



ISSN: 0976-3376

Available Online at <http://www.journalajst.com>

ASIAN JOURNAL OF
SCIENCE AND TECHNOLOGY

Asian Journal of Science and Technology
Vol. 08, Issue, 06, pp.4893-4901, June, 2017

RESEARCH ARTICLE

EFFECT OF CARBON NANOTUBE AND NANOFIBER ON MICROCEMENT MORTAR PROPERTIES

^{1,*}Yazdani, N. Ph.D., ²Narasimha Alimeneti, P. E. and ³Tanvir Manzur, Ph.D.

¹Department of Civil Engineering, University of Texas at Arlington, Arlington, Texas 76019, USA

²Bridge Designer, GM2 Associates, Inc. Bridgeport, CT

³Associate Professor, Bangladesh University of Engr. & Technology, Dhaka, Bangladesh

ARTICLE INFO

Article History:

Received 09th March, 2017
Received in revised form
26th April, 2017
Accepted 09th May, 2017
Published online 30th June, 2017

Key words:

Carbon nanotube, carbon nanofiber,
Microcement, Mortar strength,
Flowability, Setting time, Shrinkage.

ABSTRACT

Carbon nanotubes (CNT) and nanofibers (CNF) are among the toughest and stiffest materials discovered to date. Their use in cement composites is not wide spread yet and is of great interest. In this study, some important physical and mechanical properties of microcement mortar with CNT and CNF addition are investigated. Various water cement (w/c) ratios and CNT/CNF dosage rates were explored. The compressive and flexural strengths increased significantly at early ages with CNT/CNF addition. However, decrease in strengths was observed for a number of mixes at 28 days age. CNT dosage rate of 0.05% at 0.4 w/c ratio yielded the best combination for mortar strength. Nanoparticles increased flowability, decreased initial setting time and had no effect on bleeding and shrinkage rates. CNT addition resulted in more consistent effects on mortar properties than CNF addition. Additional investigation is needed to fully understand the potential usage of these nanoparticles in cement composites and remove some of the relevant inconsistencies.

Copyright©2017, Yazdani et al. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

INTRODUCTION

Various forms of reinforcements, such as steel bars, steel fibers and carbon nano particles, are used to reduce the problem of weak tensile strength of cementitious composites. Cement has a complex structure at the nanoscale, and cement hydration is a molecular process that solidifies the cement composites. Introduction of nanoparticles into cement composites can impart beneficial impact at the nano level to increase strength and ensure bridging across micro cracks. The properties of carbon nanoparticles, when imparted to the cement composite, have great potential to enhance the properties of the composites. Such prospective particles are carbon nanotubes (CNT) and carbon nanofibers (CNF). CNT is an allotrope of carbon with tube-shaped material having with length to diameter ratio of up to 132,000,000:1. The chemical bonding of nanotubes is composed entirely of sp² bond that are similar to those for graphite. CNF is the allotrope in the form of tabular microstructure called filament or fibers. Carbon atoms join each other in sp, sp² and sp³ hybridized structures to form stable structures. Table 1 presents some important common properties of CNT and CNF.

The intrinsic mechanical and transport properties of CNT and CNF make them the ultimate carbon fibers. Both are very strong in the axial direction and have both high aspect ratio and strength (Zeng *et al.*, 2004 and Yu *et al.*, 2000). The small density makes them very light material when compared to steel. They show a unique combination of strength, stiffness and tenacity compared to other fiber materials which usually lack one or more of these properties. Moreover, the very small size of these particles and large length to diameter ratios ensure finer scale distribution than that for traditional fibers. Such finer scale distribution can result in more efficient crack bridging at very preliminary stage of crack propagation within the composite matrix. The structure of CNT/CNF also shows extraordinary electrical properties due to symmetry and the unique electronic structure of graphene. They also have good kinetic and optical properties. As a result, wide spread research on CNT/CNF are being carried out in different fields, such as self-sensing sensor (Han and Kwon, 2009), reinforcement in polymer based materials (Marrs *et al.*, 2007 and Coleman *et al.*, 2006) and field emission properties (Parveen *et al.*, 2013 and Parveen *et al.*, 2012). It is well understood that mechanical properties of cement composites depend on the micro structure and mass transfer at nano level. Makar *et al.* (2005) showed that the chemistry and physical behavior of hydration products can be manipulated through nanotechnology. It is, therefore, evident that incorporating nano-scale reinforcement within cement composites has immense potential to develop next

*Corresponding author: Yazdani, N. Ph.D

Department of Civil Engineering, University of Texas at Arlington,
Arlington, Texas 76019, USA

generation high performing construction materials. Li *et al.* (2007) developed pressure-sensitive properties and mechanical properties of CNT-cement composites. The properties of both treated (with sulfuric acid and nitric acid) and the untreated CNT were studied. The surface treated CNT had more effect on pressure sensitive properties, whereas the untreated CNT had more effect on reducing the electrical resistivity. Balaguru and Chong (2008) analyzed the behavior of cementations materials with multi-walled CNT in different concentrations. Cwirzen *et al.* (2008) observed about 10% increase in mortar flexural strength through addition of multi-walled CNT (MWNT). Makar *et al.* (2004) found that the morphology of hydration products could be influenced by incorporating nanotubes. Agullo *et al.* (2009) investigated compressive strength of cement mortar by adding low concentrations of MWNT. Although early high compressive strength was observed as compared to plain mortar, the 28 day compressive strength was less than that of the mortar. Metaxa *et al.* (2009 and 2010) investigated the fracture characteristics and early strain capacity of CNT cement composites. CNT with long length at smaller quantities (0.025 – 0.048%) and short length having higher quantities (0.08%) were found to achieve a good dispersion. In another study, Konsta-Gdoutos *et al.* (2010) examined highly dispersed CNT reinforcement in cement materials. An optimum ratio of surfactant to CNT was found to be 4.0, as it controlled the mechanical properties significantly. Manzur and Yazdani (2010) found that compressive strength of cement composites could be increased through addition of CNT. Two different sizes of untreated commercially available CNT were used in this study. Yazdanbakhsh *et al.* (2009) utilized CNT for the enhancement of mechanical properties of cementations materials.

strength of CNT reinforced cementitious composites was also discussed in a different publication by Manzur and Yazdani (2016). The utilization of CNF in the cement matrix started in the early 1990s (Chen and Chen 1993) when short CNFs were introduced in cement mortar, producing an increase of 85% in flexural strength, 205% in flexural toughness and 22% in compressive strength. In 2000, Chung showed that cement-CNF composites for smart structures had increased flexural strength and toughness, improved impact resistance, reduced drying shrinkage and enhanced freeze-thaw durability (Chung 2000). Li *et al.* (2004) displayed the microstructure of the cement mortar with nanoparticles. The compressive strength and flexural strength of the cement mortar with nanoparticles were higher than plain cement paste. A break-through study (Balagurur and Chong 2008) on the integration of micro CNFs in the cement paste showed that the usage of silica fume and methyl cellulose led to decreased electrical resistivity and increased tensile strength. Li *et al.* (2007) demonstrated that the abrasive resistance, compressive and flexural strengths of concrete improved significantly with the addition of nanoparticles and polypropylene fibers. Gao *et al.* (2009) performed tests on mechanical and electrical properties of self-consolidating concrete with CNF. The concrete containing 1% of CNF produced the best performance in terms of compressive strength and electrical resistivity.

It is evident that significant research work has been undertaken to date on nanocomposites with cement and CNT or CNF. Such efforts primarily concentrated on nanoparticle dosage rates, dispersion efficiency and mechanical properties in mortar with typical Portland cement. However, considering the fineness and mechanical properties of CNT/CNF, a

Table 1. Common Properties of CNT and CNF

	CNT	CNF	References
Definition	Single or multiple graphene sheets rolled into cylindrical structure	Cylindrical allotrope with graphene sheets stacked cones, cups or relates	
Average Diameter/Length	0.4–50nm/several microns	70-200nm/several hundred microns	[Makar et al., 2005; Cwirzen et al., 2008; Manzur and Yazdani, 2010; Manzur et al., 2014; Manzur and Yazdani, 2015; Manzur and Yazdani, 2013; Manzur et al., 2016]
Modulus of Elasticity	1TPa	Ashighas 400GPa	[Makar et al., 2005; Manzur and Yazdani, 2013; Manzur et al., 2016]
Tensile strength	11-63GPa	3-7GPa	[Makar et al., 2005; Chen and Chung, 1993]
Compressive strength	100–150GPa	Not reported	[Chung, 2000]
Cost	\$130/kg	\$512/kg	

Han *et al.* (2013) observed decrease in water sorptivity and permeability coefficient of CNT reinforced cement-based materials. Manzur and Yazdani (2014) found that cement mortar reinforced with nanotubes of smaller diameter resulted in relative higher strength than that in composites with larger size nanotubes. In another study, Manzur and Yazdani (2015) recommended an optimum mix proportion for producing treated MWNT reinforced cement composites. Manzur and Yazdani (2013) also found that flow of nanotube reinforced cementitious mixes is a good measure of dispersion quality of the mix. In addition, it has been observed that MWNT reinforced cement mortar has good potential to be used as concrete crack repair material (Manzur et al. 2016). The effect of different critical parameters, such as nanotube size and type, treatment process of nanotubes and mixing technique, on

potential avenue of such application could be in composites with microcement. It is much finer (50%) than ordinary Portland cement, which may allow the formation of stronger bond and better dispersion at the micro and nano level. Past studies primarily concentrated on the application of only CNT or only CNF in cement composites, and no significant comparative performance analysis was undertaken. The study reported herein investigated the relative performance of both CNT and CNF in a microcement matrix composite. The comparison was based on various mechanical and physical properties of CNT-microcement and CNF-microcement samples through an experimental approach. Although microcement is currently used by itself in a slurry form mainly for rock crack injection and geotechnical stabilization, proven enhancement with nanoparticles could open the door for other

type of infrastructure applications. Microcement is quite a bit finer than the other cement types, and also more expensive. The size and agglomeration of cement grains play a crucial role in the dispersion of nano reinforcements within the cement matrix, and the finer grain size may assist in the bonding and dispersion of the nano additives. In this study, composites were prepared using microcement mortar reinforced with two common MWNT and CNF. The main objectives of this research were to study the behavior of microcement mortar and compare the mechanical properties of cement composites with the control samples without any nanofibers. Compressive strength, flexural strength, mix flows, setting time, bleeding and shrinkage of samples were analyzed and evaluated for the purpose of identifying changed properties due to inclusion of nanoparticles. More detailed information on the study presented herein may be found in the literature (2015).

Section 1.1

Challenges in Using Nanoparticles

Past and current research has identified several challenges for the utilization of nanoparticles in cementitious products: (1) High cost: (2) Lack of efficient dispersion techniques in the cement matrix: (3) Difficulty in effective determination of dispersion and bonding within the cement matrix. The current obstacle of high cost is likely to decrease in the future. Commercially available MWNT are cheaper at present than SWNT. Clumping and the lack of cohesion between tubes and the matrix bulk material are two problems with the addition of nanoparticles to any material. The latter causes a problem which is called sliding. The tubes tend to aggregate to form bundles or ropes due to the interaction between the graphene sheets. Sometimes the ropes can even be twisted with one another. In order to achieve good dispersion, these must be isolated from one another. Effective dispersion technique must be employed in order to avoid agglomeration. For example, when gum Arabic is used with pre-dispersion of the nanotubes, an increase in the mechanical properties is achieved. More investigation is needed in order to establish the optimum values of parameters of all nanoparticles and dispersing agents. The expectation of nanoparticle reinforcement is that one would achieve significantly greater performance from the composite. So far, the reported works have shown modest gains in mechanical properties of such composites.

Section 2.0

Materials and equipment

Section 2.1

Microcement

A typical micro fine cement was used in this research. Table 2 presents the relevant microcement properties and comparison with a typical Type II Portland cement. The selected microcement has a small particle size making it particularly well-suited for penetration into tight joints, fissures and pore spaces. Superior penetration provides a water-tight grouted rock or soil mass much more effective than ordinary Portland cement systems.

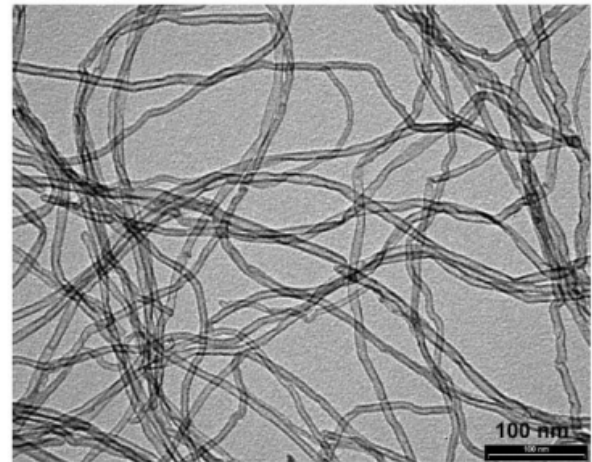
Table 2. Comparison of Microcement and Typical Type II Portland Cement

Property	Microcement	Type II Portland Cement
Blain value	6500 cm ² /g	3770 cm ² /g
% retained by 15 micron sieve	95%	95%
Initial Setting time	60-120 min	45 -375 min
Final setting time	120-150 min	60 -600 min

SECTION 2.2

CNT and CNF

Typical MWNT and CNF available in the market were used herein. Some important properties of the selected nanoparticles are presented in Table 3. The selected CNT is produced by the chemical vapor deposition method, during which exclusive catalysts are used, making it the most electrically conductive available CNT. It has highly perfect chemical surface enabling high efficiency in the embedded matrix. Scanning electron microscope (SEM) image of the CNT is shown in Fig. 1(a). Powdered form of the CNT is shown in Fig. 1(b).



(a) SEM Image



(b) Powdered form

Figure 1. CNT images

Table 3. Properties of Selected CNT and CNF

PROPERTY	UNIT	MWNT	CNF
Average Diameter	nanometers	9.5	100
Average Length	microns	1.5	1
Carbon Purity	%	90	95
Metal Oxide	%	10	5
Surface Area	m ² /g	250-300	41

The selected CNF is produced by heat-treating the fiber at 1500° C. This converts any chemically vapor deposited carbon present on the surface of the fiber to a short range ordered structure. Transmission electron micrograph (TEM) of the CNF catalytic layer is shown in Fig 2.

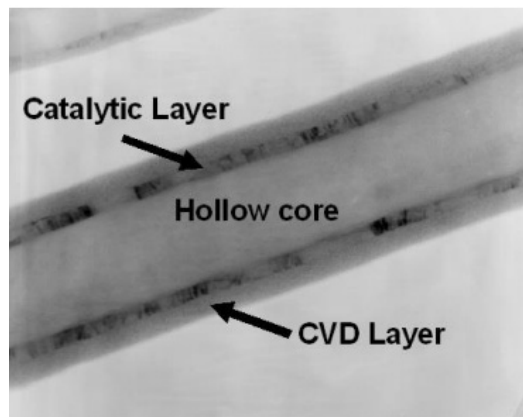


Figure 2. Catalytic layer of CNF

Section 2.3

Surfactant and Super plasticizer

A typical surfactant was used as an agent to uniformly disperse nanoparticles in water. The agent is specially formulated for creating aqueous dispersions of MWNT and CNF. Surfactant plays an active role in dispersion of nanoparticles in water. It was used herein along with a super plasticizer for enhanced dispersion. The typical super plasticizer was a commercial high range water reducing admixture.

Section 2.4

Sonicator

An ultrasonic tip sonicator was used for sonication of the aqueous medium (Fig. 3). It had a titanium tip, which is effective in dispersing the carbon nanomaterial in water, as compared to the bath or cup horn sonicator types. The tip was 12 mm in diameter and had a high intensity with amplitude of 120 μm . It had a frequency range of 1 to 100.



Figure 3. Sonicator Setup for nanoparticle Dispersion

Section 3.0

Procedure

Three different w/c ratios of 0.4, 0.45 and 0.50 were used in this research with 0.008 ratio of super plasticizer by the weight of micro-cement. The ratio of super plasticizer was chosen based on a previous study by the authors (Manzur and Yazdani). Two different ratios of nanoparticles were used in this research, 0.1% and 0.05% of both CNT and CNF, also by the weight of cement.

Section 3.1

Mortar mixing

The dispersion of CNT and CNF into aqueous medium is difficult, as they agglomerate and form bundles when mixed with water. Therefore, sonication process was used to disperse nanoparticles into water. Figure 4 presents two photographs showing improperly and properly dispersed CNT in the aqueous medium. First, the 0.8% super plasticizer was mixed with the water and then stirred well. A few drops of surfactant were added next, after which the solution was sonicated for 2 minutes with 50% amplitude. The CNT or CNF was then measured based on the intended dosage rate by weight of cement. The nanoparticle was added in sequence and was sonicated for 5 minutes for each CNT/CNF addition. The total sonication time was approximately 40 minutes. The sonicator amplitude varied between 50% - 75%. The required amount of cement was added to the sonicated aqueous solution and mixed for a few minutes in a mixer with a flat beater.



(a) Agglomerated



(b) Well dispersed

Figure 4. CNT in water

The parametric procedure consisted of varying the water/cement (w/c) ratio and the CNT/CNF dosage rates. Based on previous research [2, 3, 5, 6, 18, 25], w/c ratios of 0.4, 0.45 and 0.50 and CNT/CNF dosage rates of 0.05% and 0.1% were used in this study. The previous studies showed that these parametric combinations of w/c ratio and CNT dosage rate gave optimum performance in terms of strength and flow rates. Plain cement control samples without any nanoparticle were also prepared as a benchmark for comparison purposes.

Section 3.2

Testing Procedures

Various mechanical and physical properties of the nanoparticle enhanced microcement mortar were evaluated in the experimental program. The series of tests demonstrate well the performance of the cement composite. The ASTM C109 test procedure (ASTM C109/109m-13) was used herein to determine the mortar compressive strength using 50 mm cubes, and ASTM C348 test procedure (ASTM C348-14) was used to evaluate the mortar flexural strength with 40 by 40 by 160 mm specimens. For both tests, specimens were unmolded after 24 hours from casting and placed in saturated lime water at room temperature for moist curing. Three specimens were tested at each age of 7, 14 and 28 days at a load rate of 1334 N/s in a Universal Testing Machine until failure (Fig. 5). To determine the mortar flow at 0.5 w/c ratio with 0.05% CNT/CNF, the ASTM C939-10 method (ASTM C939-10) was used through the flow cone (Fig. 6). The time of efflux was calculated as the time taken by the mortar to pass through the nozzle outlet of the flow cone. The Vicat apparatus was used to determine the mortar setting time through the ASTM C191-13 procedure (ASTM C 191-13) with 0.5 w/c ratio and 0.05% CNT/CNF dosage rates (Fig. 7). The ASTM C596-09 method (ASTM C596-09) was used to determine the mortar drying shrinkage. In this process, a micrometer was used on 25 by 25 by 125 mm sized specimens with 0.4 w/c ratio and 0.05% of CNT/CNF. Two pins were inserted at the top face of the specimen for the micrometer readings. Initial length between these pins was noted as gauge length (Fig. 8). The specimen was stored for 48 hours in lime saturated water, allowed to dry in air for 25 days and the shrinkage calculated at different ages. Finally, ASTM 940-10a process (ASTM 940-10a) was used to determine the mortar bleeding at the surface.



(b) Flexural testing

Figure 5. Strength test setup



Figure 6. Flow cone test



(a) Compression testing



Figure 7. Setting time test

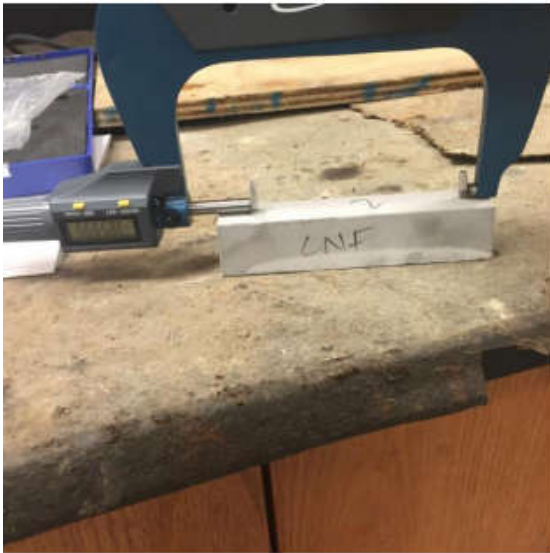


Figure 8. Shrinkage test

Section 4.0

RESULTS AND DISCUSSION

Section 4.1

Compressive strength

The compressive strength is a very important parameter for cement composites, both in a compression (masonry grout) or flexural applications (beams). Figures 9 and 10 show the compressive strengths from CNT and CNF samples in comparison with the control samples without nano addition, respectively. It can be seen from Figure 9 that the largest compressive strength was displayed by the 0.05% CNT samples with 0.4 w/c ratio at 14 and 28 days of age. The strength increase for this combination was 23% over the control samples. Additional w/c ratio at this dosage rate of CNT resulted in reduction of compressive strengths. However, the results were more inconsistent for 0.1% CNT addition, although the strength with 0.5 w/c ratio at 28 days increased from earlier ages. The maximum strength at 28 days for these samples was 57.8 MPa, which was 17% higher than that in the control samples. From Figure 9, it is clearly evident that there was no appreciable increase or trend in compressive strengths with 0.05% or 0.1% addition of CNF in the cement matrix. The initial 7 day compressive strengths were good. However, the values decreased with time in general.

Section 4.2

Flexural strength

The flexural strength is another important parameter for cement composites, for members under bending (beam-columns, slabs, beams, and footings). The flexural strength results are illustrated in Figures 11 and 12 for CNT and CNF composites, respectively, with 0.4 w/c ratio. The strengths in the samples with 0.05% CNT addition achieved significant flexural strength gain over time. In case of 0.05% CNT samples, the strength gain over the control samples was 14% at both 7 and 14 days. However, the strength reduced at 28 days for these samples. Samples with 0.1% CNF yielded more

inconsistent flexural strengths. It can be observed that there is significant increase in the flexural strength in the initial days; however, the strength reduced with time. These samples gained as much as 32% flexural strength at seven days. In case of CNF samples, the flexural strength was increased by 46% with 0.1% dosage rate and 17% with 0.05% dosage rate, respectively.

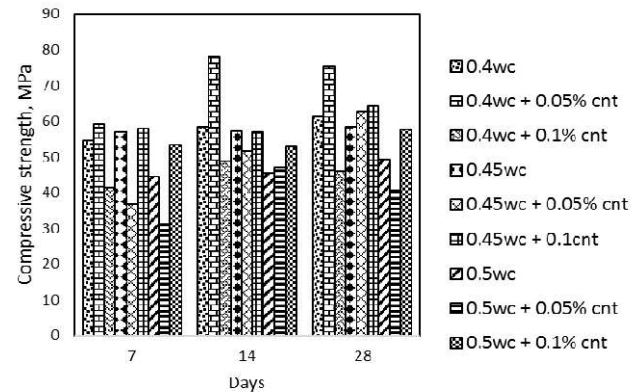


Figure 9. Compressive strengths of CNT composites

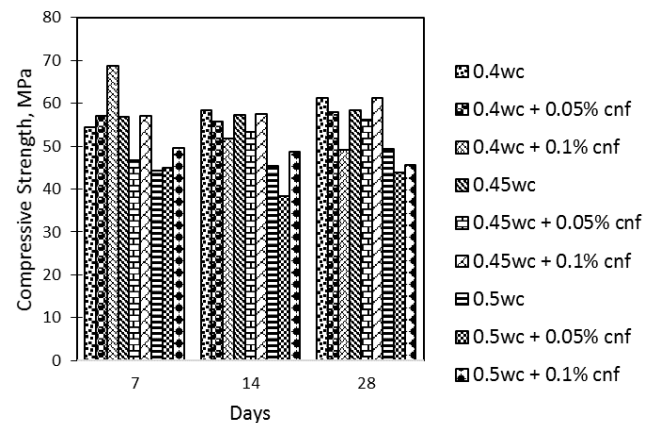


Figure 10. Compressive strengths of CNF composites

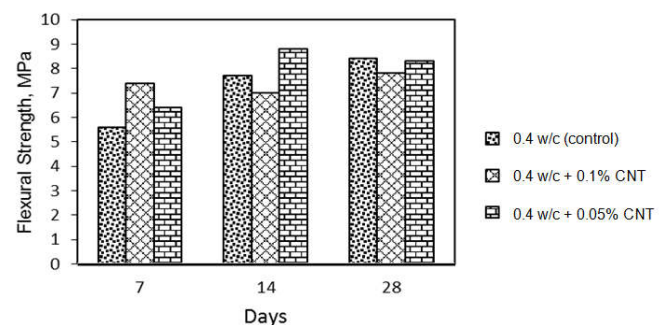


Figure 11. Flexural strengths of CNT composites

Section 4.3

Flow

The flow value is a good indicator of the workability and ease of placement of cement mortars and grouts. This is very useful, especially in casting or injecting locations with limited space or high steel rebar congestion. The flow was compared through the time of efflux value defined by ASTM, as given in

Table 4. This value is inversely related to the mortar flowability. Table 4 shows that the time of efflux decreased significantly with the addition of nanoparticles. The CNT addition resulted in a better flow rate than the CNF addition.

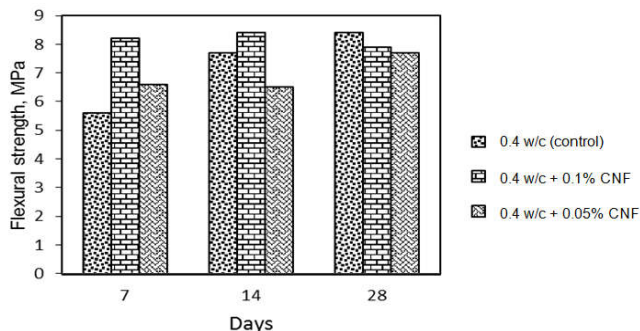


Figure 12. Flexural strengths of CNF composites

Table 4. Time of flow efflux values

Specimen	Time of efflux (sec)	% reduction from control
Control (without CNT/CNF)	16.22	N/A
CNT	12.67	22
CNF	13.65	16

Section 4.4

Setting time

Generally, initial setting is the time elapsed between the moment water is added to the cement to the time at which the paste starts losing its plasticity. Final setting time is the time elapsed between the moment the water is added to the cement to the time at which paste has completely lost its plasticity and attained sufficient firmness to resist certain definite pressures. The initial setting time is required for mixing, transporting, placing, compacting and finishing of cement composites. Figure 13 shows that control samples resulted in an initial setting time of 27 minutes, whereas samples with 0.05% CNT and CNF and 0.5 w/c ratio had setting times of 20 minutes and 18 minutes, respectively. Therefore, it is apparent that the setting time of cement mortar was highly influenced by nanoparticles and proves that presence of nanoparticles act as nucleating agent within the cement matrix to accelerate the hydration process. Quicker initial setting time could be detrimental for the construction activities mentioned above. However, faster mortar setting time could be helpful in applications such as repair work.

Section 4.5

Shrinkage

Drying shrinkage is defined as the contracting of a hardened cement composite mixture due to the loss of capillary water. This shrinkage causes an increase in tensile stress, which may lead to cracking, internal warping and external deflection, before the composite is subjected to any kind of loading. The calculated drying shrinkage per unit length at different specimen ages is presented in Figure 14. The shrinkage rate gradually increased with time. However, the rate of increase gradually decreased with time and was almost similar at about eight weeks of age. Therefore, it is expected that the drying

shrinkage rate would be asymptotic near about 10 weeks of age, as is clear from Figure 14. The microcement manufacturer specifies the drying shrinkage of the cement mortar at 21 days as 0.1. This value (for the control case without nanoparticles) is quite a bit greater than that for the expected drying shrinkage for CNT and CNF mortar specimens of about 0.045 and 0.07, respectively (Figure 14). It is apparent that the addition of nanoparticles significantly reduces the drying shrinkage in the microcement mortar that could reduce the potential for shrinkage related cracking at early ages, and to a lesser extent, at later ages.

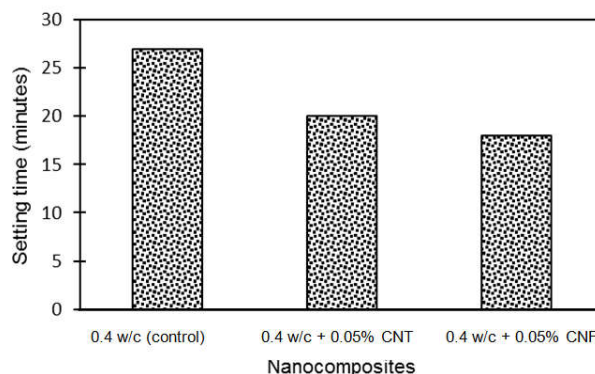


Figure 13. Setting times

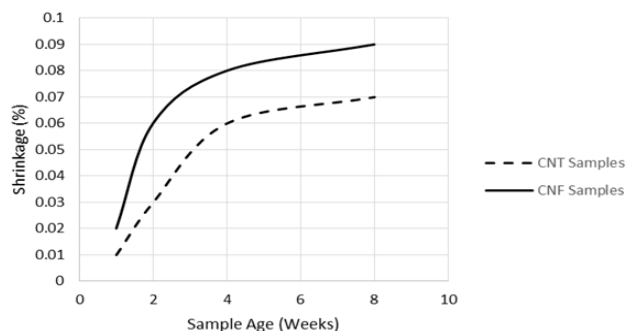


Figure 14. Shrinkage values with time

Section 4.6

Bleeding

Emergence of water from a newly placed cementations mixture caused by the settlement of the solid materials within the mass is termed as bleeding. Some bleeding is normal but excessive bleeding can be problematic. Many factors affect cement based bleeding, such as fineness of cement, sample height, and pressure. By allowing free water to migrate to the surface and evaporate, the w/c ratio may decrease, thus decreasing capillary porosity and increasing its density and durability. It can also be useful to aid in finishing operations and reduce shrinkage cracking. In addition, it is important not to begin finishing operations before most of the bleed water has evaporated. Bleeding was found to be negligible for all samples.

Section 5.0

Conclusions

The following conclusions can be made based on the findings from this study:

1. The enhancement of microcement mortar with nanoparticles such as carbon nanofiber (CNF) and carbon nanotube (CNT) is promising. Several important fresh and hardened mortar properties are affected by the nano-enhancement, most of them in a positive way. Additional research is necessary on dispersion effectiveness, w/c ratio effect, dosage rates and consideration of other properties for fully understanding and realizing this potential application.
2. Mortar samples with CNT addition resulted in a maximum compressive strength gain of 23% over the control (plain mortar) samples, for 0.05% CNT addition and 0.04% w/c ratio at 7 to 28 day age. Increased w/c ratio resulted in reduction of compressive strength. The compressive strengths for samples with 0.1% CNT was more inconsistent and did not follow any specific pattern. There was no appreciable increase or trend in compressive strengths with 0.05% or 0.1% addition of CNF in the cement matrix. The initial seven day compressive strengths were good. However, the values decreased with time in general.
3. The flexural strengths of 0.05% CNT strengthened mortar specimens with 0.4% w/c ratio were about 14% greater than the plain mortar specimens at early ages. However, similar to the compressive strengths, the flexural strengths in some samples decreased at 28 day age. The effect of CNF addition on flexural strength was more inconsistent. Although CNF caused as much as 46% increase in these strengths, the age and dosage related increases were not consistent.
4. Flow values were greatly influenced by the addition of nanoparticles. For the selected mortar mix with 0.5 w/c ratio, the flowability of the mix increased by 16 – 22% due to 0.05% nanoparticle addition. CNT resulted in 6% better flow of the mortar than CNF. So, the addition of nanoparticles cause more workable mortar and increased constructability.
5. Initial setting time values were affected by the addition of nanoparticles. The setting time decreased by about 26 – 33% with 0.05% nanoparticle addition at 0.5% w/c ratio. CNF had a larger effect in decreasing the setting time than CNT. Therefore, decreased initial setting time could be detrimental for any mortar placement and finishing activities if sufficient time is not available before the mortar sets. However, faster setting time could be desirable for small volume applications such as repair work.
6. Shrinkage rates in all samples gradually increased with time. However, the increase rate slowed with time and shrinkage rates were expected to level off at about 10 weeks of age. At earlier ages, CNF addition caused greater shrinkage in mortar samples than the CNF samples. It is apparent that the addition of nanoparticles significantly reduces the drying shrinkage in the microcement mortar that could reduce the potential for shrinkage related cracking at early ages, and to a lesser extent, at later ages.
7. The bleeding in mortar samples was negligible and unaffected by nanoparticle addition. So, finishing operations will not be adversely affected by CNT or CNF addition.
8. Several properties of Microcement mortar are positively affected by CNT or CNF enhancement, such as compressive strength, flexural strength and

flowability. The mortar setting time is negatively impacted, while shrinkage and bleeding are unaffected. CNT addition is found to result in more consistent positive effect than CNF addition. Additional investigation is needed to fully understand the potential usage of these nanoparticles in cement composites and remove some of the relevant inconsistencies.

Section 6.0

REFERENCES

- Agullo, V.J., V.C. Ligeró, D.P. Rico, M.J.G. Casas, A.G. Martínez, J.M.M. Royo and J.G. Moreno, 2009. Mortar and Concrete Reinforced with Nanomaterials, *Nanotechnology in Construction* 3, DOI: 10.1007/978-3-642-00980-8_52
- Alimeneti, N.R. 2015. Properties of Microcement Mortar with Nano Particles, M.S. Thesis, University of Texas at Arlington
- American Society for Standard Testing and Materials (ASTM), Standard Test Method for Compressive Strength of Hydraulic-Cement Mortars (Using 2 in Cube Specimens), ASTM C109/109M-13
- American Society for Standard Testing and Materials (ASTM), Standard Test Method for Flexural Strength of Hydraulic-Cement Mortars, ASTM C348-14
- American Society for Standard Testing and Materials (ASTM), Standard Test Method for Flow of Grout for Preplaced-Aggregate Concrete, ASTM C939-10
- American Society for Standard Testing and Materials (ASTM), Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle, ASTM C 191-13
- American Society for Standard Testing and Materials (ASTM), Standard Test Method for Drying Shrinkage of Mortar Containing Hydraulic Cement, ASTM C596-09
- American Society for Standard Testing and Materials (ASTM), Standard Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory, ASTM C 940-10a
- Balaguru P. and K. Chong, 2008. Nanotechnology of Concrete: Recent Development and Future Perspectives, *American Concrete Institute*
- Chen P.W. and D.D.L. Chung 1993. Concrete Reinforced with Up to 0.2 Vol% of Short Carbon Fibers, *Journal of Composites*, Vol 24, No. 1, pp. 33-52
- Chung D.D.L. 2000. Cement-Matrix Composites for Smart Structures, *Smart Materials and Structures*, Vol. 9 No. 4, pp. 389
- Chung, D.D. 2005. Dispersion of Short Fibers in Cement, *Journal of Materials in Civil Engineering*, Vol 17, No. 4, pp. 379-383
- Coleman, J.N., U. Khan, W.Z. Blau and Y.K. Gun'ko, 2006. Small but Strong: A Review of the Mechanical Properties of Carbon Nanotube-Polymer Composites, *Carbon*, DOI: 10.1016/j.carbon.2006.02.038
- Cwirzen, A., K. Habermehl-Cwirzen and V. Penttala, 2008. Surface Decoration of Carbon Nanotubes and Mechanical Properties of Cement/Carbon Nanotube Composites, *Advance in Cement Research*, doi:10.1680/adcr.2008.20.2.65
- Gao, D., M. Strum and Y.L. Mo, 2009. Electrical Resistance of Carbon-Nanofiber Concrete, *Smart Materials and Structures*, Vol. 18, No 9

- Han, B., X. Yu and E. Kwon, 2009. A Self-Sensing Carbon Nanotube/Cement Composite for Traffic Monitoring, *Nanotechnology*, DOI:10.1088/0957-4484/20/44/445501
- Han, B., Z. Yang, X. Shi, and X. Yu, 2013. Transport Properties of Carbon-Nanotube/Cement Composites *Journal of Materials Engineering and Performance*, 22, pp. 184-189
- Konsta-Gdoutos, M.S., Z.S. Metaxa and S.P. Shah, 2010. Multi-Scale Mechanical and Fracture Characteristics and Early-Age Strain Capacity of High Performance Carbon Nanotube/Cement Nano Composites, *Cement and Concrete Composites*, Vol. 32 No. 2, pp. 110-115
- Li, G.Y., P.M. Wang and X. Zhao, 2007. Pressure-Sensitive Properties and Microstructure of Carbon Nanotube Reinforced Cement Composites, *Cement and Concrete Composites*, Elsevier, Vol. 29, No. 5, pp. 377-382, DOI: 10.1016/j.cemconcomp.2006.12.011
- Li, H., H.G. Xiao, J. Yuan and J. Ou, 2004. Microstructure of Cement Mortar with Nano-Particles, *Composites Part B: Engineering*, Vol. 35, No 2, pp. 185-189
- Li, H., M.H Zhang and J.P Ou, 2007. Flexural Fatigue Performance of Concrete Containing Nano-Particles for Pavement, *International Journal of Fatigue*, Vol. 29, No. 7, pp. 1292-1301
- Makar J.M. and J.J. Beaudoin, 2004. Carbon Nanotubes and Their Application in the Construction Industry, *Special Publication-Royal Society of Chemistry*, 292, pp. 331-342
- Makar, J., J. Margeson, and J. Luh, 2005. Carbon Nanotube/Cement Composites-Early Results and Potential Application, Proceedings of the 3rd International Conference on Construction Materials: Performance, Innovations and Structural Implications, Vancouver, B.C., Aug. 22-24, pp. 1-10
- Manzur T. and N. Yazdani 2010. Strength Enhancement of Cement Mortar with Carbon Nanotubes: Early Results and Potential, *Journal of the Transportation Research Board*, No. 2142, pp. 102-108
- Manzur T. and N. Yazdani 2013. Importance of Flow Values in Qualitative Evaluation of Carbon Nanotube Reinforced Cementitious Matrix, *Malaysian journal of Civil Engineering*, Vol. 25, No. 1, pp. 71-80
- Manzur T. and N. Yazdani 2015. Optimum Mix Ratio for Carbon Nanotubes in Cement Mortar, *KSCE journal of Civil Engineering*, DOI: 10.1007/s12205-014-0721-x
- Manzur T. and N. Yazdani, 2016. Effect of Different Parameters on Properties of Multiwalled Carbon Nanotube-Reinforced Cement Composites, *Arabian Journal of Science and Engineering*, DOI: 10.1007/s13369-016-2181-8
- Manzur, T., N. Yazdani and A. B. Emon, 2016. Potential of carbon nanotube reinforced cement composites as concrete repair material, *Journal of Nanomaterials*, Vol. 2016, Article ID 1421959, Hindawi Publishing Corporation
- Manzur, T., N. Yazdani and M.A.B Emon, 2014. Effect of Carbon Nanotube Size on Compressive Strengths of Nanotube Reinforced Cementitious Composites, *Journal of Materials*, Hindawi Publishing Corporation, 8 Article ID 960984
- Marrs, B., R. Andrews and D. Pienkowski, 2007. Multiwall Carbon Nano-Tubes Enhance the Fatigue Performance of Physiologically Maintained Methyl Methacrylate-Styrene Copolymer, *Carbon*, DOI: 10.1016/j.carbon.2007.05.013
- Metaxa, Z.S., M.S. Konsta-Gdoutos and S.P. Shah, 2009. Carbon Nanotubes Reinforced Concrete, *Nanotechnology of Concrete*, 267, pp. 11-20
- Parveen, S., S. Husain, A. Kumar, J. Ali, M. Husain, Harsh and M. Husain, 2013. Enhanced Field Emission Properties of Carbon Nanotube Based Field Emitters by Dynamic Oxidation, *Current Nanoscience*, DOI: 10.2174/15734137113099990060
- Parveen, S., S. Husain, A. Kumar, J. Ali, M., Harsh and M. Husain, 2012. Study of J-E Curve with Hysteresis of Carbon Nanotubes Field Emitters, *ISRN Nanomaterials*, Hindawi Publishing Corporation, DOI: 10.5402/2012/971854
- Yazdanbakhsh, A., Z.C. Grasley, B. Tyson and R.A. Al-Rub, 2009. Carbon Nano Filaments in Cementations Materials: Some Issues on Dispersion and Interfacial Bond, *Proceedings of the American Concrete Institute (ACI'09)*, 267, pp. 21-34
- Yu, M.F., B.S. Files, S. Arepaali and R.S. Ruoff, 2000. Tensile Loading of Ropes of Single Wall Carbon Nanotubes and Their Mechanical Properties, *Physical Reference Letter*, DOI: 10.1103/PhysRevLett.84.5552
- Zheng, L.X., M. J. O'Connell, S. K. Doorn, X.Z. Liao, Y.H. Zhao, E.A. Akhadov, M.A. Hoffbauer, B.J. Roop, Q.X. Jia, R.C. Dye, D.E. Peterson, S. M. Huang, J. Liu and Y. T. Zhu, 2004. Ultra Long Single-Wall Carbon Nanotubes, *Nature Materials*, DOI: 10.1038/nmat1216
