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# Mechanical properties of Portland cement mortar containing multi-walled carbon nanotubes at elevated temperatures

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## HIGHLIGHTS

- Effect of MWCNTs dosage on the mechanical properties of Portland cement mortar.
- Elevated temperature resistance of Portland cement mortar containing MWCNTs.
- The microstructure of Portland cement mortar was characterized by petrography images.
- Optimum dosage of MWCNTs (0.1%) improved the studied mechanical properties.

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## ABSTRACT

Addition of nanomaterials to Portland cement mortars could potentially enhance their properties. It has been previously reported that addition of multi-walled carbon nanotubes (MWCNTs) to mortars can lead to considerable improvement in the mechanical properties of the binder paste. In this study, the effect of the addition of MWCNTs at the ratios of 0–0.15 wt% of Portland cement on the compressive, tensile, and flexural strength; and resistance to the elevated temperature of Portland cement mortar was evaluated. The specimens were heated up at the elevated temperatures (200–800 °C) and accordingly, the mass loss and compressive strength were determined. The microstructure of the samples was analyzed by petrography. The obtained results showed that the mortar samples containing MWCNT acquired higher mechanical strength and resistance to the elevated temperatures, in comparison with the ordinary mortar samples. The mortar containing solely 0.1 wt% MWCNT showed an increase in the compressive, tensile, and flexure strength by 35, 8 and 11.2%, respectively. Moreover, the residual compressive strength after heating showed significant improvement. Petrography image analyses also indicated that the porosity of the cement paste was reduced, whereas the cement paste became denser and more stable due to the addition of MWCNT.



## 1. Introduction

Recently, there has been a great increase in the utilization of nanotechnology to improve the performance of concrete [1]. Typically, micro cracks might cause larger cracks leading to breakdowns. One of the advantages of using nanotubes in concrete is that these nano-scale materials can fill the cracks [2]. In general, nano-materials increase the mechanical properties and durability of concrete; for instance, nano$TiO_2$ improves the distribution of hydration products [3]. The analyses of SEM images by Li et al. [4] have suggested that nano-materials are not a good filler; they act as a hydration activator promoting the microstructure of the cement paste provided that they are equally distributed.

In 1991, the first utilization of carbon nanotubes (CNTs) was reported by Iijima [5]. Multi-walled carbon nanotubes (MWCNTs) have high mechanical, aspect ratio, thermal, chemical, and electrical properties [6,7]. In a study done by Konsta et al. [8] they added 0.025, 0.048, and 0.08 wt% MWCNTs into the cement paste matrix. They found that using MWCNTs could increase the stiffness of C-S-H; also, the porosity was decreased in the cementitious matrix. Danoglidis et al. [9] used MWCNTs at 0.1% by the weight of cement in the mortar. They observed that the flexure strength, Young's modulus, flexure toughness, and first crack strength were increased about 1.9, 2, 1.8 and 1.7 times, respectively, as compared to the control mortar. In another work, Chaipanich et al. [10] added CNTs into the mortar containing fly ash, and CNTs were dispersed

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by using ultrasonication. They found the highest compressive strength for the 20% fly ash with 1% CNTs. Morsy et al. [11] produced a mortar with nano metakaolin (NMK) using 6 wt% of cement and CNTs with the amounts of 0.005, 0.02, 0.05 and 0.1 wt% of cement. The compressive strength of the mortar containing 6 wt% NMK and 0.02 wt% CNTs was increased by 29%, as compared to the control mortar. Abu alrub et al. [12] also studied the effect of the long aspect ratio (1250–3750) and the short aspect ratio (157) of CNTs in cementitious composites. Specimens containing 0.04 wt % CNTs with a short aspect ratio showed an increase of 66% in the flexural strength, as compared to the control specimens. They proposed that CNTs acted as a bridge to reduce and limit the micro cracks. Wang et al. [13] also reported that the addition of 0.08 wt % of MWCNTs in the cement-based composites led to the increase of the flexural strength by 57.5%; further, the samples containing 0.12 wt% of MWCNTs showed a decrease in porosity, by 21%. Indeed, there are several works in the literature that report on the utilization of CNTs. For instance, a study performed by Saffi et al. [14] investigated the effect of MWCNTs with the amounts of 0.1, 0.5 and 1 wt% on the mechanical and electrical properties of geopolymer composites. The MWCNTs were uniformly distributed and the stiffness and ductility were increased. However, these MWCNTs exhibited some agglomeration in the sample containing 1 wt% and consequently, the load deflection was similar to that in the control samples. Rovnanik et al. [15] also studied MWCNTs at variable amounts (0.05–1 wt%) in the alkali activated slag mortar. The optimum dosage of MWCNTs was 0.1 wt% in terms of mechanical fracture properties; compressive strength was also increased by 27%. Sun et al. [16–18] used polyvinyl alcohol to increase the interfacial interaction between CNTs and the cement matrix. Thus, the flexural strength of the composites had a significant increase. They believed that the –COOH groups of CNTs generated the surface treatment that could improve the interface between CNTs and cement.

Although concrete is generally known as a resistant material to the elevated temperatures, it is also well known that the prolonged high temperature can have a considerable influence on the concrete properties [19,20]. Theoretically, the fire resistance properties of concrete depend on its compositions characteristics at variable temperatures, particularly the cement paste and aggregates. In concrete samples subjected to the elevated temperature, depending on the type of constituents and the microstructure of the concrete, the concrete strength may be reduced considerably [21]. Morsy et al. [20,22] investigated the effect of the elevated temperatures (250–800 °C) on the mechanical properties of the mortar containing nano metakaolin. Substituting the ordinary Portland cement by nano metakaolin with amounts of 0, 5, 10 and 15 wt% led to an increase in the compressive strength at 250 °C. They proposed that the internal autoclaved effect was responsible for this increase; on the other hand, above this temperature, a decrease in the compressive strength due to the dehydration of calcium hydroxide and calcium carbonate dissociation led to giving off $CO_2$ from $CaCO_3$. At the temperature range of 100–300 °C, the hydration growth increased the concrete strength, which could be attributed to the larger mobility of water molecules in the gaseous phase [23]. The weight loss is one of the parameters investigated for the characterization of the cement matrix at the elevated temperatures. By heating the cement paste, the progress of weight loss could be increased; subsequently, physical and chemical water from the cement matrix could be evaporated. The physical evaporation occurs normally at temperatures up to 300 °C, but the chemical one could occur at 300–1000 °C. Addition of the nano-materials to the cement matrix could cause an increase in the weight loss at all temperatures [24,25]. In the temperature range of 400–1000 °C, the weakness of the microstructure and the appearance of the micro cracks due to the degradation of portlandite and the decomposition of C-S-H, as well as the formation of $CaO_2$ crystals during the cooling period, have been observed [26,27]. By using CNTs in the mortar, the cement paste was modified and boosting the compression, also increased the resistance of the cement paste at the elevated temperatures [28].

In fact, there is still not enough information regarding Portland cement mortar features containing CNTs at the elevated temperatures. The main aim of this work was, therefore, to utilize MWCNTs in the Portland cement mortar in order to investigate its effect on the strength of the mortar at the elevated temperatures.

## 2. Materials and methods

### 2.1. Materials

In this study, the ordinary Portland cement produced by Ardestan Company, in compliance with the ASTM C150 standard, was utilized. Its chemical composition and physical properties are shown in Table 1. The MWCNTs were provided by Neutrino Company. The properties of MWCNTs are shown in Table 2. Standard sand, according to the ASTM C778 standard, was used in the mixture. A polycarboxylate-based superplasticizer was used for the preparation of the mixture.

### 2.2. Mortar preparation

Cement mortar components were mixed according to the ASTM C305 standard. The cement to sand ratio of 1:2.75 and the water to binder ratio of 0.485 were prepared. MWCNTs were added with the amounts of 0.05, 0.1 and 0.15% by weight of cement. Table 3 shows classification of samples along with the materials composing the mortar. It is necessary to mention that the amounts of MWCNTs and superplasticizer are presented as a percentage of cement. In order to mix MWCNTs uniformly, they were mixed with water and the superplasticizer using an ultrasonic probe generator with 400 W power for 30 min. Then, the fresh mortar was added in oiled molds and cured for 24 h. Finally, all the specimens were demolded and cured in water at room temperature (25 °C).

### 2.3. Mortar characterization

#### 2.3.1. Mechanical strength

Three 50 × 50 × 50 mm specimens were tested for determination of the compressive strength according to the ASTM C109. The measurements were carried out after 28 days of wet curing. Also after 28 days of wet curing, a series of specimens kept in the laboratory conditions (20 ± 2 °C with 60% moisture content) up to the ages of 90 days to acquire the most of their strength and then tested. In a similar manner, the tensile strength and flexural strength of the standard specimens were also determined according to the ASTM C190 and ASTM C348, respectively, at ages 28 and 90 days.

#### 2.3.2. Measurements at elevated temperature

The measurements were performed at 20 °C, as well as after exposure to 200, 400, 600 and 800 °C. After 28 days of wet curing, the specimens were kept in the laboratory conditions (20 ± 2 °C with 60% moisture content) for 90 days to acquire the maximum strength. The specimens were heated at a heating rate of 20 °C/min in an electric furnace until reach the specific temperatures. Then, the samples were kept inside the furnace for 90 min under the temperature control; finally, after being exposed to elevated temperatures, the samples were cooled for 24 h inside the furnace. The weight loss and residual compressive strength were then determined.

#### 2.3.3. Petrography

To better understand the microstructure of the samples, optical light microscopy was employed. The benefits of optical light microscope analysis included viewing large areas of thin sections and allowing for the phase identification of the microstructure of samples [29]. To this end, a thin fraction of the sample was needed, so the thin sections of the samples were prepared at room temperature and exposed to the temperatures of 400, 600 and 800 °C. Petrography test was also carried out at 200 °C and since there was not any significant difference between petrographic images at 20 °C, they are not shown here. The areas for analyses were randomly selected. The cross polarized light with 20×, 40×, and 20× magnifications for the samples were utilized at 20, 400, and 600 °C, respectively. A 100× magnification plane polarized light was employed for the specimen at 800 °C. The MIP software was used for the calculation of the area and circularity of pores for the samples at room temperature. The units of area and circularity were $\mu m^2$ and percentage, respectively.