



## Production of Self-Cleaning SiO<sub>2</sub>/CNT Nanoparticles Substituted Cement Mortar

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### KEY WORDS

Cement Mortar, Eco-Friendly, Self-Cleaning, CNTs, nano-silica.

### ABSTRACT

*Manufacturing of building materials with unique properties is one of the most important key-parameters in the development of construction engineering and building materials. The development in the nanomaterials and nanotechnology can be utilized in this field. In this paper, production of eco-friendly, low water absorption and self-cleaning cement mortar reinforced with carbon nanotubes and nano-silica with different weight percentages (0.5%, 1.5%, and 2%) is performed. Results showed prominent improvement of hardness, compression strength and a decrease of water absorption and bending resistance in the reinforced cement mortars. The results show the possibility to control the mechanical properties of the cement mortars with minimal reinforcing agents provides the possibility to design these building materials for versatile applications.*

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### 1. Introduction

Different fields of civil engineering, ranging from building construction to materials design and engineering, are expected to benefit from nanotechnology and its products. Lighter construction materials with enhanced mechanical and thermal properties that are also fire-resistant, soundproofing (or sound absorbing), hydrophobic, self-cleaning, etc. are currently attracting engineers and researchers' interest. These properties are enhanced by using nanomaterials and nanoparticles [1].

The cement mortar properties, its durability and compatibility to certain applications are strongly affected by the addition/substitution with nano-materials. The addition/substitution is usually performed in gravitational or volumetric fractional percentages of the total prepared mortar. This must be monitored

carefully to improve the targeted properties of the mortar compound. Among the targeted properties are hardness, compression, tensile strength, corrosion resistance and fracture toughness [2]. In addition to the effect of composition, the effect of curing time of the cement mortar is an important parameter to be considered in every material characterization.

Carbon nanotubes (CNTs) is one of the most interesting materials that have been considered as additives/substitutes in cement mortars. CNTs have revolutionized the design and application of the construction materials in addition to construction engineering. CNTs unique three-dimensional molecular structure makes it possible to excite some carbon atoms and reconnect them with three other atoms in strong bonds such as diamond. Many researches proved that CNTs is one of the most powerful building materials ever discovered. When carbon fibers are mixed with concrete, the strength and durability of the reinforced concrete increases to about 300 times compare to conventional cement mortars. CNTs were also found to absorb loads in a similar way as rebar does in conventional reinforcement of cement mortars [3]. CNTs were found to be 120 – 140 times more rigid than rebar while its density is one-sixth the density of rebar. Furthermore, CNTs have improved the mechanical properties of the cement mortar giving architects the opportunity to achieve giant structures with competing altitudes [4-6].

Nano-silica, which is also known as condensed silica fume, is one of the most important materials used to replace 7 – 15 % of the total cement material in the cement mortars [7].

Nano-silica was reported to improve the packing characteristics of the cement mortar leading to improved mechanical properties [8-11].

The objective of the current study is to produce eco-friendly and self-cleaning cement mortar reinforced with carbon nanotubes and nano-silica with different weight percentages, which can be used in the manufacturing of building materials. A considerable improvement in the mechanical properties of reinforced cement mortar is obtained.

## 2. Experimental Part

The cement used in this research is United Cement Company- Tasluja-Bazian, (Sulaymaniyah-Iraq -Iq.s 5/1984 Type 2011CEM I 42,5R / BS EN 197-1 / ASTM C150, Type-1). Tables 1 and 2 describes the physical and chemical properties of the used cement.

The sand used in this research has the size of the particle between the sieves measuring 2.63-4.75 mm. Table 3 shows the physical characteristics of the sand.

Figure 1 shows that the sand used in this research (the red line) is within the limits of the Iraqi sand (IQS.NO.45).

Carbon Nanotubes were procured from Nanoshell Company; the nanotubes are multi-walled [12].

Table 4 shows some of the physical properties of nanotubes.

It was within the range of 48.8-55.3 nm where the powder was 48 nm (see Figure 3).

The examination was performed in the Faculty of Science - Ibn Al Haytham.

Figure 2 shows the carbon nanotubes which were used in experiments.

In order to verify the characteristics of the used carbon nanotubes, it was examined using the Atomic Force Microscopy (AFM) device to determine the size of the nanoparticles.

**Table 1: Physical properties of Portland cement**

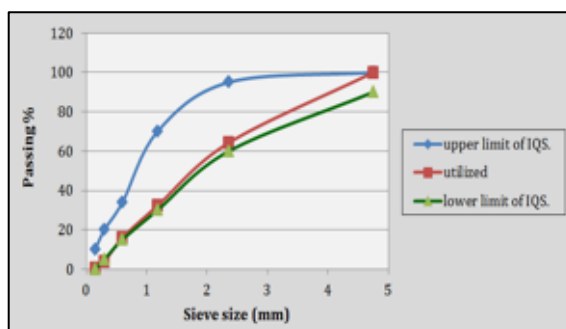
Type of measurement	Value	Limit of IQS.NO.5
Specific surface (m <sup>2</sup> /kg)	376	>230
Setting time (Vicat's method)		
-Initial setting (hrs. : min)	2:05	≥ 45min
-Final setting (hrs. : min)	4:00	≤ 10 hrs.
Compressive Strength of Mortar		
-3 Days	20 MPa	≥ 15
-7 Days	25 MPa	≥ 23
Autoclave (Soundness)	0.12	≤ 0.8

**Table 2: Chemical properties of Portland cement**

Oxidative compounding materials	Symbol	Weight ratio%	Limits of IQS No.5/1984
Lime	CaO	66.11	-
Silica	SiO <sub>2</sub>	21.93	-
Alumina	Al <sub>2</sub> O <sub>3</sub>	4.98	-
Iron oxide	Fe <sub>2</sub> O <sub>3</sub>	3.10	-
Sulphate	SO <sub>3</sub>	2.25	≤ 2.8 %
Magnesia	MgO	2.0	≤ 5 %
Loss of Ignition	L.O.I.	2.39	≤ 4 %
Lime saturation factor	L.S.F.	0.93	0.66-1.02
Insoluble residue	I.R.	1.29	1.5 ≤
Main compound (Bouge eq.)		By weight of cement	
Tricalcium silicate	C3S	58.16%	-
Dicalcium silicate	C2S	18.997	-
Tricalcium aluminate	C3A	7.95	-
Tetra calcium aluminoferrite	C4AF	9.43	-

**Table 3: Physical properties of the sand used**

Sieves (mm)	Passage percentage%	Limits of IQS No. 45/1984/ Zone 1
10	100	100
4.75	100	90-100
2.36	69	60-95
1.18	37.02	30-70
0.60	16.12	15-34
0.30	8.4	5-20
0.15	4.95	0-10

**Figure 1: The sand used according to the standard IQS.NO.45****Table 4: Characteristics of carbon nanotubes**

Physical property	Value/Units
Tensile strength	150 GPa
Young modulus	1200 Gpa
Purity	99%
Density	1.7 gm/cm <sup>3</sup>
Length	(15-30) μm
Amorphous carbon	3%
Volumetric resistance	0.1-0.15 ohm.cm
Diameter	(20-30) nm
Surface area	90 -350 m <sup>2</sup> /g

**Figure 2: Sample of the carbon nanotubes**

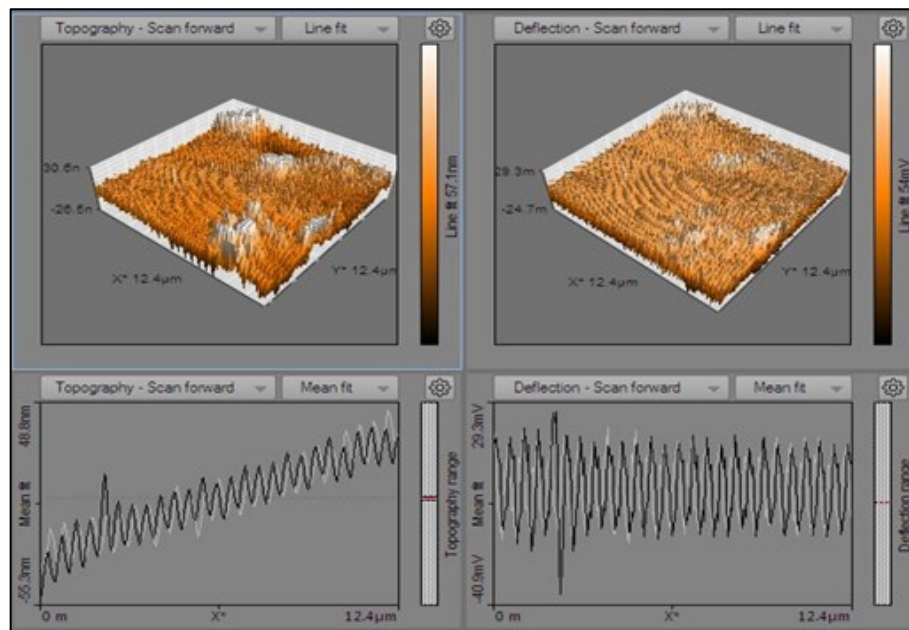


Figure 3: Atomic force microscopy for CNTs

The structural and compositional properties of the CNTs and nano-silica powders were investigated by X-ray diffraction using XRD-6000 Shimadzu diffract meter from Japan. The XRD pattern obtained from the CNTs powders is shown in Figure 4. Some of the physical properties of the nano-silica used in this work are shown in Table 5.

Silica powder was investigated using atomic force microscope to confirm the nanoscale size of the powder. Results showed that the particle size was within 60 nm with an average size of 38 nm as shown in Figure 5, indexed to a single phase of hexagonal structure associated with the crystal structure of CNTs [13]. This figure shows sharp intensity peaks indicating high crystallinity of the material. All peaks could be the results of the XRD examination showed that the high harnesses have high crystalline properties and the crystalline interfaces have a cubic shape as shown in Figure 6.

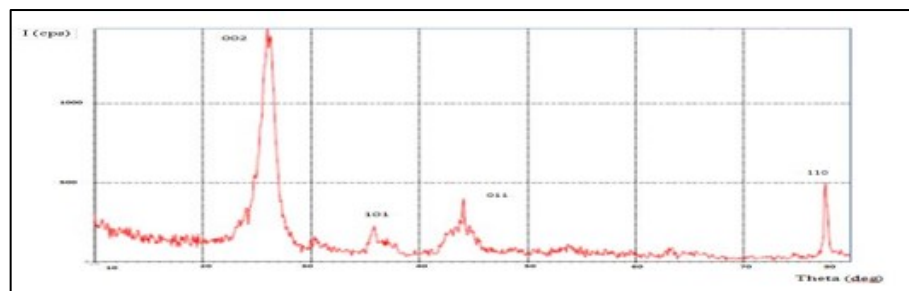


Figure 4: The XRD pattern from the CNTs powder

Table 5: Physical properties of nano-silica

Density (gm/cm <sup>3</sup> )	Surface area (m <sup>2</sup> /g)	Purity (%)	pH
2.7	170-200	99.5	6.6



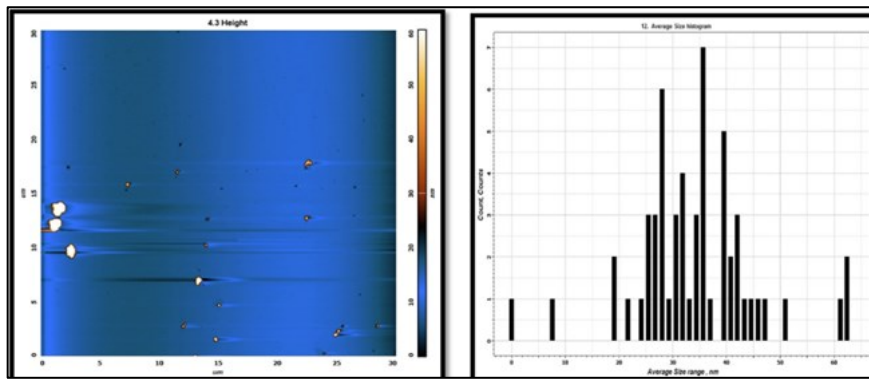


Figure 5: The ATM image of SiO<sub>2</sub> powder and its partial size distribution

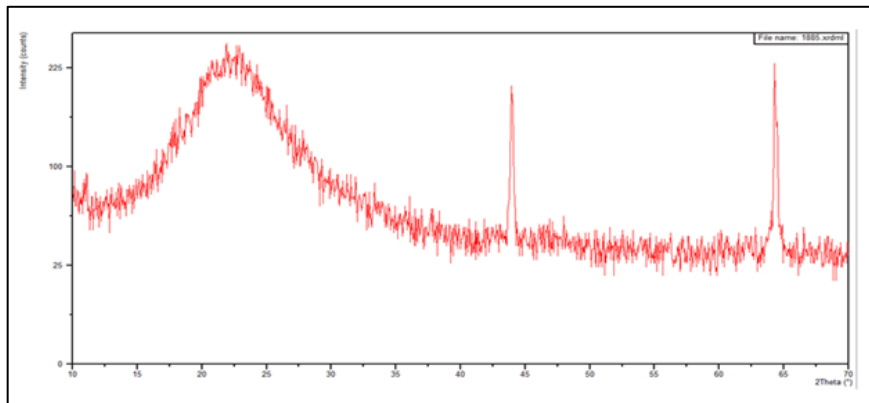


Figure 6: The XRD pattern from the SiO<sub>2</sub>

### 3. Method of Mixing

In order to prepare the specimens of the cement mortars for the compression test, cement was mixed with sand and water was added later. CNTs and nano-silica were added substituting cement component partially at gravitational ratios of 0.5, 1.0, 1.5 and 2%. For bending test specimens, mixing ratios were kept similar to those shown in Table 6. However, due to specimen size requirements the mixing weights are different as shown in Table 7. Table 6 shows the mixing ratios of these constituents. CNTs and nano-silica powders were dispersed in water ultrasonically using ASTM KQ3200 ultrasonicator to reduce aggregation and achieve a homogeneous distribution of the nanoparticles in the cement mortars. Figure 7 shows the samples of CNTs/SiO<sub>2</sub> substituted cement mortar in the compression test. Figure 8 shows the samples of CNTs/SiO<sub>2</sub> substituted cement mortar in the bending test.

Table 6: Mixing ratios of the CNTs/SiO<sub>2</sub> nanomaterials in cement mortars specimen for compression test

Specimen	CNTs/SiO <sub>2</sub> ratio (%)	Weight of CNTs/SiO <sub>2</sub> (g)	Cement weight (g)	Amount of water (ml)
1	0	0	175.95	87.98
2	0.5	0.87	175.15	87.55
3	1	1.8	174.15	87.07
4	1.5	2.63	173.31	86.65
5	2	3.51	172.43	86.22

Table 6: Mixing ratios of the CNTs/SiO<sub>2</sub> nanomaterials in cement mortars specimen for compression test

Specimen	CNTs/SiO <sub>2</sub> ratio (%)	Weight of CNTs/SiO <sub>2</sub> (g)	Cement weight (g)	Amount of water (ml)
1	0	0	400	200
2	0.5	2	398	199
3	1	4	396	198
4	1.5	6	394	197
5	2	8	392	196

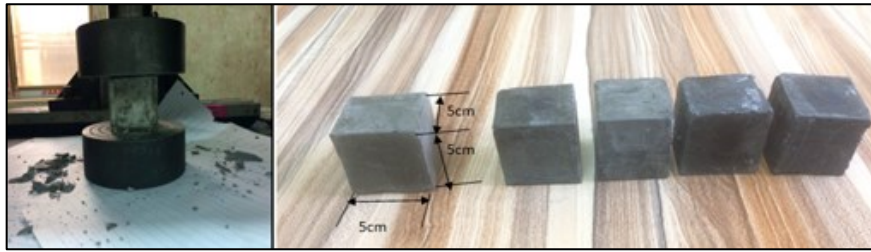


Figure 7: shows the samples of CNTs/SiO<sub>2</sub> Substituted Cement Mortar in the compression test



Figure 8: shows the samples of CNTs/SiO<sub>2</sub> substituted cement mortar in the bending test

#### 4. Results and Discussion

The first step in the preparation the samples is to ensure a good stability for dispersed particles. The ultrasonic cleaner (ASTM KQ3200) was used to disperse the CNT sand nano-silica because of the strong Vander Vals bounds between the nanoparticles and to prevent aggregations occurring. The test was carried out in the Erosion Lab/Materials Engineering Department/ Technological University. The device consists of a metal pan (25\*25\*30 cm).

The nanoparticles were dispersed for a specified time of 45 minutes. Ultrasonic waves are propagated in the material media such as air, water, solids in longitudinal vibrations far from the sound source like sea waves. It has a frequency higher than 20,000 Hz, which is higher than the acoustic sound waves and thus disperses the accumulation of the nanoparticles.

Water absorption was investigated for the cement mortars. Results showed that water absorption decreases with the increase of nano-powders in the specimen suggesting that these materials, having small dimensions, can fill the pores and voids (usually working as absorption centers) in the cement mortar during its preparation. Figure 9 shows water absorption of cement mortars reinforced with CNTs and nano-silica. The result agrees with the result in this source [14].

Hardness test was carried out using ASTM D2240-05 device. The results (Figure 10 a and b) showed that hardness increases with the increase of nano-powders in the cement mortars. This may be attributed the fact that these nanoparticles have inherent hardness that may work as centers of resistance to external mechanical influences leading to overall increase of hardness of the specimen.

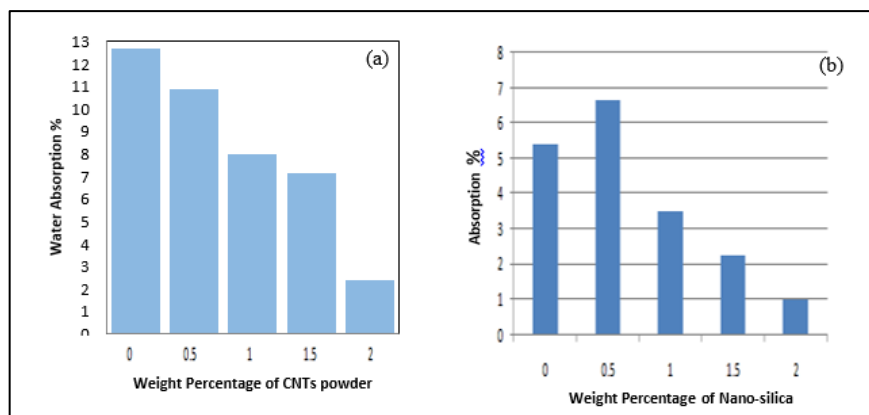


Figure 9: Water absorption of cement mortars reinforced with (a) CNTs (b) nano-silica

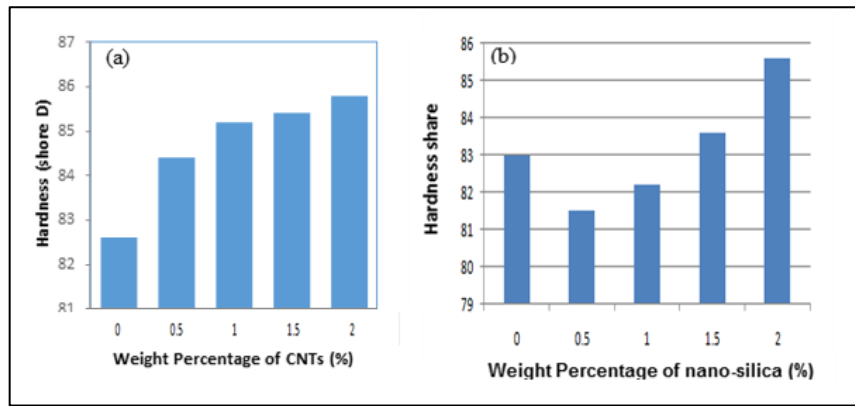


Figure 10: Hardness of cement mortars reinforced with (a) CNTs (b) nano-silica

Compression and bending of cement mortars were measured according to ASTM-A513 test. Samples were prepared as per requirements of each test. Figure 11 shows cement mortars specimens for compression test. Specimens were cubic having dimensions of 5×5×5 cm<sup>3</sup>. Results of compression test showed that this property increases with the increase in the weight percentage of nano-materials in the cement composite (i.e. CNTs and nano-silica). Compression was found to increase from 40 MPa in the pure cement mortar to about 73 MPa and 80 MPa in the CNTs and nano-silica reinforced specimens respectively. These values were measure after curing time of 28 days. Figure 12 shows cement mortar specimens for bending test measurements. Each specimen has a dimension of 4×4×16 cm<sup>3</sup>. Figure shows the bending test results for cement mortars reinforced with CNTs and nano-silica powders respectively. Results showed that bending of specimen decreases with the increase of nanoparticles in the cement mortars. This may be attributed to the fact that while specimen compression and hardness increase, its stiffness and thus resistance to strain decreases. Bending decreases to (1 MPa) and (1.5 MPa) for cement mortars reinforced with 2% weight percentage of CNTs and nano-silica respectively. The results are agreement with the results in this source [15].

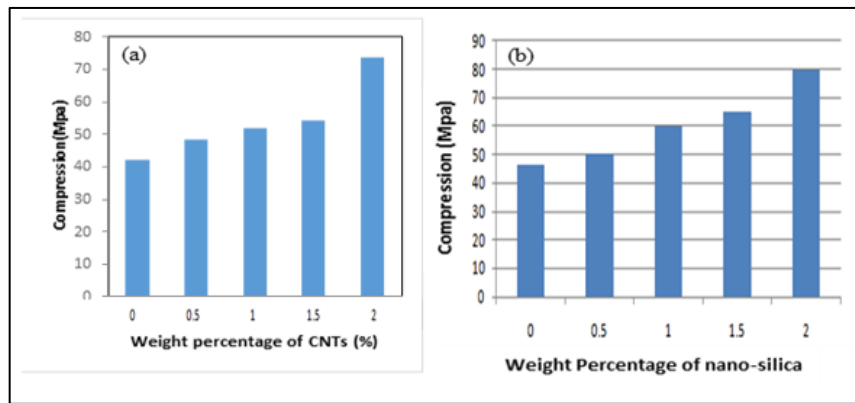


Figure 11: The compression test of (a) CNT cement (b) SiO<sub>2</sub> cement

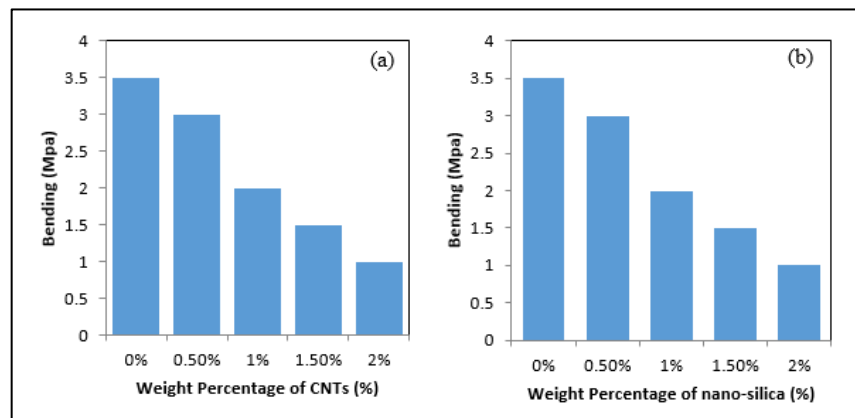


Figure12: Bending of cement mortars reinforced with (a) CNTs (b) nano-silica

## 5. Conclusions

Production of low water absorption reinforced cement mortar with CNTs and nano-silica was reported. It was possible to enhance the mechanical properties of the cement mortar with minimal additions of CNTs and nano-silica powders. Compression strength and hardness increased while bending strength and water absorption decreased with the increase of reinforcing agents in the specimens. The possibility to control the mechanical properties of the cement mortars with minimal reinforcing agents provides the possibility to design these building materials for versatile applications.

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