

## Influence of curing conditions on durability of alkali-resistant glass fibres in cement matrix

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MS received 16 October 2009; revised 13 July 2010

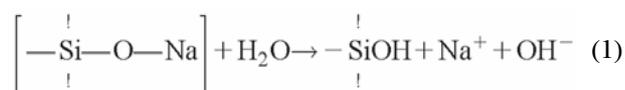
**Abstract.** Glass fibres in concrete material often increase the flexural strength. However, these fibres when in contact with cement are altered by alkali reactions due to the presence of portlandite. This study presents the results of investigation to show the effect of curing conditions on the durability of alkali-resistant glass fibres in cement matrix. Test results show that even alkali resistant fibres treated with zirconium oxide present the same degradation phenomenon. They also show that the nature of the cement has a large influence on the protection of the fibres: the Portland CEM II is less damaging than the CEM I. The substitutions of a part of cement by silica fume gave no substantial improvements to the mechanical strength of the glass fibre reinforced cement (GFRC). However, the observed microstructures in the samples show that the degradation is weakened with the addition of silica fumes. The analytical techniques used in this study are scanning electron microscope (SEM) and X-ray diffraction.

**Keywords.** Glass fibre; cement matrix; durability; curing conditions; silica fume.

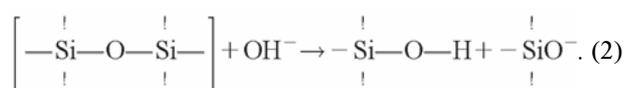
### 1. Introduction

The studies on the glass fibre reinforced cement (GFRC) showed the low durability of the fibres in the cement matrix (Daniel and Schultz 1986; Majumbar and Walton 1991; Graham 2004; Bentur and Mindess 2007). Alkali resistant glass fibres, called CemFil, have been developed using zirconium oxide ( $ZrO_2$ ) additives that inhibit the cement matrix attacks. However, they are still sensitive to a humid environment; i.e. a decrease in the flexural strength is usually observed (Kosa *et al* 1991; Xu *et al* 1998). The degradation of strongly siliceous fibres is a result of the composite internal behaviour where the cementitious matrix constitutes an aggressive environment. Although the causes of this phenomenon are still not well known, chemical and physical factors were highlighted to describe the loss of resistance and ductility of the composite in humid environments.

Charles (1958) pointed out that glass corrosion constitutes the chemical factor of the glass dissolution in humid environments. This study suggest that only the terminal end, which associates  $Na^+$  ion to the glass network, is responsible for dissolution. This initial dissolution or 'leaching' occurs according to (1). Therefore, the more alkaline is added to the bulk of the glass structure, the more it becomes chemically reactive in humid environments



The oxygen–sodium bond near the interface is broken by the removal of the  $Na^+$  ion and the remaining oxygen atom captures the hydrogen ion  $H^+$ . This causes additional stress on the glass surface because the  $H^+$  ion is larger than the  $Na^+$  and hence producing cracks, especially under the effect of the tensile forces. These cracks can get larger and propagate into the inner parts, resulting in the failure of the specimen. Free hydroxyl ion is formed in the process and enables the second reaction, (2), to take place



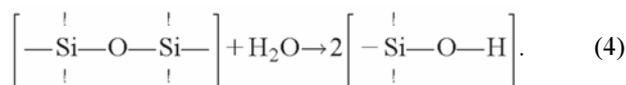
The  $SiO^-$  which is one of the by-products of the previous equation can dissociate another water molecule as shown in (3) and the  $SiOH$  by-product forms a gel on the surface of the bar, slowing down the process



As seen from the above three equations, the excess of hydroxyl ions production will increase the pH of the solution. From (4), it is clear that these ions attack the fibre surface resulting in flaws that significantly degrade strength and can result in premature fracture and failure

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of the fibres (Gonenc 2003). This reaction can also occur during the dissolution of the glass in water



However, since the glass structure has many terminal ends, the occurrence of the first three equations is much more probable.

The chemical reactions that cause glass fibre degradation are also function of alkalinity, degree of moisture, diffusivity, and void ratio (Schutte, 1994). Furthermore, the physical factor of glass fibre degradation is a result of the portlandite  $[\text{Ca}(\text{OH})_2]$  deposition and growth. Portlandite is formed during the cement hydration that fills spaces between and around the glass fibre filaments, thus, cementing them together and reducing their flexibility. This causes excessive bonding and local concentration of stresses under load at the surface of fibres, resulting in embrittlement of GFRC (Hayashi *et al* 1985; Purnell *et al* 1999).

Several techniques are used to protect fibres. One of these is to protect the fibres either by using a low alkalinity cement (Marikunte *et al* 1997; Purnell *et al* 2000; Krüger *et al* 2003; Péra and Ambroise 2003; Cuyper *et al* 2006) or by densifying the interface between fibres and matrix with polymers (PVA, AC or VAC) to prevent the lime diffusion into the fibres (Rols *et al* 2000; Li *et al* 2002). Another technique consists in limiting the alkali-silica reaction by reducing the amount of portlandite into the matrix under pozzolanic effect. For instance, fly ashes, silica fumes or granulated blast furnace slag could be added to conventional concrete in order to substitute a part of the cement (Gudmundsson and Olafsson 1999; Shehata and Thomas 2000; Paya *et al* 2007).

The aim of this study is to analyze the behaviour of fibres in the cement matrix with or without silica fume addition under different storage conditions. The fibres are observed by scanning electron microscopy (SEM) and the portlandite is quantified by X-ray diffraction.

## 2. Experimental

### 2.1 Materials

**2.1a Matrix materials:** The CEM II/B 32.5R and CEM I 52.5 were the two types of cements used in this study and manufactured by Ciment Lafarge Ltd (France), conform to EN 196-1 (the European standard). Their chemical composition is given by the manufacturer and presented in table 1. Also, the sand used was a siliceous fraction that did not exceed 500  $\mu\text{m}$  in size. Silica fume (a byproduct of the semiconductor industry brought from Bretagne area in France) was added for a series so as to reduce the amount of portlandite by pozzolanic effect (table 3). The silica fume fraction varied between 30 and

100 nm in size and its chemical composition is given in table 1. To improve the fresh mixture workability, the superplasticiser Sikament FF86, conform to NF 9342 (French standard) was added.

**2.1b Fibers:** The studied glass fibres were alkali resistant (A-R glass fibres), from Saint Gobain Vetrotex manufacturer. The fibres (12 mm in length) were  $\text{ZrO}_2$ -rich (16.6 weight %) and this could protect the fibres from the alkaline attacks of the cement matrix. The main physical and mechanical properties of these fibres are shown in table 2.

### 2.2 Sample preparation and tests

Six samples of glass fibre reinforced matrix mixtures of  $4 \times 4 \times 16 \text{ cm}^3$  were prepared and their composition is given in table 3. Samples 1–3 were prepared with CEM II/B 32.5 cement and samples 4–6 with CEM I 52.5. The rate of silica fume added to samples 1–3 was very low (3%) since the CEM II/B 32.5 cement contained already 24 wt% fly ash. On the other hand, the amount of silica fumes added to samples 4–6 was limited to 5% for comparison.

The glass fibre reinforced matrix samples were removed from mould after 24 h. They were conserved in moist room (temperature of 20°C and humidity of 95%) for 7 days, then maintained in different environments for one year: either in moist room ( $T = 20^\circ\text{C}$ , humidity = 95%), in air conditioned room considered as dry

**Table 1.** Chemical composition (weight %) of the cements and of the silica fumes: according to the manufacturer.

Composition (wt.%)	CEM II/B 32.5R	CEM I 52.5	Silica fume
$\text{SiO}_2$	25.05	19.7	97.64
$\text{Al}_2\text{O}_3$	8.6	4.9	
$\text{Fe}_2\text{O}_3$	3.5	2.8	0.1
CaO	55.4	64.5	0.14
MgO	0.9	0.9	0.15
$\text{K}_2\text{O}$	1	0.9	0.39
$\text{Na}_2\text{O}$	0.15	0.2	0.14
$\text{SO}_3$	2.7	3	
$\text{S}^-$	<0.01	<0.01	
$\text{Cl}^-$	0.022	0.01	
Insoluble	11.8	0.2	
Loss on ignition	2.2	2.7	
$\text{TiO}_2$	–	–	
MnO	–	–	
$\text{P}_2\text{O}_5$	–	–	
$\text{Mn}_2\text{O}_3$	–	–	
Undosed	0.468	0.38	
Total	100	100	
$\text{CO}_2$	1	1.9	
Free CaO	1.2	1.4	
Active alkalines	0.75	–	
Gypsum content	5	5	

**Table 2.** Physical and mechanical properties of AR glass fibre.

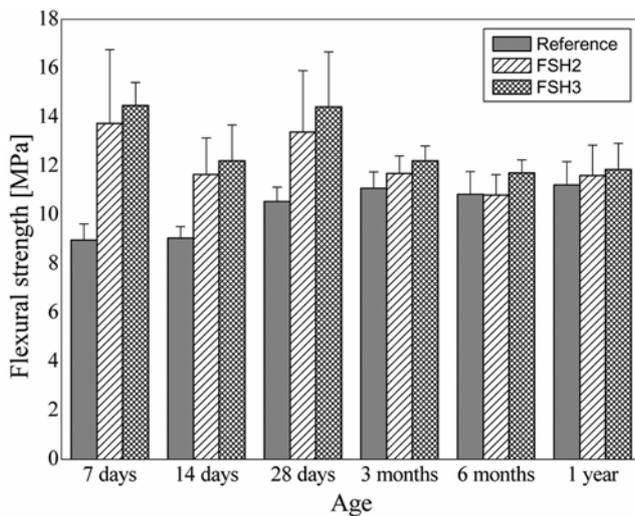
Properties	
Density	2.68 g/cm <sup>3</sup>
Tensile strength: virgin filament	3000 MPa
Tensile modulus	73000 MPa
Elongation at break	4.30%
Coefficient of linear thermal expansion (between 20 and 100°C)	5.10 <sup>-6</sup> m/m/K
Flammability	773°C

**Table 3.** Compositions of the used mortar series.

Constituents (weight %) and ratios	CEM II/B 32.5R			CEM I 52.5		
	1	2	3	4	5	6
Cement/sand	1	1	1	1	1	1
Water/cement	0.35	0.35	0.35	0.35	0.35	0.35
Fibres	0	2	2	0	2	2
Silica fumes	0	0	3	0	0	5
Superplasticizer	0	0	2	0	0	2

**Table 4.** Samples formulations and storage conditions.

Used cement	Samples	Fiber rates (weight %)	Silica fume (weight %)	Storage conditions
CEM II	FSH2	2	–	Moist room
	FCA2	2	–	Ambient air
	FSH3	2	3	Moist room
	FCA3	2	3	Ambient air
	FSC2	2	–	Dry environment
	FSC3	2	3	Dry environment
CEM I	FSH4	2	–	Moist room
	FSH5	2	5	Moist room
	FCA5	2	5	Ambient air



**Figure 1.** Flexural strength development in moist room curing.

environment ( $T = 20^{\circ}\text{C}$ , humidity = 50%), or in ambient air conditions. The sample formulations and conservations are given table 4.

The flexural strength (four-point loading test) was measured at periods of 7, 14, 28, 90, 180 and 360 days. Each value represented the average of 6 tests.

The pH of pore solutions were determined after one year of storage by a pH-meter. The tests were carried out on diluted solutions where the ratio of solid/demineralised water was 1/10 and the temperature is  $20^{\circ}\text{C}$ .

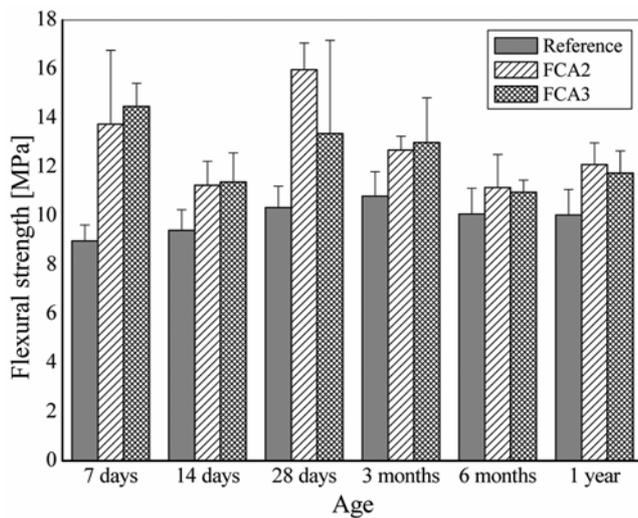
The phase composition of each sample was determined by X-ray diffraction (XRD) and the microstructures were examined using both a scanning electron microscope (SEM field effect) and an energy dispersive X-ray analysis (EDX) coupled with a conventional microscope.

### 3. Results and discussion

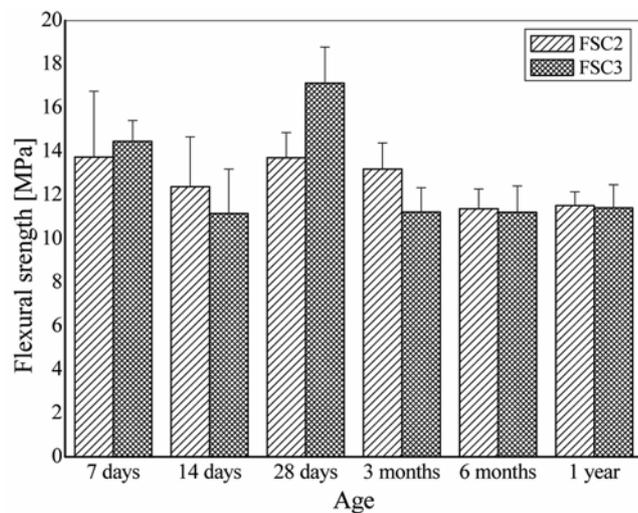
#### 3.1 Environment effect on the mechanical resistance

From figures 1–3 that show the flexural strength behaviour of cement CEM II 32.5R with age in moist room, ambient air, and dry environment conditions, it is clear that there is a marked reduction in flexural strength for all the samples reinforced by A–R glass fibres in the three conservation mediums. Also, in the same conditions, statistical data analyses of flexural strength show a rather high dispersion of the fibre-containing samples than that of the reference samples (i.e. without fibres). This dispersion seems to be related to the effect of the randomly dis-

tributed fibres which make the matrix more heterogeneous. It is also noticed that between the 7th and the 14th day, there is a marked drop of flexural strength in the fibre-containing samples followed by a subsequent increase on 28th day within the three medium conditions. This drop of flexural strength is explained by the alkaline attack of cement at the surface of the fibre beams after 14 days of conservation. Figure 4 shows that the fibres in contact with the matrix are highly corroded, while the fibres which are not in contact with the matrix are not very altered. On these last fibres, some small and scattered pittings are visible. The protective layer of fibres seems to be strongly affected by the alkaline attack which probably have a major effect on the interface, and consequently on the fibre–matrix bond. Figure 4 also shows

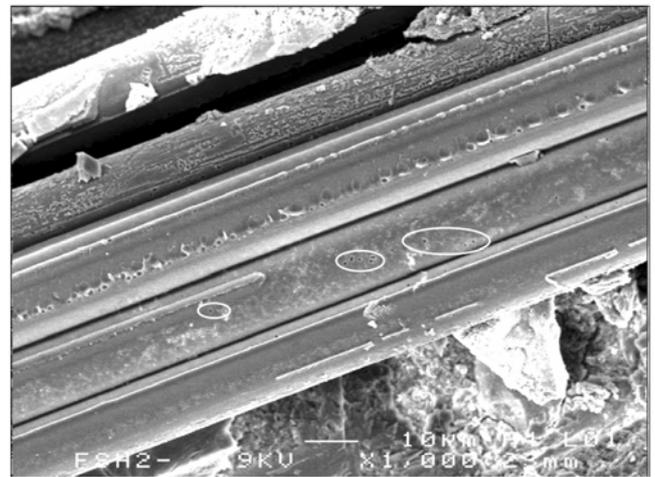


**Figure 2.** Flexural strength development in ambient air curing.

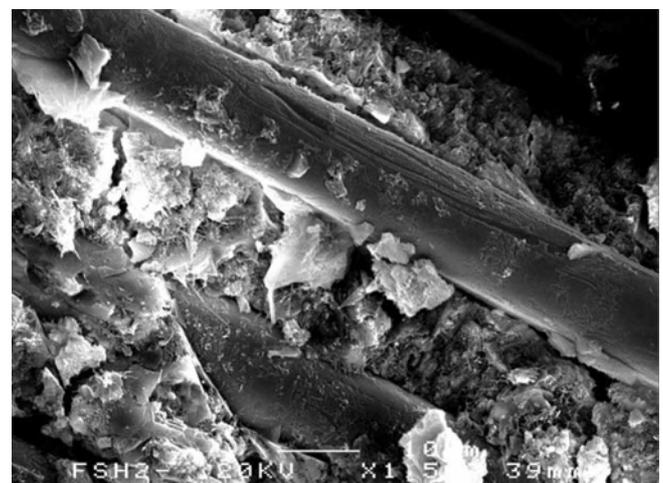


**Figure 3.** Flexural strength development in dry environment curing.

that the matrix has not yet acquired a perfect compact structure. The increase in strength in 28 days could be attributed to the development of a proper bond between fibres and cement matrix. Afterwards, the flexural strength decreases gradually until the 90th day of testing. This drop of flexural strength is attributed to the diffusion of the calcium hydroxide into the spaces between fibres as shown in figure 5. This was reported by many authors (Hayashi *et al* 1985; Purnell *et al* 1999). Two modes of attack on the glass fibres are evident: hydroxylation and notching by  $\text{Ca}(\text{OH})_2$  crystals. The surface of fibres becomes generally hydroxylated due to the high pH of the matrix, but notching attack is concentrated at points where a fibre is in good contact with  $\text{Ca}(\text{OH})_2$  crystals,



**Figure 4.** Effect of alkaline attack on AR glass fibres in cement matrix after 14 days of conservation (top-left-hand side). Note that in the middle of the figure the fibres are not very altered.

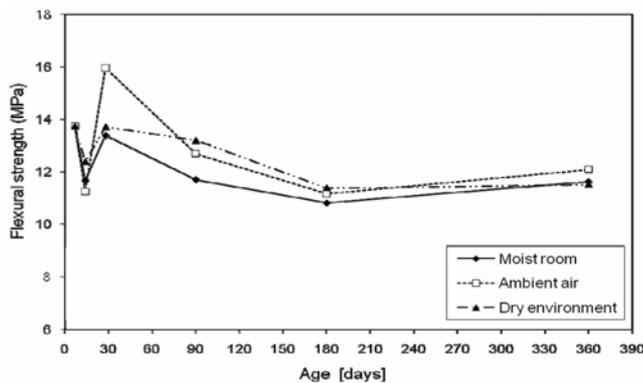


**Figure 5.** Alkali-resistant glass fibres in cement matrix after 90 days. Filling of the spaces between the filaments by calcium hydroxide. Note surface corrosion and notching by  $\text{Ca}(\text{OH})_2$ .

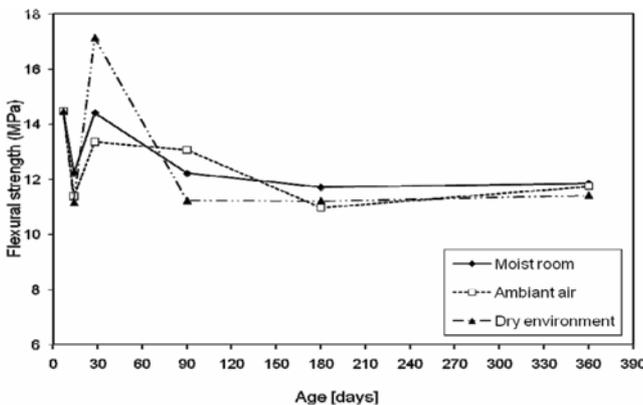
leading to their impingement on and into fibres (Yilmaz and Glasser 1992).

For the three conservation conditions, a drop of flexural strength in fibre-containing samples is observed between the 28th and 90th day; but after the 6th month, flexural strength seems to stabilize but shows a slight increase for some conservation conditions. Although there are significant reductions in strength of the GFRC after one year of hardening, the flexural strength values show that these composites tend always to give better performances in flexure than does of the reference sample.

Figures 6 and 7 show the evolution of flexural strength of the fibre-containing samples with and without silica fume under three curing conditions. The substitution of cement CEM II 32.5 at the rate of 3% by the silica fume, to reduce the alkalinity of cement and to protect A-R glass fibres from the attacks modes, gave no substantial improvements in resistances gain and also did not preserve the mechanical resistance obtained at the first month of hardening. Moreover, the inclusion of a mineral



**Figure 6.** Flexural strength development under various mediums of conservation for samples containing 2% fibre.



**Figure 7.** Flexural strength development under various mediums of conservation for samples containing 2% fibre and 3% silica fume.

addition such as the silica fume has a slow reaction effect with calcium hydroxide (pozzolanic effect), whereas the kinetics of degradation of glass fibres occur earlier. The effect of silica fume seems to appear after the 6th month of conservation. This effect is observed only for the samples conserved in moist room. It is evident that the pozzolanic effect is favoured by the presence of the interstitial solution in the porosity of the samples in high wet conditions. It should be noted that the addition of silica fume is beneficial at the interface where it is most needed to delay or prevent the development of portlandite formation. However, if the silica fume with very fine particle size ( $<0.1 \mu\text{m}$ ) penetrate the interfilament spaces where the free lime deposition mechanism has taken place and become even harder. This results in a more damaging CSH within the fibre bundle.

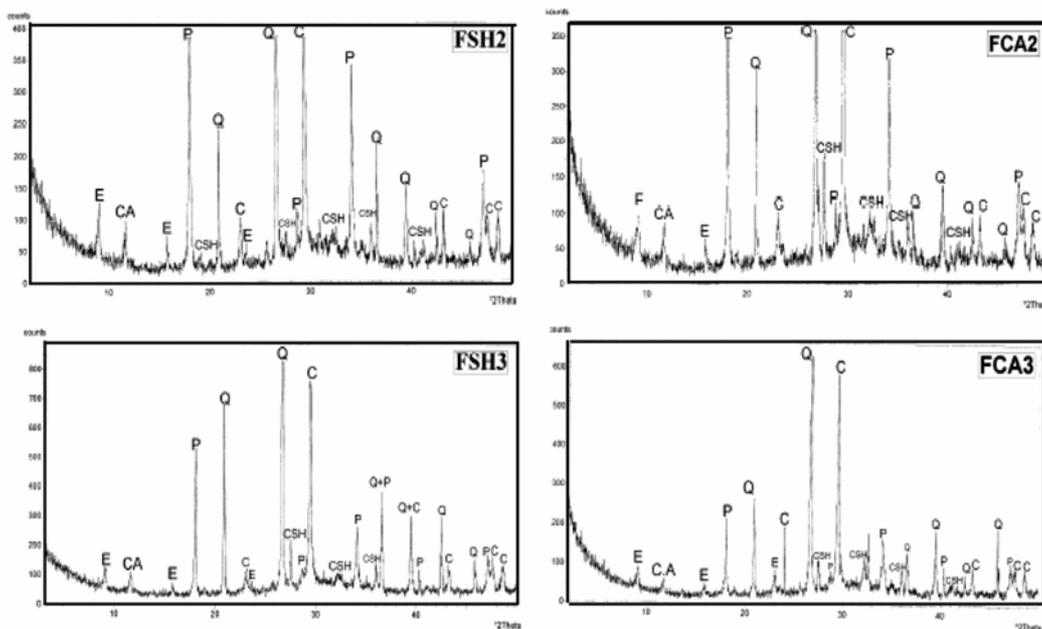
The samples conserved between 6 months and 1 year at ambient air and in dry environment have shown an improved flexural strength. The improvement in strength beyond 6 months is probably due to the portlandite transformation into calcite. This is accompanied by an increase in crystalline volume which results in a decreasing porosity of the cement paste. This can only limit the diffusion of the calcium hydroxides in the inter-fibre spaces as is pointed out by some authors (Purnell *et al* 2000, 2003). This hypothesis of carbonation supposes that the portlandite was not consumed completely by the silica fume, due probably to the lack of interstitial solution in the pores of the matrix to initiate the pozzolanic effect between the portlandite and the silica fume.

In this article, the flexural strength of CEM I 52.5 cement is not the subject of a comparison with cement CEM II 32.5R which are different cement types. The comparison is made only on observations by scanning electron microscopy.

### 3.2 Quantitative analysis of portlandite

The X-ray diffraction ( $\lambda K_{\alpha}$  Cu, Ni filter, step to step) has been performed on four samples: FSH2 and FSH3 conserved in moist room, FCA2 and FCA3 conserved at ambient air. The X-ray patterns are given in figure 8. The peak intensity of the calcium hydroxide (portlandite) on the diffraction pattern is measured by the integrated method; results are reported on table 5. It also shows the peak intensity of the calcium carbonate (calcite) in order to underline the carbonation of the samples.

The intensity of the portlandite related peaks is lower for the FCA samples, conserved at ambient air, than for the FSH samples conserved in moist room. The development of the portlandite crystals in FSH samples can be explained by the presence of water on the porosity of the samples. The water permits the  $\text{Ca}^{++}$  ions diffusion towards the matrix and then the growth of the portlandite crystals (Olafsson 1986; Kurihara and Katawaki 1989;



**Figure 8.** X-ray diffraction patterns ( $\lambda$  K $\alpha$  Cu). Q, quartz; P, portlandite (Ca(OH)<sub>2</sub>); C, calcite (CaCO<sub>3</sub>); E, Ettringite; CSH, calcium silicate hydrates; and CA, calcium carboaluminate.

**Table 5.** X-ray diffraction peaks intensity of the calcium hydroxide and the calcite.

	Formulations					
	$2\theta$	$d(\text{\AA})$	FSH2	FSH3	FCA2	FCA3
Calcium hydroxide [Ca(OH) <sub>2</sub> ]	18.08	4.90	995	504	603	171
	34.1	2.62	305	202	291	146
	47.13	1.93	125	103	114	62
Calcite [CaCO <sub>3</sub> ]	23.11	3.84	65	87	70	48
	29.45	3.02	542	728	639	517
	47.62	1.90	67	95	69	61

Tomasawa *et al* 1989). Portlandite peaks are weaker in the samples containing silica fume. This confirms a reaction between portlandite and silica, and thus the expected pozzolanic effect.

The carbonation phenomenon is observed in all the samples, regardless of the conservation environments, but is limited when samples are conserved in moist room because water in pores prevents the CO<sub>2</sub> penetration into the matrix. However, the carbonate is developed in the near surface matrix and comes from the portlandite transformation into calcite. This transformation is accompanied by an increase of the crystal volume that induces a decrease of the cement matrix porosity in the near surface. As mentioned by different authors (Purnell *et al* 2000, 2003), the carbonation limits the growth of portlandite by decreasing the diffusions in the porosity.

### 3.3 Microstructural observations

The SEM observations are displayed in figure 9 and concern matrixes made of CEM II/B 32.5. The first micrograph displays the aspect of the A-R glass fibre before mixing: a regular and smooth surface. FSH2 and FSH3 samples, conserved in moist room, present a strong degradation of the fibres, with attack spots observed along the surface. However, the actual degradations are superficial and not homogeneous. It is noted that fibres are less damaged in the FSH3 samples due to the silica fume additions. The energy dispersive X-ray (EDX) analysis of the A-R glass fibre surface support the observed results. The localized chemical analysis realized on two different points (table 6) shows a degradation of the silicic network of the glass fibre. At point A, in the FSH3, there is no degradation of the fibre body and the microanalysis

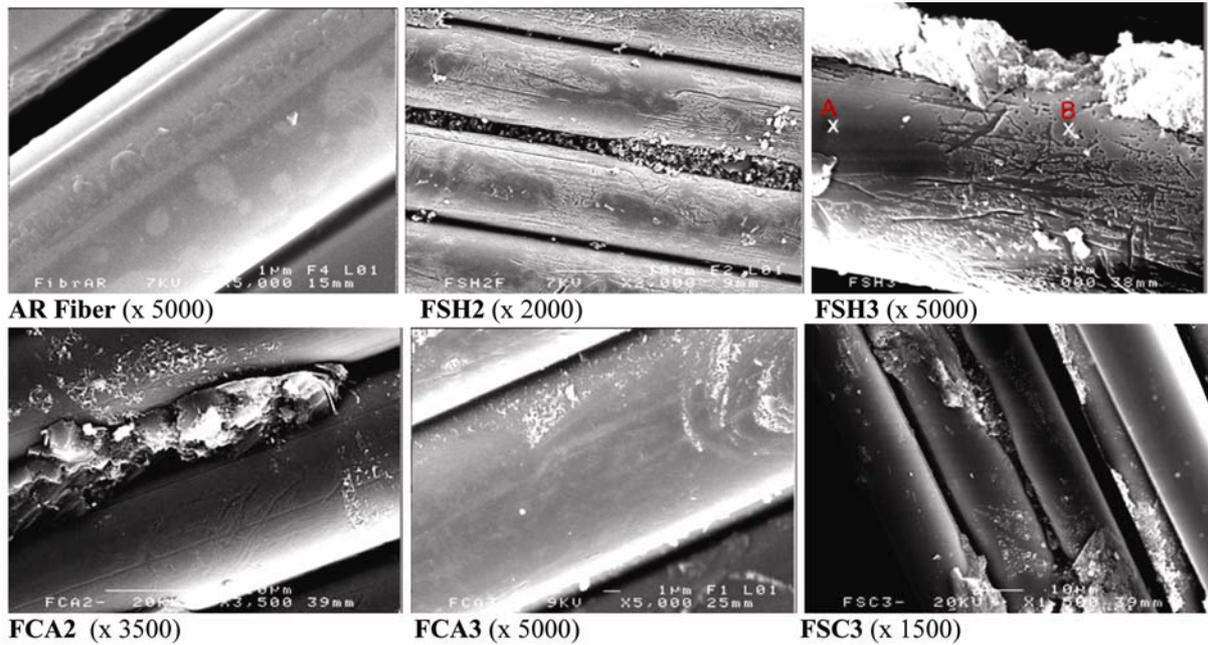
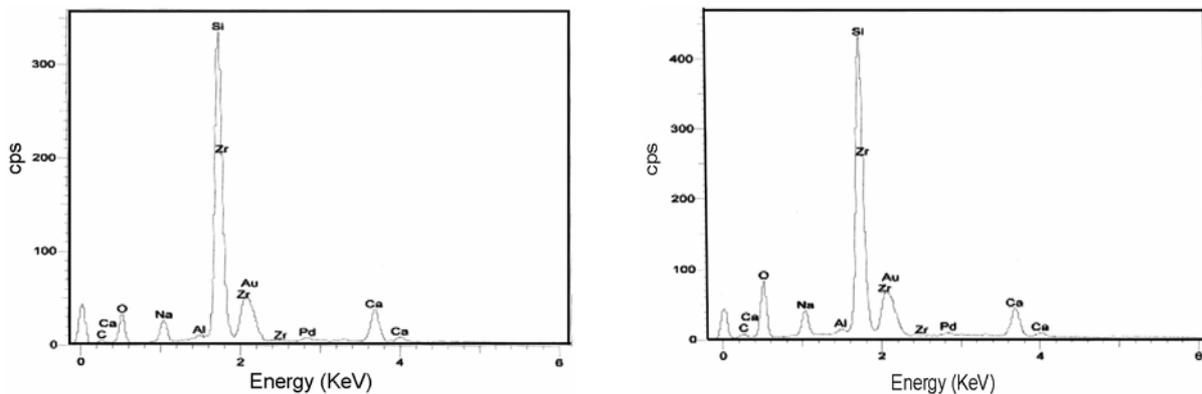


Figure 9. SEM observations of the fibre (AR) before mixing and of the glass fibres in the CEM II/B 32.5 cement matrix, conserved in different environments.

Table 6. Spectrometry X-ray analyses of the fibre body.

Element	Spectral type	Point A		Part B		
		Element (%)	Atomique (%)	Element (%)	Atomique (%)	
C	K	ED	4.37	9.34	6.07	11.63
O	K	ED	24.52	39.37	34.39	49.50
Na	K	ED	4.73	5.29	4.87	4.87
Al	K	ED	0.34	0.32	0.35	0.30
Si	K	ED	37.96	34.73	31.60	25.91
S	K	ED	-0.03*	-0.03*	0.13*	0.09*
Ca	K	ED	8.41	5.39	6.15	3.53
Fe	K	ED	0.22*	0.10*	0.11*	0.05*
Zr	L	ED	19.50	5.49	16.34	4.12
Total			100.00	100.00	100.00	100.00

\* = <math>2\sigma</math>.



shows higher silica (Si) contents than that detected at the point B. On the point B the observed degradation is marked either by erosion or notches. According to the

analysis, zirconium (Zr), which is the protective element of the fibre, is higher at the point A than that of the point B. The energy dispersive X-ray analysis of the glass fibre

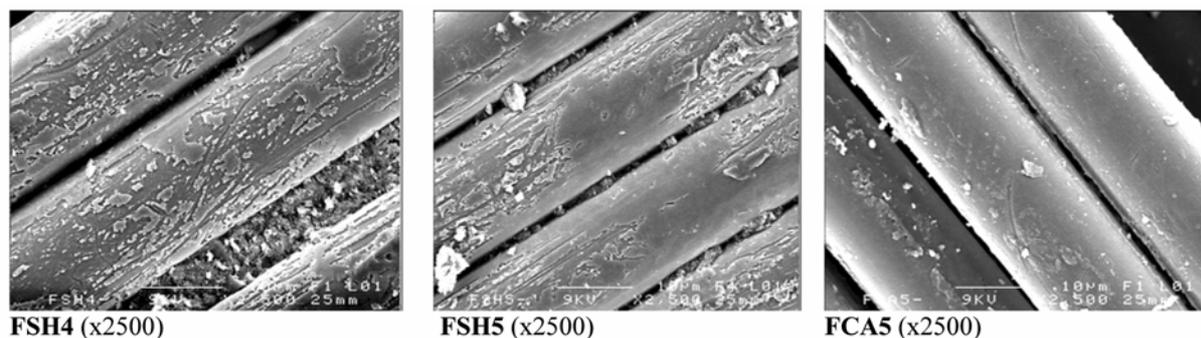


Figure 10. SEM observations of fibres in a CEM I 52.5 cement matrix, conserved in different environments.

surface indicated that dissolution of silica from the glass surface occurred. The samples conserved at ambient air, FCA2 and FCA3, and in dry environment, FSC3, remain intact.

Those observations confirm the results obtained by X-ray diffraction. First, silica fume additions permit to protect the fibres due to the pozzolanic reaction that transforms portlandite into CSH and thus decreases the matrix alkalinity. Second, the A-R glass fibre appearance remains stable in dry environment: on the one hand,  $\text{Ca}^{++}$  ion diffusion is restrained by a dry environment and the portlandite cannot develop. On the other hand, the carbonation, developed at the free surface of the samples, reduces the porosity.

Figure 10 shows the effect of a CEM I 52.5 cement on the fibres. The use of this cement amplifies the fibres degradation, as seen in FSH4 sample (as observed in the precedent samples made of CEM II cement, the fibres are ultra sensitive to a moist room). In the samples where silica fumes are added, the attack is less important and is not effective on the entire surface, which underlines the bad repartition of the silica fumes (see FSH5). Only samples conserved at ambient air, FCA5, display intact fibre surfaces. It must be noted that those fibres, announced alkali resistant by the manufacturers and introduced in a cement matrix containing up to 5% of silica fume, have however a low durability. It should be noted that CEM I 52.5 cement contains in his chemical composition higher quantities of free lime (free  $\text{CaO}$ ) and alkaline ( $\text{Na}_2\text{O}$ ) compared to the CEM II 32.5 cement. According to figure 11 it can be seen that the pH of the CEM I 52.5 cement is higher. The figure shows the pH values of pore solutions samples made from CEM II/B 32.5 and CEM I 52.5. The pH values decrease when the conservation conditions change and the silica fume is added to the samples.

#### 4. Conclusions

In this study, the obtained results permit to evaluate the behaviour of the alkali-resistant glass fibres used in a cement matrix and to observe the influence of the conservation environment.

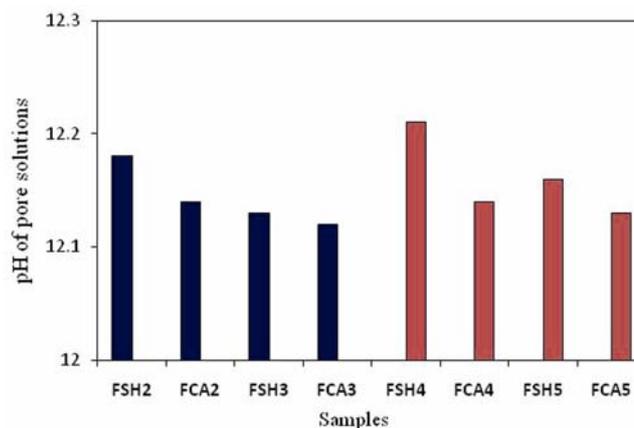


Figure 11. pH of pore solutions of the samples after 12 months of curing conditions.

The flexural strength drop is substantial during the first weeks of conservation and this is mainly related to the kinetics of cement hardening.

The addition of the silica fume at the rate of 3% contributes tardively to improve slightly the resistance, but does not give sufficient protection to the chemical attacks caused by alkaline environments on the fibres. This improvement becomes unperceivable at ambient air and dry environment conditions. However, this rate of silica fumes addition remains weak as amount to obtain of better results.

Under dry environment or low humidity, like a usual indoor building (case in ambient air), alkali-resistant fibres undergo surface modifications that look like disseminated spots. These degradations do not cast doubt on the structure stability of the glass fibre-reinforced cement.

On the contrary, the use of such materials in a humid environment (in moist room) has to be avoided. The stability of the structure would be endangered in spite of the fibre treatment and the silica fume use in the cement matrix.

The drop of flexural strength value is evident in ambient air and dry environment conservation conditions. How-

ever, using SEM, no fibre deterioration has been observed. This shows indeed the complexity of the degradation phenomenon for the glass fibres in an alkaline medium.

If degradations seem to be inevitable, they are quite reduced by the use of fly ash containing cement (CEM II/B) in comparison with cement without additions (CEM I).

### Acknowledgement

The author expresses his thanks to Joseph Le Lannic, CMEBA Rennes University, for his assistance with scanning electron microscopy.

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