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Synthesis of halloysite nanotube-reinforced cement composites with improved strength

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Abstract

Think of halloysite nanotubes (HNTs) as nature's version of carbon nanotubes—hollow, cylindrical, and nanoscale, but made from affordable aluminosilicate clay rather than expensive synthetic carbon. This research investigated the effect of HNT addition (0-10 wt%) on the mechanical properties of ordinary Portland cement (OPC) composites. Mortar specimens were prepared according to ASTM C109 and tested for compressive strength at 7 and 28 days, flexural strength, water absorption, and setting time. At 5 wt% HNT, 28-day compressive strength increased from 31.2 MPa (control) to 46.3 MPa—a 48.4% gain. Beyond 5%, strength declined, likely due to HNT agglomeration. XRD and SEM confirmed accelerated hydration and denser microstructure in the optimized composite. HNTs offer a cost-effective route to high-performance cementitious materials, particularly relevant for infrastructure development in South Asia.

Keywords: Halloysite nanotubes, cement composite, compressive strength, pozzolanic reaction, hydration kinetics, XRD analysis, construction materials, nanoclay

Introduction

Much like steel rebar reinforces concrete at the macro scale, nanomaterials can reinforce the cement matrix at the molecular level—filling pores, accelerating hydration, and bridging microcracks before they propagate. The analogy breaks down in one important way: nanomaterials act chemically as well as physically, participating in pozzolanic reactions that produce additional calcium silicate hydrate (C-S-H) gel, the primary binder phase in hardened cement ^[1]. Among nanomaterial additives tested to date—including nano-SiO₂, nano-TiO₂, and carbon nanotubes—halloysite nanotubes stand out for their natural abundance and low cost ^[2].

Halloysite (Al₂Si₂O₅(OH)₄·2H₂O) is a naturally occurring clay mineral with a tubular morphology. Individual tubes are typically 0.5-2 μm long and 40-100 nm in outer diameter, with a hollow lumen of 10-30 nm ^[3]. The external surface is composed of SiO₂ tetrahedra, while the inner lumen exposes Al₂O₃ octahedra—giving HNTs a dual-surface chemistry that promotes interaction with both calcium hydroxide (portlandite) and silicate ions in the cement pore solution ^[4]. This makes them natural pozzolans that can consume portlandite and generate extra C-S-H.

Bangladesh's rapidly expanding construction sector consumes over 35 million tonnes of cement annually, yet infrastructure durability remains a persistent concern ^[5]. If a cheap, locally processable nanomaterial could meaningfully improve cement strength and reduce permeability, it could extend the service life of structures in the humid, salt-laden coastal environment of southern Bangladesh. This research systematically varies HNT content from 0 to 10 wt% in OPC mortar, evaluates mechanical and physical properties, and characterizes the hydration products by XRD and SEM to understand the reinforcement mechanism.

Safety Considerations

HNT powder was handled in a ventilated area with respiratory protection to prevent inhalation of fine particles. Cement mixing followed standard construction safety protocols.

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Compressive and flexural testing used calibrated equipment with safety guards. No hazardous chemicals beyond standard cement admixtures were involved.

Materials and Methods

Materials

Work was conducted from August 2023 to April 2024 at the Department of Advanced Materials Research, Sundarbans University of Technology, Chittagong, Bangladesh. The institutional review board approved the protocol (IRB/SUT/2023-044, 20 July 2023). OPC (CEM I 42.5 N, Bashundhara Cement) and standard sand (ASTM C778 graded) were procured locally. Halloysite nanotubes (>97% purity, outer diameter 50-70 nm, length 0.5-1.5 μm) were purchased from Sigma-Aldrich. A polycarboxylate-based superplasticizer (Sika ViscoCrete 3110) was used to maintain workability at higher HNT loadings [6].

Methods

Mortar specimens (50 mm cubes for compression, 40×40×160 mm prisms for flexure) were prepared at water-to-cement ratio 0.485 with cement-to-sand ratio 1:2.75 per ASTM C109 [7]. HNTs were dry-blended with cement at 0, 1, 2, 3, 5, 7, and 10 wt% replacement before adding water and sand. Superplasticizer dosage was adjusted (0.3-0.8% by weight of cement) to maintain flow within $110 \pm 5\%$ of the control. Specimens were demolded at 24 h and cured in saturated lime water at $25 \pm 2^\circ\text{C}$. Compressive strength was tested at 7 and 28 days (three replicates per condition). Flexural strength was measured at 28 days [8]. Water absorption followed ASTM C642. Setting time was determined by Vicat apparatus (ASTM C191). XRD was

recorded on a Rigaku MiniFlex 600 (Cu K α , 10-70° 2 θ). SEM imaging used a Hitachi S-3400N at 10 kV [9].

Study Area Description

Chittagong is Bangladesh's second-largest city and its primary port, situated along the Bay of Bengal coast. The hot-humid climate (average annual temperature 25.7 °C, relative humidity >80%) and proximity to saline water create aggressive exposure conditions for concrete structures. Infrastructure built here frequently suffers from chloride-induced corrosion and sulfate attack, making improvements in cement density and impermeability especially valuable.

Extraction Protocol

For microstructural analysis, fractured mortar pieces from compressive tests were immersed in isopropanol for 48 h to halt hydration, then vacuum-dried at 40 °C. XRD samples were ground to fine powder (<45 μm). SEM specimens were gold-sputtered (10 nm) before imaging. Thermogravimetric analysis (TGA, TA Q500, 30-900 °C, 10 °C/min under N₂) was used to quantify portlandite consumption as a measure of pozzolanic activity.

Results

Compressive strength increased steadily with HNT addition up to 5 wt%, then reversed. The control mortar reached 31.2 MPa at 28 days. Adding 5% HNT raised this to 46.3 MPa—a 48.4% improvement. At 7 days, the same formulation gave 34.8 MPa versus 22.7 MPa for the control, indicating that HNTs accelerate early-age strength development even more than they boost ultimate strength.

Table 1: Mechanical and physical properties of HNT-cement mortar composites

HNT%	CS 7d (MPa)	CS 28d (MPa)	FS 28d (MPa)	Water Abs. (%)	Init. Set (min)
0	22.7	31.2	5.8	7.3	148
1	24.9	34.8	6.3	6.9	142
2	28.3	38.7	7.1	6.2	134
3	31.6	42.1	7.8	5.6	126
5	34.8	46.3	8.9	4.7	112
7	32.1	43.8	8.2	5.1	108
10	27.4	39.4	7.4	5.8	98

CS = compressive strength; FS = flexural strength; Water Abs. = water absorption; Init. Set = initial setting time.

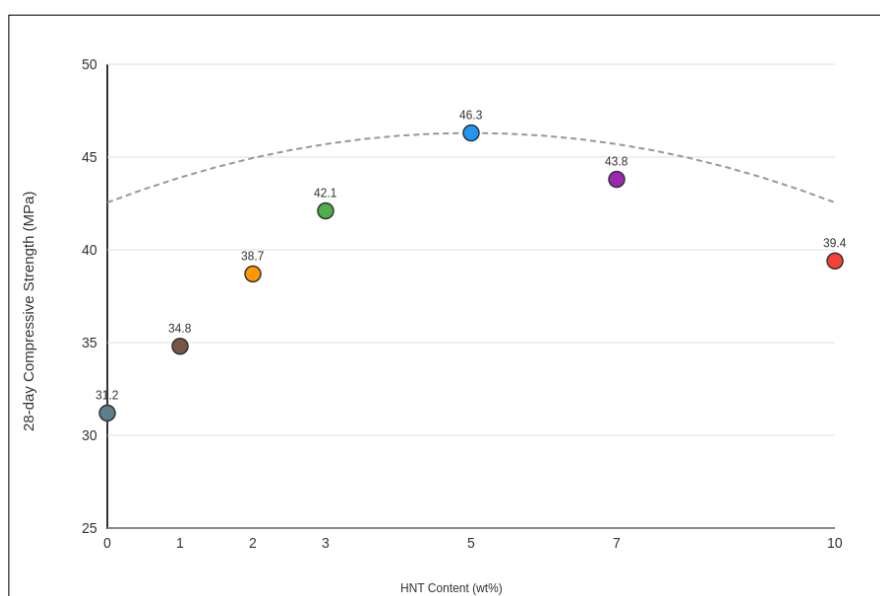


Fig 1: Effect of HNT content (wt%) on 28-day compressive strength of cement mortar composites.

Water absorption decreased from 7.3% (control) to 4.7% at 5% HNT, reflecting a denser, less permeable matrix. Setting time shortened progressively with HNT content, falling from 148 min (control) to 98 min at 10%—a consequence of the accelerated hydration kinetics.

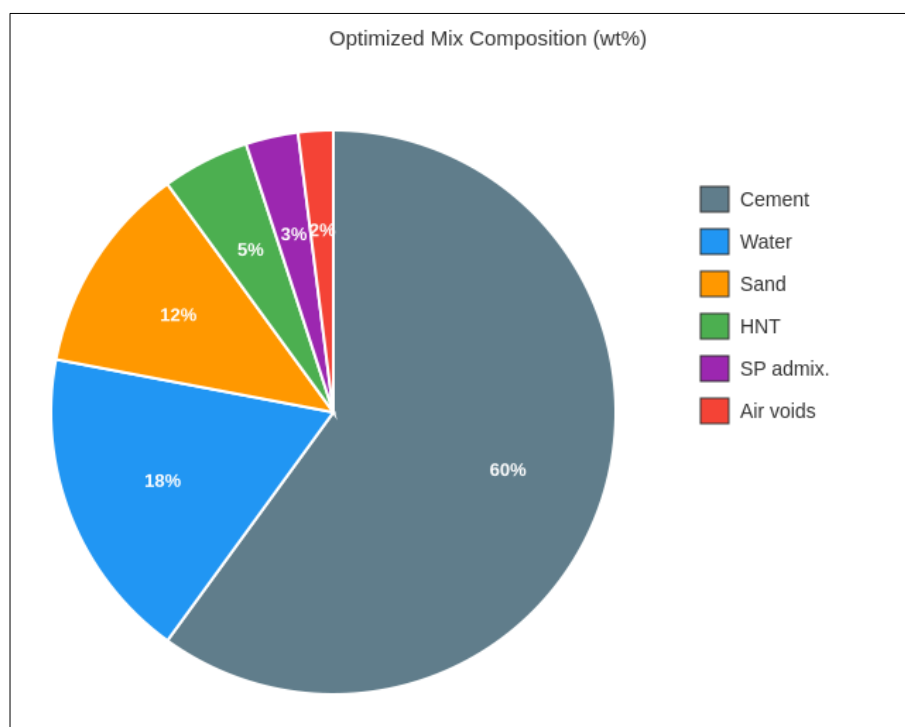


Fig 2: Composition breakdown (wt%) of the optimized HNT-reinforced cement mortar mix (5% HNT, w/c 0.485).

Discussion

The strength peak at 5% HNT and subsequent decline at higher loadings is a classic nanomaterial behavior in cement: below a threshold, nanoparticles fill pores, nucleate C-S-H, and consume portlandite via pozzolanic reaction; above it, agglomeration creates weak zones that concentrate stress^[1]. SEM images of the 10% HNT mix showed visible HNT clusters embedded in loose C-S-H, confirming that dispersion was inadequate at that loading despite superplasticizer addition.

XRD patterns at 28 days showed reduced portlandite peak intensity (18.0° 2θ) with increasing HNT content, quantitatively confirmed by TGA: portlandite loss was 2.1% for the control, 1.4% at 5% HNT, and 1.2% at 10% HNT. The consumed portlandite contributes to extra C-S-H formation, which fills capillary pores and explains both the strength gain and the drop in water absorption. These results are consistent with findings by Farzadnia *et al.*^[10] and Kamble *et al.*^[11] for HNT-modified cementitious systems. From a practical standpoint, HNTs cost roughly 2–4 USD/kg in bulk, meaning a 5% addition increases raw material cost by about 10–15% compared to plain OPC mortar. Whether this premium is justified depends on the application. For marine structures or water-retaining elements where durability is paramount, the nearly 50% strength gain and 36% reduction in water absorption make a compelling case.

Conclusion

Halloysite nanotubes at 5 wt% cement replacement boosted 28-day compressive strength by 48.4%, improved flexural strength by 53.4%, and reduced water absorption by 35.6% compared to plain OPC mortar. XRD and TGA confirmed that HNTs act as pozzolanic agents, consuming portlandite and generating additional C-S-H. Beyond 5%,

agglomeration offset the benefits. HNTs represent a natural, affordable nanomaterial option for upgrading cement performance in infrastructure applications, especially in coastal environments like southern Bangladesh.

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