

EVALUATION OF METHODS FOR TESTING CONCRETE DEGRADATION IN AGGRESSIVE SOLUTIONS

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Abstract

In order to characterise and rate relative performance of different cement-based materials in aggressive aqueous environments, reliable and reproducible test methods are necessary. Important is of course the aggressive environment one wants to simulate, which will determine the chemical and/or microbiological degradation mechanism, and the type of aggressive substances involved. Mostly considerations concerning available time and budget will guide the choice between small scale laboratory tests, large scale simulation tests and in situ tests. This choice will have a considerable effect on the test results, since it may affect different factors influencing the degradation rate or even the degradation mechanism. Different parameters which are adjusted in order to mimic the real-life situation more closely, or to accelerate the degradation and obtain results more rapidly, will influence the test results. These parameters include pH and concentration of the solution, physical state, temperature, rate of replenishment, mechanical action, alternate wetting and drying, alternate heating and cooling, pressure, etc. These parameters should be carefully selected and should always be reported together with test results. Also the choice of degradation measure may lead to different conclusions regarding relative performance of concrete types. Often a combination of multiple relevant indicators will be necessary. Furthermore sample preparation procedures and the concrete age at the time of testing are of utmost importance.

Keywords: test method, aggressive, concrete, degradation, solution, acid

1. INTRODUCTION

In the last decades several concrete structures have shown severe durability problems. Degradation mechanisms such as alkali silica reaction, chloride penetration, carbonation, acid attack, biogenic sulfuric acid attack, etc. have necessitated the renewal of complete structures. Apart from the human sorrow caused by the collapse of some bridges, these degradation problems have caused huge financial losses. These include not only the cost of repair and restoration of structures, but also the economic losses e.g. caused by traffic problems during the temporary closure of bridges, roads and tunnels. It is therefore important to tackle these problems by formulating the most appropriate concrete compositions and by taking protective measures.

On the other hand new concrete types have been developed over the last years, such as high and ultra high strength concrete, polymer cement concrete, self compacting concrete, high volume fly ash concrete, etc. Prescriptive durability specifications are not always available. Furthermore, concrete performance may highly depend on the exact mix composition and the compatibility between for instance cement and additives. Therefore, the need of suitable test methods and performance based indicators increases.

Rilem TC PAE “Performance of cement-based materials in aggressive aqueous environments” focusses especially on the problems of concrete and cement-based infrastructure having to operate in aggressive environments of an aqueous nature. These relate to natural environments such as the long-standing issue of concrete susceptibility to leaching in soft or aggressive waters; to public health environments with their associated drainage and treatment structures such as sewers, conduits, waste tanks etc; to water supply infrastructure with the provision of dams and canals; and to industrial and marine structures with natural or artificial highly aggressive environments.

In all these cases, concrete is subjected to a process of degradation whereby ion exchange reactions occur leading to a breakdown of the matrix microstructure and a weakening of the material. In some cases this can be extremely rapid and serious, such as in acidic environments, whether industrially or biogenically generated. In other cases degradation occurs over long periods of time, but the scale of the problem is such that the consequences are also severe, e.g. major dams, marine works, etc. Many industrial processes also generate aggressive aqueous solutions some of which can leach or attack concrete. Furthermore with increasing levels of acid rain being experienced worldwide, the ability of concrete to withstand this form of aggressive attack is also important.

One of the primary objectives of TC PAE is to review and define appropriate test methods to assess performance of cement-based materials in such environments, and which can be used to characterise and rate relative performance, as well as to inform long term predictions.

2. CLASSIFICATION OF TEST METHODS

Existing test methods can be classified in different ways, as listed below:

2.1. According to the type of aggressive species

Here a difference between chemical and microbiological degradation mechanisms can be perceived. Chemical degradation includes attack by soft water, acids (organic acids such as lactic and acetic acid and anorganic acids such as H_2SO_4), and salts (CO_3^{2-} , SO_4^{2-} , Mg^{2+} , NH_4^+ and Cl^- being the most important). These different ions may occur alone or in combination; in the latter case interaction may be important, the combined effect being more or less severe than the sum of individual effects. Regarding this issue NBN EN 206-1 (2001) states that “where two or more aggressive characteristics lead to the same class, the environment shall be classified into the next higher class, unless a special study for this specific case proves that it is not necessary”.

Microbiological degradation mechanisms feature aggressive substances produced by micro-organisms, such as in the sulphur cycle causing biogenic sulphuric acid attack in sewers [1]. The substances causing the degradation may be the same as for chemical degradation and act in the same way on the cement matrix, but the fact that micro-organisms are involved has an additional effect, e.g. because they favour more growing on certain

substrates than on others. This may therefore demand the use of special test methods. For instance Live/Dead® analysis during microbiological tests by Vincke [2] showed a more rapid colonisation by micro-organisms of the surface of slag cement samples compared to ordinary Portland cement samples. Monteny *et al.* [3] made a review of chemical, microbiological and in situ test methods for biogenic sulphuric acid corrosion of concrete. Furthermore they devised their own test procedure [4] implying a 4-step method, consisting of incubation of concrete samples into 250 ppmv H₂S atmosphere; incubation of samples in a solution of Thiobacilli and nutrients on a rotary shaker; rinsing with water; drying at 28°C. After every cycle, the thickness and surface roughness of the concrete blocks are measured with a high precision laser beam, mounted on an automated laser measurement table (ALM). Additionally, the material losses can be determined by weighing the samples after oven drying. This method was already used to examine the BSA resistance of several commercially available and new concrete types for cement based sewer pipes.

2.2. According to the aggressive environment

Aggressive aqueous environments may include marine environments, sewers, agricultural structures, underground structures and hydraulic structures, chemical plants, industrial structures, liquid-containing structures. The environment will determine the presence of aggressive species.

2.3. According to the scale of the test method

A difference can be made between small scale laboratory tests, large scale simulation tests and in situ tests. In the first two types of test methods, additional parameters may be changed to accelerate the degradation (see 5). For instance when configuring our own test method for biogenic sulphuric acid attack, first a test procedure for small concrete samples (size 20*20*50 mm) with limited aggregate size was developed [5]. Later on this test procedure was scaled up to obtain more representative results for concrete with large aggregates. Then, the samples were cylinders of 80 mm diameter and 15 mm height, taken some mm below the inner surface of the concrete pipes [4]. When investigating the degradation of slatted floors in animal houses by organic acids formed in acidified meal-water mixtures, we first used small concrete prisms (4 x 4 x 8 cm) as test specimens [6-8]. Later on we developed an automated test procedure on larger concrete specimens [9-10]. And to validate our results, in situ degradation experiments were carried out [11]. Also the group of Mori, when simulating biogenic sulphuric acid attack, carried out laboratory scale tests in a simulation chamber on mortar specimens of 4 x 4 x 16 cm, as well as in a demonstration plant consisting of a pipe with diameter 15 cm and a length of 20 m [12].

The scale of the test method, can have a significant effect on the test results, since it may affect factors such as the specimen surface area / liquid ratio, rate of replenishment of aggressive substances, presence of an interfacial transition zone (concrete vs. mortar specimens), use of real-life or simulated aggressive liquids, choice of an accelerated or close-to-reality test, etc. Research carried out on mortar or cement paste specimens, cannot always be extrapolated to concrete [13]. Because of the presence of aggregates a transition zone appears in concrete between the aggregates and the paste. Due to the transition zone effect, the microstructures of pure paste and paste within concrete are different. The effects of transition zones extend far beyond the aggregate surface and individual effects of surfaces in

concrete overlap. Therefore little or no paste in concrete has the same microstructure as pure paste and is not affected.

2.4. According to the combination with mechanical action or not

When only chemical action is present, a slowly growing layer of degraded material is formed and this can slow down further reactions. When the chemical action is combined with mechanical action, abrasion will remove the degraded layer and leave a new surface to chemical attack; this may accelerate the degradation process. Common ways of exerting an abrasive action in laboratory tests are manual or automated brushing and immersing in water that is shaken or stirred. The effect of mechanical action is also very clear in the case of sulphuric acid attack. When carrying out accelerated degradation tests on cylindrical test specimens rotating through a 0.5% sulphuric acid solution, an alternating increase and decrease of the radius could be noticed (see further Fig. 2), corresponding to alternating expansion of the concrete due to immersion and formation of reaction products and subsequent material loss due to brushing after each cycle of chemical degradation [14]. In a dynamic test developed by Fourie & Alexander [15] to evaluate acid resistance of concrete, a continuous brushing action during the test procedure is applied.

2.5. Parameters to accelerate degradation in simulation tests

The resistance of concrete to aggressive aqueous environments can be tested in different ways:

- realistic concentrations of the aggressive acids/salts can be used in combination with a sensitive method to detect deterioration and an extrapolation method to calculate the degradation in the future;
- accelerated tests can be performed. The degradation rate can be increased by means of higher concentrations of the aggressive medium, higher temperature, greater contact surfaces, alternated wetting and drying cycles, ...

The advantage of carrying out the experiments in a realistic way and keeping the factors responsible for the deterioration unchanged is the certainty that the deterioration process itself has not been changed. To be able to detect differences in degradation with methods close to reality within a reasonable time, sensitive methods are needed. Rombén [16] determined a relationship between the acid consumption and the attack depth of the concrete caused by hydrochloric acid. He measured the amount of acid needed to keep the pH of the solution constant and the amount of Ca released in the solution in function of the time. He also determined the Ca content of the concrete. Combination of these data allowed him to estimate the depth of attack in function of time.

A disadvantage of this kind of close-to-reality investigations is that only the very first stages of the attack can be measured. To estimate the attack of the concrete at a later stage an extrapolation of the obtained relationship has to be used. This implicates the risk that new mechanisms of a type, which was not noticeable during the measuring time, arise in a later period of the deterioration. The rate of attack in the case of sulphuric acid probably follows relationships which are difficult to extrapolate, due to the special mechanism which is characteristic for this acid [16].

One of the most common ways to investigate the chemical resistance of concrete is to carry out accelerated tests in laboratory. The advantage of this method is that the entire life of the specimen in question can be simulated. An acceleration of the process can be achieved in

different ways. The concentration of the aggressive solution can be increased and/or the reaction surface can be increased by using specimens with a large surface area-to-volume ratio, etc.

Factors influencing the rate and extent of attack include:

- *strength of the acid in the attacking solution:*

Strong acids are normally very aggressive (for a given concentration), because they decompose the cement hydrates easily. Nevertheless, some strong acids, such as phosphoric acid, are less aggressive than weak organic acids like citric, lactic or malic acid.

- *pH and concentration of the attacking solution:*

The pH value is very often used to define the concentration of an acid solution, because it is more difficult to measure concentration level than pH. When the pH decreases from 7 downwards, the solution becomes more acidic and it will become more aggressive to concrete. Therefore it is of practical interest to define the acceptable limits of the pH without exaggerated corrosion. Figure 1 [17] shows that acceptable limits for a certain concrete depend on the acid, because of the differences in acid strength. It is interesting to observe that the pH limits are lower for strong acids, like hydrochloric or nitric, than for weak acids like lactic or acetic. This can be explained because weak acid solutions have a higher pH than strong acid solutions for a given concentration. For example, 0.1 N acetic acid has a pH of 2.9, while the pH of 0.1 N hydrochloric acid equals 1. Thus, for a given pH, strong acids have a lower concentration than weak acids and may be less aggressive.

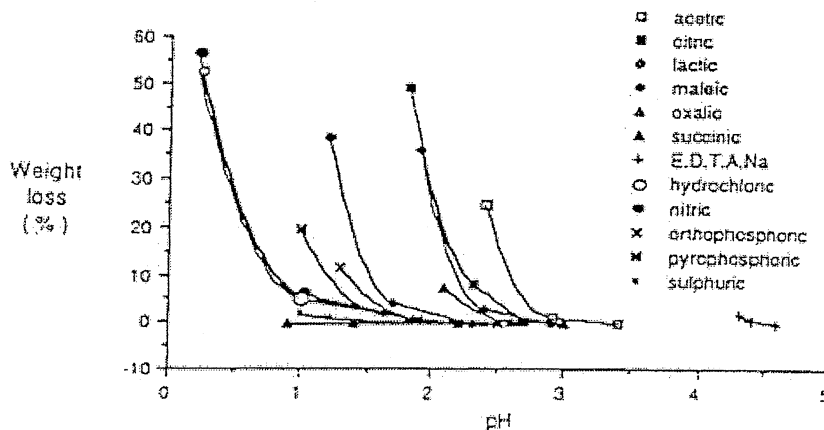


Fig. 1. Weight loss (%) of concrete samples as a function of the pH of the solution (after [17])

De Belie *et al.* [10] also found in experiments with the TAP (test equipment for accelerated degradation) that lactic/acetic acid attack on concrete was more severe than sulphuric acid attack. Figure 2 shows the average change in radius for concrete samples subjected to sulphuric or lactic/acetic acid attack. For the sulphuric acid attack, the simulation liquid consisted of 0.5% sulphuric acid in water (pH = 0.8-1.0). The concrete mix proportions of reference specimen I were based on a mix design commonly used for sewer pipes, featuring a high sulfate resistant (HSR) Portland cement (CEM I 42.5 HSR/LA) and a W/C

ratio of 0.40. For specimen II, 30 kg/m³ silica fume was added. The lactic/acetic acid degradation was carried out with a simulation liquid consisting of lactic and acetic acid in water in concentrations of both 30 g/l (pH = 2.0-2.2). The reference concrete specimen III had a mix design commonly used for production of prefabricated concrete slabs, including an ordinary Portland cement (CEM I 42.5R) and a W/C ratio of 0.39. For the same number of attack cycles (keeping in mind that furthermore the cycles for the sulphuric acid attack are twice as long), the average decrease in radius in the lactic/acetic acid solution was much more pronounced than for the sulphuric acid solution (-2.1 mm for concrete III, compared to +0.13 mm and -0.42 mm for concrete I and II, respectively). This in spite of the pH of the sulphuric acid solution (pH = 0.8-1.0) which was significantly lower than the pH of the organic acid solution (pH = 2.0-2.2).

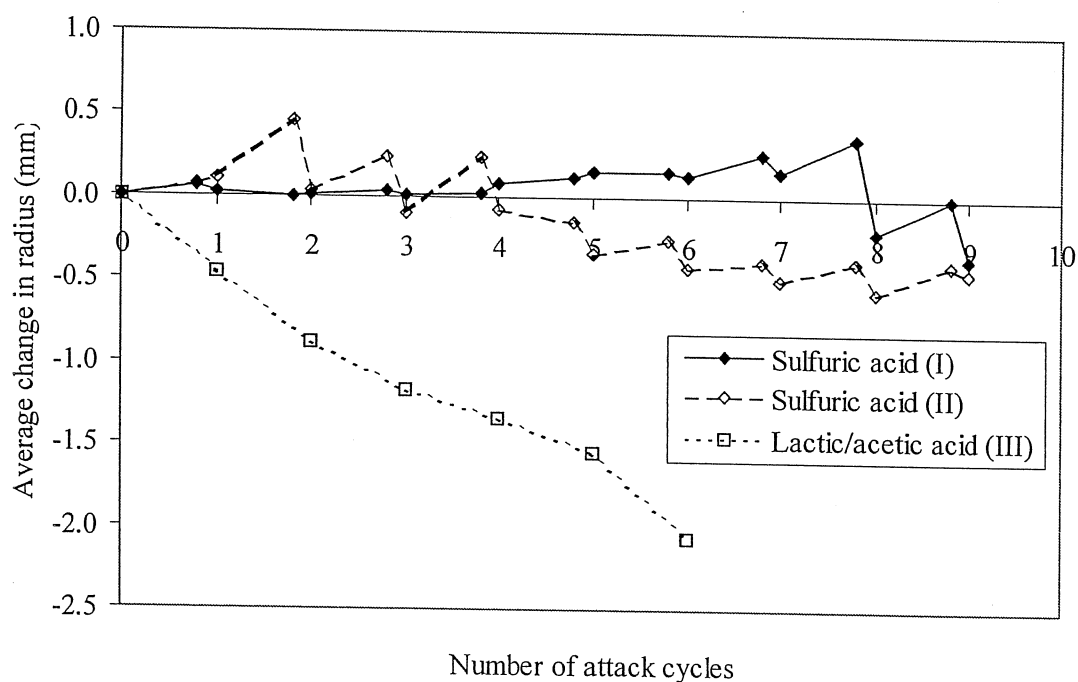


Fig. 2. Average change in radius vs. number of attack cycles for a TAP degradation test with a sulphuric or lactic/acetic acid solution, respectively

Changing the concentration of the aggressive liquid in order to obtain acceleration of the degradation process in laboratory tests, may result in unwanted effects. For instance, in the case of sulfate corrosion, changing the concentration could change the attack mechanism. Cohen & Mather [13] warn that for sulfate attack, at low SO_4^{2-} concentrations (less than 1000 mg $\text{SO}_4^{2-}/\text{L}$), deterioration of the concrete is mainly due to the formation of ettringite. At high concentrations the formation of gypsum is the main cause of deterioration. Cohen and Mather [13] conclude that in the case of sulfate attack, results obtained in laboratory under accelerated test conditions by increasing sulfate concentration cannot be used to predict actual behaviour in the field.

Bertron *et al.* [18] advises to use three parameters to compare the aggressiveness of different acids: the acids' dissociation constant, the solubility of the acid salts in water and the constants of organometallic complexes formed in solution.

A distinction between different laboratory tests can also be based on the pH being kept constant by automatic titration or not. In the latter case the pH will usually increase during the test because of leaching of free lime and alkalies from the concrete; simulation liquids can then be changed at certain intervals or not. Fourie & Alexander [15] found that allowing the pH to increase from 1.0 to 1.5 during a test with hydrochloric acid, resulted in a significantly lower mass loss and hydrogen ion consumption, compared to the situation when the pH was controlled between 1.00 and 1.05 or between 0.95 and 1.00.

Bertron *et al.* [19] investigated the effect of pH on mineralogical and chemical modifications occurring in the cement-based matrix of ordinary Portland cement and slag cement pastes submitted to organic acids mixes. They wanted to assess if use of an aggressive solution of pH 4 to accelerate alteration kinetics, would not change the degradation mechanism in comparison to a solution with a pH of 6, which is more realistic in the case of liquid manure attack. They found that the kinetics of alteration (mass losses and altered depths) were ninefold higher in the solution with a pH of 4, but that the alteration mechanisms were sensibly identical. The modifications in both cases included decalcification, disappearance of crystallized and amorphous hydrated phases and probable formation of silica gel containing aluminium and iron. Minor alteration mechanism differences mainly concerned the stability of anhydrous phases such as C_4AF which is stable at pH of 6, but not at a pH of 4. Also a slight decalcification of slag anhydrous phases was seen at a pH of 4.

- *anion/cation combined with the aggressive ion in a salt solution*

The anion or cation combined with the aggressive ion in a salt solution will partly determine the aggressiveness. Some examples:

- Magnesium ions are particularly detrimental in combination with sulphate ions. The reaction produces calcium sulphate, which can cause further damage by formation of the expansive ettringite.
- The anion combined with NH_4^+ is also important, as it determines the solubility of the calcium salt produced. Ammonium oxalate, ammonium acetate and ammonium carbonate have a limited aggressiveness. Ammonium bicarbonate and nitrate are more harmful and the anion of ammonium sulphate and chloride results in an extra detrimental effect.
- The cation combined with NO_3^- determines the aggressivity of the nitrate. A 10% calcium or alkali nitrate solution would not attack concrete, while aluminium nitrate will. Ammonium nitrate would be most harmful.
- Chlorides which produce unstable and water soluble compounds when reacting with $Ca(OH)_2$, for example $MgCl_2$, $CaCl_2$, NH_4Cl , $FeCl_3$, $CuCl_2$ and $AlCl_3$, could damage concrete. Other chlorides, such as $NaCl$ and KCl , which do not react with lime would only degrade concrete in very high concentrations.
- Magnesium sulphate has a much more damaging effect than either calcium or sodium sulphate.

- *physical state of the attacking medium:*

Dry, non-hygroscopic solids do not attack dry concrete, but some will attack moist concrete. A moist, reactive solid can attack concrete, as can aggressive liquids and solutions. Dry gases,

if aggressive, may come into contact with sufficient moisture within the concrete to make attack possible. Moist, aggressive gases would tend to be more destructive.

- *temperature:*

Temperature may effect the rate of attack in two different ways. The common effect is that chemical activity usually increases exponentially, approximately doubling with each 10°C rise in temperature. Temperature may also affect the rate of attack indirectly. As temperature rises, the moisture content of the concrete is reduced, making it drier, but more permeable to additional fluid. As temperature falls, it may sometimes cause sufficient normal shrinkage to open small cracks and allow greater penetration of liquid into the concrete.

- *replenishment of the aggressive medium:*

For example, a concrete structure may be placed in an acidic soil, but if the acidity is not replenished, the available acid may be quickly neutralized with little or no damage to the concrete.

- *alternate wetting and drying:*

In some cases the process can be accelerated by means of continuous wetting and drying cycles which allows uptake of aggressive agents through convective processes, being an order of magnitude faster than diffusion. De Belie *et al.* [10] applied an alternate wetting and drying procedure in their TAP equipment, by mounting concrete cylinders on rotating horizontal axes (about 1 revolution per hour) and making them turn through containers with simulation liquids. Cycles of chemical attack are followed by abrasion using rotary brushes. The change in dimension of the concrete specimens and the surface roughness are determined through a non-contact distance measurement with laser sensors. Alternate wetting and drying may also increase the crystal pressures. Dissolved substances may migrate through the concrete and deposit at or near the surface from which evaporation occurs. The deposit may be the original substance or a reaction product formed in the concrete. This effect can be seen in the familiar 'efflorescence' on walls of concrete, brick or stone. Progressive disposition can be disruptive.

- *alternate heating and cooling:*

Alternate heating and cooling may be harmful. Freezing and thawing can damage concrete through physical effects.

- *pressure:*

Pressure is seldom of consequence in its effect on chemical reaction rates, but can greatly increase the degree to which attacking substances can penetrate the concrete. Partial pressures of aggressive gases may be of importance, because they are a measure of concentration.

2.6. According to the way of measuring the degradation of specimens

Different measures for quantifying degradation may be implemented in test procedures. Regarding different experimental methods to determine sulphate resistance for instance, the Koch/Steinegger method implies measurement of the residual flexural strength, while Wittekint and ASTM C1012-84 implement a criterium based on length changes.

In general, degradation measurements may include thickness change, mass loss, expansion, residual strength of specimens, pH change of liquid, calcium released in liquid,

hydrogen ion consumption, loss of elastic modulus, etc. The former measurements may be supplemented by SEM or XRD analysis to examine the microstructure. Bertron *et al.* [20] measured the altered depth visualised with phenolphthalein, in relation to the time of exposition, to investigate the kinetics of progression of the dissolution front for cement pastes immersed in a mix of five organic acids. Chemical modifications in different zones were characterised with an electron microprobe, analysing Ca, Si, Al, Fe, Mg, S, K, Na. Schneider & Chen [21] also used phenolphthalein indicator to determine penetration depths of ammonium nitrate in concrete specimens subjected simultaneously to flexural loads. Maltais *et al.* [22] also used microprobe analysis together with SEM and XRD analyses to establish chemical composition of phases after degradation of Portland cement systems in deionised water and sodium sulphate solutions. Neuenschwander *et al.* [23] and Schmidt [24] examined the use of a non destructive method using ultrasonic surface waves (leaky Rayleigh waves) to quantify degradation of mortar samples in sulphuric acid solutions.

The choice of the degradation measure may lead to different conclusions regarding the relative performance of concrete types. For instance in microbiological tests De Belie *et al.* [4] used both mass loss and thickness change as parameters to judge degradation. Samples with limestone aggregates showed a somewhat higher weight loss than samples with inert aggregates, while their thickness change was lower. This effect could be partly explained by taking the density of the concrete constituents into account. For concrete with limestone aggregates, the removed material consisted of “concrete” (= aggregates + cement mortar) with a density of about 2400 kg/m³. For the concrete with inert aggregates, on the contrary, the removed material consisted in this stage only of cement mortar with a density of around 2000 kg/m³. This partly explains why limestone concrete with a lower average attack depth, can still figure a higher weight loss. This effect would probably disappear when the degradation process proceeds and large aggregates are removed from the matrix. The large differences between thickness and weight results could also suggest that concrete with limestone aggregates underwent a somewhat larger expansion than concrete with inert aggregates.

Therefore, one single measure may not suffice to characterise the degradation sufficiently. Mass loss, for instance, may be well used when no secondary products precipitate, otherwise it may be the result of a combination of several phenomena. It is therefore recommended to use multiple relevant indicators to investigate the resistance of concrete to a particular degradation mechanism [13, 25]: for instance for sulphate attack, mass loss, expansion, dynamic modulus of elasticity and flexural strength can be monitored. [4] combined measurements of thickness with an automated laser profilometer, mass loss, and calcium and sulphate concentrations in the microbial suspension, to quantify biogenic sulphuric acid degradation.

2.7. Other important points to be considered

Other important points to be considered include the effect of the age of test specimens on the results of accelerated tests. For instance if we consider concrete with fly ash compared to ordinary Portland cement concrete, it may be that the resistance in an accelerated test is higher for OPC concrete at the age of 28 days, while it is higher for fly ash concrete after some years. Under real conditions the aggressive species may exert their effect over a certain time period. Concentrating the effect at a certain age of the specimens favour some types over others. Fig. 3 for instance [26] shows the non steady-state chloride migration coefficient D_{nssm} in a CTH test (NT Build 492, 1999) for four concrete types with 400 kg/m³ cementitious

material (CM) and a W/CM ratio of 0.4 prepared according to NBN B15-001 (2004). The reference concrete (REF) was made with ordinary Portland cement CEM I 52.5 N; in FA35, FA50 and FA67, respectively 35%, 50% and 67% of the cement was replaced with type F fly ash (1.96% CaO). The concrete was stored at 20°C and more than 90% relative humidity until the age of testing. CTH tests were carried out at the age of 1, 3 and 6 months. At one month, chloride migration coefficients are much higher for FA50 and FA67 than for REF and FA35, whereas at 3 months they become similar as for the reference concrete.

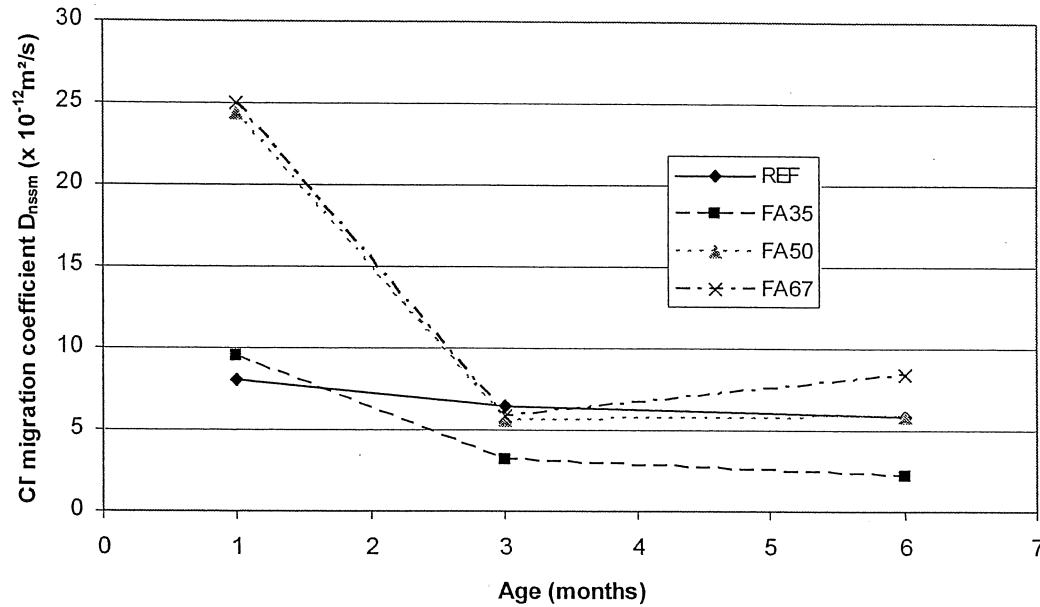


Fig. 3. Non steady-state chloride migration coefficients for concrete with fly ash to cementitious materials ratio of 0% (REF), 35% (FA35), 50% (FA50) or 67% (FA67)

Also the choice of the reference should be considered. Often tests results are relative results, in comparison with a reference concrete. The choice of this reference is therefore of great importance. For example, the relative sulphate resistance of a certain concrete type may be completely different when compared to a reference with an ordinary Portland cement or with a high sulphate resistant (Portland) cement. Factors such as mix procedure, curing, specimen size, etc. should always be detailed.

3. CONCLUSIONS

The choice of a test method to rate relative performance of different cement-based materials in aggressive aqueous environments, will have a major effect on the test results. Parameters such as the scale of the test method, physical state of the attacking medium, the pH and concentration of the solution, temperature, rate of replenishment, mechanical action, alternate wetting and drying, alternate heating and cooling, pressure, etc. should be carefully selected and should always be reported together with test results. Also the choice of degradation measure may lead to different conclusions regarding relative performance of concrete types. Often a combination of multiple relevant indicators will be necessary.

Furthermore sample preparation procedures and the concrete age at the time of testing are of utmost importance.

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