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Durability of mortars made with sand washing waste

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HIGHLIGHTS

- An industrial waste emanating from sand washing, rich in kaolinite, is used as a pozzolanic material.
- Durability of mortars containing 0%, 10% and 30% of calcined waste is carried out in water, in HCl and Na_2SO_4 solutions.
- Durability of mortars is evaluated by weight loss, expansion test, compressive strength and XRD analyses.
- In the HCl solution, the decalcification of C—S—H and the dissolution of portlandite are observed.
- Sulfates react with portlandite to produce gypsum and expansive ettringite.

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ABSTRACT

The industrial waste emanating from sand washing, rich in kaolinite, was used as a pozzolanic material. The durability of mortars containing 0%, 10% and 30% of calcined waste was carried out in water, in HCl and Na_2SO_4 solutions. In the HCl solution, the decalcification of C—S—H and the dissolution of portlandite were observed. Sulfates react with portlandite to produce gypsum and expansive ettringite. The durability of mortars evaluated by weight loss, expansion test, compressive strength and XRD, showed that mortars containing 10% of waste develop the highest durability in both solutions thanks to the consumption of portlandite by the pozzolan.

1. Introduction

Over the last years, pozzolanic materials have been frequently used. Indeed, pozzolans are known for their significant reduction in CO_2 emissions, in the unit cost of concrete production, and in the heat evolution. The corresponding concretes are characterized by a low permeability, and therefore good durability [1–15]. Metakaolin is one of the most known pozzolanic materials, which improves both strength and durability of mortars and concrete by its reaction with calcium hydroxide and production of C—S—H in the pores structure [16–24].

Among the most aggressive chemicals, sulfates and hydrochloric acid can affect the durability of concretes and mortars structures. Sulfate environment, which come from the soil, ground water, and seawater, are found in combination with other ions such as sodium. It can affect the long-term durability of concrete structures, and leads to expansion, cracking, and deterioration of concrete specimens. The sulfate reacts with calcium hydroxide and calcium aluminate hydrate to form gypsum and secondary ettringite that are more voluminous than the initial reactants [23]. This medium can attack also the structure of calcium silicate hydrate (C—S—H) gel through leaching of the calcium compounds so that stiffness and overall deterioration of the cement paste matrix are occurred [25]. Thaumasite ($CaSiO_3{\cdot}CaCO_3{\cdot}CaSO_4{\cdot}15H_2O$) can be also produced after sulfate attack by the reaction between calcium silicate hydrate, ettringite and carbonates ions [26–34].

The mechanism of sulfate attack is mostly influenced by the concentration of sulfate ions, the ambient temperature, the cement composition, water/cement ratio, porosity and permeability of concrete, and the presence of supplementary cementitious materials [35].

For the hydrochloric acid, the attack takes place immediately when the specimens are immersed in solution. . The reaction with

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the CH produces a calcium salt soluble in water, leading to high mass loss. Calcium silicate hydrate is also affected by this acid and causes its decalcification. This attack depends on the acid nature and concentration [17,36–39].

In a previous work, sand washing waste from the region of Sfax, Tunisia, which contains natural minerals, was studied as pozzolanic material to substitute Portland cement after thermal treatment. An optimum formula of blended cement containing calcined waste was proposed [40].

In the present study, we developed the resistance of composite mortars based on blended cements to aggressive environments and more precisely to chloride (hydrochloric acid HCl) and sulfate (sodium sulfate Na_2SO_4) environments in order to simulate the real conditions (sewage) on which the mortar will be exposed, especially that we intend to include such waste in cement to make sewage pipes.

2. Materials and experimental techniques

2.1. Materials

The materials used in this study were essentially an Ordinary Portland Cement CEM I 42.5 produced by a dry process, a standard sand conforming to European standard EN-196-1 (EN 196-1, 1995) for the mortar preparation, and a waste generated by sand washing. It should be noted that during sand washing, a flocculent (PRAESTOL® 56540, polyelectrolyte based on acrylamide and sodium acrylate) is used in order to flocculate the clay particles, and therefore to facilitate the sand washing operation. The waste was calcined at 650 °C for 3 h in a static bed and then quenched by air to room temperature. The density and Blaine fineness of cement, crude and calcined waste are reported in Table 1.

2.2. Experimental methodology

The chemical composition of samples was determined by X-ray fluorescence (ARL 8400, software XRF 386). X-ray diffraction (XRD) was carried out to determine the mineralogical composition of mortars, of crude and calcined wastes. The X-ray diffractometer used in this investigation was a Brucker D8. The generator settings were 45 kV and 40 mA and the wavelength (λ) was 1.5418 Å (CuKα). The scanning rate was 1° (2θ/min) from 5° to 60°. The crystalline phases were identified from the powder diffraction files (PDF) of the International Center for Diffraction Data (ICDD).

The durability was carried out on 40 * 40 * 160 mm mortar bars containing sand washing waste calcined at 650 °C for 3 h [40]. The water/binder ratio used for all binders was 0.5 and the sand/cement ratio was equal to 3, conforming to European Standard EN 196-1 (EN 196-1, 1995).

All mortar preparations were processed according to the European Standard EN 196-1. Potable water was first introduced in the mechanical mixer. The dry mix solids (cement and calcined waste) were then added to the water and mixed for 30 s at low speed. Finally, sand was added and mixed for 30 s. Then, the mixing proceeds in a sequence of three steps: 30 s mix at high speed, 90 s in rest and 60 s mix at high speed. The mortars were cast into molds for 24 h and cured with plastic sheet.

The specimens were demolded after 24 h and left in water for 28 days at the moist curing room until compressive strength measurement [41,42]. They were then immersed in the aggressive solutions: 16 g/l of sodium sulfate [42] or 5% of HCl [41]. Meanwhile, another series of similar compositions remained stored in water as a reference to make a comparative study. The sulfate solution (Na_2SO_4) was renewed every two weeks during the first three months, and monthly during residual testing period [43]. However, the chloride solution was renewed every two weeks for all test period [44].

The attacked mortar specimens were cleaned with deionized water and then the acid or sulfate attack was evaluated through the measurement of the weight loss of three specimens [42,44,45]. The sulfate attack was also evaluated by expansion on three mortars bars [44].

Strength tests were also performed at 7, 28, 90, 180 and 364 days for the specimens kept in water, in HCl solution or in sulfate solution. All values of the compressive strength reported in this study are mean values of six measurements. For the chloride environment, the study was limited for 90 days. The microstructural aspect of the degraded fraction was evaluated by XRD and SEM. The studied mortars are identified in Table 2.

Table 1
Blaine fineness and density of cement, crude and calcined waste.

	Density (g/cm^3)	Blaine fineness (cm^2/g)
Cement CEM I	3.1	3700
Crude waste	2.7	5095
Calcined waste	2.63	3536

Table 2
Identification of the studied mortars.

Conservation solution	Cement mortar	Pozzolanic mortars (containing sand washing waste)	
		10%	30%
Water	MCW	MPW10	MPW30
Chloride medium	MCCl	MPCl10	MPCl30
Sulfate medium	MCS	MPS10	MPS10

For example

- MCW: Mortars made with pure Cement conserved in Water.
- MPCl10: Mortars containing 10% of calcined sand washing waste (Pouzzolan) immersed in HCl solution.
- MPS30: Mortars containing 30% of calcined sand washing waste (Pouzzolan) immersed Sulfate solution.

3. Results and discussion

3.1. Material characterization

The results of X-ray fluorescence of the crude waste and of cement are reported in Table 3. It is clear that the waste is an alumino-silicate material containing a high amount of SiO_2. In addition, this waste is rich in CaO with some Fe_2O_3. X-ray diffraction spectrum (Fig. 1a) indicates that this material contains a fraction of clay mineral based on kaolinite and illite. High quantities of associated minerals (quartz and calcite) are also present.

The presence of kaolinite in the mineralogical composition of waste washing sand indicates that this material can play the role of pozzolan after specified treatment.

The heat treatment causes the disappearance of the crystalline structure of kaolinite (Fig. 1b) and the appearance of an amorphous phase, potentially reactive with $Ca(OH)_2$. Moreover, the heat treatment causes the decarbonation of calcite proved by the disappearance of the peaks relative to $CaCO_3$ in the calcined waste spectrum.

3.2. Durability in the HCl solution

Fig. 2 presents the weight loss of specimens immersed in the chloride solution. With immersion period, a continuous weight loss for all mortars is observed. The highest weight loss was recorded after 90 days for the sample without mineral admixture (MCCl) ($\approx$12%), and the lowest for the sample with 10% of pozzolan (MPCl10) ($\approx$4%). For the mortar containing 30% of pozzolana (MPCl30), the loss in weight is 8%. We expect from these values that the durability in HCl solution is in the following order: MPCl10 > MPCl30 > MCCl.

In order to confirm this result, compressive strength measurements are performed. Fig. 3 presents the evolutions of the compressive strength of mortar bars conserved in water and others in HCl solution. From this figure, it is clear that the compressive strength of mortars conserved in water increases with curing

Table 3
Chemical composition of the cement and crude waste.

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3
Crude waste(%wt)	45.93	13.49	5.31	12.45	2.11	1.46	1.11
Cement (%wt)	21.1	4.73	2.86	65.31	–	0.50	2.34