IV.10

Compatibility of Repair Mortars with 19th Century Natural Cement Cast Stone from the French Rhône-Alpes Region

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Abstract In the French Alps, near Grenoble, in the middle of the 19th century, natural cements were massively used to produce “cast stone” (concrete block), to simulate natural yellowish to reddish cut stone. In a first project, several ancient concrete buildings were studied and a major decay mechanism was identified: erosion, leading to the loss of the original fake stone appearance. Today, due to a lack of appropriate repair materials, grey Portland-cement-based mortars, combined with paintings are used, leading to a complete loss of the original aspect. Therefore, the aim of this second study was to develop and to test compatible repair materials to conserve the cultural heritage of this region. Based on the results of the first project, specifications concerning the composition and main properties of compatible repair materials were established. Then 4 mortars were selected, 2 of them being specifically formulated. In a second step the intrinsic properties of those mortars were characterised and finally, their mortar/concrete compatibility was assessed.

1 Introduction

The oldest concretes encountered in France date back to the middle of the 19th century and were made with natural cements. These cements were produced in the Rhône-Alpes region and notably used to cast concrete blocks or quite complex
ornaments, which were intended to imitate the colour and the texture of natural stone. One of these is an ochre colour, varying from light brown to red.

In a previous project [1, 2], a preliminary survey revealed that this cultural heritage was, on the whole, quite well-preserved. However, an erosion phenomenon was affecting the majority of the surfaces leading to a gradual disappearance of the concrete skin, which is detrimental to the initial “natural stone aspect.” The current rehabilitation techniques consist of the use of gray Portland cement-based mortars combined with a yellow or brownish paint finish. In fact, as the colour and composition of these concretes are very specific, there is a lack of suitable repairing mortars. Therefore, based on the analysis of the composition and properties of several historic concretes in this region, the aim of this study was to formulate and test natural cement-based repair mortars to restore eroded surfaces and to compare their performances to that of the Portland cement-based mortar currently used.

2 Protocol

The protocol of the study was divided into three steps. The first step consisted of the selection of four repairing mortars, two of which were specifically formulated from the specifications established in the first project, based on the analysis of historic concrete.

In a second step, the mortars were characterised in terms of transfer, physical and mechanical properties, microstructure, and performance. The intrinsic properties of the four selected mortars were characterized in terms of shrinkage, water porosity, water vapour permeability, capillary suction, dynamic modulus of elasticity, bending, and compressive strength measurements. The microstructure was characterised by optical microscopy (on polished section) and scanning electron microscopy. The performance evaluation was conducted with visual analysis observations.

The third step of the project was dedicated to the evaluation of the compatibility of the selected mortars with historic concrete and to the assessment of the durability of the repair mortar/concrete system. Therefore, natural cement-based slabs were cast using a 19th century concrete formula and were artificially eroded. After applying the four mortars to the slabs, visual observations and pull-out tests were carried out before (A) and after three sorts of artificial aging (B, C, and D): 10 heating and stormy shower cycles (B), 10 freeze-thaw cycles (C), 10 heating and stormy shower cycles followed by 10 freeze-thaw cycles (D).
3 Requirements and mortars selection

The requirements for mortar selection took into account the main characteristics of the historic concrete, the repair type (fine mortar for erosion) and the monument type [3]. The concrete monuments to be repaired are among the first buildings made of concrete in France, with a specific architecture and in cultural heritage context. As a consequence, criteria such as the preservation of the historic support and the need to use repair material with a colour and a texture close to those of the historic concrete had to be considered.

As quite high alkali contents were measured in the historic concretes to be restored, the use of alkali reactive (even potentially reactive) aggregates had to be avoided. The aggregate size also had to be adapted to the quite small thickness of eroded concrete to be repaired. As a consequence of the high sulphate contents observed in binder of the historic concrete to be restored, the cement to be used had to show a good sulphate resistance in order to be compatible. To assess the durability of the restoration and to avoid further decay of the historic concrete, the properties of the repair mortars had to be adapted to those of the historic support, in terms of transfer properties (water vapour permeability higher than that of the support) or mechanical performances (modulus of elasticity comparable to that of the support). However, the mortars also had to present a good durability and be able to resist the main stresses that repair mortars usually face (low shrinkage, high tensile strength) [4-7]. Finally, to fit with the aesthetic requirements, cements had to be used that exhibited an ochre colour, either combined with mineral pigments or not.

Based on these requirements, two mortars were specifically formulated and two others were selected among repair mortars available on the market (Table 1). It is to be noted that in the Alps region of France, there is a natural cement (so-called Prompt cement) that is still produced using the 19th century industrial process and whose composition is very close to that of the cements encountered in preliminary characterization of the historic concrete. Therefore, this Prompt cement was used in the composition of the two specific formulations and in one of the ready-to-use mortars. Furthermore, this cement has a good sulphate resistance. The fourth mortar selected was a Portland cement-based product containing fibres, which is currently used for rehabilitation operations.

<table>
<thead>
<tr>
<th>Table 1 Repairing mortars selected</th>
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<td>Mortar</td>
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<td>1</td>
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<td>4</td>
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4 Mortars characterisation

4.1 Transfer, mechanical and physical properties

The evaluation of the intrinsic properties of the four selected mortars was presented in a previous paper [8]. The main conclusions are the following:

- concerning the transfer properties, the results of porosity measurements were quite scattered, mortar 4 being less porous (less than 15%) and mortar 1 being excessively porous (more than 40%). Water vapour permeability was quite high for mortar 2 and, on the contrary, very low for mortar 4. Finally, the water capillary sorption tests showed that mortar 2 presented a capillary coefficient that was too high.
- regarding shrinkage after 1 year, mortar 1 exhibited the highest values (0.2%). The best results were obtained with mortars 2 and 3, in which shrinkage was quite low and stable with time. Surprisingly, mortar 4, which contains fibres meant to limit the shrinkage phenomenon, showed values higher than mortars 2 and 3.
- the results of bending and compressive strength indicated very low performances of mortar 2 (even though they were increasing with time). On the contrary, the Portland cement-based mortar (mortar 4) presented much higher bending and compressive strength than the three Prompt cement-based mortars. Finally, the dynamic moduli of elasticity were lower than 27 GPa in all mortars, which is the lowest value measured on the historic concretes. No incompatibility was therefore evidenced.

4.2 Microstructure

Optical microscopy observations performed on polished section after borax attack revealed differences in non-hydrated phases. In mortar 4, which is Portland cement-based, clinker grains presented well-crystallised alite and belite, with no clear separation between C4AF and C3A. In mortars 1, 2, and 3, anhydrous residual grains were poorly crystallised, with small alite and belite crystals and well-separated C4AF and C3A phases.

The hydrated phases observed by scanning electron microscopy also varied depending on the binder:

- mainly calcium silicates hydrates (CSH) and ettringite with fresh portlandite for Prompt cement-based mortar
- and mainly CSH, portlandite, and fresh ettringite for Portland cement-based mortar (mortar 4).
These observations indicated that the microstructure of the Prompt-cement based mortars was quite close to that of the historic concretes; on the other hand, mortar 4 had a clearly different microstructure. Sulphate resistance was undetermined.

4.3 Aestheticism

The colours of the four mortars can be seen in Fig. 1a. Mortars 1 and 3 have an adapted colour but mortar 1 presents pigments traces (Fig. 1b). Mortar 2 is too white, but it is easy to tint. Mortar 4, whose colour is too grey and difficult to tint, is clearly not suitable.

![Fig. 1 a) Colours of the mortars and b) Pigment trace on mortar1.](image)

4.4 Slabs manufacture

Twenty slabs (50 cm x 50 cm x 8 cm) were cast for the purpose of mortar/concrete compatibility characterisation. The slabs’ manufacture has been presented in a previous paper [8]. A formula extracted from documents dating back to the end of 19th century was used, using Prompt-cement as a binder. To reproduce a surface similar to the most commonly encountered erosion facies, deactivation products were pulverised on the 20 slabs surfaces just after their casting. After manufacture, the slabs were kept in a room at 20°C and 95% RH and dried in the open air for 28 days.

4.5 Mortars application

The four selected mortars were applied to the slabs (four slabs per mortar). In terms of workability, mortar 1 was very fluid, mortars 2 and 3 were easy to apply, and mortar 4 was sticking to the tools and therefore was quite hard to apply (Fig. 2).
After its application, mortar 1 showed immediate shrinkage cracks (Fig. 3a). After setting, white efflorescence appeared on mortar 4 (Fig. 3b).

After the application of mortars and before the artificial ageing, the slabs were kept for 28 days in a room at 20°C and 65% RH.

4.6 Artificial aging

The artificial aging cycles are presented in Fig. 4.
After artificial aging, visual analysis was conducted, and then the slabs were kept for 7 days in a room at 20°C and 65% RH before the pullout tests. For each type of artificial aging one slab per mortar was tested, and five pullout tests were performed per slab. The pullout tests were performed according to the French standard NF EN 1015-12, which consists of sampling a core through the entire thickness of the mortar and up to 3 mm in the concrete support with a core drill (5 cm inside diameter). Then, circular metal pellets (5 cm diameter) are glued to the mortar surface, and the pullout tests are performed using a 500 daN capacity dynamometer. The load is applied monotonically, increasing to the breaking point. For each test, the breaking load and the breaking site are noted. Finally, the adhesion strength is calculated as ratio between the breaking average load and the pellet surface (the result is expressed in MPa).

In Fig. 5 the adhesion strengths obtained for each mortar before and after the different artificial aging are presented. The breaking sites for each mortar before and after the different artificial aging are given in Table 2.

![Fig. 5 Adhesion values before and after artificial aging.](image-url)
Table 2 Breaking sites for each mortar before and after the different artificial aging.

<table>
<thead>
<tr>
<th>Mortar</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
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<tbody>
<tr>
<td>2</td>
<td>Interface breaking</td>
<td>Interface breaking</td>
<td>Mortar breaking</td>
<td>Mortar breaking</td>
</tr>
<tr>
<td>3</td>
<td>Interface breaking</td>
<td>Interface breaking</td>
<td>Support breaking 60%</td>
<td>Mortar breaking</td>
</tr>
<tr>
<td>4</td>
<td>Support breaking</td>
<td>Support breaking</td>
<td>Support breaking 60%</td>
<td>Support breaking 60%</td>
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</table>

Support breaking 60%    mortar breaking 40%

With a simple cure without artificial ageing (A), the adhesion strength was similar for mortars 2 (0.8 MPa) and 3 (0.6 MPa), while the adhesion strength for mortar 4 was much higher (2 MPa). After the freeze-thaw cycles (B), no important adhesion evolution was noticed. After the heating and stormy shower cycles (C), the adhesion values of mortars 2, 3, and 4 increased (respectively 1.2, 1.6 and 2.9 MPa). After the D cycles, the adhesion strengths for mortar 2 (from 1.2 to 1.3 MPa) were similar to the results obtained from the C cycles, although they decreased for mortar 3 (from 1.6 to 0.4 MPa) and increased for mortar 4 (from 2.9 to 3.7 MPa).

Fig. 6 illustrates interface and support breaking sites. With a simple cure (A) and after the freeze-thaw cycles (B), mortars 2 and 3 presented an interface breaking location, whereas mortar 4 presented a support breaking location. After the C cycles, only mortar 2 presented a mortar breaking location, and mortars 3 and 4 presented a break both in the support and in the mortar. After the D cycles, mortars 2 and 3 presented a mortar breaking location, whereas mortar 4 presented a support/mortar breaking location.

To summarise, mortar 4 was too adhesive (with breaking in the support), mortars 2 and 3 showed suitable adhesion properties (adhesion strength higher than the 0.4 MPa threshold of the initial specifications); mortar 1, which shrank immediately after its application to the slabs, could not be tested.

After the freeze-thaw cycles (B), no apparent damage was noted for mortars 2, 3, and 4. After the heating and stormy shower cycles (C), cracking was observed for mortars 3 (Fig. 7) and 4, and no damage was noticed for mortar 2. After the
heating and stormy shower cycles, followed by the freeze-thaw cycles (D),
 cracking was observed for mortars 3 and 4 and no damage was noticed for mortar
 2. Therefore, no additional damage was noted after the (D) cycles.
 Mortar 2 showed a good durability whatever the artificial aging, while mortars
 3 and 4 suffered degradations after the heating and stormy shower cycles (C),
 which may be linked to a deformability deficiency.

Fig. 7 Cracking observed on mortar 3 after heating and stormy cycles.

5 Conclusions

The aim of this study was to develop and test repair mortar for historic concrete
 of the 19th century encountered in the French Alps area, which is affected by
 serious erosion. After a first study focused on these historic concretes analysis [1,
 2], a list of specifications was established. Based on these specifications, four
 mortars were selected, their intrinsic properties were characterised, and the
 compatibility of the system mortars/concrete was tested.

Firstly, the intrinsic properties tests revealed that some mortars were unable to
 match the specifications. Actually, shrinkage was clearly too high in mortar 1.
 Mortar 4, which is Portland cement-based, was clearly too impermeable to water
 vapor. Its mechanical performances also were much higher than the three Prompt
cement-based mortars. Only mortars 2 and 3 (specially formulated) presented
 transfer and mechanical adaptations. In terms of microstructure, the use of
 Prompt-cement in the mortars leads to a microstructure close to those of the
 historic concrete. Only mortar 3 (on site mortar) presented an adapted color.

Secondly, the tests performed on slabs presented some incompatibilities.
 Mortars 1 and 4 were difficult to apply on the slabs simulating the historic
 concrete. The adhesion strength for mortar 4 was too high, with breaking sites in
 the support, which is incompatible with the problem of the support conservation.
For mortars 2 and 3, the adhesion strengths were sufficient, with breaking sites principally at the mortar/concrete interface or in the mortar.

Finally, in terms of resistance to the artificial aging, only mortar 2 (ready-to-use and specially formulated) showed no degradation for all the cycles types, while the heating and stormy cycles caused cracking in mortars 3 and 4.

To conclude, mortars 1 and 4 are incompatible for the repair of the eroded historic concrete of the 19th century of the French Alps area. Mortar 2 corresponds to all the specifications except the colour, but it can be easily tinted. Mortar 3, the “on site” mortar, presents only a deficiency of durability for the heating and stormy cycles. The final step of this study will consist of testing the two best mortars (mortars 2 and 3) on-site in cooperation with skilled operators and with a follow-up in time.

6 References