EFFECT OF ADDING POLAR IMPURITIES ON CARBON NANOTUBES AND CONCRETE BONDING STRENGTH

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Summary: The addition of nanotubes (CNT) in the production of concrete has gained attention in recent times due to the ability of the nanotubes in increasing the desired properties of concrete. However, one of the major drawbacks in using nanotubes is the lack of dispersion and bonding between the tubes and cement matrix material. This research experimentally investigates the effect of adding polar impurities to even the dispersion of carbon nanotubes in the cement matrix and examining its influence on concrete strength for the same cement to water ratio. The cementitious material used in the study was composed of Portland cement, crushed rocks and natural sand. A control mix was prepared using 0% CNTs, while other mixes contained varying percentages of CNTs, CNTs with polar -OH and -COOH impurities and CNTs initially dispersed in water. Concrete samples in the form of cubes of 5cm sides were prepared and cured for two different time periods (7 and 56 days) and then tested for compressive strength. Results show the effect of the polar impurities of the dispersion as well as the strengthening of the concrete. CNTs with polar –COOH impurity reveal the highest improvement in the concrete’s mechanical properties. The 56-day results indicate a 60% increase in compressive strength.

1 INTRODUCTION

Concrete is one of the most widely used and economical materials of construction. Concrete is a multi-phase composite material that is known to age over time. Improving its performance has been a major goal for many researchers. A relatively new field of study is the use of nanoparticles in concrete to improve its workability, durability and strength in addition to adding new functionalities. The outstanding mechanical properties of carbon nanotubes (CNTs) highlight them as potential candidates for concrete reinforcement. The strength of the CNTs is directly related to the strong C=C bond and the relatively small number of defects present in the tubes. It is said to possess “a hundred times the strength of steel at one sixth of the weight” [1]. The CNT are characterized by thermal stability up to
2800°C [2]. However, their surfaces have very low friction, so it is very difficult for them to bind together or with the cement matrix material [3]. In addition, carbon nanotubes are packed together by Van der Waals attraction forces into crystalline ropes during production. These ropes tend to aggregate and result in lack of ability of CNT powder to disperse in aqueous or organic solutions [2]. Incorporating the unique mechanical properties of CNTs in cement composites is complex, and results vary between studies. One way to increase the solubility of CNTs in water is through the addition of polar impurities such as OH or COOH end groups.

2 EXPERIMENTAL

2.1 Material

The concrete mix consisted of Type 1 Portland cement, crushed sand, water and CNTs. Type I normal Portland cement with a surface area of 355 m²/kg was used as binder material. Crushed sands, with particle size in range of 0.15-4.75mm, a specific gravity of 2.65 and water absorption of 5% were used as fine aggregate.

Four different types of CNTs were used in the experiment to compare the effect of polar impurities on improving the CNTs dispersion in concrete and thereby its mechanical properties. Four Industrial grade multiwall carbon nanotubes - CNTs, CNT-OH, CNT-COOH and CNT-water dispersed - were used for the study. All grades has 88±% purity, an outside diameter that varies from 20-40 nm, an inner diameter of 5-10 nm and a length varying between 10-30 µm, as shown in Figure 1.

![Figure 1: Scanning electron Microscope (SEM) image of Industrial grade CNTs at different magnifications.](image)

The industrial-grade -OH functionalized multi-walled carbon nanotubes (CNT-OH) contained 1.55-1.71 wt% -OH. The industrial-grade -COOH functionalized multi-walled carbon nanotubes (CNT-COOH) contained 1.36-1.50 wt% -COOH. The water dispersed CNTs have a dispersant content of 1.4-1.6% and are stable for up to 6 months at room temperature. Aromatic modified polyethylene glycol ether was used as solvent for the dispersion procedure. All CNTs were purchased from [4].

2.2 Specimen Preparation

To ensure consistency in sample preparation and avoid variance in results the same
procedure was adopted in preparing all samples. A water/binder ratio of 0.3 and a sand to cement ratio of 1.3 was used for all experiments. CNTs % was fixed to 1% in all tests.

After properly weighting the sand and cement as per the mix design, ASTM standard Hobart mixer was used for mixing process. All the dry material was added to the mixer and mixed for 4 minutes. Meanwhile the correct weight of carbon-nanotubes was mixed with water. The CNTs-water mix was whisked for 5 minutes and then mixed with the dry mix in the mixer for 5 minutes to ensure a consistent sample. The concrete paste was then used to fill 1/3 of the mold and placed on the shaker table. Concrete was then compacted while shaking the mold. The process was repeated 3 times until the mold was full. The concrete was placed in water for curing, and then crushed by a universal testing machine after 7 and 56 days from samples preparation date.

![Sample preparation equipment](image)

Figure 2: Sample preparation equipment.

### 2.3 Testing Procedure

Table 1 summarizes the list of 10 sets of mixtures that were prepared to test compressive strength of the material. Each set of experiment was repeated three times. Several factors were examined, including impurities and number of days. The CNTs concentration was kept constant at 1 % while maintaining same water to binder ratio.

<table>
<thead>
<tr>
<th></th>
<th>Days</th>
<th>CNT %</th>
<th>Polar impurities</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7</td>
<td>Control</td>
<td>No CNTs</td>
</tr>
<tr>
<td>2</td>
<td>7</td>
<td>1.00</td>
<td>CNT</td>
</tr>
<tr>
<td>3</td>
<td>7</td>
<td>1.00</td>
<td>OH</td>
</tr>
<tr>
<td>4</td>
<td>7</td>
<td>1.00</td>
<td>COOH</td>
</tr>
<tr>
<td>5</td>
<td>7</td>
<td>1.00</td>
<td>Dispersed</td>
</tr>
<tr>
<td>6</td>
<td>56</td>
<td>Control</td>
<td>No CNTs</td>
</tr>
<tr>
<td>7</td>
<td>56</td>
<td>1.00</td>
<td>CNT</td>
</tr>
<tr>
<td>8</td>
<td>56</td>
<td>1.00</td>
<td>OH</td>
</tr>
<tr>
<td>9</td>
<td>56</td>
<td>1.00</td>
<td>COOH</td>
</tr>
</tbody>
</table>

Table 1: Different sets of mixtures prepared.
3 RESULTS

Figure 3 shows the effect of the functionalization group on the compressive strength ratio of concrete. The compressive strength ratio was calculated by normalizing the compressive strength value by the compressive strength value of control experiment (with no CNTs) to give a clear indication of the increase/decrease in strength. A ratio less than 1 indicates a decrease in strength and vice versa. As seen in Figure 3, a 1% CNT addition does not increase the strength of concrete. This was attributed to agglomeration effect and non-dispersion of CNTs, leading to areas of stress concentrations and weakening of the concrete. On the other hand, adding -OH and -COOH functionalized CNTs have a significant effect on improving the strength especially with time. After 56 days, the compressive strength of concrete reinforced with COOH-functionalized CNTs increased by 65%, while that functionalized with OH group increased by 35%.

Figure 3: Effect of polar impurities on the compressive strength of concrete.

Figure 4 compares the increase in strength due to COOH functionalization of CNTs with the use of dispersed CNTs water mixture. Results indicate that the effect of COOH functionalization is more significant than that of dispersed CNTs (65% versus 42%). This shows that using functionalized CNTs also adds to the bonding strength between the CNTs and cement hydrates, not just the dispersion.
Figure 4: Compressive strength ratio for different polar impurities.

Figure 5 shows the increase in strength that develops in the concrete with time. The functionalized CNTs exhibit highest slope followed by the dispersed CNTs and then CNTs.

![Compressive strength ratio with time.](image)

**Figure 5:** Compressive strength ratio with time.

## 4 CONCLUSIONS

Functionalizing the CNTs has a significant influence on its dispersion in the concrete mix, thereby greatly affecting its mechanical properties. Results from this research illustrate that using CNTs with functionalized groups does not just add to the dispersion but also enhances the bonding strength with the concrete mix. This positive effect was also seen to increase with time. In order to improve the mechanical properties of concrete using CNTs, it is necessary to consider the COOH functionalized CNTs since preliminary results showed an
increase of strength of 60% in 56 days. This is the focus of ongoing research.

5 ACKNOWLEDGEMENTS

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REFERENCES