Residual strength and degradation of cement mortar containing polypropylene fibers at elevated temperature

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Abstract. The elevated temperatures caused by fire affect the physical and chemical properties of the mortar and thus, influence its mechanical properties. This study presents the results of an experimental investigation on the mechanical properties and microstructure of cement based mortar composites contained polypropylene (PP) at high temperatures. The mortar was prepared and cured as per the relevant international standards. Upon curing, tested specimens were exposed to 250, 450, 600 and 900 °C for 2 h. The influence of the high temperatures on the physical and chemical changes of the blended cement mortar was also analyzed by Scanning Electron Microscopy. Mortar specimens exposed to different high temperatures were tested for compressive and flexural strengths, and compared with control specimens (i.e. unheated). Results revealed that even low fiber dosage of PP fibers with mortars was helpful in completely preventing explosive spalling due to significant increase of permeability. Mechanical results and microstructural analysis of mortar specimens showed that both compressive and tensile strengths were initially increased at 250°C, followed by considerable reduction at higher temperatures.

Keywords
High temperature; residual strength; mortar

1. Introduction

One of the most important advantages of Concrete and mortar is the ability to withstand high temperatures and combustion because of the low thermal conductivity properties as well as high specific heat characteristics [1]. In spite of that, high temperatures and fire may influence concrete properties. The durability of concrete structures can also be affected by physical damage processes due to exposure to high temperature, which may cause color changes as well as affect the concrete’s mechanical properties, concrete density, elastic modulus and its surface appearance [2, 3].

Changes in mortars that are exposed to high temperatures and even fire could include loss in compressive and tensile strengths, morphological changes, surface discoloration, cracking and finally spalling, depending on the temperature level and the duration of exposure time to the high temperatures or fire [4, 5].

The importance of these properties in assessments of heated cement based composites has led engineers to evaluate decline in the mechanical properties [6, 7], color [8, 9], development of cracking...
and spalling [10, 12] of fire-damaged mortar under the effect of various temperatures, and to develop an examining procedure to assess fire-damaged concrete [7, 13].

Due to the hydration reaction of cement particles with water, two major constitutes are produced. They are 60%–70% C-S-H gel and 20%–30% portlandite (Ca(OH)$_2$) [14, 15]. Previous studies in this field clearly show that the internal structure of cement particle hydrates vary upon exposure to elevated temperatures. Firstly, the free water evaporates at about 30 to 100 °C; secondly, the bound water losses due to the decomposition of C-S-H and the hydration of carboaluminate occurs at about 180 to 300 °C [16]. Subsequently, dehydroxylation of portlandite (CH) takes place at 450 to 500 °C, while C-S-H gel eventually decomposes at 700 °C or above [17]. At temperatures more than (900°C), a complete C-S-H decomposition takes place [18].

Throughout these stages, the dehydration of cement hydrates causes significant changes in the pore microstructure and leading to significant changes in the physical and mechanical properties of hardened cementitious mortar.

At normal temperatures, concrete thermal properties remain regular. However, these thermal properties are changed at high temperatures because of change in moisture content in concrete components and decomposition of the hydration products. The rate of temperature rise, time, and maximum temperature of exposure are highly responsible for the degradation process associated with possible strength reduction. [13]. On the contrary, the influence of high temperature on the hydrated cement paste be based on the degree of hydration and its moisture content.

Furthermore, the exposure of low permeability mortar to high heating rate may result in surface spalling. This may happen because of the steam vapor pressure rising inside the mortar at a faster rate than the pressure relief that results from steam release into the atmosphere.

Nevertheless, hard damage may occur also owing to the expansion of lime throughout the cooling period [19]. This deterioration can be noticed during process of fire putting out, where CaO reacts with water and turns into Ca(OH)$_2$ which is associated with a large amount of expansion.

The use of polypropylene fibers with concrete mixture is proved to be valuable to prevent concrete spalling and the inclusion of even minimum percentages of PP fibers may help to mitigate explosive spalling in cement based composites [20, 21].

Exposure of the cement-based composite to elevated temperatures affects its durability characteristics and final performance. This destructive impact can be minimized by using appropriate mix proportions or adding other materials such as mineral admixtures [2]. The results of this study are expected to provide a better understanding of this effect to the scientific community, and it will help explore the fire resistance and mechanical strength degradation of cement-based composites due to elevated temperatures.

The aim of this research work is to assess the physical and mechanical changes in terms of microstructure, compressive, flexural strengths residues of hardened cementitious mortar containing polypropylene (PP) fibers for 2 hours of exposure to various elevated temperatures and to obtain basic data for assessing the flaw and damage on cement-based materials by fire. Mortar specimens were evaluated before and after exposure to temperatures of up to 900 °C.

2. Experimental work
2.1. Materials
Ordinary Portland cement (OPC) commercially known as MASS conforming to ASTM C 150 [22] was used in this study (see Table 1). Standard sand (S) conforms to ASTM C778-13 [23] was employed as a fine aggregate. Super plasticizer (SP) admixture Type F based on modified polycarboxylic ether was used to increase the flowability of the mixture and reduce the water demand.

Table 1. Chemical composition of OPC used.

| Oxide composition | OPC (%) |
|-------------------|---------|
| CaO               | 62      |
| SiO₂              | 20      |
| Al₂O₃             | 4.73    |
| Fe₂O₃             | 3.22    |
| MgO               | 3.55    |
| SO₃               | 2.44    |
| Na₂O              | 0.24    |
| K₂O               | 0.64    |
| Total             | 96.21   |
| Ignition loss     | 2.31    |
| Insoluble residue | 0.91    |

Table 2. Mixture proportion of mortar (g).

| code of specimens | w/c | Water | OPC | S   | SP | PP |
|-------------------|-----|-------|-----|-----|----|----|
| p                 | 0.484 | 242  | 500 | 1375 | 1.5 | 0.9 |

2.2. Mortar Preparation and Identification

Mixing procedure and mix proportion implemented in this study were according to ASTM C305 [24] and ASTM C109 [25], respectively, and as also followed in the study of Hassan et al. [26-28]. Table 2 shows mix proportions and contents for each component. Mortar specimens were made using cement: sand proportion of 1:2.75 and water to cement ratio (w/c) of 0.484. The mortar specimens were cast into 50 mm cubes for compressive and 40×40×160 mm prisms for flexural strengths tests. The form works were vibrated for 30 s to expel all air bubbles from the fresh mixtures. The specimens were kept in molds for 24 h, and then water cured until testing age. The water was extracted from hardened cement mortar specimens by piece of cloth prior to testing. Thereafter, cube and prism specimens were exposed to a sequence of increasing temperatures, 250, 450, 600, and 900°C for two hours, to explore the thermal stability of cement hydrate phase. The specimens were then left inside the furnace to cool steadily to normal temperature. Both compressive and flexural tests were executed on specimens before and after heating.

3. Results and discussion
### 3.1 Compressive strength

This test was implemented to assess the residual compressive strength after exposure to elevated temperature. The average value of three cubes was recorded at 28, 90 days of ages and presented in Table 3 and Figures 1 to 4. As can be seen, the compressive strength values increased with temperature up to 250 °C then declined as the temperature increased up to 900 °C. Figures 2 to 4 show the improvements in compressive strength at 250 °C were 10.48% and 4.68% while the corresponding losses in compressive strength at 450 °C, 600 °C and 900 °C were 48.1%, 53.8%, 90.48%, 36.17%, 56.17% and 84.68% for 28 and 90 days, respectively. In general, there is a slight increase in compressive strength at 250 °C followed by sharp reduction at 450 °C and 900 °C while at 600 °C it reduces slightly. This trend is consistent and agreed with the findings of Morsy [29]. The initial improvement in compressive strength could be attributed to the further hydration of unhydrated cement particles due to the steam pressure inside the pores occurring during phenomena called Internal Autoclaving Influence [29]. Over 250 °C, the reduction in compressive strength with temperature is related to the desiccation of CH at a temperature close to 450 °C generating CaO and H₂O. The losses in strength after 600 °C mostly occur due to the dehydration of calcium carbonate and escape of CO₂ from CaCO₃ components. Essentially, at 900 °C the strength loss is attributed to CSH decomposition as well as the increased microcracking resulting from stresses generated from thermal changes, which are formed due to temperature rises from 250 to 900 °C. In another words the reduction in compressive strength with temperature ranging from 250 to 900 °C was associated with the evaporation of free water and a part of the hydration water leading to induce micro-cracks. The decompositions of CH and CSH are the other possible causes of such reductions.

| TEMPERATURE (°C) | COMPRSSIVE STRENGTH (MPA) | CHANGE (%) | TEMPERATURE (°C) | COMPRSSIVE STRENGTH (MPA) | CHANGE (%) | 90/28 d of Change |
|------------------|---------------------------|-------------|------------------|---------------------------|-------------|------------------|
| 25               | 21                        | 0           | 23.5             | 0                         | 0           | 0.44             |
| 250              | 23.2                      | 10.48       | 24.6             | 4.68                      | 0           | 0.75             |
| 450              | 10.9                      | -48.1       | 15               | -36.17                    | -3.6        | 1.04             |
| 600              | 9.7                       | -53.8       | 10.3             | -56.17                    | -84.68      | 0.93             |
| 900              | 2                         | -90.48      | 3.6              | -84.68                    | -84.68      |                   |
3.2. Flexural strength.

This test was implemented to assess the residual Flexural strength after exposure to elevated temperature. The average value of three prisms was recorded at 28, 90 days of ages and presented in table 4 and figures 5 to 8. As can be seen, the flexural strength initially increased with temperature reaching to 250 °C. With progressive temperature increase from 250 to 900 °C, the flexural strength reduced accordingly. Figures 6 and 8 show the percentages of development in flexural strengths at 250 °C was 5.64% and 14.04% while the corresponding losses in flexural strength at 450 °C, 600 °C and 900 °C were 49.94%, 80.7%, 92.3%, 50.15%, 80.87% and 89.73% for 28 and 90 days, respectively. There is a slight increase in flexural strength at 250 °C followed by sharp reduction at 450 °C then it decreases gradually with temperature increasing up to 900 °C. This trend is similar to that observed in compressive strengths and agreed with the finding of other researchers [27].

The development in flexural strength at initial stage of heating (i.e. up to 250 °C) could be attributed to the further hydration of unhydrated cement particles caused by the influence of steam pressure inside the paste pores. Morsy [29] mentioned that possible explanation of this effect is the condition of the so-called internal autoclaving phenomena. In a similar way to compression behavior, the results of the residual flexural strength showed a significant reduction at temperature over 250 °C. This is again associated with the desiccation of calcium hydroxide when the temperature reached about 450 °C leading to form further CaO and H₂O. It is also worth mentioning that the formation of micro-cracks and decomposition of hydration products especially CSH at heating range from 250 to 900 °C were behind the reduction in flexural strength. This behavior is correlated with the residual in compressive strength results discussed in the preceding section.

To explain the effect of age of test (28 and 90 d) and strength types (compressive and flexural) on the residual strengths due to exposure to a series of increasing temperatures in the current study, the residual strength ratios of later age to earlier age (90/28) are reported in tables 3 and 4. In the compressive strength case, this ratio (90/28) increased as the temperature increased from 250 to 450 and then to 600 °C indicating higher CH contents in later ages when compared with earlier ages. In contrast, 90/28 ratios of flexural strength residues of specimens exposed to similar temperature were close to one.

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**Figure 1.** Residual compressive strength at elevated temperature on 28 day.

**Figure 2.** % Change of compressive strength according to unheated control mortar.

**Figure 3.** Residual compressive strength at elevated temperature on 90 day.

**Figure 4.** % Change of compressive strength according to unheated control mortar.
indicating that with time mortars lose more compressive strength than they lose in flexural strength when exposed to fire.

**Table 4.** Flexural strength after temperature exposure.

| TEMP (°C) | Flexural Strength (Mpa) | Change (%) | Flexural Strength (Mpa) | Change (%) | 90/28 d of Change |
|-----------|-------------------------|------------|-------------------------|------------|------------------|
| 25        | 6.09                    | Increase (+). | 6.14                    | Increase (+). | 0                |
| 250       | 6.43                    | Decrease (-). | 7                       | Decrease (-). | 0                |
| 450       | 3.1                     | -49.94     | 3.1                     | -50.15     | 0.99             |
| 600       | 1.18                    | -80.7      | 1.18                    | -80.87     | 1.00             |
| 900       | 0.47                    | -92.3      | 0.63                    | -89.73     | 0.97             |

**Figure 5.** Residual flexural strength at elevated temperature on 28 day.

**Figure 6.** Change of flexural strength according to unheated control mortar.
3.3. Microstructure
To specify changes in the microstructure of the specimens which may supply a link to changes in compressive and flexural strengths at normal temperature and after exposure to four temperatures, SEM was carried out.

The SEM images of 28 cured mortar samples at (25) °C and subjected to (250 and 900 °C) temperatures are shown in figure 10. As can be seen, obviously, the features of the hydrated cement mortar at age of 28 days and before exposing to increased temperature, figure 10a, shows the presence of nearly amorphous, CSH and homogeneous cement paste matrix without any appearance of micro-cracks. Upon exposure of hydrated cement mortar specimens to a temperature of 900 °C, the decomposition of hydration products could be observed clearly as well as the formation of narrow micro-cracks (Figure 10c). Moreover, a gap between the aggregate edge and cement paste could be observed clearly owing to the heating effects up to 900 °C leading to separation of the cement paste from the aggregate. The poor microstructure shown in figure 10c is related to the creation of unwanted arrangement of CSH layers of crystals, and excess cracking at higher temperature. In general, the CSH crystals develop an unordered spaces occupied in the matrix at elevated temperatures leading to less densification of the microstructure [20]. The developed micro-cracking is resulting from elevated thermal stresses that are created because of temperature differential.

![Figure 7](image1.png)  
**Figure 7.** Residual flexural strength at elevated temperature on 90 day.

![Figure 8](image2.png)  
**Figure 8.** % Change of flexural strength according to unheated control mortar.
Figure 9. SEM images of hydrated cement mortar at (a) 25 °C, (b) after exposure to 250 °C and (c) after exposure to 900 °C.

The inclusion of polypropylene fibers (PP) seems to help reduce the pore pressure through releasing internally trapped moisture and vapor, and then prevent explosive spalling. Figure 11 did not show spalling even after exposure to 900 for 2 h. The macro-cracks observed in figure 10c disintegrated the specimen into several sections. Such macro cracks could be responsible for creating free surfaces for water vapor to release. Furthermore, use of polypropylene fibers created micro paths and channels, which could also help to release trapped vapor and improve specimen’s microstructure strengths against spalling.

Figure 10. Spalling extent of hydrated cement mortar with PP fibers after exposing to elevated temperature
4. Summary and conclusions
In the current study, the influence of elevated temperature on the residual mechanical strength and microstructural characteristics is studied experimentally. Based on the test results and the microstructural observations, the following conclusions can be drawn:

1. The residual mechanical properties (compressive and flexural strengths) improved initially until reaching a specific temperature; identified here in this study to be 250 °C. Thereafter, these properties reduced continuously as the temperature of exposure increases up to 900 °C. This reduction may be owing to appearance of micro- and macro-cracks in the hydrated cement paste phase due to elevated temperatures.

2. The SEM images of the hydrated cement paste phase heated at 900 °C indicate formation and development of micro-cracks.

3. Further amounts of hydration products including CSH and CH are increased with heating until the temperature reached 250 °C. Possible explanation is the continuous hydration of unhydrated cement particles in the hardened cement paste.

4. Comparison of residual compressive and flexural strength results revealed that with time mortars lose more compressive strength than they lose flexural strength when exposed to fire.

5. Differential thermal stresses under increased heat exposure are responsible for the formation of macro and micro-cracks, which result in the loss in compressive and flexural strength.

6. The addition of polypropylene fibers seems to be helpful in reducing pore pressure by releasing internally trapped moisture and vapor, and then to prevent explosive spalling.

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