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Assessment of the single and combined effect of superabsorbent particles and porogenic agents in nanotitania-containing mortars

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A R T I C L E I N F O

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A B S T R A C T

This article reports an investigation on the development of multifunctional mortars with enhanced potential to passively adjust indoor RH levels and thermal conductivity, while reducing aerial pollutants (NO_x) through photocatalysis. Superabsorbent polymer (SAP), Vermiculite (VER) and TiO_2 nanoparticles (nT) were used as functional additives for mortars. Single and binary mixtures with 0–0.9 wt.% SAP and 0–15 wt.% VER were established based on flow table measurements (165–180 mm), while 1.0 wt.% nT remained invariable in all tested samples. In addition, the workability was adjusted by distinct amounts of water in the mixtures. In general, the effect of the VER particles predominated for most of the properties. SAP-single mixtures with similar workability showed distinct rheological behavior over time, while the yield stress represented better the continuous absorption of water molecules by SAP. VER single mixtures imposed deeper changes on the flow, hindering the rheological features. Changes on the apparent porosity, water absorption and capillarity limited the admissible VER level, but for SAP the compositional range is less restricted. In fact, no adverse effect or gain on the flexural strength was observed on SAP-containing mixtures. The internal pores created by VER reduced in 50% the thermal conductivity of 0.9SAP + 10VER in comparison to 0.9SAP + 0VER. In addition, a significant increment of moisture buffering value in VER-containing mortars was also reached by using SAP particles. Finally, all samples were photocatalytically active against NOx, reaching up to 80% reduction of its initial concentration, after 60 min, regardless the concentration of additives.

1. Introduction

The ability of materials to interact with the internal environment of buildings allows extensive worldwide industrial applications due to novel functionalities that might be achieved. Application of additives in cement based materials, aiming to passively modulate the hygrothermal conditions and assure healthier breathable conditions of interior envelopes, is growing and several studies have been reported on that [1–18]. Examples include the individual or combined use of expanded vermiculite (VER), superabsorbent polymers (SAP) or nano-TiO_2 (n-TiO_2) with other additives. In fact, cement pastes containing expanded VER show higher thermal resistance than pure ones [1], while the thermal conductivity remained lower than EPS [2]. In addtion, the workability and rheology of VER-containing mortars are negatively affected regarding to perlite [3]. The superabsorbent polymers (SAP) proved to be highly effective in the moisture buffering of mortars [4,5]. It can also be used as an alternative to the internal curing agent for the prevention of self-desiccation, avoiding crack formation, increment of hydration and reduction of permeability [6–8]. The combined use of SAP particles and colloidal silica induces positive effects on the compressive strength [9], while the gradual release of additional water stored by SAP, combined with pozzolanic reaction of fly ash may create a denser matrix [10,11]. However, the swelling effect is severely reduced in SAP-containing cement paste due to formation of calcium carbonate precipitates [9].

Innovative applications have been also reached by using n-TiO_2 in cementitious materials, such as environmental pollution remediation, self-cleaning and self-disinfection [12], but the agglomeration effect of n-TiO_2 in cement media tends to minimize the benefits [13]. At early ages, n-TiO_2 acts as a catalyst in the cement hydration reactions [14] and can improve the mechan-

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Table 1
Chemical composition of Portland cement CEM I—42.5 determined by XRF.

Component (in wt.%)	Portland Cement
SiO_2	20.37
CaO	63.05
Al_2O_3	4.78
Fe_2O_3	2.96
SO_3	3.70
MgO	2.02
Cl^-	0.018
K_2O	–
Loss On Ignition	2.37

ical properties and microstructure of black rice husk ash-containing mortars [15]. Results showed that the decline of the contact angle and the pore structure refinement observed with the addition of nano-TiO_2 slowed down the water loss of hardened cement paste, which resulted in a similar drying shrinkage to that of the control sample at 1 month age [16]. In self-compacting mortars containing fly ash, the effect estimates revealed that ternary combination of n-SiO_2, n-Al_2O_3 and n-TiO_2 (3% wt each) assures the best chloride permeability results [19].

Those studies suggest that the use of multiple additives requires a complete evaluation of potential interactions between them. As previously mentioned, VER-containing mortars show remarkable thermal insulation properties but the additional dosage of water required to keep the workability of fresh mixes under acceptable limits tend to damage the mechanical performance of cured samples. SAP particles assured optimal moisture buffering performances [4,20], while the internal porosity and density of mortars seem not to be much affected. In this way, the combination of VER and SAP can assure complementary functionalities, preserving the usual properties of the material, though.

Therefore, the purpose of this experimental research work is estimating the individual and combined performance of VER and SAP particles in n-TiO_2-containing mortars in order to define optimized formulations for targeted applications. Several properties in fresh (rheometry and flow table), and hardened state features (apparent porosity, water absorption, capillary index, flexural strength and weight variation) were evaluated up to 28 days of curing and complementary functionalities were assessed (moisture buffering capacity, thermal conductivity and NO_x photocatalytic degradation).

2. Experimental

2.1. Materials

Portland cement (OPC-type I 42.5R, according to EN 197-1 [21]) was used as binder in the mortars. It has a specific surface area (SSA) of 0.35 m^2/g (Blaine fineness), average particle size of 14 mm and its chemical composition is given in Table 1. Hydrated lime (Calcidrata, Portugal) has about 90% $Ca(OH)_2$ and humidity below 0.1%. The sand employed as aggregate was composed by particles in the range of 0.08–1.25 mm. Vermiculite (VER, Aguiar e Melo, Portugal) was selected to change the internal porosity of the mortar, while a superabsorbent (SAP, T 5066 G, Evonik, Germany) was used due to its strong absorption capability. A commercial photocatalytic titania powder (Evonik Aeroxide P25) was added. This n-TiO_2 is a mixture of anatase, rutile and amorphous phase (76.3; 10.6 and 13.0 wt%, respectively), with a SSA of ~50 m^2 as reported elsewhere [22].

VER and SAP particles size range between 315 and 1230 μm and ~100 μm, respectively, while the average size value of n-TiO_2 particles is 21 nm, as indicated by the manufacturer. The fresh mortar adjustment was achieved using a superplasticizer (SP, Glenium 52, BASF, Germany) admixture based on a polycarboxylic acid, with density of ~1.05 g/cm^3 and containing 20 wt% solids.

Table 2
Experimental program.

Measurements	Time (days)
Spread on table and rheology	0
Water absorption, apparent porosity and capillary index	28
Flexural strength	28
NO_x photocatalytic degradation	28
Moisture buffering capacity	28
Thermal conductivity measurements	28

Table 3
Formulation of mortars.

Composition	CEM (g)	Lime	SAP	VER	nT	Sand	W	SP	Spread (mm)
0.00SAP + 0VER + 0.7W/B	100	100	–	–	9.6	600	140.0	1.5	178
0.00SAP + 5VER + 1.1W/B	100	100	–	30	9.6	570	220.0	1.5	171
0.00SAP + 10VER + 1.5W/B	100	100	–	60	9.6	540	300.0	1.5	165
0.00SAP + 15VER + 2.0W/B	100	100	–	90	9.6	510	400.0	1.5	168
0.15SAP + 0VER + 0.7W/B	100	100	1.2	–	9.6	600	140.0	1.5	165
0.30SAP + 0VER + 0.7W/B	100	100	2.4	–	9.6	600	140.0	1.5	178
0.30SAP + 5VER + 1.1W/B	100	100	2.4	30	9.6	570	220.0	1.5	171
0.30SAP + 10VER + 1.5W/B	100	100	2.4	60	9.6	540	300.0	1.5	169
0.30SAP + 15VER + 2.0W/B	100	100	2.4	90	9.6	510	400.0	1.5	165
0.60SAP + 0VER + 0.7W/B	100	100	4.8	–	9.6	600	140.0	1.5	167
0.90SAP + 0VER + 0.8W/B	100	100	7.2	–	9.6	600	160.0	1.5	170
0.90SAP + 5VER + 1.1W/B	100	100	7.2	30	9.6	570	220.0	1.5	170
0.90SAP + 10VER + 1.6W/B	100	100	7.2	60	9.6	540	320.0	1.5	168
0.90SAP + 15VER + 2.0W/B	100	100	7.2	90	9.6	510	400.0	1.5	165

2.2. Testing procedures

Several properties were evaluated (Table 2), attempting to compare the impact of the SAP and VER additives on the overall behavior of the mortar. The workability of all mortars was kept invariable, but the strong impact of VER on the workability limited its admissible dosage when W/B ratio was held unchanged. However, this adverse condition was overlapped by using distinct dosages of water. The preparation of mortars with binder/aggregate weight ratio (B/A) of 1:3 involved: (a) weighing the components, (b) drying mixing solid components bag for 1 min, (c) adding superplasticizer into water, (d) pouring the solid components into water, (e) mixing the components mechanically for 3.5 min. Complete mortar formulations are shown in Table 3.

After demoulding, samples were cured in a room with controlled relative humidity (65% RH) and temperature (23 °C) and then tested after 28 days of curing in terms of flexural strength (EN 1015-11:1999) [23], and water capillary absorption coefficient (EN 1015-18:1999) [24]. Unrestrained shrinkage and weight variation [25], water absorption and apparent porosity values were also determined by an immersion method [26]. The variance of experimental error was checked by using one single replication for the spread on table and rheometry, while 3 replications were carried out for flexural strength, water capillary absorption coefficient, water absorption and apparent porosity determinations.

2.3. Flow table and rheological characterization

Flow table or consistency test is a traditional method to evaluate the workability of mortars. It gives only qualitative information about the consistency of the mixture and allows the evaluation of the influence of fine materials added [3,4,27,28]. The flow table test (Fig. 1a) was performed after mechanical mix, following EN 1015-3:2007 [29]. Slump values between 165 and 180 mm were achieved by using distinct dosages of water.