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Accelerated test design for biodeterioration of cementitious materials and products in sewer environments

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Abstract – A new test method for evaluating the resistance of cementitious products and materials to biogenic acid attack has been developed. It consists of inoculating pipes with a highly diverse microbial consortium (from an urban wastewater treatment plant), and trickling a feeding solution containing a safe and soluble reduced sulfur source over the inoculated surface in order to select a sulfur-oxidizing activity. A sulfur substrate, thiosulfate, was used in the feeding solution, which facilitated the monitoring of the bacterial activity, and of the leaching of cementitious ions. The functioning of the test in terms of selection of sulfur oxidizing microorganisms, acid and sulfate production, and degradation mechanisms occurring in the cementitious materials has been validated previously. This paper aims (i) to evaluate the reproducibility and repeatability of the test when the source of the sludge used for the inoculation is changed and (ii) to optimize the test design. The following changes were carried out with a view to intensifying the biological and chemical reactions: change of sulfur substrate (tetrathionate instead of thiosulfate), increased sulfur flow rate at the surface of the biofilm, and increased temperature. The results highlight good reproducibility of the test: the change in the inoculum and in the sulfur substrate led to the same phenomena in terms of (i) transformation of reduced sulfur species into sulfate and production of acid and (ii) degradation mechanisms of the cement linings. Moreover, the change of the sulfur substrate combined with the reduction of the exposed surface locally intensified the alteration kinetics. In contrast, in the experimental conditions, the increase in temperature did not seem to have any positive influence on the microbial activity.

Key words: Biodeterioration / sewers / biological test / mortars / microstructural changes

1 Introduction

Biodeterioration of cementitious materials in sewer networks is due, in particular, to the biological oxidation of the hydrogen sulfide (H_2S) released in the gas phase of the sewers into sulfuric acid (H_2SO_4) [1]. This concrete deterioration leads to major problems during the collection and treatment of wastewaters, needing considerable financial investment for restoration [2]. Cementitious products intended for sewer networks are currently qualified only by chemical tests in dedicated standards although it is acknowledged that these tests are not representative of phenomena occurring in sewer environments [3–5]. In this context, several tests with different designs and principles have been proposed in the literature so far, with no consensus being reached on any test method [6].

This study proposes a new biological test intended for products and materials in sewer systems. It consists of inoculating the inner surface of cementitious linings of pipe products with a highly diverse microbial consortium (activated sludge) and in trickling a feeding solution, containing a reduced sulfur source, over the inoculated surface in order to select sulfur-oxidizing activity [7]. Safe, soluble sulfur species, identified as chemical and biological intermediates in the oxidation of H_2S species into SO_4^{2-}, were chosen to replace H_2S (toxic gas) as sulfur sources. Thiosulfate ($S_2O_3^{2-}$) was the sulfur species tested in the first series of experiments.

Previous studies validated the representativeness of the test in terms of microbial selection, and chemical, mineralogical and microstructural changes in the cementitious matrices, and its ability to discriminate between

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two different manufactured linings (CEM III and CAC mortar linings) through short-term experiments [7–9].

The present paper aims to present further experimental campaigns carried out to evaluate the reproducibility of the test and to optimize its design. The following parameters were varied: the nature of the sulfur substrate (tetrathionate ($S_4O_6^{2-}$) instead of thiosulfate), the inoculum, the area of the exposed surface, the hydraulic retention time of the biogenic acid, and the temperature. The composition of the leaching solution was monitored over time (tetrathionate, sulfate, calcium, pH). At the end of the test (4 months), the deterioration of the mortar linings was analysed by SEM observations coupled with analyses by EDS and EPMA fitted with a BSE detector.

2 Experimental aspects

2.1 Pipe products

The pipe segments were 200 mm long and 200 mm in internal diameter. A pipe of Saint-Gobain PAM is composed of a ductile cast-iron tube with a cement mortar lining. The cementitious lining acts as a protective layer for the ductile iron structure of the sewer pipe (chemical, sacrificial protection against aggressive environments). The lining was a few millimetres thick and was produced by centrifugation of mortar on the inner surface of the iron pipe (Saint Gobain PAM production process [10]). The placing of cementitious linings inside the pipes by centrifugation leads to spatial segregation of the different mortar compounds. Figure 1 shows SEM observations of cross sections of control specimens: CAC (Calcium Aluminate Cement with siliceous aggregates) and BFSC (Blast Furnace Slag Cement with siliceous aggregates).

The linings show a heterogeneous, double-layered structure composed of:

(i) A paste layer (inner part of the lining and in contact with the biofilm). The paste layer is 1 mm thick for the CAC lining and 0.8 mm thick for the BFSC lining.
(ii) An outer layer (in contact with the ductile cast iron tube) made of mortar. The CAC mortar layer is 3.8 mm thick, whereas the BSFC one is 3.0 mm thick.

No initial cracking was observed in either of the control linings.

The composition of the hydrated matrix of the paste layer is given in Reference [9].

2.2 Test set-up

2.2.1 Architecture of the test

The pilot apparatus was made up of two vertical parallel pipe-reactors (Diameter: 200 mm; Length: 200 mm), each mainly composed of a segment cut from a real sewer pipe (Fig. 2).

Before the start of the experiment, a fraction of the surface of each cementitious lining was inoculated with an activated sludge consortium (high diversity mainly composed of heterotrophic and autotrophic bacteria from organic matter and the nitrogen removal, associated with fungi). The efficiency of this type of inoculation, in terms of implementation of a sulfur oxidizing activity in controlled conditions had been highlighted previously [7]. The surface inoculated was delimited with grooves created by longitudinal sawing (with a hand grinder). The grooves were filled with an epoxy resin (Fig. 3). The initial area of each longitudinal inoculated zone was 100 cm^2 (5×20 cm^2) (Fig. 3a). A constant flow of a mineral solution (Tab. 1 for the composition), 6 in pH, containing a sulfur source (tetrathionate from the dissolution of $Na_2S_4O_6$) was trickled onto the cement surface (Fig. 3b), leading to an active exposed surface of 0.002 m^2. The aim was to produce a liquid phase that could be considered as a condensate at the surface of the exposed linings (reactive zone at the top of a pipe in real sewers). The surface exposed to this trickling was considered as the reactive area. Soluble sulfur sources were used in this device to enable fast and quantifiable selection of sulfur-oxidizing activity at the lining surfaces. Moreover, the use of safe sulfur sources made the laboratory handling easier than the use of H_2S [7–9].

2.2.2 Optimization of test design

The aim of the present study was (i) to test the repeatability and the reproducibility/robustness of the test method, (ii) to intensify the microbial reactions and the deterioration of the material in order to accelerate the kinetics of the test. The robustness of the test method could be reduced by a possible variability in the test response linked with non-controlled biodiversity. An activated sludge collected in a wastewater treatment plant (Villefranche du Lauragais, near the city of Toulouse, France) other than that of our previous study was therefore used here. The repeatability was assessed by performing two tests at the same time on two CAC linings in the same conditions (marked CAC_a and CAC_b in what follows).

In order to intensify the reactions with regards to our previous campaign [7,9], the design of the test was modified as follows:

– Thiosulfate, an intermediate in the oxidation of sulfur into sulfate was used as the sulfur source in our previous study. Tetrathionate was tested as the sulfur source in the present experiment. As thiosulfate, tetrathionate is a soluble compounds (easly used, leading to safe experimental conditions), stable in abiotic test (during the experiment and in the range of feeding solution concentrations), involved in the biological oxidation of reduced sulfur compounds by numerous sulfur oxidizing bacteria. In other hand, tetrathionate compare to thiosulfate was choosen in order to increase the proton production of the biological reaction: 1 mole of sulfur oxidized from tetrathionate $S_4O_6^{2-}$ produces 1.5 mol of H^+ whereas one mole of

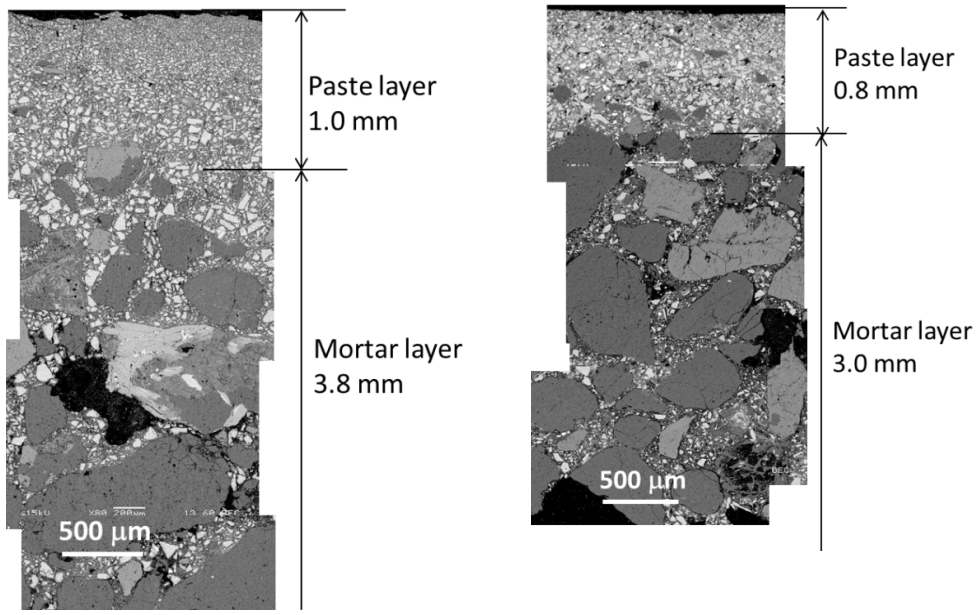


Fig. 1. Observation with SEM (BSE mode) of control specimens, cross sections of mortar linings, (a) the CAC lining and (b) the BFSC lining (×80). The paste layer is in contact with the biofilm, the mortar layer is in contact with the ductile cast-iron tube.

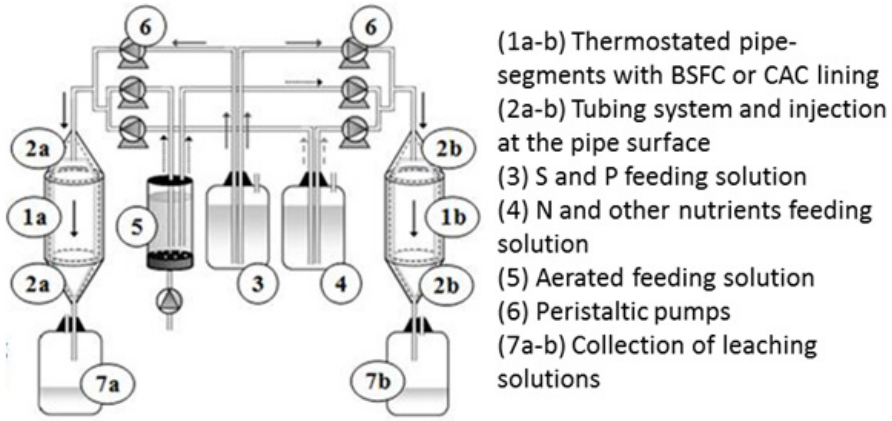


Fig. 2. Pilot for biodeterioration test.

Table 1. Chemical compositions of the feeding solution.

Mineral	S-$S_4O_6^{2-}$	N-NH_4^+	P	Mg^{2+}	Mn^{2+}	Fe^{3+}	Cl
Concentration (mol/L)	5×10^{-3}	2.60×10^{-4}	1.3×10^{-5}	5.1×10^{-6}	4.3×10^{-7}	2.6×10^{-7}	2.7×10^{-4}

sulfur oxidized from thiosulfate $S_2O_3^{2-}$ produces 1 mol of H^+.

– The sulfur substrate flow was increased: it was set at about 7.44 molS/m^2/d vs. about 0.15 molS/m^2/d in our previous study. To this end, the concentration of the sulfur substrate of the feeding medium was kept similar in both studies (4.00×10^{-3} molS/L for tetrathionate and 6.32×10^{-3} molS/L for thiosulfate at the end of the experiment) but the flow of leaching solution was increased (155 ml/h vs. 50 ml/h respectively) and the area of exposed surface was decreased (0.002 m^2 vs. 0.05 m^2 from [7]).
– One test, performed on a BSFC lining (labelled BFSC_30 °C), was carried out at 30 °C (the feeding solution and the pipes were thermostated) in order to explore temperature as a possible way to intensify the biological and chemical reactions. The other tests (performed on a BSFC and on the CAC linings)

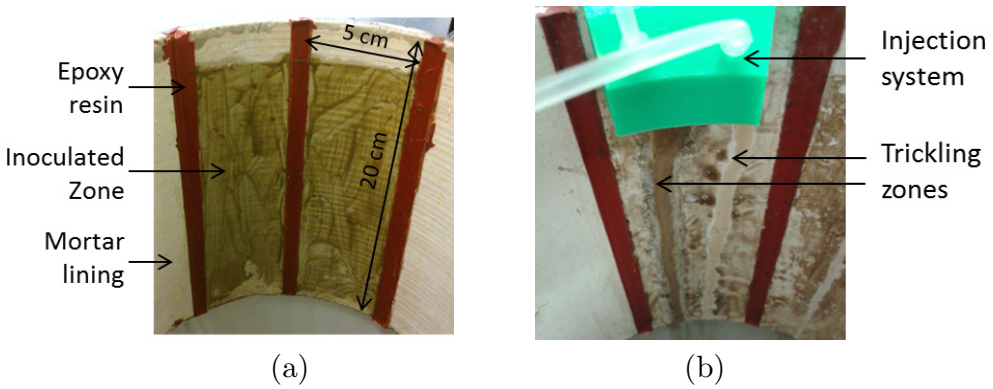


Fig. 3. Inoculated zones of the pipe segments (a) and trickling of the S-bearing substrate solution (b).

were carried out at 20 °C and respectively labelled BFSC_20 °C, CAC_a and CAC_b.

The composition of the feeding solution for the four lines is given in Table 1. The test was run for 112 days.

2.3 Chemical analyses of the leaching solution

The leaching solution was regularly sampled and analysed: pH and concentrations of tetrathionate, sulfate and dissolved calcium were measured.

The concentration of SO_4^{2-} was measured by anionic chromatography (DIONEX: IC25, IonPacTM AS19, at 30 °C with eluent generator cartridge EGC III KOH). The dissolved calcium concentration was quantified by cationic chromatography (DIONEX: ICS 2000, IonPac CS12, at 30 °C with eluent generator cartridge EGC III MSA for methanesulfonic acid).

Without biological activity, thiosulfate was found to be stable in a solution trickling at the surface of cementitious linings [7], as was tetrathionate (data not shown). Consequently, biological acid production could be calculated from the sulfate production, with the hypothesis of a low proportion of sulfate trapped inside the cementitious matrix (controlled by sulfur mass balances in the leaching solution as shown in Ref. [7]).

2.4 Microstructural characterization of the cementitious linings

Microstructural observations and chemical analyses of exposed and control mortar lining specimens were performed using a scanning electron microscope (Jeol JSM-6380LV, pressure: 60 Pa, accelerating voltage: 15 kV) fitted with an EDS detector (Rontec XFlash® 3001). The observations were made on fractures and on flat polished sections previously coated with a carbon film. The flat polished sections were prepared as described in References [9, 11]. The flat polished sections were also analyzed with an electron microprobe analyzer (CAMECA SX-Five-WDS detector) fitted with a BSE detector in order to produce quantitative chemical mapping. The accelerating voltage and current were respectively 15 keV and 20 nA so as to collect enough signal for quantification with low counting times. The interaction volume of the beam was about 10 μm^3. Data were acquired on a 101 × 126 point grid (step size between 2 points: 2 μm, counting time: 0.25 s on peak and 0.06 s on background for each point). Calibration was performed on natural and synthetic materials.

3 Results

3.1 pH and composition of leaching solution

Figure 4 presents the evolution of pH, and cumulative sulfate, calcium and H^+ of the different trickling solutions collected at the bottom of the pipe segments according to time.

For all the linings tested (BSFC linings at 20 and 30 °C, CAC_a and CAC_b), the initial pH of the leaching solution started around 9, probably due to the surface carbonation of the materials during the storage, miming the initial abiotic decrease of surface pH in sewers environment (Islander et al. 1991) associated to the pH of the trickling solution (pH = 6). The pH of the leaching solution decreased from 9 to 4 in the first 30 days of the experiment (Fig. 4a). The decrease of pH was linked with the transformation of tetrathionate by the biological activity, which produced H^+ and SO_4^{2-} in the trickling solution (Figs. 4b and 4d). Despite some instability of the pH at 53, 60 and 103 days due to occasional clogging of the tubing, an overall acidification was observed, pH being around 3.5 between day 50 and the end of the experiment. The sulfate production showed a two-stage evolution (0–50 and 50–117 d) with an intensification of the kinetics between the two stages. On the second period (50–117 d, matching pH ≈ 3.5), the sulfate production was almost linear according to time with an average sulfate production rate of 1.12 mmol/d (or 560 mmolSO_4^{2-}/d/m^2). The rate of H^+ production ion the same period (calculated from the sulfate production [7]) was 1.68 mmol/d (or 840 mmol/d/m^2). The leaching of calcium showed significantly different behaviour between CAC and BFSC linings. A two-stage evolution was observed for BSFC_20 °C and the two CAC: an initial period

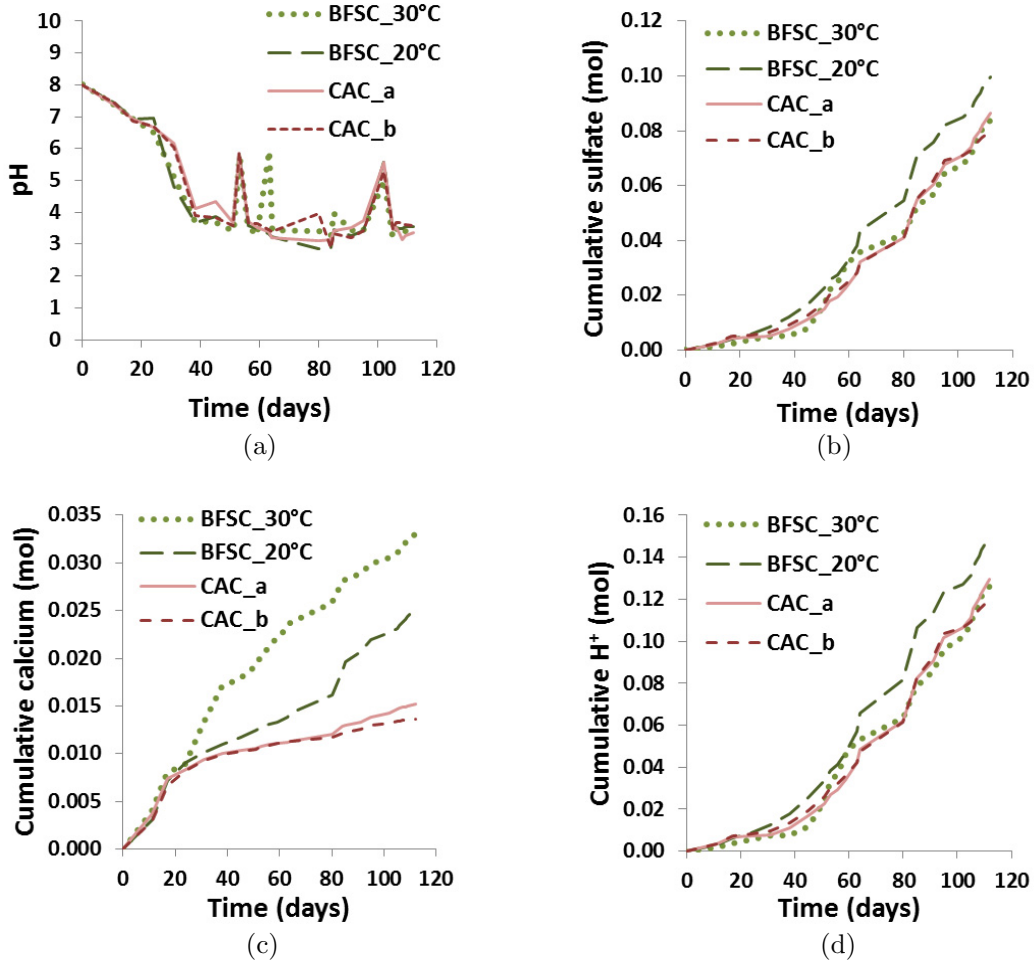


Fig. 4. Evolution of (a) pH, (b) cumulative sulfate, (c) calcium and (d) H^+ of the leaching solution according to time.

(0–20 d) with sharp release of calcium (surface dissolution phenomena) and a second period (20–117 d) with smoother kinetics, probably due to diffusion kinetics becoming installed. The increase of temperature did not cause any increase in the biological activity (same sulfate production for BSFC_20 °C and BSFC_30 °C – the biofilm suffered drying), but it impacted the chemical processes by increasing the leaching of calcium (Fig. 4c) (discussed in 4.2). Finally, the repeatability of the test was tested on CAC_a and CAC_b data, with very similar evolutions of pH and cumulative sulfate and calcium for the two linings.

3.2 Intensification of biological process responsible for mortar lining degradation

Figure 5 compares the acid production and the calcium leaching of the cementitious materials exposed to different biodeterioration tests, according to the sulfur substrate: H_2S for the "Hamburg test" presented in Reference [4], $S_2O_3^{2-}$ for our previous study [7] and $S_4O_6^{2-}$ in the present paper. Mortars or linings of BSFC and CAC were studied in each case. The intensification of biological processes obtained from the change of sulfur substrate nature ($S_2O_3^{2-}$ replaced by $S_4O_6^{2-}$) and the increase of sulfur substrate flow (0.15 to 7.44 molS/m^2/d) is clearly visible when the results in Reference [7] are compared with those of the present study (plots C to F vs. plots A and B). Figure 5 also confirms that the increase in temperature had no positive effect on the biological reactions in the testing conditions (plots C and D) and that the repeatability of the test was quite good (CAC_a and CAC-b, plots E and F). Moreover, the total quantity of acid to which the specimens were exposed in this study (65.7 molH^+/m^2 on average for the different specimen types) is significantly higher than that in the Hamburg chamber (31.3 molH^+/m^2) despite the shorter duration of the test (3.7 months vs. 6.7 months).

3.3 Microstructural analysis on BFSC mortar

The surface of the BFSC linings exposed to the biodeterioration test was not regular (Fig. 6). In the areas where the solution trickled, so where biofilm was implanted ("run-off zone" in Fig. 6), the cement paste layer was dissolved to a depth of 700 μm compared to the zones

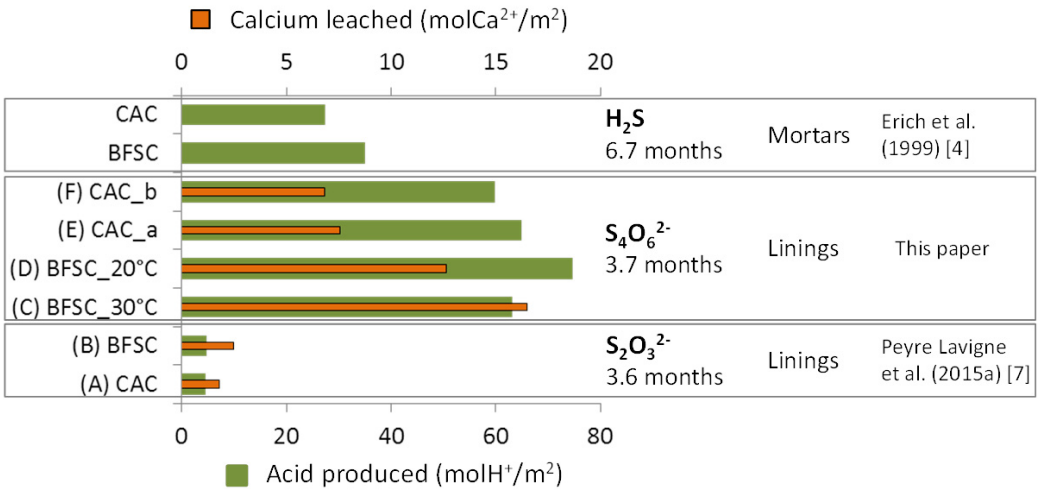


Fig. 5. Comparison of biogenic acid produced and calcium leached from cementitious materials (mortars or linings made of CAC or BSFC) exposed to biodeterioration tests with different sulfur substrates.

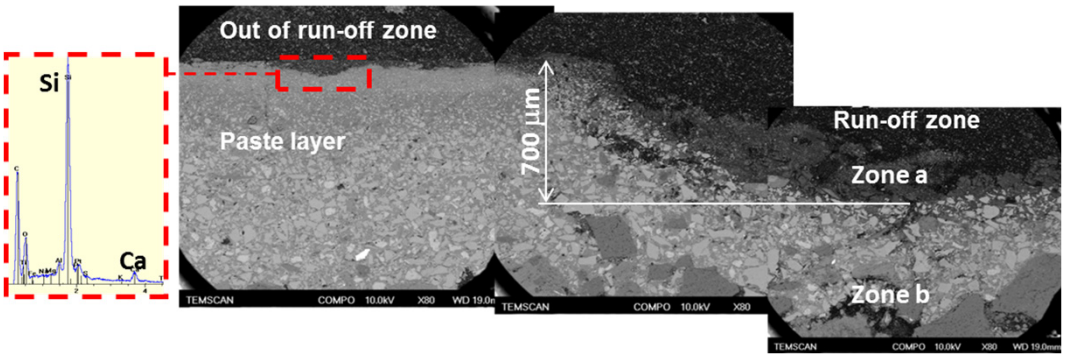


Fig. 6. Observation with SEM (BSE mode) of the surface of the BSFC_20 °C lining (flat polished section, ×80) and EDS analysis in the rectangular red zone.

where no feeding solutions were trickling. The first aggregates under the paste layer were thus uncovered. These hollowed out zones were visible to the naked eye on the cross sections of the specimens.

Outside the run-off zones, the outer layer of the paste (near the zone where the biofilm was developed, dotted red rectangle in Fig. 6), 20 μm to 70 μm thick, was strongly decalcified and mainly composed of Si (EDS spectrum on the left-hand side of the figure). It was thus probably composed of a silica gel. The zone under this surface was slightly decalcified, probably due to the biogenic acid solution produced nearby outing sometimes of the classic way. No cracks were observed in the paste layer or deeper in the mortar layer.

In the run-off-zone, where the biofilm was active throughout the entire duration of the test, the lining showed two main zones from the surface to the core of the specimen:

– Zone a (surface zone), 100 to 300 mm thick, had a low density and was made of silicon only (probably a silica gel).
– Zone b, under zone a, with a similar density to the sound specimen, showed intense cracking, typical of secondary expansive ettringite formation. These cracks were observed at many places and at different depths in the matrix (Fig. 7). EDS analyses and observations in SEI mode confirmed the formation of ettringite (Fig. 8).

3.4 Microstructural analysis on CAC mortar

The surface of the CAC linings exposed to the test was regular. Notably, no loss of material was observed in the run-off zones. The outer layer of the specimen in contact with the biofilm showed chemical and density zonation (Fig. 9).

From the core to the surface:

– Zone 1 had density and composition similar to those of the control specimen (Fig. 1). The density of residual anhydrous grains was high.
– Zone 2, with lower density, 100 to 150 μm thick, was decalcified and composed mainly of aluminium (Figs. 9 and 10) (probably in the form of AH_3 [8,9]), most residual anhydrous grains had been dissolved.

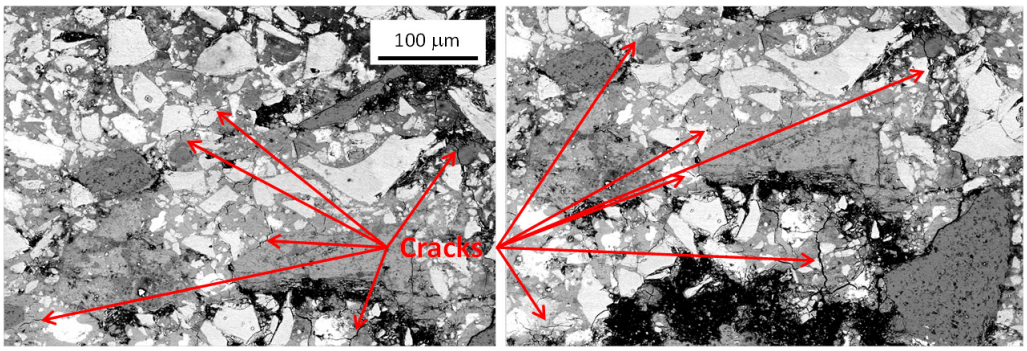


Fig. 7. Crack network observed with SEM (BSE mode) of the mortar layer of the BSFC_20 °C lining under the run-off zone (flat polished sections, ×200).

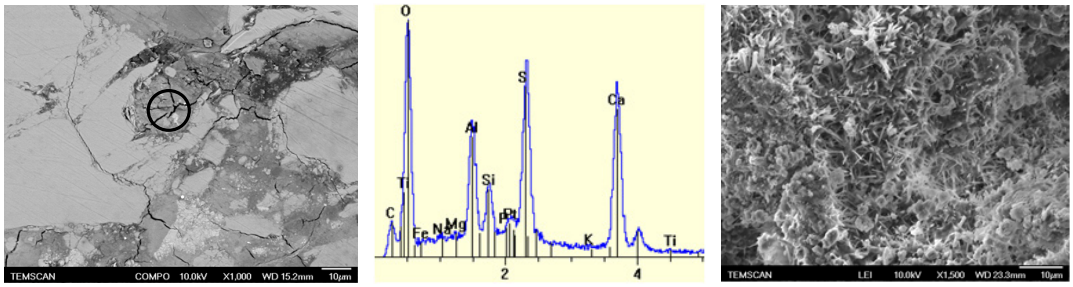


Fig. 8. Observation with SEM of the mortar layer of the BSFC_20 °C lining under the run-off zone. Left: typical aspect of massive ettringite on flat polished section (BSE, ×1000), middle: EDS analysis of the circled zone (typical spectrum of ettringite). Right: ettringite needles observed on fractures (SEI, ×1500).

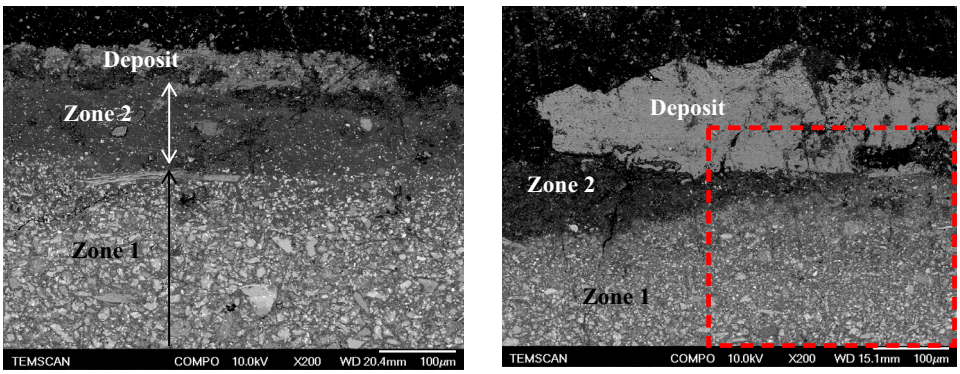


Fig. 9. Observation with SEM of the mortar layer of the CAC lining exposed to the test (flat polished sections, BSE mode, ×200), red square zone: area analysed with EPMA and shown in Figure 10.

– At the surface, a deposit was observed in some places close to the run-off zones. EPMA chemical mapping showed that the stoichiometry was typical of gypsum.

No cracks were observed in the specimen as a whole.

4 Discussion

Our previous study showed that, from an unspecified environmental inoculum, sulfur-oxidizing bacteria (Thiomonas sp. and Thiobacillus sp. identified from 16% to 63% of the total 16S rDNA by samples analysed) were selected at the surface of BFSC and CAC linings by the use of thiosulfate as a sulfur source and the control of the operating conditions. Thiosulfate is an intermediate in the oxidation of reduced sulfur compounds [7, 12] and a sulfur substrate used by numerous sulfur-oxidizing bacteria, including acidophilic bacteria [13–15]. The use of a soluble sulfur source, such as thiosulfate or tetrathionate, as the sulfur substrate for a test intended to evaluate the behaviour of cementitious materials subjected to biogenic acid attack in a sewer-like environment has been discussed previously [7]. The representativeness of the test in terms of degradation phenomena occurring in the cement matrix that was exposed and its ability to discriminate two cement linings, BFSC and CAC mortars, known to have different behaviours when faced with biogenic acid [3, 4, 8, 9, 16, 17] has also been validated [9, 17].

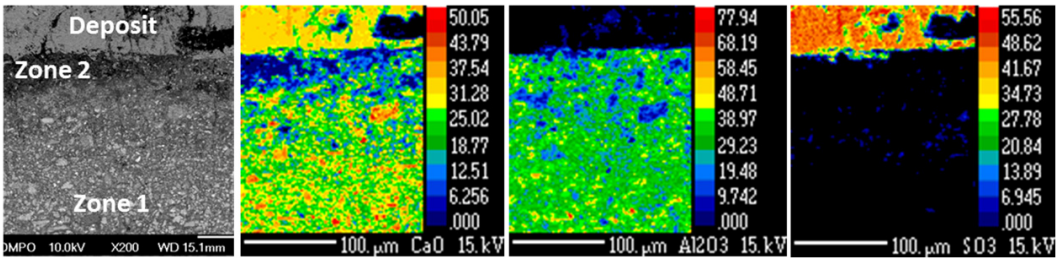


Fig. 10. Observation in BSE mode of the zone in the red square of Figure 9 (left) and quantitative chemical mapping obtained with EPMA (CaO, Al_2O_3 and SO_3) (right).

This paper first aimed to test (i) the reproducibility of the test design when the source of the sludge used for the inoculation was changed and (ii) its repeatability. The second aim of the study was to optimize the test design. Some changes made with a view to intensifying the reactions (biological activity and chemical changes of the materials).

4.1 Analyses of the test design

Despite the sludge source change, the type of metabolism selected in the test (sulfur oxidizing) was the same as in our previous study: sulfate and acid were produced from the oxidation of the reduced form of sulfur used (tetrathionate). The same types of deterioration mechanisms were also observed:

- The BFSC lining showed intense cracking of the outer zone of the paste layer because of the precipitation of secondary ettringite. The outer layer of the paste zone was also decalcified and mainly amorphous.
- For the CAC lining, an amorphous layer was observed at the surface of the material. This layer, in contact with the biofilm, was decalcified and probably composed of AH_3 (which is consistent with the literature [4, 18]). No cracks were observed in the lining.

The reproducibility of the process was tested, and showing some good results.

The repeatability was also tested on CAC_a and CAC_b specimens in the same conditions with interesting results. The analyses of the leaching solutions showed the same production of sulfate (thus the same biogenic acid production (65 and 60 $molH^+/m^{-2}$ for the CAC_a and CAC_b respectively)), in terms of total, as well of the dynamical evolution (Figs. 4 and 5). The leaching of calcium showed the same tendencies (7.6 and 6.8 $molCa^{2+}/m^{-2}$ for the CAC_a and CAC_b respectively). Moreover, the observations on the specimens showed the same patterns of chemical and microstructural degradation.

4.2 Optimization of test design

The changes made to the test design in this study were: a change of sulfur substrate, an increase in sulfur flow rate at the surface of the biofilm, and an increase in temperature. Intensification of the biological reactions and of the chemical reactions within the cementitious matrix was obtained through the change of sulfur substrate (thiosulfate replaced by tetrathionate) and the increase in the flow rate of sulfur substrate (7.44 $molS/m^2/d$ vs. 0.15 $molS/m^2/d$ in our previous study, obtained mainly by the increase of the leaching solution flow and the decrease of the area of exposed surface). The oxidation of tetrathionate produces 1.5 times more H^+ per atom of sulfur oxidized than the oxidation of thiosulfate. Moreover, if no nutritional limitation occurs, the higher the flux of the reduced sulfur source, the higher the biological activity (in our case, the excess of mineral nutrients such as nitrogen and phosphorous was adjusted to obtain the highest biological activity and to avoid any precipitation of unrepresentative minerals such as complex phosphorous salts (struvite, hydroxyapatite, etc.).

The differences observed between this study and our previous one [9] were:

- Higher biogenic acid production (15 folds higher production on the same BFSC lining, and 13 folds higher on the same CAC lining) and greater calcium leaching (5 folds higher on BFSC lining and 4 folds higher on CAC lining) (Fig. 5).
- More intense alteration phenomena: the outer layer of BFSC lining was dissolved in the zones exposed to the trickling solution, which was not observed previously. This may have been due to higher local concentration of acids at the biofilm-cementitious matrix interface. Moreover, greater degraded layer thicknesses were obtained: whereas the deterioration phenomena were formerly limited to the paste layer of the BSFC lining (the degraded layer thickness was 700 μm at the end of the test), here, precipitation of secondary massive ettringite was observed deeper in the specimen at many locations in the mortar layer (degraded layer depth greater than 1 mm).

Moreover, the representativeness of the test, in terms of deterioration phenomena in the material, was improved through the changes made in this study. Precipitation of gypsum, typical of concrete deterioration in sewer environments [5, 19, 20], was observed at the surface of the specimens. The formation of gypsum was favoured by the creation of super-saturation areas close to the run-off zone (through the limitation of the trickling area), as already suggested [9].

The discriminating power of the test was also increased since the difference in behaviour between the two linings was accentuated: the degraded layer depth for the CAC was similar in both our campaigns (about 150 μm), whereas the degradation phenomena were intensified (local dissolution of the paste) and the degraded layer depths were increased for the BFSC lining.

Finally, no positive effect was observed of the increase in temperature from 20 °C to 30 °C. The tests carried out on BSFC_20 °C and BSFC_30 °C did not show any intensification of biological activity (no increase of sulfate) mainly because the biofilm suffered drying in these conditions of temperature and relative humidity (50%). Nevertheless, even if the dissolution constant (for portlandite for example) decrease with the an increase of the temperature, some accelerating effects were obtained on the chemical phenomena (larger quantity of calcium leached at 30 °C than at 20 °C), probably due to the modification of the thermodynamical and transport propoerties (diffusion coefficient) with the temperature.

5 Conclusions

Based on a previous test design for industrial cementitious linings for sewage applications, this study focused on (i) evaluating the reproducibility of the use of an unspecified microbial consortium as inoculum and (ii) optimizing the test design by the intensification of the biological acid production and its impact on the cementitious matrix. The intensification of the biological acid production was brought about by changing the reduced sulfur source (from thiosulfate to tetrathionate) and increasing the fluxes of reduced sulfur at the surface of the cementitious linings. The increase of the aggressiveness of the acid attack on the cementitious matrix was obtained by decreasing the area of the exposed surface. First, the use of an unspecified microbial consortium from wastewater treatment as the inoculum was positive. Secondly the optimization of the test design enabled an increase of the biological acid production (from 4.6 $molH^+/m^2$ for the previous test with thiosulfate and large exposed surface area, to 74 $molH^+/m^2$ in this paper for BFSC at 20 °C). This led to an increase in the leached calcium (from 1.8 $molCa^{2+}/m^{-2}$ to 12.6 $molCa^{2+}/m^{-2}$ for a BFSC lining). Moreover, the new test design intensified the discrimination between Portland based linings and calcium aluminate linings. At the same time, the chemical and microstructural evolutions of the cementitious linings were still representative of those observed in a sewer environment, with the observation of gypsum at the surface and ettringite in the depth of the deteriorated lining. The reproducibility and repeatability of the test were evaluated with positive first results. Further works are under way as part of the French research program DURANET, including the validation of the test design with respect to field investigations.

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