

## Research Article

# The Characteristics of CNT/Cement Composites with Acid-Treated MWCNTs

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Received 24 April 2015; Revised 1 July 2015; Accepted 2 July 2015

Academic Editor: Luigi Nicolais

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This study investigated the effects of acid-treated MWCNTs on the workability, compressive and tensile strength, porosity, and microstructure of CNT/cement composites. While workability decreased with acid treatment of CNTs, compressive and tensile strength improved significantly. Strength was also enhanced by using acid-treated CNTs alone, compared to using a surfactant with acid-treated CNTs. MIP analysis revealed that the porosity decreases from using acid-treated CNTs and that using acid-treated CNTs without a surfactant was more effective in reducing the size of micropores. A SEM analysis revealed improved CNT dispersion and dense hydration products in cement composites containing acid-treated CNTs. Ultimately, the use of acid-treated CNTs improved the CNT dispersion within CNT/cement composites, enhanced the pore structure, and formed denser hydration products around CNTs.

## 1. Introduction

Carbon nanotubes (CNTs) are cylindrical nanostructures formed from rolling graphene hexagonal nets into nanometer diameter tubes. Graphene can be rolled as a single layer or multiple layers. The former is known as single-walled CNTs (SWCNTs), and the latter as multiwalled CNTs (MWCNTs), as shown in Figure 1. The elasticity coefficient of carbon nanotubes is approximately 1TPa and more than five times greater than that of steel. The tensile strength is about 60 GPa, which is more than 100 times larger than that of steel. On the other hand, the specific weight is only one-sixth that of steel [1–3]. Following the 1991 discovery of carbon nanotubes by Iijima during his analysis of a carbon mass formed on the cathode of graphite using the electric discharge method, CNTs have been widely used in electronic materials, medicine, energy, chemistry, and high-functional composites on the basis of advantages such as outstanding mechanical properties, thermal conductivity, electrical conductivity, low specific weight, and high resistance to corrosion [4–8]. The use of carbon nanotubes in polymer-based composites is known to significantly improve mechanical properties [9, 10].

Meanwhile, mortar, concrete, and other cement-based materials are commonly used in construction because of their low costs and high compressive strength. However, cementitious materials exhibit brittle behavior and have very low tensile strength compared to compressive strength. The most general method of overcoming these weaknesses is to introduce fiber. Steel fiber, carbon fiber, glass fiber, and synthetic fiber are some materials that have been used to enhance the mechanical properties of cementitious materials.

Common characteristics of fiber introduced to enhance mechanical properties include high tensile strength, large aspect ratio, and adhesion. Considering these points, carbon nanotubes are outstanding candidates for fiber and are expected to greatly enhance mechanical properties. Many researchers have conducted experimental studies on the compressive strength or tensile strength of cement composites mixed with carbon nanotubes. In general, MWCNTs are more widely used than SWCNTs because they require lower manufacturing costs and offer better reinforcement in cement composites. Brenner et al. [11] used 0.2% MWCNTs and improved flexural strength by 7% with cement paste as a reinforcement. Chan and Andrawes [12] improved flexural strength and tensile strength by 25% using 0.25% MWCNTs.

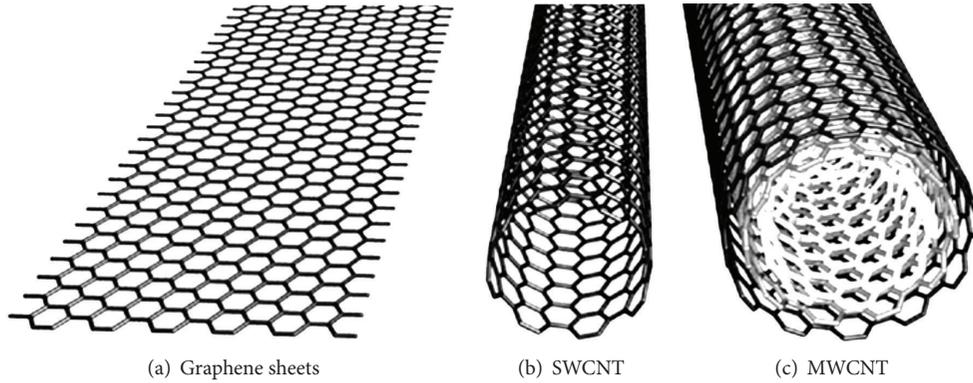


FIGURE 1: Graphene versus carbon nanotubes [31].

Hunashyal et al. [13] performed a direct tensile test and used 0.5% MWCNTs, achieving a 19% improvement in tensile strength. Using only small amounts of MWCNT at 0.08%, Konsta-Gdoutos et al. [14] enhanced flexural strength by 25%.

Past research shows that the use of MWCNTs as reinforcement improves both compressive and tensile strengths by 10 to 25%. However, the reinforcement effect is not as prominent when we consider the outstanding mechanical properties and geometrical shape of CNTs. The disadvantages of using CNTs as reinforcement for cementitious materials have been widely reported. First, the strong van der Waals force between CNT particles makes it difficult to ensure uniform dispersion. Second, the hydrophobicity of CNTs leads to weak bonds between the cement matrix and CNTs. Three methods are available to improve dispersion. First, ultrasonic waves can be applied to a solvent and CNT-containing solution to facilitate dispersion. Second, a surfactant is introduced between carbon nanotubes and matrix to improve affinity. These two methods have been employed in past research on cement composites reinforced with CNTs. The third method, which involves chemical modification of carbon nanotubes, places functional groups on the surfaces of carbon nanotubes, thereby facilitating dispersion as well as improving the bonding between carbon nanotubes and the matrix. Other methods include surface modification with exposure to ozone gas at high temperatures [15] and the formation of carboxyl groups through acid treatment [16–18]. The formation of carboxyl groups on the surface improves the bonding by inducing chemical reactions with hydraulic cementitious materials.

This study applied acid treatment to maximize the strength enhancement of MWCNT-reinforced cement composites by improving MWCNT dispersion and adhesion onto cement. Comparisons were made with consideration of acid treatment and use of surfactants.

## 2. Experiment

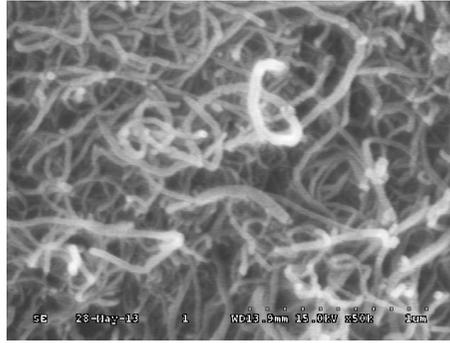
**2.1. Materials and Mix Design.** This study mixed CNTs into cement paste and used Type I Portland cement. To obtain a more uniform CNT dispersion, a certain proportion of the cement was substituted with silica fume, which has relatively

TABLE 1: Properties of cement and silica fume.

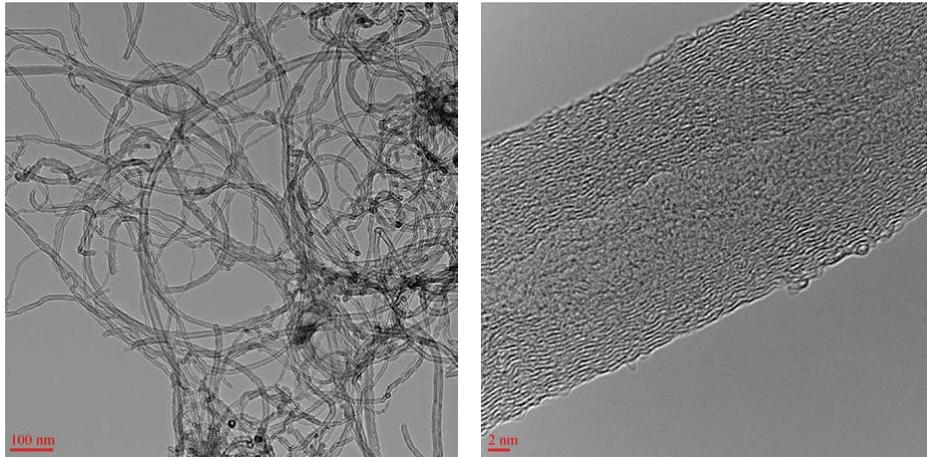
	Chemical composition (%)					Specific gravity (g/cm <sup>3</sup> )
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	
Cement	21.91	5.25	3.51	63.38	2.10	3.15
Silica fume	99.47	0.40	0.05	0.01	0.01	2.65

smaller particles. Sanchez and Ince [19] reported that silica fume contributes to improving CNT dispersion. By virtue of the very small particle size of silica fume, the particles can be located between individual CNTs during the mixing process, separating CNTs from one another enough to weaken the van der Waals forces between CNTs that agglomerate them as clumps. Silica fume neighboring CNTs also generate Ca-Si rich phase and calcium hydroxide plates, which improve the bonding between CNTs and hydration products [19, 20]. The physical and chemical properties of cement and silica fume are presented in Table 1. To improve the CNT dispersion and acquire workability, high-performance polycarboxylic superplasticizer was used as a surfactant, at 0.4% of the weight of the binding material. Surfactant is widely used to facilitate deagglomeration and stabilization of nanomaterials in aqueous solution, often with sonication process. The CNTs used in this study were MWCNTs, and their characteristics are given in Table 2. SEM and TEM images of a MWCNT are shown in Figure 2.

Specimens were prepared, with acid treatment and use of a surfactant as experimental variables. The water to binder ratio was 0.4 for all mixtures, and the ratio of cement to silica fume to mixing water was 9 : 1 : 4. The amount of CNTs was 0.1% of binder in weight. A small amount of high-performance superplasticizer was added at 1% to the amount of the mixing water. The same amount of superplasticizer was added to the plain specimen, prepared without mixing CNTs, for accurate comparison of strengths with other CNT-containing specimens. NAT-S is the specimen prepared by mixing CNTs without acid treatment. AT-S represents the specimen with acid-treated CNTs and containing the high-performance superplasticizer and AT-NS without the superplasticizer. Sonication was not performed except for the sonication process included in acid treatment.



(a) SEM image



(b) TEM images

FIGURE 2: (a) SEM and (b) TEM images of pristine MWCNTs.

TABLE 2: Properties of MWCNT.

Purity (wt.%)	Avg. diameter (nm)	Length ( $\mu\text{m}$ )	Metal oxide (wt.%)	Bulk density ( $\text{g}/\text{cm}^3$ )	Specific surface area ( $\text{m}^2/\text{g}$ )
>95	20	1–25	<5	0.03–0.05	150–250

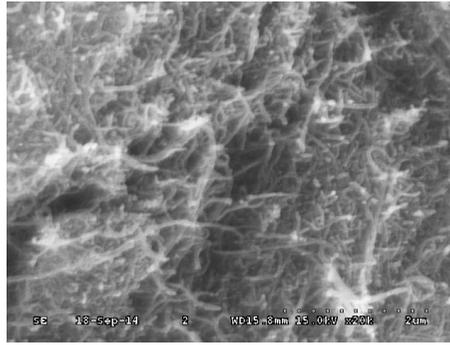
After dry mixing of silica fume and cement, mixing was carried out again by adding a mixture of CNTs, high-performance superplasticizer, and mixing water. Following demolding at one day after placement, wet curing was conducted at room temperature.

**2.2. Acid Treatment.** Acid treatment was taken for mixing 5 g of CNTs in a solution of 150 mL of sulfuric acid and 50 mL of nitric acid. One cycle of the CNT acid treatment process was defined as 10-second sonication and a 10-second rest. Acid treatment was performed for two hours. After sonication, the mixture is diluted for 24 hours in 1000 mL of water, which is 5 times the amount of the acidic solution. Residual acidic materials on the surface of CNTs are first removed through filtration and completely removed from washing CNTs with water following storage in a drying oven for 24 hours.

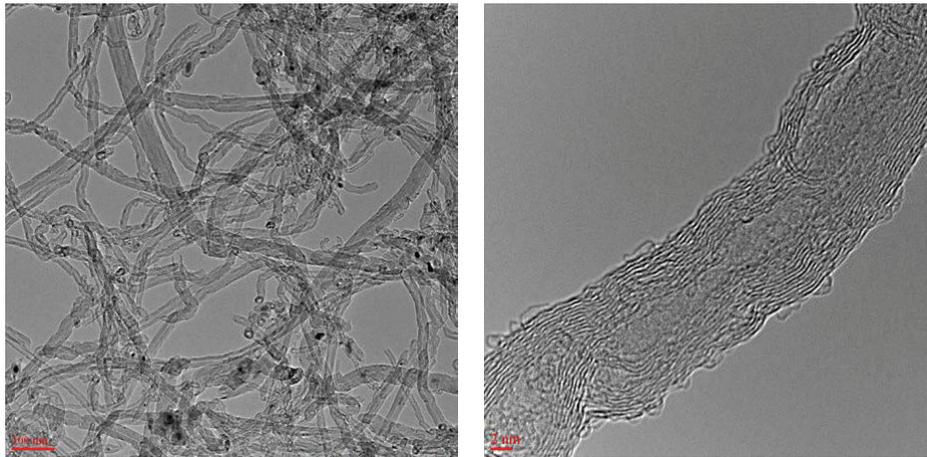
Figure 3 shows SEM and TEM images of MWCNTs after acid treatment. Compared to the MWCNTs before acid treatment (Figure 2), it can be found that the CNTs have a shorter length, a smaller diameter, and a roughen surface after

acid treatment. According to past research on acid treatment of CNTs, the length of CNTs shortens during sonication, and defects on the CNT surface induced by acid treatment tend to break carbon-carbon bonds while promoting bonding with carboxyl groups. This leads to a decrease in thickness and weight of CNTs [21, 22].

To determine whether carboxyl groups have been successfully formed on the surface of acid-treated CNTs, a FT-IR analysis was performed using a Nicolet 380 spectrometer. The infrared absorption spectrum is shown in Figure 4. CNTs without acid treatment hardly show any absorption peaks, but the CNTs with acid treatment show many peaks across regions. From the figure, we can see absorption peaks at 580, 1180, 1280, 1640, and 3400  $\text{cm}^{-1}$ . Absorption peaks are found at 1180  $\text{cm}^{-1}$  and 1280  $\text{cm}^{-1}$  for C-O bonds of the carboxyl group, at 1640  $\text{cm}^{-1}$  for C=O bonds, and at 3400  $\text{cm}^{-1}$  for O-H bonds [16, 21, 23–25]. The absorption peaks corresponding to the three types of bond indicate that acid treatment has successfully combined the carboxyl group onto the surface of the CNTs.



(a) SEM image



(b) TEM images

FIGURE 3: (a) SEM and (b) TEM images of acid-treated MWCNTs.

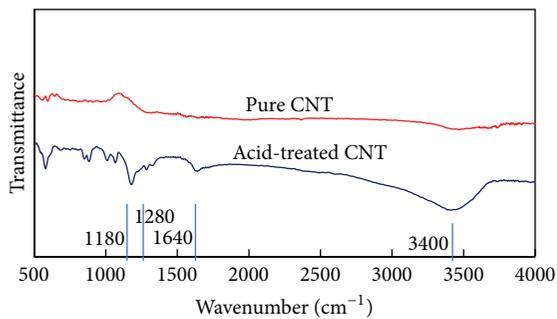


FIGURE 4: Results of FT-IR analysis for CNT.

**2.3. Test Method.** Using acid-treated MWCNTs, strength characteristics of a CNT-reinforced cement paste in the fresh and hardened states were estimated. Workability was measured with a flow table of ASTM C 1437, and strength at 14 days was evaluated through compressive and tensile tests. According to ASTM C 109 specifications, the mortar specimen for compressive strength was prepared in the form of a 50 mm × 50 mm × 50 mm cube, and the tensile strength specimen used in the splitting tensile test was a cylinder with the dimension of  $\Phi 25$  mm × 50 mm. The test was performed using a universal testing machine under the displacement

control condition. Figure 5 shows the experimental setup for the compressive test and tensile splitting test.

### 3. Results and Analysis

**3.1. Workability.** Figure 6 shows the flow value for each specimen measured through the flow table test. As shown in the results, flow values decrease for CNT-containing specimens, but the difference is not significant enough to affect workability and strength. The decrease in flow for CNT-containing specimens compared to the plain specimen can be explained by the fact that incorporating fibers into concrete generally reduces workability regardless of the type of fiber [26].

Comparisons of workability with and without acid treatment show that flow values decrease in the former case. It is thought that acid treatment leads to the formation of hydrophilic carboxyl groups and hydroxyl groups on the surface of CNTs, and their adsorption to water reduces the workability of cement composites.

**3.2. Strength.** Figure 7 shows the results of the compressive test and tensile splitting test. From the results of the compressive strength in Figure 7(a), it can be seen that the plain

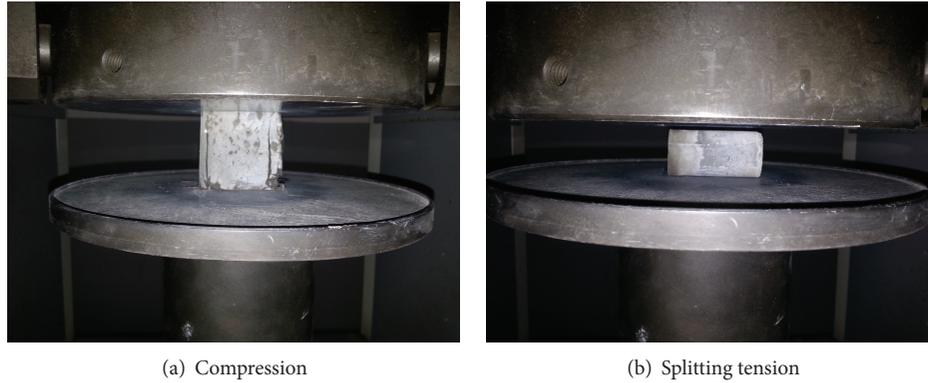


FIGURE 5: Test for compressive and splitting tensile strength.

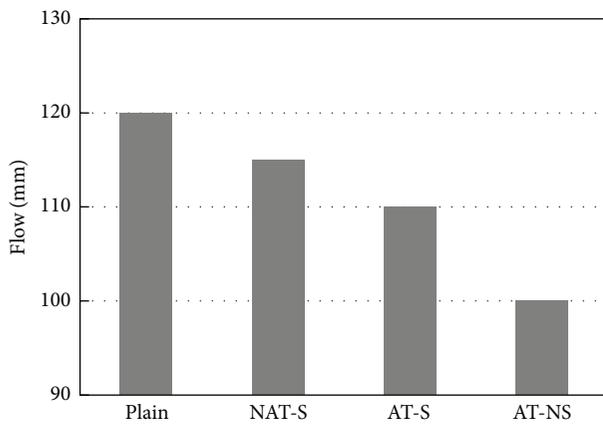


FIGURE 6: Flow test results.

specimen has the lowest strength among them, and that the CNT-reinforced specimens have greater strength.

When the compressive strength of NAT-S and AT-S was compared under the same mixing conditions, the acid-treated AT-S had a compressive strength higher than that of NAT-S by 30%. Strength improvement in relation to acid treatment of CNT can be explained by the schematic structure formation illustrated in Figure 8 [16, 18, 27–29]. Acid treatment forms hydrophilic carboxyl groups on the surface of CNTs, which are originally of a hydrophobic nature. This allows water to be effectively adsorbed onto the CNT surface, generating hydration products such as a C-S-H gel or  $\text{Ca}(\text{OH})_2$ . The chemical reaction between hydrophilic carboxyl groups bonded on CNTs and the hydration products densifies and strengthens the interfacial area around CNTs [30]. The improved adhesion between CNTs and cement hydration products enhances the overall strength.

Higher aspect ratio of fiber, in general, provides more beneficial performance in mechanical properties of the composites but causes more difficulty in dispersing them uniformly. Clumped fibers without effective dispersion detract the strengthening effect of fiber reinforcement. Even though the aspect ratio of CNT became lower after acid treatment, better dispersion of the CNTs and the enhanced bonding

between CNTs and hydration products enabled obtaining the improved mechanical performance. In addition, the fracture and strength of CNT/cement composites or most fiber-reinforced composites are commonly not dominated by the rupture of CNT but strongly dominated by the pullout of CNT from the matrix. Therefore, even though the generation of defects in CNT after acid treatment reduced its strength, the defects acted as nucleation sites for production of carboxyl groups which could induce a strong bond between the CNTs and the matrix, and consequently the improved bond could lead to the increase in strength.

AT-NS, prepared by using acid-treated CNTs and without the high-performance superplasticizer, had greater strength compared to not only the NAT-S but also the surfactant-containing AT-S. Even without the surfactant, acid treatment generates electrical repulsion between CNTs, thus facilitating uniform dispersion. The introduction of the surfactant was found to impede rather than improve strength.

As shown in Figure 7(b), the change in strength by specimen in relation to acid treatment and use of surfactant is similar to that of tensile test results.

**3.3. Porosity and Microstructure.** Porosity and microstructure analyses were performed to determine changes in the microstructure of CNT/cement composites with acid treatment. The results of the MIP analysis are shown in Figure 9. All CNT/cement composites had smaller porosity compared to the plain specimen. Specimens prepared by acid-treated CNTs presented lower porosity than those without acid treatment. A slight increase in porosity was observed when a surfactant was introduced for acid-treated CNT/cement composites. While the improvement in strength is closely related to decreased porosity, the porosity of AT-NS compared to AT-S did not decrease despite having greater strength than other specimens. Unlike the three types of specimens, AT-NS exhibited a significant decrease in average pore size. The enhanced strength of AT-NS was thought to have been achieved by the improved pore distribution, and not the overall reduction in porosity.

Figure 10 shows the analysis results of the pore distribution by specimen. The size of the pore distribution by specimen is around  $100 \mu\text{m}$ . Pores of this size correspond to

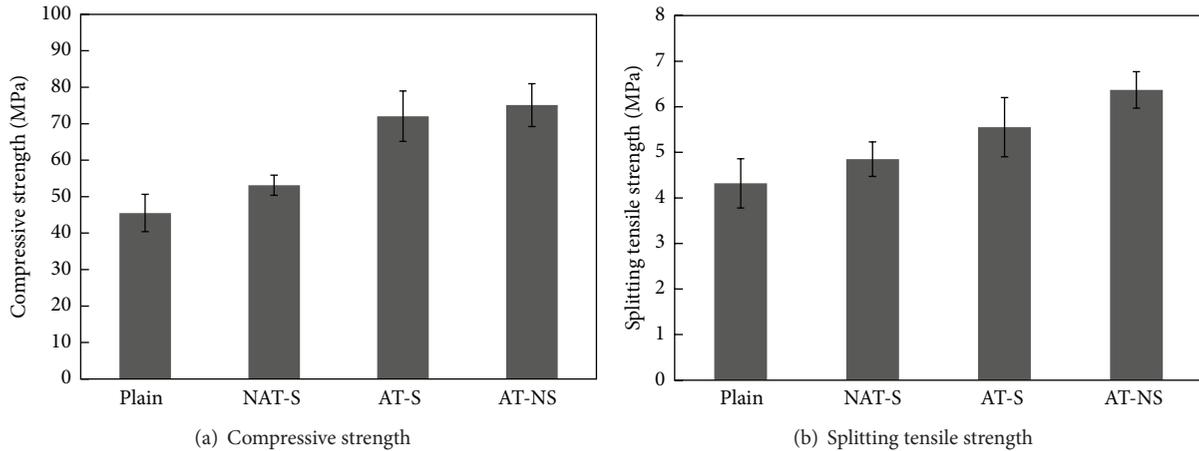


FIGURE 7: Comparison of strengths with different mixes.

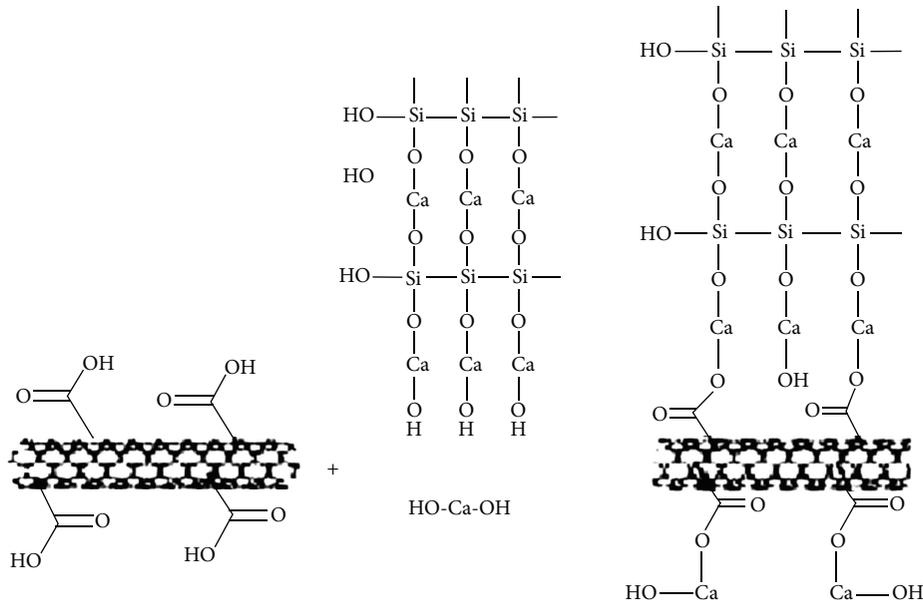


FIGURE 8: Reaction scheme between carboxylated nanotube and hydrated production ( $\text{Ca}(\text{OH})_2$  and C-S-H) of cement [16].

entrained air. The use of high-performance superplasticizer in acid-treated CNT increases the amount of entrained air. As such, specimens lacking the superplasticizer exhibited the smallest entrained air volume. Differences in the pore distribution were observed in a range of 10 to 100 nm which corresponds to capillary pores. Similar pore distributions were exhibited by the plain, NAT-S, and AT-S specimens, whereas the pore distribution of AT-NS shifted slightly to the left. The shift from 40~80 nm to 10~40 nm indicates a change from macrosized capillary pores to micro-sized pores. In general, capillary pores of 10~100 nm in size are related to strength. The decreased size of capillary pores is considered to have influenced the strength enhancement.

Figure 11 shows SEM images of the NAT-S and AT-NS specimens. As presented in Figures 11(a) and 11(b), hydration

products generated around CNTs are less dense for NAT-S, and some pores can be seen between hydration products and CNTs. On the other hand, Figures 11(c) and 11(d) show that the AT-NS specimen contains hydration products that are very densely packed. This is directly related to the improved adhesion between CNTs and hydration products. In Figure 11(a), CNTs are not completely dispersed and are only partially concentrated. Even though the specimen in Figure 11(c) was prepared without a surfactant, CNTs were found to be well dispersed based on acid treatment alone. In Figure 11(d), multiple CNTs can be observed on the cracked surface, which contributes to a more efficient load distribution, thereby enhancing the compressive and tensile strength of composites. A SEM analysis confirmed that acid-treated CNTs influence the formation of hydration products and CNT dispersion.

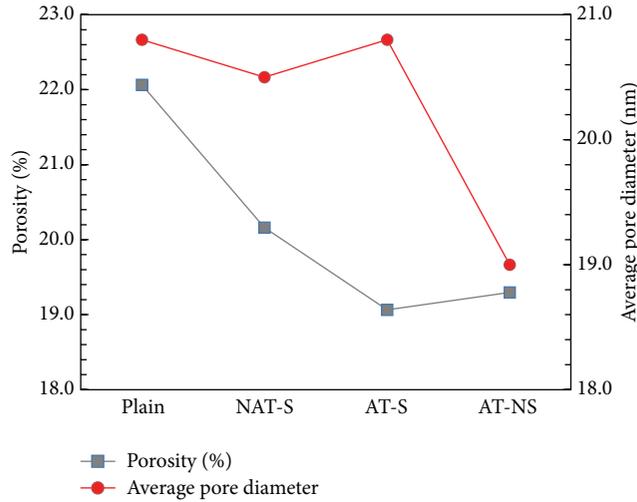


FIGURE 9: The results of MIP analysis.

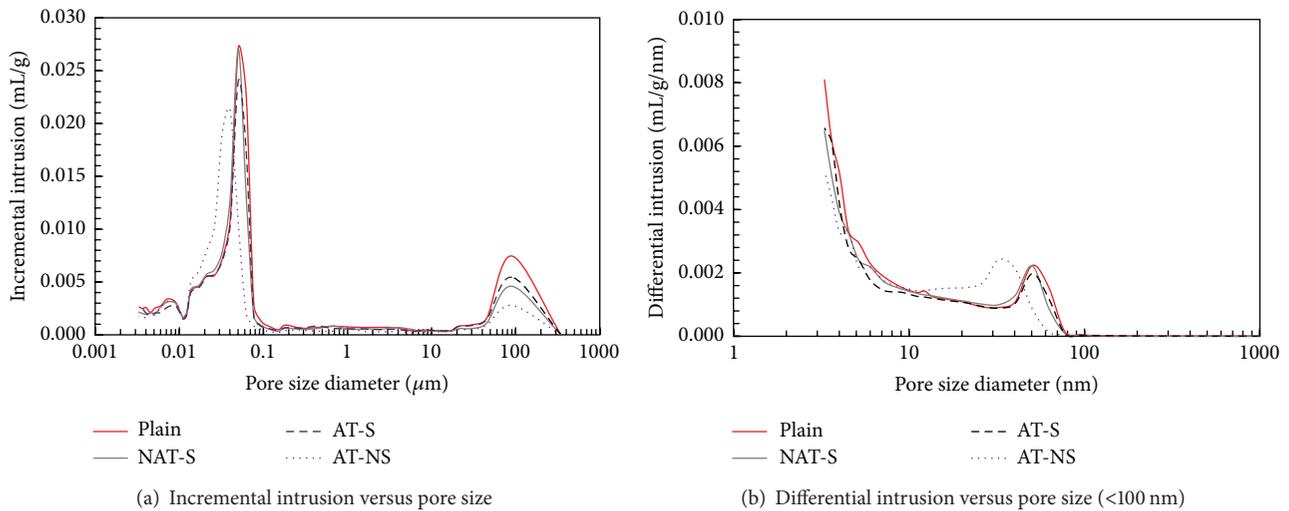


FIGURE 10: Pore size distribution with different mixes.

#### 4. Conclusions

This study applied acid treatment to maximize the strength enhancement of MWCNT-reinforced cement composites by improving the MWCNT dispersion and adhesion onto cement. The effects of using acid-treated MWCNTs were estimated by analyzing workability, strength, porosity, and microstructure. The following conclusions were derived:

- (1) Flow values in flow table decreased for specimens prepared with acid-treated CNTs. This can be explained by the reduced workability resulting from improved adsorption of hydrophilic carboxyl groups, formed on the surface of CNTs, onto water.
- (2) The use of acid-treated CNTs improved compressive and tensile strength by more than 30%. In this case, strength was negatively affected when an additional surfactant was introduced.
- (3) A pore analysis of CNT/cement composites showed that the use of acid-treated CNTs lowers porosity. Applying acid-treated CNTs only without a surfactant was more effective in reducing the size of micropores than adding a surfactant to acid-treated CNTs.
- (4) A SEM analysis revealed that cement composites prepared with acid-treated CNTs had improved CNT dispersion and denser hydration products.
- (5) Ultimately, the use of acid-treated CNTs improved the CNT dispersion in CNT/cement composites, enhanced the pore structure, and formed denser hydration products around CNTs.
- (6) In order to have a comprehensive understanding of the acid treatment effect on the CNT/cement composites, the in-depth investigation of microstructural characteristics, long-term behavior, durability, and so on might be also needed in further studies.

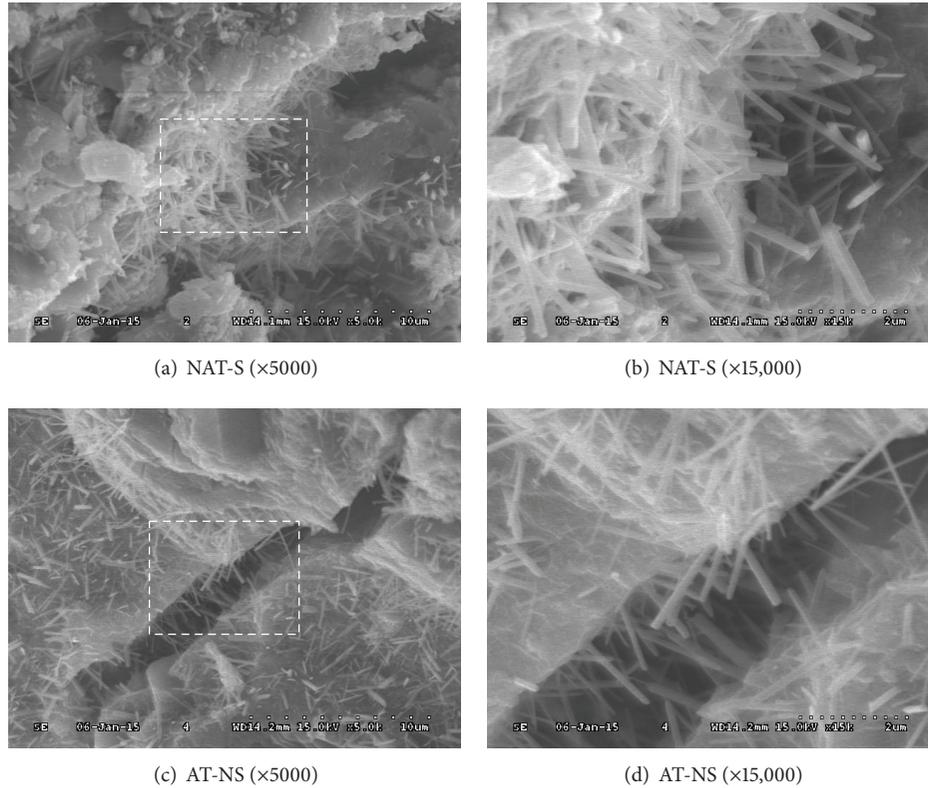


FIGURE 11: Representative SEM images taken from NAT-S and AT-NS specimens.

## Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

## Acknowledgments

This research was supported by a grant (14RDRP-B076268) from Regional Development Research Program funded by Ministry of Land, Infrastructure and Transport of Korean government as well as by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MEST) (NRF-2012R1A1A1010626).

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