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Chemical and microbiological tests to simulate sulfuric acid corrosion of polymer-modified concrete

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Abstract

In certain industrial activities sulfuric acid is used during the production process, which may cause degradation of concrete structures. Another important phenomenon where sulfuric acid is responsible for concrete corrosion is biogenic sulfuric acid corrosion, which occurs often in sewer systems. Because previous investigations have already pointed out the difference between purely chemical sulfuric acid corrosion and biogenic sulfuric acid corrosion two different tests were performed: a chemical test and a microbiological test. Five different concrete compositions were used in the tests, including a reference mixture with high sulfate resistant portland cement and four different polymer cement concrete with a styrene–acrylic ester polymer, an acrylic polymer, a styrene butadiene polymer and a vinylcopolymer, respectively. The concrete composition with the styrene–acrylic ester polymer showed in both tests a higher resistance than the reference mixture while the compositions with the acrylic polymer and the styrene butadiene polymer had a lower resistance than the reference mixture. The concrete composition with the vinylcopolymer did not induce the same results in both tests. The results of the chemical test indicated a slight increase in resistance compared with the reference mixture while the opposite was noticed for the microbiological test.

Keywords: Corrosion; Durability; Sulfate attack; Biogenic sulfuric acid

1. Introduction

Sulfuric acid is a very aggressive acid that reacts with the free lime [$Ca(OH)_2$] in the concrete forming gypsum ($CaSO_4 \cdot 2H_2O$). This reaction is associated with an increase in volume of the concrete by a factor of 2.2 [1,2]. An even more destructive action is the reaction between calcium aluminate and the formed gypsum crystals. These two products form the less soluble reaction product ettringite ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$). These very expansive compounds cause internal pressure in the concrete, which leads to the formation of cracks [3]. The corroded surface becomes soft and white. When the corrosion continues, the corroded concrete structure loses its mechanical strength. Although the important role of bacteria in biogenic sulfuric acid corrosion may not be ignored it is the reaction with the produced sulfuric acid that causes the corrosion of the concrete. This type of corrosion is often seen in sewer systems. Concrete structures, normally built to last for at least 30 years, fail sometimes after only a few years when biogenic sulfuric acid corrosion is involved. Due to different chemical and microbiological reactions, hydrogen sulfide can be released in the atmosphere of sewer structures above the water level. This gas reacts with oxygen to form elemental sulfur, which is deposited on the walls of the sewer structures. In the slime layer coating these walls, aerobic sulfur-oxidizing bacteria (*Thiobacillus* sp.) metabolize the sulfur to sulfuric acid. Repair and sometimes complete replacement of the corroded structures becomes necessary. These repairs are expensive and cause several discomforts to the community. Therefore, it is important to find ways to control this specific kind of concrete corrosion.

Compared with ordinary concrete, polymer-modified concrete has the advantage of having a higher resistance against aggressive liquids such as lactic, acetic and sulfuric acid [3–5]. In the experiments discussed in this paper four different polymer-modified concrete compositions were subjected to purely chemical sulfuric acid and microbiolo-

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gically produced sulfuric acid. The resistance of these compositions was compared with a reference concrete composition without addition of polymer.

2. Material and test methods

2.1. Materials

All concrete mixtures tested were made with 350 kg/m^3 high sulfate resistant portland cement (CEM I 42.5 HSR/LA). The composition of all experimental mixtures is shown in Table 1. For the polymer-modified concrete mixtures four polymer types were used: a styrene–acrylic ester polymer, an acrylic polymer, a styrene butadiene polymer and a vinylcopolymer. The polymers were added to the mixing water as latex at a polymer–cement ratio of 7.5% (weight of solid phase of polymer emulsion/weight of cement). The reference mixture without polymer was made with a water–cement (W/C) ratio of 0.4 while for the polymer-modified concrete mixtures, the water content was adjusted till a slump of class S1 (10 to 45 mm) was obtained [Belgian standards NBN B15-232 (1982)]. In spite of the low W/C ratio (0.34), the mixture with the acrylic polymer showed a slump of class S3 (100 to 150 mm) due to high air entrainment. Due to the stickiness of the mixture with the vinylcopolymer, it was not possible to work with an S1 class and more water had to be added to create a workable concrete mixture. The final W/C of the different mixtures was calculated taking into account the water content of the added latexes. These values are shown in Table 2.

Because of the water-reducing properties of the polymers, the use of a superplasticizer was not necessary for the polymer-modified concrete mixtures (Concrete Mixes II to V). Slump, flow and air content of the fresh concrete mixtures, were measured according to the Belgian Standards NBN B15-232 (1982), NBN B15-233 (1982) and NBN B15-224 (1970), respectively (Table 2).

Table 1
Mix proportions of the different concrete mixtures

	Concrete mix				
Material (kg)	I (P)	II (SBR)	III (SA)	IV (A)	V (VPV)
CEM I 42.5 HSR/LA	350	350	350	350	350
Gravel 4/14	1120	1124	1119	1124	1092
Sand	840	843	839	843	819
Water	140.0	90.6	97.3	86.9	104
Total water content[a]	140.0	119.0	122.5	119.0	143.5
Polymer (as latex)	–	54.7	51.5	58.3	65.6
(polymer type)		(SBR)	(SA)	(A)	(VPV)
Superplasticizer	2.5	–	–	–	–

CEM I 42.5 HSR/LA: portland cement; SBR: styrene butadiene; SA: styrene–acrylic ester polymer; A: acrylic polymer; VPV: vinylcopolymer.

[a] Including the water contained in the polymer latex.

Table 2
Slump, flow, air content and water–cement (W/C) ratio of the fresh concrete

Concrete mix no.		Slump (mm)	Flow (mm/mm)	Air content (%)	W/C ratio[a]
I	(P)	20	1.69	3.0	0.40
II	(SBR)	40	1.62	4.4	0.34
III	(SA)	40	1.47	4.0	0.35
IV	(A)	105	2.04	9.7	0.34
V	(VPV)	75	1.71	5.2	0.41

[a] Including the water contained in the polymer latex.

From each concrete mixture three cylinders (∅230 mm, height 70 mm) were prepared for the chemical tests and three small prisms (20 × 20 × 50 mm) to perform the microbiological tests. The small prisms were cut out of a larger prism in such a way that one face of the small prisms (20 × 50 mm) coincided with a moulded face of the larger prisms. The small prisms were glued on a glass plate in such a way that the glued face was opposite to the moulded face. Because the glass plate did not corrode during the test, it acted as a reference for the measurements.

2.2. The microbiological test method

During the microbiological test the small concrete prisms glued on a glass plate were subjected to a microbiological suspension. This suspension contained all necessary elements (bacteria, sulfur and nutrients) to produce sulfuric acid in a microbiological way. The test method consisted of a cyclic procedure of four subsequent steps, which simulate the phenomenon of biogenic sulfuric acid corrosion in sewer pipes [6].

Step 1 simulated the sorption of H_2S gas in the surface layer of the concrete. During this step, the test specimens were placed in an H_2S atmosphere for 2 days. The initial gas concentration of the atmosphere was around 250 ppmv H_2S.

Step 2 simulated the production of sulfuric acid by sulfur-oxidizing bacteria present in the slime layer coating the pipe walls. Each test specimen was suspended during 10 days in a separate glass recipient containing 600 ml of a simulation solution. The test specimens were completely immersed in the solution, and constantly stirred. The solution consisted of 950 ml of tap water with an additional N and P source (100 mg/l $(NH4)_2SO_4$ and 10 mg/l K_2HPO_4) and 50 ml of a biological sulfur suspension. The biological sulfur suspension consisted of complex aggregates containing elemental sulfur and other reduced sulfur compounds, biomass (*Thiobacillus*-like bacteria) and biopolymers. During the incubation, the bacteria use the elemental sulfur to produce sulfuric acid. This phenomenon also happens in practice. For every tested concrete mixture, a control test specimen was also investigated. The control test specimen was treated in the same way as the other test specimens, with the difference that the simulation solution was inactivated by adding the biocide glutaraldehyde. Therefore, the bacteria in the control did not produce sulfuric acid during the incubation period. Furthermore, a reference was investigated, which contained only the simulation solution and no concrete test specimen.

Step 3 simulated the filling up of sewer pipes during rainy days by stirring the test specimens during 1 day in separate glass recipients containing milli-Q water.

During step 4 the test specimens were dried for 4 days to simulate dry periods in the sewer pipes. Furthermore, the test specimens were slightly brushed to remove weakly adhering concrete particles.

One complete cycle lasted for 17 days and the complete test programme consisted of four subsequent cycles. To investigate the corrosion process the change in pH and sulfate concentration during step 2 were measured. The total Ca^{2+} content released during steps 2 and 3 was also measured. The corrosion of the different concrete specimens was quantified by two different parameters: the change in height and the change in weight of the specimens. The change in height of the specimens was measured after the fourth step of every cycle. For every test specimen, the change in height was the average of the measurement on 10 different points. The change in weight was measured after the total test procedure was finished.

2.3. The chemical test method

A standardized and automated chemical exposure procedure to sulfuric acid was performed using a testing apparatus for accelerated degradation test (TAD) [5,7]. Three concrete cylinders of each concrete mix were mounted on rotating axles fixed on a frame. The cylinders were turned through containers with a solution of 0.5% sulfuric acid (pH of around 1.0) at a speed of 1.04 revolutions per hour, in such a way that only the outer 50 mm was submerged. One exposure cycle lasted 12 days and in total nine cycles were completed. After each exposure cycle, the following steps were taken: first, the change of the radius of the concrete cylinders was measured with laser sensors connected to a computer. For each concrete cylinder, six profiles equally distributed along the cylinder height were scanned. The change in radius of the concrete cylinders (the combined effect of swelling and a limited destruction of the surface) was calculated by comparing the measured profiles with the initial profiles at the same place. Next, the cylinders were brushed with rotary brushes to remove the weakly adhering concrete particles. After brushing, the change of the radius of the cylinders was measured again. By measuring before and after brushing a distinction could be made between the expansion of the cylinders and the loss of material, which implies a decrease in the radius of the cylinders. After each cycle, the solutions were renewed and after cycles 2, 4, 6 and 8 the total Ca^{2+} content of the different sulfuric acid solutions, in which the cylinders had rotated, was determined.

3. Results and discussion

3.1. Mechanical properties of the concrete mixtures

All polymer-modified concrete mixtures had a lower density than the reference mixture without polymer. This was mainly due to the higher air entrainment of the concrete with addition of polymer. The addition of the styrene butadiene polymer, the styrene–acrylic ester polymer and the vinylcopolymer caused an increase in air content of the fresh mixture by 1% to 2% compared to the reference mixture (Mix I). The addition of the acrylic polymer resulted in an increased air content of almost 10%.

3.2. Microbiological tests

Concerning the change in pH of the simulation solutions, there was a significant difference between the active and the inactive solutions. The pH of the inactivated simulation solutions showed a small increase up to a value of almost 9 due to the presence of the concrete blocks. The pH stayed at this level during the following days. During all cycles the pH of the active solutions decreased from 8 to around 1 after 10 days. This decrease confirmed the production of sulfuric acid by the bacteria. No significant difference could be noticed between the different concrete mixtures.

The change in sulfate concentration of the simulation solution during step 2 of all four cycles is given in Fig. 1. For the first two cycles the change in sulfate concentration for the inactivated simulation solutions, containing the control test specimens, is also given. Because all lines of the different specimens coincided the average sulfate concentration of these solutions was plotted. No increase of sulfate concentration could be noticed for these solutions. This confirms the fact that the bacteria were indeed inactivated and did not produce sulfuric acid. The sulfate concentration of the active simulation solutions, of the different test specimens, increased from around 0.2 to almost 2.5 g/l. This increase also reflected the action of the bacteria. For the

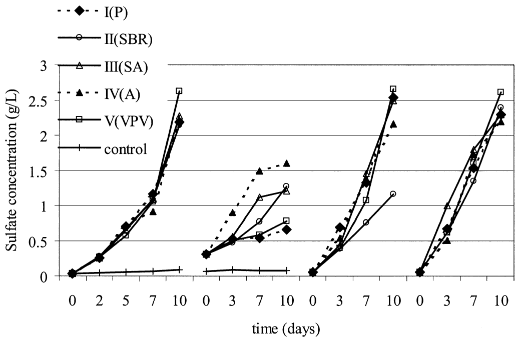

Fig. 1. Change of the sulfate concentration of the simulation solution.

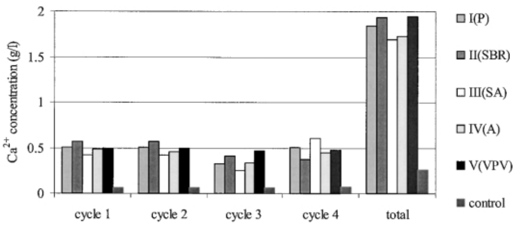

Fig. 2. Ca^{2+} concentration of the simulation solution.

different cycles, no repetitive pattern could be found for the different concrete mixtures concerning the amount of sulfate produced during the second step.

The Ca^{2+} content of the simulation solutions (activated and inactivated solutions) measured after every cycle and the total Ca^{2+} release during all four cycles is given in Fig. 2. For the inactivated control solutions with the different concrete mixtures, only a slight (< 0.2 g/l) Ca^{2+} release was measured. This indicated that the control test specimens were indeed not affected by the inactivated simulation solution. Because of the small difference between the different inactivated simulation solutions, the average of these values is plotted as a result for the control group. The concrete specimens in the active simulation solutions, on the other hand, showed a Ca^{2+} release of 0.2 to 0.5 g/l per cycle. This confirmed the corrosion of the concrete due to the sulfuric acid formed by the bacteria. The mixtures with styrene–acrylic ester polymer and acrylic polymer had a lower total Ca^{2+} release (sum of the Ca^{2+} contents of the different cycles) than the reference mixture. The mixtures with styrene butadiene and vinylcopolymer had a higher total Ca^{2+} release than the reference mixture. However, these differences were not statistically significant.

Fig. 3 shows the change in height per cycle and the cumulative change in height at the end of the test of the different mixtures. The test specimens with styrene–acrylic ester polymer and those with acrylic polymer showed during the first cycle, even after brushing, a swelling. The largest swelling (0.15 mm) was measured for the mixture with acrylic polymer. On the other hand, this was also the mixture that had the largest loss in height during all other cycles. This resulted in the largest cumulative loss in height of all mixtures at the end of the test (0.76 mm). The mixture with the styrene–acrylic ester polymer showed a moderate decrease in height during all cycles. At the end of the test, this concrete mixture had the smallest decrease in height (0.32 mm). The reference mixture, the mixture with vinylcopolymer and the mixture with styrene butadiene polymer showed the same pattern concerning the change in height. A moderate decrease was measured during cycles 1, 2 and 4 (for the reference mixture a slight increase during cycle 7) and a relatively high decrease was noticed after the third cycle. No significant difference was observed between these three mixtures at the end of the test. The control test specimens of all mixtures showed no significant change in height (not shown in the figure). This was also a confirmation of the fact that the inactivated solution was not corrosive to the concrete.

At the end of the total procedure, the change in weight of the different test samples was measured. The results of these measurements are given in Fig. 4 together with the results of the control test specimens (concrete test specimens that were treated with an inactivated simulation solution). All the control test specimens showed a slight increase in weight. This was probably due to the deposition of dead biomass. For the test specimens subjected to the active solution, the largest weight loss was measured for the mixture with acrylic polymer. The mixtures with the other polymers showed smaller weight losses than the reference mixture. Between these three polymer cement concrete mixtures no significant difference could be found.

Taking all measurements into account the addition of the styrene–acrylic ester polymer resulted in a slight increase in resistance of the concrete: the Ca^{2+} release, the decrease in height and the weight loss were 8%, 33% and 9% lower than for the reference concrete, respectively. On the other hand, the addition of the acrylic polymer had a negative influence on the resistance against the action of the bacteria. The addition of the styrene–butadiene and the vinylcopolymer resulted in neither an increased nor a decreased resistance.

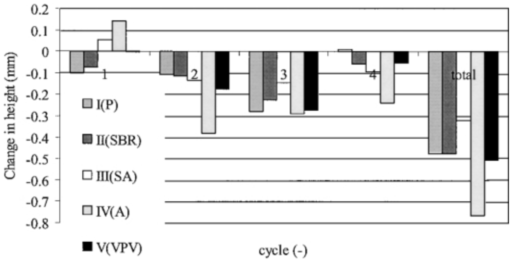

Fig. 3. Change in height of the different concrete mixtures.

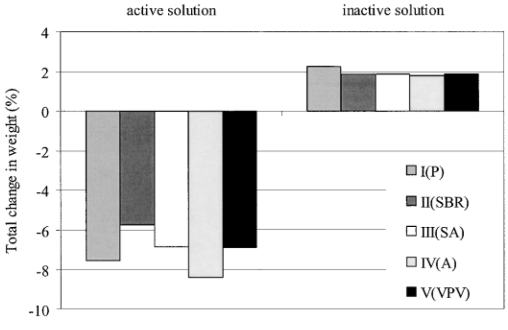

Fig. 4. Change in weight of the different concrete mixtures.

3.3. Chemical tests

The average change in radius (either decrease or increase of the radius with regard to the initial radius) of the different cylindrical specimens versus the number of measurements is shown in Fig. 5. A positive value means that the concrete swelled and a negative value indicates a loss of material of the concrete. For every cycle, the cylinders were measured twice: once before (odd measurements in Fig. 5) and once after brushing (even measurements). The alternating increase and decrease of the radius corresponds to alternating expansion and material loss of the concrete. The expansion of the cylinders occurred during the immersion in the sulfuric acid due to the production of expansive reaction products, such as gypsum. The decrease of the radius was mainly due to brushing of the cylinders but also occurred partially during immersion. Once the expansion of the concrete became too large, loss of adhesion of the expanded part occurred.

In Table 3, the concrete mixtures are classified in order of increasing average degradation for the successive cycles (based on the results of the measurements after brushing). Concrete mixtures of which the average degradation is not significantly different (determined by a Student–Newman–Keuls test with level of significance .05) are connected by a line.

The mixtures with styrene butadiene polymer and acrylic polymer seemed to be most vulnerable to degradation. Initially they showed the highest swelling (increase of the average radius with 0.3 mm after three cycles) and this was followed by the highest material loss of all mixtures (Fig. 5 and Table 3). The change from large expansion to loss of material occurred for the mixture with styrene butadiene polymer after the third cycle only. For the mixture with acrylic polymer, the change occurred after the fifth cycle. The large decrease of the average radius of these mixtures was confirmed during the following cycles. The same evolution was found for the reference mixture and the mixture with the vinylcopolymer. Yet, for these mixtures the change between increase and decrease of the radius occurred only after eight and seven cycles, respectively. The mixture styrene–acrylic ester polymer showed for almost every cycle a resulting increase of the average radius. This implies that the increase of the average radius due to expansion exceeded the decrease of the average radius due to brushing.

Table 3
Significant differences in average degradation (change in radius) between the different mixtures for the successive cycles

1	A	VPV	SA	SBR	P
2	SBR	VPV	A	SA	P
3	A	SA	VPV	P	SBR
4	A	SA	VPV	P	SBR
5	SA	VPV	P	A	SBR
6	SA	VPV	P	SBR	A
7	SA	P	VPV	SBR	A
8	SA	VPV	P	SBR	A
9	SA	VPV	P	A	SBR

Concrete mixtures are classified in order of increasing radius change and connected by a line if not significantly different (level of significance .05).

Fig. 6 shows the cumulative changes of the average radius of the different mixtures due to alternated immersion in the sulfuric acid solution. These values represent the sum of all differences between the values of the radius measured before and after brushing of the previous cycle. Hence, the effect of brushing is excluded and only the swelling remains. All polymer-modified mixtures, with the exception of the mixture with the acrylic polymer, showed a larger cumulative expansion than the reference mixture. For the mixture with the acrylic polymer, no expansion was measured after cycles 6, 7 and 8. Probably an expansion of the concrete had taken place during the immersion but the expanded material did not possess enough cohesion so that, during the immersion stage, loss of surface material had already taken place. Hence, for this mixture, the measured total increase of the average radius (Fig. 6) was in fact an underestimation of the real expansion that took place. Up to cycle 5, the reference mixture showed only a slight increase of the average radius (0.15 mm). However, after the fifth cycle, an increased expansion of the cylinders was measured during the immersion periods. The cumulative increase of the average radius after nine cycles had the same magnitude

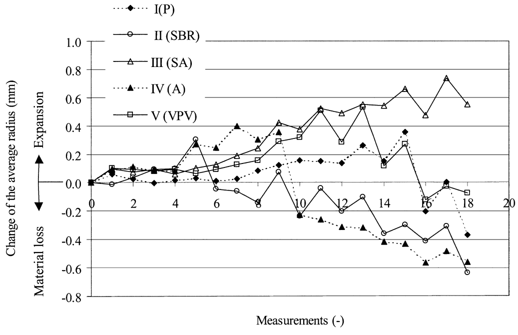

Fig. 5. Change of the average radius of the different concrete mixtures during the chemical test.

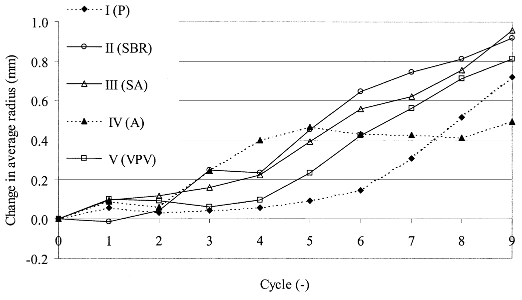

Fig. 6. Change of the average radius of the different concrete mixtures only due to alternated immersion in the sulfuric acid solution.

as the values measured for the mixtures with the styrene butadiene polymer, the styrene–acrylic ester and the vinylcopolymer.

Fig. 7 shows the cumulative changes of the average radius of the different mixtures due to brushing of the cylinders. It represents the sum of all differences between the values of the radius measured after brushing and before brushing during the same cycle. The addition of the styrene butadiene polymer resulted in increase of material loss due to brushing. After nine cycles a total cumulative decrease in the average radius of almost 1.6 mm was measured. Although the mixture with acrylic polymer showed a large cumulative decrease in the average radius (Fig. 7), this was mainly due to the fairly high material loss after the fifth cycle (decrease of average radius by 0.6 mm). For all other cycles a relatively low amount of material loss was measured (decrease of average radius by 0.1 mm for every cycle). The reference mixture and the mixture with the vinylcopolymer showed the same pattern concerning the loss of material due to brushing. Up to cycle 6, almost no loss of material was measured. From this cycle onwards, the brushing of the cylinders caused an increasing amount of material loss. Although the mixture with the styrene–acrylic ester polymer showed the highest expansion during the alternated immersion, little material loss due to brushing was measured.

The average Ca^{2+} contents of the sulfuric acid solutions in which the different mixtures had rotated are presented in Fig. 8. The values for all mixtures were always larger than the initial Ca^{2+} content of the solutions. This indicates that all specimens were affected by the sulfuric acid solution. The values followed the same evolution as the changes of the radius of the specimens. The change between increase and decrease of the radius of the reference mixture and the mixture with the vinylcopolymer, which occurred after 16 weeks (8 cycles), was also reflected in the values of the Ca^{2+} content. Only the specimens with the styrene–acrylic ester polymer had, for all measured cycles, a Ca^{2+} content around or less than 1000 mg/l in the solution. These specimens also showed no decrease of the average radius compared to the initial radius (Fig. 5).

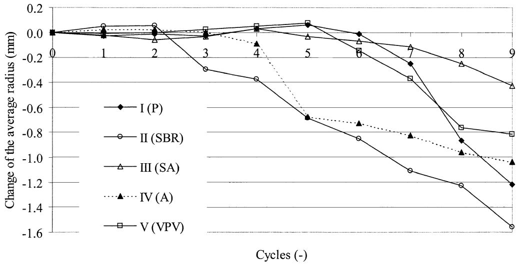

Fig. 7. Change of the average radius of the different concrete mixtures only due to brushing of the cylinders.

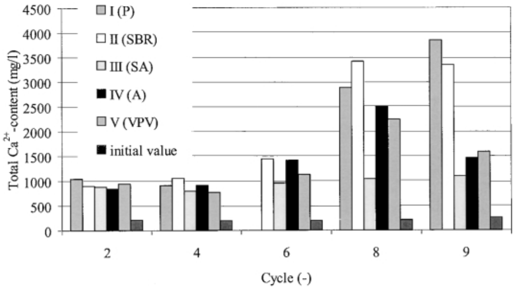

Fig. 8. Ca^{2+} content of the different solutions.

3.4. Comparison between the microbiological tests and the chemical tests

Relative to the change of dimension of the test specimens, the microbiological test was much more aggressive for the concrete than the chemical test. For both tests, the immersion in the aggressive solution lasted 10 days per cycle. At the end of the microbiological test (after four cycles), a cumulative change in height ranging from 0.3 to 0.8 mm was measured. These values were also reached at the end of the chemical test, but this test lasted for nine cycles. During the microbiological test, the concrete was subjected to an environment that changes smoothly from pH 8 to pH 1. In the chemical test, the specimens were immediately subjected to a pH of about 1. Although the pH at the end is the same in both tests, the sulfate concentration of the solutions was much higher in the chemical test than in the microbiological test. The concentration in the chemical test was about 9.3 g/l whereas for the microbiological test it started at about 0.2 g/l and increased up to 1 to 2.5 g/l. Yet, there was a much higher corrosion rate in the microbiological test.

Stirring the test specimens in the solution during the microbiological test was probably more destructive than the brushing of the test specimens during the chemical test. This proposition was somewhat confirmed by the fact that, with the exception of the first cycle, no expansion of the concrete was measured during the microbiological test in comparison with the chemical test. In this test, even after nine cycles, some mixtures still showed an increase in dimension of the test specimens. Furthermore during one cycle of the microbiological test, the concrete was constantly immersed whereas during the chemical test the immersion was alternated with drying in the air.

Concerning the difference in resistance of the different concrete mixtures against both types of corrosion, the concrete mixtures at the extremes were the same in both tests. The mixture with the acrylic polymer had the lowest resistance and the mixture with the styrene–acrylic ester polymer showed the highest resistance in both tests. The mixture with the acrylic polymer showed the same corrosion pattern during the microbiological test as well as during the chemical test. Concerning the change in dimension of the test specimens, for both test methods, first a swelling was noticed followed by a loss of material. The higher resistance of the mixture with the styrene–acrylic ester polymer in the chemical test was mainly expressed by the small decrease in dimension due to brushing (65% smaller than for the reference) and the high expansion due to immersion. Both phenomena together led to the highest expansion. For the microbiological test, the higher resistance was expressed in terms of low material loss (small decrease in height).

For the resistance of the three other mixtures, some discrepancy for the two tests was observed. In both tests, these concrete mixtures showed an average corrosion. The mixture with the styrene butadiene polymer tended to be more resistant than the reference mixture in the microbiological test, although this was not the case for the chemical test. The opposite could be said for the mixture with the vinylcopolymer.

4. Conclusions

The addition of selected polymers to the concrete mixtures was associated with an increase of air content of the fresh concrete. This resulted in a higher slump and flow of the fresh concrete and, also, in a lower density. The addition of the acrylic polymer was associated with a high level of air entrainment (9.7%), which resulted in concrete with poor mechanical properties.

All measured parameters of the microbiological test provide evidence that the simulation of the corrosion process works well. Within a time span of four cycles (4 × 17 days), differences between the concrete mixtures concerning the resistance could be noticed. Depending on the type of polymer, slight increase (styrene–acrylic ester polymer) or decrease (acrylic polymer) in resistance against the microbiological corrosion was noticed.

The addition of the polymers caused an increase in expansion of the concrete during the immersion in the sulfuric acid solution in the chemical test. For some polymer types (styrene butadiene and acrylic polymer), the increased expansion was associated with an increase of material loss due to brushing of the concrete after the immersion cycles. The superposition of these two phenomena led to a more unfavorable behavior regarding the resistance of these concrete mixtures compared to the reference mixture. For other polymers (styrene–acrylic ester polymer and the vinylcopolymer), the increase in expansion was not associated with an additional material loss due to brushing. This led to better results concerning the resistance of the concrete.

Although both test methods gave roughly similar tendencies, there was some divergence for a few of the concrete types.

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