

## **O 127. COMPARISON OF FIBER REINFORCED AND CARBON NANOTUBE MODIFIED CEMENT MORTARS**

Oğuzhan Öztürk\*<sup>1</sup>, Ülkü Sultan Keskin<sup>1</sup>

Department of Civil Engineering, Konya Technical University, Konya, Turkey

*E-mail: oozturk@ktun.edu.tr, uskeskin@ktun.edu.tr*

**ABSTRACT:** Brittleness of concrete has been tailored to higher bending attribute thanks to reinforcement elements which mostly include steel bars and several types of fibers. Inclusion of fibers into cementitious matrix has become a well-known practice in terms of improving engineering properties. Recent developments in nanotechnology products have led to the utilization of nano-scale materials in cementitious composites for different purposes. Among nanomaterials, carbon nanotubes (CNT) possess superior properties for the benefit of the mechanical properties compared to conventional additives. Taking into consideration the fact that there is an likeness between the micro-scale and bulk structure of the material, uniting both scales in an engineering manner is significant especially in the use of nanomaterials. In this paper, researchers performed an experimental study on the reinforcing of cement-based materials. To do this, CNTs were incorporated homogeneously in cement mortars. Also, conventional fiber reinforced cement mortars were produced by using polymeric fibers together with specimens that do not contain any reinforcing elements as reference. Produced specimens were tested under compression and flexural loadings. Results of each mixture were discussed in terms of basic engineering properties taking into account of microstructural investigations.

*Keywords: Nanomaterials, sustainability, fibers, cement mortars, mechanical properties*

### **1. INTRODUCTION**

Cementitious materials are brittle due to low strength at tension zone and inadequate in strain capacity. Homogeneously distributed fibers serve the purpose of eliminating the brittleness of concrete and its weak resistance to crack initiation and progress. Improved tensile strength, ductility and toughness can be obtained owing to fiber inclusion into cement-based materials. (Vandewalle, 2007; Jamshidi and Karimi; 2010; Yao et al. 2003; Öztürk et al. 2018). Favorable mechanical properties can be gained by hindering or controlling the different crack patterns by means of metallic or polymeric fibers in concrete mixture design. Many studies can be found in the literature with the intent of improving the ductility and toughness of cementitious composites by utilizing different fibers (Brandt 2009). Since fiber reinforced concrete comprises different stages of crack formation from micro-crack occurrence to propagation of a macro-crack (Banthia and Nandakumar 2009; Ahmed et al. 2003), cementitious composites can be tailored to advanced properties approximately in the given production cost by combination of different fibers (considering type, ratio and amount) as reinforcement.

Beside conventional reinforcing elements such as polymeric fibers, limited attempts to date have been presented in incorporating nanoscale reinforcement agents in cementitious material systems. Carbon-based nanomaterials have proved to significantly increase the mechanical performance of cement-based materials due to their unique ability to fill pores, physically supporting the hydration process and effectively bridging micro / nano-cracks under loadings (Öztürk et al. 2018; Öztürk, 2015).

In this study, different fiber reinforced cement-based materials have been developed by the incorporation of polymeric fibers and nanoscale materials into cement mortars. The compressive and flexural strengths of cement mortars reinforced by the carbon nanotubes (CNTs) and microscale (polymeric) fibers have been investigated at 28 days. Furthermore, microstructural analysis has been conducted to ensure the findings of abovementioned properties.

## 2. EXPERIMENTAL DETAILS

### 2.1. Materials

In the preparation of specimens, Portland cement (PC) Type I CEM-42.5/R, similar to ASTM Type I, was used as a hydraulic binder. Specific gravity and Blaine fineness were 3.1 and 325 m<sup>2</sup>/kg, respectively. Physical properties and chemical composition of PC was presented in Table 1. Standard silica sand with a maximum aggregate size of 2 mm was used in conformity with CEN (CEN 196-1, 2009). The particle size distribution of the sand was within limits of Table 2. Polycarboxylic ether-based high-range water reducing admixture (HRWRA) was used to obtain similar flow for each mixture.

**Table 1.** Physical and chemical properties of PC

Chemical composition, %	PC
CaO	61.12
SiO <sub>2</sub>	21.63
Al <sub>2</sub> O <sub>3</sub>	5.12
Fe <sub>2</sub> O <sub>3</sub>	3.45
MgO	2.41
SO <sub>3</sub>	2.39
K <sub>2</sub> O	0.69
Na <sub>2</sub> O	0.22
SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub>	30.2
Specific gravity	3.10
Blaine fineness (cm <sup>2</sup> g <sup>-1</sup> )	3250

**Table 2.** Particle size distribution of the silica sand in mortars

Particle size (mm)	Cumulative retained sand (%)
2.00	0
1.60	7 ± 5
1.00	33 ± 5
0.50	67 ± 5
0.16	87 ± 5
0.08	99 ± 1

Polymer type synthetic fibers with lengths of 6 mm, diameters of 27 μ, specific gravity 1.14, and tensile strength of 962 MPa was used to the reinforce cementitious system. The other reinforcing element was carbon nanotubes, 20-30 nm in diameter, 10-30 μm in length and had a surface area of more than 200 m<sup>2</sup>/g with purity greater than 90%. Polymeric fibers (PF) and carbon nanotubes (CNTs) were given in Figure 1.



**Figure 1.** Synthetic fibers in a bundle and randomly distributed form (at left) SEM micrograph image of CNT (at right)

## 2.2. Preparation of Mixtures

A standard cement mortar fabrication was applied in accordance with the relevant Turkish Standard (TS EN 196-1, 2009). Apart from standard mortar preparation, and polymeric fibers (PF) were added in cement mortars. To start with, water was put into mortar mixer together with the cement. Mixing started for 30 seconds in 62 round per minute (rpm). Then, silica sand was gradually added into the mixer in 30 seconds and mixing continued for another 90 seconds in 125 rpm. PFs were gradually added into the mixer tank during 60 seconds for relevant mixtures. Together with fiber addition, HRWRA was used to provide similar consistency (flow diameter  $20\pm 1$ ) for each mixture.

A different mixing procedure was applied for CNT bearing cement mortars. Since CNTs tend to agglomerate due to very high surface area ( $200 \text{ m}^2/\text{g}$ ) it requires a special distribution process to achieve uniformly dispersed CNTs in cement mortars (Öztürk, 2015). To address this issue, a separate production process for CNT modified cement mortars was followed. CNTs were first mixed with an entire amount of mixing water and HRWRA with another blender for 15 minutes at 3000 rpm. After that dry materials were mixed in a similar way with the former procedure. The separately prepared solution was then gradually to raw materials and additional mixing continued for 10 minutes at 300 rpm. Detailed mixing method of CNTs into cement-based materials can be found in the former study of corresponding author (Öztürk, 2015). The preparation of specimens can be seen in Figure 2. A total of 3 mixtures were molded to prismatic specimens with the dimension of  $40*40*160 \text{ cm}$ . Three specimens were fabricated for each type of mixture. After 24 hours, mortars were taken from molds and kept in thermostat controlled curing tank for 27 days in lime-saturated water at  $20\pm 2 \text{ C}$ . Ingredients of the total 3 mixtures were given in Table 3 to be easily followed throughout the study.



**Figure 2.** Preparing CNT solution (at left) and CNT incorporated cement mortars (at right)

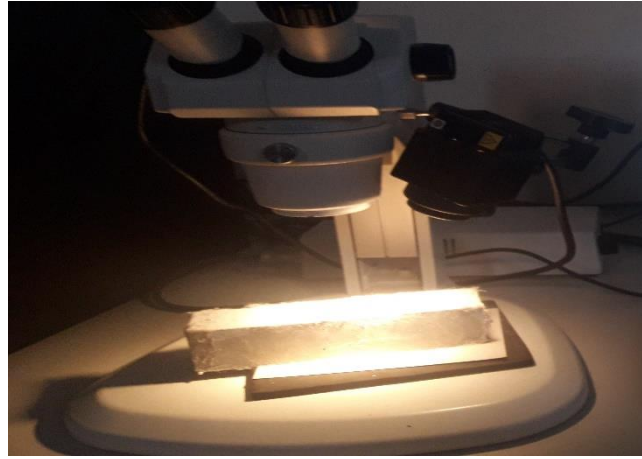
**Table 3.** Ingredients of the mixtures by weight (gr)

Mix ID	Cement	Fly ash	Water	w/b ratio	Sand	PFs	CNTs
M-Ref.	450	-	225	0.5	1350	-	-
M-PF	450	-	225	0.5	1350	1%	-
M-CNT	450	-	225	0.5	1350	-	0.5%

## 2.3. Experiments

### 2.3.1. Microstructural analysis

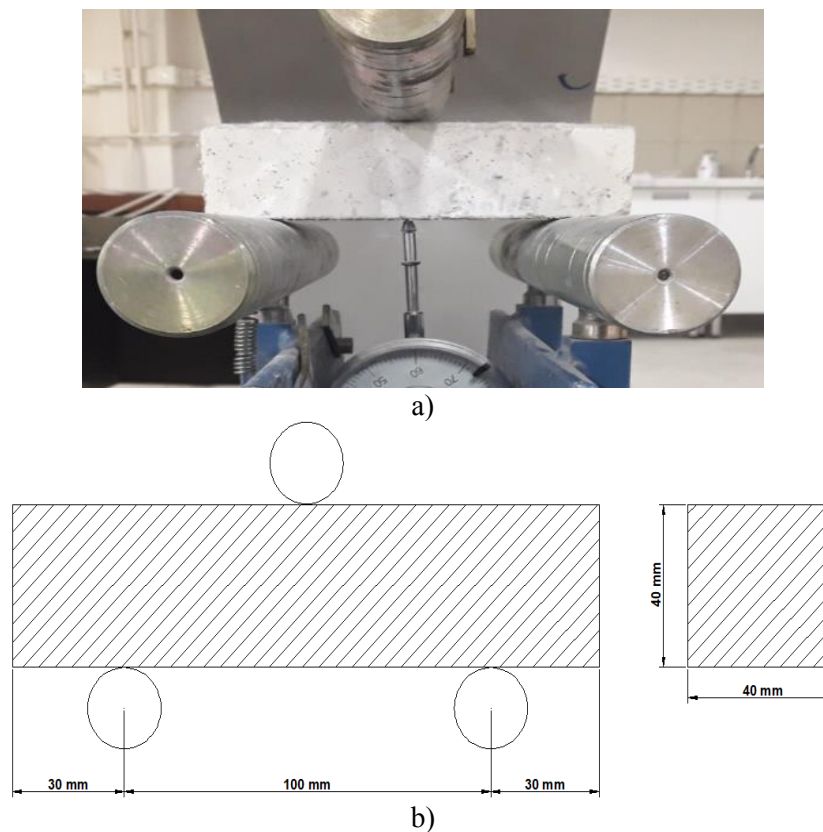
Microstructural analysis was performed to confirm the physical structure of cement mortars at 28 days as a validation of mechanical results. To this end, microscope analysis was used as given in Figure 3.



**Figure 3.** Microstructural investigations of specimens

### 2.3.2. Mechanical Properties

Flexural and compressive strength of each mixture was evaluated in accordance with TS-EN 1015-11 (TS-EN 1015-11, 2000). Compressive strength was determined on each half of 160 x 40 x 40 mm prism specimen by uniaxial loading at 2400 N/second. The flexural strength of mortars was determined by three-point loading of a 160 x 40 x 40 mm prism specimen (Figure 4-a). The loading rate was applied at 50 N/second. Specimens were placed on supports of the testing machine as details were given in Figure 4-b. Both mechanical tests were carried out for 28 days-old specimens.

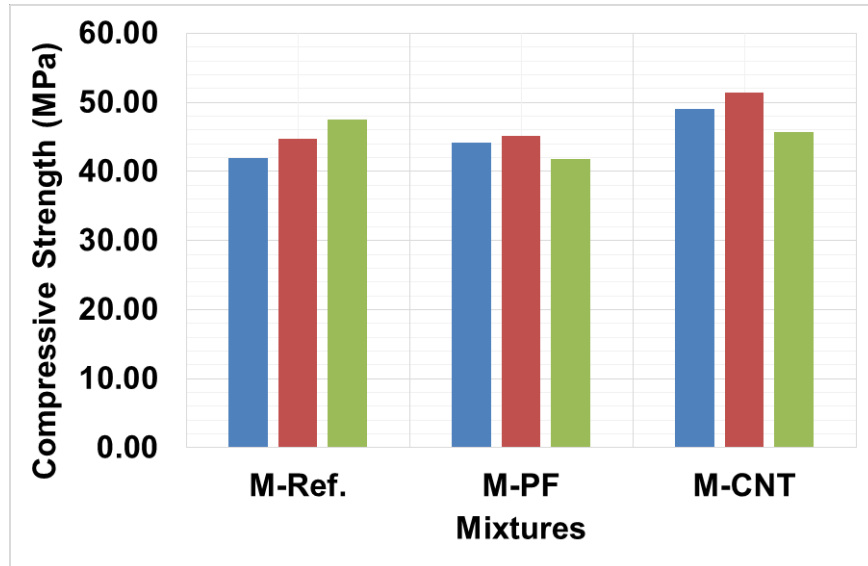


**Figure 4.** Flexural tests (a) schematic details for flexural tests (b)

### 3. RESULTS and DISCUSSIONS

#### 3.1. Compression Tests

Mechanical properties of 3 different cement mortars were determined. Experimental results of basic mechanical properties made with evaluating compression strength were given in Figure 5.

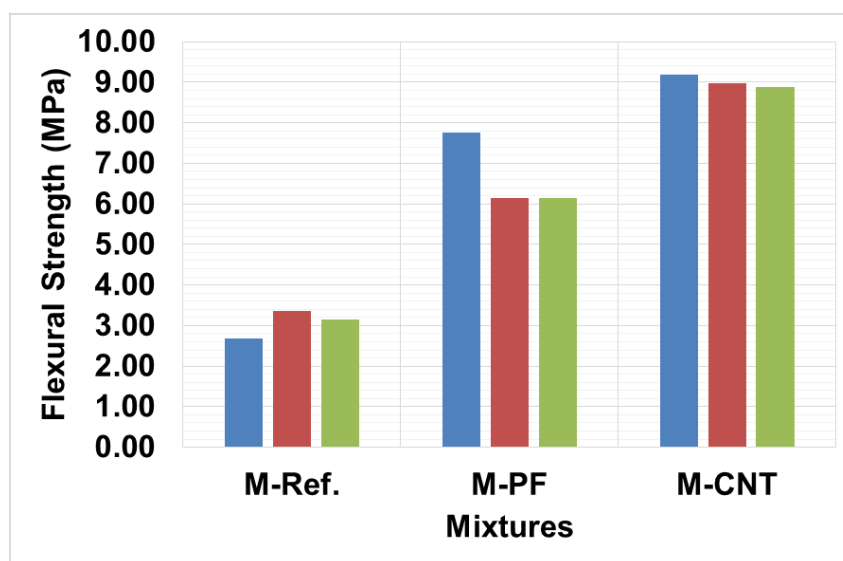


**Figure 5.** Compressive strength of specimens at 28 days

As given in Figure 5, the average compressive strengths of M-PF mixture (43.7 MPa) were comparable with the M-Ref mixture (44.8 MPa). The negligible reduction in the average compressive strength of M-PF mixture compared to the reference mixture can be attributed to entrapped air due to the inclusion of polymeric fibers into mixtures. CNT incorporated mixtures exhibited 8.8% higher compressive strength than M-Ref specimens. The higher compressive strength of M-CNT specimens can be related to higher specific surface areas of CNT particles, which may have provided more nucleation sites with filler effects and flaw-bridging effect at nano/scale.

#### 3.2. Flexural Tests

Flexural strength results of different mixtures under 3-point bending tests were illustrated in Figure 6.



**Figure 6.** Flexural strength of specimens at 28 days

As can be clearly inferred from Figure 6, the average flexural strength of M-PF specimens was higher (119%) than control specimens. Also, the average flexural strength of CNT based mixtures was 195.4% higher than reference mixture. Compared to compressive strength, the inclusion of fibers enhanced the properties of M-PF under flexural loadings. However, the homogeneous dispersion of CNT into cement-based materials was more promising in terms of flexural strength. This result can be ascribed to the capability of CNT in the arresting of crack growth. CNTs were probably likely to act as internal pore and/or microcrack bridging reinforcing elements. Superior characteristic properties of CNTs may have played a significant role in modifying pre- and post-microcracking behavior thus higher bending strengths were obtained. Both in compressive and flexural behavior of CNT based mortars were stronger in packing through densification of matrices so that improved particle size distribution was achieved.

### **3.3 Microstructural Tests**

Microstructural tests were conducted on  $1 \times 1 \times 1$  cm sliced specimens by using a microscope. Microstructural analysis confirms that reduction of compressive strength in M-PF mixture compared to reference specimens is due to flocculation of polymeric fibers (Figure 7). Although the reduction in compressive strength is negligible, different dispersion methods for polymeric fibers can be further investigated to increase the mechanical properties.



**Figure 7.** Flocculation of polymeric fibers in mortars

Another microstructural finding suggests that incorporation of polymeric fibers led to entrapped air voids as can be clearly seen in Figure 8. The air voids on the surface of the sliced specimen by  $1 \times 1 \times 1$  cm (Figure 8) support that entrapped air was responsible for the reduction in compressive strength due to decreased cross-sectional area.



**Figure 8.** Entrapped air voids due to the inclusion of polymeric fibers

#### 4. CONCLUSIONS

An experimental study was undertaken to evaluate the mechanical properties of polymeric fibers and CNT incorporated specimens. After determining the compressive and flexural strength, the microstructural analysis was performed on mortars. The following conclusions were reached.

- The addition of polymeric fibers caused a negligible reduction in compressive strength although polymeric fibers triggered higher flexural strength thanks to the bridging of cracks thereby retarding post-cracking. Dispersion method of polymeric fibers can be improved to obtain higher compressive strength. However, the average compressive strength of M-PF specimens was comparable with the reference specimens.
- CNT based mortars were promising in compressive strength compared to M-PF and M-Ref. specimens. The reason can be related to the seeding effect of CNTs so that dense microstructure assisted to increment of compressive strength. Also, CNT based mortars were clearly better in flexural strength compared to the other two mixtures. It can be said that CNTs are highly preferable to improve mechanical properties provided that homogeneous dispersion is conducted.
- Microstructural analysis supports the abovementioned findings that polymeric fibers slightly reduced the compressive strength due to non-uniform orientation in mixtures. However, this behavior can be accepted as tolerable since the results are comparable. Also, despite delaying crack growth, polymeric fibers may lead to entrapping air in mortars as microstructural analysis suggests.

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