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Étude de la biocolonisation des composites mortier-polymère
Biocolonisation of polymer-modified mortars

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Résumé

Les composites mortier-polymère sont des matériaux cimentaires incorporant des polymères dans des proportions allant de 5 à 20 % en masse, par rapport à la masse de ciment. Ces matériaux sont de plus en plus utilisés pour des applications de protection et/ou de réparation des surfaces en béton telles que des façades, des ouvrages d'art ou encore des canalisations de réseaux d'assainissement. De nombreuses études ont en effet montré la participation des polymères à l'amélioration des propriétés fonctionnelles de ces matériaux (e.g., l'augmentation de la résistance mécanique et de l'adhésion, la diminution de la porosité et de la perméabilité). Toutefois, à ce jour, aucune étude ne traite de l'interaction entre les composites mortier-polymère et les micro-organismes. Or, une telle interaction peut conduire à la modification non désirée des propriétés de ces matériaux, entraînant des modifications esthétiques ou structurelles. Ainsi, l'objectif principal de cette étude est d'évaluer la bioréceptivité des composites mortier-polymère lorsqu'ils sont utilisés pour la protection de surfaces en béton, dans des conditions proches de celles rencontrées *in-situ*. Deux formulations de composites mortier-polymère et un mortier exempt de polymère sont caractérisés à l'état durci, avant d'être exposés à des conditions représentatives d'une application en mortier de réparations. Ces matériaux sont alors testés via deux essais accélérés, le premier visant à conduire à la bioaltération des matériaux, le second visant à les biodétériorer. Ces essais permettent alors d'évaluer la résistance de chaque formulation à la biocolonisation de manière à établir des préconisations d'utilisation de ces matériaux.

Abstract

Polymer-modified mortars are cementitious materials that integrate polymers from 5 to 20 wt%. Those materials are widely used for protecting and/or repairing concrete surfaces among which building façades, civil engineering structures or sewage networks. Indeed, different studies demonstrated the participation of polymer in the improvement of the functional properties of such materials (e.g., an increase in both mechanical resistance and adhesion, a decrease in porosity and permeability). However, the existing studies do not take into account the interaction between polymer-modified mortars and microorganisms. Nevertheless, such interaction can lead to undesired aesthetical or structural modifications of those materials. As a result, the main objective of this paper is to evaluate the bioreceptivity of polymer-modified mortars in environments that are representative of the on-site conditions. Two formulations of polymer-modified mortars and a polymer-free mortar are characterised in the hardened state. Then, the resistance of those mortars to biocolonisation is tested by means of two laboratory accelerated essays. The first experiment is performed in order to recreate

biofouling at the surface of the specimens, while the second one exposed the materials to biodeterioration. Both tests allow then establishing recommendations for the use of polymer-modified mortars as repair and/or protecting materials.

Mots-clés : composites mortier-polymère ; bioréceptivité ; biodétérioration ; réseau d'assainissement ; bioaltération ; micro-algues.

Keywords: polymer-modified mortars; bioreceptivity; biodeterioration; sewer network; biofouling; micro-algae.

1. Introduction

Repairing and protecting cement-base surfaces is becoming essential in order to prevent the appearance of early disorders and to extend the service life of infrastructure. In this context, polymer-modified mortars are widely used as repair and protective materials for concrete surfaces in a large variety of environments such as building façades, civil engineering structure or sewage networks. To do so, polymer-modified mortars are applied into thin layers (about 1 – 3 cm) directly on the structure either in a localised or generalised way, depending on the required application. The properties of those innovative materials have been widely studied over the past few years. The great improvements obtained by the addition of polymers when it comes to formulate mortars were highlighted. Indeed, previous laboratory studies showed that a polymer/cement co-matrix is formed within the microstructure of the material, allowing increasing the adhesion while decreasing both permeability and porosity [1]–[4]. Besides, the durability of polymer-modified mortars was also studied in laboratory through durability indicators (i.e. permeability, carbonation, chloride ion migration, freeze-thaw cycles, etc.). Results have stressed that the presence of polymer makes it harder for aggressive agents to penetrate within the mortar. On a practical level, this evolves the increase in the resistance to both carbonation and chloride ion diffusion [1].

However, polymer-modified mortars present early modifications of their properties, resulting in their surface coloration, delamination, or swelling. The early deterioration of polymer-modified mortars may have its origins on the differences that exist between the actual on-site conditions during their application and the environment in which laboratory tests are performed. Firstly, their application into thin layers is not taken into account on the laboratory field, and the experiments are generally carried out on massive pieces (4x4x16 cm³ for most tests). In addition, during their service life, polymer-modified mortars are exposed to a wide range of microorganisms. This interaction can result in the modification of the surface and functional properties of the material. In this respect, two phenomena can take place. First, from an aesthetical point of view, the colour of the material can be modified due to the presence of microalgae established on its surface. This phenomenon is called biofouling, and is mainly encountered on building façades. Secondly, the properties of the protective material and its integrity can be affected by the presence of microorganism, resulting in its biodeterioration [5], [6]. The latter phenomenon may be encountered especially in aggressive media among which sewage networks. Yet, so far, biofouling and biodeterioration have only been studied on classic cement-based materials, allowing the identification of the main properties favouring biocolonisation (which defines the bioreceptivity of a material) [7], [8].

Hence, in this study, the proposed area of study is the resistance of polymer-modified mortars to biocolonisation when they are applied into thin layers. With this in mind, two experiments were carried

out: a capillary ascent test and a biodeterioration test. By performing both essays, recommendations will be made regarding the use of polymer-modified mortars in a wide range of applications from façades coating to sewage network repair.

2. Materials and methods

2.1. Materials and sample preparation

All mortars were prepared with ordinary Portland cement (CEM I 52.5 N) and siliceous sand, with a particle size of 0 – 2 mm. The granular skeleton of all mortars was fixed, with a volume fraction of sand of 0.5. Two formulations of polymer-modified mortars were considered in this study and compared with an original Portland mortar (identified as PF in the rest of this document).

Two polymers were used: a styrene-acrylate copolymer and an acrylic polymer. Both of them were in the form of latexes, which means that polymer particles were initially dispersed in an aqueous phase. Polymers characteristics are detailed in Table 1. Both polymers have a negative glass transition temperature, allowing them to be in a rubbery state at room temperature. The minimum film forming temperature (MFT) was determined in both cases by allowing the latexes to dry at a given temperature. The results obtained confirmed that the two selected polymers were able to form a homogeneous, continuous and transparent polymer film at different temperatures. Indeed, results showed that the acrylic polymer was the one with the lowest MFT.

Table 1 – Polymer characteristics

Nature	Solid content (wt%)	Average diameter (nm)	Tg (°C)	MFT (°C)
Styrene-acrylate copolymer	50 ± 2	220	-6.2 ± 0.7	20 – 25
Acrylic polymer	49 ± 1	255	-1.8 ± 0.2	13 – 20

Thus, two polymer-modified mortars were formulated with each previously mentioned polymer. In what follows, both polymer-modified mortars are referred as SA and A corresponding to the formulation with the styrene-acrylate copolymer and the acrylic polymer respectively. Both of them had a polymer-to-cement ratio of 10 wt%. A third mortar was formulated, without polymers, referred as the polymer-free mortar (PF). All mortars were formulated with the same initial amount of water, maintaining the water-to-cement+polymer ratio at 0.4. Consequently, the water-to-cement ratio (W/C) varies in the presence of polymer: the polymer-free mortar has a W/C ratio of 0.4 while both polymer-modified mortars have a W/C ratio of 0.44.

Once formulated, all mortars were poured into rectangular moulds (15x60x2 cm³) and vibrated to evacuate the air from the mortars. All formulations were isolated from the exterior for 24 hours and before being demoulded. Curing conditions were adapted in order to favour both cement hydration and polymer film formation [9], [10]. Therefore, the polymer-free mortar (PF) was maintained under water for 27 days whereas both polymer-modified mortars were conserved under water for 4 days, then put into sealed bags and conserved at room temperature for 23 days.

All mortars aged 28 days were cut either into cubic samples (2x2x2 cm³), either into rectangular samples (10x5x2 cm³) depending on the following tests.

2.2. Hardened mortars characterisation

2.2.1. Characterisation of the surface state of mortars

A roughness test was carried out in order to evaluate the impact of each polymer on their surface state. This test was performed on the rectangular samples using a contactless optical 3D surface measurement system based on Focus-Variation. A mapping was performed on three consecutive zones (10x10 mm²), defined on each samples. The surface parameter considered was the mean arithmetical height (Sa). Measurements were completed on an Alicona Infite Focus.

2.2.2. Characterisation of physical properties

Capillary absorption of the mortars was determined according to the NF EN 13057 standard. The rectangular samples were placed into a tank and their basis (5x2 cm²) was immersed over a height of 2 ± 1 mm, the highest dimension (10 cm) being in vertical position. The specimens were weighted at 0, 12, 30, 60, 120, 240 and 1,440 minutes. Water absorption per unit of area is computed and plotted against the square root of the immersion time. If the gradient obtained with those curves is linear, it is defined as the sorption coefficient.

The previous essays were performed on three specimens of each formulation. The porosity, bulk density and water absorption of each formulation is the average of the three specimens tested.

Porosity and bulk density of each mortar were determined based on the NF P18-459 standard. The rectangular samples were placed into a tank where the vacuum is made until the pressure reaches approximately 5 mbar. The samples are maintained in such conditions for 4 ± 0.5 hours. Then, they are saturated with water under vacuum for 44 ± 1 hours. A hydrostatic weighing (m_1), followed by a weighing in air (m_2) are carried out. The mortars are then dried in an oven at $105 \pm 5^\circ\text{C}$ until stabilisation of the mass of the mortars (m_3). The porosity and bulk density of the mortars are determined according to the equations (1) and (2) respectively.

$$\Phi_w = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \quad (1)$$

$$\rho_{app} = \frac{m_3}{m_2 - m_1} \times \rho_w \quad (2)$$

where Φ_w is the porosity, ρ_{app} is the apparent bulk density and $\rho_w = 1,000 \text{ kg/m}^3$ is the water density.

2.3. Accelerated biocolonisation devices

Biocolonisation of the mortars was studied through two phenomena commonly encountered by repair and/or protecting applications. The first one affects building façades. It is the result of the interaction between the materials and micro-algae, resulting in the biofouling of the mortars. The second one occurs in sewage networks. It is due to the presence of hydrogen sulphide and a consortium of bacteria, resulting in a modification of the inner properties of the materials.

2.3.1. Biofouling set-up

The biofouling of mortars by micro-algae was performed on three rectangular samples (10x5x2 cm³) of each formulation, dampened by capillary ascent. This method was used in previous studies [11]–[13], by placing the highest dimension of the specimens horizontally on a vermiculite layer, placed into

a hermetic chamber. On the contrary, in this study, the test was adapted it in order to cope with the on-site reality. As a result, three mortars of each formulation were placed so that the highest dimension of the samples is in vertical position (Figure 1). Moreover, the vermiculite layer was moistened with 4 times its weight in culture medium (modified B_{3N} medium, with a vitamin solution taken from the Jaworski medium). Specimens were previously inoculated by dropping 2 ml of a suspension of *Klebsormidium flaccidum* directly on the surface of the mortars, and letting it dry in air. The inoculated samples were finally placed into the chamber and maintained at 25°C, and 90 % of relative humidity. A day/night cycle was programmed with a photoperiod of 12 h/12 h, for 12 weeks. Biofouling was estimated overtime by visual observation, coupled with colorimetry measurements on the surface of the specimens. The colour monitoring was performed using a sphere spectrophotometer X-Rite Ci4200. The lighting applied at the surface of the samples was uniform and corresponded to daylight. Three parameters were selected in the CIELab space, corresponding to the chromaticity a* (traducing the evolution from green to red) and b* (traducing the evolution from blue to yellow), and the luminosity L*. For each face of a specimen, 18 measuring points of 8 mm diameter were performed. The final values for a formulation were the average of all measuring points of each of the three specimens.

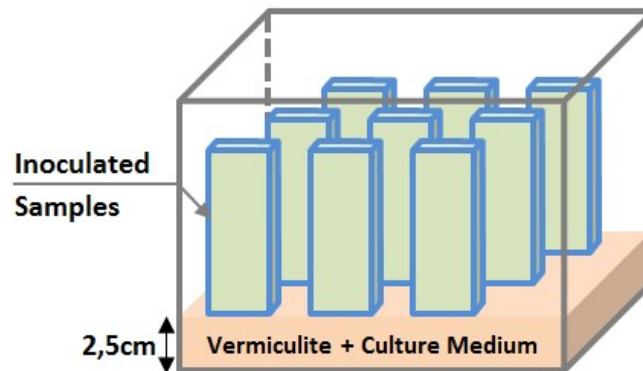


Figure 1 – Biofouling device

2.3.2. Biodeterioration set-up

The biodeterioration test was performed on five cubic specimens (2x2x2 cm³) of each formulation. The acceleration laboratory test used in this work was developed in previous studies in order to recreate the conditions encountered in sewage networks [14], [15]. According to the testing protocol, all mortars were submitted to an abiotic pre-treatment with hydrogen sulphide for 2 weeks in order to reach a suitable surface pH that allows microorganisms development. During the pre-treatment, the concentration of H₂S was maintained at 100 ppm, the temperature and relative humidity were fixed at 23°C, 100 % respectively. Then, activated sludge, containing high microbial diversity from water treatment plants, was spread on the surface of the mortars by means of a brush. Inoculated mortars were finally placed in the biodeterioration cell, and maintained at a concentration of H₂S of 30 ppm, 23°C and 100 % relative humidity for 16 weeks (Figure 2).

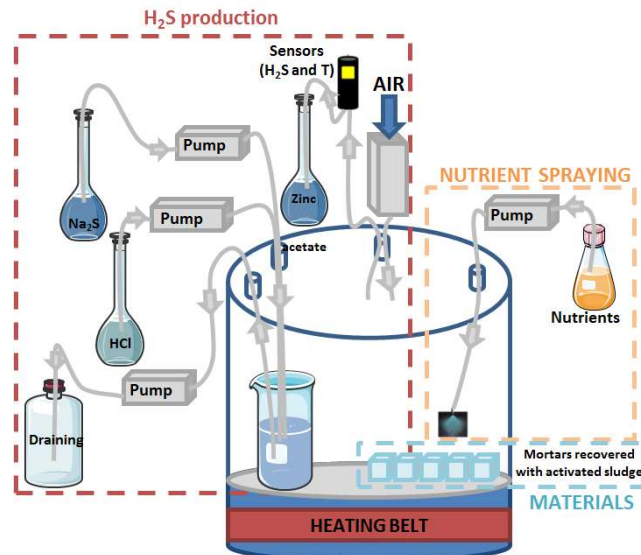


Figure 2 – Biodeterioration cell

Biodeterioration was first evaluated for all mortars by means of visual observations, coupled with surface pH measurements and dimensions measurement performed by means of pH paper and a calliper. Then, a specimen of each formulation was resinated and cut in profile. The samples were then scanned to be observed and the deteriorated surface was estimated with image processing software. Finally, three samples of each formulation were sonicated in order to remove the non-cohesive layer. To do so, all mortars were submitted to an ultrasonic treatment applied for 1 hour at 20 KHz. The global weight loss, resulting from the comparison between the mass of the specimens before sonication and after the ultrasonic treatment, was estimated in order to evaluate the biodeterioration of the mortars. For all quantitative tests, the final values were the average of all of the specimens tested.

3. Results

3.1. Characteristics of hardened mortars

3.1.1. Surface state

The surface state of the hardened mortars was first evaluated qualitatively by visual observation. It was noted that the surface of all mortars in contact with the mould presented surface bubbling. The extent of the phenomenon varied from one formulation to another (Figure 3). Indeed, the presence of bubbles is generalised to the entire surface in the case of the polymer-modified mortar SA. On the contrary, the phenomenon is localised for the formulation A. The reference mortar PF is somewhere in between. Besides, the morphology of the bubbles observed was also different. As a result, this phenomenon was quantified using image processing software.

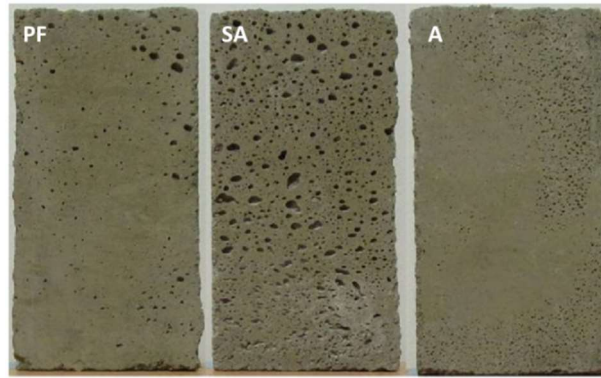


Figure 3 – Visual observation of the surface of all mortars

The diameter of the bubbles and the percentage of the total surface affected by the phenomenon were estimated (see Table 2). It can be seen from the results that the polymer-modified mortar SA is the most affected by the phenomenon, with approximately 9 % of its surface affected by the presence of bubbles. In addition, this formulation presents bubbles with the largest diameter (up to 5.0 mm). As observed from a visual point of view, the polymer-modified mortar A is the least affected (about 3 % of the total surface), with bubbles of small diameter.

Table 2 – Characteristics of the surface bubbling

	PF	SA	A
Diameter of the bubbles (mm)	0.3 – 3.5	0.5 – 5.0	0.1 – 1.0
Percentage of the total surface affected (%)	7.3 ± 1.1	8.6 ± 2.2	3.2 ± 1.0

In addition, the roughness of the mortars was studied by evaluating the parameters Sa and Sq. The results presented on the Figure 4 show that the polymer-modified mortar SA is the roughest. Indeed, for that formulation, both parameters are twice the values obtained for the polymer-free formulation. Regarding the polymer-modified mortar A, it appears to be as rough as the polymer-free mortar (PF) when taking into account the standard deviation.

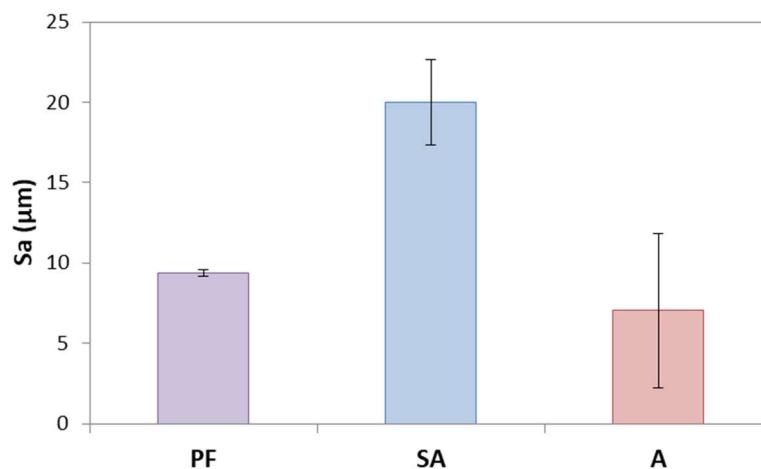


Figure 4 – Sa Roughness parameter of the set of mortars evaluated by optical 3D surface measurement

3.1.2. Physical properties

Water absorption was determined for all formulations of mortars (Figure 5). Results show that the polymer-free mortar (PF) has higher water absorption in comparison with the two polymer-modified mortars. In addition, the polymer-modified mortars behave in the same way. In all cases represented on the graph below, the gradient is linear. It is then possible to determine in each case the sorption coefficient (see Table 3). It can be noticed that the sorption coefficient obtained for the polymer-modified mortars is half that of the PF formulation.

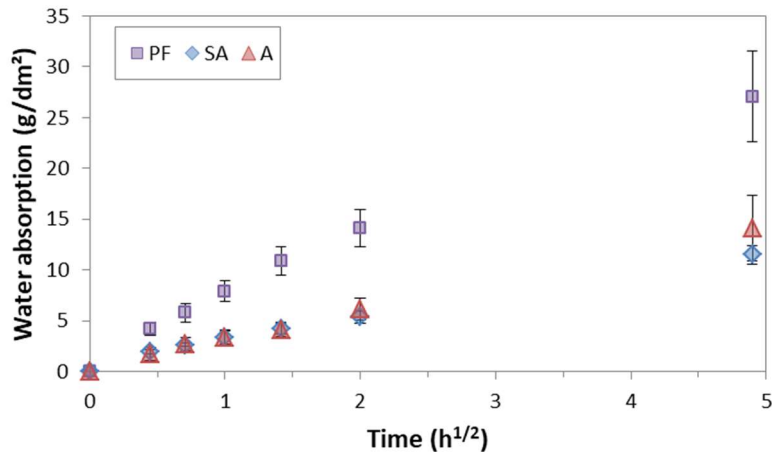


Figure 5 – Water absorption of mortars evaluated by capillary absorption

The porosity and bulk density of each formulation is presented in Table 3. Results show that the presence of polymer leads to a decrease in both porosity and bulk density of mortars. Besides, for both polymer-modified mortars, the decrease in porosity and bulk density is in the same extent.

Table 3 – Sorption coefficients, porosity and bulk density of mortars

	PF	SA	A
Sorption coefficient (g/dm².h^{1/2})	5.03 ± 0.91	2.13 ± 0.15	2.77 ± 0.71
Porosity (%)	18.0 ± 0.6	15.0 ± 0.6	14.0 ± 0.2
Bulk density (g/cm³)	2.100 ± 0.004	1.860 ± 0.012	1.810 ± 0.008

3.2. Biofouling of polymer-modified mortars

3.2.1. Qualitative aspects

Biofouling of polymer-mortars were first evaluated from a qualitative point of view through visual observation (see Figure 6). When samples are compared before the start of the experiment and after 12 weeks of conservation in the biofouling device, there are few differences. After 12 weeks, a slight green colouration can be observed at the surface of all mortars, but none of them have really been colonised by the micro-algae. Accordingly, the visual observation does not allow discriminating the formulations.

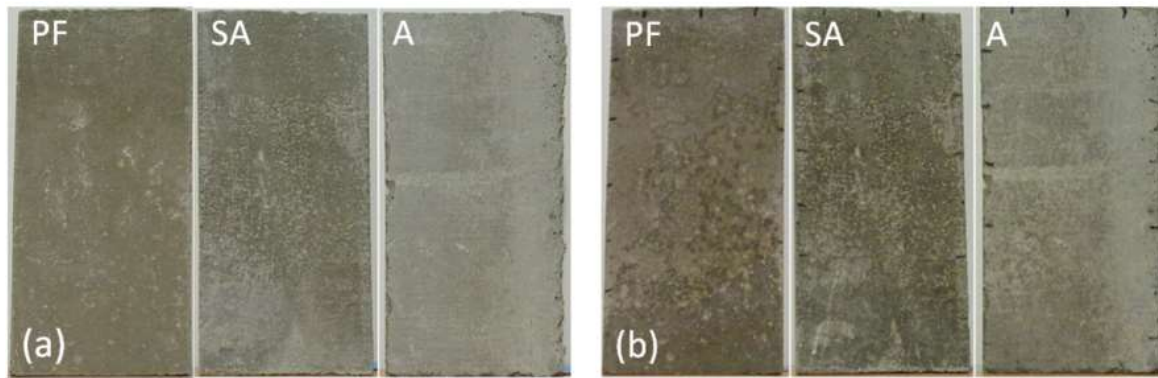


Figure 6 – Visual observation of the mortars (a) before the biofouling test and (b) after 12 weeks in the biofouling device

3.2.2. Quantitative aspects

The slight changes in colour appearing at the surface of the samples were quantified overtime in the CIElab space by means of colorimetry. Three parameters were extracted: the chromaticity a^* and b^* , and the luminosity L^* (Figure 7). The results show that chromaticity parameters a^* and b^* slightly increase overtime for all formulations. Besides, the luminosity tends to decrease with time independently of with formulation is considered.

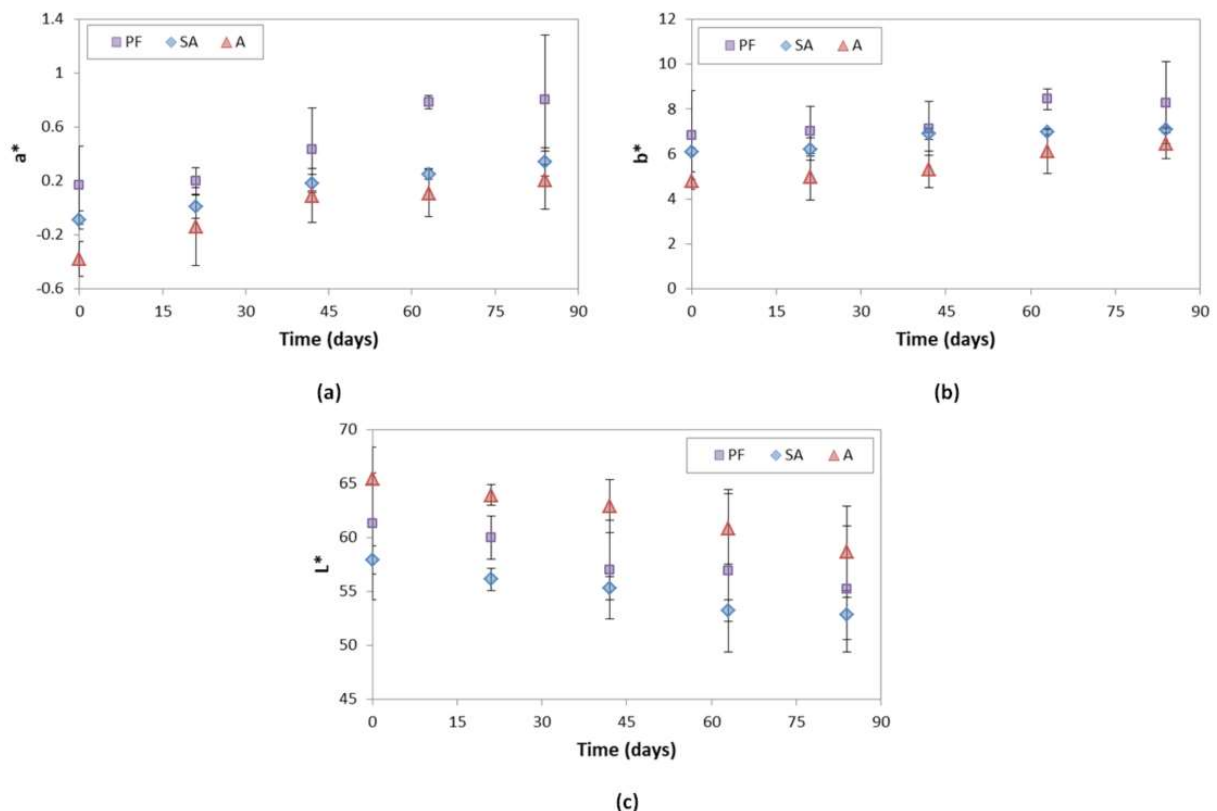


Figure 7 – Evolution of the colour of mortars overtime through different parameter, in the CIElab space: (a) chromaticity a^* ; (b) chromaticity b^* ; (c) luminosity










3.3. Biodeterioration of polymer-modified mortars

3.3.1. Qualitative aspects

The biodeterioration evolution of the samples was estimated qualitatively by their visual aspect and the estimation of their surface pH using pH paper (Table 4). It can be noticed that after 2 weeks of

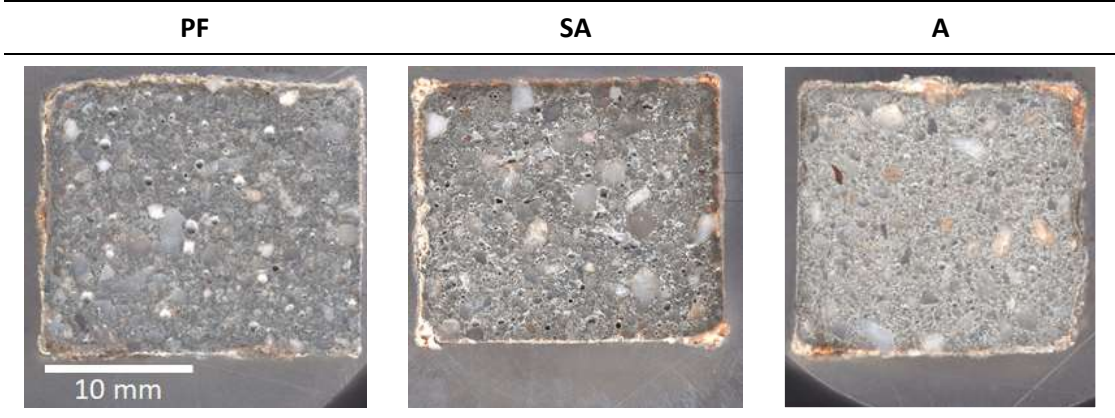
abiotic pre-treatment, all mortars blacken and their surface pH reaches 7. After 16 weeks of exposure to the hydrogen sulphide in the presence of activated sludge, the deterioration becomes significant for all mortars. Streaks can be observed on the surface of the samples, and deterioration products can be distinguished by their ochre colour. The surface pH measurements confirm that all mortars still have identical evolution overtime, reaching a pH around 5 after 16 weeks spent in the biodeterioration chamber.

Table 4 – Visual evolution of the samples over the experiment and pH range associated

	Initial state	After pre-treatment	After 16 weeks in the cell
PF	 pH = 9 – 10	 pH = 6.8 – 8	 pH = 5 – 5.3
SA	 pH = 9 – 10	 pH = 6.8 – 7.1	 pH = 4.7 – 5
A	 pH = 9 – 10	 pH = 6.8 – 7.1	 pH = 5 – 5.3

The observation of the specimen in profile allows distinguishing the deterioration depths, which appear on the rim the samples with ochre colour (see Table 5). Regarding these observations, it may be noted that the polymer-modified mortar SA seems to be the most deteriorated, while the one formulated with the polymer A appears to be as affected as the polymer-free formulation (PF).

Table 5 – Profile of the samples after 16 weeks in the biodeterioration cell



3.3.2. Quantitative aspects

It can be noticed from the previous photographs that the deterioration fronts are not homogenous over the samples height. This front was estimated for each profile, as shown on Figure 8. The results confirm that the polymer-modified mortar SA is the most deteriorated. Meanwhile, the one with the polymer A is the most resistant to biodeterioration. It is worth noting that after 16 weeks of exposure to hydrogen sulphide in the presence of micro-organisms, swelling was observed on the samples. The measurements of the dimensions of the specimen showed that all mortars dimensions increased by 0.5 – 0.7 mm.

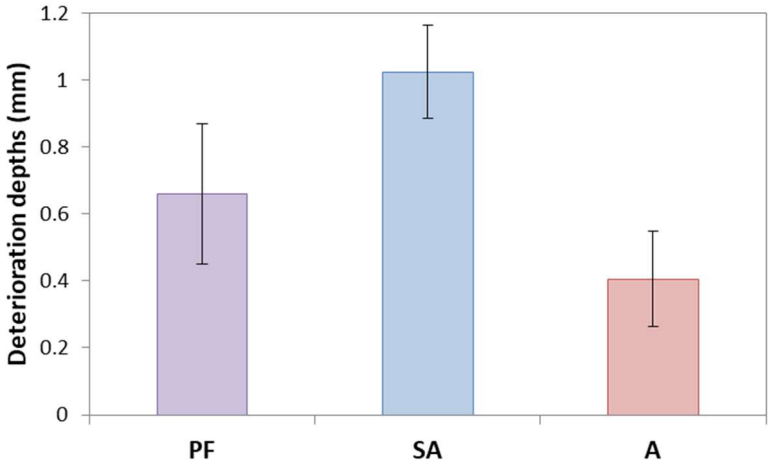


Figure 8 – Estimation of the mean deterioration depths by image processing

The global weight losses obtained after sonication show comparable values for all mortars (see Figure 9). Indeed, weight loss for all specimens is around 0.6 to 0.8 g, which is not significant considering the standard deviations. Besides, according to the standard deviations, differences between the three formulations are not significant.

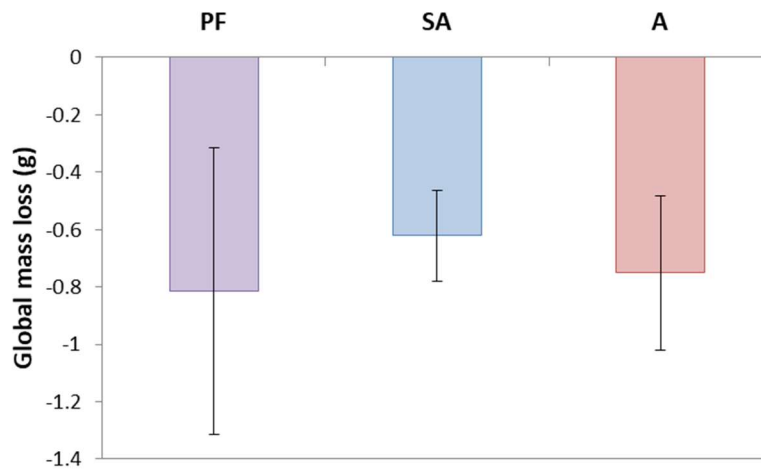








Figure 9 – Global mass loss after sonication treatment

When the specimens are observed before and after the ultrasonic treatment, a difference in the efficiency of the treatment seems to exist between the polymer-free mortar and the two polymer-modified mortars (Table 6). It appears indeed that, in the presence of polymer, the global material is more cohesive. As a result, the non-cohesive layer may be less important than in the case of an ordinary Portland mortar.

Table 6 – Visual observation of mortars before and after sonication

	PF	SA	A
Before sonication			
After sonication			

4. Discussion

Characterisation campaign highlighted that polymer-modified mortars have higher roughness than polymer-free mortars. This property may lead to an increase in the potential anchor sites for microorganisms. At the same time, the presence of polymer leads to a decrease in porosity, bulk density and capillary absorption of the mortars. This result was already stressed in previous studies [16]–[18]. As a result, an improvement on the resistance of polymer-modified mortars to the penetration of aggressive agents may be expected. However, since both phenomena influence the bioreceptivity of a mortar [19]–[21], it is not possible to predict the behaviour of a polymer-modified

mortar considering only those results. This preliminary results support the necessity to perform a test that place the material in direct interaction with microorganisms.

Bicolonisation was first evaluated considering the case of a building façade by performing a biofouling test. After 12 weeks of testing, no sample was colonised, even if different surface states were obtained for each formulation. This result was mainly attributed to the surface pH of the specimens, which remained around 9 after 12 weeks of testing for all mortars regardless of the formulation. Thus, it appears that an abiotic pre-treatment is essential in order to reach a surface pH that is suitable for the growing of microalgae. However the high surface pH may not be the only reason for which microalgae were not able to colonise the specimens since it was stressed in previous studies that *Klebsormidium flaccidum* is able to grow on surfaces whose pH is around 9.5 [22], [23]. On this basis, it was also observed that the hermetic chamber was contaminated by moulds. From a quantitative perspective, the monitoring of the surface colour of all mortars showed that the chromaticity tended to increase for all mortars while their luminosity tended to decrease. Nevertheless, it is worth noting that the variations obtained remained small in comparison with the results found in other studies [24], [25]. As a consequence, the evolution of colorimetric parameters shall be regarded as insignificant, which is in accordance with the fact that no biofouling was shown on these samples.

Biodeterioration test showed that the exposure of mortars to hydrogen sulphide in abiotic conditions led to a blackening of the surfaces of the specimens and a decrease in the surface pH around 7. It was highlighted that the polymer-modified mortars tested behaved in the same way as the polymer-free mortar. However, the blackening phenomenon appeared to be more pronounced in the case of the ordinary Portland mortar. This result is in accordance with the observations made during the characterisation campaign, considering that the hydrogen sulphide is an aggressive agent. When a consortium of microorganisms representative of the population present in a water treatment plant was applied at the surface of the mortars, all mortars were deteriorated after 16 weeks. Quantitative measurements showed that the extent of the biodeterioration depended on the considered formulation. There are no conclusive evidences that polymer-modified mortars are generally more resistant to an interaction with microorganisms. Indeed, the formulations with the polymer SA was the most degraded while the one incorporating the A polymer seemed to be the least affected in such conditions. Regarding the properties of the hardened mortars, previously determined, it seems that the balance between surface state and physical properties is responsible for the behaviour of the mortars in such severe conditions. It is indeed interesting to note that the most degraded formulation is also the roughest one. Thereupon, after these experiments, it was not possible to state whether porosity, roughness or capillary absorption was the most decisive parameter regarding bicolonisation. Finally, it appears after the ultrasonic treatment that polymer-modified mortars may have an improved inner cohesion. The microstructure of the materials was not studied but it may play an important role when it comes to interactions between the material and microorganisms. In particular, a highly cohesive material may be of great interest since even if the material is degraded, it may guarantee its structural handling.

5. Conclusions and perspectives

This study showed that the nature of the polymer influences the capacity of a polymer-modified mortar to resist bicolonisation. This parameter should then be taken into account when formulating and applying polymer-modified mortars as repair and/or protective material on façades, civil engineering structure or sewage networks. This study also confirmed the role the roughness, porosity and capillary

absorption on microorganism settlement and biodeterioration. Further experiments shall be performed in order to determine which one of these factors is the most decisive regarding the establishment and growth of microorganisms. In addition, this study suggests that the presence of polymer may improve the inner cohesion of the global material. This observation is of primary importance because it may assure that even if a polymer-modified mortar is more bioreceptive compared to an ordinary Portland mortar, it will conserve its integrity. Finally, concerning the biofouling test performed on mortars, it may be improved by adding a pre-treatment stage allowing reaching a suitable surface pH for the growth of microorganisms.

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