



The strength of limestone mortars with quaternary binders: Leaching effect by demineralized water

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HIGHLIGHTS

- ▶ Limestone mortars present an increase in resistance to the demineralized water attack.
- ▶ Weight and compressive strength losses of mortars with mineral additions was minimal.
- ▶ pH of conservation solutions of mortars with mineral additions decreased over time.
- ▶ Different mineral phases produced in this attack.

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ABSTRACT

Some cementitious additions, such as limestone filler, blast furnace slag and natural pozzolana incorporated simultaneously in cement, have a beneficial effect on the durability of limestone mortars immersed in demineralized water. The percentages of the three mineral admixtures combined in cement are 30%, 10% and 10% in turn, comparing them to a Portland cement without mineral admixtures. The lime water serves as reference environment. The specimens were subjected to two protocols of degradation. The first consists to immersing series of specimens in the environment storage for 180 days at 23 ± 1 °C, it is a continuous immersion, while the second protocol is a procedure of accelerated degradation which consists in subjecting the samples of mortars to seven drying-immersion and heating-cooling cycles during 42 days, followed by a continuous immersion of specimens in their exposure environment up to 180 days. The follow-up of the degradation kinetics was achieved in terms of weight change, evolution of compressive strength and monitoring of the pH of storage solutions of mortar sample until 180 days. Several mineral phases are determined by XRD. The concomitant combination of the above mentioned mineral additions in Portland cement showed that the development of mechanical properties and durability of limestone mortars with quaternary binders were better than those of mortars without additions.

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1. Introduction

Indeed, when the cementitious materials are placed in an aggressive environment as demineralized water or weakly mineralized, which are defined as chemically aggressive and correspond to exposure classes XA according to European Standard EN 206-1, several complex physical and chemical phenomena, chemical reactions, processes of dissolution and an exchange with the environment are produced. This dissolution causes an increase in porosity and transport properties, resulting over time in a deterioration of

mechanical properties and decoherence of the material and leads to the loss of bearing capacity and quality of concrete structures. These phenomena are generally grouped under the name of leaching [1]. It concerns all immersed structures, and more particularly those exposed to weakly mineralized water or acids: hydraulic structures, pipes and rain water or sewage tanks.

Previous studies highlighted a progressive dissolution of principal hydrates: initially portlandite, which is the most soluble hydrate, then calcium monosulfoaluminate, and finally the ettringite. After that the portlandite is completely dissolved, the C–S–H decalcify themselves until forming, in the ultimate state of degradation a silica gel [2–5]. Since the pH of the interstitial solution is close to 13.5, the most natural aqueous environments are potentially aggressive with respect to this material and an ionic exchange with the outside can occur within the capillary porosity.

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The degradation kinetics are highly variable and depend in particular on the material and the aqueous environment. For example, Badouix et al. [6] reports that for similar exposure times and an aggressive environment (53 and 66 years), the layers of the degraded surface of a dam and a lock of which thicknesses are 6–8 mm and 1–3 mm, respectively. This difference is attributed to the cement used, a CEM I/CPA for the dam and CEM II/CPD for the lock. Fujiwara et al. [7] showed that the presence of dissolved ions in water also modifies strongly the degradation kinetics of concrete buried in soil.

Indeed, some of these concretes, aged from 60 to 100 years, have been degraded over a few centimeters in presence of a saline water, so a thickness ten times more important than in freshwater. This difference is explained by the aggressive or protector character of certain (some) ions such as CO_3^{2-} which induce the formation of a protective layer on the surface of material. Thus, the high variability of the degradation kinetics observed in existing structures, has led to many studies in laboratory on perfectly reproducible environments. These studies led to works on cement pastes subjected to the pure water [4,8], to mineralized water [9], or an ammonium nitrate solution [10,8]. The aggressive reactions were also accelerated by an electric field [5,11]. Some materials were completely hydrated [4], and others partially hydrated [12].

Many authors have studied the influences of chemical deterioration on the mechanical behavior of cementitious materials. These studies evaluate the mechanical behavior of samples which preliminary underwent a chemical attack. This type of test allows the study of the influences of chemical alteration factor and time of chemical degradation on the mechanical behavior of structure [13,14].

The drying-immersion cycles play an important role in the acceleration of degradation of materials exposed to aggressive environments. The US Office of Reclamation followed during 20 years the dimensional variations of concrete cylinders subjected on the one hand to drying-immersion cycles and on the other hand to a continuous immersion. The results show that 1 year of alternate immersion would be equivalent to 8 years of permanent immersion [15].

The simultaneous use of two or three mineral admixtures as a partial substituent of Portland cement in mortars and concretes has several advantages, in terms of mechanical performance and durability, compared with binary cements [16,17]. However few works have been carried out, until now, on the effect of incorporation of three mineral admixtures in cement of limestone mortar with respect to aggressive environment in particular leaching with demineralized water. In this experimental study, crushed limestone sand was used with quaternary binders in order to study the resistance of mortars to the attack of demineralized water during 180 days. Five mortars were studied, of which a reference mortar manufactured with ordinary Portland cement (OPC), without mineral additives, and four quaternary binder mortars, simultaneously incorporating limestone filler, granulated blast furnace slag and natural pozzolana in partial replacement with Portland cement. The samples were regularly examined by the change of weight and compressive strength. We also proceeded to the follow-up of pH of cure solutions and several mineral phases are determined by XRD.

2. Materials used

The granular class of limestone sand is 0–2.5 mm. The clinker with 5% gypsum and mineral admixtures were ground separately in a laboratory ball mill. The Blaine fineness of cement is $3500 \pm 100 \text{ cm}^2/\text{g}$ and that of mineral additions is $3800 \pm 100 \text{ cm}^2/\text{g}$. The densities of cement, limestone, slag and natural pozzolana are 3, 2.7, 2.6 and 2.8, respectively. The chemical properties of constituents are detailed in Table 1. The sand/cement ratio is equal to 3 and the water/cement ratio is 0.50. The plasticizer added to the mixture is from 2% to 2.5% by weight of the binder

Table 1
Chemical and mineralogical compositions of different compounds.

Composition	Liant			
	Clinker	Limestone	Slag	Pozzolana
SiO_2	21.38	0.76	39.38	57.10
Al_2O_3	5.59	0.41	5.64	15.82
Fe_2O_3	3.21	0.23	2.3	6.16
CaO	65.26	54.9	40.3	5.95
MgO	1.72	0.61	4.50	2.09
K_2O	0.47	0.24	0.46	2.0
Na_2O	0.19	0.04	0.13	1.1
SO_3	0.56	0.61	0.90	0.28
Cl^-	0.02	0.005	-	1.40
LOI	0.58	36.3	0.8	1.2
CClinker mineralogy (%)	C_2S 15.64	C_3S 60.97	C_3A 9.39	C_4AF 9.76

Table 2
Composition of binders.

Composition (%)	0	1	2	3	4
Clinker	95	47.5	47.5	47.5	47.5
Gypsum	5	2.5	2.5	2.5	2.5
Limestone	0	30	10	10	16.67
Slag	0	10	30	10	16.67
Pozzolana	0	10	10	30	16.67

to maintain a plastic workability of fresh mixtures. The mortars were made according to European Standard EN196-1. The samples were cast into $4 \times 4 \times 16 \text{ cm}$ prisms using steel molds, where they stayed for 24 h in a chamber at $23 \pm 1 \text{ }^\circ\text{C}$. After that time, they were unmolded and introduced in a saturated lime solution to prevent any premature loss of calcium during the remaining 28 days of curing.

Five mortars containing limestone aggregates have been made. The compositions of the various mortars are given in Table 2.

3. Experimental methods

The simple leaching process with demineralized water occurs very slowly. To obtain 4 cm thick of degraded cement paste with $\text{W/C} = 0/4$, it would take about 300 years [4]. Moreover, the phenomenon of concrete degradation is even slower because of the aggregates that hinder the diffusion process. So to have acceleration, it is necessary to accelerate either the dissolution process or the diffusion process. There are three methods that can be followed:

- The forced transport of ions by the application of a hydraulic pressure or an electric field.
- The accelerating of the diffusion of ions by increasing concentration gradients between the interstitial solution and the environment.
- The Increasing of the temperature of the aggressive solution which accelerates the diffusion process.

The slow degradation kinetics of mortars by the action of weakly mineralized water, leads us to seek a way to accelerate the degradation process for achieving relatively significant results. The procedure followed in order to accelerate the degradation kinetics consists in applying drying-immersion and cooling-heating cycles to the samples of mortars.

The acceleration of leaching by increasing temperature is used by Prené et al., quoted by Le Bellégo et al. [11]. A campaign of leaching tests on cement paste with CEM I was carried out. The kinetics was controlled by the diffusion for the three studied temperatures (20 °C, 50 °C and 85 °C). They observed an acceleration of the leaching of calcium at 50 °C compared to 20 °C. On the other hand, the total leaching kinetics is identical at 50 °C and 85 °C.

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