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Effect of Cellulose Nanofibers on the Compressive Strength Enhancement of Cement Pastes at Early Ages

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Abstract **-** This study investigated the effect of cellulose nanofibers (CNFs) on the early-age compressive strength development of cement pastes. CNFs were incorporated at concentrations of 0.05%, 0.1%, and 0.15% by weight of cement using a combined ultrasonication and magnetic stirring technique to ensure proper dispersion. The impact was evaluated at 3 and 7 days of curing, with microstructural analysis conducted using scanning electron microscopy (SEM). Results showed that all CNF-modified mixtures exhibited enhanced compressive strength compared to the control, with 0.1% CNF concentration showing optimal performance. At 3 days, improvements of up to 49.7% were achieved with 0.05% CNFs, while at 7 days, the 0.1% CNF mixture demonstrated the highest strength enhancement of 69.6%. The strength development analysis revealed significant improvements in hydration rates, with CNF-modified mixtures showing up to 265.5% higher strength gains compared to the control. SEM observations confirmed the formation of a well-integrated fibrous network and enhanced matrix densification in CNF-modified samples.

Keywords**:** Bio-derived nanomaterials, cellulose nanofibers, nano-reinforcement, cementitious materials, mechanical properties

1. Introduction

Nanotechnology has transformed the construction industry by enabling the development of high-performance materials with enhanced properties [1], [2]. Among these advancements, carbon-based nanomaterials, such as carbon nanotubes and graphene oxide, have demonstrated significant potential to improve concrete properties [3]. However, their practical application is limited by challenges such as high production costs, energy-intensive manufacturing processes, and finite graphite reserves [4]. These limitations have led to increased interest in sustainable, bio-based alternatives like cellulose nanofibers (CNFs). CNFs are derived from renewable sources such as wood, agricultural residues, and bast fibers, making them an environmentally friendly and cost-effective material for construction applications. Known for their exceptional mechanical properties [5], high aspect ratios, and hydrophilic nature, CNFs are effective in enhancing the mechanical performance of cementitious composites. These nanoscale reinforcements complement broader advancements in fiber-reinforced concrete (FRC), where the inclusion of discrete fibers has long been employed to enhance mechanical performance and control cracking [6], [7].Studies have shown that CNFs improve the hydration process, matrix densification, and mechanical strength [8]. Their hydrophilic properties allow them to absorb and retain water during mixing, facilitating internal curing and improving hydration, particularly at early ages [9]. These advantages position CNFs as a promising reinforcement for enhancing cementitious composites.

Compressive strength enhancements have been observed with CNF concentrations in the 0.05–0.15 wt.% range. For instance, Hisseine et al. [10] reported a 26.5% increase within this range, while Liang et al. [11] achieved a 46.7% improvement at 0.15 wt.%. However, higher concentrations often lead to agglomeration and reduced performance, as demonstrated by Mejdoub et al. [12], where strength reductions were observed at concentrations exceeding 0.5 wt.%. Additionally, differences in CNF sources significantly influence their properties, which in turn affect their performance in cementitious composites. While CNFs have shown considerable promise, variations in reported results indicate a need for further investigation to establish an optimal concentration range. Discrepancies in findings highlight the influence of factors such as dispersion, dosage, and material properties, necessitating more detailed studies on their effect on mechanical performance. This paper focuses on evaluating the effect of cellulose nanofibers (CNFs) on the compressive strength of cement pastes. The study investigates varying CNF concentrations within the 0.05–0.15 wt.% range and assesses their impact on compressive strength at early curing ages (3 and 7 days), with a constant dispersion duration of 10 minutes.

2. Experimental Program

Ordinary Portland Cement (OPC) Type I, classified as CEM I, Class 42.5 N under BS EN 197-1:2011, was sourced from Sharjah Cement Factory and used as the primary binder. Commercial cellulose nanofibers (CNFs) from Nanoshel LLC, USA, were incorporated as nano-reinforcements at concentrations of 0.05%, 0.1%, and 0.15% by weight of cement. A control mixture without CNFs was also prepared, with all mixtures using a fixed water-to-cement (w/c) ratio of 0.35, as previous studies have shown that wood-derived CNFs are most effective in cement composites with w/c ratios between 0.35 and 0.45. CNF dispersion was achieved using ultrasonication and magnetic stirring. For each mixture, 400 ml of the total mixing water was used to prepare the CNF suspension, which was subjected to a probe sonicator operating at 40% amplitude for a duration of 10 minutes. Simultaneously, magnetic stirring at 450 RPM ensured a uniform and homogeneous suspension throughout the dispersion process.

Cement paste mixtures were prepared in accordance with ASTM C305-20. The CNF suspension, along with the remaining water, was added to the cement, and the mixture was subjected to 30 seconds of low-speed mixing, 1 minute of high-speed mixing, and an additional 30 seconds to ensure homogeneity. The fresh paste was cast into steel molds, vibrated to remove entrapped air, and demolded after 24 hours. Specimens were then wet-cured under controlled conditions. The compressive strength of cement paste specimens was evaluated at curing ages of 3 and 7 days, following ASTM C109/C109M-20. Testing was performed using an MTS compression machine with a 300 kN capacity. For each mixture, three 50 mm cubic specimens were tested at each curing age. A constant loading rate of 7 kN/sec was applied, and the load-strain response was recorded until failure.

3. Results and Discussion

3.1 Compressive Strength

The compressive strength of cement pastes with varying concentrations of CNFs is presented in Fig. 1 for curing ages of 3 and 7 days. The results indicate that incorporating CNFs significantly improved compressive strength compared to the control (0% CNFs) for both curing ages.

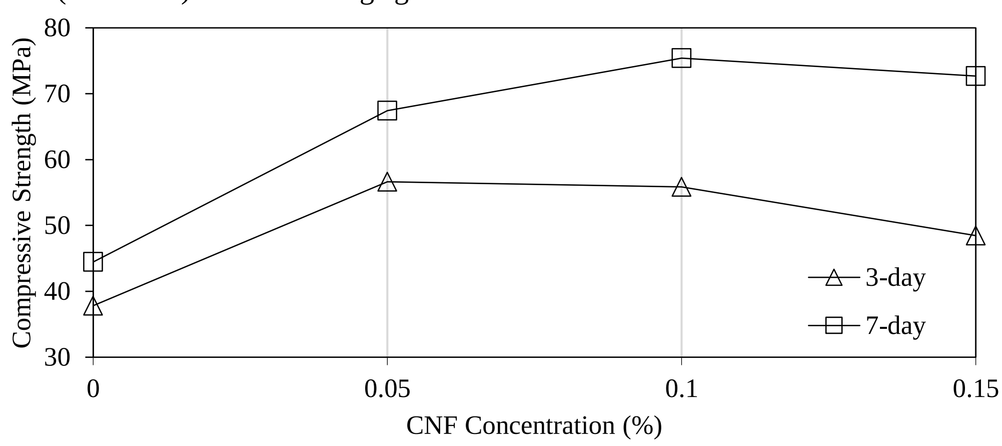


Fig. 1: Effect of CNF concentration on compressive strength development of cement pastes at 3 and 7 days.

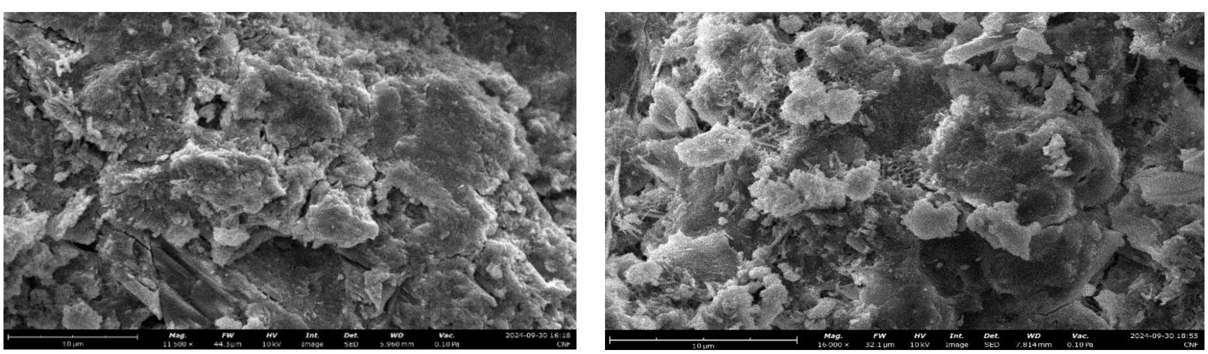
At 3 days, the control mixture achieved a compressive strength of 37.82 MPa. The addition of 0.05% CNFs resulted in an increase to 56.63 MPa, reflecting a 49.7% improvement over the control. Further increasing the CNF concentration to 0.1% yielded a strength of 55.86 MPa, a 49.7% improvement compared to the control. At 0.15% CNF concentration, the strength was 48.47 MPa, showing a 28.2% increase over the control. The decline at higher CNF concentrations may be attributed to agglomeration, which can reduce the effectiveness of nanofiber dispersion and compromise matrix integrity. These findings align with previous studies that reported optimal CNF concentrations between 0.05-0.1% for enhanced mechanical properties [10], [11]. At 7 days, the control mixture achieved a compressive strength of 44.45

MPa, while mixtures with 0.05% and 0.1% CNFs demonstrated higher strengths of 67.42 MPa and 75.40 MPa, representing increases of 51.7% and 69.6% respectively. The mixture with 0.15% CNFs exhibited a compressive strength of 72.70 MPa, showing a 63.5% increase over the control but lower than the 0.1% mixture. This behavior can be attributed to the optimal dispersion achieved at 0.1% CNF concentration, which provides maximum surface area for nucleation while maintaining adequate spacing between fibers to prevent agglomeration

The strength development between 3 and 7 days revealed the remarkable influence of CNFs on cement hydration and matrix densification. While the control mixture showed a moderate strength gain of 6.63 MPa, the CNF-modified mixtures exhibited substantially higher development rates. The incorporation of 0.05% CNFs led to 62.7% higher strength gain compared to the control mix. The mixture with 0.1% CNFs demonstrated superior performance with a 194.7% improvement in strength development compared to the control. Notably, despite its lower early-age performance, the 0.15% CNF mixture achieved the highest strength gain corresponding to a 265.5% enhancement over the control. This enhanced strength development can be attributed to several mechanisms: CNFs acting as nucleation sites for cement hydration products [13], bridging of micropores and microcracks due to their high aspect ratio [10], and the interaction between CNF surface functional groups and cement hydration products promotes stronger interfacial bonding. The exceptional strength gain at 0.15% CNF content suggests that initial agglomerates might serve as concentrated nucleation sites for delayed hydration, eventually contributing to matrix densification. These findings of strength enhancement compared to the plain counterpart mix were more significant than other outcomes found in similar studies investigated CNFs at similar concentrations, likely due to the moderate dispersion duration, which indicate that the CNFs structure were not degraded, resulting in effective bridging and nucleation effects [10], [12], [13].

3.2 Scanning Electron Microscopy (SEM)

The SEM images provide valuable insights into the microstructural modifications induced by CNF incorporation in cement paste. Fig. 2(a) shows the control sample exhibiting characteristic features of plain cement paste, including heterogeneous surface texture, visible pores, and occasional microcracks, with the typical rough, flaky appearance of cement hydration products. In contrast, Fig. 2(b) reveals the significant microstructural enhancement in the 0.1% CNF-reinforced cement paste, characterized by a well-integrated fibrous network creating a more complex and intricate surface morphology. The CNF incorporation resulted in notable pore refinement and increased surface roughness, suggesting improved matrix densification. Additionally, the modified microstructure indicates CNFs' role as nucleation sites for hydration products, evidenced by the more homogeneous distribution of hydration products and enhanced matrix density.



(a) (b)

Fig. 2: SEM images of cement paste specimens at 7 days: (a) control mixture showing typical cement hydration products and porous structure; and (b) 0.1% CNF-reinforced mixture exhibiting enhanced matrix densification and integrated fibrous network.

4. Conclusion

This study investigated the influence of CNFs on the compressive strength development of cement pastes at early ages (3 and 7 days). All investigated CNF concentrations (0.05%, 0.1%, and 0.15%) enhanced the compressive strength compared to the control mixture, with 0.1% identified as the optimal concentration. SEM analysis revealed a well-integrated fibrous network and enhanced matrix densification in CNF-modified samples. The findings are summarized as follows:

- CNF incorporation significantly enhanced compressive strength at both ages, with optimal performance achieved at 0.1% concentration (49.7% and 69.6% improvement at 3 and 7 days, respectively). Higher concentration (0.15%) showed reduced early-age effectiveness due to potential agglomeration, though maintaining strength improvements of 28.2% and 63.5% at 3 and 7 days.
- CNF-modified mixtures exhibited enhanced strength development between 3 and 7 days, with 0.1% and 0.15% CNF mixtures achieving remarkable gains of 194.7% and 265.5% higher than the control, respectively. SEM analysis confirmed the improved matrix densification through refined pore structure and homogeneous distribution of hydration products.

Further research should focus on optimizing the dispersion duration, particularly at higher CNF concentrations, and investigating the effects of CNF source, synthesis method, and functionalization. Surface functionalization may improve interfacial bonding with the cement matrix and enhance the nucleation of hydration products, leading to better mechanical performance.

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