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Evaluation of Adding Carbon Nanotubes to Lime Mortar: Mechanisms and The Effect of their Addition on Modifying the Internal Structure for Repairing Cracks in Heritage Buildings

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Abstract

Lime mortar is one of the oldest building materials used since ancient times in temples and historical monuments that still stand today. It is characterized by its role as a natural binder, its relative flexibility, and its compatibility with traditional stone materials, which makes it suitable for restoration and maintenance of historical structures. However, lime mortar faces fundamental challenges that limit its use in advanced construction applications. With the evolving need to preserve historical heritage and develop more sustainable and environmentally friendly building materials, research has focused on integrating nanomaterials with traditional mortar to enhance its mechanical and physical properties. Carbon nanotubes (CNTs) have shown positive effects when incorporated with various materials due to their unique mechanical and physical properties. The combination of lime mortar as a traditional and environmentally friendly material with CNTs as a modern reinforcing material enables the development of a composite that merges traditional authenticity with high mechanical performance. This research aims to investigate the effect of incorporating CNTs in varying proportions within lime mortar and to evaluate their impact on compressive and flexural strength, as well as internal structure and porosity. CNTs have been synthesized and purified, then SEM-EDX and TEM analyses were conducted prior to mixing with lime mortar in ratios of 0.01%, 0.03% and 0.3%, followed by mechanical and physical testing of the prepared samples, but the results did not achieve the desired outcomes.

Keywords

lime mortar, CNTs, SEM-EDX, TEM, mechanical and physical properties

1 Introduction

Lime mortar is a traditional binding material widely used in construction and heritage conservation because of the compatibility of its physical and mechanical properties with traditional stones and bricks, and its ability to adapt to old substrates without causing stress or physical damage [1]. It is classified into air lime mortar, which hardens gradually under natural conditions, and natural hydraulic lime mortar (NHL), which contains silicate or aluminate components allowing it to set in the presence of water [2]. Lime mortar is characterized by high vapor permeability, flexibility, and porosity, along with relatively low compressive strength compared to cement mortar, making it suitable for restoration applications [3], such characteristics make it effective in treating cracks, such as those observed in the Sakkakini Palace, (Fig. 1 (a), (b) and (c)). Its workability, porosity, early-age hardness, and long-term durability are influenced by factors such as water-to-binder ratio, aggregate characteristics, pozzolanic additives, and preparation and curing methods. The use of suitable pozzolans and optimized mix proportions improves fresh and hardened properties while maintaining historical compatibility [4]. Beyond restoration, lime mortar is increasingly used in sustainable hybrid mortars to reduce environmental impact. Studies have shown that incorporating organic waste ash or industrial residues into NHL mixtures enhances sustainability while maintaining

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Fig. 1 Cracks observed in the case study: (a) cracks in the basement wall; (b) cracks in the first floor; (c) cracks between the blocks

performance, and proper manufacturing practices ensure predictable behavior under different climatic conditions [5].

Despite these advantages, the mechanical and durability performance of lime mortar still requires improvement, which has encouraged the use of advanced materials such as nanomaterials. Recent research has focused on modified nanomaterials, particularly CNTs are nanoscale materials with exceptional properties that have been widely used to enhance the performance of cementitious materials [6]. CNTs are classified into single-walled (SWCNTs) and multi-walled (MWCNTs), with chirality influencing their electronic and mechanical properties, making proper selection essential for applications [7]. CNTs can be produced using several techniques, with their properties being strongly influenced by production and purification processes [8]. Purification is required to remove impurities while preserving CNT quality, although some treatments may introduce defects or functional groups [9].

The effectiveness of CNTs strongly depends on their dispersion within the mortar. Proper dispersion refines the pore structure and improves mechanical and durability properties, while agglomeration can negatively affect performance. Optimal improvements are typically achieved at very low CNT dosages, as higher contents may reduce performance due to dispersion challenges [10–12]. Previous studies showed that adding dried wood ash to lime mortar improves compressive strength and drying behavior due to its internal structure [13]. The incorporation of fly ash also enhances compressive strength over time, particularly at later ages [14]. The addition of silica nanoparticles to hydraulic lime has been reported to influence porosity and water absorption [15]. Nano-lime has been shown to improve bonding properties and structural performance, particularly in limestone restoration applications [16].

2 Research objectives

This research investigates the incorporation of carbon nanotubes (CNTs) into lime mortar to improve its mechanical and physical properties for crack repair in heritage buildings. Multi-walled carbon nanotubes (MWCNTs) are incorporated at low dosages (0.01%, 0.03%, and 0.3% by binder weight) to evaluate their influence on mortar behavior. These percentages were selected to assess the effect of very low CNT concentrations, with the expectation that low percentages enhance properties, while higher percentages may cause agglomeration and reduce performance. Standard and modified samples are tested to determine the optimal CNT content.

3 Material and methods

MWCNTs were added to lime mortar to enhance its properties and durability. The nanotubes were treated and characterized using SEM and TEM, and mortar samples with different CNT contents were tested to evaluate their effect on performance.

3.1 Characterization of MWCNTs

MWCNTs produced by CVD (10–40 nm diameter, up to 5 μm length) were supplied by the Egyptian Petroleum Research Center. Fig. 2 illustrated that TEM revealed impurities affecting mixing, so the nanotubes were functionalized and dispersed before use. Post-treatment SEM and TEM confirmed impurity removal and suitability for lime mortar. TEM images showed tubular structures with some agglomeration, residual impurities, and structural defects that may influence their properties [17, 18].

3.2 Functionalization and Dispersion of MWCNTs

MWCNTs were purified and dispersed to improve their properties. One gram was treated with 350 mL of a 1:3 HNO_3/H_2SO_4 mixture for 12 hours to remove metallic

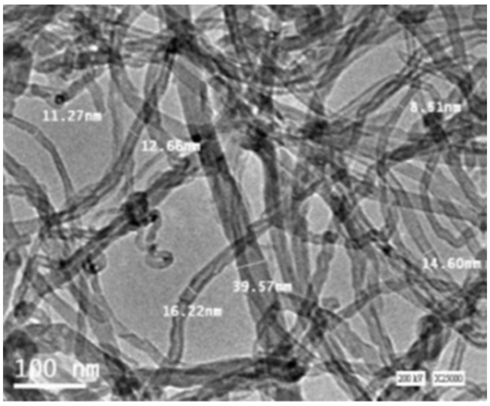
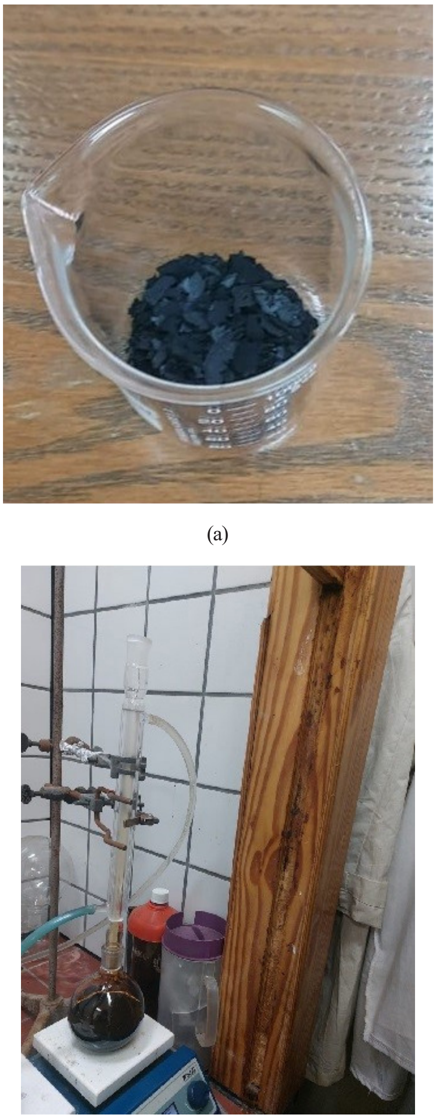


Fig. 2 TEM of MWCNTs as a powder before functionalization

and carbonaceous impurities, reducing the mass to 0.7 g Fig. 3 (a). The acids were removed by repeated washing with deionized water and standing for 24 hours. The sample was then ultrasonically dispersed in 700 mL of deionized water to achieve homogeneous dispersion Fig. 3 (b), a method widely reported to enhance dispersion in cementitious matrices [19].



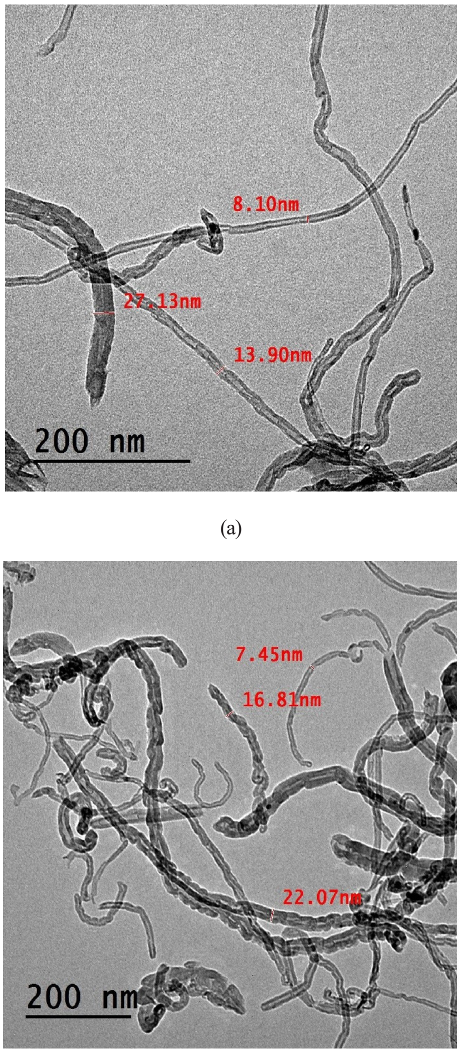
(b)

Fig. 3 Characterization of MWCNTs: (a) Morphology and volume of MWCNTs post-functionalization; (b) The ultrasonication-assisted dispersion process.

3.3 Characterization of purified MWCNTs

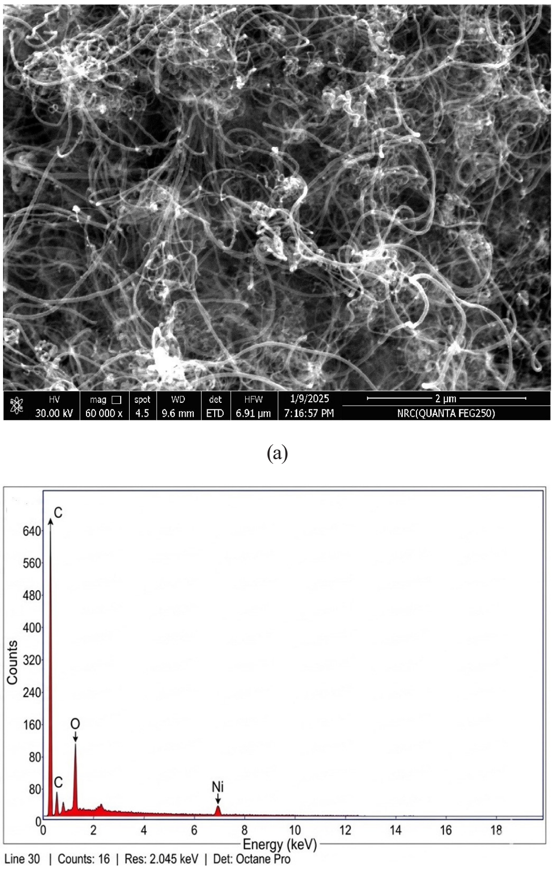
TEM analysis (Fig. 4) confirmed the structure and quality of MWCNTs, showing impurity-free nanotubes after functionalization. The measured diameters ranged from 8.10–27.13 nm for the solid sample and 7.45–22.07 nm for the liquid sample. The results also verified the removal of amorphous carbon compared to pre-functionalization [20].

SEM (Fig. 5 (a)) revealed a complex MWCNT network with morphological changes after treatment, including random structure and noticeable agglomeration at the nanoscale [21]. EDX analysis (Fig. 5 (b)) confirmed the presence of carbon and oxygen, indicating successful functionalization by acid oxidation [20].



(b)

Fig. 4 TEM micrograph of MWCNTs after functionalization (magnification 200nm): (a) MWCNTs are in the solid condition; (b) MWCNTs are in the liquid condition



(a)

(b)

Fig. 5 Characterization of functionalized MWCNTs:
(a) SEM micrograph showing surface morphology (magnification = 2 µm);
(b) EDX spectrum indicating elemental composition

3.4 Preparation of samples

The mortar samples consisted of hydraulic lime, sand, and MWCNTs. A standard mix with a sand-to-lime ratio of 3:2 was adopted based on commonly reported proportions for lime mortars used in conservation applications, with 100 mL of water. MWCNTs were initially added at 0.5%, 1%, and 1.5% by weight. However, these higher dosages resulted in internal cracking and shrinkage during curing. Repetition of the mixes yielded consistent results (Fig. 6 (a) and (b)). Consequently, lower MWCNT contents (0.01%, 0.03%, and 0.3%) were used. These samples remained intact during curing and after demolding, showing no visible cracking or damage (Fig. 6 (c), (d), (e), and (f)). Table 1 summarizes the composition of the mortar samples. After casting, all specimens were cured under controlled laboratory conditions at a temperature of 20 ± 2 °C and relative humidity of 60 ± 5% until the testing age (28 days). A total of 96 specimens were prepared, including 24 control samples and 24 samples for each MWCNT ratio, in addition to extra specimens to account for potential damage. Mechanical tests were subsequently conducted on the stable samples.

3.5 Physical and mechanical tests

The prepared mortar samples were evaluated following the specifications of the Construction and Building Research Center. Tests included:

Fig 6 Description of the samples after casting: (a) explain the thin cracking samples inside molds according the three highest percentages; (b) show the wide cracking samples inside molds according the three highest percentages; (c) show the standard samples; (d) show the percentage of 0.01% after preparation; (e) show the percentage of 0.03% after preparation; (f) show the percentage of 0.3% after preparation

Table 1 describes the prepared lime mortar samples and the percentage of MWCNTs in each sample

Symbol	Compound	Percentage of MWCNTs
A	Lime- Sand	Standard (0%)
S	Lime- Sand- MWCNTs	0.01%
F	Lime- Sand- MWCNTs	0.03%
M	Lime- Sand- MWCNTs	0.3%

- Compressive strength (Fig. 7 (a)): to determine load-bearing capacity and select the optimal mortar ratio;
- Flexural strength (Fig. 7 (b)): to assess resistance to bending and ensure durability;
- P-wave: to evaluate sample density and identify causes of deterioration and through it we will determine the porosity;
- Water absorption: to determine the effect of water on sample hardness;
- Shrinkage cracking (Fig. 7 (c)): to measure the ability to resist cracking under dry or humid conditions.

4 Results

4.1 Physical and mechanical tests

Mortar specimens were prepared in different dimensions according to the test type: 9 × 4.5 cm for compressive strength, P-wave, and water absorption; 12 × 6 cm for flexural strength; and 12 × 2.5 × 2.5 cm for shrinkage (ASTM C157) [22]. To assess durability, samples underwent an accelerated aging cycle involving immersion in 3% sodium sulfate, drying at 105 °C, washing, and final weight measurement. Mechanical tests were then repeated to evaluate the long-term performance of CNT-modified mortar (Fig. 8 (a) and (b), Table 2).

Compressive strength results showed that the control mix achieved the highest value before aging (1.39 kN), while strength decreased progressively with increasing CNT content, reaching 1.12 kN (−19%), 0.91 kN (−34%), and 0.63 kN (−55%) for 0.01%, 0.03%, and 0.3% CNTs, respectively. After aging, the control sample recorded 1.22 kN, and a similar decreasing trend was observed with CNT addition. The 0.01% mix showed no reduction (1.22 kN), while the 0.03% and 0.3% mixes decreased to 1.11 kN (−9%) and 0.50 kN (−59%), respectively.

Flexural strength results before aging showed that the control sample recorded 1.39, with a gradual decrease upon CNT addition to 0.50 (−64%) at 0.01%, 0.37 (−73%) at 0.03%, and 0.31 (−78%) at 0.3%. After aging, the control sample reached 0.56 kN, while the 0.01% sample increased to 1.08 (+93%), and the 0.03% and 0.3% samples decreased to 0.40 (−29%) and 0.24 (−57%), respectively.

P-wave results showed that the control sample had a velocity of 63.87 m/s. This slightly decreased to 63.53 m/s (−0.53%) at 0.01%, then dropped to 51.50 m/s (−19.37% *vs*. control; −18.93% *vs*. previous) at 0.03%, and further to 46.97 m/s (−26.46% *vs*. control; −8.79% *vs*. previous) at 0.3%.The variation in flexural strength values is attributed to the intrinsic sensitivity of the flexural test to minor variations in specimen preparation, while the overall trend remains consistent.

Water absorption results showed variations with CNT content. The control sample recorded 22%, while the 0.01% sample slightly decreased to 21.40% (−2.70%). The 0.03% sample increased to 22.37% (+1.70%), whereas the 0.3% sample decreased to 20.53% (−6.70%) compared to the control.

Shrinkage results at 28 days showed that the control sample recorded 0.31%. The 0.01% CNT mix increased to 0.40% (+29%), while the 0.03% mix decreased significantly to 0.20% (−35%). The 0.3% mix recorded 0.24% (−23% *vs*. control), although it remained higher than the 0.03% sample. The observed variations in mechanical and durability properties are interpreted in light of microstructural considerations and CNT dispersion.

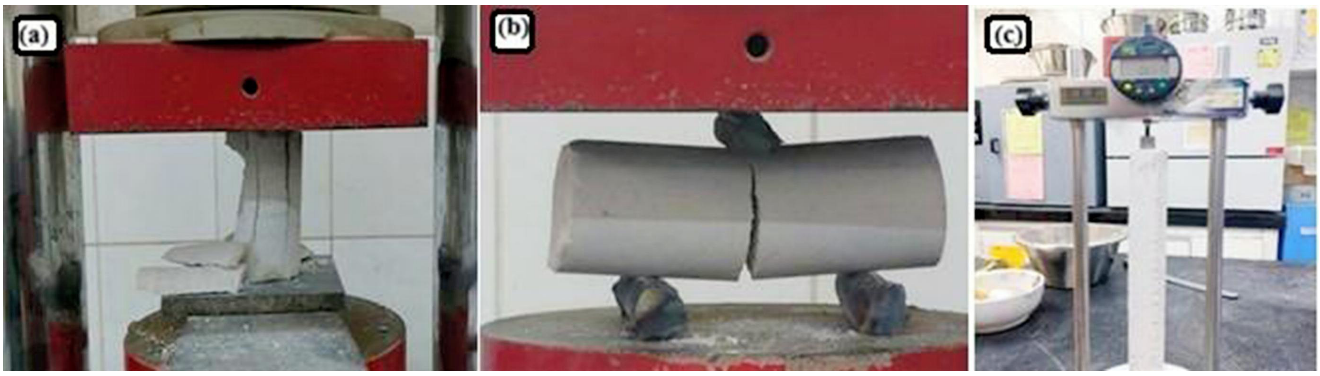


Fig. 7 Explains the mechanical tests: (a) shows compressive strength test; (b) shows flexural strength test; (c) shows shrinkage cracking test

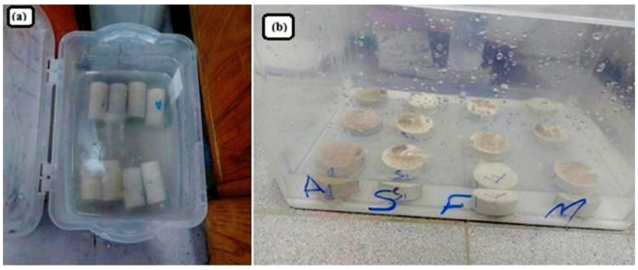


Fig. 8 Sample preparation for aging tests: (a) indicate the immersion of samples in saline solution in preparation for testing after aging; (b) show the prepared samples for testing

While direct measurements of carbonation depth, pore structure, and CNT distribution were not performed, the experimental trends are consistent with established literature. These aspects are acknowledged as limitations and represent opportunities for future research.

4.2 Discussion

This section analyzes the effect of CNT dosage on mortar performance and the interrelationship between the investigated properties. Increasing CNT content reduced compressive and flexural strength, while water absorption and shrinkage showed variable behavior. Low CNT content (0.01%) did not significantly improve strength or shrinkage resistance, suggesting that CNTs alone could not form an effective reinforcing network within the lime matrix [23]. At 0.03% CNTs, no improvement in strength was observed, and shrinkage increased due to non-uniform dispersion and nanotube agglomeration, which create micropores, increase porosity, and reduce mortar density [24]. At 0.3% CNTs, mechanical properties declined markedly because of increased porosity, consistent with previous studies attributing this to poor dispersion and agglomeration, which enhance permeability and reduce carbonation resistance [25].

Previous studies on CNT incorporation with various materials reported both positive and negative outcomes. Negative results are often linked to poor dispersion, causing agglomeration, weak points, and reduced mechanical performance [26]. Although CNTs were treated with concentrated HNO_3 and H_2SO_4 to remove impurities and introduce oxygen-containing functional groups to enhance dispersibility, acid treatment can induce structural defects and leave residual acids or ions, which must be removed to avoid negatively affecting performance in mortar [27]. SEM analysis confirmed that the MWCNTs were free of impurities and residues, indicating successful acid treatment, after which compressive strength was evaluated for the four mixes.

Table 2 Illustrate mechanical and physical properties of lime mortars

Sample	Percentage of MWCNTs	Test	Average of three samples		Final average	
			Before ageing	After ageing	Before ageing	After ageing
A	Standard 0%	Compressive strength (KN)	(1.90), (1.00), (1.28)	(1.40), (0.85), (1.42)	(1.39)	(1.22)
		Flexural strength	(0.72), (0.73), (2.72)	(0.55), (0.57), (0.57)	(1.39)	(0.56)
		P-wave (m/s)	(45.80), (56.50), (38.60)		(63.87)	
		Water absorption (%)	(19%), (23%), (24%)		(22%)	
		Shrinkage cracking (%)	(0.19%), (0.23%), (0.31%)		(0.24%)	
S	0.01%	Compressive strength (KN)	(1.10), (1.16), (1.10)	(1), (1.45), (1.22)	(1.12)	(1.22)
		Flexural strength	(0.49), (0.51), (0.51)	(0.40), (0.41), (2.43)	(0.50)	(1.08)
		P Wave (m/s)	(62.20), (42.90), (49.30)		(63.53)	
		Water absorption (%)	(21%), (21.50%), (21.70%)		(21.4%)	
		Shrinkage cracking (%)	(0.23%), (0.15%), (0.23%)		(0.20%)	
F	0.03%	Compressive strength (KN)	(0.83), (0.90), (1.01)	(1.11), (1.07), (1.16)	(0.91)	(1.11)
		Flexural strength	(0.33), (0.37), (0.41)	(0.41), (0.40), (0.39)	(0.37)	(0.40)
		P-wave (m/s)	(70.1), (59.7), (60.8)		(51.50)	
		Water absorption (%)	(23.20%), (20.90%) ,(23%)		(22.37%)	
		Shrinkage cracking (%)	(0.40%), (0.36%), (0.45%)		(0.40%)	
M	0.3%	Compressive strength (KN)	(0.62), (0.63), (0.63)	(0.49), (0.56), (0.44)	(0.63)	(0.50)
		Flexural strength	(0.30), (0.29), (0.34)	(0.24), (0.25), (0.23)	(0.31)	(0.24)
		P-wave (m/s)	(65.70), (70.10), (55.80)		(46.97)	
		Water absorption (%)	(18.90%), (19.10%), (23.60%)		(20.53%)	
		Shrinkage cracking (%)	(0.20%), (0.28%), (0.44%)		(0.31%)	

Understanding the mechanical and physical behavior of CNT-modified samples requires considering porosity, its distribution, and the effects of carbonation on lime mortar. Porosity is not only a quantitative measure of void volume but also depends on pore shape, size, and connectivity. Connected capillary pores govern water and ion transport, affecting absorption and shrinkage, while closed pores or clusters may not influence absorption directly but reduce stress transfer and ultrasonic wave velocity [28]. Carbonation alters chemical structure, decreases alkalinity, and can affect bond stability and internal porosity. The penetration of CO_2 depends largely on connected porosity and permeability, making the porous structure critical for carbonation depth and rate [29]. CNT addition modifies porosity and thus carbonation: at low concentrations (0.01%) that reduce connected porosity, CNTs can slow CO_2 penetration, block fine pores, improve internal distribution, and reduce permeability [30]. Conversely, at higher CNT concentrations, agglomeration or additional voids may increase connected porosity, facilitating CO_2 penetration, accelerating carbonation, and potentially weakening internal bonds, leading to decreased compressive and flexural strength over time, even if initial results show minimal deterioration [31]. Carbonation alters pore structure by initially dissolving calcium hydroxide (CH), generating new porosity, followed by precipitation of calcium carbonate ($CaCO_3$), which fills some pores and can reduce total porosity. This dynamic balance explains variations in shrinkage, water absorption, and compressive strength [32].

Reduced P-wave velocity at higher CNT content indicates increased internal defects and ineffective porosity, leading to lower compressive and flexural strength. Despite possible lower water absorption, agglomeration and poor dispersion weaken internal cohesion and load-bearing capacity [33].

Studies have shown a clear relationship between P-wave velocity, porosity, and density, where a decrease in wave velocity indicates increased porosity, while higher velocity reflects greater density [34, 35]. Widodo also confirmed that P-wave velocity and porosity are key indicators for estimating compressive and flexural strength, as reduced velocity corresponds to higher porosity and consequently lower mechanical strength [36].

High porosity facilitates CO_2 penetration, accelerating carbonation and increasing its depth, leading to $CaCO_3$ formation that alters pore structure and chemical stability [37]. Additionally, the hydrophobic nature of CNTs hinders proper dispersion, promoting pore formation at higher contents, which enhances CO_2 ingress, resulting in weight loss and reduced mechanical properties [30].

CNTs influence porosity in a dual manner: at low contents, they may seal fine pores and improve internal distribution, reducing permeability, while at high contents, agglomeration creates microcracks and new pathways, increasing effective porosity and affecting shrinkage and water absorption [30]. Carbonation can enhance drying by consuming water, leading to increased shrinkage and cracking over time [38]. It also alters water absorption, as some zones become denser and less permeable, while crack formation in other areas increases permeability, resulting in variable absorption behavior [39].

Water absorption results showed values of 22.00% for the control sample and 21.40% (−2.7%), 22.37% (+1.7%), and 20.53% (−6.7%) for 0.01%, 0.03%, and 0.3% CNTs, respectively, reflecting the influence of CNT dispersion and pore structure [40, 41]. SEM analysis confirmed good dispersion and high purity of MWCNTs; thus, the decrease at 0.01% may be due to partial filling of fine pores, while the increase at 0.03% suggests agglomeration or higher open porosity. The greater reduction at 0.3% indicates possible capillary pore blockage; however, the simultaneous decline in mechanical properties may reflect increased total porosity or microcracks despite reduced connected porosity [42, 43].

Previous studies showed that CNTs can act as bridges to reduce shrinkage cracks and amounts below 0.1% improve internal structure and reduce self-shrinkage compared to the control [29]. CNTs function as nano-fillers, filling pores and slowing water evaporation, but higher additions increase shrinkage due to greater porosity and lower water content [44]. Shrinkage tests revealed that 0.01% CNTs reduced shrinkage from 0.24% to 0.20% (~16.7%), 0.03% increased it to 0.40% (+66.7%), and 0.3% resulted in 0.31% (+29.2%), with variations dependent on dispersion quality and CNT content [29–45].

The results indicate a nonlinear relationship between CNT addition and mortar performance. (Figs. 9 and 10) show that increased porosity reduces compressive strength, flexural strength, and P-wave velocity, while water absorption, shrinkage, and carbonation increase. Over time, increased carbonation further decreases mechanical strengths. Overall, the effect of CNTs on lime mortar properties depends on the added proportion, with porosity and carbonation playing a key role, as connected pores regulate water and CO_2 transport, influencing compressive and flexural strength, shrinkage, and P-wave velocity.

Standard Sample: The results showed that the standard sample maintained its original compressive and flexural strength, water absorption, shrinkage, and P-wave velocity, serving as the baseline for comparison. Carbonation in

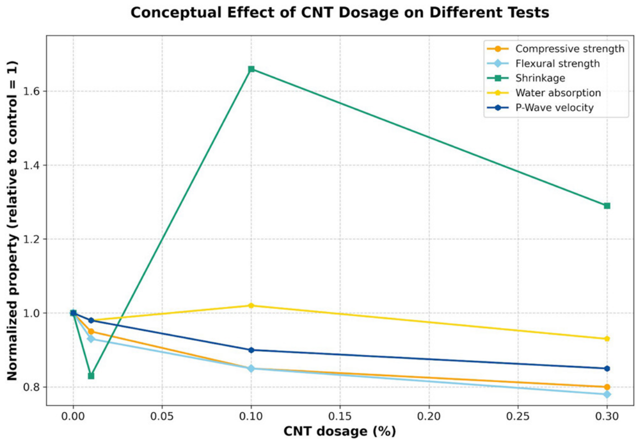


Fig. 9 A conceptual chart showing the relationship between the percentage of added carbon nanotubes (CNTs) and the results of various tests

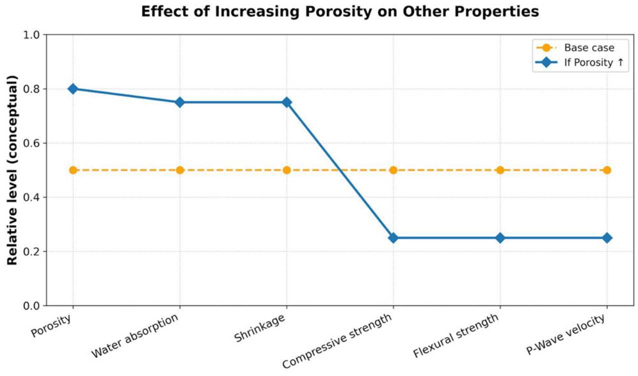


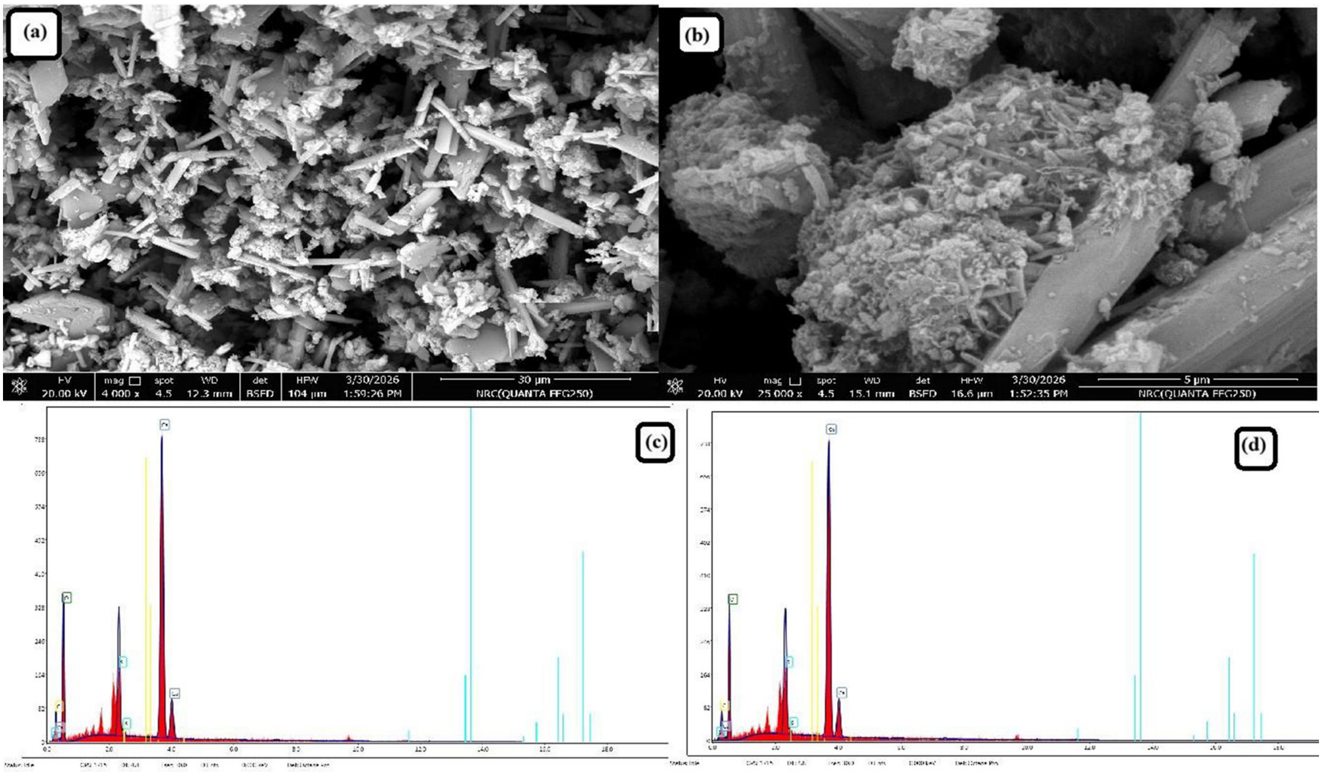
Fig. 10 A conceptual diagram showing how increased porosity affects other tests: Increased porosity is associated with higher water absorption and shrinkage, accompanied by reductions in compressive strength, flexural strength, and P-wave velocity

this sample was governed by its natural porosity, as connected pores controlled CO_2 penetration, influencing the depth of carbonation and internal structure over time.

- 0.01% CNTs: Adding a very low CNT content (0.01%) did not significantly improve compressive or flexural strength, nor notably reduce shrinkage cracks, indicating that nanotube distribution was insufficient to form an effective crack-limiting network. However, water absorption slightly decreased from 22.00% to 21.40% (−2.7%) and shrinkage reduced from 0.24% to 0.20% (−16.7%), likely due to partial filling of fine pores and reduced permeability. The reduction of connected pores at this CNT level may also have slowed CO_2 penetration, limiting structural changes and reaction rates, thereby partially mitigating carbonation effects such as shrinkage and increased dryness.
- 0.03% CNTs: Increasing the CNT content to 0.03% did not improve compressive or flexural strength, while shrinkage significantly increased to 0.40% (+66.7%). This is attributed to non-uniform distribution and agglomeration of nanotubes, creating fine pores that increase porosity and reduce mortar density. Higher connected porosity at this level also facilitates CO_2 penetration, leading to dissolution of calcium hydroxide and deposition of calcium carbonate, which alters internal structure and increases localized drying. This accelerates shrinkage, promotes microcrack formation, and corresponds with the observed decrease in P-wave velocity.
- At the 0.3% CNTs ratio: At a CNT content of 0.3%, a significant decline in compressive and flexural strength was observed due to increased porosity. Water absorption decreased to 20.53% (−6.7%) as some capillary pores were blocked, while shrinkage increased to 0.31% (+29.2%) compared to the reference sample. The reduction in P-wave velocity indicated internal defects and ineffective pores or microcracks. Carbonation was more pronounced at this ratio, as agglomeration and higher pore connectivity facilitated rapid CO_2 penetration, accelerating carbonation. Although calcium carbonate deposition may partially reduce absorption, the resulting shrinkage and drying cracks contributed to the marked decline in mechanical properties.

SEM analysis was conducted on the standard lime mortar sample and the sample containing 0.3% MWCNTs to interpret mechanical performance. The standard sample exhibited a heterogeneous microstructure with numerous needle-like crystalline structures and significant intermolecular voids, indicating a relatively porous matrix. In contrast, the 0.3% MWCNT-modified sample displayed a denser and more cohesive structure, with partially clumped regions. Filamentous carbon nanotubes were not clearly visible in SEM images due to their nanoscale size and embedding within the matrix. Observed agglomeration and uneven distribution of MWCNTs may increase porosity and create localized weak areas, potentially explaining the observed reduction in compressive strength (Fig. 11).

Although effective functionalization and dispersion of MWCNTs were achieved in the aqueous phase, as confirmed by TEM and SEM analyses, such dispersion is not necessarily preserved after incorporation into the cement-based system. The highly alkaline and ion-rich environment of the cement matrix, particularly the presence of Ca^{2+} and OH^- ions, significantly increases the likelihood of re-agglomeration of nanotubes. This phenomenon results from



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
O K	45.78	66.78	31.04	14.10	0.0471	1.0577	0.0974	1.0000
S K	11.31	8.23	154.64	6.56	0.0915	0.9636	0.8112	1.0357
CaK	42.92	24.99	470.82	2.41	0.3782	0.9360	0.9393	1.0027

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	12.97	19.54	20.72	13.57	0.0408	1.0590	0.2967	1.0000
O K	59.54	67.35	132.13	11.14	0.0854	1.0218	0.1404	1.0000
S K	6.16	3.48	194.28	4.43	0.0489	0.9275	0.8325	1.0278
CaK	21.32	9.63	556.15	1.92	0.1902	0.9000	0.9841	1.0067

Fig. 11 SEM micrographs and corresponding EDX spectra: (a) SEM micrographs of the standard sample (magnification=30µm); (b) SEM micrographs of the sample with 0.3% MWCNTs (magnification=5µm); (c) EDX spectrum of the standard sample; (d) EDX spectrum of the sample with 0.3% MWCNTs.

the screening of repulsive forces between particles, ultimately leading to the formation of localized agglomerates.

These agglomerates act as microstructural defects rather than reinforcing elements. Instead of contributing to crack-bridging or pore-filling mechanisms, poorly dispersed MWCNT clusters create weak zones that facilitate the initiation and propagation of microcracks under compressive loading, thereby reducing the overall mechanical performance.

In addition, the high specific surface area of MWCNTs leads to increased water demand, which adversely affects the workability of the mortar. Reduced workability can result in inadequate compaction and an increase in entrapped porosity, both of which contribute to the observed decline in compressive strength.

The absence of clearly identifiable MWCNTs in SEM micrographs of the hardened mortar can be attributed to several factors, including the low dosage of nanotubes, their encapsulation within hydration products (such as C–S–H gel), and the inherent limitations of SEM resolution in detecting nanoscale features within a dense cementitious matrix. Therefore, complementary techniques such as EDS mapping or high-resolution microscopy may be required to confirm their presence and distribution.

Based on these findings, it can be concluded that the negative impact of MWCNT addition observed in this study is not an inherent limitation of the nanomaterial itself, but rather a consequence of inadequate dispersion stability within the cementitious environment. This highlights the critical importance of optimizing both the dosage and dispersion strategy—such as the use of polycarboxylate-based superplasticizers—to fully exploit the reinforcing potential of carbon nanotubes.

Pivák et al. [46] reported that incorporating CNTs up to 0.5% in lime (CL) and hydraulic lime (HL) mortars significantly improved mechanical and structural properties. In CL mortar, compressive strength increased by over 30% at 0.3% CNTs, whereas HL mortar showed ~65% increase in compressive strength and ~19% in flexural strength. Microstructural analysis revealed that total porosity slightly increased (~2%) in CL, while open porosity decreased in

HL, indicating improved internal cohesion and reduced large voids. Some CL samples showed lower water absorption, and HL samples exhibited enhanced drying rates, which is relevant for restoration applications. The study emphasized high-quality CNT dispersion using sonication, surfactant, and defoamer to ensure optimal nanoscale distribution. Despite these improvements, the study did not address CNT performance under real and variable moisture conditions, such as wetting–drying cycles or salt exposure, which remain important for future research.

1. The effect of CNTs on mortar under real and variable moisture conditions, such as repeated wetting–drying cycles or salt exposure, was not evaluated, which is crucial for heritage building restoration.
2. Mechanical properties were measured only after a short period (28 days), without assessing the long-term stability of CNTs, changes in porosity, or water absorption.
3. Detailed microstructural analysis using techniques such as SEM–EDS was not performed, limiting understanding of CNT distribution and its relationship with pores and calcium crystals.
4. The study focused on relatively high CNT concentrations (0.1–0.5%) and did not investigate very low dosages (0.01% or 0.03%), which may be more cost-effective, reduce agglomeration, and improve dispersion.
5. The interaction of CNTs with mineral impurities or natural additives in the mortar was not addressed, although real lime mortars often contain silica, clay, or other impurities affecting CNT performance.
6. No mathematical or analytical models were provided to explain mechanical and porosity improvements; the study was limited to experimental observations.

These gaps provide opportunities for complementary research, such as the current study, which focused on very low CNT dosages (0.01%, 0.03%, 0.3%) and achieved complete and homogeneous dispersion using mechanical mixing and sonication. Porosity was assessed via the Widodo method using ultrasonic pulse velocity (UPV), and water absorption and saturation were measured. The effect of CNTs on carbonation was analyzed, showing that nanotubes promote more uniform $CaCO_3$ formation, enhancing long-term stability and mechanical properties. All mechanical properties, including compressive strength, flexural strength, and shrinkage, were evaluated and directly linked to CNT content. Precise curves were plotted to show the effect of different dosages on mechanical, water-related, and microstructural performance, allowing the optimal dosage to be identified.

This study demonstrates how complete CNT dispersion, appropriate dosage, reduced porosity, decreased water absorption, and enhanced carbonation collectively improve the overall performance of lime mortar.

5 Conclusions

This study investigates the effect of adding multi-walled carbon nanotubes (MWCNTs) to lime mortar on its mechanical and physical properties, including compressive strength, flexural strength, longitudinal wave (P-wave) velocity, water absorption, and shrinkage behavior. The results showed that the effect of the nanotubes depends significantly on their concentration and the quality of their distribution within the mortar, and that the relationship between the addition ratio and performance is not linear.

It appears that porosity is the controlling factor that links all measured properties, while carbonation plays an additional role by modifying the pore structure. At low concentrations, CNTs may contribute to the closure of some fine pores and delay carbonation, whereas higher concentrations enhance pore connectivity and the formation of microcracks, facilitating carbon dioxide penetration and accelerating carbonation, thereby weakening the mechanical properties over time.

At low concentration (0.01%), a slight improvement in shrinkage resistance and a decrease in water absorption were observed, which can be attributed to a partial improvement in capillary pores and a reduction in their connectivity compared to higher concentrations. However, this improvement did not reflect on compressive strength or flexural strength, which remained close to the control sample. At a concentration of 0.03%, the addition of tubes led to increased shrinkage and water absorption, likely due to the formation of interconnected pores that increased permeability. At the higher concentration (0.3%), the mechanical properties clearly deteriorated, with decreases in compressive and flexural strength as well as P-wave velocity, reflecting the negative effect of poor distribution and increased agglomeration, which raised total porosity and weakened the internal structure.

In general, the incorporation of carbon nanotubes into lime mortar does not necessarily guarantee improved performance; it largely depends on the concentration, distribution quality, and resulting pore structure. While small amounts may help reduce shrinkage and permeability, high amounts can lead to agglomeration, increased porosity, accelerated carbonation, and thus deterioration of mechanical properties. These results emphasize the need to carefully control the concentration of nanotubes and their distribution methods to achieve reliable improvements in lime-based composites.

6 Future recommendations

Based on the results of this study, it is recommended that future research focuses on:

1. Improving dispersion methods: Using surfactant additives or advanced mixing techniques (such as chemical dispersants) to ensure a homogeneous distribution of carbon nanotubes and prevent their agglomeration before mixing.
2. Enhancing dispersion within the mortar using a Superplasticizer, which is a chemical additive to the mortar that increases fluidity without adding extra water. It is also known as: High Range Water Reducer (HRWR).
3. Microscopic modeling: Using numerical simulation techniques (such as molecular dynamics or finite element methods) to link the effect of nanotubes on porosity with mechanical properties more precisely.

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