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## Effect of mixing duration on flexural strength of multi walled carbon nanotubes cementitious composites



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#### HIGHLIGHTS

• Long mixing durations improve CNT dispersion within the cement paste.

• Effect of CNTs on the flexural strength of cement pastes was investigated.

• Surfactant/superplasticizer overdosing causes a flexural strength reduction in CNT cementitious composites.

• Quantification of CNT dispersion in cement was performed.

• Well dispersed CNTs results in lower voids content in CNT cementitious composites.

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#### ABSTRACT

This paper investigates the effect of mixing duration on the flexural strength of multi-walled carbon nanotubes (MWCNTs) reinforced cementitious composites. Each batch containing 0.03%, 0.08%, 0.15% or 0.25% MWCNT to cement weight fractions was prepared using mixing durations of 1.5, 15, 30, and 60 min. On the 28th day, the flexural strengths of the specimens prepared from the batches, were obtained using three-point bending tests. The specimen microstructures were investigated using scanning electron microscope (SEM) and image analysis techniques. The purpose of the investigation was to identify potential relationships between strength, dispersion, void ratio, and mixing duration. The results showed that increasing the mixing duration improved the dispersion and strength of most MWCNT reinforced cementitious batches. They also showed a high flexural strength increase for MWCNT reinforced cementitious batches containing a 0.25% CNT to cement weight fraction and mixed for 60 min. Moreover, the results showed that increasing the mixing duration resulted in a reduction of the percent void ratio of MWCNT reinforced cementitious batches. These findings are significant for a wide variety of concrete applications where higher flexural strength and small void are needed.

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#### 1. Introduction

Carbon nanotubes (CNTs) are cylindrical shell shapes of carbon atoms positioned in episodic hexagonal arrangements. They possess attractive properties such as high strength and stiffness, chemical stability, and electrical conductivity [1-4]. These properties make CNT-based composites suitable for several applications. In the last few years, the effect of CNT addition to cement-based materials has been investigated [5–17]. Incorporation of CNTs in cementitious composites can achieve higher flexural strength gains than adding minerals or reducing water. Fly ash, silica fumes as well as fibers of different types and sizes could delay crack development on micron scale which may result in optimizing the flexural strength as well as other mechanical properties. However, working with carbon nanotubes or nanofibers may help in preventing the cracks initiation. Evidences in literature studies showed the ability of nanofilaments to bridge cracks at nano scale (Kumar et al. [18] and Tyson et al. [19]). This may be the reason for the remarkable flexural strength increases of up to 200% that were reported in previous studies (Al-Rub et al. [8], Yazdanbakhsh et al. [20]). These

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findings may also be developed with the aid of extra minerals and less water content to produce even higher flexural strength. Even though a number of researchers reported improvements in the mechanical properties of CNT-reinforced cementitious composites, others did not.

The contradiction in the findings may be due to the dispersion of the CNTs within the composite matrix. CNTs have a tendency to agglomerate due to their high van der Waals forces. Nonuniformly dispersed CNTs will not form a continuous network within the composite matrix to sustain load transfer and/or prevent crack growth. Moreover, CNT agglomeration could behave as local defects because of their low strength in the direction normal to their longitudinal axes. Methods, such as ultrasonication of CNTs and functionalization, were successful in providing good dispersion in aqueous solutions. However, when mixed with the cement paste, low dispersions were still observed. To the authors' best knowledge, no research was conducted on studying the effect of mixing patterns on the dispersion and flexural strength of CNTreinforced cementitious composites. The mixing pattern is investigated herein as a major factor in improving CNTs dispersion quality by reducing the size of cement grains and allowing for better dispersion medium (Fig. 1).

Two essential physical processes, intensive and extensive, are used in cement mixing operation studies [21]. Intensive mixing efficiently reduces the agglomeration scale of particles bonded by surface tension. This is achieved by providing hydrodynamic stresses that exceed the strength of the inter-particle bond in the agglomerates. On the other hand, extensive mixing deforms the fluid by applying high shear rates, which results in increasing the interface area between the cement particles and in reducing the fluid plastic viscosity. The selection of the mixing process is governed by the need to obtain a homogeneous cement mix that is free of clumps and exhibits a low viscosity.

In CNT cement composite studies, several mixing methodologies with various durations, sequences, and intensities were used to produce homogenous batches with dispersed CNT particles. Luo and Koo [22] used Kenwood KNM20 planetary mixer with a speed of 98 rpm for mixing CNT-reinforced cement pastes. The sequence of mixing consisted of mixing for 2 min, resting for 2 min, and then mixing for another 2 min. The results have shown a reduction in the compressive strength of all composite batches. Luo and Koo [23] used a multispeed planetary mixer to mix the cement with the dispersed solutions containing carbon nano fibers (CNFs) and carbon nano tubes (CNTs) for 7 min in order to improve the uniformity of the paste. To remove any entrained air, the paste was placed in a vacuum chamber for 3 min after the mixing process. The obtained results illustrated an improvement in the strain capacity, flexural strength and modulus of elasticity of some batches. Makar et al. [24] and Li et al. [25] mixed the materials according to ASTM C305 using a standard Hobart mixer and

obtained improvement in the mechanical properties of some batches. On the other hand, Li et al. [26] prepared mortar mixes with CNTs by manually mixing the chemical admixtures with water. Subsequently, the CNTs were manually and mechanically blended with the mortar mixes for 5 and 2 min, respectively. Initially, the fine aggregates and cement were manually blended and then dumped into a 120 L concrete mixer. The CNT solution was gradually added to the mix and stirred with the cement and fine aggregates for 10 min. Their results show an increase in the compressive strength, splitting tensile strength and secant modulus of elasticity in the batches of the lowest CNTs content. Sanchez and Ince [27] used a 600W Oster BVCB07-Z blender for mixing cement with nano fibers at approximately 7500 RPM. The obtained results demonstrated a significant increase in the flexural strength, resilience and modulus of elasticity of all composite batches. Corinaldesi et al. [28] mixed silicon fume, cement, sand, water reducing agent and other ingredients in a I-160A rotary mixer for 2 min. Then, the remaining two-fifth of the water was added and the mixer was stirred for additional 2 min. The prepared CNT solution was placed into the mixer. Then, the mixture was stirred for 3 min. The results showed a slight increase in the compressive strength and modulus of elasticity. However the results illustrated a decrease in the flexural strength of all batches.

Despite the fact that a significant number of mixing methodologies were reported in the literature, there is no standard and systematic procedure that can yield consistent dispersion of the CNTs within the cement matrix. The standard ASTM C305 mixing procedure used to prepare laboratory samples of cement pastes or mortars includes a batch rotation at a speed of 140 rpm for 0.5 min followed by a rotation at a speed of 285 rpm for 1 min. The procedure is investigated herein to determine its suitability for batches with high CNT weight fractions. Therefore, the aim of this study is to investigate the effect of mixing duration on the dispersion and flexural strength of CNT cementitious composites. The factors affecting the flexural strength are investigated by inspecting the microstructure (e.g., CNT dispersion, void volume fraction, and micro-cracks) of all batches using qualitative and quantitative analysis techniques.

#### 2. Testing methodology

In this study, four mixing durations were applied to cement batches with several CNT weight fractions. Table 1 summarizes the experimental design. It includes the prepared batches, mixing durations, CNT weight fractions, and prepared specimen numbers. It is worth noting that ASTM C305 mixing duration is 1.50 min. The testing methodology started with the preparation of the batches and specimens, followed by the measurement of their flexural strength using three-point bending tests. Then, the microstructures

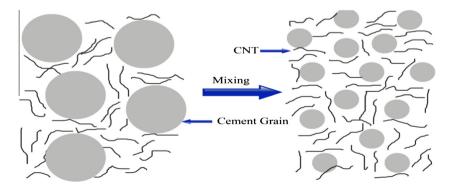


Fig. 1. Schematic of mixing effect on cement grain size.

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