

Influence of Carbon Nanotubes on Building Materials

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Abstract: In this study, a modified solid-phase pyrolysis procedure was used to create multi-walled carbon nanotubes (MWCNTs). Using ultrasonic energy without and with surfactant for two distinct sonication durations (15 min and 40 min), the MWCNTs are successfully disseminated. The impact of MWCNT concentration (0.001, 0.01, 0.05, and 0.1 wt%) on the compressive strengths of cement mortars has been explored in the current work. At ages 7 and 28, compression tests were performed on an automated pressure machine (C089) using a 0.5 kN/s loading rate. It is demonstrated that the ideal concentration of nanotubes does not exist for 15 minutes of sonication, but the optimal concentration for 40 minutes of sonication, both with and without surfactant, is 0.01%. Additionally, the specimen's strength throughout 7 days of hardening rose by 13% in the absence of surfactants and by 19.5% in their presence. After 28 days of curing, the compressive strength improved by 6.3% and 13.8%, respectively.

Keywords: carbon nanotubes; sonication time; surfactant; compressive strength; cement mortar

1. Introduction

The characteristics of cement composites, such as their adaptability in terms of architecture, good mechanical properties, and durability, have been continuously improving as a result of the rising demand for high-performance cement composites [1].

To that aim, numerous industrial wastes [4,5], agricultural wastes [2,3], natural minerals [6,7], and synthetic materials [8] have all been effectively included in the literature. These binders improved the mechanical, microstructural, shrinkage, fresh, and durability qualities of building materials. Carbon nanotubes (CNTs) are an example of a substance that is occasionally utilised in literature. The number of graphene layers in CNTs allows for classification [8]. CNTs may be thought of as seamless cylinders made of one or more graphene roll sheets. Single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs) are the two varieties, which differ based on the quantity of graphene layers [9]. MWCNTs are made up of graphene sheets that have been manifold-wrapped and are organised in hollow tubes that are concentric and have outer

diameters between 2 and 100 nm [10]. The physical and mechanical properties of MWCNTs include high modulus of elasticity (1 TPa), extraordinary tensile strength (65-93 GPa), great thermal conductivity (two times that of a diamond), high aspect ratio (100-2500), and excellent electrical conductivity [11]. The mechanical characteristics of composites might be significantly improved by including CNTs into Portland cement matrices used in civil engineering [12–14]. MWCNTs are employed more commonly than SWCNTs because to their superior reinforcing and reduced manufacturing costs. The flexural and tensile strengths of cement-based mortars were enhanced by 25% by adding 0.25 percent to the cement mass, per Lai and Basem [15]. The authors found an increase in flexural strength of 25% when using very modest concentrations of MWCNTs (0.08%) [16]. MWCNT-reinforced mortars have better engineering qualities than regular mortars, claim Jeevanagoudar et al [17]. In order to get the best compressive strength, it has been shown that 0.4 percent is the ideal concentration of MWCNT.

One of the crucial elements in the creation of improved cement-based composite materials is the

appropriate dispersion of CNTs. Mechanical and chemical dispersion of nanotubes are the two basic approaches [18,19]. In this study, mechanical methods, particularly ultrasonication, have been used to disperse nanotubes. It is important to note that a variety of variables, including time, sonicator type, energy, temperature, and MWCNT characteristics, affect how well MWCNTs disperse during ultrasonication [20–23]. MWCNTs have a propensity for agglomeration because of their high surface energy, which might result in the formation of weak zones in the finished product. The effectiveness of sonication is improved by the use of surfactants, which leads to a more uniform dispersion of MWCNTs in cementitious matrices [24]. Further research is also needed to determine how varying MWCNT concentrations, the length of sonication, and the usage of surfactants affect the mechanical characteristics of cement-based materials, notably cement mortar.

The effects of MWCNT content, sonication time, and surfactant usage on the compressive strength of cement-based mortar were examined in the current work.

2. Experiment

2.1. Materials

As a binder in the mortars utilised in this study, regular Portland cement 52.5 (GOST 31108-2020), which is available at the Araratcement Factory in Yerevan, Armenia, has been employed. The solid-phase pyrolysis of cobalt phthalocyanine was modified to create the short MWCNTs (Figure 1). Using pyrolysis analogues of Ni and Fe phthalocyanines, the pyrolysis was conducted in a closed quartz ampoule for 30 minutes at a temperature of 900 C. Table 1 displays the physical characteristics and chemical makeup of the used cement (GOST EN 196-1-2002, 196-2-2002, and 196-3-2002), whereas Table 2 displays the physical characteristics of the utilised sand.

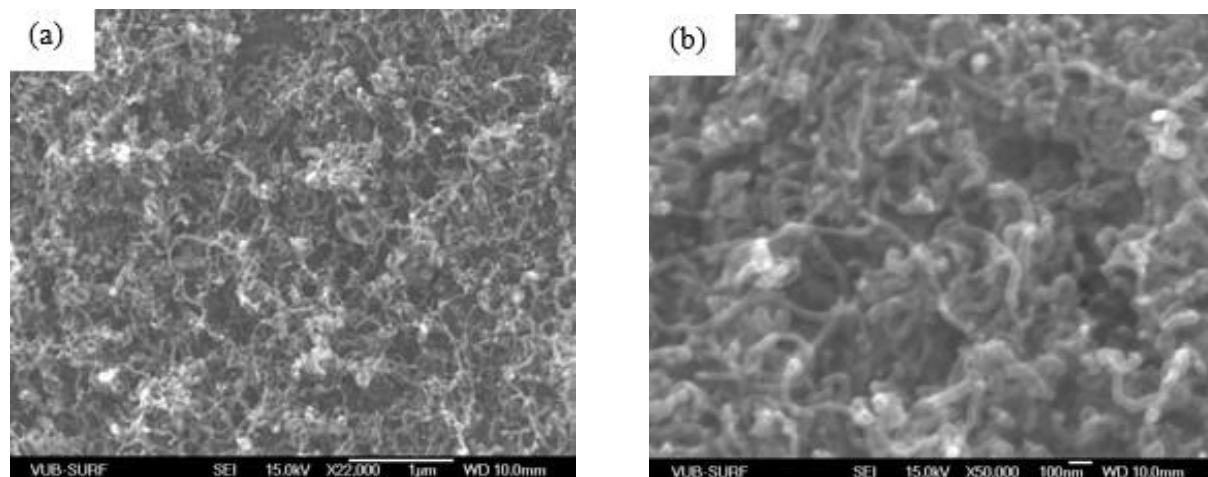


Figure 1. Scanning electron microscope (SEM) images (a) $\times 22,000$ (b) $\times 50,000$ of MWCNTs.

Table 1. Physical properties and chemical composition of cement.

Characteristics	Days	Results Obtained
Standard consistency (%)	-	31
Specific gravity (g/cm^3)	-	3.1
Blain's fineness (m^2/kg)	-	354.8
Compressive strength (MPa)	3 days	23
	7 days	38
	(EN 196-1)	
	28 days	52
	Initial	60

Setting time (min)		n)					330	
		Final						
Chemical composition of cement (wt.%)								
Al ₂ O ₃	SiO ₂	Loss of Fe ₂ O ₃	CaO	MgO	SO ₃	Ignition	Insol. Resid.	Free CaO
3.21	23.2	1.25	57.5	5.1	2.9	3.7	2.1	1.13

Table 2. Physical properties of sand and MWCNTs.

Zone	Fineness	Specific	Bulk Density in	Bulk Density in
Sand	Modulus	Gravity	Compact State (kg/m ³)	State (kg/m ³)
II	2.43	2.17	1829	1609
Outer diameter				
MWCNTS	Length < 1 μm		Purity > 90%	
40–50 nm				

2.2. Dispersion of MWCNTs

MWCNT dispersion has been documented by several researchers using a variety of methods. In this experiment, MWCNTs were added to water in the right quantity and continually agitated to achieve adequate mixing. We evaluated two distinct sonication durations (15 and 40 min). The UP400S ultrasonic device is used to sonicate materials at ambient temperature. To improve the effectiveness of the MWCNT dispersion procedure in the water in the current experiment, DISPERBYK 199 was also applied. Similar steps were taken for solutions containing varied weight percentages of MWCNT (0.001, 0.01, 0.05, and 0.1).

2.3. Mixing and Sample Preparation

The cement to sand ratio employed in the current job was 1:4, and the water to cement ratio was 0.47. The MWCNTS/water combination was

introduced after the cement and sand had been mixed for 2.0 minutes in an E095 Mortar mixer from Matest in Treviolo, Italy. The moulds have dimensions of 40 mm by 40 mm by 160 mm. A vibration machine (C278 Vibrating table, Matest, Treviolo, Italy) was used to compress the mortar for 30 seconds. Similar to this, a series of mortars with varying MWCNT contents—0.001%, 0.01%, 0.05%, and 0.1% by weight of cement—were cast alongside the control mortar. The MWCNTS/water surfactant DISPERBYK-199 (manufactured by business BYK, Wesel, Germany) was also added to the specimens to prepare them. The surfactants were each 4.4, 44, 220, and 440 mg in weight. The samples were demolded after 24 hours, and the mortar sample was submerged in water that was 20 ± 0.2 °C in temperature (Figure 2).

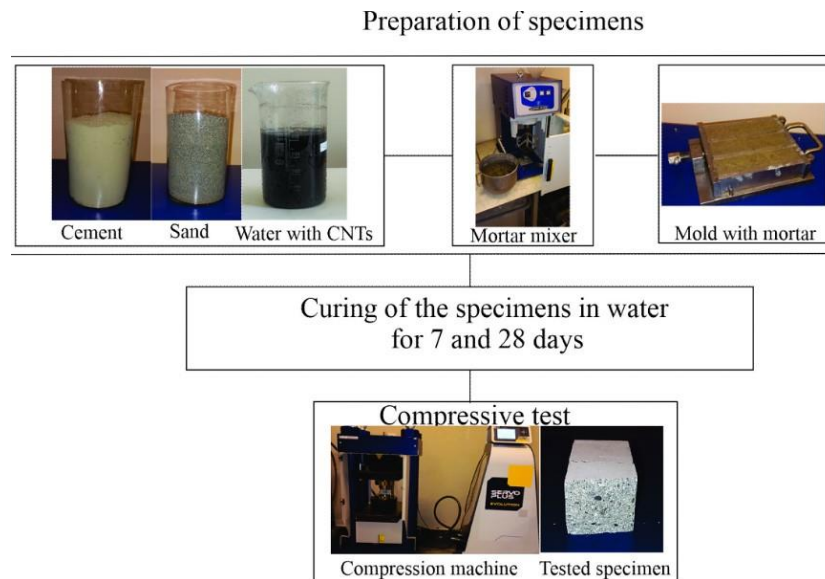


Figure 2. Diagram of the experimental procedure.

2.4. Compressive Strength Testing

The Concrete Compression Machine (Matest, Treviolo, Italy) 2000 kN automated, Servo-Plus Progress (following the norm EN 196-1, and specimen sizes were 40 mm 40 mm) was used to assess the average compressive strength of three cubes of each specimen from each batch. At the ages of 7 days and 28 days, compression tests were performed on an automated pressure machine (C089) (Matest, Treviolo, Italy) with a loading rate of 0.5 kN/s.

3. Results and Discussions

The findings obtained after 15 minutes of sonication without the use of a surfactant are first

shown. The compressive strength of the mortar with various weight percentages of MWCNTs for 7 and 28 days is shown in Figures 3a and 3b, respectively. The findings show that the best value of MWCNT concentration does not exist for either situation of the cement mortar's curing days. Additionally, the cement mortar's compressive strength is lower in the presence of MWCNTs than it is in the reference specimen. This can be explained by the MWCNTs' ineffective dispersion in the water, which will lower the degree of hydration. As a result of increased hydration over time, the results show that compressive strength rises with extended curing time.

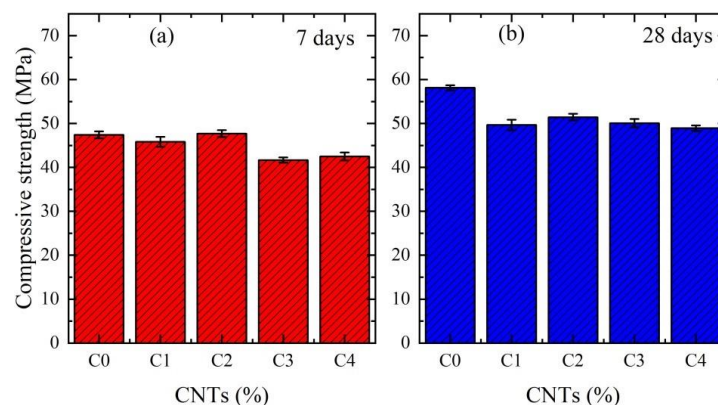


Figure 3. Compressive strength of cement mortars with different wt.% of MWCNTs. The results are for 15 min of sonication time without surfactant. (a) for 7 days, (b) for 28 days.

The compressive strength of the mortar with various weight percentages of MWCNTs for 7 and

28 days is shown in Figures 4a and 4b, respectively. The percentages of MWCNTs that are C0, C1, C2,

C3, and C4 are 0%, 0.001%, 0.01%, 0.05%, and 0.1%, respectively. The outcomes show that as the curing period lengthens, each specimen's compressive strength grows. Over time, this is connected to increasing hydration. Compressive strength rises with the inclusion of nanotubes containing the right amount of MWCNTs. After reaching its peak, the compressive strength begins to decline. In other words, it was possible to determine the ideal nanotube concentration at which the compressive strength achieves its maximum value in both scenarios of curing days. Nanotubes' recommended ideal value is 0.01 weight percent. This is caused by the mortar's selected chemical make-up as well as the nanotubes' mechanical and physical characteristics (structure and size). It is well known that a homogenous distribution of nanoparticles

throughout the volume is required in order for the influence of nanotubes on the physical and mechanical characteristics of cement-based mortars or concretes to be successful. Each time the concentration of nanotubes rises, the degree of homogenous dispersion declines under the same circumstances, which in turn lowers the compressive strength.

The maximum compressive strength was only discovered at 0.4 weight percent of MWCNTs for 28 days of curing (in this work, the ideal values of MWCNTs were not attained for 7 curing days). In another study [17], the scientists looked at fairly high values of nanotube concentration. The findings show that specimens with 7 curing days rose in strength by 13%, whereas specimens with 28 curing days increased in strength by 6.3%.

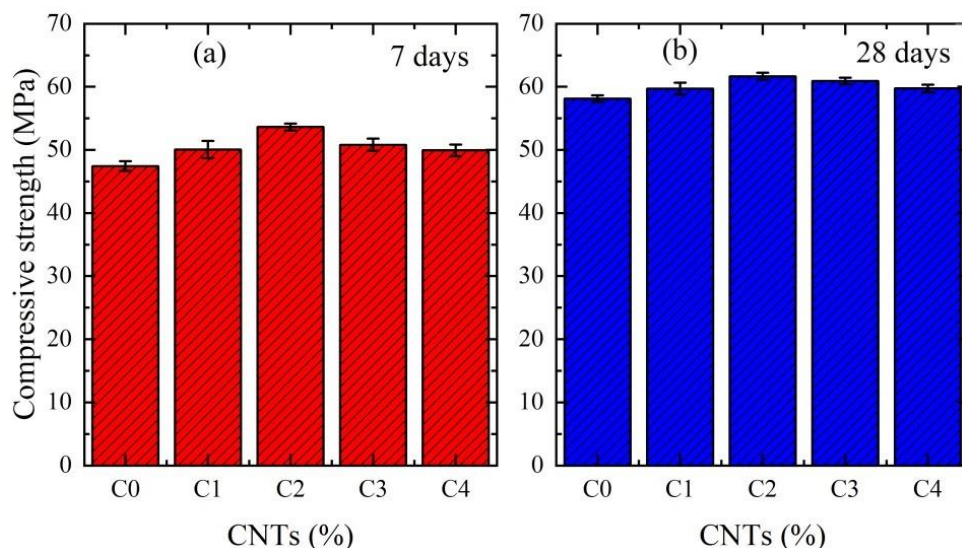


Figure 4. Compressive strength of cement mortars with different wt.% of MWCNTs. The results are for 40 min of ultrasonication time without surfactant. (a) for 7 days (b) for 28 days.

Figures 5a,b illustrate the compressive strength of the mortar after adding different weight percentages of MWCNTs for 7 and 28 days, respectively.

The MWCNT concentration was found to be optimum in both cases of curing days at 0.01 wt%.

The maximum value of compressive strength increased as a result of the MWCNTs' water dispersion process's increased efficacy. In particular, a sample with 7 curing days strengthened by 19.5%, and a sample with 28 curing days strengthened by 13.8%.

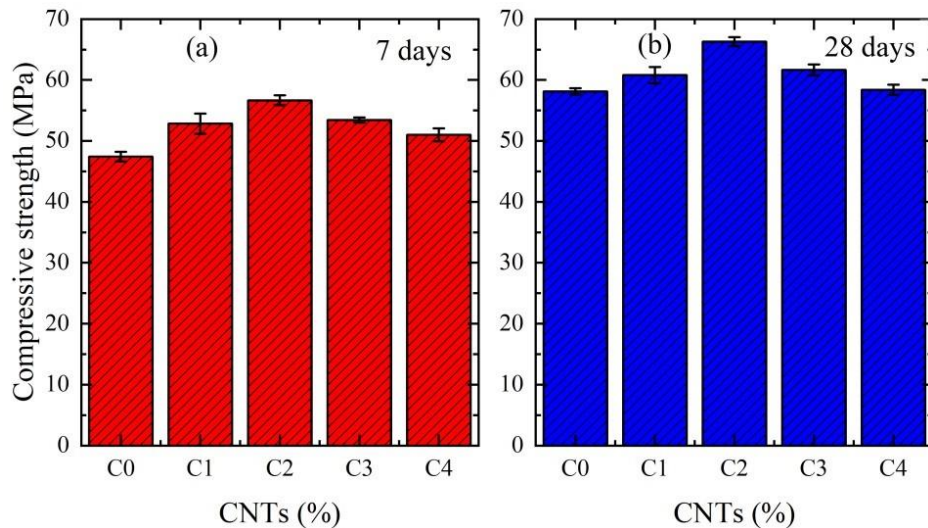


Figure 5. Compressive strength of cement mortars with different wt.% of MWCNTs. The results are for 40 min of ultrasonication time with surfactant. (a) for 7 days (b) for 28 days.

4. Conclusions

The mechanical characteristics, such as compressive strength, of cement mortar with various MWCNT concentrations have been studied in the current research. In specifically, DISPERBYK-199 and the sonication method with and without surfactant were used. The ideal concentrations of MWCNT to achieve maximum compressive strength for 15 minutes of sonication do not exist, however for 40 minutes of sonication, it was discovered to be 0.01%, which is substantially lower than the optimal value attained in previous

investigations. In specifically, the specimen's strength rose by 13% after 7 days of hardening in the absence of surfactants and by 19.5% in their presence. After 28 days of curing, the compressive strength improved by 6.3% and 13.8%, respectively. The ideal mass of surfactants was thus identified, and it was demonstrated that adding them to MWCNTs/water may enhance the maximum value of compressive strength by up to 7.5%.

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