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Effect of biochemical environment on fiber concrete containing silica fume

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Abstract. The durability of a concrete depends on several factors, one can quote: final mechanical characteristics, operating stresses, environmental stresses… Specific conditions of surrounding environment generate disorders. This is the case of structures and equipment of wastewater treatment which pose some problems not being in the other traditional uses of the concrete, the phenomenon is bacterial in origin. The impact of these degradations is very harmful for our hygiene of life. The impact of wetting / drying (control environment) Three tests were carried out: strength, depth of pH reduction using phenolphthalein, and micro-structural analysis by SEM. The mixes studied had various water cement ratios (0.5; 0.65; 0.8), with a 0.5% steel fibre content and 10% silica fume content. To characterize the durability performance of the mortars studied, samples were exposed to one-year cyclic wetting and drying: control samples under laboratory conditions and exposure samples in a sewage pumping station sump. SEM observations were carried out on samples with 0.8 w/c ratio to study the micro-structural changes in the mortar’s matrix. The SEM observation shows that there is a deterioration of the external surface of the samples by biochemical phenomena which are caused by a bacterial attack and sulphate attack.

1 Introduction

These last years we have seen a lot of degradation in wastewater systems [1]. This can have a negative impact for hygiene humans, health and economy. To remake a sewerage is very expensive and hence the orientation of the solutions to repair.

A low oxygen level in the wastewater can lead to the release of hydrogen sulphide gas, which can be oxidized to sulphuric acid in a bacterial layer; the acid with a pH of the order of 2 can produce deterioration of concrete by the dissolution of certain components of the cement paste and a sulphatic reaction which leads to the formation of expansive ettringite [2], [3], [4], [5], [6].

These reactions only take place in sections of the structure that are not permanently submerged. Deterioration is particularly aggressive in environments that are poorly ventilated (humid and elevated temperatures). The deterioration progresses from the exposed surface to the heart of the concrete; ultimately depassivation of the reinforcement can occur, further weakening the structure.

Durability depends essentially on porosity and cracking and therefore the compressive strength [7]; additionally the difference in the alkalinity of the interstitial solution and the exterior environment influences durability. Previous studies have shown that the addition of fibres limit the development of cracks and can lead to an improvement in durability [8], [9]. Other studies have shown that the addition of silica fume contributes to not only a reduction in porosity but also an improvement in compressive strength; these two advantages potentially contribute to improving durability [10], [11], [12].

In this study four series of mortar samples with and without silica fume addition (MN, MFs), with and without steel fibres (MNfms, MFsfms) are subjected to cyclic wetting and drying: control samples in the laboratory and exposure samples in the sump of a sewage pumping station. The principal difference in the environments being that the samples in the sewage pumping station sump are exposed to bacterial action, chemical attack and a higher humidity whereas those in the control were exposed to tap water and ambient air condition. It is also highly probable, given the conditions in the sump, that the samples were exposed to sulphate attack and a low pH.

After an exposure period of one year the modifications to the mortar were characterized by compressive and flexural strength, phenolphthalein test and examination by Scanning Electron Microscopy. To examine kinetics and accelerate reactions three mortars were tested with water cement (w/c) ratios, 0.5, 0.65 and 0.8; that is to say increasing mortar porosities.

2 Experimental Method

2.1 Materials
The cement used is a portland cement CEM I 52.5 from Saint Pierre Lacour (France); the chemical composition is set out in Table 1. The siliceous sand used complies with the standard CEN EN 196 N1 ISO 679; the granulometric range is between 0.08 and 2mm. The
The chemical properties are also set out in Table 1. The steel fibres used are 25mm long and 0.25mm diameter, their physical and mechanical properties are set out in Table 2. The fibre content used is 0.5% by volume of the mortar and the silica fume content is 10% by volume of cement.

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Cement</th>
<th>Silica Fume</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>64.50</td>
<td>0.14</td>
</tr>
<tr>
<td>SiO2</td>
<td>21.01</td>
<td>97.64</td>
</tr>
<tr>
<td>Al2O3</td>
<td>4.90</td>
<td>0.1</td>
</tr>
<tr>
<td>MgO</td>
<td>2.80</td>
<td>0.15</td>
</tr>
<tr>
<td>SO3</td>
<td>0.90</td>
<td>0.39</td>
</tr>
<tr>
<td>K2O</td>
<td>3.00</td>
<td>0.14</td>
</tr>
<tr>
<td>Na2O</td>
<td>0.90</td>
<td>0.14</td>
</tr>
<tr>
<td>Insoluble solids</td>
<td>0.20</td>
<td></td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>1.10</td>
<td></td>
</tr>
<tr>
<td>CaO free</td>
<td>0.14</td>
<td>1.10</td>
</tr>
</tbody>
</table>

Table 1. Chemical and mineralogical composition of cement and silica fume.

<table>
<thead>
<tr>
<th>Property</th>
<th>Steel Fibre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg/m³)</td>
<td>7500</td>
</tr>
<tr>
<td>Flexural strength (GPa)</td>
<td>1 ÷ 3</td>
</tr>
<tr>
<td>Module of elasticity (GPa)</td>
<td>200</td>
</tr>
<tr>
<td>Fire resistance (°C)</td>
<td>1500</td>
</tr>
<tr>
<td>Expansion coefficient (μm/m)</td>
<td>11</td>
</tr>
</tbody>
</table>

Table 2. Physical and mechanical properties of fibres.

2.2 Preparation and testing of samples
Four mortar mixes were used both complying with the standard (EN 106 – 1), these are:

- MN: Normal mortar
- MFs: Normal mortar + silica fume
- MNfm: Normal mortar + steel fibres
- MFsfm: Normal mortar + silica fume + steel fibres.

For each series of samples three water cement ratios were used 0.5; 0.65 and 0.8. After mixing 4x4x16 cm³ moulds were filled, compacted and stored in an environmental chamber at 20°C and 95% relative humidity (RH) for 24 hours. Once struck from the moulds, samples were conserved for a further seven days under the same conditions. Subsequently, the samples were stored in a chamber at 20°C and 50% RH to an age of 28 days. After the period of cure, to monitor the evolution of strength and durability characteristics, one series of samples was placed in a sewage pumping station sump, mid-way between high and low water levels and a control series of samples was subjected to cycles of wetting and drying under laboratory conditions; 8 hours cycles – 4 hours in water 4 hours drying, that is to say 3 cycles per day, in total 1100 cycles. In the control, samples were immersed in tap water and drying occurred under ambient conditions in the laboratory (approximately 18°C, 55% RH).

At an age of 365 days samples were recovered for testing: Three point flexural tests and compression tests were carried out. The depth of pH reduction was measured after failure by spraying the freshly fractured surface with a solution of phenolphthalein which revealed the depth of pH reduction in the mortar. Finally, observations by scanning electron microscopy (SEM) were carried out on the samples with a w/c ratio of 0.8 to visualize the modifications to micro-structure.

3 Results and discussion

3.1 Strength Characteristics

The stress at failure was determined in flexure and compression for the various water cement ratios. Fig 1 show the results obtained for mortars conserved in the two environments (pumping station sump and cyclic wetting / drying).

The stress at failure was determined in flexure and compression for the various water cement ratios. Figure 1 show the results obtained for mortars conserved in the two environments (pumping station sump and cyclic wetting / drying).
With regard to the compressive strengths: The mortar with fibres (MNfm, MFsfm) showed slightly higher strengths, when compared with the same mortars without fibres (MN, MFs), for both exposure environments and for all water cement ratios. In the case of the control samples, exposed to wetting and drying under laboratory conditions, the samples with fibres (MFsfm) showed a higher flexural strength than the control samples without fibres (MFs). On the other hand the flexural strength of the MNfm and MFsfm mortars was lower than the MN and MFs mortar when exposed to sewage. These samples have been exposed to aggressive agents in the sewer; the steel fibres have corroded and suffer a loss in section and consequently a reduction in strength and bond; this also resulted in an increase in porosity and surface cracking with a consequential reduction in flexural strength. On the basis of these observations it is noted that the use of steel fibres in a wastewater environment may have a detrimental effect on performance except if the fibres are well coated.

2 Phenolphthalein Test

The depth of pH modification of the mortar was determined by the phenolphthalein test (color indicator); a sprayed solution turns pink if the pH is greater than 9 which allow the visualization of the modification to the mortar. Five measurements were taken from each face of the sample, ignoring the measurements at the corners; this allowed a mean value of 20 readings for each sample. Figure 2 sets out the average depth of pH reduction.

It is interesting to note the variation of depth of pH reduction for the three water cement ratios: For a water cement ratio of 0.5 the steel fibres mortar had a greater depth of pH loss whereas with the higher w/c ratios the converse was true. This can be explained by two factors being responsible for the alteration of pH. Firstly, as previously noted the corrosion of the steel fibres engenders an opening of the structure; secondly as the w/c ratio increases the cement paste becomes more porous. Given a low w/c ratio the C-S-H resulting from the pozzolanic action of Portlandite and silica fume will fill pores in the matrix. In cases where the matrix has an initial high volume of voids, higher w/c ratios, the secondary C-S-H is insufficient to block the pore structure. In the cases of high w/c ratios (0.65 and 0.8) the high porosity of the cement paste has a dominant effect with respect to depth of pH reduction, i.e. steel fibre corrosion occurs but the subsequent effect is secondary to the cement paste porosity.

3.3 SEM Observations

Observations by scanning electron microscope (SEM) are shown in Figure 3 and 4; these observations were restricted to the MFsfm samples, w/c ratio 0.8, conserved in the sewer. The aim of this exercise was to study a series which had undergone the most marked modification to microstructure.
On photograph 1, we can see the dried organic material which gives this aspect of wrinkling. The aspect of C-S-H degraded on the surface of sample is shown in photo 2. We can notice that the degradation of C-S-H was apparent on a small thickness of about 4 microns.

Fig 3. Aspect of Surface degradation.

Fig 4 shows the phase of sound concrete. The photos shows the undegraded plates of C-H-S which one also calls “Tobermorite”.

Fig 4. Healthy phase of the concrete (C-S-H).

4. Conclusion

In all series of tests it is noted that an increase in water cement ratio leads to a deterioration in characteristics and therefore potential durability.

The depths of pH alteration in the samples exposed in the sump are significant. It is normal that the humidity in a closed space such as a sump would be high, a condition not favouring carbonation. Given the reduced portlandite content in the mortars and the high humidity it is suggested that carbonation is not the reason for the reduction in pH. It is probable that bio-chemical effects are responsible for the alterations to the external mortar surface under sewer conditions.

The compressive strength of the mortars subjected to both environments are sensibly of the same order, however marked differences are seen in the flexural strength. This disparity between compressive and flexural behavior implies a surface modification to the mortar; an observation borne out by the pH testing and SEM observation.

Comparing the mortars with silica fume (MFs), it is interesting to note that the flexural strength of the samples exposed to sewage is significantly higher than those exposed to laboratory conditions. This effect suggests that a surface hardening has occurred in the case of sewage exposure; once again the aggressive bio-chemical environment results in structural modification of the surface layers of the mortar. It is suggested that the increase in flexural strength may be due to an accumulation of organic and secondary crystallites forming in the skin of the mortar, which would have the effect of filling the pore structure and inhibiting the propagation of tensile cracking.

The duration of one year allow us to see the effect of fibers and silica fume on improving the durability. The SEM observations revealed a slight thickness degraded and healthy phase of the matrix. This aspect of the study merits further investigation.

References

11. M. Regourd, B. Mortureux, H. Hornain, Use of condensed silica fume as filler in Blended Cements.