Influence of perlite and aerogel addition on the performance of cement-based mortars at elevated temperatures

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Abstract. Structures confront severe damages when subjected to fire, related to the characteristics of the fire (i.e. maximum temperature, duration) and the constructional types and materials confronted. During the last decades, the influence of fire and extreme temperatures on building materials has been induced. In this paper, the influence of perlite and aerogel addition on the resistance of cement-based mortars at elevated temperatures is studied. Four compositions of cement-mortars have been manufactured and tested at 200°C, 400°C, 600°C, 800°C and 1000°C. The mortar mixtures was based on cement I42.5 and siliceous sand (0-4mm) in a Binder/Aggregate ratio of 1/2. A proportion of 20% w/w of aggregates was substituted by perlite, while an addition of aerogel at a proportion of 0.5% w/w of binders was performed. After their exposure at the elevated temperatures, the physico-mechanical properties of the specimens were recorded, concerning: volume change, weight loss, porosity, apparent specific gravity, dynamic modulus of elasticity, flexural and compressive strength. Additionally, macroscopic and microstructural observation was conducted. From the comparative evaluation of the results it was concluded that although the perlite and aerogel addition, reduced the initial strength of the mortar compositions, they had a positive effect on their resistance at elevated temperatures, especially regarding their volume stability and mechanical properties above 800°C.

1. Introduction
From the ancient times and until nowadays, fire has been a significant decay factor of structures, leading to extreme damages of constructional elements and materials. The damage degree is usually related to the characteristics of the fire (i.e. maximum temperature attained, fire duration), as well as the type and properties of building materials. When subjected to fire, specific properties of materials are influenced, regarding physical, mechanical and chemical characteristics [1-6]. The impact of fire on concrete and concrete structures has been studied from the beginning of the 20th century (1922) [7], while during the last decades research has been induced [3-5, 8-9]. It is focusing on various aspects, concerning the analysis and prediction of the fire scenarios (heating rate, exposure temperature and time), the design and testing of fire-resistant materials, as well as the development and application of post-fire assessment and repair strategies. To this direction, relevant Regulative frames and Recommendations provide critical data (RILEM TC 129-MHT, RILEM TC 200-HTC, EN 1991-1-2 Eurocode 1, EN 1992 Eurocode 2, ISO 834-11:2014, BS 476-3:2004) [10-15]. The temperature/time development curves, as given by the Standards (Fig. 1), show that the maximum temperature attained during a fire, is around 1100-1200°C and the temperature rate may vary [16]. It is generally accepted that during the first 30 minutes of the action, temperature is rapidly increased at 822°C. In order to study the performance of building materials at elevated temperatures and due to the
difficulty of simulating such an extreme temperature rate at laboratory level, researchers propose a development rate ranging from 5 to 10°C/min, [1-6].

Since concrete and cement-based mortars are heterogeneous materials, elevated temperatures influence both their paste and aggregates in different ways and according to their constituents and thermal strains (expansion of aggregates, shrinkage deformations of the cement paste) [1-6]. Their post-fire pathology symptoms, usually concern colour change, spalling, cracking, delamination, and deformation [7-9].

According to literature, between 100-200°C, the free moisture of the cement-based materials evaporates, while above 250°C the dehydration or loss of the bonded water begins [1-6]. Above 300°C the silicate hydrates of the cement paste (C–S–H gel) decompose and above 500°C portlandite, influencing the stability of the matrix [4]. Aggregates function according to their origin, whereas siliceous aggregates seem to be more resistant than calcareous ones [4].

Regarding the mechanical characteristics of concrete and cement-based mortars, for temperatures up to 300°C the residual strength is maintained, while between 300°C and 500°C it is decreased around 15-40% [1-6]. Above 550-600°C, it is minimized and secondary chemical reactions occur (decarbonisation of carbonates in both the cement paste and the aggregates) [4]. Usually above 800°C, complete disintegration of the constituents begins.

In the present paper, an effort has been made to study the resistance of cement-based mortars at elevated temperatures. To this direction, the impact of expanded perlite and aerogel addition was envisaged. During the experimental work, four compositions of cement-based mortars were manufactured and subjected to extreme temperatures (200°C, 400°C, 600°C, 800°C and 1000°C). A proportion of 20% w/w of the aggregates was substituted by expanded perlite, while aerogel at a proportion of 0.5% w/w of binders was added. All mortar specimens were tested before and after their exposure at the elevated temperatures and their physico-mechanical properties were recorded. From the comparative evaluation of the results it was concluded that although the perlite and aerogel addition, reduced the initial strength of the mortar compositions, they had a positive effect on their resistance at elevated temperatures, especially regarding their volume stability and mechanical properties above 800°C.

2. Materials and Methods

During the experimental work, four compositions of cement-based mortars were manufactured and tested. They were based on cement I42.5 and natural, siliceous sand of gradation 0-4mm, in a Binder/Aggregate (B/A) ratio 1/2. In an effort to enhance the resistance of the mortars at elevated temperatures, a proportion of 20% w/w of aggregates was substituted by expanded perlite, while aerogel at a proportion of 0.5% w/w of binders was added. All mortar specimens were tested before and after their exposure at the elevated temperatures and their physico-mechanical properties were recorded. From the comparative evaluation of the results it was concluded that although the perlite and aerogel addition, reduced the initial strength of the mortar compositions, they had a positive effect on their resistance at elevated temperatures, especially regarding their volume stability and mechanical properties above 800°C.
Regarding perlite, it is an amorphous, alumino-silicate mineral, coming from volcanic deposits. After its industrial process, the outcoming product, named expanded perlite (EP), presents light weight and insulating properties, rendering it an alternative raw material used in constructions [17-18]. China, Greece, Turkey and US are the most important perlite producers, resulting in the 95% of the worldwide production (4.6 million tons for the year 2016) [18]. Perlite mining has generally little overburden to manage and minimal environmental impact, while during the last 60 years less than the 1% of the perlite reserves have been exploited [18].

Aerogel on the other hand, is a synthetic lightweight, porous material that mainly consists of air (98%). The pores are of nano-dimension and contain still air. Due to this property, aerogel offers exceptional thermal insulation capabilities as it is almost weightless (low density is around 0.020g/cm³). Aerogels alone are hydrophilic, but chemical treatment can turn them into hydrophobic.

In order to reduce the water demand, a sulphate free, polycarboxylate superplasticizer was added in a proportion of 1% w/w of binders. The Water/Binder (W/B) ratio was adjusted for achieving workability of 15±1cm, according to EN1015-3. The manufacture and curing of the mortar specimens was according to EN1015-11, while totally 16 specimens (dimensions 4x4x16cm) of each mortar series were manufactured and tested. In Table 1 the constituents and proportions of the mortar series are presented.

Table 1. Constituents and proportions of the mortar series.

| Raw materials                        | Mortar series / Parts of weight | C       | C-P     | C-A     | C-P-A   |
|--------------------------------------|---------------------------------|---------|---------|---------|---------|
| Cement I42.5                         |                                 | 1       | 1       | 1       | 1       |
| Sand of siliceous origin, pale colour (0-4mm) |                                 | 2       | 1.6     | 2       | 1.6     |
| Expanded perlite (0-4mm)             |                                 | -       | 0.4     | -       | 0.4     |
| Aerogel (% w/w of binders)           |                                 | -       | -       | 0.5%    | 0.5%    |
| Super plasticizer (% w/w of binders) |                                 | 1%      | 1%      | 1%      | 1%      |
| W/B ratio                            |                                 | 0.36    | 0.69    | 0.38    | 0.68    |
| Workability (cm) (according to EN1015-3:1999) |                                 | 15.5    | 15.7    | 16.0    | 15.8    |

Twenty-eight days after their manufacture, the physico-mechanical properties of the mortar compositions were recorded, regarding porosity and apparent specific gravity (RILEM CPC 11.3), water absorption coefficient due to capillary action (EN 1015-18:2002), dynamic modulus of elasticity (BS 1881-203:1986), flexural and compressive strength (EN1015-11:1999).

Subsequently, two specimens of each mortar composition were subjected to elevated temperatures (200°C, 400°C, 600°C, 800°C and 1000°C), according to former research work [6]. For the experiment, an electric furnace was used, where the temperature rate and duration time could be manually set (Fig. 2). The heating scheme followed (maximum temperature, heating rate, exposure time and cooling rate) was based on former research work and was aligned with the contemporary research on cement-mortars and concrete [1-6]. The methodology concerned heating rate 5°C/min, exposure duration to the maximum temperature 2h and cooling rate 2°C/min (Fig.2).
Figure 2. Electric furnace (left) and heating scheme (rate, exposure time, cooling rate) (right).

After the exposure to each temperature level, the specimens maintained for 24h at laboratory conditions and their weight and dimensions were recorded, in order to estimate their weight and volume changes. Their physico-mechanical characteristics were afterwards tested, regarding porosity, apparent specific gravity, dynamic modulus of elasticity, flexural and compressive strength. Macroscopic observation of the specimens and colour determination (according to the Munsell chart) were also attained. All the results were comparatively evaluated in order to identify the impact of the perlite and aerogel addition on the resistance of cement-based mortars subjected at elevated temperatures.

3. Results and Discussion

The physico-mechanical properties of the mortar specimens at the age of 28 days are presented in Table 2. The characteristics concern porosity, apparent specific gravity, dynamic modulus of elasticity, flexural and compressive strength, while the water absorption coefficient due to capillary action is presented in Figure 3.

| Mortar series | Porosity (%) | Ap. Spec. Gravity | Dyn. Mod. of Elasticity (GPa) | Flexural strength (MPa) | Compressive strength (MPa) |
|---------------|--------------|--------------------|------------------------------|-------------------------|---------------------------|
| C             | 8.68         | 2.153              | 37.546                       | 6.20                    | 60.79                     |
| C-P           | 15.72        | 1.706              | 18.925                       | 3.91                    | 21.97                     |
| C-A           | 9.45         | 2.097              | 33.342                       | 5.20                    | 54.23                     |
| C-P-A         | 18.60        | 1.607              | 16.847                       | 4.61                    | 20.65                     |

Figure 3. Water absorption coefficient due to capillary action of the mortar compositions (28d).
From the evaluation of the results (Table 2) it was asserted that the substitution of the 20% w/w of aggregates with perlite (C-P), resulted in a significant increase of porosity (~80%) and reduction of apparent specific gravity (20%). Correspondingly, the water absorption coefficient due to capillary action (Fig. 3) was extremely increased. Regarding the mechanical characteristics, a drop of strength was recorded (around 60%), mainly influencing compressive strength.

The addition of a 0.5% w/w of binders of aerogel in the reference mortar (C-A), also influenced the physico-mechanical properties. Porosity was slightly increased (~9%), as well as compressive strength (~10%). Water absorption coefficient due to capillary action (Fig. 3) was slightly increased.

When both perlite and aerogel were added in the mixture (C-P-A), the highest increase of porosity was attained (~115%), as well as the lowest strength values (reduction around 66%). Water absorption coefficient values were increased, however below the C-P ones (Fig. 3).

Regarding the analysis results taken after the exposure of the specimens at the elevated temperatures (200°C, 400°C, 600°C, 800°C and 1000°C), the following remarks could be asserted:

The volume change of the specimens presented fluctuations (Fig. 4), whereas a volume increase was recorded in all cases, ranging from 2-9%. The highest volume increase was observed for composition C-P, with the pick to be at 400°C, while C-P-A showed the lowest fluctuations. Regarding the mass change, the results were more concentrated, showing gradual increase of weight (5-20%) until 1000°C (Fig. 4). Compositions C and C-P presented the same trend, while C-P-A presented higher weight increase. On the other hand, C-A and C-P-A followed the values given by C and C-P respectively, providing a significant weight loss at 400°C.

![Figure 4](image1.png)

**Figure 4.** Volume and mass change of the specimens exposed at elevated temperatures.

Regarding porosity and specific gravity changes throughout the tested temperatures (Fig. 5) it was observed that compositions C and C-A followed the same trend, as well as compositions C-P and C-P-A.
A. Generally, C and C-A showed lower porosity values, following the initial ones, which in all cases were gradually increased (almost doubled) up to 800°C. At 1000°C a drop of porosity was attained (almost the same with 600°C). The remarks regarding porosity were in accordance with the apparent specific gravity values.

![Porosity and apparent specific gravity of the specimens exposed at elevated temperatures.](image)

**Figure 5.** Porosity and apparent specific gravity of the specimens exposed at elevated temperatures.

Concerning the mechanical characteristics of the specimens exposed at elevated temperatures (Fig.6), it was generally observed that all values were decreased throughout the temperature increase. Regarding Modulus of elasticity, up to 400°C the values were gradually decreased around 45% for all compositions, while at 600°C a significant drop was observed, especially for composition C (reduction around 90%). For the rest of the compositions, the decrease was less intense, ranging from 70-75%. At 800 and 1000°C all values were at the same level (2-4GPa).

The flexural and compressive strength values of the specimens followed the same trend. The highest loss of strength at 800°C was shown for composition C (85% reduction for flexural and 65% for compressive strength), while the lower one for composition C-P-A (around 60-65%). It was generally attained that although the initial compressive strength values of composition C were significantly higher (60MPa) than those of the modified ones (20MPa), they all provided the same range of results at the highest temperatures of 800 and 1000°C (5-15MPa). In the case of C-A, up to 600°C the strength level was maintained (42-55MPa), while at 1000°C the highest strength value was recorded (15MPa).
Figure 6. Mechanical characteristics (Dyn. Modulus of elasticity, flexural and compressive strength) of the specimens exposed at elevated temperatures.

The structure of the specimens, after their exposure at the elevated temperatures was generally maintained, as presented in Figure 7. At 600°C limited spalling and colour alterations were observed, which were more intense at 800 and 1000°C. Regarding the more extreme color changes (according to the Munsel scale), composition C was converted from GLEY 2, 7/1 light bluish gray to GLEY 2, 8/1 light greenish gray, and C-P from GLEY 2 8/1 light bluish gray to 2.5Y 8/2 pale yellow.

Figure 7. Macroscopic photos of the specimens exposed at elevated temperatures.
4. Conclusions
The design and testing of alternative building materials, resistant to extreme temperatures, is a research field that has been induced during the last decades and is nowadays of great interest. According to the research results of the present study, the substitution of natural aggregates by expanded perlite in cement-based mortars, could result in producing performable, fire resistant and lightweight materials.

The proportion of perlite in the mixtures is of great importance, since it has a direct impact on the initial strength capacity of the materials. The tested substitution of 20% w/w of aggregates, resulted in a decrease of apparent specific gravity around 30% and a drop of the initial strength around 60%. However, it maintained the residual strength after the exposure to the highest temperatures at the same level (800-1000°C). It is therefore considered that a lower substitution of aggregates by perlite (i.e. 10%) could offer more favourable results regarding both the initial and the residual strength capacity.

On the other hand, the addition of aerogel (0.5% w/w of binders), resulted in an initial strength decrease of 8%, while it positively influenced the behaviour of the mortars at the extreme temperatures. The parallel addition of perlite and aerogel had the most favorable impact on the final mortars’ properties.

Concluding, it is therefore noticed that both the perlite and aerogel addition in cement-based mortars, could significantly enhance their resistance to elevated temperatures. Their proportion in the mixtures is of great importance and should be further envisaged.

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