Novel Dispersion Technique for Carbon Nano Tubes within Cement Matrix

Mohamed I. Serag

Mohamed I. Serag. Author is with the Department of Material Strength, Faculty of Engineering/ Cairo University, Egypt

Abstract—The major problem in utilizing Nano-particles is that they are highly agglomerated particles which cause loss in their high-surface area due to grain growth. Effective de-agglomeration and dispersion for Nano-particles is needed to overcome the bonding Van Der Waals forces after wetting which results in the formation of agglomerations in the form of entangled ropes and clumps that are very difficult to disentangle. The dispersion problem has been combated by methods like using surfactants, usually in combination with sonication. The present study focuses on the effectiveness of superplasticizers (high-range water-reducing admixtures), homogenization and ultrasonic processing (direct/indirect) for the purpose of dispersing carbon Nano tubes in water and finally pastes. A qualitative analysis using compressive strength test will be conducted in order to investigate the effect of dispersion on the mechanical properties of cement composites in corroborating CNT. In addition particle size distribution, SEM and TEM images will be carried out to observe the surface morphology and microstructure of cement composites with CNT addition.

Keywords—Carbon Nanotube; Sonication; Optimization; Novel Technique, Agglomeration.

I. INTRODUCTION

The major problem in utilizing Nano-particles is that they are highly agglomerated particles which cause loss in their high-surface area due to grain growth. Effective de-agglomeration and dispersion for Nano-particles is needed to overcome the bonding Van Der Waals forces after wetting which results in the formation of agglomerations in the form of entangled ropes and clumps that are very difficult to disentangle. The dispersion problem has been combated by methods like using surfactants, usually in combination with sonication.

The present study focuses on the effectiveness of superplasticizers (high-range water-reducing admixtures), homogenization and ultrasonic processing (direct/indirect) for the purpose of dispersing carbon Nano tubes in water and finally pastes. A qualitative analysis using compressive strength test will be conducted in order to investigate the effect of dispersion on the mechanical properties of cement composites in corroborating CNT.

In addition particle size distribution, SEM and TEM images will be carried out to observe the surface morphology and microstructure of cement composites with CNT addition.
II. EXPERIMENTAL WORK

The research discusses the dispersion of optimum mixing sequence of carbon Nanotubes within cement matrix. Eight methods are used to examine the compressive strength for cement pastes (5*5*5 cm$^3$). After 7 days; water curing. Superplasticizer is added to carbon Nanotubes during sonication as it helps carbon Nanotubes particles to disperse well. The paste contained of 523 gm., water/cement ratio is 0.4, 0.02% CNT% of cement weight and 0.4 wt. % SP. Indirect sonication method is used. The first sample is chosen to study the compressive strength of a paste contained of cement, water and SP. The second sample studies the compressive strength for different methods of carbon Nanotubes dispersion in water using indirect sonication and homogenizer. The first method is 1 hr. sonication; the second 1 hr. homogenizer, the third 40 min. sonication then 10 min. homogenizer, the forth 10 min. homogenizer then 40 min. sonication, the fifth 40 min. homogenizer then 10 min. sonication and the sixth 30 min. sonication then 30 min. homogenizer. Table 1 shows the constituents of the 6 mixtures. Figure 1 shows the mixing sequence of the samples.

Table 1: Mixtures composition (gm.)

<table>
<thead>
<tr>
<th>MIX</th>
<th>CEMENT</th>
<th>WATER</th>
<th>SP</th>
<th>CNT</th>
<th>SONICATION/HOMOGENIZER TIME</th>
</tr>
</thead>
<tbody>
<tr>
<td>C/SP</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>---</td>
</tr>
<tr>
<td>C/CNT/SP</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>---</td>
</tr>
<tr>
<td>S60</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>60 min CNT sonication</td>
</tr>
<tr>
<td>H60</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>60 min CNT homogenizer</td>
</tr>
<tr>
<td>S40H10</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>40 min sonication then 10 min homogenizer</td>
</tr>
<tr>
<td>H10S40</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>10 min homogenizer then 40 min sonication</td>
</tr>
<tr>
<td>H40S10</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>40 min homogenizer then 10 min sonication</td>
</tr>
<tr>
<td>S30H30</td>
<td>523</td>
<td>210</td>
<td>2.3</td>
<td>0.1</td>
<td>30 min sonication then 30 min homogenizer</td>
</tr>
</tbody>
</table>

Where:

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Figure 1: Schematic diagram showing differences between mixing sequences

III. RESULTS AND DISCUSSION

This paper presented a novel dispersion method for de-agglomeration of CNT particles. The dispersion level was determined through evaluating the CNT particle size distribution, specific surface area, and cement pastes compressive strength results. In addition micro-structural will be introduced in order to expand our knowledge about the used techniques and their effect on the dispersion level.

A. Compressive Strength

Figures (2-4) showed the early age compressive strength of different CNT de-agglomeration methods
and their gain as compared to the control batches. The following points were observed:

- The application of either sonicator or homogenizer enhanced the dispersion of CNT particles.
- All methods increased the compressive strength of the cement paste as compared to sample containing SP.
- The method S30H30 (30 min sonication then 30 min homogenizer) got a loss in the compressive strength as compared to the sample containing SP and CNT.
- The optimum treatment method of CNT (S40H10, 40 minutes sonication then 10 minutes homogenizer).
- The optimum method obtained a gain 18% in compressive strength as compared to cement paste contained carbon Nanotube and superplasticizer (C/CNT/SP) and 38% as compared to cement paste contained superplasticizer only (C/SP).
- For the mix H60 (60 minutes homogenizer), the homogenizer increased the compressive strength by 35% as compared to the mix containing SP. This can be attributed to the homogenizer effect in decreasing the agglomerates but at the same time it influenced the particle size distribution changing it to a narrower distribution (Hugo Santos et al. 2009 and Rashad, Alaa M. 2014), as it breaks the CNT particles into equal sizes which is not recommended to act as bridges for different sizes of cracks into the matrix.
- Gain in compressive strength as compared to C/CN/SP refers to the effect of method of dispersion.

Particle size distribution shown in figures (5 and 6) noted that:

- 20% of particles in the size of nanometer varies between 0.01 to 0.1 um, however 20% of particles for the as received sampled in the range of sizes 100 to 1000 um.
Figure 3: Gain in 7 days compressive strength for cement pastes studying different methods for CNT treatment as compared to cement paste containing superplasticizer and CNT.

Figure 4: Gain in 7 days compressive strength for cement pastes studying different methods for CNT treatment as compared to cement paste containing superplasticizer only.
Figure 5: Particle size distribution of as received and optimum method for CNT treatment

Figure 6: Cumulative density of as received and optimum method for CNT treatment
Figures (7 and 8), shows a 3D curve and its projection relating the sonicator application times and the homogenizer times with the resultant compressive strength of cement composites, while figure 9 shows the dispersion status of the carbon Nano tubes before and after applying the optimum dispersion technique. The figures reveal that the proposed technique is highly effective not only in dispersing the CNT particles but also in keeping it well dispersed for longer times.

Figure 7: 3D graph between time of sonication and homogenizer and compressive strength

Figure 8: Contour graph presents the relation between time of sonication and homogenizer and compressive strength
B. Microstructure Analysis

SEM micrographs showed that:
- The plain cement composite which contained a lot of voids as compared to sample contained treated CNT which had lower voids and well dispersed carbon Nanotubes, shown in figure 10.

TEM shown in figure 11 indicates a better dispersion performance for the CNT sample after treatment using S40H10 method as compared to the as received sample.
Figure 10: SEM micrograph of the plain cement paste (a) as compared to optimum CNT treatment method (S40H10) cement paste (b)

Agglomerated CNT
IV. CONCLUSION

- The optimum treatment method of CNT (S40H10) obtained a gain 62% in compressive strength.

- Homogenizer (H60) breaks the CNT particles into equal sizes which are not recommended to act as bridges for different sizes of cracks into the matrix.

REFERENCES
