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Effect of freeze – Thaw cycles on the physicomechanical properties of a pozzolanic mortar

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HIGHLIGHTS

- Pozzolanic materials may improve the mortar's frost resistance.
- The treated clay has not improved the mortar's behavior when exposed to 300 F-T cycles.
- Despite its pozzolanic potential, damage increased with the clay content.
- The clay fineness (only 3870 cm^2/g) was the main factor of its low efficiency.
- The F-T cycles have favored the progress of cement hydration reactions.

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ABSTRACT

This paper presents an assessment of the frost resistance, after 300 freeze – thaw cycles, of mortars made from cements having different amounts of pozzolan addition. Calcined Algerian clay was used, as clinker replacement, at 0, 10, 15 and 20% by weight. In order to limit its physical pozzolanicity, the fineness of the treated clay, at 700 °C for 5 h, was chosen very close to that of clinker. The aim of this work is to study the influence of the pozzolanic addition on the physical, mechanical and thermal properties of mortars undergoing this kind of cyclical loading. Experimental results show that compressive strength and thermal conductivity decreased, while porosity, water absorption and hydration progress increased. The deteriorations were in an absolute increase with the increase of treated clay level. Although the variant with 10% of addition was the least affected, it was found that whatever the substitution level, the clay fineness has not led to an improvement in pozzolan mortars behavior against the frost attack compared with the control CEM I.

1. Introduction

The resistance of the blended Portland cement mortar and concrete to the freezing and thawing action depends on a number of parameters such as: material strength, its saturation degree and its pore system [1]. It is well known that the principal hydration product affecting the performances of Portland cement products is the Portlandite ($Ca(OH)_2$). However, this phase can be transformed into calcium silicate hydrate phases (C-S-H), if we substitute a part of clinker by pozzolanic addition such as calcined clays. The nature, amount and reactivity of the pozzolan addition have a decisive role in determining the final performance of the blended Portland cement products [1].

In a porous material, such as mortar and concrete, the frost action occurs when water molecules occupying the micro-cracks freeze. The transformation of aqueous solution into ice, in which the volume increase by about 9% [2], leads to the creation of tensile stresses and an increase in the cracks size [3], which causes a loss in mechanical performances of the material. The propagation of microcracks through paste, aggregate, and the interfacial transition zone between paste and aggregates are the main factors in the frost damages [4]. This disadvantage of mortar and concrete can be reduced if we substitute a part of Portland cement by pozzolanic addition which may lead to stronger paste and denser interfacial transition zone [5,6]. This can be explained by the formation of new C-S-H following the chemical reaction between portlandite, derived from cement hydration, and reactive minerals SiO_2 and Al_2O_3 of pozzolan material [7]. The enhancement of mortar and concrete frost resistance was recently reported by many researchers, using different pozzolanic materials, such as: Zeolite [8], Waste

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glass sludge [9], Waste clay brick [10], Metakaoli, Fly ash and Silica fume [11].

The most crucial factors in pozzolanic materials efficiency are: fineness and reactivity. As the Portland cement Blaine fineness varies between 3000 and 4000 cm^2/g, researchers often choose higher finenesses for these materials, to ensure their physical pozzolanicity. Furthermore, traditional pozzolans (MK, FA, SF, Brick waste, etc.) are widely studied and their reactivity has been sufficiently demonstrated. It is known that the calcination of some other clays (montmorillonite, muscovite, illite, etc.) may lead to artificial pozzolans [12–14]. Unfortunately the effect of these materials on mortars behavior against the frost action is not well experienced, especially for moderate fineness.

So, to obtain artificial pozzolan, we used in this paper an abundant clay deposits from Algeria. Mineralogical analysis of the clay showed that it contains 29% of phyllosilicates (Illite, Muscovite and Chlorite) and 46% of carbonates (calcite and dolomite). In a previous research, the clay's pozzolanic activity was studied, where it was subjected to various heat treatments in order to get the most optimal one. It has been found that the treatment which ensures maximum dehydroxylation and minimum decarbonation was at 700 °C for 5 h [15]. The aim of this work is to evaluate the effect of artificial pozzolan on mortar's behavior after 300 freeze – thaw cycles. The mortars were made from modified cements containing 10, 15 and 20% of clinker substitution by treated clay. In this work, compressive strength, hydration progress, water porosity and absorption and thermal conductivity were studied before and after the frost test.

2. Materials and methods

2.1. Materials

An ordinary Portland cement clinker is used in this study, it comes from Meftah cement factory in Algeria. In order to obtain blended cement, we used the calcined clay as substitution of clinker with different amounts. To regulate cements setting, we used gypsum in all cement variants; all materials were separately ground until obtaining very close finenesses (3000 to 4000 cm^2/g), chemical analysis and physical characteristics of clinker, gypsum and clay are shown in Table 1. After that, the materials were sieved then mixed and homogenized according to the selected formulations. In this study, three types of Portland pozzolan cement (CEM II) were used. The CEM II cements were prepared using clinker, gypsum (5% by weight of binder) and calcined clay (10%, 15% and 20% by weight of binder), as control, a Portland cement (CEM I) was produced by mixing 95% of clinker and 5% of gypsum.

2.2. Methods

Cements were used in the confection of mortar specimens (40 × 40 × 160 mm) by using normalized sand, with a binder-sand (b/s) ratio of 1/3, and a water/binder (w/b) ratio of 0.5 by weight.

Mechanical strength, weight, water porosity, water absorption, thermal conductivity and hydrates amount of hardened specimens, at 28 days, were assessed. After 300 freeze and thaw cycles, these tests were done again in order to evaluate the frost resistance of mortar variants. Furthermore, these performance's losses were measured, hence a damage factor was calculated.

Mortar specimens prepared in accordance with EN 196-1, were demolded after 24 h, and preserved in water at room temperature until 28 days. Freezing and thawing (F-T) test was carried out according to the French standard NF 18-425 and without using water. For that, all specimens were covered by a plastic film, to prevent all evaporation phenomena, before submitting them to the test. For driving the test, the climatic room has been coupled to an automatic acquisition central, which allows to record the temperature through a thermocouple. Each day, two cycles were carried out, the mortar specimens were frozen from 9 ± 3 °C to −18 ± 2 °C within 4 h 30 mn and were thawed in 9 ± 3 °C within 3 h. A cycle consists of freezing the sample for 2 h 30 mn in air, followed by 2 h thawing in air also, according to Fig. 1.

Compressive strengths were evaluated by using a 3R test apparatus according to the EN 196-1 standard. Furthermore, and in accordance with EN P18-459 standard, tests of water porosity and absorption coefficient at 44 h were done. Thermal conductivity test was conducted by a non-standardized method based on the proportionality between heat flux and time. The hot-wire method is a measurement test (Fig. 2), which consists in the determination of the temperature rise as a function of time and the distance between a given point and a thermal source embedded between two mortar specimens of the same variant. The thermal conductivity presents the quotient of the heat flux density by the temperature gradient. For each measurement, the test was repeated six times, the average of these values was taken as the result.

The cement hydration is the process allows for minerals C_3S, C_2S, C_3A and C_4AF to react with water and produce hydrated phases of which the most important are C-S-H and Portlandite. Thermogravimetric analysis TGA involves to set samples under the effect of temperature between 20 and 1000 °C with a heating rate of 20 °C/min and cooling of 50 °C/min. The mass losses, corresponding to the evaporation of molecules of water chemically bound in hydrated phases (C-S-H, Portlandite,...), are taken in temperature ranges selected from the TGA curves. This analysis leads to measure the content of each sample in C-S-H and Portlandite before

Table 1
Chemical and physical analyses of materials.

Materials		Clinker	Clay		Gypsum
			Natural	Calcined at (700 °C-5 h)	
Chemical analysis	SiO_2	22.62	34.68	39.91	8.01
	Al_2O_3	5.12	9.16	9.63	2.91
	Fe_2O_3	3.38	3.44	3.42	1.29
	CaO	64.76	22.52	25.96	30.10
	MgO	1.68	4.66	4.94	1.96
	SO_3	0.65	0.94	0.98	35.82
	K_2O	0.49	1.10	1.02	0.48
	Na_2O	0.18	0.14	0.11	0.50
Physical properties	LOI (%)	0.96	22.98	13.24	18.83
	SSB (cm^2/g)	3250	3630	3870	3750
	Density (g/cm^3)	3.12	2.68	2.56	2.49