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Thermal response characterization and comparison of carbon nanotube-enhanced cementitious composites

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A B S T R A C T

Carbon nanotubes (CNT) can be integrated with cement-based composites to modify their electrical and thermal characteristics. In this study, the objective is to characterize the thermal characteristics of CNT-based cement mortars cast using two unique CNT mixing methods. The mixing method, CNT content, curing age, and applied electrical excitation were systematically varied during experiments. The results of the temperature tests with the CNT cement mortars indicated that mixing CNTs in sand more effectively dispersed the CNTs throughout the mortar, and thus more effectively modified the thermal characteristics of CNT cement mortar. Furthermore, cross-sectional image analysis using field emission scanning electron microscopy supports increased coherence inside the cement due to strong covalent bonds with C-S-H, a hydrate from the temperature tests.

1. Introduction

Recently, the integration of nanomaterials in cement-based materials has demonstrated the possibility of engineering multifunctional structural materials. Specifically, in the aerospace and machinery fields, nanomaterials are being used to develop mechanically superior and functionally new composite systems [1]. Since the mid-2000's, next-generation construction materials have been developed by incorporating nanomaterials into existing cement-based materials, where a significant focus is on enhancing their physical and mechanical characteristics to broaden the use of these materials for new applications [2–5]. For example, Kim et al. [6] used silica fumes to modify the mechanical and physical characteristics of cement mortar specimens enhanced with carbon nanotubes (CNTs). Cement specimens of dimensions of $25 \times 25 \times 150\,mm^3$ with silica contents of 10 wt%, 20 wt %, and 30 wt% were tested. Scanning electron microscope (SEM) images of the specimen demonstrated that the silica fumes increased the interfacial contact between the CNTs and hydration products, thus improving the dispersibility of CNTs [6]. Despite these advances, the realization of nanotechnology-enabled multifunctional construction materials remains to be a relatively new field of study, where the goal is to encode other functionalities (such as sensing, energy harvesting, and energy dissipation, among others) in addition to their load-bearing capabilities.

Among the variety of nanomaterials broadly available today, carbon nanotubes have received significant attention in the composite and polymer materials fields. In short, CNTs are tube-shaped carbon allotropes, where allotropes are materials composed of a single element but possess very different characteristics due to differences in atom counts, sequencing of the atoms, or methods of combination. In particular, CNTs are characterized by an approximately one-dimensional physical structure, low densities, near-ballistic-transport electronic behavior, and larger length-to-diameter ratios than other carbon allotropes. More importantly, they exhibit intrinsic piezoresistivity, which makes them ideal for engineering self-sensing materials. In fact, CNTs were first discovered in the form of multi-walled carbon nanotubes (MWCNTs) while examining carbon byproducts on graphite electrodes in Japan [7].

The potential for utilizing the excellent mechanical properties, electrical conductivity, and heat conductivity of CNTs to modify and functionalize various materials has been studied [2–4,8–18]. While CNTs can be readily incorporated in polymer-based nanocomposites, it becomes considerably more challenging to incorporate them in cement-based materials. This is because cement-based materials tend to be less microscopically homogeneous as compared to polymer-based systems. Nevertheless, experimental studies to improve the mechanical strength of cement mortars by mixing with nanomaterials have been conducted [2–4,8–13]. A study by Seo [14] on improving the mechanical strength

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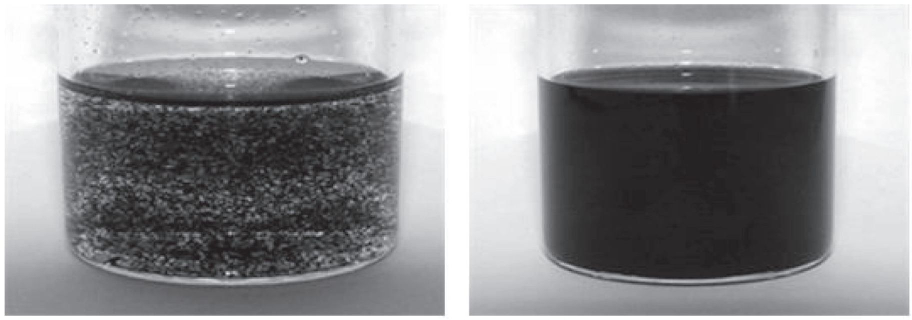
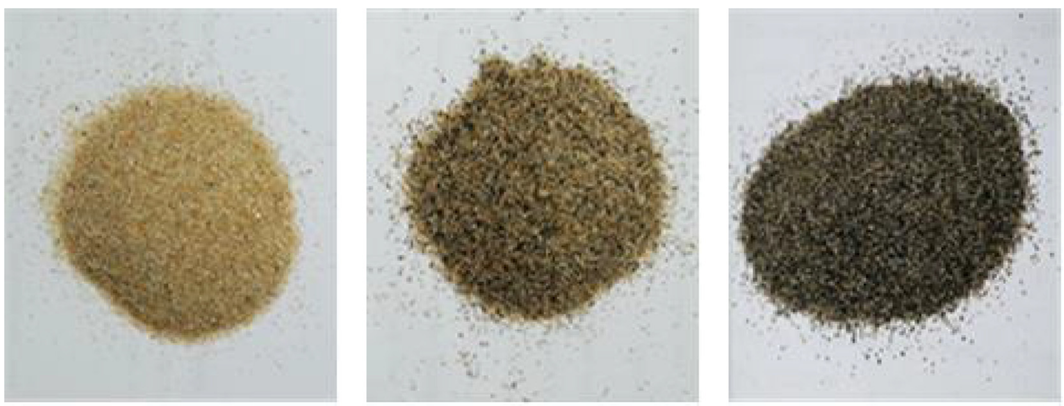


(a) Undispersed CNT solution (b) Dispersed CNT solution

Fig. 1. Dispersion of CNT solutions.



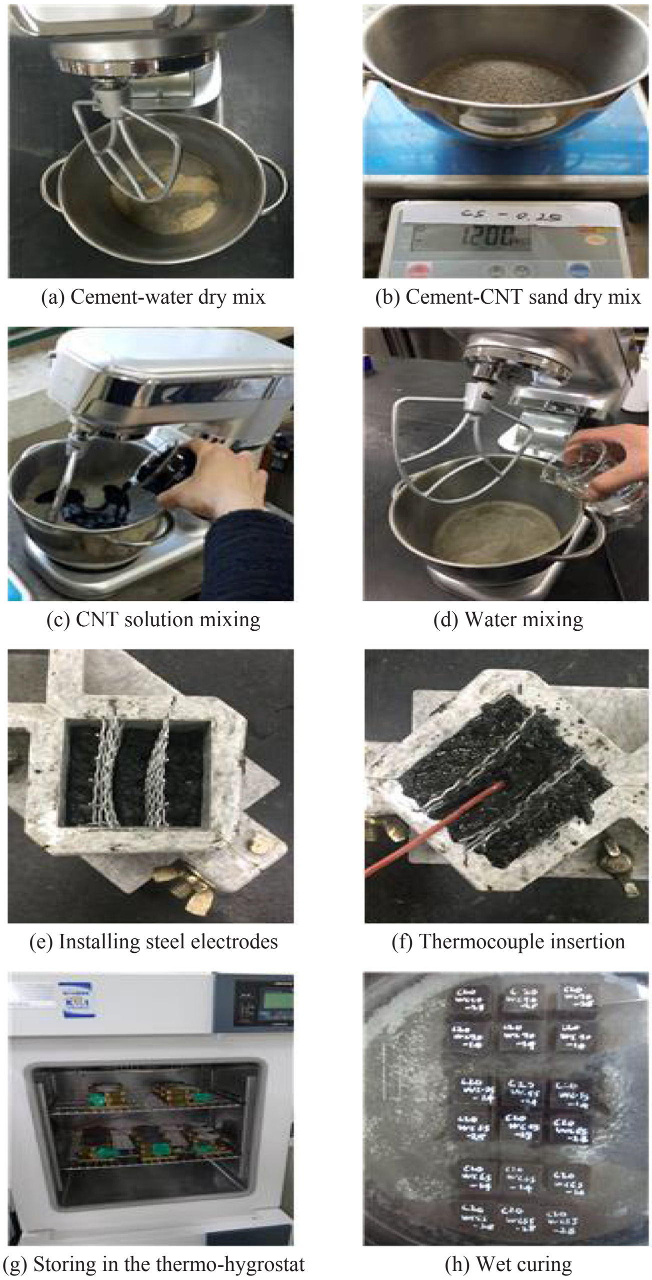
(a) Standard sand (b) 0.125 wt% (c) 0.250 wt%

Fig. 2. Comparison of standard sand and CNT-coated sands.

of cement mortars involved mixing with a MWCNT solution. Compressive strength experiments were conducted with 0.004 wt%, 0.005 wt%, 0.006 wt%, 0.8 wt%, 1.0 wt%, and 1.2 wt% MWCNT cement mortars. The results indicated that the compressive strengths of all weight fractions of MWCNT cement mortars were higher than that of a normal cement mortar. The study showed that the MWCNTs were distributed like a network within the hydration material, acting as a scaffold for the microscopic cracks within the cement and ultimately slowing the cracking process [14]. Choi et al. [15] performed compressive strength experiments for MWCNT cement mortars that contained a diluted MWCNT solution. The diluted 0.5 wt% MWCNT solution used in the study was made with 1.0 wt% and 0.75 wt% MWCNT solutions and distilled water. The results indicated that the compressive strength of the MWCNT cement mortar made from the 0.5 wt% solution (diluted from a 1.0 wt% solution) was lower by up to 4.8% compared to the MWCNT cement mortar made from the undiluted 0.5 wt% solution. The study concluded that dilution of the MWCNT solution did not significantly influence the condensation or dispersion of the solution. Camacho et al. [9] conducted experiments testing the compressive and flexural strength of CNT cement mortars while varying the CNT content and the curing age. This study confirmed that the compressive and flexural strength of a CNT cement mortar increased in proportion to the curing age, in a way similar to the increments of compressive and flexural strength of a normal cement mortar. The compressive and flexural strength of the CNT cement mortar were the highest when cement was aged for 28 days and CNT content was 0.05%. Once CNT content exceeded 0.05%, the compressive and flexural strengths of the CNT cement mortar declined. Kang et al. [16] analyzed the microscopic structure of a cement mortar mixed with MWCNTs using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The results indicated that the addition of CNT led to minimal changes in hydration products within the cement mortar. However, the study also concluded that the increase in compressive strength of the MWCNT cement mortar was due to the decrease in porosity in its microscopic structure, and the reduction of pore size in the cement mortar was due to the addition of MWCNTs. However, fundamental research is still required to understand and devise effective methods to best homogeneously distribute CNTs within cement-based materials.

Although, as mentioned earlier, it is known that an even distribution of CNTs in cement-based materials can improve their mechanical strength, the thermal and electrical conductivities of CNTs can also be utilized to transform cement-based materials from a nonconductive to a conductive state. These modified characteristics may result in cement-based materials with rapid curing, heating, and thawing capabilities. Massive concrete is characterized by a large temperature differential across the concrete volume, due to the byproduct heat that is produced from the hydration reactions during the curing process. Large concrete structures must be built to reduce and dissipate this heat. Therefore, the modified characteristics of CNT mixtures may be disadvantageous for mass concrete. However, in cold regions, the water and aggregate used in construction must either be heated or electricity must be applied to the steel reinforcements in order to raise the temperature until suitable for curing. It could be comparatively more effective for both heating and curing if the modified physical characteristics of CNT-enhanced concrete are utilized for constructing concrete buildings in cold areas. Moreover, when snow accumulates on the surface of a structure after its completion, electricity can be applied to the CNT-enhanced concrete, increasing its surface temperature, and subsequently melting the accumulated snow.

Aside from the improvement of mechanical strength, studies focused on modifying the electrical and thermal conductivities of nanocement mortars are also growing in number [14,15,9,16,17]. Gomis



(a) Cement-water dry mix (b) Cement-CNT sand dry mix (c) CNT solution mixing (d) Water mixing (e) Installing steel electrodes (f) Thermocouple insertion (g) Storing in the thermo-hygrostat (h) Wet curing

Fig. 3. Specimen production process.

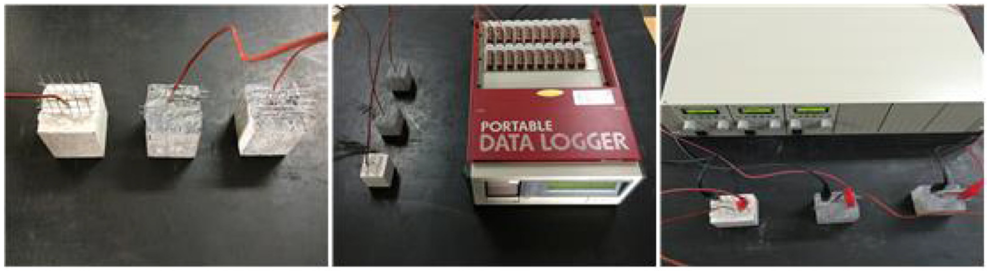
Table 1
The parameters of CNT cement mortar temperature tests.

Test group	Specimen name	CNT mixing method	CNT type	CNT content (wt%)	Curing age (days)	Supplied voltage (V)
Group#1	OPC-7	–	–	–	7	50 100
	OPC-28				28	50 100
Group#2	MW-Water-0125-7	CNT Solution	MWCNT	0.125	7	50 100
	MW-Water-0125-28				28	50 100
	MW-Water-0250-7			0.250	7	50 100
	MW-Water-0250-28				28	50 100
Group#3	MW-Sand-0125-7	CNT Sand	MWCNT	0.125	7	50 100
	MW-Sand-0125-28				28	50 100
	MW-Sand-0250-7			0.250	7	50 100
	MW-Sand-0250-28				28	50 100

et al. [18] placed a 100 × 10 mm^2 silver-plated electrode on a nano-mixed specimen with an area of 100 × 100 mm^2 and a thickness of 10 mm, and then measured its surface temperature. They studied the effects of varying water/cement mix ratios, different contents of various nanomaterials (i.e., carbon nanofibers, carbon nanotubes, carbon fiber powders, graphite powders, etc.), and supply voltage (i.e., 50 V, 100 V, and 150 V). Approximately 110 g of ice was piled on top of a nano-mixed specimen with 1 wt% CNT, which melted within 1500 s while applying 90 V to the specimen, which can be applicable for transportation infrastructure. Zhao et al. [19] used a concrete slab with embedded carbon fiber heating wires (CFHWs) to study thawing methods. The concrete slab, with 4 CFHWs spaced 100 mm apart, was stored in a refrigerator at −25 °C. When current was applied to the frozen concrete slab, its temperature reached 0 °C after 2.5 h. Zhang et al. [20] studied thawing systems on paved roads based on MWCNT cement. The heat conducting layer with a thermal conductivity of 2.83 W/m·K was produced by mixing 3 wt% MWCNT with cement. The thawing experiments began when the specimens reached −30 °C, −20 °C, or −10 °C after placing them in a freezer. The temperatures of the specimens rose to 5 °C within 4000 s when 1800 W/m^2 of electricity was applied. They concluded that thawing systems in highways and bridges could be constructed with MWCNT-mixed cement supplied with electricity.

In terms of enabling electrical conductivity and encoding self-sensing properties, Chung [21] studied cement polymers by attempting to increase their heat resistance for thawing and heating purposes (i.e., using carbon fiber mat, carbon fiber cement, carbon fiber epoxy-matrix, flexible graphite, etc.). The thermal conductivity of cement can be increased by mixing electrically conductive fibers with cement. They concluded that a cement polymer using a carbon fiber mat is most effective for self-heating (temperatures up to 134 °C), except for flexible graphite [21]. Han et al. [22] studied the electrical resistance of MWCNT cement mortars subjected to repetitive compressive loads with a range of moisture contents (0.1%, 3.3%, 5.7%, 7.6%, and 9.9%). The results indicated that the piezoresistivity of an MWCNT cement mortar was closely related to its internal moisture content. Han et al. [23] produced CNT cement mortars, 50 × 50 × 50 mm^3 in size, as a basic experiment to produce a self-sensing concrete structure. The compressive strength-resistance experiments were conducted using specimens with two steel nets inserted at 10 mm intervals. The experiment results suggested that the electrical resistance of a CNT cement mortar is directly correlated to the applied compressive load. Wong et al. [24] showed that ultra-low-concentrations of MWCNTs could be dispersed in surfactant solutions and used for casting cementitious composite specimens with piezoresistive properties.

The purpose of this study is to experimentally analyze the thermal characteristics of cement mortar specimens that are produced by two different CNT incorporation techniques. The hypothesis is that the thermal characteristics of CNT cement mortar vary depending on the methods used to mix CNTs and cement. Here, CNT cement mortars are prepared by: (1) using MWCNTs dispersed in water and then used directly during and (2) modifying the interfacial transition zones of mortar by pre-coating dry sand with MWCNT-latex thin films. The rationale is to compare two drastically different CNT incorporation techniques (i.e., dispersion, which is more established, versus a



(a) Specimen placement on insulating rubber (b) Data logger connected to the specimen (c) Power supply connected to the specimen

Fig. 4. Specimen installation.

Table 2
Temperature test results for the CNT cement mortars.

Specimen Name	CNT mixing method	CNT Type	CNT content (wt%)	Curing age (days)	Supplied Voltage (V)	Temperature change (°C)	Specimen/Control group
OPC-7	–	–	–	7	50	1.4	–
					100	11.0	–
OPC-28				28	50	0.5	–
					100	1.1	–
MW-Water-0125-7	CNT Solution	MWCNT	0.125	7	50	33.3	23.8
					100	45.9	4.2
MW-Water-0125-28				28	50	3.7	7.4
					100	21.5	19.6
MW-Water-0250-7			0.250	7	50	19.4	13.9
					100	43.2	3.9
MW-Water-0250-28				28	50	5.7	11.4
					100	14.7	13.4
MW-Sand-0125-7	CNT Sand	MWCNT	0.125	7	50	42.0	30.0
					100	61.7	5.6
MW-Sand-0125-28				28	50	3.5	7.0
					100	29.4	26.7
MW-Sand-0250-7			0.250	7	50	38.6	27.6
					100	55.6	5.1
MW-Sand-0250-28				28	50	2.4	4.8
					100	18.7	17.0

significantly different approach by pre-coating fine aggregates) and to study their corresponding thermal properties for civil infrastructure applications. This experiment entails supplying electrical current to the specimens and measures the internal temperature changes due to resistive heating. The variables in these experiments are the CNT incorporation method, CNT content, curing age, and applied voltage. The results of the experiments are the measured differences between the initial and the maximum temperatures (temperature change, ΔT) for each set of parameters.

2. Experimental details

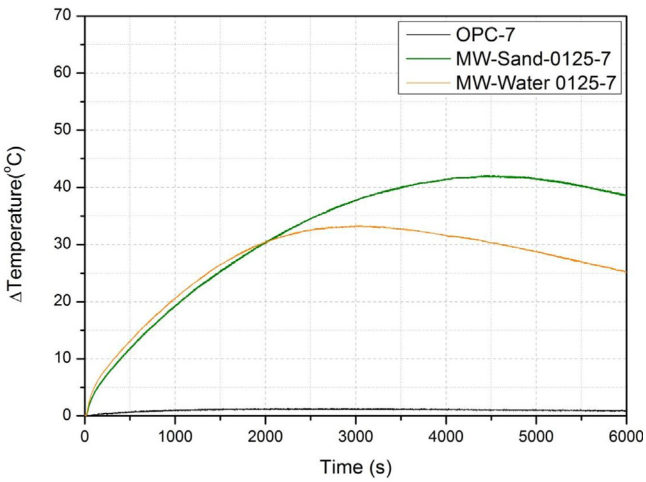
2.1. Carbon nanotube dispersion

Successful dispersion of CNTs in cement-based materials is critical for achieving the desired mechanical, physical, electrical, and thermal characteristic enhancements. Because of strong attractive Van der Waals (vdW) force interactions, CNTs prefer and tend to agglomerate to form bundles. Agglomeration prevents one from leveraging the unique properties of CNTs and from scaling these properties to the bulk scale. Thus, the cohesion between CNTs must be stabilized by dispersing them in a suitable medium before incorporating them in CNT cement mortar.

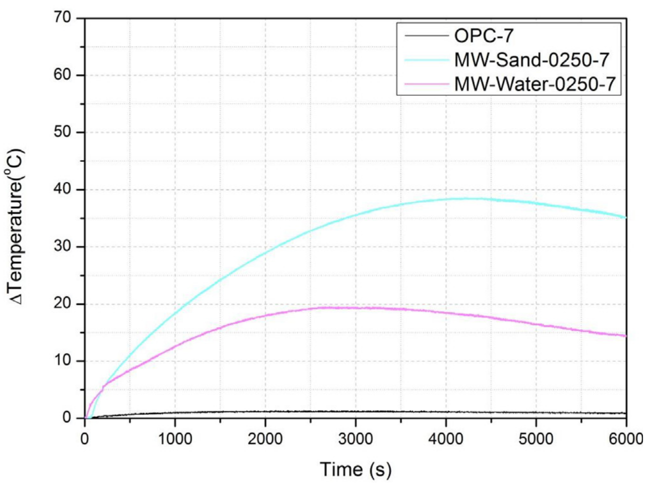
Methods of counteracting vdW forces in order to evenly disperse CNTs have been studied extensively. The existing literature mostly deals with a combination of surface chemical processing and mechanical processing [25]. This mixed method uses both a chemical process to weaken the strong attractive force between CNTs and mechanical processing to disperse them. Recent studies have found that chemical processing with surfactants and physical methods that use ultrasonic waves have increased the dispersibility of CNTs. There is ongoing research to identify the optimal CNT dispersion method for mixing with cement-based materials [26,27].

As mentioned earlier, two different techniques were employed in this study to disperse and incorporate CNTs within cement mortar. The first method makes use of chemical and physical dispersion by mixing MWCNTs in water to create CNT solutions. The content of the mixed MWCNT is 0.125% and 0.250% as a percent of the total cement weight. Because of the vdW forces between the CNTs, they agglomerate to one another when they are mixed in a solution (See Fig. 1(a)). Then, surfactants were added to the solution, which then went through ultrasonic processing [5]. Fig. 1(b) shows the evenly dispersed CNT solution. In this study, MWCNT was dispersed in polyacrylic acid copolymer in beaker glass at room temperature for 8 h at a frequency of 22 kHz using an ultrasonic processor VC-505 (Sonic) made of titanium. These specimens are referred to as "CNT solution cement mortar".

On the other hand, the second method of dispersion involves pre-coating sand with MWCNT-latex thin films. This technique was developed in previous studies and is reported in detail in Gupta et al. [28] and Gonzalez et al. [29]. In short, the procedure entailed dispersing MWCNTs in a 2 wt% poly(sodium 4-styrenesulfonate) (PSS) aqueous solution via ultrasonication. The solution was then mixed with a latex solution to obtain the MWCNT-ink, which was then directly spray-coated onto sand using an airbrush. It should be mentioned that the concentration of the MWCNT-latex ink was prepared such that the same

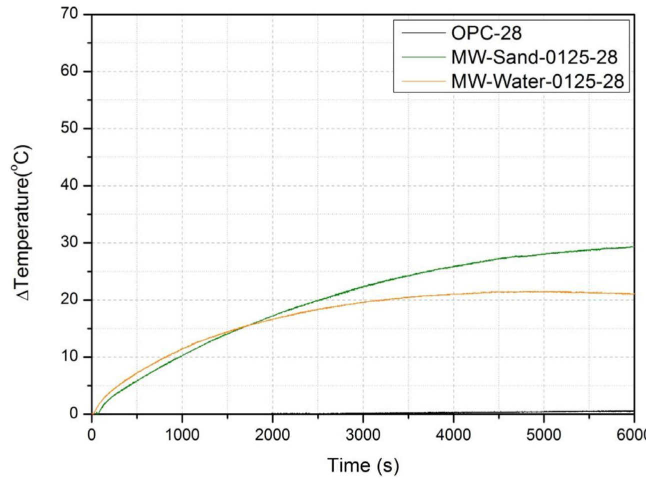


(a) 0.125 wt% CNT content

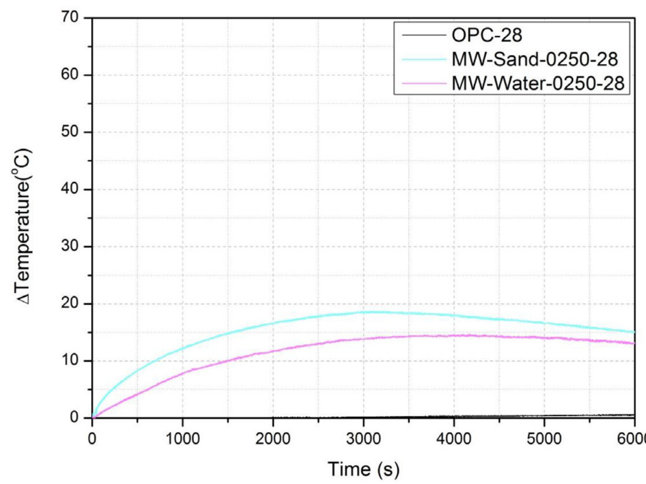


(b) 0.250 wt% CNT content

Fig. 5. Temperature – time curve under 50 V of voltage applied to cement mortars cured for 7 days.



(a) 0.125 wt% CNT content



(b) 0.250 wt% CNT content

Fig. 6. Temperature – time curve under 100 V of voltage applied to cement mortars cured for 28 days.

amount of MWCNTs were incorporated in the cement mortar specimens. Fig. 2(a) shows the pristine sand; Fig. 2(b) and (c) show sand after it has been pre-coated with the MWCNT-latex films, where the MWCNT content was 0.125 wt% and 0.250 wt%, respectively. The darkness of the sand became more pronounced as MWCNT content increased. These specimens are referred to as "CNT sand cement mortar" hereafter.

2.2. Cement mortar casting

Cement mortar specimens were cast using dispersed MWCNT solutions and sand pre-coated with MWCNT-latex thin films, as was mentioned in Section 2.1. First, both types of cement mortar were produced by dry-mixing cement and sand (1:2.5 by weight) for 2 min using a mixer with a maximum output of 700 W (Fig. 3(a)). For CNT sand cement mortar, the film-coated sand was used, as shown in Fig. 3(b). Second, the dry-mixes of cement and sand were combined with distilled water and mixed for 2 min, except the CNT solution cement mortar employed the CNT aqueous dispersion. Fig. 3(c) shows the mixing process of the CNT solution cement mortar, while Fig. 3(d) represents the mixing process of the CNT sand cement mortar.

Compaction after the mixing process was divided in two phases, similar to the process of producing regular cement mortars. The mold was filled halfway with mortar and compacted, followed by the insertion of two steel electrodes within the CNT cement mortar, as shown in Fig. 3(e). Here, the electrodes were placed in the specimen at 10 mm intervals, with dimensions of 50 × 70 mm^2. After the mold was complete filled, a T-type thermocouple was then inserted in the center of each specimen, as shown in Fig. 3(f). Thereafter, the mold was compacted using a tamper, and the remaining mortar was piled on top of the mold. After the compaction process, special care was taken to ensure that there were no openings around where the thermocouple was placed. The mold was then placed in a thermo-hygrostat for 24 h, as shown in Fig. 3(g). Fig. 3(h) shows the wet curing of the specimen in a tank with clean water (23 ± 2 °C) after removing it from the mold. The specimens were cured in water throughout curing and completely dried afterwards. The specimens were rectangular of 50 × 50 × 50 mm^3 in size, and the water/cement ratio was fixed at 0.5 for all specimens.

2.3. Thermal response characterization

This study then proceeded with characterizing the thermal characteristics of both sets of cement mortar specimens by measuring their internal temperature change as a function of the type of specimen, CNT content, curing age, and supplied voltage. In total, three specimens of each type were fabricated for each parameter set in order to characterize their internal temperature change under different conditions. Since, there is no standard for temperature tests for cement mortar, the

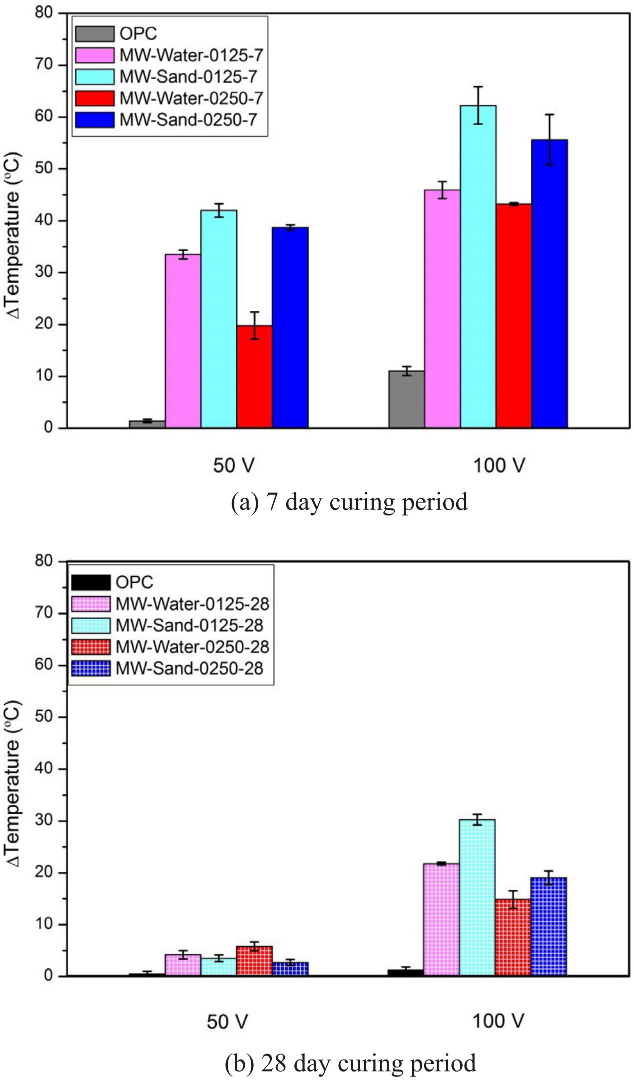


Fig. 7. Temperature changes of CNT cement mortars with changes in voltage.

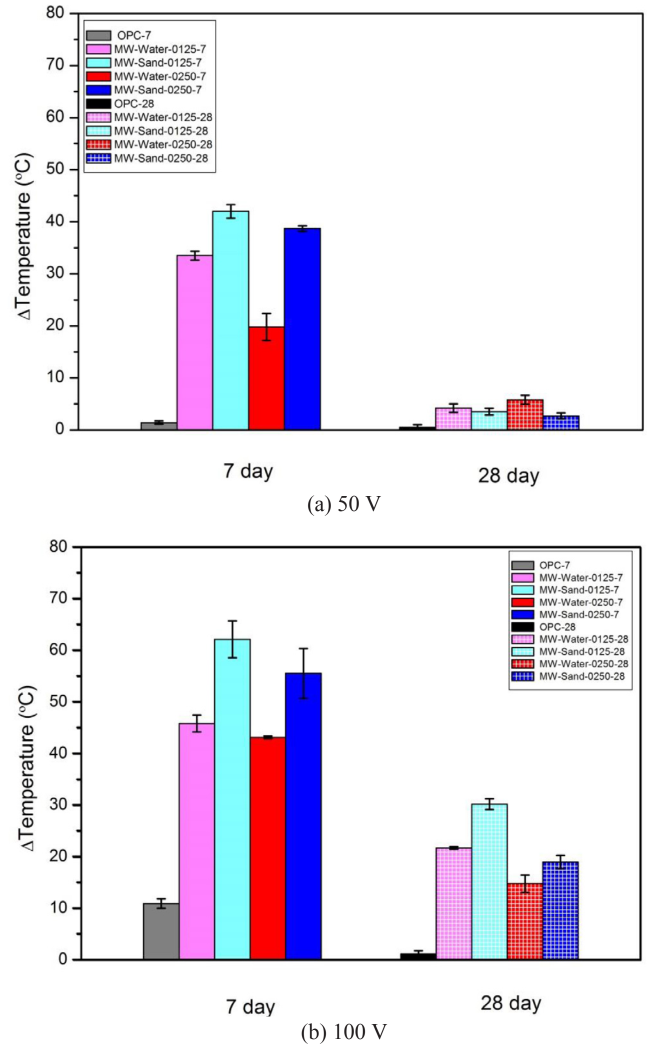


Fig. 8. Temperature changes of CNT cement mortars with changes in curing age.

specimens were prepared to be the same size as those used in compressive strength experiments, which were in accordance with the ASTM C109 [30].

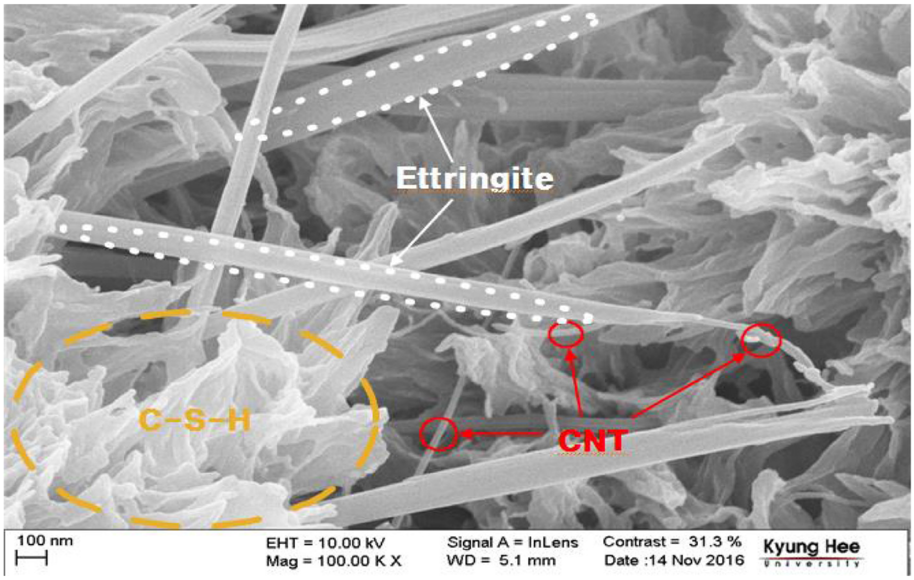
Table 1 shows the parameters of the nano-cement mortar temperature tests. The test groups were divided according to production method. Group #1 is the control group with ordinary portland cement (OPC) mortar. Group #2 is the CNT solution-based cement mortar. Group #3 is the CNT sand-based cement mortar. The CNT content in Groups #2 and #3 were 0.125 wt%, and 0.250 wt% to facilitate direct comparisons. The wt% is the weight ratio of MWCNT particles to cement weight. The cement mortars in all groups were assigned a curing age of either 7 or 28 days, as well as a supplied voltage of either 50 V or 100 V. The first segment of each specimen name indicates the material used. As mentioned earlier, 'OPC' refers to ordinary Portland cement mortar and 'MW' indicates MWCNT. The second segment indicates the CNT mixture method. "Water" indicates that specimens were casted using MWCNT dispersed solutions, whereas "Sand" indicates that sand pre-coated with MWCNT-latex films were used. The third segment in the specimen names, namely "0125" and "0250", refers to CNT content, which are 0.125 wt% and 0.250 wt%, respectively. The last segment indicates the curing age; "7" indicates a 7-day curing period, while "28" means a 28-day curing period. The specimens were produced based on methods used for compressive strength experiments [30].

To ensure that the electricity supplied to the CNT cement mortar does not flow externally, the specimens were placed on insulating rubber as shown in Fig. 4(a). Fig. 4(b) shows the thermocouple inside the CNT cement mortar connected to the data logger. The steel electrodes within the CNT cement mortar are connected to a power supply, as shown in Fig. 4(c). The positive and negative electrodes of each specimen were set equally, and a certain amplitude of voltage was supplied. The temperature tests involved supplying voltage to each specimen for 6000 s and measuring the corresponding temperature changes throughout the entire duration of the experiment.

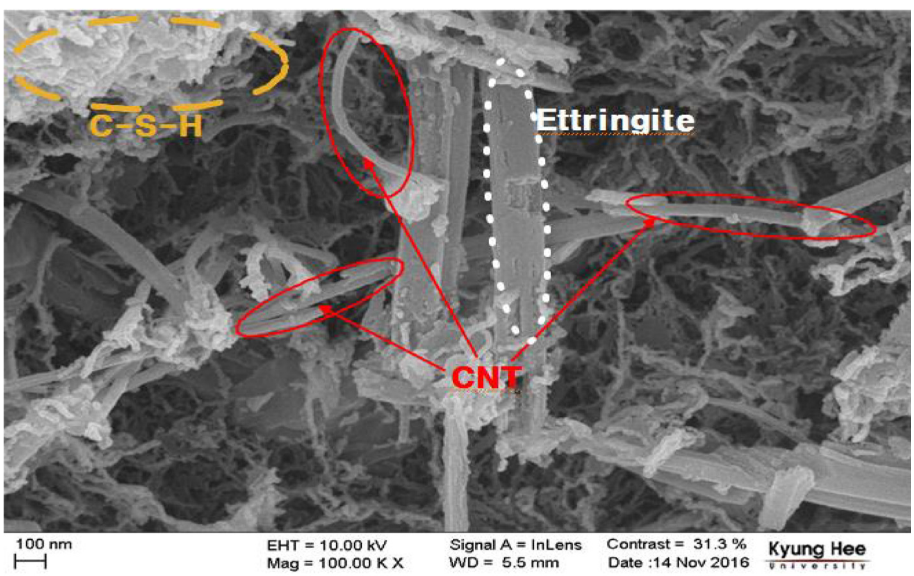
3. Results and discussion

3.1. CNT cement mortar thermal characteristics

This study analyzed the effects of how MWCNTs were incorporated in cement mortar, CNT content, curing age, and applied voltage on the thermal characteristics of CNT cement mortar specimens. The maximum temperature was measured in specimens for each parameter set, and an average value of the three specimens tested in each sample set was reported as the temperature change of the specimen (ΔT). Based on the temperature changes of the specimens for each parameter set, the temperature test results of the CNT cement mortar were compared and analyzed. This study also used field emission scanning electron microscopy (FE-SEM) to image the inner cross-section of the CNT cement



(a) CNT solution cement mortar



(b) CNT sand cement mortar

Fig. 9. SEM image of CNT cement mortar.

mortar to analyze the effect of CNT dispersion on the inner temperature changes of the CNT cement mortar.

The temperature test results of the CNT solution cement mortar are presented in Table 2. In all cases, the changes in temperature of the CNT cement mortar were greater than the changes in temperature of an OPC specimen, regardless of the CNT mixing method, CNT content, curing age, or supplied voltage. This result confirms that the incorporation of CNTs enhance the self-heating capabilities of cement mortars.

The temperature change of the specimen cured for 7 days and subjected to 50 V is shown in Fig. 5. While the temperature change for the OPC mortar was only 1.4 °C, the results suggest that mixing CNTs with the nonconductive OPC mortar enhances their thermal properties. The change in temperature for the 0.125 wt% CNT sand cement mortar was 42.0 °C and for the 0.125 wt% CNT solution cement mortar was 33.3 °C. In the case where CNT content was 0.250 wt%, the temperature change for CNT sand cement mortar was 38.6 °C while for the CNT solution cement mortar was 19.4 °C. With all other parameters equal, the rise in temperature of the 0.125 wt% CNT sand cement mortar was 26% higher than the 0.125 wt% CNT solution cement mortar. The rise in temperature of the 0.250 wt% CNT sand cement mortar was 192% higher than that of the 0.250 wt% CNT solution cement mortar. It is hypothesized that this is due to more effective dispersion of CNTs when CNTs were pre-dispersed in a thin film matrix and used to modify the interfacial transition zone of concrete. Results from Gupta et al. [28] also showed that this procedure yielded electrically conductive cementitious composites suitable for sensing applications. In general, spray-coating films onto sand led to more effective modifications of the thermal characteristics of cement mortar.

Fig. 6 presents the change in temperature for specimens cured for 28 days and excited with 100 V. The temperature change for the OPC mortar (control) case was 1.1 °C. The temperature changes of the CNT cement mortar specimens, regardless of CNT content, were all greater than the OPC control case. In particular, when CNT content was 0.125 wt%, the temperature change of the CNT sand cement mortar was greater than the average value for CNT solution cement mortar by approximately 37%. The rise in temperature of the 0.250 wt% CNT sand cement mortar was 27% higher than that of the 0.250 wt% CNT solution cement mortar. Similar to before, these results confirmed that pre-coating sand with the MWCNT-latex thin films was a more effective technique for modifying the thermal characteristics of CNT cement mortar, as compared to directly incorporating CNT dispersions during casting. With the increase in CNT content from 0.125 wt% to 0.250 wt %, the temperature of the CNT sand cement mortar was 10.7 °C lower while that of the CNT solution cement mortar showed a 6.8 °C decrease. It is hypothesized that the temperature change of cement mortar decreased as CNT content was increased due to agglomeration of CNTs, thereby leading to ineffective conductive pathways within the cement mortar.

The results for temperature changes as voltage increased from 50 V to 100 V is shown in Fig. 7. As supply voltage was increased, the temperature change of CNT cement mortars increased. This result makes sense since a higher voltage would induce greater resistive heating. For specimens cured for 28 days, the temperature change of CNT sand cement mortar was more than 19.7 °C, and the temperature change of CNT solution cement mortar was more than 9.0 °C (with respect to OPC mortar). The temperature changes of CNT solution cement mortar was slightly lower than that of CNT sand cement mortar.

Fig. 8 shows the temperature changes in CNT cement mortars cured for 7 and 28 days. For the 7-day sample sets, all cases exhibited self-heating response, and the temperature of CNT sand mortar (due to 100 V) increased up to 61.7 °C. However, after curing for 28 days, the temperature of CNT sand cement mortar increased up to 29.4 °C. As hydration of cement mortar progressed, the thermal conductivity of all cement mortars decreased. Thus, the internal temperature of cement mortar did not increase as much.

3.2. CNT dispersion

A cross-sectional analysis was conducted with FE-SEM in order to qualitatively assess the level of CNT dispersion in CNT cement mortar. The FE-SEM (Merlin) applies electronic beams to the sample and collects images from the secondary and backscattered electrons, enabling structural analysis via X-ray. Unlike other SEMs, FE-SEM allows for high-definition scanning, thereby enabling detailed examination and analysis of nanoparticles. The specimens for scanning were small fragments of the cement mortar, which were attached to a carbon tape and then inserted in the vacuum chamber of the microscope. Because imaging occurred in a vacuum, moisture was removed from the specimen prior to imaging to avoid technical errors. The magnification of the images was set to 100,000×, and specimens with the highest temperature changes were examined. These specimens were MW-Water-0250-7 in Group #2 and MW-Sand-0125-7 in Group #3.

Fig. 9(a) shows the FE-SEM image of the CNT solution cement mortar (MW-Water-0250-7). The image shows that CNTs, in the form of a thin thread, bound with calcium silicate hydrate (C-S-H) or Ettringite in a straight line. However, the bond between CNTs and C-S-H or Ettringite was only partially confirmed. It should be noted that the bonding of C-S-H and Ettringite was more frequently observed. It appeared that CNTs in the CNT solution cement mortar exhibited some agglomerations, which were likely due to inadequate dispersion during processing or agglomeration during mixing/casting. The FE-SEM image of the CNT sand cement mortar (MW-Sand-0125-7) is shown in Fig. 9(b). Aside from the CNTs binding with C-S-H and Ettringite, it was also observed that CNTs were in general evenly dispersed. In general, Fig. 9(b) suggests that better dispersion of CNTs in cement mortar could be achieved by initially incorporating CNTs in a polymer-based nanocomposite and then spray-coated onto dry sand for scale-up. By achieving better CNT dispersion, the result of the bulk material (i.e., CNT cement mortar) exhibited better thermal response characteristics and confirmed the main hypothesis of this study.

4. Conclusions

In this study, the thermal characteristics of two different CNT cement mortars were investigated experimentally. The first sample set entailed pre-dispersing CNTs in an aqueous solution and then directly using the dispersed solution to cast cement mortars. The second sample set focused on modifying the interfacial transition zones in cement mortars. This was achieved by preparing a CNT-latex-based ink solution and then spray-coating the ink onto dry sand to form thin films. Numerous specimens were cast by varying different parameters, namely how CNTs were incorporated in cement mortar, CNT content, curing age, and applied voltage. Based on the temperature test results, the effects of each parameter on the thermal characteristics of CNT cement mortar were analyzed, which led to the following conclusions:

1. CNTs were more effectively dispersed when they were incorporated in thin films and pre-coated onto dry sand, as opposed to dispersing them in water and using the solution during casting. Scanning electron microscopy studies showed that CNTs were more effectively dispersed in CNT sand cement mortar. Enhanced CNT dispersion led to more effective modifications of the thermal characteristics of CNT cement mortar. Moreover, the SEM images indicated that CNTs mixed with sand led to a denser, percolated network in cement mortar.
2. The temperature test results indicated that 0.125 wt% CNT cement mortar exhibited higher changes in temperature, as compared to the 0.250 wt% sample sets. This signified that a CNT content over a certain threshold leads to agglomeration within the CNT cement mortar, resulting in compromised thermal characteristics.
3. After curing the cement mortar mixes, CNT cement mortars could exhibit modified thermal properties by exciting the specimens with an applied voltage signal. In order to observe significant changes in thermal properties, at least 100 V of direct current should be applied to raise the temperature regardless of other parameters. After specimens cured for 28 days, CNT sand cement mortar could self-heat to higher than 29° C (by supplying 100 V).

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