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The Influence of the Ageing Process on the Mechanical Properties of Cement Mortars with Nano-SiO_2 Admixture Initially Subjected to Thermal Treatment †

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Abstract: The research aimed to evaluate the effect of nano-silica (NS) on the strength recovery of the previously heated cement mortars. One hundred twenty cement mortar specimens were prepared with 1% to 5% of NS. The specimens were heated at temperatures ranging from 200 °C to 800 °C. Half of the samples, after heating and cooling, were mechanically tested. The other half of the specimens were stored for 24 months in 90% relative humidity. The presence of NS fostered partial recovery of the compressive strength of the heated mortars. The cement mortars regained more than 40% of their compressive strength after heating at 400 °C and about 25% after heating at 600 °C. The strength recovery phenomenon faded away at 800 °C.

Keywords: cement mortars; compressive strength; high temperature; nano-silica

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1. Introduction

Despite good thermal resistance, cement composites are subject to physical and chemical changes at high temperatures. These changes lead to the worsening of mechanical performance and structural defects. The research described in [1] and [2] has confirmed that a temperature lower than 300 °C does not affect the cement composites' strength. A significant strength drop is observed when the temperature reaches 400 °C [3], and at 800 °C, the strength decreases to be below 30% of the initial strength [4]. The limit temperature is 1000 °C, above which the cement composites lose most of their total strength [5]. The worsening of the mechanical performance of the cement composites, including concrete, at a high temperature is connected to the decomposition of hydrates, and mainly, the C-S-H phase and portlandite [6], a non-uniform thermal gradient, which causes shrinkage of the cement matrix and expansion of the coarse aggregate [7], as well as the loss of bound water, which creates voids [8].

Recent studies showed [9–12] that cement concrete damaged by fire can regain its original strength if exposed to moisture for a long time. The process of mechanical property recovery by concrete is called the after-fire hardening technique. The main reason for regaining the strength of the cement composites is the rehydration of the cement matrix dehydrated during the fire [11–13].

The tests on the cement mortars containing 1% to 5% of colloidal nano-silica (NS) have shown that NS can support the partial strength recovery of the cement composite following its heating at a high temperature.

2. Materials and Methods

2.1. Materials

The mortars to be tested were prepared with the use of Portland cement CEM I 42.5N, a magnetite aggregate with a grain size up to 2 mm and apparent density of 4.77 g/cm^3, and the water dispersion of nano-silica. The water dispersion of NS with the density of 1.4 g/cm^3 contained 50% of the colloidal NS by volume. The diameters of the NS particles were from 40 to 140 nm. The TEM images of NS particles are presented in Figure 1.

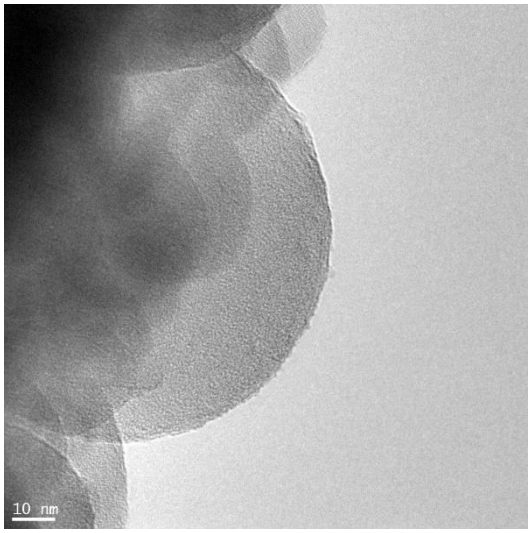

Figure 1. TEM images of nano-silica (NS) [14].

2.2. Samples and Their Storage

The tests were carried out on the cement mortars whose compositions are presented in Table 1. The water content was reduced in mortars containing nano-silica in the form of dispersion. Specimens of the size 40 mm × 40 mm × 160 mm were prepared according to the European Standard PN-EN 196-1 [15].

Table 1. Composition of the tested mortars.

Mortar Designation	Cement (kg/m^3)	Water (kg/m^3)	Magnetite (kg/m^3)	NS (% of Cement Mass)	NS Dispersion (kg/m^3)
M0	450	225	2439	0	0
M1	450	220.5	2439	1	9
M2	450	216	2439	2	18
M3	450	221.5	2439	3	27
M4	450	207	2439	4	36
M5	450	202	2439	5	45

The demoulded specimens were stored in a climate chamber on the grate above water at 20 ± 2 °C. After 28 days of curing, the specimens were heated. Then, part of the samples were tested, and the others were stored in the chamber for 24 months until further testing. In total, sixty specimens of the tested mortars were conditioned for two years, from which two specimens of each mortar were initially heated at the given temperature.

2.3. Test Methods

After 28 days of curing, the specimens were heated in the medium-temperature oven. In the first stage, the temperature in the oven rose at the rate of 1 °C/min until reaching the specified value. The specimens were heated at 200, 400, 600, and 800 °C. The final heating temperature was maintained for 60 min so that the entire volume of the specimens

could achieve the proper uniform temperature. Then, the specimens were cooled down at the rate of 1 °C/min. The cooled samples were removed from the oven and weighed. The compressive strength of the remaining specimens was determined after 24 months of storage in the climate chamber. The compressive strength was determined according to the European Standard PN-EN 196-1 [15].

3. Results and Discussion

3.1. Compressive Strength after Heating

The results of the compressive strength testing after heating are presented in Figure 2. The positive effect of the NS admixture becomes visible with heating at an increasing temperature. The highest compressive strength within the tested temperature range was determined for M3 mortar specimens.

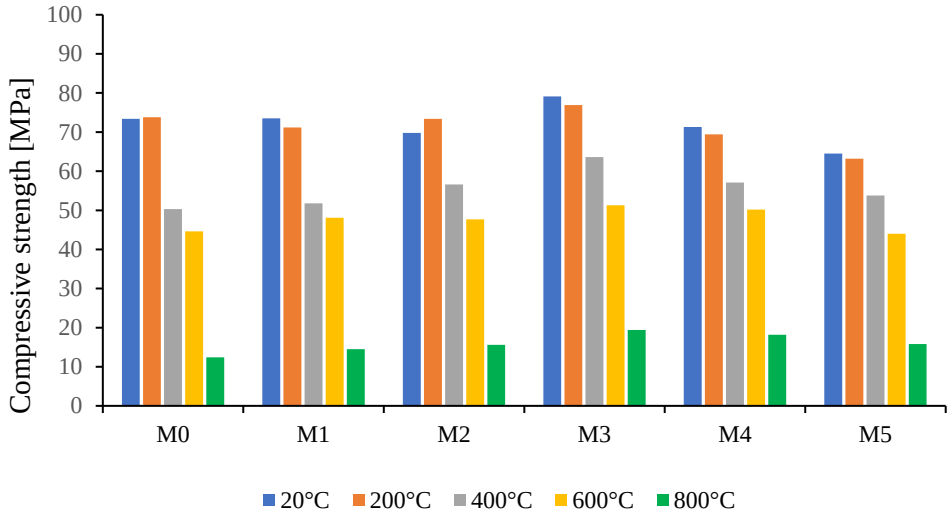

Figure 2. Compressive strength of the heated mortars.

3.2. Mechanical Property Recovery by Mortars Damaged at High Temperature

Some of the heated mortar specimens were stored for 24 months in the moisture conditions (see Point 2.2) and then tested. The results of compressive strength determination for these specimens are presented in Figure 3. The highest growth of the compressive strength was observed for M3 mortar specimens within the entire range of temperatures. However, the significant recovery of the strength after 24 months of storage was noted for all tested mortars.

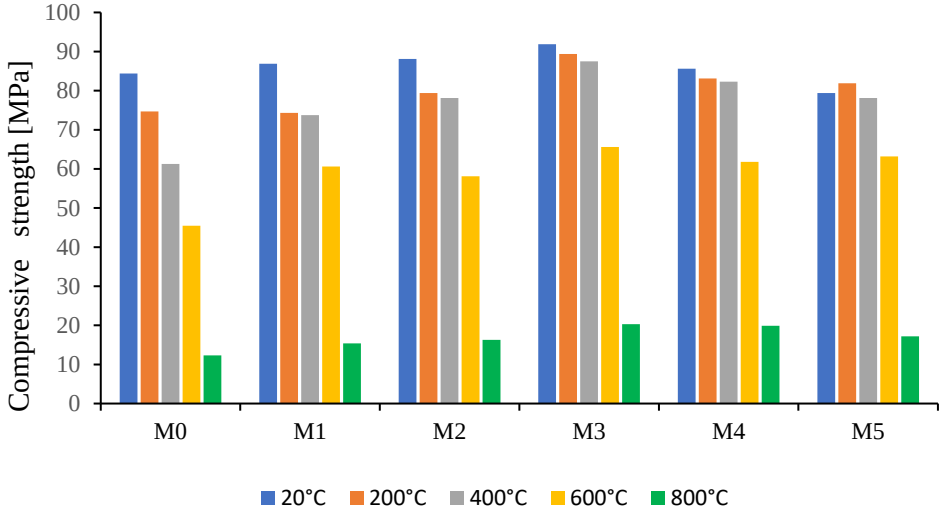

Figure 3. Compressive strength of the mortars after heating and curing for the next 24 months.

The recovery of the mechanical properties of the heated cement mortars after 24 months of storage in the moisture conditions is presented in Figure 4. The compressive strength determined after heating was accepted as the reference strength, and the strength after 24 months of storage was compared to it.

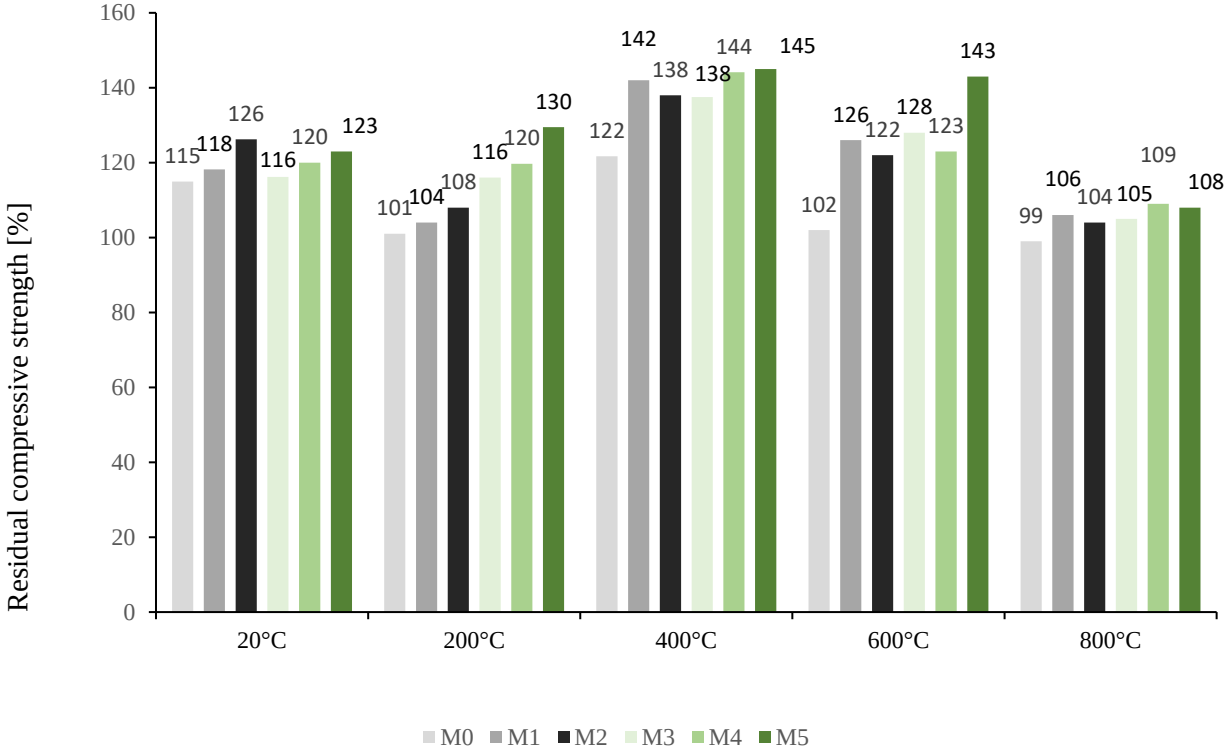

Figure 4. Relative compressive strength of the heated mortars after 24 months of further curing.

After 24 months of storage, all mortar specimens achieved a compressive strength higher than that obtained immediately after heating, except for the M0 mortar, which demonstrated a slight decrease in the average strength (by 1%) after heating at 800 °C. The positive effect of the NS's presence on the mortars' structure recovery is visible. The increase in the mortars' strength after heating at 200 °C was proportional to the NS content. The moisture delivered during storage in the climate chamber after heating made it possible for the NS to hydrate fully, which led to the growth of strength. The highest increase in the strength was observed for the specimens heated at 400 °C. The compressive strength of the mortars containing NS increased by 38% to 45%, while the compressive strength of the mortar M0 without NS increased by only 22%. The strength of the mortars with NS, heated at 600 °C, also rose after 24 months of storage. However, the increase was smaller (23% to 28%), except for in mortar M5, where a strength increase of 43% was noted. The phenomenon of a strength increase fades away after heating at 800 °C. Some compressive strength growth was observed only for the mortars with the higher NS content; it was 9% for the mortar M4 and 8% for the mortar M5. For the other mortars, the strength increased by about 5%, which is on the border of the measurement error.

The compressive strength increase in the heated cement mortars is caused by the rehydration of the C-S-H phase and hydration of other, previously not hydrated, components, such as cement grains, NS, and CaO [16,17]. The partial regaining of the compressive strength by the mortars containing the NS is possible when the volume of delivered water is sufficient for the hydration of yet unhydrated nano-silica. As shown in the study described in [18], the NS agglomerates forming in the cement matrix at the NS content above 3% are the reason for decreasing strength after heating the mortars compared to the mortars without nano-silica. The downfall of the strength is caused, among other reasons, by the air voids formed in the area of NS agglomerates. The tests presented in this paper

have shown that sufficient water delivery leads to the hydration of yet unhydrated NS and, consequently, the filling of the created air pores and voids. The filling of the empty spaces causes, in turn, the higher compressive strength increase for the mortars M4 and M5. At the temperature of 800 °C, the damages in the material's internal structure are too big for significant strength recovery. The authors plan to continue the investigation using more sophisticated testing methods, including scanning electron microscopy, X-ray diffractometry, and X-ray computer tomography.

4. Conclusions

The presented research aimed to evaluate the nano-silica (NS) influence on the strength recovery of the previously heated cement mortars. The tests have shown that NS fosters compressive strength recovery by the mortars heated up to 600 °C. The composites regain more than 40% of the compressive strength after heating at 400 °C and about 25% after heating at 600 °C. The main reason for the strength recovery is the rehydration of the C-S-H phase and the hydration of other, previously not hydrated, components. Additionally, the hydration of yet unhydrated nano-silica contributes to regaining the compressive strength when the volume of delivered water is sufficient. The strength recovery phenomenon fades away at 800 °C when the damages in the material's internal structure are too big for significant strength recovery.

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