Surface Modification of Carbon Nanotubes Using Carboxymethyl Cellulose for Enhanced Stress Sensing in Smart Cementitious Composites

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***Abstract*— Carbon nanotubes (CNTs) when used as additives in cementitious materials are known to enable the piezo-responses making them smart materials. The piezo-responses can potentially be used to monitor the applied forces, stress, and the resulting performances of the associated materials. An effective and reproducible method for smart cementitious material production needs to ensure uniform dispersion of CNTs within the cementitious base, and that should lead to better sensitivity and consistency in sensing by the smart cementitious materials. This paper presents a surface modification method of CNTs using carboxymethyl cellulose (CMC) to improve the performance of the CNT-modified smart cementitious materials for stress sensing. Compared to the two commonly used CNTs dispersion methods, the direct mixing and the surfactant surface treatment methods of dispersing CNTs, the new CMC surface modification method developed within this study significantly increased the stress detection sensitivity and consistency, and reduced the measurement hysteresis.**

***Index Terms*— Carbon Nanotube (CNT); Carboxymethyl Cellulose (CMC); Cementitious Material; Dispersion Effectiveness; Piezo-response.**

I. Introduction

With the extensive application of cementitious materials in critical infrastructure, it has become imperative to be able to remotely observe, monitor, and evaluate of the structural health conditions of these structures in real time, and thus minimize in-person inspections and associated human errors [1]. Civil structures' structural health monitoring (SHM) is essential to enable timely maintenance and inspection and enhance the structural life expectancy and structural safety [2]. SHM makes the predictions of calculations and theoretical structural model possible to verify with the results from the actual measured stresses in the structures.

The application of low-cost sensors on the civil infrastructure to accurately detect and acquire structural performance data such as stress or strain plays a critical role for an effective SHM system [3]. To detect the resulting stress in a structure from external loading, various tools have been provided with the recent advances of smart sensor technologies which include sensors based on acoustic or ultrasonic waves, guided waves, and electrical resistance [4], [5]. However, with a large number of structures to be monitored, the substantial costs and complex signal processing algorithms involved limit the numbers of local sensors to be applied in the structures and made the individual sensor-based solutions hardly scalable, restricting the measurement accuracy and impeding the wide applicability of such individual sensor- based solutions [6].

To overcome the current limitations of the SHM system, smart materials with specific electrical sensing properties of materials can be developed by adding additives. The use of nanomaterials as additives can make cementitious materials smart, with not just improved engineering properties only but also impart sensing properties to the materials. One of the popular nanomaterials which has been investigated for smart cementitious materials is carbon nanotube. The seamless hexagonal network and unique C-C covalent bonding enables carbon nanotubes (CNTs) to exhibit extraordinary mechanical properties. The specific strength of CNTs is reported to be as high as 48,000 $kN \cdot m \cdot kg^{-1}$ in comparison to 154 $kN \cdot m \cdot kg^{-1}$ from high-carbon steel [7], [8]. CNTs high strength will introduce compatible or improved mechanical properties of the

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smart materials compared to the materials without CNT addition. More importantly, CNTs also exhibit high electrical conductivity (10^6 -10^7 S/m) [9] and piezo effects under loading [10], [11] in the axial direction [12], [13], which make them promising candidates for developing smart cementitious materials.

For any CNT-enabled smart cementitious materials, the percentage of CNTs plays a significant role on the electrical properties and mechanical properties and also microstructural characteristics of the resulting composite CNT-cement materials [14]. Specifically, the piezo electric properties of CNTs are of interest for sensing. The water/cement (W/C) ratio and the CNT concentration level (% CNT) affect the piezo responses of smart cementitious composites with the piezo responses first increasing and later decreases with the increase of CNTs concentration. Higher (W/C) ratio helps in improving the piezo responses [15]. Approximately 0.6 W/C ratio and 0.1% CNTs by the weight of cement are reported to yield the highest compressive strength with good piezo electric properties of CNT-cement composites [15]–[19].

When mixing CNTs with cement for enhancing either mechanical or sensing properties, it is commonly assumed that by simply mixing of untreated CNTs into water and then into cement will result in a uniform and homogenous mixture [20]. However, untreated CNTs agglomerate into lumps in water as they are hydrophobic in nature and because of the existence of large Van der Waals force, and thus, may lead to non-uniform dispersion in cementitious materials. This phenomenon would lead to a potentially disconnected networks of CNTs making the composite less conductive or non-conductive. This would result in low piezo responses and inconsistency in sensing the applied forces as well as low structural performance of the composites [21]–[23]. More effective methods to mix CNTs into the cement matrix (to achieve uniform dispersion) are needed for developing better CNT-enabled smart cementitious materials [24].

The dispersion of CNTs in cement matrix can be done by two common approaches to improve the dispersion, one mechanical approach and the other being chemical approach. For the mechanical approach, high-shear mixing devices or ultrasonification were employed to mix CNTs in the (W/C) matrix. However, the use of these mechanical device is time-consuming and less efficient [25]. Furthermore, ultrasonication may lead to fragmentation of CNTs which would result in decreasing the aspect ratio of CNTs leading to unstable (poor) dispersion [26]. The chemical and physical approach to improve CNTs mixing effectiveness includes both covalent and noncovalent surface treatments of CNTs [27]. Various chemical moieties using the covalent surface treatment method to functionalize CNTs such as acids to improve the CNTs' solubility in aqueous solution [28]. The chemical functionalization of CNTs may also increase defects on the nanotube surfaces consequently altering the electrical properties of carbon nanotubes to enhance its sensing properties [29]. Acid treatments have significantly improved the sensitivity of the treated CNTs-cement composite for force sensing [19], [30]. However, the use of corrosive and hazardous chemicals such as acid in the chemical functionalization process and the requirements of high temperature have environmental consequences and limit the wide use of this approach.

In noncovalent surface treatment method, CNTs adsorb chemical moieties (e.g., uncharged surfactants) onto surfaces and that helps in mixing CNTs in water/cement matrix [31], and has shown to improve the piezo-response of the cementitious composite to some extent [32]. Surfactants such as Sodium Dodecylbenzene Sulfonate (NaDDBS), Sodium Dodecyl Sulfate (SDS), superplasticizer, and silica fumes have been investigated [33], [34]. The comparisons on various surfactants indicate that NaDDBS is a more stable surfactant and have higher sensitivity to the external force compared with other surfactants [33]. Recently, a co-polymer (octenyl succinic anhydride (OSA) modified tapioca starch) was reported to be effective in dispersing iron nanoparticles in water [35]. The authors investigated using the OSA-modified tapioca starch as noncovalent surface treatment may have potential to enhance CNTs' dispersion in cementitious matrix [36]. OSA-modified tapioca starch coated CNTs showed good sensitivity toward applied force and that was compatible with NaDDBS surface-treated CNTs, but OSA-modified tapioca starch coated CNTs exhibited better consistency. In all these investigations, multi-walled carbon nanotubes (MWNTs) were employed as they are more sensitive to stress changes compared with single-walled nanotubes (SWNTs).

Although various CNTs surface treatments were investigated to improve and enhance the sensitivity of smart cementitious materials incorporated with CNTs, the application of smart cementitious materials such as smart concrete is not yet used in practice mostly due to high cost, low sensitivity, inconsistent measurements, poor durability, poor compatibility, and signal processing inaccuracy [37]. For this study, a new surface modification method using the carboxymethyl cellulose (CMC) polymer was developed to surface treat the CNTs to be incorporated in smart cementitious composites to improve the stress sensing sensitivity, consistency, and hysteresis for its potential wider or larger-scale use of SHM in concrete or any structures involving cementitious materials. Comparisons between the new CMC surface modification method, the NaDDBS surfactant, and the direct mixing method were made to validate the effectiveness of the new method.

The organization of the paper is as follows: Section 2 introduces the polymer selection and the CNT surface treatment method, Section 3 presents the methodology for the laboratory validation, Section 4 discusses the experimental setup, Section 5 discusses and presents the experimental results, and Section 6 lists the conclusions and outlines the potential future work.

II. Polymer Selection and CNT Surface Modification

A. Polymer Selection

A more effective noncovalent surface treatment on CNTs requires the appropriate polymers. Several polymers which had been previously applied in cementitious materials as concrete additives to increase durability or for changing the setting/hardening time were considered for this study.

Polyacrylamide (PAA), sodium polyacrylate (SP), polyethylene glycol (PEG), and carboxymethyl cellulose (CMC) were screened based on their properties (Table I) for their potential use for surface modification.

Among the screened polymers mentioned above, the PAA reacts with metals such as reinforcement steels (Table I), thus, its application is limited in conventional cementitious materials [38]–[40]. In addition, all the PAA, SP, and PEG have high water absorbance [41]–[45], and the high water-absorbance by the surface modifier is not desired for smart composites as that may increase the Van der Waals force among the coated CNTs and resulting in agglomeration [46].

On the other hand, the organic polymer, CMC, has been used as retarder to extend the setting time of concrete [46]. When added as additive (retarder), CMC is reported to improve bending and compressive strengths as well as fracture toughness of the Portland cement concrete in addition to decreasing porosity and water absorbance of the mix [47]–[49]. A bare 0.5% CMC by mass of cement in cementitious materials has been claimed to be the optimal for enhanced compressive strengths (2,200 Psi) and acceptable setting time (4 h). Above 0.5% CMC, the setting time of the cement mix increases dramatically [46]. The CMC in combination with CNTs is reported to increase the interfacial bonding of CNTs to cementitious materials and the CNTs remained stable for more than three months and does not experience significant changes in physical or chemical characteristics [24], [50]. Given its promising results in improving structural properties, the CMC was selected as the candidate polymer to modify the surface of CNTs to be used and to enable the smart cementitious materials for potential stress sensing in this study.

B. Surface Modification of CNTs using CMC

In general, the electrical and mechanical properties of single walled carbon nanotubes (SWNT) can change when functionalized, due to the structural defects occurred by C=C bond breakages during chemical processes. However, intrinsic properties of carbon nanotubes can be preserved by the surface modification of MWNTs, where the outer wall of MWNTs is exposed to chemical modifiers. In addition, the cost of the MWCNTs is much cheaper compared to SWCNTs. For potential massive amounts of CNTs required in the application of smart cementitious materials as in this study, the cost-effective solution of MWCNTs was used for this study that are supplied by SkySpring Nanomaterials, USA with predetermined properties have been used in a previous study [36]. The MWNT particulates have an outer diameter of 50-100nm with an inner diameter of 5-10nm and has lengths of 5-20μm.

According to the literature, 0.1% of CNTs by weight to cement was the most effective for mechanical properties and sensing purpose [15-19], which was selected to be the CNTs percentage in this study. Although it was observed from literature that the optimal percentage of CMCs for mechanical properties is 0.5% by the mass of cement [46], since for the first time, this study used CMCs to coat CNTs for improving its sensing performance, the influences of various CMCs percentages were investigated by changing the CMCs from 0.1%, 0.3%, 0.5%, and 1%.

For the 0.5% CMC-water solution, it was prepared by mixing 0.5% (5 g) of dry CMC with 1,000 mL of deionized (DI) water. While the DI water was stirred with the magnetic stirrer, the CMC was slowly added into the center of the vortex until the CMC was completely mixed in the DI water. To modify the CNTs, 50 mL of CMC-water solution was mixed with 0.4 g of CNTs in a 50-ml test tube and placed on a rotator for 72 hours to ensure a proper coating of the polymer onto the CNTs. In this study, we adopted the 72 hours of rotating to ensure proper surface coating of CNTS with CMC according to practices used for co-polymer surface modification on nano particles [35, 36]. Future work to optimize the rotating time of the CNT-CMC solutions for proper dispersion is needed to be performed. After 72 hours, the test tube with CMC coated CNT solutions were placed into a centrifuge for approximately 5 minutes and rinsed with an ample amount of DI water to eliminate excess/unattached CMC.

The CMC coated CNTs for other CMC percentages (0.1%, 0.3%, and 1%) followed similar procedures using corresponding weights of CMCs. For ease of storage and handling, separate plastic vial was used to transfer and store the content of the test tubes. For future use, each vial was added with fresh DI water before storing in the plastic vial.

C. Surfactant Modified (SM) CNTs

To compare the effectiveness of the CMC coated CNTs for sensing, in this study, surfactant modification using NaDDBS, was also applied to modify the CNTs. For the preparation of surfactant, NaDDBS, modified (SM-) CNTs, the input surfactant concentration NaDDBs (supplied by Sigma-Aldrich, USA) with a critical micelle concentration in water was 1.4 x 10^{-2} mol/L [33]. The NaDDBS (1.17g) was mixed with 240 ml of DI water for 5 minutes using a magnetic stirrer. The surfactant modified (SM-) CNTs (0.4 g) were ultrasonicated for 2 hours in an aqueous solution to get the SM-CNTs solution.

D. Microstructure of Bare and Modified CNTs

To investigate the microstructure and morphology of the bare (not coated with CMC), the CMC coated CNTs (0.5% of CMC), and surfactant surface treated CNTs, the Scanning Electron Microscope is the best tool to observe the changes in microstructure and morphology of the CNTs [51]–[53]. The bare and modified CNTs samples were mounted on aluminum mounts using colloidal silver paint or coverslip and then coated with a conductive layer of carbon in a high-vacuum evaporative coater (Cressington 208c, Ted Pella Inc., Redding, California, USA). Images were obtained with a JEOL JSM-7600F field emission scanning electron microscope (JEOL USA Inc., Peabody, Massachusetts) operating at 2 kV. The SEM micrographs produced are as shown in Fig. 1(a, b, c), which were taken under the same resolution. Sample charging is a common problem in SEM imaging. Charging occurs when there is no conducting path for electrons to flow from the sample surface to ground, typically the sample holder. As such, the white spots that are present in Fig. 1(a, b, c) are due to the charging issues.

From Fig. 1 (a), it can be seen that the bare CNTs have outer diameters varying from 50 nm to 100 nm and they are noticed to be randomly agglomerated together. Fig. 1 (b) shows that once coated with CMC, the outside diameters of the coated CNTs are almost uniform (~100 nm). The coated CNTs are

observed to be not being agglomerated. For the surfactant surface treated CNTs in Fig. 1(c), the fragmentation of CNTs due to the ultrasonication can be noticed.

III. Sample Preparation and Experimental Setup for Sensing Tests

To evaluate the stress sensing performances of the CMC coated CNT impregnated cementitious materials, mortar samples were prepared with CMC coated CNTs, the bare CNTs, and the surfactant, Sodium Dodecylbenzene Sulfonate (NaDDBS) modified CNTs. Bare and NaDDBS-modified CNTs are used in mortars for currently available smart cementitious materials [19], [20]. The preparation of the samples and the experimental setup are detailed below.

A. Preparation of CMC Wrapped CNT-, Surfactant Modified (SM) CNT-, and Bare CNT-Cement Mixtures

The preparation of the CMC coated (CMC-) and surfactant modified (SM-) CNTs aqueous solution followed the same procedure as in Section II-B and II-C. The bare CNTs-water solution was prepared by directly adding the CNTs (0.4g) into the 240ml of DI water and mixed with a magnetic stirrer.

With the CNTs-water solution prepared, 400g of Portland cement were then added to the prepared CNT (CMC-, SM-treated, and bare) aqueous solutions to produce the CMC-, SM- and bare CNT-cement mixtures with 0.1% CNTs of cement weight. Specific properties of Portland cement (Holcim, Inc, USA) used for this study can be found on Table II. All cement mixtures had a W/C ratio of 0.6 in all samples in this study. As indicated in Section II-B, all three methods use the same CNTs percentage of 0.1% [15]–[23]. In addition, a control group without CNTs (CN) was also prepared by mixing cement of 400g and water of 240ml.

B. Sample Matrix

CMC-, SM-, bare CNT-, and CN- cement mixtures test mortar blocks were prepared using a mold with dimension of 50mm × 50mm × 50mm. Two electrical wires with their naked ends are placed and embedded 2.5cm apart in each block for piezo-response measurement (Fig. 2). All samples were made and prepared at room temperature (22°C ± 2°C) and cured in water for 7 days [54]. The curing time of 7 days was selected based on the common practice of smart cement paste samples used in field so that the results can be compatible with previous studies.

As shown in Table III, for CMC testing groups, three test samples were made Group CMC (A) that has a 0.1% CMC, Group CMC (B) with 0.3% CMC, Groups CMC (D) and (CMC (E) which have 1% of CMC. These testing groups will be used for CMC concentration sensitivity study. In addition, six test samples were made for Groups CMC (C)-, SM-, and bare CNTs for the ease statistical validation. For the control group, Group CN, three samples were prepared. Samples in Group CMC (C) had a CMC concentration of 0.5% and they were used for both CMC concentration study and method comparison study. Fig. 3 shows the samples for method comparison study. Thus, a total of 33 block samples were prepared for sensing tests.

C. Experimental Setup

To test the stress sensing performances of mortars impregnated with CNTs modified with different methods, dynamic compression loads were applied on individual sample blocks (Fig. 4(a)) using MTS 809 Axial/Torsional Tester (Materials Testing Systems Inc., USA). The loading profile were subjected to all samples at room temperature (Fig. 4(b)). 12 cycles with an average value of 1,000 N and an amplitude of 2,000 N were used for the applied cyclic compressive load. Compressive forces applied was indicated with the negative sign. The frequency of 0.1 Hz was used for the loading cycle. The applied stress (σ in Pa) can be calculated by dividing the applied load (P in N) with the area of the sample (A in mm, which is 2,500 mm^2). Thus, the applied dynamic stress was varying between 0 and 0.8 Pa. A digital bench multi-meter (BK 5492B, B&K Precision Inc., USA) was used to record the piezo-responses changes under the dynamic forces subjected to further analyses.

D. Data Analysis Method

The recorded piezo-responses (R(t) in microvolts, μV) from the digital bench multi-meter were input into a commercial statistical software, SAS, for further data analysis. Since each sample undergo 12 cycles of cyclic compressive loads and each group of CMC (A), CMC (B), CMC (D) and CMC (E) had three samples, a total of 36 cycles were recorded for each group while each group of CMC (C), SM, and DM had six samples, a total of 72 cycles were recorded for each group. The CN group had three samples and generated 36 cycles of recorded data. To obtain the loading and the unloading sensitivity of the stress sensing CNT-impregnated cement mortar, the recorded piezo-responses were subjected to post-analyzed by having the piezo-responses divided based on the maximum stress obtained from the applied loads, σ_{max} (with unit in Pa, 0.8 Pa). A function of piezo-response and stress (Eq. 1) can be used to describe a cement mortar block's dynamic response ($\Delta R(t)_i$, the trough response) [36],

$$\Delta R(t)_i = R(t)_i/\sigma_{max} \quad (1)$$

The i loading cycle for loading and unloading stress sensitivity ($S_{L,I}$, $S_{U,i}$) of the smart cementitious material was calculated (Eq. 2) as [36]:

$$S_{L,i} = \left| Min(\Delta R(t)_i) - Max(\Delta R(t)_i) \right|;$$
$$S_{U,i} = \left| Max(\Delta R(t)_i) - Min(\Delta R(t)_{i+1}) \right|. \quad (2)$$

In Eq. 2, Min(ΔR(t)i) and Max(ΔR(t)i) is defined as the trough response and peak response of each cycle respectively. The average loading and the average unloading stress sensitivities, hysteresis and consistency/repeatability were employed to evaluate the dynamic responses of the smart cementitious materials.

IV. Experimental Results

A. The CMC Surface Modification Method

Figs. 5 (a~e) show the dynamic responses (ΔR) of the samples in Groups CMC (A~E) under the cyclic loads based on Eq. (1), respectively. The Groups CMC (A~A) had CMC concentrations of 0.1%, 0.3%, 0.5%, 1%, and 1% CMC but no

CNTs. Figs. 6 (a~e) illustrates the calculated average loading and unloading stress sensitivity of the samples in Groups CMC (A~E) following Eq. (2), respectively. The average loading sensitivity of Groups CMC (A~E) were 0.0209 μV/Pa, 0.0327 μV/Pa, 0.0754 μV/Pa, 0.0319 μV/Pa, and 0.0025 μV/Pa. The average unloading sensitivity of Groups CMC (A~E) were found to be 0.0201 μV/Pa, 0.0326 μV/Pa, 0.0757 μV/Pa, 0.0301 μV/Pa, and 0.0025 μV/Pa. It is clearly seen that for the same CNTs concentration (0.1%), the stress sensitivity of the CMC-CNT modified cement paste increases as the CMC concentration increases before CMC concentration of 0.5%. However, after 0.5% of CMC concentration, adding more CMC would negatively impact the stress sensitivity of the modified smart cement paste. Thus, 0.5% of CMC had been identified as optimal CMC concentration which agrees with previous studies on the optimal concentration for mechanical properties. Group CMC (C) will be used to compare with other dispersion methods in the following sections. In addition, the comparison between Figs. 6 (d) and (e) also indicated that adding bare CMC in cement mixture increased the piezoresponses of the samples, which is one of the reasons for the improved stress sensitivity for the CMC-CNTs modified smart cement paste in addition to the potential enhanced dispersion effectiveness.

B. The Direct Mixing Method

The ΔR for Group DM under cyclic loads varied between 0.005 and 0.05 μV/Pa (Fig. 7(a)). The average loading sensitivity ($S_{L,i}$) of Group DM samples was 0.0055 μV/Pa. The unloading sensitivity ($S_{U,i}$) was 0.0051 μV/Pa. The standard deviation for the average loading sensitivity is 0.0051 μV/Pa and the standard deviation for the average unloading sensitivity is 0.0048 μV/Pa (Fig. 7(b)). The low stress sensitivities and the relatively high standard deviations of Group DM of the direct mixing method might be induced by its ineffective dispersing of CNTs in the piezo-sensitive cementitious material.

C. The Surfactant Surface Treatment Method

The ΔR of the SM Group varied between 0.02 to 0.052 μV/Pa (Fig. 8(a)). For Group SM samples, the average loading sensitivity ($S_{L,i}$) was 0.0077 μV/Pa. The unloading sensitivity ($S_{U,i}$) was 0.0072. The standard deviation for the average loading sensitivity is 0.0058 μV/Pa and the standard deviation for average unloading sensitivity is 0.0060 μV/Pa (Fig. 8(b)). Compared with Fig. 7(b), the stress sensitivity of Group SM is slightly higher (~40%), indicating that the surface modification using surfactant might slightly increase the dispersion of CNTs in cement base. However, compared with Fig. 5 (d), the stress sensitivities of Group SM were demonstrated to be much lower than Group CMC (0.5% CMC). This could be due to the fragmentation of CNTs during the ultrasonification process when mixing the CNTs with NaDDBS resulted in less effective dispersion of CNTs.

D. Control (Direct Mixing Method)

The ΔR from Group CN (without the addition of CNTs) was negligible and varied between 0.004 and 0.012 μV/Pa (Fig. 9(a)). For the samples from Group CN, the average loading sensitivity ($S_{L,i}$) was 0.0019 μV/Pa. The unloading sensitivity was 0.0017 μV/Pa. The standard deviation for the average loading sensitivity is 0.0014 μV/Pa and the standard deviation for average unloading sensitivity is 0.0012 μV/Pa (Fig. 9(b)).

V. Comparisons between Different Methods

A. Stress Sensitivity

Average stress sensitivities of the cement mortar samples were determined and compared (Fig. 10 and Table IV). For the samples from Group CMC (C)-, SM-, and DM- CNTs, the average stress sensitivity was 0.0756 μV/Pa, 0.0075 μV/Pa, and 0.0055 μV/Pa, respectively. For the control (Group CN), the average stress sensitivity observed was almost negligible (0.0018 μV/Pa and a standard deviation of 0.0013 μV/Pa). The percentage difference between any two methods can be calculated using formula percentage difference [36], Eq. (3):

$$Percentage\ difference = 100 \times \frac{(M_{method\ 1} - M_{method\ 2})}{M_{method\ 1}} \quad (3)$$

in which, $M_{method\ i}$ is the measured value from the ith method. Based on Eq. (3), the CMC surface modification of CNTs was found to increase the average stress sensitivity by 14 times in comparison to the bare CNTs samples (Group DM), and approximately 10 times for surfactant treated CNTs samples (Group SM). The improved stress sensitivity of the CMC surface modification of CNTs might be resulted from a combined effect of the slight piezoresistence induced by the bare CMC as indicated in Fig. 6 (e) and more dispersed CNTs in the cement mixture.

B. Hysteresis

Hysteresis for a sensing material indicates the lag in responding under loading and unloading conditions. For a cyclic loading, hysteresis describes recovery rate of a material when the initial load applied is withdrawn. Thus, the output of response with high hysteresis is a function of the loading history. A material's hysteresis is heavily influenced by the load history by demonstrating how nonlinear or plastic the material is. For a material with high hysteresis, the recovery of strain in a material subjected to a stress during its unloading cycle is incomplete due to energy consumption. A smaller hysteresis is a good indicator of a more uniform material. To detect the stress more accurately, a smaller hysteresis is preferred. For this study, smart cementitious materials' average hysteresis was calculated and used as an indicator to determine the effectiveness of the different dispersion methods (Fig. 11 and Table V). The average hysteresis (H) is calculated by using the stress sensitivities differences between the loading and unloading of the materials divided by the average force sensitivity (Eq. 4):

$$H = \frac{\frac{1}{N}\sum_{i=1}^{N}(S_{L,i} - S_{U,i})}{\frac{1}{N}\sum_{i=1}^{N}\frac{(S_{L,i} + S_{U,i})}{2}} \quad (4)$$

For Groups CMC (C)-, SM-, and DM- CNTs samples, the hysteresis was 0.4%, 6.7%, and 7.3%, respectively. It can be seen that both Group SM and Group DM have relatively large hysteresis for the stress sensing. The percentage difference of hysteresis between the methods calculated using Eq. (3) found that the new CMC surface treatment method reduces hysteresis ~95% and 94% when comparing to bare CNTs (Group DM) and surfactant treated CNTs (Group SM) respectively, which is a significant improvement compared to the current practices.

C. Consistency/Repeatability

Consistency/repeatability can be defined as a measure repeatability of measurements on a single sample. For a sensing material, the uniformity of the material can be a good indicator. A high consistency of stress measurement is highly preferable for the new modified nanomaterial (CNTs) incorporated smart cementitious materials. Consistency/repeatability can be measured using the standard deviations (σ) in the stress sensitivity data (Eq. 5). A smaller standard deviation for smart cementitious materials indicates a consistent, repeatable, and predictable as well as quality of the sensing.

$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(x_i - \mu)^2} \quad (5)$$

In Eq. 5, N is the total number of testing on stress sensitivity, x_i is the individual stress sensitivity for each cycle and μ is mean of all stress sensitivity data.

72 cycles of measurements from each group (SM, CMC (C), and DM) were used to compute the standard deviations in the stress sensitivity data for loading and unloading(Table VI). For Group CN, it was calculated based on 36 cycles of measurements. The coefficient of variation (CV) is then calculated by having the standard deviation divided by mean following Eq. (6) below:

$$Coefficient\ of\ Variation = \frac{\sigma}{\mu} \times 100\% . \quad (6)$$

The coefficient of variation for the samples with bare CNTs (Group DM) were 92.7% and 94.1% for loading and unloading respectively. The results indicate a poor consistency/repeatability in stress sensing. Samples made with surfactant-dispersed CNTs (Group SM), the coefficient of variation were 75.3% for loading and 83.3% for unloading, which is slightly better than Group DM but still very high. For the samples with CMC coated CNTs (Group CMC (C)), a smaller coefficient of variation of 15.1% for loading and 14.9% for unloading was observed.

The average coefficient of variation between loading and unloading stress sensing data were also computed (Fig. 12 and Table VI). The percentage difference of average coefficient of variation was calculated using Eq. (3). When comparing with samples made from bare CNTs (Group DM), CMC coated CNT impregnated samples (Group CMC (C)) showed higher repeatability/consistency of the average force sensing with an increase of 84%. The new method (Group CMC (C)) have increased repeatability/consistency of the average force sensing by 81% when comparing with surfactant dispersed CNTs (Group SM).

VI. Conclusion and Future Work

In this paper, a new CNTs dispersion method for smart cementitious material was developed using the CMC surface modification of CNTs. Based on a comparison with the other two commonly used CNTs dispersion methods, viz, direct mixing (bare CNTs) and the surfactant method (surfactant dispersed CNTs), we can conclude the following:

1) the new CMC surface modification method developed within this research significantly improves the dispersion effectiveness and increases the stress sensitivity of smart cementitious material by up to 10 times;

2) The optimal CMC concentration for modifying CNTs in smart cementitious materials for stress sensing was found to be 0.5%;

3) the CMC surface modification method reduces the hysteresis approximately 95% compared to the direct mixing method and surfactant method and 94% compared to the surfactant method;

4) the CMC surface modification method significantly increased the consistency in force sensitivity by 84% compared to the direct mixing method and 81% compared to the surfactant method.

This study validated that the developed CMC surface treatment method can enhance stress sensing with higher stress sensitivity, smaller hysteresis, and better consistency. More studies will be needed to further investigate the optimal mixing progress, the dispersion effectiveness, chemical reactions, bonding mechanisms, and the mechanical property of the smart cementitious materials made with the CMC coated CNTs.

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FIGURES

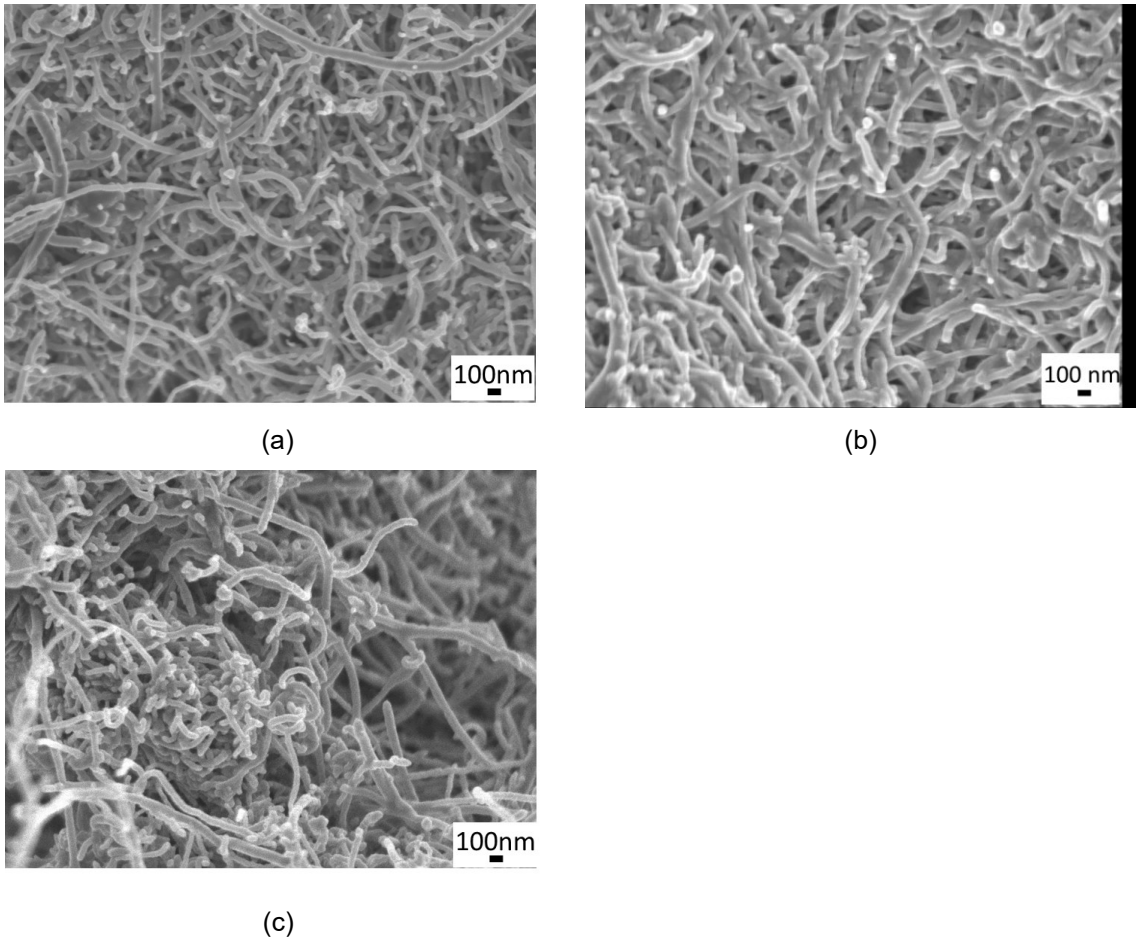


Fig. 1. SEM micrographs of (a) bare/uncoated CNTs and (b) CMC surface coated CNTS (c) Surfactant surface treated CNTs under x30,000 magnification. The uncoated CNTs are agglomerated. The coated CNTs are uniformly distributed compared to uncoated CNTs.

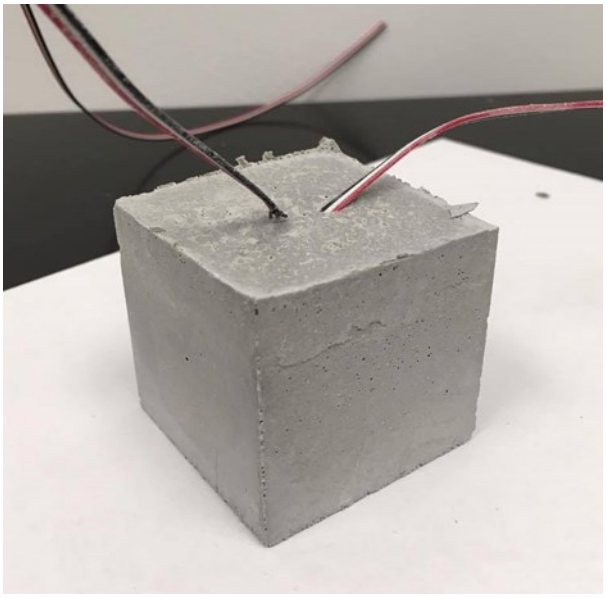


Fig. 2. 50mm x 50mm x 50mm test mortar blocks prepared with CMC-, SM-, bare CNT-, and CN-cement mixtures. Two electrical wires with their naked ends are placed and embedded 2.5cm apart in each block for piezo-response measurement.

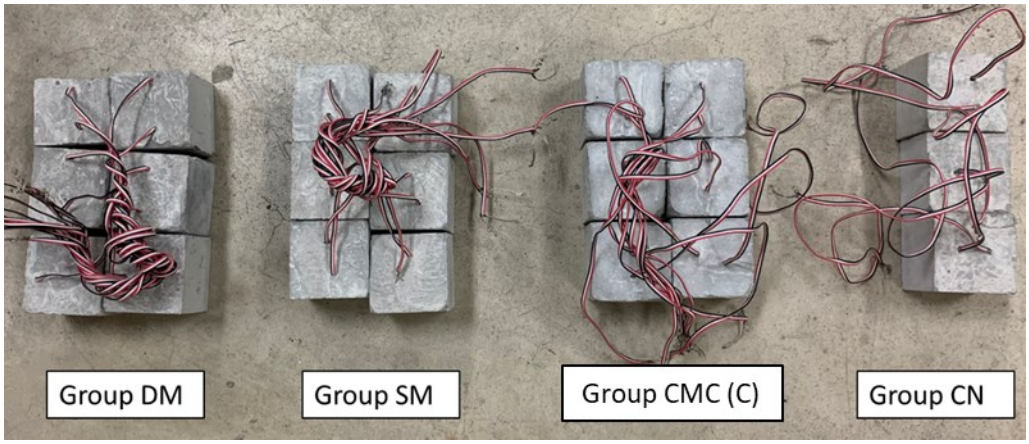
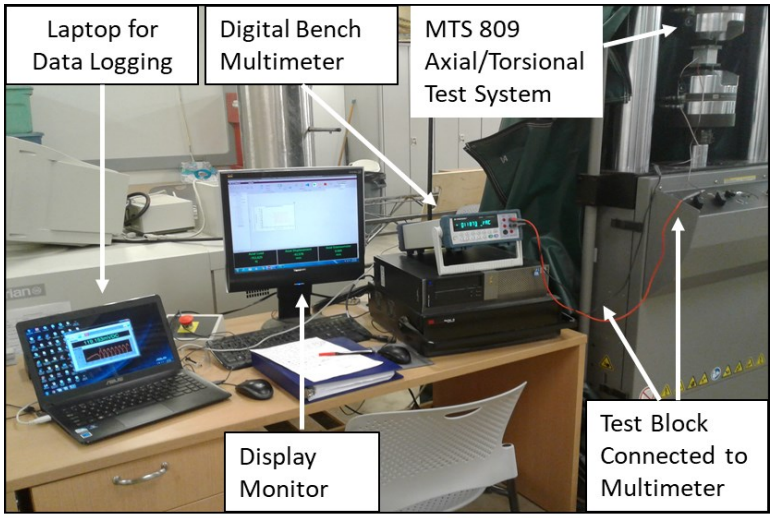
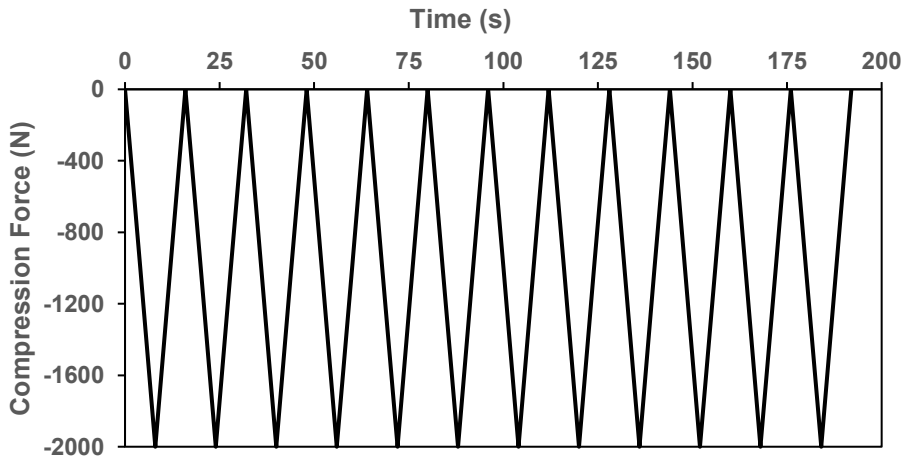


Fig. 3. The sample groups for various dispersion methods. Group DM with bare CNTs using direct mixing method, Group SM was made by CNTs treated with the NaDDBS surfactant method, Group CMC (C) was made by CNTs surface modified using CMC, and Group CN includes control samples without CNTs.



(a)



(b)

Fig. 4. Measurement of piezo-responses in the samples: (a) Sample testing laboratory set-up and (b) test samples dynamic loading scheme for 12 cycles.

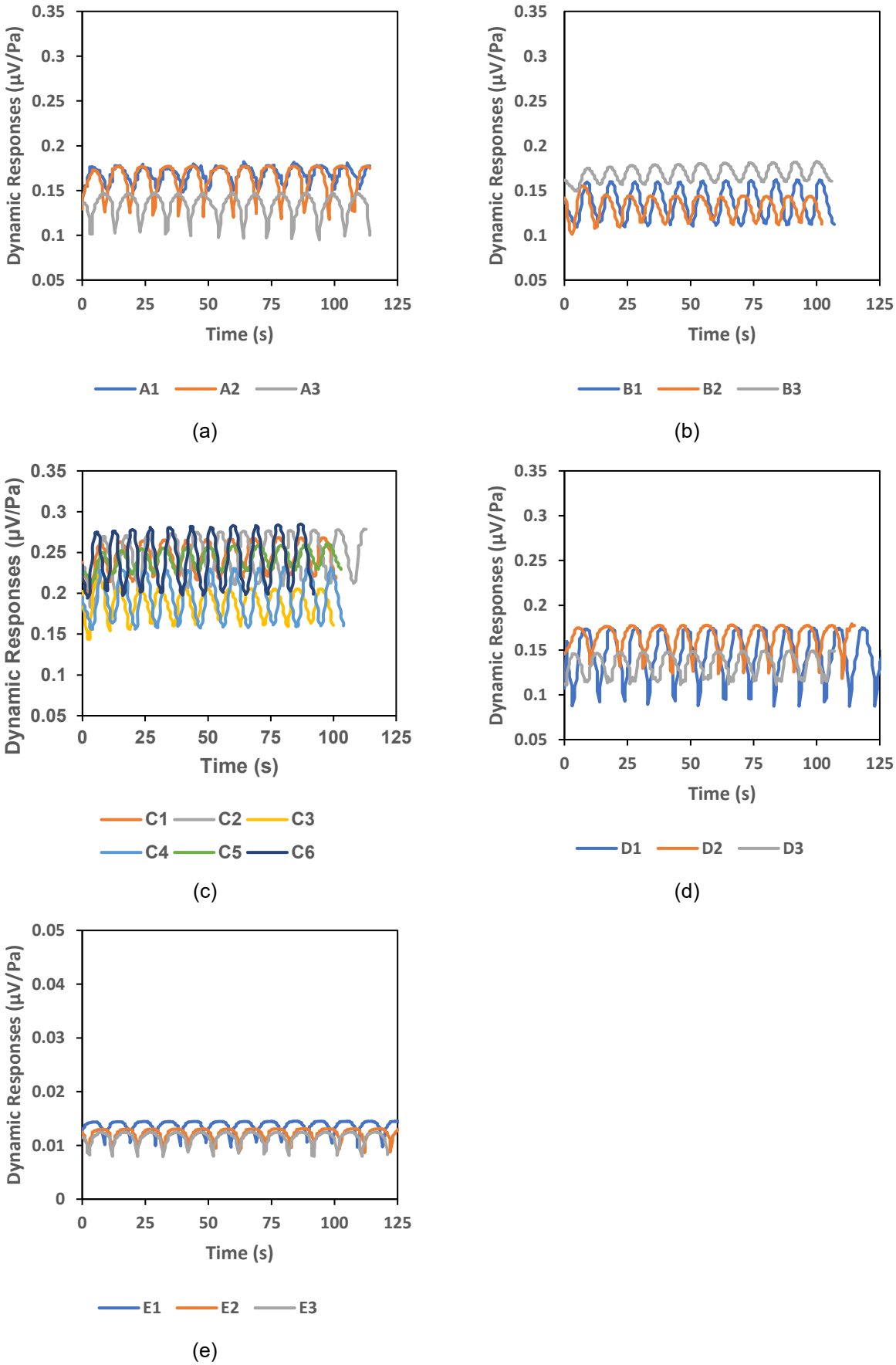


Fig. 5. (a) Dynamic responses in Group CMC block samples. Each sample's responses from Group CMC is represented by the multiple plots represent responses (a) with 0.1% CMC; (b) with 0.3% CMC; (c) with 0.5% CMC; (d) with 1% CMC; (e) No CNT with 1% CMC

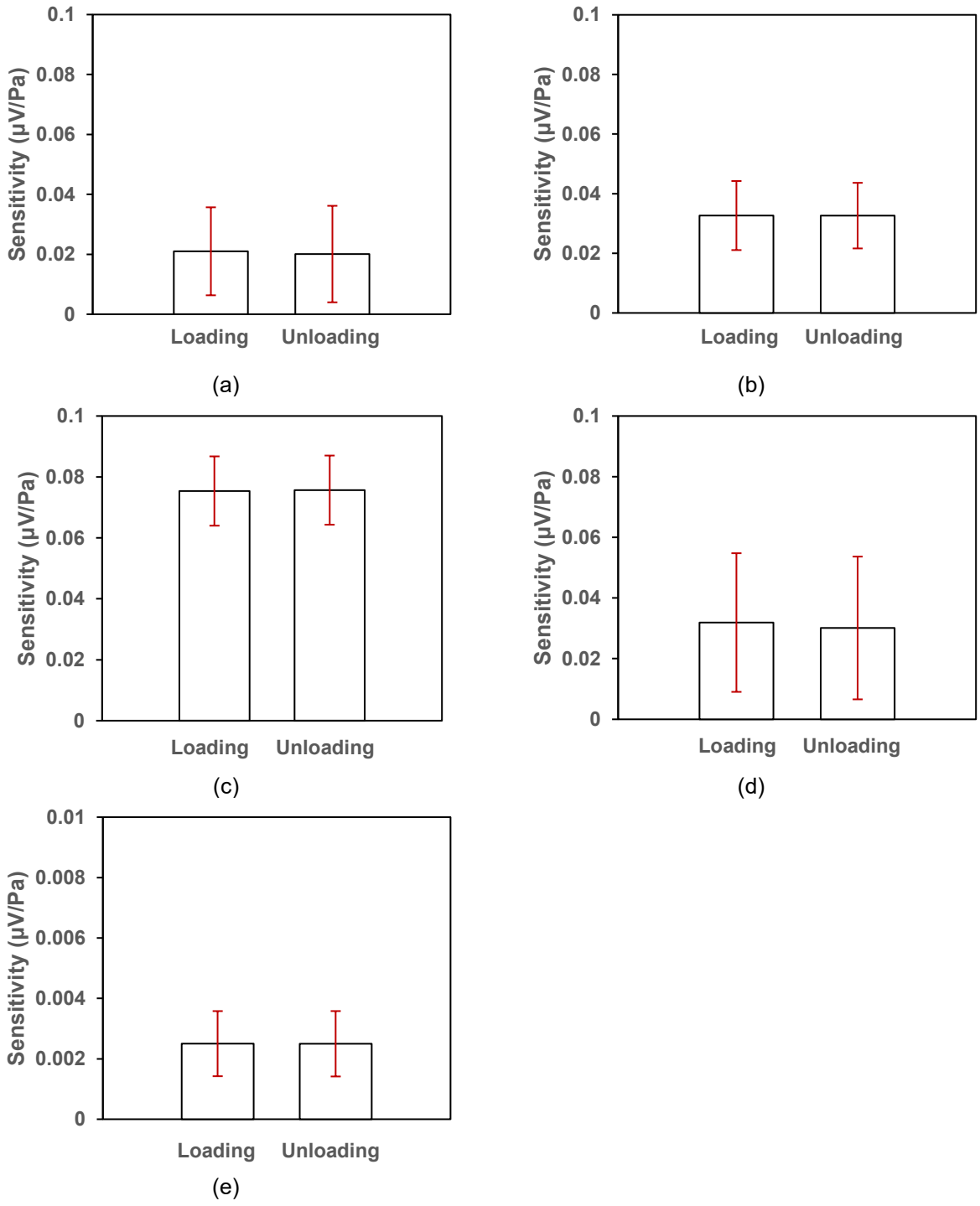
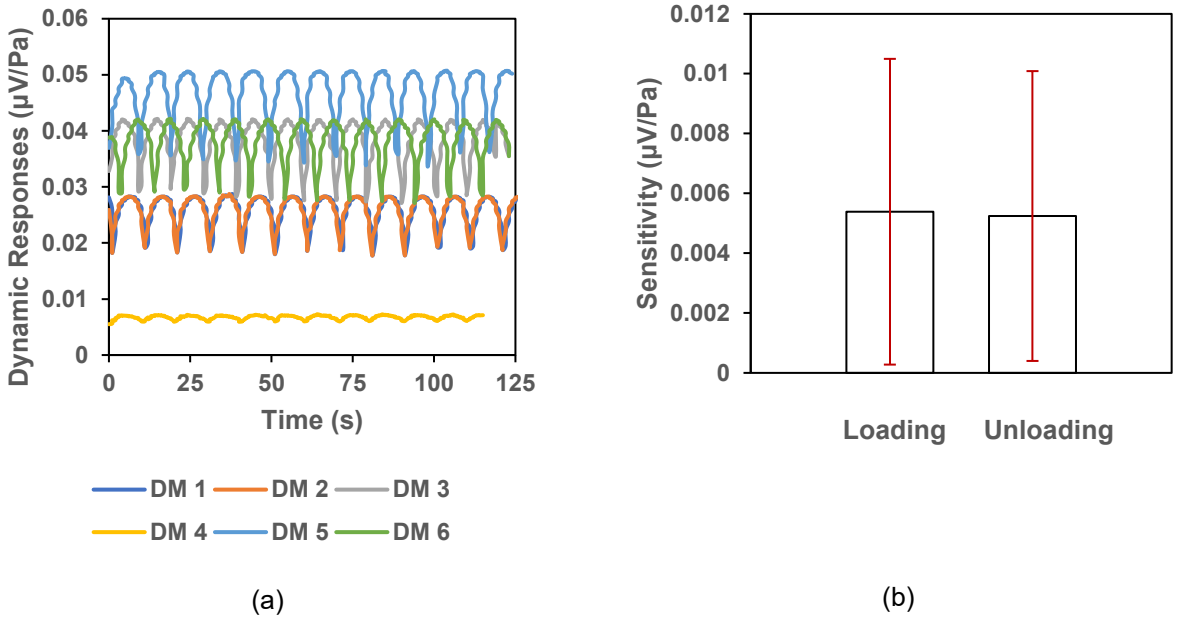


Fig. 6. Group CMC block samples average loading and unloading sensitivities. Standard deviation is represented by the vertical error bars (a) with 0.1% CMC; (b) with 0.3% CMC; (c) with 0.5% CMC; (d) with 1% CMC; (e) No CNT with 1% CMC



(a) (b)

Fig. 7. (a) Dynamic responses in Group DM block samples. Each sample's responses from Group DM is represented by the multiple plots represent responses and (b) Group DM block samples average loading and unloading sensitivities. Standard deviation is represented by the vertical error bars.

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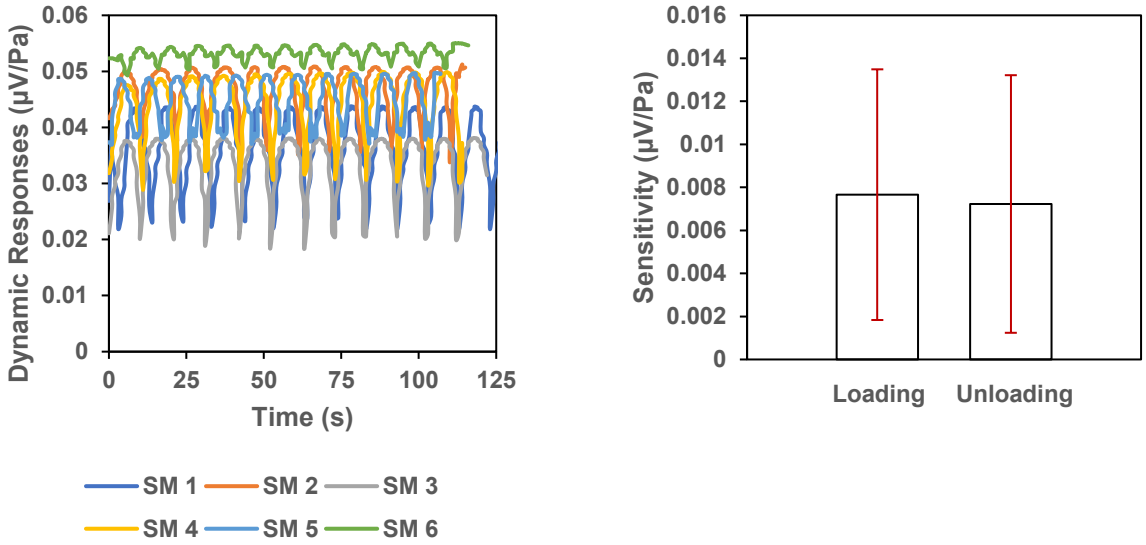

(a) (b)

Fig. 8. (a) Dynamic responses in Group SM block samples. Each sample's responses from Group SM is represented by the multiple plots represent responses and (b) Group SM block samples average loading and unloading sensitivities. Standard deviation is represented by the vertical error bars.

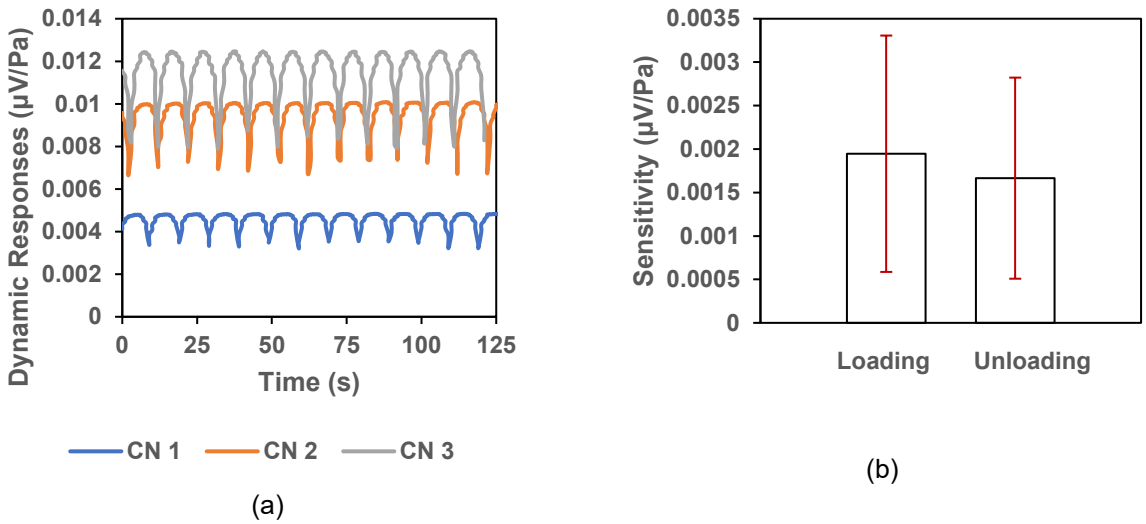

Fig. 9. (a) Dynamic responses in control sample block Group CN (no CNTs). Each sample's responses from Group CN is represented by the multiple plots represent responses and (b) Group CN block samples average loading and unloading sensitivities. Standard deviation is represented by the vertical error bars.

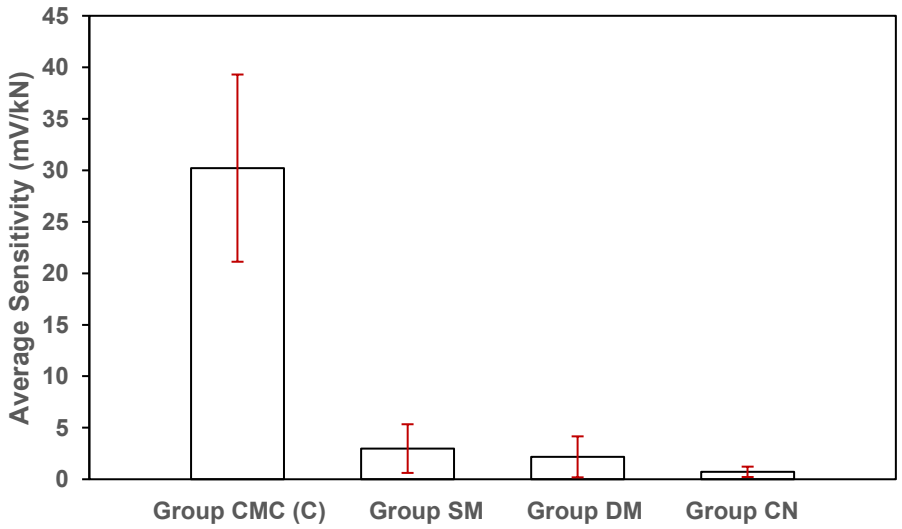

Fig. 10. Loading and unloading average stress sensitivity in the sample blocks made with CMC coated CNTs (Group CMC (C)), surfactant modified CNTs (surfactant method, Group SM), bare CNTs (direct mixing method, Group DM) and no CNTs (Group CN). Standard deviation is represented by the vertical error bars.

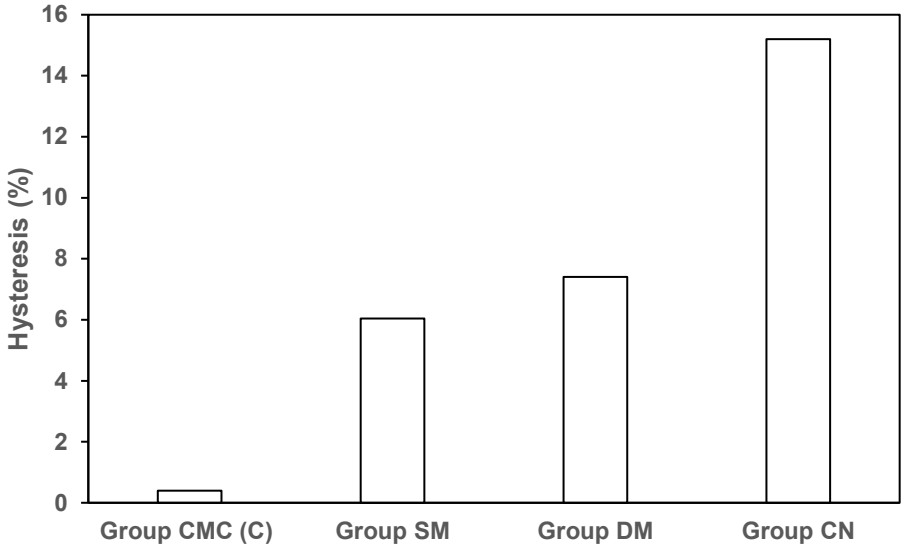

Fig. 11. Average percentage hysteresis from different dispersion methods. The new CMC surface treatment method reduces hysteresis approximately 95% compared to the direct mixing method (Group DM) and 89% compared to the surfactant method (Group SM).

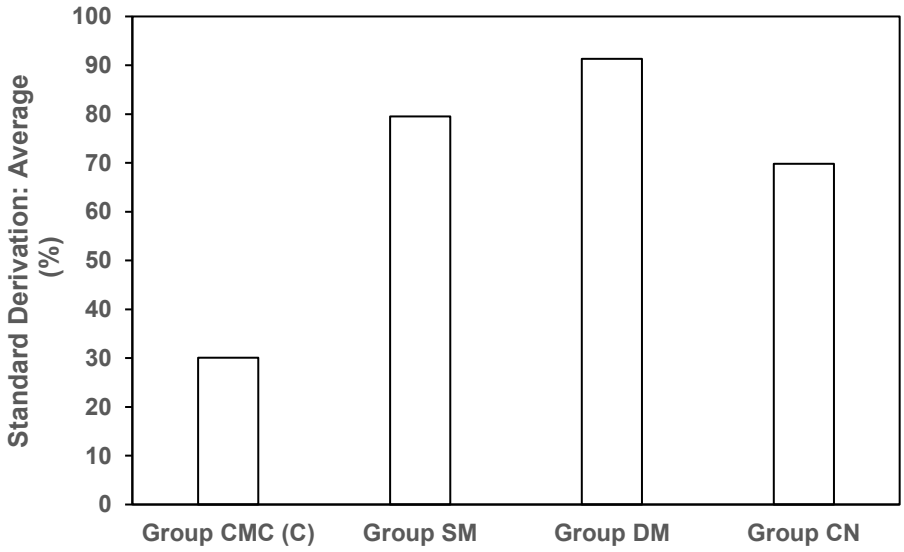

Fig. 12. The average Coefficient of Variation between loading and unloading stress sensitivity. Group CMC (C) showed increased repeatability/consistency of the average stress sensing for loading and unloading by 84% compared to Group DM and 81% compared to group SM.

TABLES

TABLE I
Properties of Polymers Used in Cementitious Materials

	Setting Time	Reaction with Metal	Water Absorbance	Bio-polymer	Solution Preparation	Cost
Polyacrylamide (PAA)	Long	Yes	Very High	No	Complex	$8,150/1 kg
Sodium Polyacrylate (SP)	N/A	No	Extremely High	No	Complex	$91.50/1 kg
Polyethylene glycol (PEG)	N/A	No	High	Yes	Easy	$32.50/1 kg
Carboxymethyl Cellulose (CMC)	Medium	No	Medium	Yes	Easy	$106/1 kg

TABLE II
Holcim Cement Physical Properties Per ASTM C150 Requirements

Property	
Fineness, m/g	
Turbidimeter (min)	160
Air permeability (min)	280
Time of set	
Vicat (minutes)	
Initial (min)	45
Final (max)	375
Gilmore (minutes)	
Initial (min)	60
Final (max)	600
Air content (max)	12%
Autoclave expansion (max)	0.80%
Compressive strength (min)	
1 day, MPa (psi)	-
3 days, MPa (psi)	12 (1740)
7 days, MPa (psi)	19 (2760)
28 days, MPa (psi)	-

TABLE III
Samples Made and Used in This Study.

Dispersion Method	Group	Number of Samples	Description
CMC Surface Treatment	CMC (A)	3	0.1% CNTs / 0.1% CMC
	CMC (B)	3	0.1% CNTs / 0.3% CMC
	CMC (C)	6	0.1% CNTs / 0.5% CMC*
	CMC (D)	3	0.1% CNTs / 1% CMC
	CMC (E)	3	Only Cement / 1% CMC**
Surfactant Method	SM	6	0.1% CNTs
Direct Mixing Method	DM	6	0.1% CNTs
Direct Mixing Method*	CN	3	Only Cement**

*CMC (C) uses the same samples for method comparison and CMC concentration study
**No CNTs

TABLE IV
Comparisons of stress sensitivity and hysteresis

Group Name	Stress Sensitivity: Loading (μV/Pa)	Stress Sensitivity: Unloading (μV/Pa)	Average Stress Sensitivity (μV/Pa)	Percentage of Changes Compared with DM method
CMC (C)	0.0754	0.0757	0.0756	1,326%
SM	0.0077	0.0072	0.0075	41.5%
DM	0.0055	0.0051	0.0053	0%
CN	0.0019	0.0017	0.0018	-66%

TABLE V
Comparison of hysteresis

Group Name	Sensitivity Difference (μV/Pa)	Average Stress Sensitivity (μV/Pa)	Hysteresis
CMC (C)	0.0003	0.0756	0.4%
SM	0.0005	0.0075	6.7%
DM	0.0004	0.0055	7.3%
CN	0.0002	0.0018	11.1%

TABLE VI
Comparison of consistency/repeatability

Group Name	Standard Deviation: Loading (±μV/Pa)	Coefficient of Variation: Loading (%)	Standard Deviation: Unloading (±μV/Pa)	Coefficient of Variation: Unloading (%)	Standard Deviation: Average (±μV/Pa)	Coefficient of Variation: Average (%)
CMC (C)	0.0114	15.1%	0.0113	14.9%	0.0113	14.9%
DM	0.0051	92.7%	0.0048	94.1%	0.0050	90.9%
SM	0.0058	75.3%	0.0060	83.3%	0.0059	78.7%
CN	0.0014	73.7%	0.0012	70.6%	0.0013	72.2%