



Freeze-thaw effect on the durability of binary cements containing activated coal-mining waste



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HIGHLIGHTS

- The starting coal waste presented aluminosilicate properties.
- Once the waste had been activated, the presence of kaolinite was not identified.
- The addition 20% ACW to the blended cement has an influence on the hydrated phases.
- After 300 freeze/thaw cycles, the 20% ACW mortar presented a higher resistance.
- After 300 freeze/thaw cycles, the 20% OPC present less surface microfissuring in the crystals.

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ABSTRACT

The present work contains an approach to the evaluation of binary cements prepared with a 20% substitution of coal-mining waste and their behaviour under a maximum of 300 freeze/thaw cycles. Mineralogical studies are conducted, as well as tests on mass variations, compressive strength, microporosity, the dynamic modulus of elasticity, and SEM morphologies. It is important to highlight that the partial replacement of cement by 20% of activated coal waste, does not affect the durability against freeze/thaw cycles, since they show a fairly similar tendency to the reference mortar under extreme temperature changes.

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1. Introduction

One of the main causes for the deterioration of stone and therefore of construction materials in the natural world is the effect of abrupt temperature change in certain geographical regions where frequent freeze/thaw cycles alternate with high diurnal temperatures. Those processes generate microcracking differential alteration and the reduction of crystallinity on rock surfaces and, therefore, a significant loss of their engineering properties [1]. Various factors and specific topological features intervene in the

deterioration process, such as the prevailing climate, thermal oscillations [2], slope inclination and orientation, the frequency of the freeze/thaw cycles, and the location of primary cracking and microcracking [3].

These parameters and their variables are repeated in zones with similar geomorphological and lithological parameters, such as the Yosemite National Park (USA), the National Park of Spitzkoppe (Namibia), the Natural Reserve of Val di Mello (Italy) [4], and the Ladakh batholith (India). In Spain, these deterioration processes are very well represented in the Pedriza de Manzanares (Madrid) and the Montes Gallegos (Galicia). Similar microcracking processes may be observed in all of some landscapes that are predominantly formed of granite lithologies. On sandstone samples, under the effect of freeze–thaw cycles, the sandstone pores become connected with each other, some of the micro-pores and mini-pores

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are changed into meso-pores and macro-pores and the rock pore structures have changed, which would lead to the change of mechanic properties [5].

Investigations in which the extent of cracking can be estimated through ultrasonic wave propagation (V_p) establish parameters [6] that can also quantify the changes provoked by microcracking due to the freeze/thaw effect [3]. The variation of V_p throughout the freeze/thaw cycles shows a perceptible change as the microcracking occur [7]. The deterioration is not in the first stages (num. of freeze/thaw cycles 0, 70, 140, 196, 210, 280), although intergranular microcracking appears, at approximately 140 cycles and thereafter (at cycle 280), the microcracking is differential, depending on the mineralogy of the rock, and grain sizes. The quartz veins of polymineralic rocks, such as the granitoids, undergo microcracking under pressure, while the biotites, due to their greater flexibility undergo deformation with little or no cracking.

Concretes, due to their porous nature and their high content of natural aggregates have always presented serious structural problems, when simultaneously exposed to saline water at low temperatures [8–10]. If robust materials that endure throughout the lifecycle are not considered in the design phase, then the service life of concrete structures that are continually exposed to the elements and the aggressive environments described earlier in the context of rocks will be shorter. Hence, durability, especially in the context of freeze–thaw cycles, is therefore an essential concept [11].

The deterioration of paste matrixes due to abrupt temperature changes (freeze/thaw cycles) is still one of the most significant problems for the scientific community today and their durability is a priority line of research. Research has therefore been conducted over decades on the effects of freeze–thaw cycles on deterioration so that new and sufficiently robust materials may be produced that can withstand them better [12]. In the 1940s, Powers [13], one of the pioneering researchers in this field, had already described the effects of freezing capillary pore (pore sizes between 10 and 10,000 nm) water in hydrated paste matrixes and the consequent deterioration. Numerous researches have since defined the deterioration mechanisms due to freeze/thaw cycles through various models to predict the results [14]. They have all contributed to the development of current standards and to the design of paste matrixes capable of withstanding freeze/thaw cycles [15].

Different solutions may be highlighted from the bibliography to solve or to limit the deterioration process. It has been studied a poro-elastic–plastic model to predict changes in the pore structure and reduction in the mechanical property of cement-based materials when subjected to freeze–thaw cycles [16]. They range from the incorporation of air-entrainment agents to produce air spaces that serve as expansion chambers when the water freezes within the porous network [17–19], to predominantly silane-based hydrophobic surface treatments [18], as well as the incorporation of pozzolanic additions (silica fume, metakaolin, calcinated clays, ladle-furnace slags, and fly ash) [20–28]. The pozzolanic additions behave well in so far as they are related with a more refined pore structure, lower permeability, the presence of anhydride cement particles [29,30] and even, lower amounts of portlandite [31].

However, one drawback is their wide variety of sources and properties (natural, natural activated, industrial, agro-industrial) [32,33]. It leaves open a still somewhat unknown field of investigation, above all, for future pozzolans with hydraulic properties, which present a very promising future within the global strategy for a Circular Economy.

Recently, one line of research has centred on coal-mining waste as an innovative means of obtaining recycled metakaolin (MK) and using it in the manufacture of future eco-efficient cements. Studies [34,35] have previously demonstrated the highly pozzolanic properties of these thermally activated products, a kinetic reaction that

is similar to a natural MK one, and a good physical–mechanical behaviour of the binary cements that incorporate them. However, there is a significant scientific gap in reference to improvements in durability.

In the present paper, the results are presented of up to 300 cycles of accelerated freeze/thaw tests on cement mortars prepared with a 20% substitution of cement by pozzolans (consisting of thermally activated coal-mining waste). To the best of the authors' knowledge, this is the first study on paste matrixes with pozzolanic materials that have undergone freeze/thaw tests. It includes their mineralogical characterization, and measurements of mass variation, compressive strength, microporosity, the dynamic modulus of elasticity, and SEM morphologies.

2. Materials and methods

2.1. Raw materials

A coal waste (CW) supplied by the Sociedad Anónima Hullera Vasco – Leonesa (La Robla, León, Spain) was used for the present work. Following grinding of the waste to a particle size of under 65 μm , it underwent a process of thermal activation at 600 °C over 3 h in a furnace [36]. Conditions that are considered appropriate (in both economic and energetic terms) for the conversion of an industrial waste into a highly pozzolanic Activated Coal Waste (ACW) product.

An ordinary Portland cement (OPC) type CEM I 52.5 N was used, in accordance with current regulations [37]. It was mixed with a standard siliceous sand with a content of >98% SiO_2 for the manufacture of the cement mortars. Table 1 and Fig. 1 show the chemical composition by XRF and the mineralogical phases by XRD, respectively.

The analysis and the quantification of the mineralogical phases performed by XRD spectrometry revealed the presence of phyllosilicates 2:1 (mica 25%), phyllosilicates 1:1 (kaolinite 14%), quartz (29%), calcite (15%), and amorphous materials (17%) in the coal waste, with values of $R = 24.6$ and $X^2 = 5.7$ in the Rietveld quantification (Fig. 1); while mica (20%), quartz (35%), calcite (10%), and amorphous material (35%) were present in the ACW pozzolan with $R = 13.2$ and $X^2 = 6.9$. In the case of the OPC cement, the components were alite (35%), bredigite (25%), C_3A (15%), C_4AF (8%), bassanite (7%), and calcite (1%) with $R = 26.8$ and $X^2 = 14.7$.

The cement mortars for the durability tests were completed in accordance with the specifications in standard UNE EN 196-1 2005 [38], with a water/binder ratio of 0.5 and a binder/sand ratio of 1:3. The blended cements were prepared with the partial substitution of 20% of the cement by ACW, to obtain a II/A CEM type cement (6–20%), in accordance with regulation UNE EN 197-1 (2011) [37] and, on the other hand, to a previous work published by the authors [39].

2.2. Testing methods

As in previous research works [39,40], several test methods were used to assess concrete deterioration after successive freeze/thaw cycles [23,42–44], all of which are detailed in standard ASTM C666-03 (2008) [15]. That formed the basis of the test procedures in this study.

Prismatic and cubic test specimens were manufactured for each type of cement:

- 1) 3 prismatic specimens of $80 \times 80 \times 400$ mm were manufactured for each type of cement, to monitor variations in their dynamic modulus.
- 2) 12 cubic specimens of 100 mm were manufactured to assess the evolution of compressive strength at 0, 94, and 300 freeze–thaw cycles and were determined according to the specifications in standard EN 12390-3 (2002) [45].

Table 1
Chemical composition of the starting materials by XRF analysis.

Oxides (%)	CW	OPC	ACW
SiO_2	49.79	20.00	53.63
Al_2O_3	21.77	5.70	25.29
Fe_2O_3	4.07	3.20	4.64
MnO	0.08	0.03	0.08
MgO	0.64	1.70	0.77
CaO	3.84	57.90	4.20
Na_2O	0.13	0.77	0.17
SO_3	0.27	4.30	0.27
K_2O	2.74	1.60	3.09
TiO_2	1.07	0.15	1.17
P_2O_5	0.13	0.21	0.14
L.O.I.	15.18	2.57	3.09

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