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Durability of alkali-sensitive sisal and coconut fibres in cement mortar composites

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Abstract

This paper presents the results of an experimental program designed to assess the durability of sisal and coconut fibres exposed to alkaline solutions of calcium and sodium hydroxide. In addition, the durability and microstructure of the cement mortar composites reinforced with these fibres aged under tap water, exposed to controlled cycles of wetting and drying, as well as to the open air weathering have been studied. The possibility of biological attack of fibres was investigated by conditioning them in tap water for 420 days. It was found that sisal and coconut fibres kept in a calcium hydroxide solution of pH 12 completely lost their flexibility and strength after 300 days. The composites manufactured with short sisal or short coconut fibres and ordinary Portland cement "OPC" matrix presented a significant reduction in toughness after six months of exposure to the open air weathering or after being submitted to cycles of wetting and drying. The embrittlement of the composites can be mainly associated with the mineralisation of the fibres due to the migration of hydration products, especially calcium hydroxide, to the fibre lumen, walls and voids.

Keywords: Sisal fibres; Coconut fibres; Cement; Mortar; Composite materials; Durability; Strength; Toughness; Weathering

1. Introduction

During the last two decades a considerable effort has been directed toward the application of vegetable fibres, which exist in abundance in the tropical and subtropical parts of the world, as reinforcing material for the production of building components with thickness up to 20 mm in combination with cement mortar or cement paste matrix, for low-cost housing. The main objective in this field is finding a substitute for the asbestos-cement products. Although enormously successful throughout seven decades of the 20th century, their use began to be prohibited by law in industrialised countries because of the serious health hazard they present to humans and animals. Unfortunately, asbestos-cement products are still commonly produced and used in the under-developed and developing countries.

In the production of composites made of brittle cement matrices, the main contribution of the fibres is to improve their toughness and post-cracking performance. There has been concern about the applicability of vegetable fibres in cement-based matrices mainly due to the long-term durability of the composite that may undergo reduction in strength and toughness. It has been suggested [1–5] that this is associated with a decrease in the amount of fibre pull-out due to a combination of a weakening of the fibres by alkali attack, fibre mineralisation provoked by migration of hydration products to the fibres' lumen and voids in addition to the volume variation in the fibres due to their high water absorption. The extent of the attack depends on the fibre type, cement matrix composition, porosity of the matrix and ageing environment.

The degradation of natural fibres in cement-based composites occurs because the alkaline pore water dissolves the lignin and hemicellulose existing in the middle lamellae of the fibres, thus weakening the link between the individual fibre cells. An additional mechanism is the

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Table 1
Physical and mechanical properties of sisal and coconut fibres [3,4]

Property	Sisal fibre		Coconut fibre	
	Lower–upper	Mean–CV (%)	Lower–upper	Mean–CV (%)
Diameter (mm)	0.08–0.30	0.12–23.8	0.11–0.53	0.25–27.30
Density (kN/m^3)	7.50–10.70	9.00–8.90	6.70–10.00	8.0–7.60
Natural moisture content (%)	10.97–14.44	13.30–8.80	11.44–15.85	13.5–10.00
Water absorption after 5 min under water (%)	67.00–92.00	82.00–14.50	22.00–38.00	28.00–16.0
Water absorption to saturation (%)	190.00–250.00	230.00–16.00	85.00–135.00	100.0–19.5
Tensile strength (MPa)	227.80–002.30	577.50–42.66	108.26–251.90	174.00–24.20
Modulus of elasticity (GPa)	10.94–26.70	19.00–29.50	2.50–4.50	3.50–27.00
Strain at failure (%)	2.08–4.18	3.00–29.15	13.70–41.00	25.00–29.10

alkaline hydrolysis of cellulose molecules, which causes degradation of molecular chains, therefore leading to a reduction in degree of polymerisation and lower tensile strength [1].

This paper is part of a series of paper related to the behaviour of vegetable fibre-reinforced mortar composite (VFRMC). This paper concerns the durability of sisal and coconut fibres exposed to tap water and alkaline solutions of calcium and sodium hydroxide. In addition, the microstructure and durability of VFRMC aged under tap water, exposed to open air weathering and to controlled cycles of wetting and drying are presented. The durability of sisal and coconut fibres was measured as the loss of strength in time to the mentioned treatments. The durability performance of the composites is discussed using results of flexural tests carried out on specimens of dimensions $400 \times 100 \times 15$ mm^3 before and after 6 and 12 months of ageing, and from observations of the photo-micrographs at the fibre–matrix interfaces. Backscattered electron imaging in the scanning electron microscopy "SEM" combined with the image analyser was used in order to detect any changes in the microstructure of the composites after ageing.

The long-term performance of the VFRMC in different environments is of significant importance in finding the reliability of this newly developed composite. The microstructural studies carried out on samples taken from the composites exposed to different environments gave important information concerning the deterioration and hence the durability of VFRMC.

2. Experimental study

2.1. Materials

The natural sisal and coconut fibres used in this investigation were of Brazilian production. The upper, lower, mean and coefficients of variation (CV) of the physical and mechanical properties of these fibres based on a minimum of 20 tests are given in Table 1 [3,6,7]. The characteristics of the Thames valley sand used in this investigation, determined using the British Standard [8], are presented in Table 2. Chemical and physical properties of the used ordinary Portland cement "OPC" are presented in Table 3. Tap water was used in all mixes.

2.2. Test methods

The durability of sisal and coconut fibres was measured as strength loss occurred over the time of fibres submitted to three types of treatments: fibres stored in tap water of pH 8.3; fibres stored in a solution of cal-

Table 2
Characteristics of the aggregate

Fineness modulus	Specific gravity	Total moisture content (%)
2.81	2.65	0.35

Table 3
Chemical and physical properties of the cementing material

Chemical properties										Physical properties		
SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Loss on ignition	Soluble residue	Fineness (m^2/kg)	Setting time (initial – min)	$\sigma_{c,14d}$ (MPa)
20.70	3.00	4.61	64.70	1.00	3.00	0.13	0.65	1.30	0.38	353	135	59.20

cium hydroxide of pH 12; and fibres stored in a solution of sodium hydroxide of pH 11.

Sisal and coconut fibres were stored in small containers with tap water or chemical solutions for up to 420 days. The containers were covered and the pH of the solutions was checked at regular intervals; solutions which had not retained the initial pH value were replaced. The tensile strength of fibres was determined using a Branco-Marcallo (Varese) tensile testing machine with the maximum capacity of 200 N. Load was applied at a constant rate of about 20 mm/minute. The tests were carried out after 0, 30, 60, 90, 120, 180, 210, 300 and 420 days of immersion in the solutions or in water. Sixty fibres were tested at each period. The fibres of 65 mm length were tested after being dried in the laboratory at room temperature for 24 h.

The durability of the VFRMC was evaluated from the behaviour in bending of the specimens of dimensions 400 mm × 100 mm × 15 mm over a span of 300 mm subjected to third-point loading. The tests were ended when the displacement at mid-span reached about 4.0–5.0 mm. Twenty-one test specimens were cast for each mix. Three samples were tested after 28 days of curing as reference. The other 18 specimens were subjected to the three different ageing conditions. In each condition, three specimens were tested after six months and the rest after 10–12 months. The ageing conditions were: stored in water with temperature of about 18°C, controlled cycles of wetting and drying and London open air weathering.

To define the period of the cycle of wetting and drying of VFRMC, an extra sample was cast and then completely saturated in water at 18°C and left to dry in a conditioned laboratory room at 23°C and 40.1% relative humidity. Readings of gain and loss of weight were made during this period. Fig. 1 shows the total mass of the specimen during the cycles of wetting and drying. Considering that the saturation of the sample occurred in the first 24 h and the loss of about 72% of the gained mass in six days, a 7-day cycle was chosen. In this cycle, the samples were left one day under water at 18°C and six days drying in the conditioned laboratory room.

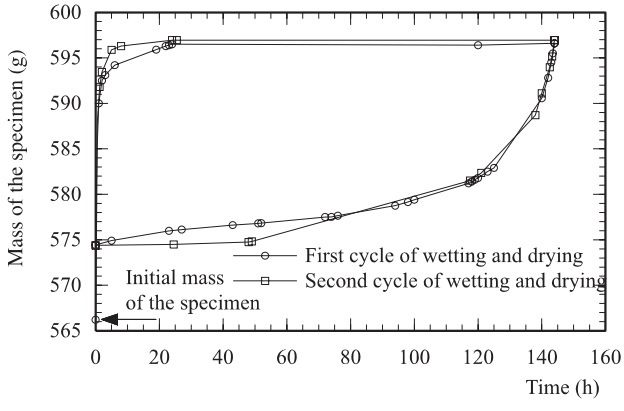


Fig. 1. Definition of the length of the wetting and drying cycle.

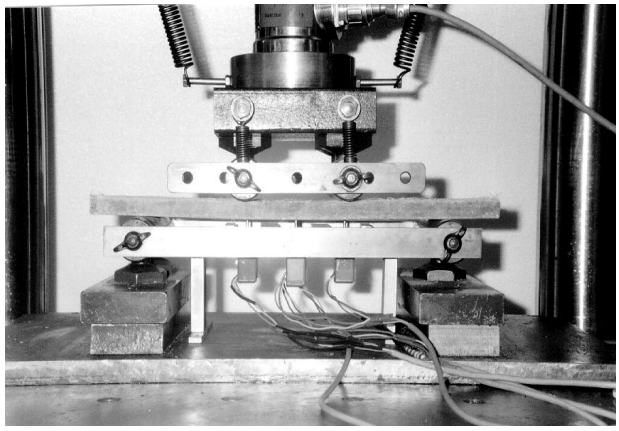


Fig. 2. Test set-up for the bending test.

The bending tests were carried out on a 2500 kN Mayes testing machine at a cross-head rate of 0.1 mm/min. Deflections at mid-span and at the load-points were measured using three and two electrical transducers, respectively. Load and deflections were continuously recorded through a 16 bit data acquisition system capable of taking up to 40 readings/s. Fig. 2 shows the test set-up for the bending test.

Table 4 presents the mixes studied. In this table, the following designations are used to represent fibre type, fibre length, fibre arrangement, volume of fibre and OPC mortar mix: M1 – cement mortar mix proportions by weight 1:1:0.4 (cement:sand:water); S – sisal fibre; C – coconut fibre; number after the fibre type – volume fraction of fibre; number after the fibre volume symbol-length of fibre; l – length of fibre; 2s + 1s – 2% of randomly arranged short sisal fibre (25 mm) plus 1% of aligned continuous sisal fibre (375 mm) and 2c + 1s – 2% of randomly arranged short coconut fibre (25 mm) plus 1% of aligned continuous sisal fibre (375 mm).

2.3. Composite production

Short sisal and coconut fibres with 25 mm length and long sisal fibres with 375 mm length were used in this investigation. Fibre arrangements were either only randomly mixed or randomly mixed plus aligned fibres along the length of the specimen. A conventional pan mixer was used to make the VFRMC. The specimens with random fibres were prepared as follows: all the sand was placed in the mixer which was then switched on; 40% of the total amount of water was added to the operating mixer; the chopped fibres were added to the

Table 4
Matrix and composite mix proportions

Mix	Mortar mix proportions (by weight)	Fibre type	Fibre volume, V_f (%)	Fibre length, l (mm)	Fibre arrangement
M1	1:1:0.4	–	–	–	–
M1S325	1:1:0.4	Sisal (s)	3s	25	Random
M1S2S1	1:1:0.4	Sisal (s)	2s + 1s	25 + 375	Random + aligned
M1C325	1:1:0.4	Coconut (c)	3c	25	Random
M1C2S1	1:1:0.4	Coconut (c) + sisal (s)	2c + 1s	25 + 375	Random + aligned\

running mixer little by little in order to avoid clumping. During this stage 30–35% of the total amount of water was progressively added to keep the mix wet. After adding all volume of fibres, the cement was added to the mixer. The remaining amount of water was then added to the running mixer and the mixing process was continued for about 5 min to enhance fibre dispersion in the mix. Samples with short and long aligned fibres were cast as follows: a short fibre-reinforced mortar layer about 3–4 mm thick was placed in the bottom of the mould and then the layer of long and aligned sisal fibre was uniformly distributed over it. The remaining volume of the mould was then filled with short fibre-reinforced mortar. A conventional vibration table was used after placing the layer of long fibre in the mould and then after completely filling the mould with short fibre composite randomly mixed. Time of vibration was established according to the recommendation made by ACI 544.2R [9].

The test specimens were cast in wooden moulds having $400\times100\times15$ mm^3 internal dimensions. The moulds were made in such a way that seven specimens could be cast at the same time. The specimens were covered with a damp cloth and a polythene sheet in their moulds for 24 h. After this time, they were demoulded and cured for 28 days in a curing box at 18°C and 97% relative humidity.

2.4. Methods for the microstructural analysis

Two methods of obtaining images on "SEM" were used in this study: backscattered electron imaging (bse) and secondary electron imaging (se). The microscopic studies were carried out on a system which consists of a JOEL 35CF scanning electron microscope, equipped with a solid-state backscattered electron detector, a LINK AN10000 energy dispersive X-ray analysis (EDXA) system and a KONTRON SEM-Image Processing System. The image analyser controls the incident beam of the SEM and can collect the signal from the secondary electron, from the backscattered electron detector, or the counts from up to four windows of the X-ray spectrum via the EDXA system. The images are digitised into an array of 512×512 pixels with 256 grey levels.

The samples used for bse imaging were slices taken from the same specimens used for the flexural test. The slices were cut from the region of the plate where the bending moment was minimum. Then they were vacuum resin impregnated using an Araldite MY 753 epoxy resin and hardener. After impregnation, slices of about 2 mm were cut, lapped and polished with successively decreasing grades of Hyprex diamond pastes down to 0.25 μm. The polished specimens were then coated with approximately 15 μm of evaporated carbon. This type of coating was used because it prevented electrical charging. In addition it had little effect on atomic number contrast or X-ray production for elemental identification. The fibres used for bse imaging work were obtained also from the plates tested in flexure. In this case, piece of fibres pulled out of the matrix and cut-out from the fractured surface were mounted on SEM stubs, gold coated and then their surfaces studied.

2.5. Methods of evaluating the durability of VFRMC

From the load–deflection curves, three parameters were calculated to evaluate the reinforcing effect of the fibre and consequently its durability or embrittlement with time:

1. The flexural strength of the composite (σ_b) – determined from the maximum post-peak load using the bending formula given by Eq. (1)
$$\sigma_b = 6M/bd^2, \quad (1)$$
where M is the failure moment of beam and d and b are the depth and width of the beam, respectively.
2. The Japanese toughness index (T_{JCI}) [10] – defined as the energy required to deflect the VFRMC beam to a midpoint deflection of 1/150 of its span i.e. 2 mm in this study.
3. The French and Belgian load ratios $P^* = P_n/P_f$. The applied load at deflections of 0.5, 1.0, 1.4, 2.0 and 2.8 mm is defined as P_n and that at the first visible crack is defined is P_f [11,12].

Tests carried out after 28 days of curing in damp cloth were considered as reference or control tests. Indices calculated from the load–deflections curves of these tests were considered as reference or control indices.

3. Test results and discussion

3.1. Vegetable fibre durability

A significant reduction in strength was observed for sisal and coconut fibres conditioned in a calcium hydroxide solution "$Ca(OH)_2$" as it is shown in Fig. 3. It has been found that after 210 days sisal and coconut fibre retained, respectively, 33.7% and 58.7% of their original strength which have been lost completely after 300 days. The flexibility of the air-dried fibres was reduced to such an extent that the fibres could be pulled apart fairly easily by finger force. The sisal and coconut fibres immersed in sodium hydroxide "Na(OH)" retained, respectively, 72.7% and 60.9% of their initial strength after 420 days. The higher alkaline attack by $Ca(OH)_2$ is probably associated with crystallisation of lime in the pores of the fibres [1,2,13–15]. This can be explained considering that the loss of strength with time is in less extent for fibres immersed in tap water. The reduction of 17–23% of the initial fibre strength, after 420 days in water, is attributed to the microbiological action.

3.2. Embrittlement of mortar matrix reinforced with short fibres

Typical examples of bending load–deflection curves obtained from specimens of the mixes M1S325 and M1C325 are presented in Fig. 4. It can be seen that they are seriously embrittled with ageing. Although the loss of reinforcing capacity has been more severe when the specimens were subjected to alternately wet and dry conditions in the laboratory (Fig. 4(c)) or outdoors (Fig. 4(b)), some embrittlement was also observed when the specimens were stored in water (Fig. 4(a)). One of the main reasons is that the transport of OH^- ions or Ca^{2+} ions from the cement matrix to the fibres occurs slowly when the environment is kept constant. When the sisal composites are subjected to alternating wet and dry conditioning, the capillary pore system of the cement matrix in turn would be alternately filled and emptied with alkaline pore water. This movement increases the transport of hydration products from the matrix to the fibres as has been observed by Gram [1].

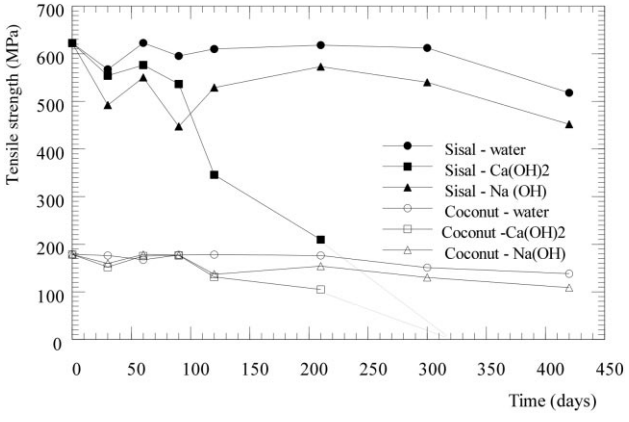


Fig. 3. Behaviour of sisal and coconut fibres aged in alkaline solutions and in tap water.

The specimens of the mix M1S325 aged in water for 322 days presented a reduction in the parameters σ_b and T_{JCI} of about 53% of the initial values as shown in Fig.5(a) and (b). A strain hardening behaviour can be observed in the load–deflection curves presented in Fig. 4(a). After the appearance of the first crack it has been observed that the gradient of the work hardening for the control specimen is much higher than those of the specimens kept in water for 180 and 322 days. This tendency is more intense for the mixtures reinforced with coconut fibre. As it can be seen in Fig. 5(c) and Table 5 the parameter $P^*_{2.8}$ indicates that the mix M1S325 maintains 57% of first crack load whereas this value for the mix M1C325 is only 29%.

For the cases when the specimens were aged outdoors and submitted to 25 and 46 cycles of wetting and drying, the dominant post-cracking behaviour is work softening. The drop in the $P^*_{2.8}$ is more significant and this index reaches only 0.21 and 0.17, respectively, for the mixes reinforced with sisal and coconut fibres (see Table 5 and Fig. 5(b) and (c)). Although a reduction in the toughness of the composite has occurred, an overall increase in the first crack strength was observed. This is in agreement with the observations mentioned by Akers and Studinka [16] for cellulose fibre cement composites aged in natural weather and in a CO_2-rich environment.

3.3. Embrittlement of mortar matrix reinforced with short and long fibres

The toughness of the mortar matrix reinforced with long and short fibres stored in water and subjected to natural weathering is higher than for the short fibre composites as can be seen in Fig. 6(a) and (b). However, this trend has not been followed when the specimens were exposed to 46 cycles of wetting and drying (see Fig. 6(c)). Quantitative values for the embrittlement parameters can be found in Table 5 and Fig. 7. For example, after 322 days of exposure to the open air weathering the specimens of the mix M1S2S1 retained, respectively, 75% and 87% of the values of σ_b and T_{JCI}

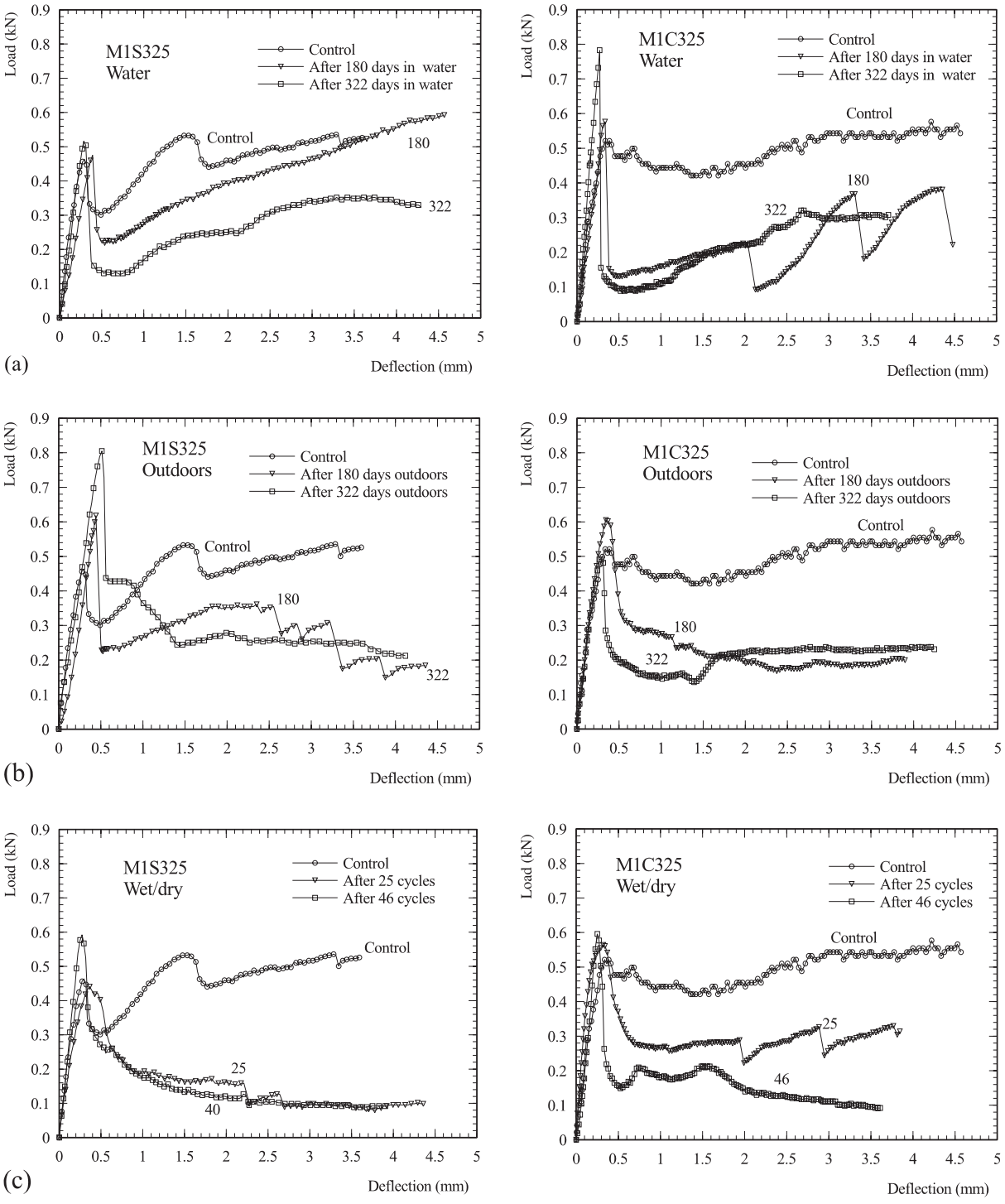


Fig. 4. Load–deflection curves with time for mixes reinforced with short fibres. (a) Stored in water; (b) aged outdoors; (c) after 25 and 46 cycles of wetting and drying.

observed for the control samples whereas the specimens submitted to 46 cycles of wetting and drying retained only 40.7% and 49.5% of those observed before ageing.

It has been found that when the M1S1S1 and M1C2S1 are kept in water up to 322 days and aged outdoors present a dominant work hardening behaviour after the appearance of the first crack as compared with the composite control specimen (Fig. 6(a) and (b)). This trend has not been observed when the same composites were subjected up to 46 cycles of wetting and drying in

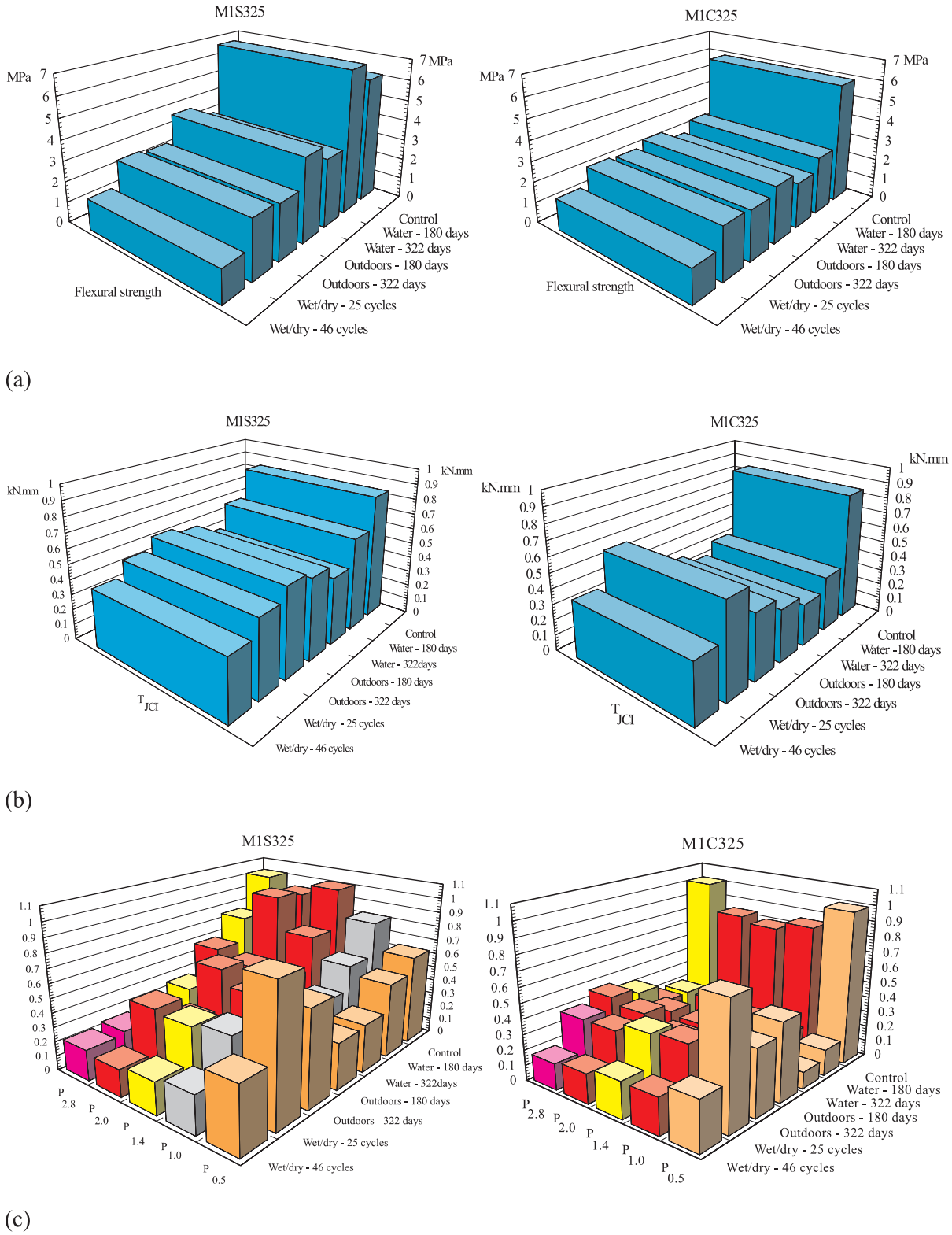


Fig. 5. Embrittlement parameters for specimens of the mixes M1S325 and M1C325. (a) Flexual strength – σ_b; (b) toughness index parameter – T_{JCI}; (c) load ratio parameter – P^*.

the laboratory. In this situation, the drop of the load due to cracking is relatively higher and the load becomes nearly constant with the increasing bending deflection.

For the specimen M1C2S1, a work hardening with a very small gradient occurred. Referring to Table 5 and Fig. 7(c), the load ratio parameter P^* up to 2.8 mm is

Table 5
First crack strength, σ_b, T_{JCI}, and P^* for specimens after various lengths of time conditioned in water, outdoors and alternately wet and dry

Mix	Ageing condition	Ageing time (days)	FCS (MPa)	σ_b (MPa)	T_{JCI} (KN mm)	$P^*_{0.5}$	$P^*_{1.0}$	$P^*_{1.4}$	$P^*_{2.0}$	$P^*_{2.8}$
M1S325	Control	28	5.47	6.09	0.83	0.63	0.81	1.00	0.92	1.01
	Water	180	5.56	6.93	0.62	0.51	0.57	0.72	0.95	0.74
	Water	322	5.42	3.31	0.44	0.33	0.40	0.48	0.51	0.57
	Outdoors	180	6.70	4.01	0.53	0.32	0.39	0.46	0.57	0.35
	Outdoors	322	8.40	2.64	0.57	0.64	0.35	0.30	0.29	0.24
	Wet/dry	25 cyc	4.81	2.77	0.49	0.91	0.49	0.45	0.48	0.23
	Wet/dry	46 cyc	6.19	1.53	0.39	0.44	0.26	0.22	0.19	0.21
M1C325	Control	28	4.96	5.85	0.83	0.99	0.84	0.79	0.83	1.01
	Water	180	6.28	2.79	0.36	0.17	0.19	0.24	0.29	0.29
	Water	322	6.98	2.17	0.27	0.11	0.09	0.14	0.17	0.29
	Outdoors	180	5.78	2.71	0.34	0.50	0.37	0.34	0.36	0.42
	Outdoors	322	5.04	2.37	0.42	0.43	0.35	0.34	0.42	0.45
	Wet/dry	25 cyc	4.94	2.45	0.60	0.79	0.45	0.44	0.38	0.38
	Wet/dry	46 cyc	5.86	1.56	0.37	0.32	0.23	0.23	0.19	0.17
M1S2S1	Control	28	6.11	7.52	0.99	0.87	0.89	0.94	1.02	1.08
	Water	180	5.78	5.65	0.84	0.45	0.81	0.81	0.88	0.69
	Water	322	6.68	7.15	0.82	0.43	0.60	0.69	0.87	0.78
	Outdoors	180	6.27	6.94	1.10	0.55	0.77	0.92	0.90	0.88
	Outdoors	322	5.20	5.60	0.86	0.59	0.70	0.95	0.79	0.67
	Wet/dry	25 cyc	5.61	6.40	0.81	0.72	0.72	0.74	0.87	0.97
	Wet/dry	46 cyc	6.03	3.06	0.49	0.46	0.39	0.39	0.46	0.47
M1C2S1	Control	28	5.69	7.42	0.99	0.99	0.99	1.04	1.02	1.11
	Water	180	6.43	6.39	0.72	0.40	0.53	0.64	0.80	0.82
	Water	322	6.78	7.52	0.73	0.42	0.57	0.69	0.88	1.08
	Outdoors	180	5.58	6.38	0.85	0.72	0.78	0.92	0.84	0.87
	Outdoors	322	6.05	5.00	0.66	0.62	0.49	0.56	0.63	0.65
	Wet/dry	25 cyc	5.79	5.51	0.73	0.58	0.55	0.65	0.81	0.76
	Wet/dry	46 cyc	6.31	3.79	0.57	0.58	0.39	0.39	0.51	0.56

about 0.46 for the mix M1S2S1 and about 0.56 for the mix M1C2S1.

Comparing the behaviour under ageing of the mixes M1S325 and M1S2S1 it can be seen that the mix reinforced with long fibres presented a smaller reduction in the embrittlement parameters. This fact suggests that the short fibres become mineralised earlier than the long fibres. One of the reasons could be attributed to the fact that in the short fibre-reinforced composites there are more end points which create facilities for the penetration of cement hydration products accelerating the loss of flexibility of the fibres.

4. Microstructural studies

4.1. Analysis of the backscattered images

In Fig. 8(a) comparison is made between backscattered electron images of the short coconut fibres obtained from unaged specimens with those submitted to 25 cycles of wetting and drying. Observing the microstructural characteristics of the interface of the composites, it can be seen that the matrix around the fibres of the unaged specimen (Fig. 8(a) and (b)) is relatively more porous and presents more cracks than that of the aged specimen (Fig. 8(c) and (d)). It can also be seen that the mortar zone delineated by the fibre surface and the crack around the fibre perimeter is larger in an unaged specimen than in the specimen submitted to the cycles of wetting and drying. A somewhat weak matrix in the vicinity of the fibre, which can yield or crack, can be favourable from the point of view of the ductility of the fibre composite. The densening of the matrix around the fibres or fibre embrittlement due to migration of hydration products from the matrix to the fibres could be the main factor contributing to the increase in the first crack strength and reduction in toughness, by reducing the flexibility and deformation capacity of the fibres. Stronger, more rigid and brittle fibres, and improved bond strength, are known to lead to composites of higher strength and lower ductility.

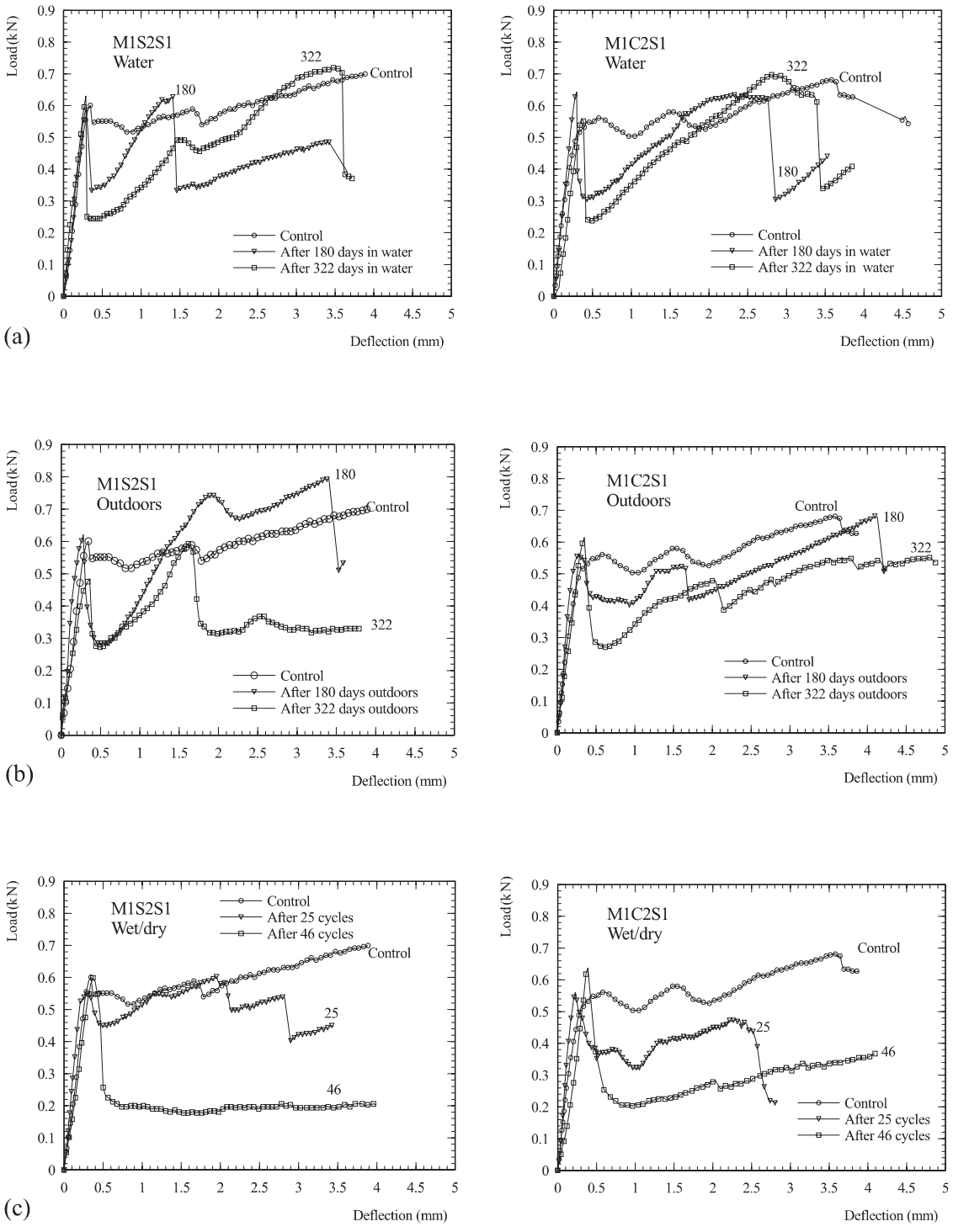


Fig. 6. Load–deflection curves with time for mixes reinforced with short and long fibres. (a) Stored in water; (b) aged outdoors; (c) after 25 and 46 cycles of wetting and drying.

In order to verify whether cement products had migrated to the lumen, pores and spaces of the fibres, a dotting map of chemical elements was carried out. The chemical elements searched were calcium, Ca, sulphur, S, silicon, Si, and sodium, Na. Fig. 9 presents the fibre–matrix interfacial zone where the dot mapping of elements was carried out.

Fig. 10 shows the dotting map of chemical elements. Results show that there is a considerable transport of cement chemicals, mainly calcium products, to the

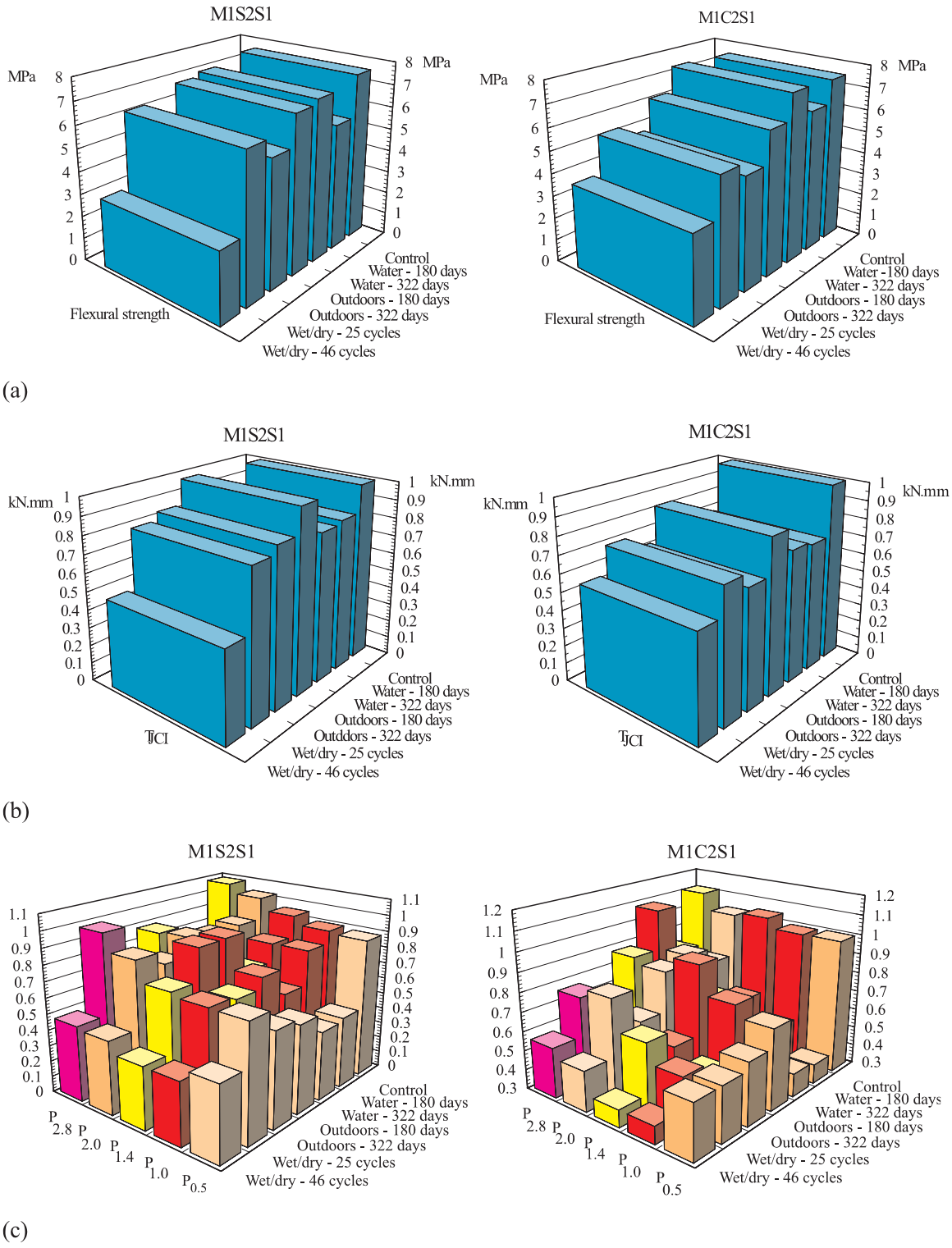


Fig. 7. Embrittlement parameters for specimens of the mixes M1S2S1 and M1C2S1. (a) Flexural strength parameter – σ_b; (b) toughness parameter – T_{JCI}; (c) load ratio parameters – P^*.

fibres during the cycles of wetting and drying. These results confirm the speculation of Gram [1] and Singh [2] about the mineralisation of the sisal fibre by calcium hydroxide deposited in the lumen and voids of the fibre cells. They also confirm the findings of Bentur and Akers [17,18] about the mineralisation of wood

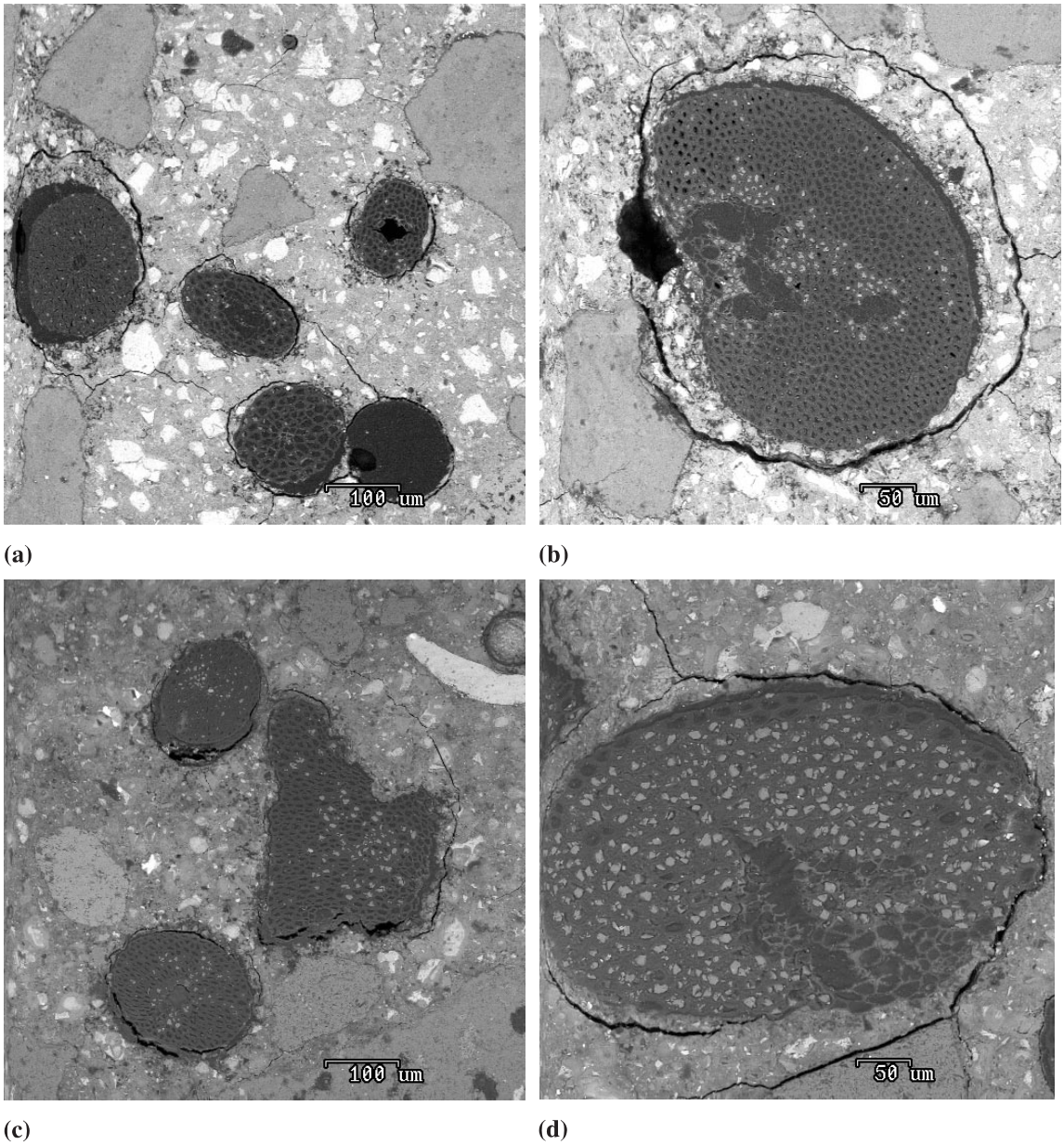


Fig. 8. Backscattered images of cross-sections of coconut fibres. (a) Images from a 28-day specimen; (b) higher magnification of the typical fibre–matrix interface before ageing; (c) images from a specimen submitted to 25 cycles of wetting and drying; (d) higher magnification of the typical fibre–matrix interface after 25 cycles of wetting and drying.

fibres that had their core filled with hydration products after natural ageing and accelerated ageing in a CO_2-rich environment. Thus, the natural and wood fibres, which are originally a flexible and low modulus reinforcing unit, become stiff. This change in the fibre structure can account for increase in strength at first crack and reduction in toughness, during ageing.

Fig. 11 compares backscattered electron images of cross-section of long sisal fibres obtained from specimens 28 days old with those submitted to 25 cycles of wetting and drying. When observing the microstructural characteristics of the interface of the aged and unaged composites (Fig. 11(a)–(d)), no significant differences can be noted in matrix porosity and volume of matrix cracks around the fibres.

A dotting map of chemical elements was also executed in order to verify whether transport of cement products had already happened to the lumen and voids in the fibre cells. The chemical elements mapped were calcium, sulphur, silicon and aluminium. Fig. 12 presents the fibre–matrix interface where the mapping of

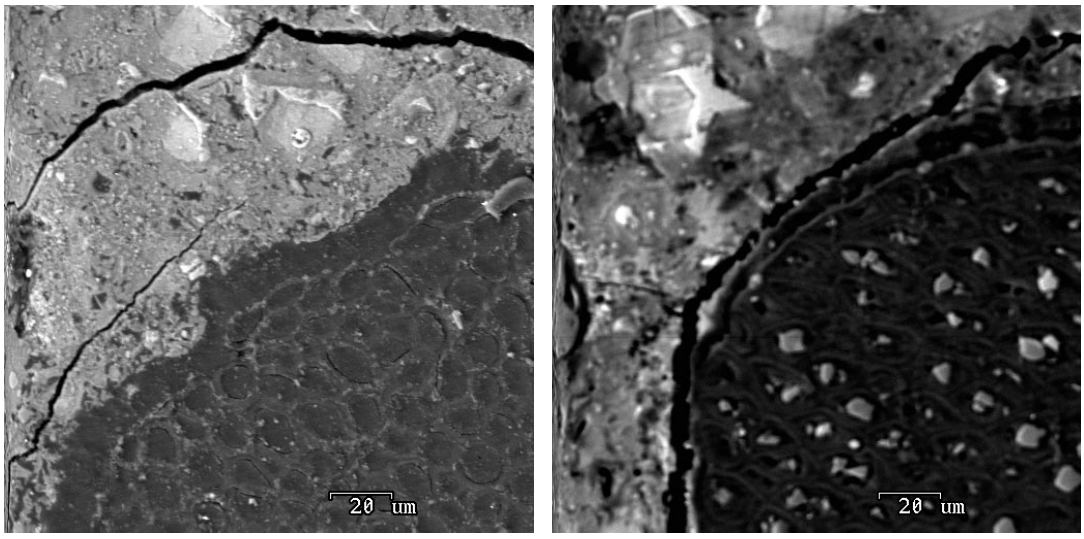


Fig. 9. Fibre–matrix interfacial zone where the dot mapping of elements was carried out. (a) Coconut fibre–mortar interface zone for a specimen 28 days old; (b) coconut fibre–mortar interfacial zone for a specimen subjected to 25 cycles of wetting and drying.

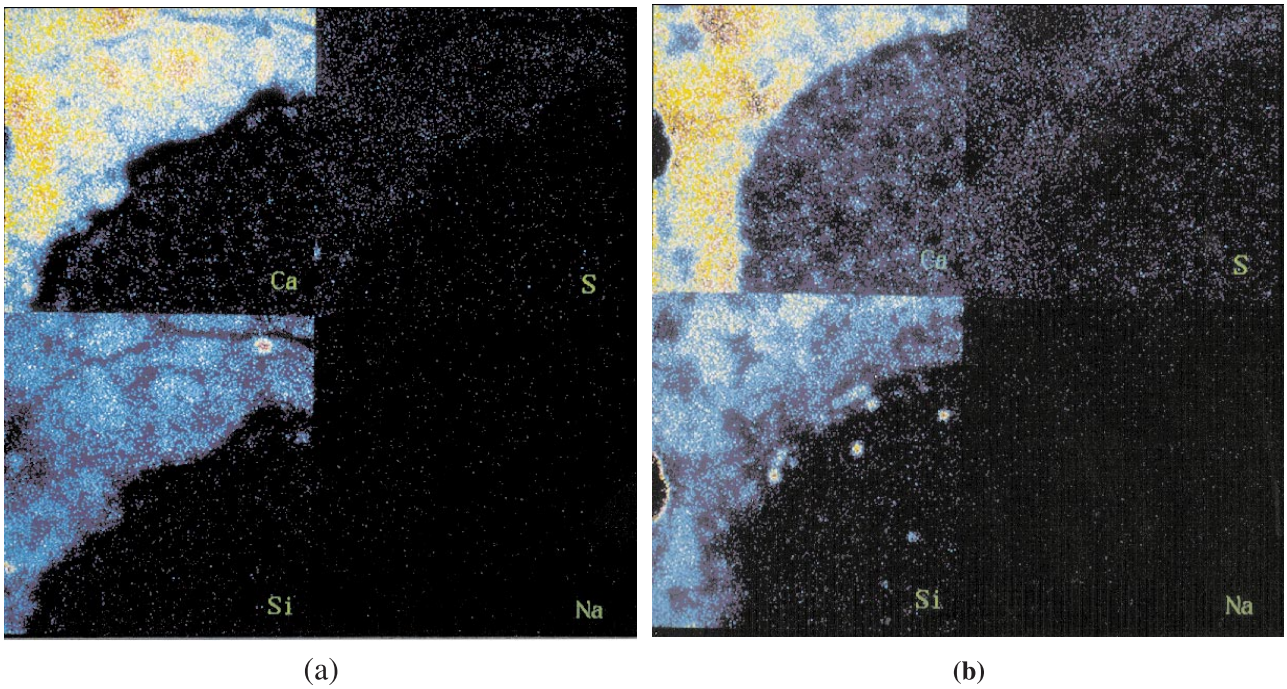


Fig. 10. Dotting map of chemical elements in coconut fibre–mortar interface. (a) Mapping of chemical elements for the specimen 28 days old (reference); (b) mapping of chemical elements for the specimen submitted to 25 cycles of wetting and drying.

elements was conducted. Fig. 13 shows the dotting map of chemical elements. The results do not show significant differences between the maps obtained for the long sisal fibre at 28 days and after the first 25 cycles of wetting and drying.

4.2. Analysis of the SEM images

Scanning electron micrographs of coconut fibres showing the surface changing with ageing are presented in Fig. 14. The micrographs reveal that some deterio-

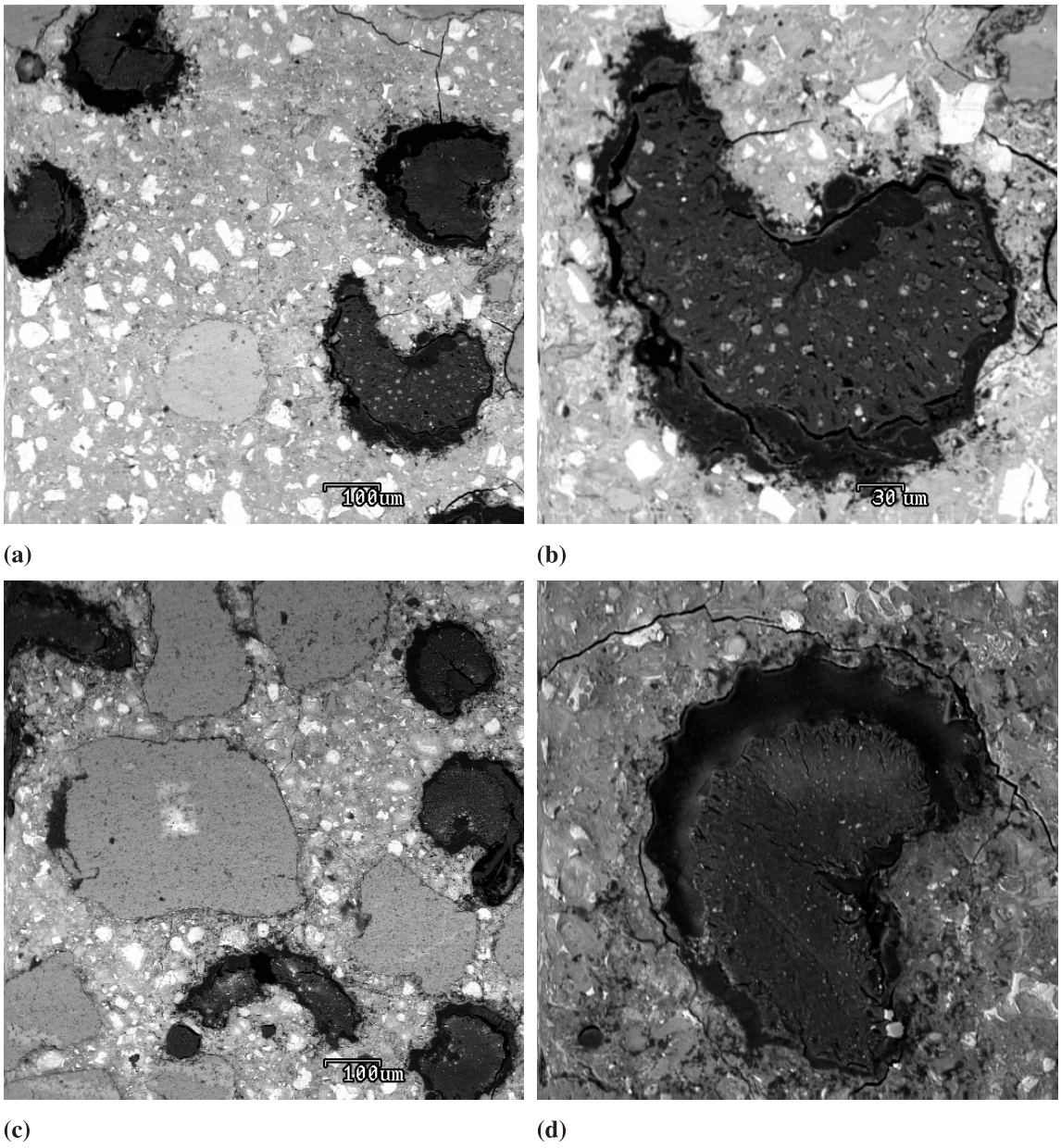
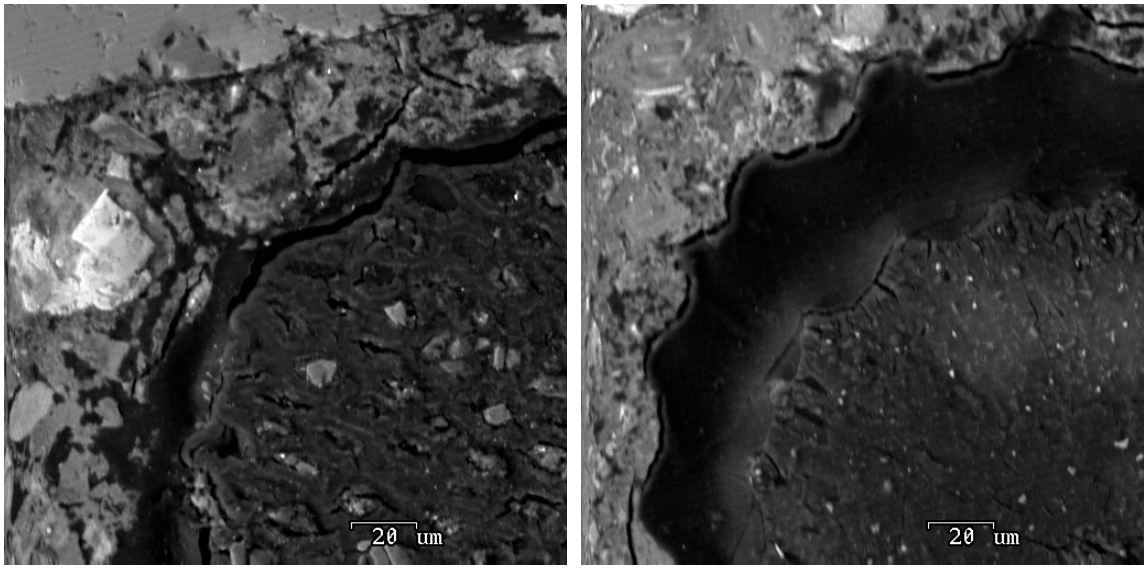


Fig. 11. Backscattered images of cross-section of long sisal fibres before and after ageing. (a) Images from a 28-day specimen; (b) higher magnification of a typical fibre–matrix interface before ageing; (c) images from a specimen submitted to 25 cycles of wetting and drying; (d) higher magnification of a typical fibre–matrix interface after 25 cycles of wetting and drying.

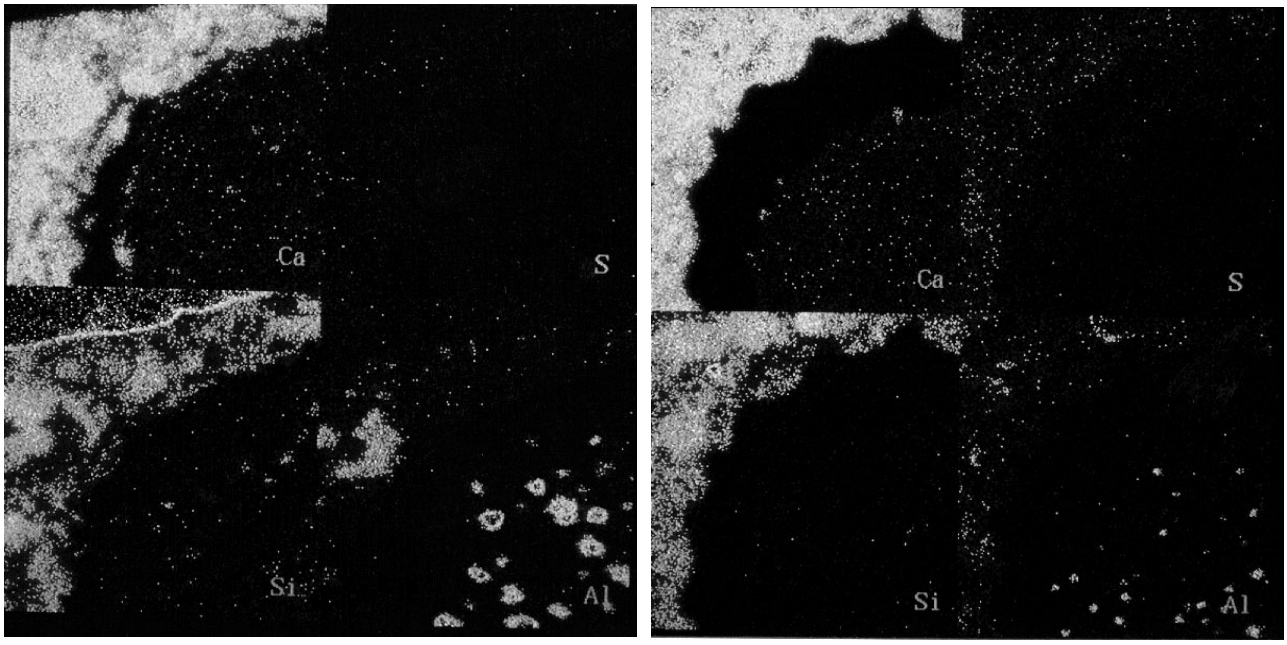
ration occurred in the fibre surface. These images, however, do not indicate enough superficial abrasion to cause fibre decomposition and, thus, fibre mineralisation seems to be the main mechanism of embrittlement of VFRMC with ageing. Visual observations on the failure surface of the composites after 46 cycles of wetting and drying indicated that the pull-out length of the fibres from aged specimen was considerably smaller than that observed from unaged samples and that the fibres in the aged specimens could be easily broken off at their anchorage in the mortar. The fibres also demonstrated a loss of flexibility, being easily broken by finger force.

Scanning electron micrographs of sisal fibres showing the surface changing with ageing are presented in Fig. 15. The micrographs do not reveal a considerable deterioration of the fibre surface after ageing, although the surface of the fibre extracted from the specimen cured in water indicates some alteration. Visual observations on the failure surface of the composites after 46 cycles of



(a) (b)

Fig. 12. Sisal fibre–mortar interface where the dotting map of chemical elements was executed. (a) Sisal fibre–mortar interface for a specimen 28 days old; (b) sisal fibre–mortar interface for a specimen subjected to 25 cycles of wetting and drying.



(a) (b)

Fig. 13. Dotting map of chemical elements obtained for the mix M1S2S1. (a) Mapping of chemical elements for the specimen 28 days old (reference); (b) mapping of chemical elements for the specimen subjected to 25 cycles of wetting and drying.

wetting and drying indicated that the pull-out length of the fibres from aged specimen was smaller than that observed from unaged samples. The long fibres also demonstrate a loss of flexibility and could be easily broken off at their anchorage in the mortar.

5. Conclusions

Based on the results presented in this paper it can be concluded that the vegetable sisal and coconut fibres are highly sensitive to the alkalinity of the cementitious

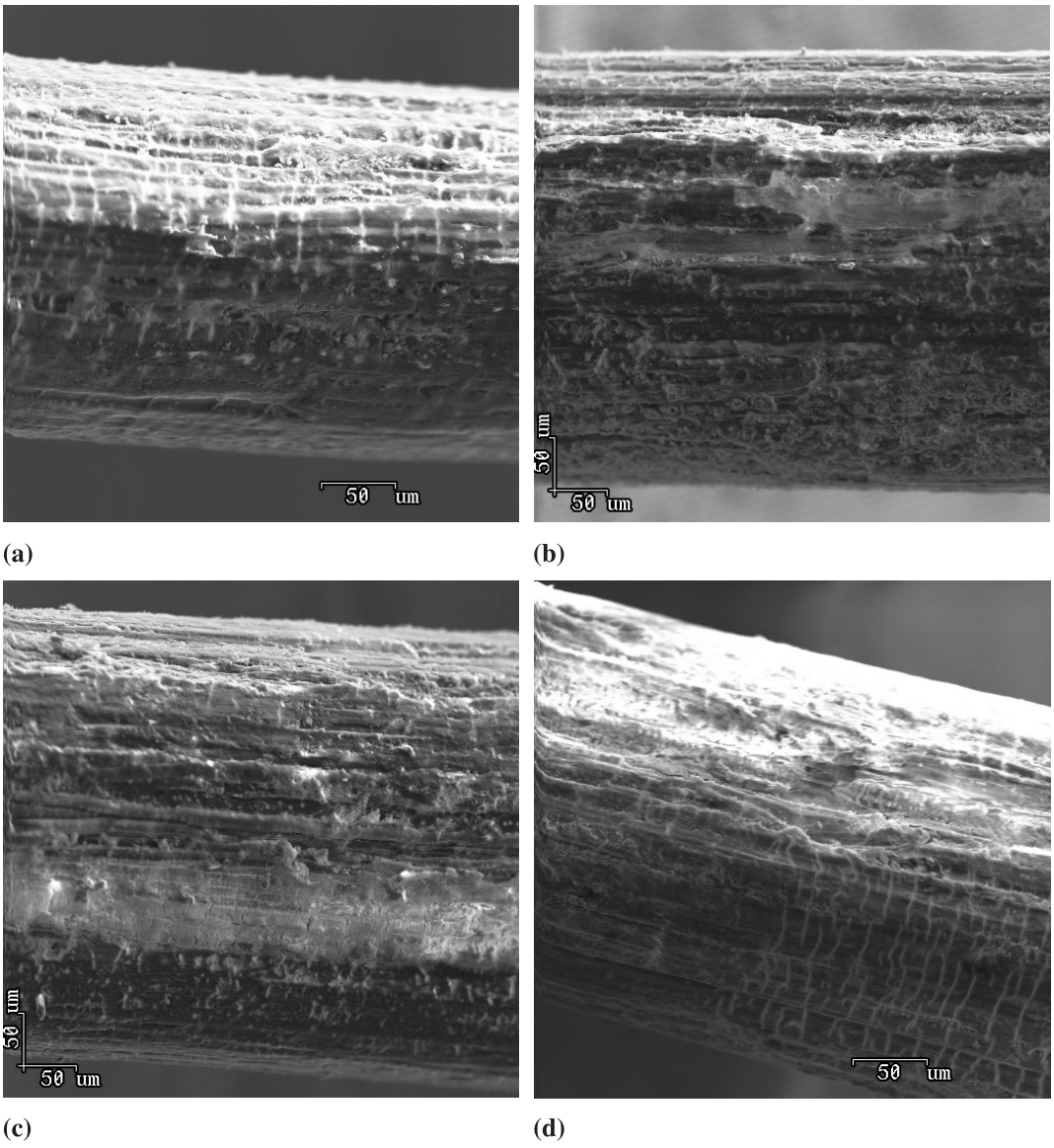


Fig. 14. Scanning electron micrographs of coconut fibres showing the surface of the fibre before and after ageing. (a) Before ageing; (b) fibre extracted from a specimen aged for 322 days in tap water; (c) fibre extracted from a specimen aged for 322 days outdoors; (d) fibre extracted from a specimen submitted to 46 cycles of wetting and drying.

matrix. Sisal and coconut fibres immersed in a calcium hydroxide solution with pH 12 for 300 days lost completely their flexibility and could be pulled apart fairly easily by finger force. This can be attributed mainly to crystallisation of lime in the lumen, walls and voids in the fibre. The extent of alkaline attack was smaller when the fibres were conditioned in the sodium hydroxide solution of pH 11. Fibres conditioned in tap water also lost strength with time, probably due to microbiological action. The results show that after 420 days, sisal and coconut fibre retained, respectively, 83.3% and 77.2% of their original strength.

Composites manufactured with short sisal or coconut fibres and OPC mortar matrix presented a significant reduction in toughness after six months of ageing outdoors or submitted to cycles of wetting and drying. Some embrittlement was also observed in specimens aged in water. The embrittlement is mainly associated with the mineralisation of the fibres due to migration of hydration products, especially calcium hydroxide, to the fibre lumen, walls and voids.

Embrittlement of composites manufactured with short sisal fibres was greater than that observed in specimens reinforced with long fibres. This may be attributed to higher number of end points and surface area of the short fibres that allow a faster penetration of cement hydration products and consequent mineralisation of the fibres.

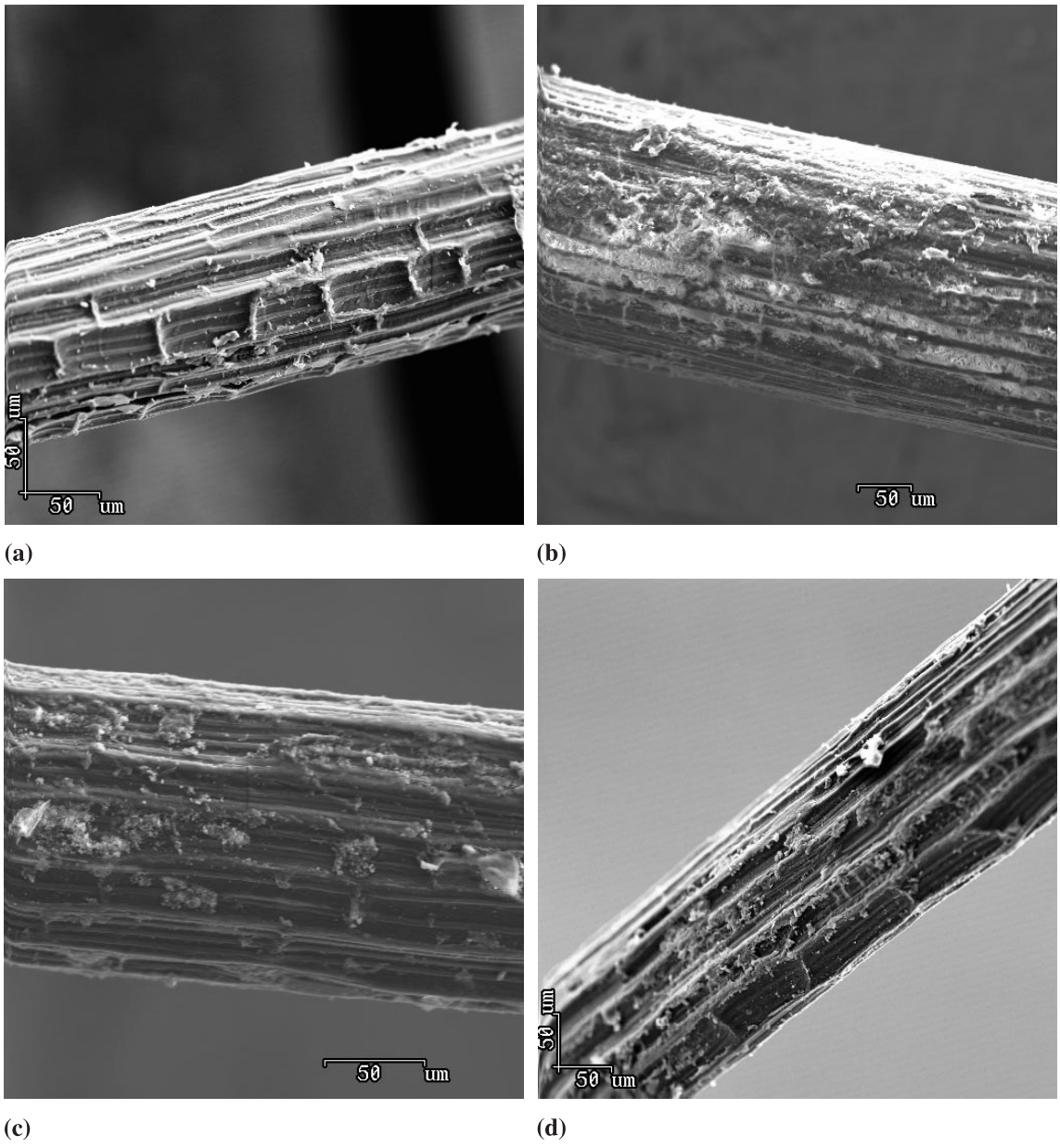


Fig. 15. Scanning electron micrographs of sisal fibres before and after ageing. (a) Before ageing; (b) surface of a fibre extracted from a specimen aged for 322 days in tap water; (c) surface of a fibre extracted from a specimen aged for 322 days outdoors; (d) surface of a fibre extracted from a specimen submitted to 46 cycles of wetting and drying.

To enhance the durability of the composites, several treatments have been studied, including: (i) modifications to the matrix through the replacement of certain part of Portland cement by undensified silica fume and by blast-furnace slag; (ii) carbonation of the cementitious matrix and (iii) immersion of the fibres in slurry silica fume prior to being incorporated in the Portland Cement matrix and the results will be presented in a forthcoming paper [19].

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