

**19TH CENTURY CAST STONE FROM THE FRENCH RHÔNE-ALPES  
REGION: MATERIALS CHARACTERISATION AND RESTORATION  
SOLUTIONS**

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**Abstract**

In the French Alps near Grenoble, in the middle of the 19th century, within the dynamics of development of the natural cements industry, a new building material was created: cast stones. Until the end of the 19th century, cast stones were massively produced and used as an alternative to cut stone, that they were perfectly simulating, due to their ochre colour. Today, due to a lack of appropriate repair materials, the restoration of the vast heritage built using these cast stones is not satisfactory, both in terms of compatibility and aesthetics. The purpose of this study was to identify the different uses of this peculiar material, to study its composition, properties and specific decays in order to find and develop compatible restoration solutions.

**Keywords:** Cast stone, natural cement, erosion, repair mortar

**1. Introduction**

Natural cements from Isere are among the first cements produced in France, after the discoveries of Louis Vicat in 1818 on hydraulic limes. Obtained after calcination and grinding of limestones naturally containing clay, natural cements from Isère were massively produced and exported worldwide from the middle of 19<sup>th</sup> century to the beginning of the 20th century (Hadley 1999; Cornier 1904). Depending on their composition and calcination conditions (temperature and duration), some of these cements, such as “Prompt cements”, were characterized by a quick setting and a clear ochre surface colour (Hadley 1999; Botton 1997; Cayol-Gerin 1994). At that time natural cements were used for numerous purposes, but their most characteristic application was so-called “artificial stone” obtained by casting concrete blocks imitating the colour and texture of stones. 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.” Moreover, few data are currently available on these cements, their microstructure, composition or their uses.

In the 19th century, the formulation and implementation of concrete were very empirical. Consequently, the properties of concrete from this period are generally quite distinct (low mechanical strength and high porosity) from those of more recent products, more efficient every day. Unfortunately, repair mortars, are generally formulated for modern concrete, and therefore, they are, in most cases, too rigid and impermeable to be compatible with these ancient materials. Consequently, it is now very difficult to restore these concrete, or cast stone, for physico-chemical, mechanical, but also aesthetic compatibility issues.

In this context, this study was aimed at finding restoration solutions, by testing and developing repair mortars compatible with the concrete used in the 19th century in the Rhône-Alpes region.

In a first step, buildings constructed using natural-cements-based-concrete in the Rhône-Alpes region were inventoried and their alterations were listed. Then samples were taken from a series of representative buildings, both in sound and decayed areas, in order to characterise the composition, the microstructure, the main physical and mechanical properties, and the alteration mechanisms of these natural-cement-based-cast-stones.

The second step was dedicated to develop and to test compatible repair materials. Based on the preliminary series of results on ancient concrete, specifications concerning the composition and the main properties of repair materials which could ensure both chemical, physical and mechanical compatibility with the ancient substrate were established. Three natural-cement and one Portland-cement based mortars were selected, two of them being specifically formulated. The appearance, the workability and the transfer, mechanical and physical properties of those mortars were characterised. The compatibility performances of the 4 chosen mortars were evaluated by applying them on natural-cement-based concrete slabs, specially formulated and artificially decayed. On those slabs, visual observations and pull-off tests were carried out before and after different artificial ageing.

## **2. Cast stone survey**

### **2.1 Inventory**

First an inventory was carried out by collecting data from archival sources, collaborating with historians and performing several surveys in the field. More than 60 buildings using natural cements were listed both in the city of Grenoble and in the Rhône-Alpes region. Most of them were located in Grenoble and situated in districts which were strongly developed in the 19th century because of both industrial and demographic expansions. The majority of those buildings corresponded to residential blocks but several religious buildings were also noted. Three main specific application domains of these cements were distinguished :

- Sculptures (Fig. 2.1), sometimes with very complex shapes. They were mainly located on building fronts of the city of Grenoble. Their design was generally very close to those met in the case of buildings made of freestones.
- Ornamental elements (Fig. 2.2) using geometric patterns repeated all over the building fronts. This application was very specific to the Rhône-Alpes region. In some cases, specific design of the concrete surface reproducing the surface aspect of other building materials such as brick or stone was noticed.
- Building structural components (Fig. 2.3): in this case, natural cements were used to produce concrete blocks which were then assembled as stone. This application was very specific to the Rhône-Alpes region too.

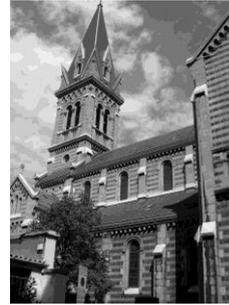
For numerous buildings and notably residential blocks, it was noted that they were mainly constituted of masonry and only some particular parts such as doors and windows frames, or sculptures were made using natural cements.



**Figure 2.1.** Example of sculpture made of natural cements and located on the fronts of a residential building.



**Figure 2.2.** Example of a geometric pattern repeated all over building fronts of the Saint-Bruno church.



**Figure 2.3.** Example of building made of natural cements: the Saint-Bruno church.

Archival sources indicated that pre-casting of concrete elements in factories and their assembling on the building site was the most developed technique for the manufacturing of sculptures or concrete blocks, but natural cements could also be directly applied to the building front preferentially for rendering.

From historical literature, several kinds of materials could be used for the mould such as concrete or steel depending on the number of specimens and the precision of the shape (Mettra 1997; Fontenay 1880). Historical documents also mentioned that as a function of the application (sculpture, pipe, ...), the cement firing temperature and duration could be increased and hydraulic limes could be added to the cement, specially with prompt natural cements which presented an extremely rapid hardening. The aim was to significantly increase the setting time of the cement from few minutes to several hours in order to facilitate its casting (Gobin 1889).

From these data, it is therefore probable that numerous kinds of cements with likely different compositions and properties were used in building construction. Nevertheless, knowledge about these cements (compositions, formulations, properties, ...) is today very poor and their identification is mainly based on their specific ochre.

## 2.2 Decays

The majority of the monuments visited exhibited a good state of preservation. As nowadays most of them are more than 100 years old it is therefore possible to conclude that these natural cements have shown a good durability. Nevertheless, two kinds of surface damages were mainly identified:

- An erosion phenomenon (Fig. 2.4) developed on the surface of the majority of the buildings and conducting to the progressive disappearance of the concrete skin and revealing the aggregates. Consequently, the original aspect of the concrete blocks freestone-imitating is progressively lost and a gradual degradation of the details of the sculptures is generally noticed.
- A spalling phenomenon (Fig. 2.5) observed only on few buildings and which could be associated with black crusts or white efflorescences.

In order to rehabilitate those buildings, gray Portland cement-based mortars, combined with a yellow or brownish painting as finishing, are generally used. In some

cases, painting is even directly applied on the ancient concrete, without preliminary application of a surfacing mortar.



**Figure 2.4.** Example of erosion phenomenon on a cast concrete block from a wall in Grenoble.



**Figure 2.5.** Spalling phenomenon associated with black crust on the Champollion high school in Grenoble.

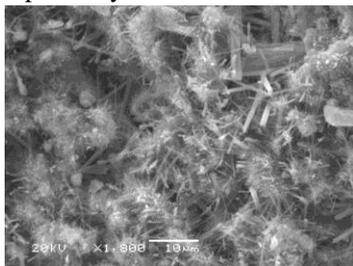
### 3. Materials characterisations

In order to characterise the microstructure, the weathering mechanisms and the main physical and mechanical properties of these ancient concretes, samples were taken from three representative buildings : Saint-Bruno church and Champollion high school in Grenoble and La Mure's church.

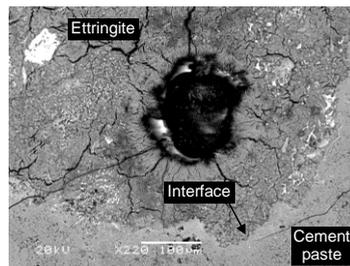
#### 3.1 Microstructure

Scanning electron microscopy (SEM) was performed on both fractures and polished cross-sections. For the cement identification, non-carbonated areas are necessary, which was quite difficult as phenolphthalein tests revealed important depths of carbonation (from 15 to 35mm). A more complete microstructural and chemical descriptions of the concrete samples are reported in previous papers (Cailleux 2005, 2006).

Concrete samples from Saint-Bruno church and and Champollion high school were characterised by numerous ettringite crystals (thin needles or fine fibres) mainly located in the cement paste of the concrete (Fig. 3.1). On cross-sections (Fig. 3.2), some large areas mainly composed of ettringite crystals were detected. Moreover, filaments of hydrated calcium silicate were noticed in the cement paste but neither portlandite or mono-sulphate crystals were observed.



**Figure 3.1.** Hydrated calcium silicate filaments and ettringite needles in the cement paste of the Saint-Bruno church.



**Figure 3.2.** Interface between a high ettringite content area and the cement paste (Saint-Bruno church).

Concerning the concrete samples from La Mure's church, less ettringite crystals were observed. Some of these ettringite crystals were constituted of fine fibres dispersed in the cement paste, but most of them were detected in the pores in association with massive portlandite. Several platelets which might correspond to mono-sulphate or calcium aluminium hydrates, but also some hydrated calcium silicate were also noticed in the paste.

The presence of large amount of ettringite and the absence of portlandite in the concrete of Saint Bruno church and Champollion high school indicated the use of a rapid setting cement. In contrast, the large quantity of portlandite and the small amount of ettringite in concrete of La Mure's church indicated a Portland cement based concrete. These observations fitted with the data from historical literature. In fact, it was indicated that Saint-Bruno Church was built using a natural cement classified as a prompt natural cement, characterised by rapid setting and hardening processes (Viallet 1900). For La Mure's church, historical data mention that a natural cement classified as a Portland cement was used. This cement was characterised by low setting and hardening rates of about 5 hours (Gobin 1889).

Two kinds of cements have been therefore identified for the concrete samples of Saint-Bruno church, Champollion high school and La Mure's Church. Finally, it appears from these results that if the natural cements could be characterised by an ochre colour, this feature does not seem to be exclusively characteristic of the Prompt natural cements.

### **3.2 Physical and mechanical properties**

The main physical and mechanical properties of Saint-Bruno and La Mure's churches were determined according to a testing protocol detailed in a previous paper (Cailleux 2006). Results of these tests are given in Table 3.1.

Carbonation depths ranging between 15 and 35mm were measured, which is relatively low for monuments built in the 19th century.

Density (2200 to 2300kg/m<sup>3</sup>) and porosity (15 to 17%) measured were indicative of an average compactness. Dynamic Young's Modulus evaluated on these ancient concrete were consistent with the latest and indicative of average mechanical properties.

These results indicate that despite a probable lack of mechanical vibration during the casting, a careful manufacturing of the concrete blocks resulted in a quite good quality of the concrete explaining the absence of important defect noted on the buildings examined.

Finally, sulphate (4 to 4.3%) and equivalent alkali (4.8 to 5.45 kg/m<sup>3</sup>) contents determined for these ancient concrete were slightly higher than those actually recommended for Portland cements (sulphate threshold: 3.5%, alkali threshold: 3.3kg/m<sup>3</sup>, both by weight of cement). These results are consistent with the numerous ettringite crystals observed in the cement pastes and the sodium sulphate crystals detected in the concrete of the La Mure's church.

**Table 3.1.** Main characteristics of the concrete sampled

Site	Carbonat ion depth (mm)	Density (kg/m <sup>3</sup> )	Water porosity (%)	E dynamic (GPa)	Cement content (kg/m <sup>3</sup> )	Sulphates content (%)	Equivalent alkali content (kg/m <sup>3</sup> )

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<b>Saint-Bruno</b>	15-20	2300	15	32	435	4.3	4.8
<b>La Mure</b>	35	2200	17	32	432	4	5.45

#### **4. Restoration solutions**

##### **4.1 Requirements and selection of repair mortars**

As the main decay phenomenon observed on these ancient buildings was erosion, the research was focused on repairing superficial loss of material. The requirements definition for suitable repair mortar selection took into account the main characteristics of the historic concrete, the repair type and the monument type (Daubresse 1998). The monuments to be repaired being among the first buildings made of concrete in France, with a specific architecture, in a cultural heritage context, criteria such as the preservation of the historic materials and the need to use repairing products with a colour and a texture close to those of the historic concrete, had to be considered.

Firstly, the characteristics of the historic concrete to be restored, conditioned the composition of the restoration mortars. Thus as the ancient concrete were containing quite high alkali contents, the use of alkali reactive (even potentially reactive) aggregates had to be avoided. As the ancient concretes also exhibited high sulphate contents, the cement to be used had to show a good sulphate resistance in order to be compatible.

Secondly, the aggregate size had to be adapted to the quite thin layers of eroded concrete to be repaired.

Thirdly, 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 substrate) or mechanical performances (modulus of elasticity comparable to that of the substrate). However, the mortars also had to present a good durability and to be able to resist to the main stresses that repair mortars usually have to face (low shrinkage, high tensile strength) (Thomasson 1982; Emmons 1994; Vaysburd 2000). Finally, to fit with the aesthetic requirements, the restoration mortars had to exhibited an ochre colour.

Based on these requirements, two mortars were specifically designed and two others were selected among repair mortars available on the market (Table 4.1). It is to be noted that in the Alps region of France, there is a natural cement (“Prompt<sup>®</sup> cement”) that is still produced using the 19th century industrial process and which composition is very close to that of the cements encountered in the preliminary characterisation of the historic concrete. Furthermore, this cement has a good sulphate resistance. Therefore, this Prompt<sup>®</sup> cement was used in the composition of the two specifically designed and in one of the ready-to-use mortars. The fourth mortar selected was a Portland cement-based product containing fibres, which is currently used for rehabilitation operations.

**Table 4.1** Repairing mortars selected

Mortar	Cement type	Mortar type	Comments
1	Prompt <sup>®</sup> cement	Ready-to-use	Available on the market
2	Prompt <sup>®</sup> cement	Ready-to-use	Specially formulated

3	Prompt <sup>®</sup> cement	“On site” mortar	Specially formulated, for skilled operator
4	Portland cement	Ready-to-use	Available on the market

## 4.2 Repair mortars characterisation

### 4.2.1 Moisture transports, physical and mechanical properties

The evaluation of the intrinsic properties of the four selected mortars was presented in a previous paper (Bouichou 2008). Results of these tests are given in Table 4.2. The main conclusions are the following:

- concerning the moisture transport properties, the results of porosity measurements were quite scattered, mortar 4 being the less porous (WP water porosity < 15%) and mortar 1 being excessively porous (more than 40%). Water vapour permeability (WVP) 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, for which shrinkage was quite low and stable with time. Surprisingly, mortar 4, which contains fibres meant to limit shrinkage, showed values higher than mortars 2 and 3.
- the results of bending (BS) and compressive (CS) 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<sup>®</sup> cement-based mortars. Finally, the dynamic modulus of elasticity were lower than 27 GPa for all mortars, which is the lowest value measured on the historic concretes.

**Table 4.2.** Intrinsic properties of mortars.

Mortar	WP(%)	Capillary C. (g/cm <sup>2</sup> min <sup>2</sup> )	WVP(g/m <sup>2</sup> . h.mmHg)	Shrinka ge (%)	BS (MPa)	CS (MPa)	E dyn (GPa)
1	41	20	0.53	0.20	4,3	28,5	21,4
2	38	1.35	0.72	0.07	2,3	6,9	11,1
3	27	3.94	0.36	0.08	4,9	36	24,5
4	14	0.10	0.08	0.13	7,7	45,2	23,3

### 4.2.2 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 (C<sub>3</sub>S) and belite (C<sub>2</sub>S), with no clear separation between C<sub>4</sub>AF and C<sub>3</sub>A. In mortars 1, 2, and 3, anhydrous residual grains were poorly crystallised, with small alite and belite crystals and well-separated C<sub>4</sub>AF and C<sub>3</sub>A phases. The hydrated phases observed by scanning electron microscopy also varied depending on the binder:

- mainly calcium silicates hydrates (CSH) and ettringite with primary portlandite for Prompt<sup>®</sup> cement-based mortars,

- and mainly CSH, portlandite, and primary ettringite with the presence of fibres for the Portland cement-based mortar (mortar 4).

The mortars 1, 2 and 3 presented a microstructure close to those of the historic concrete.

#### **4.2.3 Aesthetics**

Mortars 1 and 3 had a suitable colour but mortar 1 presented pigments stains. Mortar 2 was too white, but it would be easy to tint. On the contrary, Mortar 4, which colour was too grey would probably be difficult to tint, and was clearly not adapted.

### **4.3 Compatibility cast stone - repair mortars**

#### **4.3.1 Slabs manufacture and mortar application**

Twenty slabs (50 cm x 50 cm x 8 cm) were cast to test mortar/concrete compatibility. The slabs' manufacture has been presented in a previous paper (Bouichou 2008). A composition extracted from documents dating back to the end of 19th century was used, using Prompt<sup>®</sup> cement as a binder. To reproduce a surface similar to the most commonly encountered erosion facials, a deactivation product was pulverised on the slabs surfaces just after their casting. The slabs were then kept in a room at 20°C and 95% RH and dried in the open air for 28 days.

Afterwards, the four selected mortars were applied on the slabs (four slabs per mortar). In terms of workability, if mortars 2 and 3 were easy to apply, mortar 1 was too fluid, and mortar 4 was sticking to the tools and therefore both latest were quite hard to apply. After its application, mortar 1 showed immediate shrinkage cracks, when after setting, white efflorescence appeared on mortar 4.

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

#### **4.3.2 Artificial ageing and adhesion tests**

Visual observations and pull-off tests were carried out both after applying the four repairing mortars on the slabs, (A) and after three sorts of artificial ageing (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). The experimental protocol and the results of those test has been presented more precisely in a previous paper (Bouichou 2010). Results of these tests are given in Table 4.3. The main conclusions concerning the adhesion tests (fracture site and adhesion values) are the following:

- mortar 4 was too adhesive (rupture in the substrate), whatever the artificial ageing type,
- mortars 2 and 3 showed suitable adhesion properties ; with ruptures principally at the mortar/concrete interface or in the repairing mortar, whatever the ageing type.
- mortar 1, which shrank immediately after its application , could not be tested.

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

**Table 4.3.** Adhesion (Ad. MPa) values and fracture sites (FS) for each mortar before and after the different artificial ageing. Fracture location : IF = interface ; MF = Mortar ; SF = Support

Mortar	A		B		C		D	
	Ad. MPa	FL	Ad. MPa	FL	Ad. MPa	FL	Ad. MPa	FL
2	0.8	IF	0.6	IF	1.2	MF	1.3	MF
3	0.6	IF	0.6	IF	1.6	SF 60 % MF 40%	0.4	MF
4	2	SF	1.8	SF	2.9	SF 60 % MF 40%	3.7	SF 60% MF 40%

#### 4.4 Back to the specifications

In Table 4.4, the compatibility requirements given in chapter 3.1 are recapitulated, and a comment is given on the ability of the different mortars to fit or not with the specifications.

**Table 4.4.** Requirements and ability of mortars to met the specifications

Characteristics	Specifications	Mortars
Aggregates	sand non alkali-reactive	All mortars
Binder	sulphate resistant	All mortars except mortar 4
Shrinkage	Low, < 0.1% after 1 year	Mortars 2 and 3
Transfer and mechanical properties	Comparable to those of the ancient concrete	Mortars 2 and 3
Aesthetics	Comparable colour and texture	Mortar 3
Durability	Adhesion (>0.4MPa), but substrate preservation (Interface fracture)	Mortar 2

Concerning the type of aggregates, all the mortars contained non alkali-reactive sand. The binder of mortars 1, 2 and 3 was Prompt<sup>®</sup> cement, which is sulfate-resistant, when mortar 4 was Portland-cement-based, and therefore more sensitive to the sulphate attack.

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 was Portland cement-based, was clearly too impermeable to water vapour, and its mechanical performances were also much higher than that of the three Prompt<sup>®</sup> cement-

based mortars. Only mortars 2 and 3 (specially designed) presented moisture transfer and mechanical properties adapted.

In terms of microstructure, the use of Prompt<sup>®</sup> cement in 3 of the mortars leads to a microstructure close to those of the historic concrete.

Concerning the aspect, only mortar 3 (on site mortar) presented an adapted colour.

Finally, the tests performed on slabs evidenced some incompatibilities, mortar 4 being too strong for ancient concrete and mortar 1 being to shrinkage susceptible.

To conclude, Mortars 1 and 4 were clearly unfitted for repairing the eroded historic concrete of the 19th century of the French Alps area. Contrarily, Mortar 2 corresponded to all the specifications except the colour, but it could be easily tinted. Finally, Mortar 3, the “on site” mortar, that exhibited quite compatible properties, only presented a deficiency of durability after the artificial heating and stormy ageing cycles.

## 5. Conclusions

In a preliminary survey, more than 60 buildings using natural cements were listed in the French department of Isère, and the main application domains of these materials on the monuments were described, the most characteristic application being cast stone or concrete block. A series of visit 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.” Characterisations of the concrete microstructure and the damaging mechanisms were then performed on samples taken from three representative buildings. Results showed important variations in ettringite content and two different cements were identified. In addition, the main physical and mechanical properties of the ancient concrete were evaluated and several propositions were advanced for the development of a compatible repair mortar. Based on these specifications, four mortars were selected, their intrinsic properties were characterised, and the compatibility of the system mortars/concrete was tested. Two mortars, mortars 1 and 2, presented the best compromise between mechanical and physical properties and mortar-substrate compatibility and could be used *in situ* to repair eroded cast stone.

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