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Standardizing Protocol for Incorporating Cnts in Concrete

Noha M. Hassan
American University of Sharjah/Department of Industrial Engineering
PO Box 26666, Sharjah, United Arab Emirates
nhussein@aus.edu

Kazi Fattah, Adil Tamimi
American University of Sharjah/Department of Civil Engineering
PO Box 26666, Sharjah, United Arab Emirates
kfattah@aus.edu; atamimi@aus.edu

Abstract **-**Concrete is a core element in the construction industry. Billions of tons of concrete are produced worldwide every year. The growing demand in emerging economics compels the construction industry to embrace new technologies in order to deliver safer, more sustainable and better performing structures. A relatively new field of study is the use of carbon nanotube (CNTs) in concrete to improve its workability, durability and strength in addition to adding new functionalities. Research reveals that the incorporation of nanomaterials may avail concrete, although there are mixed results in the literature within this regard. To take advantage this addition, effective dispersion of the CNTs must be ensured to avoid aggregation of the CNT powder into unreacted sacks that leads to stress concentration in the concrete. Using different protocols to prepare the concrete affects nanomaterial dispersion and thereby concrete properties. This research compares the degree of variability that exists in concrete blocks from CNTs dispersions when prepared using different protocols for the same cement to water ratio. Different protocols were proposed to examine the effect of initial wet mixing of CNTs with water, dry mixing of CNTs with cement, sonication exposure time, whisking, grinding and CNTs concentration. Experiments were performed to physically examine and compare the proposed protocols. Mechanical tests were performed on the prepared blocks to determine its resultant compressive strength. Results revealed which proposed protocol was effective in overcoming the weak bonding and low dispersion. This research clearly illustrates that for the purpose of data comparability, there is a need to provide the exact details of all steps involved in a dispersion protocol and standardize the methodology for preparation for optimum dispersion.

Keywords**:** Carbon NanoTubes (CNTs), dispersion, concrete/ cementitious materials, Compressive strength, Microstructural examinations (SEM).

1. Introduction

Concrete is a common construction material. The properties of which may be predetermined by design, selection of constituent materials and quality control. The constituent materials of concrete are cement, aggregates, water and admixtures (Somayaji, 2001). Ordinary concrete exhibits good compressive strength but weaker properties when subjected to tensile, flexural or shear forces. For this reason, it is often reinforced with materials that possess high tensile or flexural strength. Concrete structures also suffer from crack formation induced by shrinkage, creep, or thermal changes that degrades its performance during service (Kelsall, et al., 2006). Fiber reinforced concrete offers solution to this problem by making the concrete tougher and more durable by incorporating 3-D reinforcement within the concrete.

There is a significant potential of nanotechnology in terms of development of construction and building materials. Nanotechnology refers to the understanding and manipulation of materials at the nanoscale (<100 nm). The properties of the material at this level are governed by the rules of quantum mechanics (Hanus and Harris, 2013). Research reveals that the incorporation of nanomaterial avails concrete by increasing its strength and durability, reducing pollution, enhancing its self-cleaning ability and prolonging its service life through sensing and self-repairing ability (Pacheco-Torgal and Jalali, 2011). High strength concretes often incorporate silica fumes. The main role of this additive is due to its small particle size, which allows it to fill the space between the cement grains and increase the compactness of the concrete (Scrivener and Kirkpatrick, 2008). Vera-Agullo, et al. (2011) confirmed that the use of nanoparticles contributes to higher hydration degree of cementitious compounds as long as higher nanoparticle dispersion can be achieved.

The outstanding mechanical properties of carbon nanotubes (CNTs) highlight them as potential candidates for concrete reinforcement as well. The strength of the CNTs is directly related to the strong C=C bond and the relatively small number of defects present in the tubes. It is said to possess “a hundred times the strength of steel at one sixth of the weight” (Kelsall, et al., 2006). Young modulus is estimated to vary between 1-5 TPa while density is around 2000 Kg/m3. The CNT are characterized by thermal stability up to 2800 0C (Cwirzen, et al., 2009). However, their surfaces have very low friction, so it is very difficult for them to bind together or with the cement matrix material (Scrivener and Kirkpatrick, 2008). In addition, carbon nanotubes are packed together by Van der Waals attraction forces into crystalline ropes during production. These ropes tend to aggregate and result in lack of ability of CNT powder to disperse in aqueous or organic solutions (Cwirzen, et al., 2009). A number of methods have been investigated to improve dispersion and to activate the graphite surface in order to enhance the interfacial interaction through surface functionalization and coating, optimal physical blending, and/or the use of surfactant and other admixtures (Makar, et al., 2005). Tantra, et al. (2014) compared the effect of different protocols on the particle size distribution of TiO2 dispersion and concluded that they represent potential sources of variations, with final particle concentration being the most significant factor. This research will follow a similar methodology to determine the most influential protocol on CNTs dispersion in concrete when preparing sample in an aim to conclude a standardized procedure to be followed during sample preparation.

2. Methods and Materials

2. 1. Materials

Industrial grade multiwall carbon nanotubes were used for the study. The outside diameter varies from 20-40 nm, an inner diameter 5-10 nm and length varying between 10-30 μm, as shown in figure 1. They were purchased from Nanostructured and Amorphous Materials, Inc. The binder mix consisted of Type 1 Portland cement, crushed sand and water.

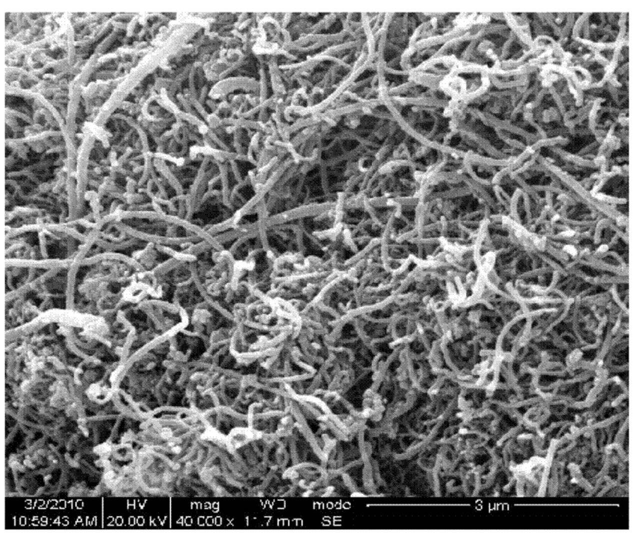

Fig. 1. SEM of Multiwall carbon nanotubes(40000x)

2. 2. Dispersion Protocols

Table 1 summarizes the list of 15 sets of mixtures that were prepared. Several factors were examined including effect of wet versus dry mixing, use of pre-grinded CNTs, Whisking the CNT mixture, effect of sonication exposure time and effect of varying CNTs concentration (0.5 % , 1 %) while maintaining same water to binder ratio. Each set of experiment was repeated three times, standard deviation between the results from the 3 samples was recorded. A water/binder ratio of 0.3 and a sand to cement ratio of 1.3 was used for all experiments.

Table. 1. Protocols examined through different experimental sets.

Set	*wet / dry*	*Sonication time*	*grinded*	*Whisked*	*Solvent*	*days*	*CNT %*
1	control	control	control	control	water	7	***Control- 0% CNTs***
2	wet	***5***	No	No	water	7	0.50
3	wet	***20***	No	No	water	7	0.50
4	wet	5	***Yes***	No	water	7	0.50
5	wet	20	***Yes***	No	water	7	0.50
6	wet	5	No	***Yes***	water	7	0.50
7	wet	20	No	***Yes***	water	7	0.50
8	***dry***	0	No	No	water	7	0.50
9	wet	***5***	No	No	water	7	1.00
10	wet	***20***	No	No	water	7	1.00
11	wet	5	***Yes***	No	water	7	1.00
12	wet	20	***Yes***	No	water	7	1.00
13	wet	5	No	***Yes***	water	7	1.00
14	wet	20	No	***Yes***	water	7	1.00
15	***dry***	0	No	No	water	7	1.00

2. 3. Experimental Procedure

The following procedure was adopted to ensure consistency in sample preparation. Proper weight of sand and cement were measured and mixed together as per the mix design. All the dry material is added to the mixer and mixed for 3-5 minutes. Meanwhile the correct weight of carbon-nanotubes is mixed with water after being grinded as per experimental set up. The CNTs-water mix is then sonicated or whisked then mixed with the dry mix in the mixer for 5-7 minutes to ensure a consistent sample. The concrete paste is then used to fill 1/3 of the mold and placed on the shaker table. Concrete is then compacted while shaking the mold. The process was repeated 3 time until mold is full. After fully filling the mold, all the extra concrete was removed from the top of the mold and the surface was scrapped to smoothen it. 24 hours later, concrete was removed from mold and labelled. Concrete was placed in water for curing, and then crushed by a universal testing machine after 7 days from samples preparation.

3. Results and Discussion

Results from the 15 sets of experiments were investigated in more details. Compressive strength of each of the three replicates was averaged for each experiment and standard deviation was calculated. In case standard deviation between replicates exceeded 3, experiment set was repeated. Standard deviation varied between 0.58- 3 depending on the experiment set.

3. 1. Agglomeration Of Cnts In Water

The dry nanoparticles form large clusters in the range of hundereds of micromemters when added to water, as illustrated in Figure 2. In order to avoid these agglomeration when using nanomaterials, three steps must be followed; wetting, deagglomeration and then stabilization using surfactants and thickners.

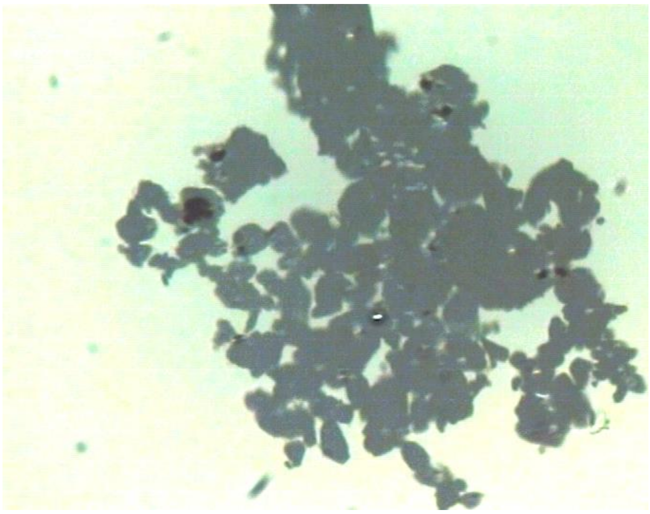

Fig. 2. illustrates the agglomeration of the carbon nanotubes in water forming larger particle sizes. The CNTs particle size varied between 40 μm- 380 μm with an average size of 240 μm.

3. 2. Pre-Grinding The Cnts Versus Whisking The Wet Cnts-Water Mixture

Figure 3 shows the concrete samples compressive strength in case CNTs were grinded before mixing with water or whisked after mixing. Results are compared with control experiment when similar sample was prepared without adding any CNTs. It is clear that the compressive strength did not increase in as the specimens were not grinded or whisked, it exhibits the same compressive strength as that of the control experiment. Both whisking or grinding show similar effects and they are more dominant as the CNTs concentration increase. After seven days the compressive strength of concrete with 1 % CNTs reached almost 1.45 its strength in case no CNTs were added, which is a significant increase in the compressive strength of the material. In case of lower CNs percentage (0.5%) grinding the CNTs has more influence than whisking it after. An increase of strength of 1.26 versus a 1.06 respectively.

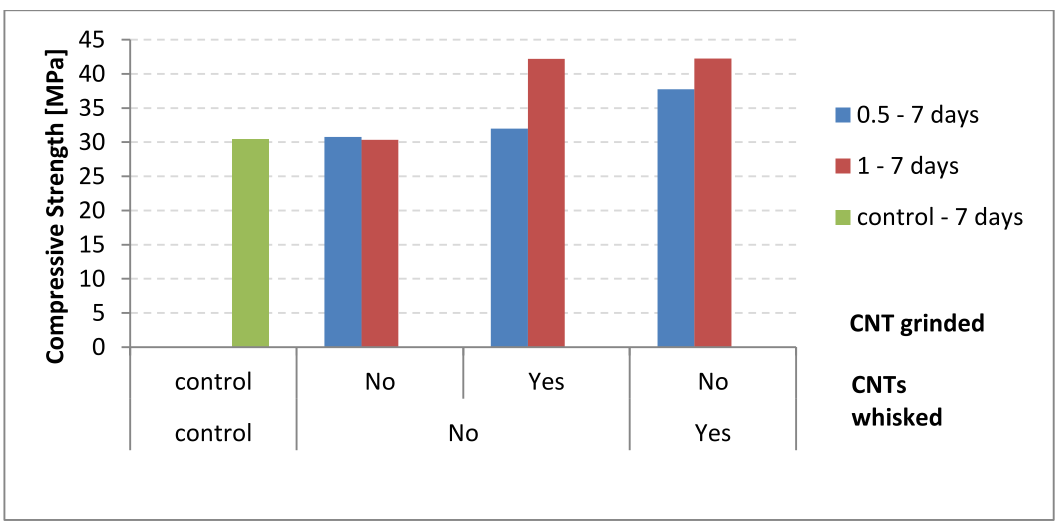

Fig. 3. Pre-grinding CNTs versus whisking the wet CNTs water mixture on compressive strngth of concrete samples

3. 3. Sonication Time Exposure

As seen in Figure 4, an increase in the sonication time results in samples with higher compressive strengths. This could be attributed to the effect of sonication on dispersing the CNTs as well as decreasing the particle size or de-agglomerating the CNTs.

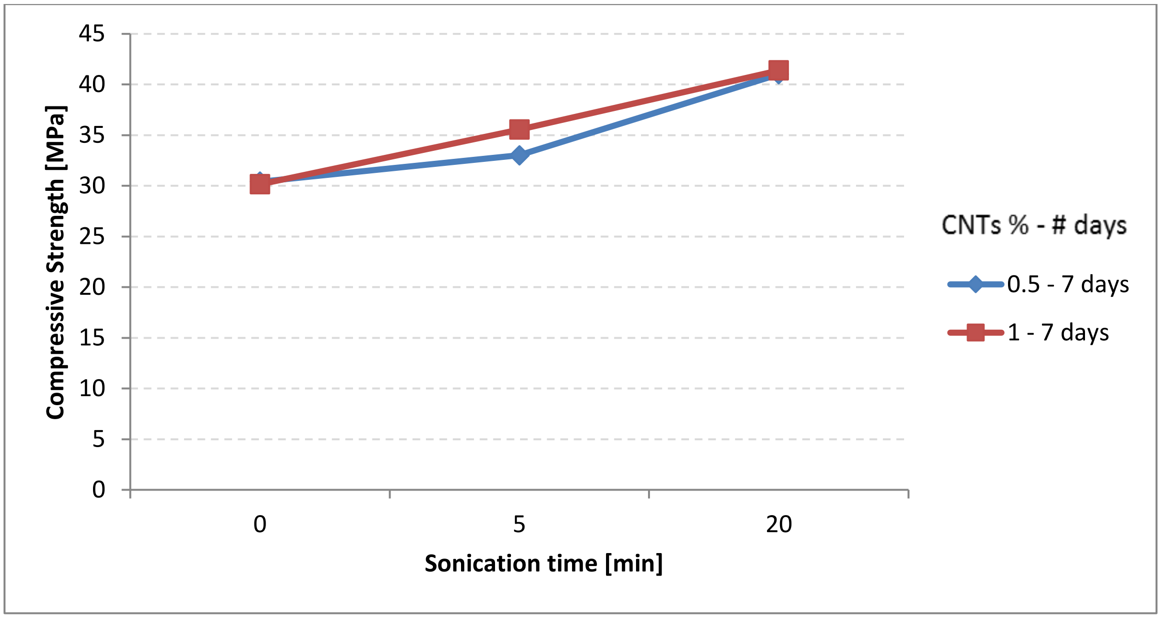

Fig. 4. Effect of sonication time exposure on compressive strngth of concrete samples.

3. 4. Dry Versus Wet Mixing

Even though one of the standard ways to prevent de-agglomeration of the CNTs is through wetting, experimental results reveal otherwise in terms of effect on compressive strength. Figure 5 depicts the effect of pre-wetting with water by mixing CNTs with water before adding it the aggregate mix versus dry mixing the CNTs with the aggregates (sand and gravel) then adding the water at a later stage. As illustrated in Figure 5, dry mixing results in a higher compressive strength samples for all CNTs concentrations examined.

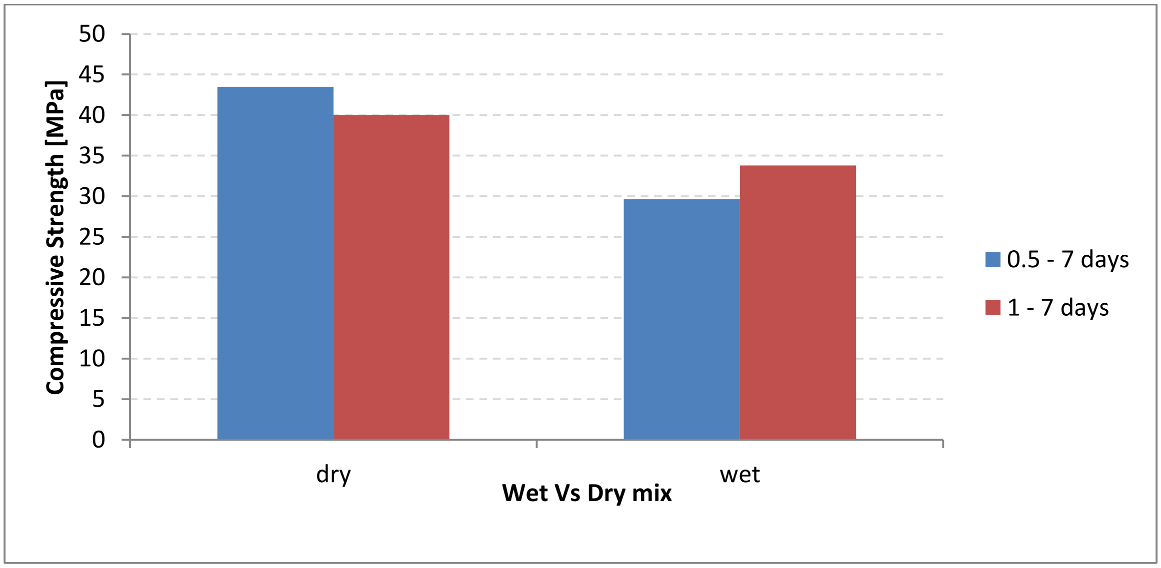

Fig. 5. Effect of preweting versus a dry mix on compressive strngth of concrete samples.

4. Conclusion

Sample preparation procedure has a significant influence on the dispersion mechanism of the CNTs in the concrete mix, thereby greatly affecting its mechanical properties. Results from this research illustrate the necessity to standardize the procedure for sample preparation in order to have a fair picture on the effect of adding CNTs on concrete. Mixed results in literature about the influence of CNTs on the mechanical properties of concrete are very well justified since varying the procedure significantly affects the result. In order to improve the mechanical properties of concrete using CNTs, some suggested procedures included grinding the CNTs to de-agglomerate it, dry mixing the CNTs with the aggregate mix prior to adding water, followed by whisking the wet mix vigorously to ensure proper dispersion of CNTs.

Acknowledgements

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