of these vegetable fibres in concretes is limited by the problems of durability in alkaline environment. This has been revealed in most of the research carried out on several vegetable fibres: sisal, jute and coir [11–18].

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This research is a part of a global trend concerning the study of the performance of building materials in the hot dry climate zones using locally available natural resources. As an example of a hot dry environment, the temperature of Ouargla from June to November varies from a minimum of about 10 °C to a maximum of 43 °C. For the same time period, the relative humidity varies from 24% to 57% and the wind speed varies from 8 km/h to 18 km/h [19,20]. In a former work [19], it has been shown that a direct exposure of palm fibre-reinforced concrete to the hot dry conditions can reduce its mechanical performances.

In this paper, the results of an investigation using the surface date palm fibres from Ouargla (south of Algeria) are presented. Several alkaline solutions are used to study the durability of date palm fibres: calcium hydroxide (Ca(OH)₂), sodium hydroxide (NaOH) and Lawrence solution which is based on a mixture of calcium, potassium and

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sodium hydroxides. In addition, the effect of curing (in wet and hot dry environments) on concrete durability have also been examined.

2. Materials and methods

2.1. Materials

The male date palm surface fibres (MDPSF) investigated are pulled out from trunk in a form of nearly rectangular mesh (length 300–500 mm, width 200–300 mm) formed with three superposed layers as shown in Fig. 1. It is easy to separate them in water into individual fibres of 0.2–0.8 mm diameter. Tables 1 and 2 give the physical and the mechanical properties of MDPSF [19].

CPA-CEM I 52.5 cement was used with the following physical properties: fineness = $385 \text{ m}^2/\text{kg}$, initial setting time = 200 min and compressive strength $\sigma_{c'}$ at 28 days = 50 MPa. Table 3 gives the chemical properties of this cement.

Natural sand and coarse aggregates were used with maximum sizes of 5 mm and 10 mm, respectively. The bulk densities of the fine and coarse aggregates were 1660 kg/m³ and 1610 kg/m³, respectively.

2.2. Concrete specimens and curing conditions

2.2.1. Specimens preparation

For the production of flexural concrete specimens of $70 \times 70 \times 280$ mm the method recommended by Lesage [21] and Gorisse [22] was used. For all fibre-reinforced concrete mixes, the masses of cement and sand were the same as that of simple ordinary concrete. The volume fraction of fibres was varied (2% and 3%) but the total mass of fibres plus aggregates was maintained constant. The fibre lengths were 15 and 60 mm. The water quantity was adjusted to obtain workability in terms of a VB time equal 20 ± 5 s and slump equal to 70 ± 10 mm. With the increase of volume fraction of fibres the amount for water also was



Fig. 1. Typical MDPSF mesh.

Table 1
Physical properties of MDPSF [19]

Property	Lower- upper	Mean–CV (%)
Diameter (mm)	0.1 - 1.0	0.5-54.4
Bulk density (kg/m ³)	512.2-	900-17.6
	1088.8	
Absolute density (kg/m ³)	1300-1450	1383.3-5.5
Natural moisture content (%)	9.5 - 10.5	10-5
Water absorption after 5 min (%) under water	60.1–84.1	74–14
Water absorption to saturation (%)	96.8–202.6	132.5–20.6

increased due to water absorption by fibres. No significant variation of workability was observed for changes in fibre length. Table 4 gives the mix proportions of the three concretes considered in this research. The volume fraction of fibres was: Mix 1: 0%; Mix 2: 2%; Mix 3: 3%.

A conventional Zyklos mixer with a horizontal axis and maximum capacity 100 l was used to produce the concrete composites. The fibres were washed with tap water and dried in air, before being added to the mix. The composite was produced using the following sequence: the fibres were initially immersed in 10% of the total volume of water and kept for 5 min, in order to facilitate their separation. At this stage the rate of saturation of fibres was about 56%. The sand, coarse aggregates, plus 30% of water were mixed for 30 s in a running mixer. Then, the fibres were added together with 30% of the water and mixed for 3 min. This was done slowly in order to obviate the possible clumping of fibres. After adding all cement, the remaining water was added during 2.5 min. The fresh fibre-reinforced concretes were placed in moulds on two following parts using a conventional vibration table. The time of vibration was 30 s for each part, according to standards NFP18-409 [23].

The fibre-reinforced concretes are referenced by the following notation N%–L, where N is the volume fraction of fibres and L is the length (mm) of fibres. Four types of fibre-reinforced concretes were prepared in addition to plain unreinforced concrete (2%–15, 3%–15, 2%–60, 3%–60 and 0%–0).

2.2.2. Curing conditions

The specimens were initially cured in the laboratory for 24 h under normal climatic conditions: temperature $T=20\pm2\,^{\circ}\mathrm{C}$, and relative humidity RH = 65 ± 5%. After demoulding, they were cured until the date of test in two types of environment. For the first type of environment (referenced: wet curing), the test specimens were placed 28 days in water at $20\pm2\,^{\circ}\mathrm{C}$ followed by curing in a wet chamber at $20\pm2\,^{\circ}\mathrm{C}$, with the RH = 100%. For the second type of environment (referenced: hot dry curing), they were placed 14 days in water at $20\pm2\,^{\circ}\mathrm{C}$ followed by a hot dry room at $32\pm2\,^{\circ}\mathrm{C}$ and RH = $28\pm2\%$. The hot dry room was used to simulate the mean temperature and humidity of the environment of Ouargla. The concrete specimens were tested at 28 and 180 days. Six replicate specimens were tested for each parameter setting.

Table 2 Mechanical properties of MDPSF [19]

Fibre length (mm)	Dry			Wet			
	Tensile strength (MPa)	Elongation (%)	Modulus of elasticity (GPa)	Tensile strength (MPa)	Elongation (%)	Modulus of elasticity (GPa)	
100	170 ± 40	16 ± 3	4.74 ± 2	175 ± 30	17.4 ± 2	3.78 ± 2	
60	240 ± 30	12 ± 2	5.00 ± 2	250 ± 25	13 ± 2	3.25 ± 1.5	
20	290 ± 20	11 ± 2	5.25 ± 3	300 ± 20	12 ± 2	3.55 ± 2	

Table 3 Chemical properties of cement

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	SO ₃	K ₂ O	Loss on ignition
20.29	4.60	2.98	1.66	65.24	0.30	2.70	0.99	2.80

Table 4
Mix proportions

Materials (kg/m ³)	Mix 1	Mix 2	Mix 3
Cement	400	400	400
Aggregate	1000	982	973
Sand	750	750	750
Water	240	270	290
MDSF	0	18	27

2.3. Test methods

The mechanical properties (tensile strength, elongation and the modulus of elasticity) of fibres were determined in accordance with standards NF EN ISO 5079 [24], at 20 ± 2 °C and RH = $65\pm2\%$, in an Instron universal testing machine at a cross-head speed of 0.5 mm/min. The tensile test lengths were 20, 60 or 100 mm. Fig. 2 shows the set-up of the fibres in the tensile test device, in which the cylinders were used to grip the fibre ends. Thirty fibres were tested for each parameter setting.

A Hitachi S 800 scanning electron microscope was used to study fibre surface topographies and to examine the section of fibres and the fibre–matrix interface. The fibres and the fibre–matrix samples were attached to conducting silver plates then were sputter coated with a layer of gold 20–30 nm thick in a vacuum chamber. The SEM images were obtained by the secondary electron imaging method, with a tension of acceleration of the beam of 15 kV.

The flexural strength has been determined using an Instron universal testing machine and a four-point test configuration, with 210 mm span, and with an experimental set-up in accordance with the NFP 18-409 standard [23]. The displacements have been measured using two LVDT displacements transducers and the deflection at mid-span has been recorded as a function of loading.

2.4. Evaluation of fibre durability in alkaline solutions

The durability of MDPSF is evaluated using two methods. The first one is related to the loss of tensile strength, occurred over time, of fibres submitted to two alkaline

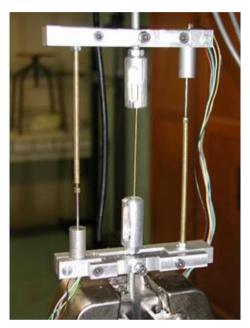


Fig. 2. The tensile strength test set-up.

solutions: in a calcium hydroxide (Ca(OH)₂) and sodium hydroxide (NaOH), with pH 12.5 for both solutions. The pH is regularly controlled. The fibres are tested after duration of immersion in solution of one, two, three and six months. Sixty fibres of length of 100 mm for each term and diameter (0.8, 0.6, 0.4 mm) are tested, after having been dried in the laboratory for 24 h.

The second one is based on some microscopic observations with a scanning electron micrograph of the fibres immersed for six months in the following alkaline solutions:

- Ca(OH)₂ pH 12.5
- NaOH pH 12.5
- Lawrence, with following proportions: Na = 0.22 mol/l, K = 0.061 mol, Ca = 0.0065 mol/l and pH 12.95 at 20 °C.

According to Houget [25], Lawrence solution is among the best solutions, which simulate experimentally the interstitial pore fluid of ordinary cement composites.

2.5. Evaluation of fibre durability in the concrete

The durability of fibres as reinforcement in the concretes is estimated by the variation of their flexural properties.

From the load-deflection curves, the flexural properties were evaluated using three parameters: (a) the first cracking strength (FCS); (b) the maximum post-cracking flexural load carried by the composite after the first cracking $(P_{\rm max})$; and (c) the maximum load ratio $D_{\rm max} = P_{\rm max}/P_0$ where P_0 is the load at the first visible cracking. It has been assumed to correspond to the maximum load of the load-deflection curve in the first elastic part.

Microscopic observations are given for fibres extracted from concretes kept under wet and hot dry curing conditions for six and 12 months. In addition, other microscopic observations are also made at the fibre–matrix interface.

3. Results and discussion

3.1. Tensile strength of fibres

Tables 5 and 6 indicate that the MDPSF has poor resistance to alkaline solution attack. The mechanical performance (tensile strength and elongation) is affected by immersion time in alkaline solutions. The loss of the tensile strength and elongation of fibres in Ca(OH)₂ solution is higher than that in NaOH solution. The higher alkaline attack Ca(OH)₂ is associated with crystallisation of lime in the pores of the fibres. In fact, as a function of time of immersion in alkaline solutions, the fibres become increasingly brittle in Ca(OH)₂ solution compared to those in NaOH solution. Moreover, when the diameter of fibres decreases the mechanical performance decreases. It has been found that in Ca(OH)₂ solution the MDPSF fibres retained about 76%, 48% and 33% of their original strength

Table 5
Tensile strength and elongation of MDPSF the length 100 mm immersed in Ca(OH)₂ solution

Time (months)	Tensile st	rength (M	Elongation (%)			
Diameter of fibre (mm):	0.8	0.6	0.4	0.8	0.6	0.4
0	180 ± 20	160 ± 18	60 ± 15	20 ± 2	18 ± 2	12 ± 3
1	151 ± 11	92 ± 13	39 ± 15	19 ± 2	16 ± 3	09 ± 2
2	146 ± 15	83 ± 15	27 ± 14	17 ± 2	15 ± 2	07 ± 2
3	137 ± 12	77 ± 14	20 ± 07	15 ± 2	13 ± 3	06 ± 2
6	125 ± 12	65 ± 11	05 ± 04	12 ± 2	08 ± 2	01 ± 1

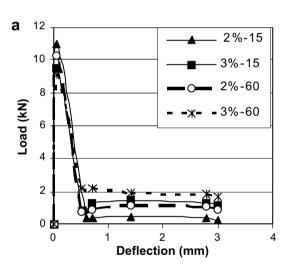
Table 6
Tensile strength and elongation of MDPSF the length 100 mm immersed in NaOH solution

Time (months) Diameter of fibre (mm):	Tensile str	rength (MP	a)	Elongation (%)			
	0.8	0.6	0.4	0.8	0.6	0.4	
0	180 ± 20	160 ± 18	60 ± 15	20 ± 2	18 ± 2	12 ± 3	
1	159 ± 14	106 ± 12	58 ± 16	19 ± 2	17 ± 3	10 ± 2	
2	153 ± 12	95 ± 15	52 ± 15	18 ± 2	16 ± 2	08 ± 3	
3	149 ± 13	88 ± 13	25 ± 13	17 ± 2	14 ± 3	06 ± 2	
6	137 ± 16	74 ± 18	10 ± 06	13 ± 2	10 ± 2	02 ± 2	

for the diameters 0.8, 0.6 and 0.4 mm, respectively, at three months, whereas, they retained only 69%, 40% and less then 10% at six months. In NaOH solution they retained about 83%, 55% and 40% at three months and 76%, 46%, and 16% at six months for the diameters 0.8, 0.6 and 0.4 mm, respectively.

These results are in concordance with those reported by Lewis and Premalal [4] and Aziz et al. [7], Bentur and Akers [14], and Tolêdo Filho et al. [15,16]. As an example: Tolêdo Filho et al. [15] reported that the sisal and coconut fibres are highly sensitive to the Ca(OH)₂ and NaOH solutions of pH 12 and 11, respectively. Lewis and Premalal [4] reported, about the tensile strength of some vegetable fibres immersed in Ca(OH)₂ solution with pH 10, that elephant grass fibres retained nearly 91% of their original strength at three months, and then followed by a constant rate of reduction, water reed fibres retained nearly 57% and 14% of their original strength at three and six months, respectively.

From that it has been thought that the durability of MDPSF in Portland cement may be affected, due to calcium hydroxide release during the hydration process.



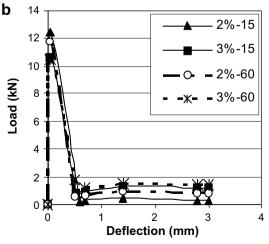


Fig. 3. Load-defection curves of MDPSF-concretes in hot dry conditions: (a) at 28 days, (b) at 180 days.

3.2. Flexural properties of fibre-reinforced concretes

Fig. 3 presents the mean bending load–deflection curves of MDPSF-reinforced concrete maintained 28 and 180 days in hot dry conditions. The load–deflection curves for specimens under wet conditions are similar to those for the hot dry conditions. The beams behaved elastically up until the first cracking strength (FCS). Beyond the FCS the recorded at a deflection of about 0.04 mm, the initiated cracking exhibited instable growth leading either to separation of the body into parts when there is no fibre, or to a macro-cracked beam with a deflection around 0.5 mm when the fibres could stop the cracking growth. After that the load became nearly constant with the increasing bending deflection.

Table 7 provides the mean and the coefficient of variation (CV) of the first cracking strength (FCS), the maximum post-cracking flexural load ($P_{\rm max}$) and the maximum load ratio ($D_{\rm max}$), in wet and hot dry conditions. The results show that, for both type of curing and for each concrete type, the FCS continues to increase with ageing. However, when the length and the volume fraction of fibres increases the FCS decreases.

The examination of the $P_{\rm max}$, and $D_{\rm max}$, show that for each age (28 or 180 days) the $P_{\rm max}$, and $D_{\rm max}$ increase with increasing length and volume fraction of fibres. For each concrete type, the $P_{\rm max}$, and $D_{\rm max}$ decrease with ageing due to loss of tensile strength of fibres in the alkaline environment, because failure of the bend test specimens is caused by fibre rupture.

Table 7 FCS, P_{max} and D_{max} of MDPSF–concretes as function of time and curing conditions

Flexural properties	Ageing conditions	Ageing time (days)	Concretes type					
			0%-0	2%-15	3%-15	2%-60	3%-60	
FCS (CV) (MPa) (%)	Wet	28	7.60 (7.11)	6.47 (11.59)	5.50 (14.54)	6.02 (10.82)	5.34 (12.47)	
	Wet	180	8.35 (5.98)	7.40 (8.78)	6.30 (11.11)	6.82 (8.95)	6.21 (12.86)	
	Hot dry	28	7.90 (7.28)	6.74 (4.68)	5.80 (5.60)	6.30 (4.92)	5.60 (6.81)	
	Hot dry	180	8.70 (3.08)	7.60 (4.15)	6.50 (3.54)	7.22 (6.12)	6.40 (2.66)	
P_{max} (CV) (kN) (%)	Wet	28	_ ` `	0.80 (19.32)	1.69 (11.77)	1.18 (11.06)	2.96 (2.57)	
	Wet	180		0.70 (26.67)	1.40 (11.14)	0.99 (29.69)	2.02 (19.04)	
	Hot dry	28		0.59 (25.22)	1.50 (25.62)	1.15 (17.84)	2.23 (20.15)	
	Hot dry	180		0.49 (23.19)	1.20 (12.35)	0.96 (17.03)	1.50 (13.37)	
D_{max} (CV) (%)	Wet	28	_	0.08 (2.85)	0.19 (27.58)	0.10 (12.63)	0.34 (14.57)	
max (= v) (v)	Wet	180	_	0.06 (11.19)	0.14 (11.25)	0.09 (24.30)	0.20 (8.36)	
	Hot dry	28		0.05 (16.27)	0.15 (13.15)	0.11 (9.17)	0.24 (11.38)	
	Hot dry	180		0.03 (27.14)	0.11 (17.04)	0.07 (15.39)	0.14 (10.22)	

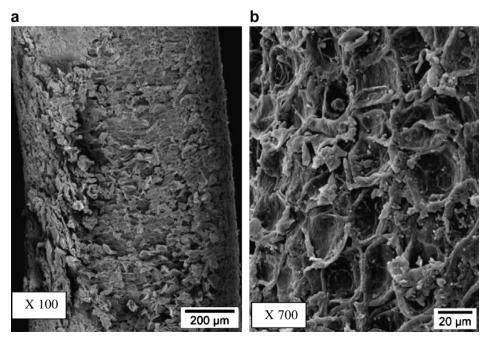


Fig. 4. SEM images of typical natural MDPSF.

3.3. Microscopic observations of fibres

To examine the effect of alkaline attack, Fig. 4 presents SEM images of typical natural MDPSF before their immersion in alkaline solution. The surface of fibres is irregular with many filaments and cells, allowing for good adhesion between the fibre and matrix. Figs. 5–7 present the SEM images of typical fibres immersed during six months in Ca(OH)₂, NaOH and Lawrence alkaline

solutions, respectively. Fig. 5 shows the mechanism of calcium hydroxide attack, the uniform deposition of this last on all fibre surface and its penetration into the cells and pores of the fibres can be distinguished, and hence the mineralization of fibres. In fact Fig. 5b shows clearly the deposition of portlandite (P) at the fibre surface. This helps explain the drop in their resistance and their elasticity. The deterioration of the filaments of the fibres is also evident.

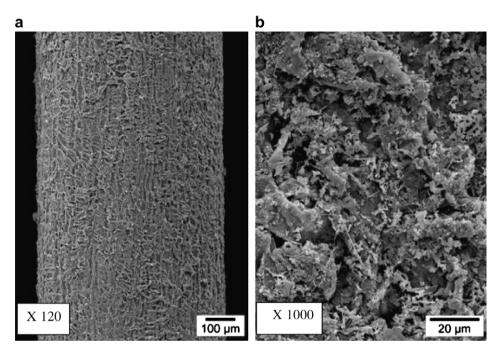


Fig. 5. SEM images of typical MDPSF immersed during six months in Ca(OH)₂ solution.

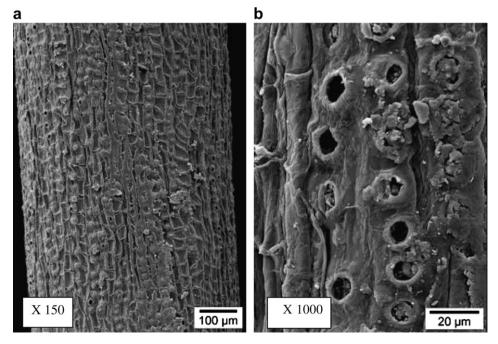


Fig. 6. SEM images of typical MDPSF immersed during six months in NaOH solution.

In addition, Fig. 6 visualizes the mechanism of NaOH attack. Contrary to the mechanism of calcium hydroxide attack, NaOH attack is characterized by a local penetration of sodium hydroxide into the cells and pores of the fibre while burning, continuously, the organic matter. This leaves local holes that appear like bird nests in Fig. 6b and the fibres become increasingly fine and porous (Fig. 6a). That explains the decease in fibre resistances and their section area as a function of immersion time.

Thus, the mechanism of Ca(OH)₂ attack is diffuse, whereas, the mechanism of NaOH attack is local. For the crystallisation of lime in the pores of the fibres in Ca(OH)₂ solution the flexibility of fibres was reduced. That is a probable explanation for why the tensile strength and elongation of fibres in Ca(OH) solution are lower than those in NaOH solution. It appears that rate of degradation of fibres in Ca(OH)₂ solution is about ten times greater than that in NaOH solution.

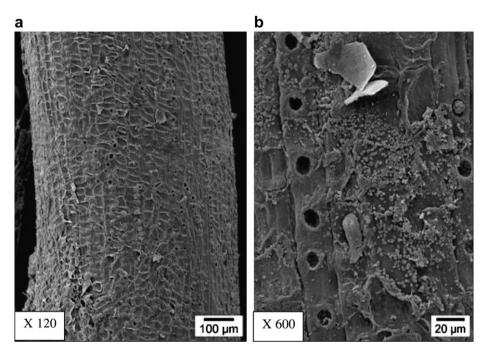


Fig. 7. SEM images of typical MDPSF immersed during six months in Lawrence's solution.

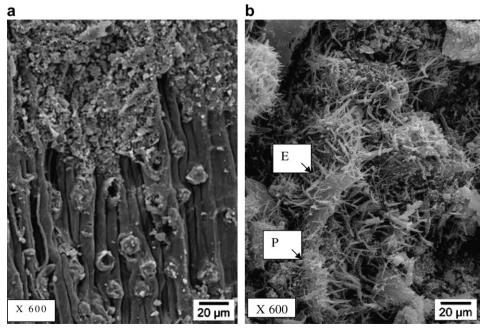


Fig. 8. SEM images of typical MDPSF extracted from fibre concretes conserved in wet conditions for 12 months; P: portlandite, E: ettringite.

Fig. 7 shows the mechanism of attack due to exposure to Lawrence's solution. The previous mechanism of Ca(OH)₂ and NaOH attack are both active. Indeed, the mechanism of Ca(OH)₂ attack by uniform deposition can be distinguished, as well as the penetration of calcium hydroxide in several pores of fibre and the mechanism of the local attack of NaOH solution (Fig. 7b). That is logical since this solution is composed, of alkalis of calcium, sodium and potassium (Table 2).

Fig. 8 presents a SEM image of a typical fibres extracted from the concretes after 12 months in wet conditions. The

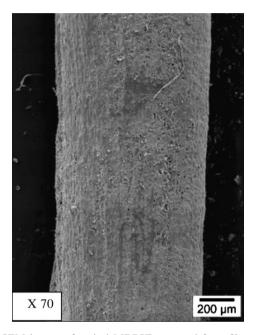


Fig. 9a. SEM images of typical MDPSF extracted from fibre concretes conserved in wet conditions for six months.

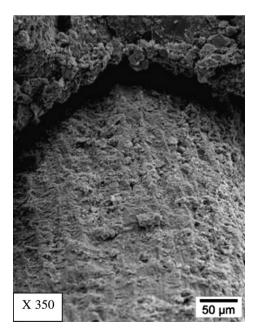


Fig. 9b. SEM images of typical MDPSF-matrix interface of fibre concretes after six months in wet conditions.

penetration of the calcium hydroxide and the local sodium hydroxide attack can be observed (Fig. 8a). The formation of portlandite (P) and needles of ettringite (E) at the fibre surface (Fig. 8b) are also evident. The mechanism of attack is similar to that due to Lawrence solution exposure. That helps confirm the similitude between the Lawrence solution and the interstitial composition of the cement matrix, and secondly it explains the loss of durability in wet conditions.

Figs. 9 and 10 present SEM images of the fibres extracted from concrete and of fibre-matrix interface after

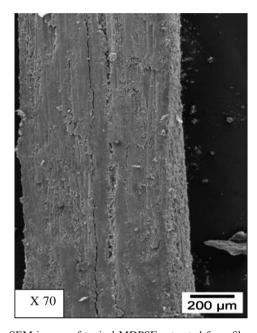


Fig. 10a. SEM images of typical MDPSF extracted from fibre concretes conserved in hot dry condition for six months.

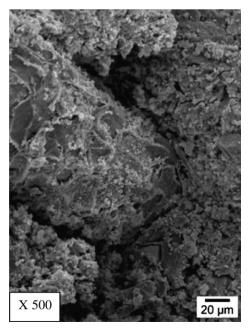


Fig. 10b. SEM images of typical MDPSF-matrix interface of fibre concretes after six months in hot dry conditions.

six months in wet and hot dry conditions, respectively. Figs. 9a and 10a present the SEM of typical fibres extracted from fibre concrete conserved in wet and hot dry conditions, respectively, after six months. Figs. 9b and 10b present some SEM of typical fibre-matrix interface of fibre concretes after six months in wet and hot dry conditions, respectively. The fibres extracted from the concrete conserved in hot dry conditions are less affected compared to those conserved in wet conditions (Figs. 9a and 10a). In the same way, at the broken surface, and at the fibrematrix interface. Figs. 9b and 10b show that, the cracks around some fibres are relatively smaller for the concrete in hot dry conditions. It is thus confirmed that the quantity of alkaline hydration products in hot dry conditions is greater than that in wet conditions. This may also explain why the post-cracking flexural properties of MDPSF-reinforced concretes, after the first cracking, in hot dry conditions are slightly lower than that in wet conditions condition.

4. Conclusion

Experimental work was conducted to study the resistance of male date palm surface fibres (MDPSF) to highly alkaline solutions. The durability of these fibres was also studied through flexural testing of the fibre-reinforced concrete specimens kept under either wet or hot dry curing conditions. The following conclusions can be made:

- (a) Like more common vegetable fibres, the MDPSF are severely affected by the alkaline solutions (calcium hydroxide and sodium hydroxide with pH 12.5, and Lawrence solution with pH 12.95). The results show that after six months in Ca(OH)₂ solution the MDPSF retained about 69%, 40% and less than 10%, and in NaOH solution they retained about 76%, 46%, and 16% of their original strength, for the diameters 0.8, 0.6 and 0.4 mm, respectively.
- (b) The mechanism of Ca(OH)₂ attack is diffuse and relatively uniform, whereas the mechanism of NaOH attack is localized. The degradation of fibres in the Lawrence solution is similar to that of fibres exposed to interstitial fluids of the cement matrix.
- (c) The MDPSF extracted from concrete conserved in the hot dry conditions is slightly more affected than that cured under wet conditions. This may be attributed to greater effects of the hydration products in the hot dry conditions.
- (d) For both curing conditions and for each concrete type, the first cracking strength continues to increase with ageing. For each testing age (28 or 180 days), however, the first cracking strength of conventional concrete is greater than that of the fibre-reinforced concretes.
- (e) The results show that peak load (P_{max}), and load ratio (D_{max}) increase with increasing length and volume fraction of fibres. For each concrete type,

- however, P_{max} , and D_{max} decrease with ageing, which is caused by the loss of tensile strength of fibres in alkaline cement products.
- (f) The hot dry environment has a beneficial effect on the performance of fibre concretes at early age. For the longer term, however, it has a beneficial effect only on the first cracking strength. Some other treatments of MDPSF and cement–matrix are then necessary to improve their durability, especially after the first cracking.

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