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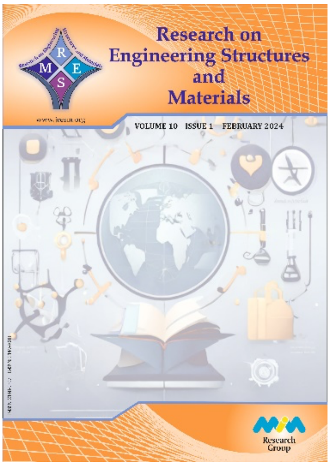

The effect of multi-walled carbon nanotubes on mechanical properties and water adsorption of lightweight foamed concrete

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Research Article

The effect of multi-walled carbon nanotubes on mechanical properties and water adsorption of lightweight foamed concrete

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Abstract

The paper investigates the impact of carbon nanotube-based modifiers on the performance characteristics of lightweight foamed concrete (LFC). The method involves saturating quartz sand with a solution containing a catalyst for carbon nanotube (CNT) growth, followed by the subsequent chemical vapor deposition (CVD) synthesis of CNTs. Evaluation of nanomodified sand samples was conducted using SEM and TEM, thermogravimetry, Raman spectroscopy, and XRD. Compression and flexural strength tests of (LFC) specimens indicated that the optimal proportion of nanomodified sand introduced is 1% by weight with a particle size of 0.16 mm. This resulted in a notable 35% increase in compressive strength and an approximately 32% improved in flexural strength. Furthermore, the modified sample with CNT-based sand exhibited a 27% reduction in water absorption. The paper also presents a potential mechanism to explain the impact of carbon nanotube-based modified sand on the evolving structure of (LFC).

1. Introduction

Cementitious materials have emerged as the predominant choice for structural applications in civil engineering, primarily attributed to their economical pricing and consistent operational effectiveness. Globally, Portland cement has garnered substantial utilization as one of the most prevalent commodities. [1-4].

The key characteristics of a structural material encompass its strength, rigidity, resistance to fractures, energy absorption potential, and ductility. Nevertheless, enhancing all these attributes poses a challenge when it comes to traditional cement-based materials [5-8]. These materials are associated with certain established drawbacks, such as relatively limited tensile strength and suboptimal viscosity. Concrete is prone to the formation of cracks, providing pathways for the infiltration of water and corrosive substances like de-icing salts. This, in turn, initiates the deterioration of both the material and the commonly employed reinforcing steel, giving rise to various concerns regarding their long-term resilience and safety [9,10]. The addition of nanoscale particles can improve the properties of concrete mainly because of the effect of increased surface area on reactivity and because the cement paste fills the nanopores. Nano-silica and nano titanium dioxide are two of the most well-known additives in the field of nanomodified concrete [11]. Moreover,

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nanomaterials may improve the ductility and compressive strength of concrete. Concrete's strength and ductility can also be improved by using carbon nanotubes (CNTs) and nanofibers. Furthermore, the use of nanomodified cements or nano-sized paste additives can improve shrinkage characteristics and reduce permeability, both of which can increase the strength of concrete [12–15]. Concrete's long-term and early shrinkage could be effectively reduced with the addition of CNTs, that can be attributed to the filler, nucleation, and bridging properties of CNTs. One way that CNTs reduce capillary stresses is through their filler and nucleation effect, which reduces the amount of fine pores between hydration products because of their small diameters (20–40 nm) and high surface area [16,17].

The utilization of nanomaterials within cement, aimed at enhancing its mechanical properties, stands as a highly promising research frontier. Numerous investigations have delved into the integration of aluminum and clay in nano size [18-21]. When a small quantity of nanoparticles is uniformly distributed throughout a cement paste, hydrated cement compounds precipitate onto the nanoparticles due to their elevated surface energy. Consequently, these nanoparticles serve as nucleation sites, expediting the growth of the cement structure. C3S dissolution and the details of the C-S-H in cement paste have been observed to occur more quickly when colloidal nano silica is added a) Nanoparticle filling of the concrete pores and (b) the pozzolanic reaction of nano-SiO2 with Ca(OH)2 yielding additional C-S-H are two more mechanisms for improving performance. Particle size and the degree to which nanoparticles are effectively dispersed within the cement determine which method is best; colloidal dispersions work better than powdered versions [22,23].

In the research conducted by Sedaghatdoost et al. [24], it was documented that the incorporation of 0.1 wt. % the cement slurry that contain carbon nanotubes (CNT) maybe led to improve the flexural and compressive strength, with increases of up to 35%, 8%, and 11.2%, respectively, observed after a 28-day curing period. It is possible to explain this phenomenon by the nucleation effects of CNTs, which speed up hydration reactions. Hydrated products attach themselves to the carbon nanotubes (CNTs) and create a cohesive layer that covers the whole surface. Its filling qualities also enable CNTs to lower the average diameter and total volume of pores in cementing materials [25-27].

However, their limited dispersibility presents a challenge when using carbon nanotubes (CNTs) as nano-reinforcing agents in cementitious materials. When using nanofillers, such as carbon nanotubes (CNTs), in cement systems, the best reinforcement happens when the nanofillers are distributed uniformly and evenly. Isfahani et al.'s research indicates that a number of factors contribute to poor dispersion, including CNTs' large surface area and nano diameter as well as their propensity to form tightly bound bundles as a result of strong van der Waals forces. Furthermore, highly entangled agglomerates form within the liquid phase due to the high form factor and flexibility of CNTs [28-29]. Similarly, the existing literature underscores the recurrent issue of CNT agglomeration, which hampers their uniform distribution in the cement slurry. This lack of even distribution limits the CNTs' capacity to create an effective and continuous network within the substrate, one that can adequately support load transfer and mitigate crack formation [30-32].

In addition, the agglomeration of CNTs contributes to the creation of stress concentration points, resulting in the emergence of cracks [33]. Thus, achieving a consistent dispersion of CNTs is imperative, serving as a prerequisite for the effective utilization of CNTs in binding materials [34]. It can be confidently asserted that the enhancement of mechanical properties in the ultimate composite is closely linked to the uniform and effective dispersion of CNTs, allowing them to serve as connecting bridges between voids and cracks within porous cement-based structures.

To achieve this goal, the processes of introducing CNTs into cement-based materials (paste, mortar, and concrete) have to be complicated, in particular, by the obligatory use of additives or surfactants, as well as by preliminary chemical functionalization of CNTs [35-38].

In this paper, the authors investigated the properties of foamed concrete. Foamed concrete is a type of concrete that has a set of some useful properties (special porous structure, excellent anti-seismic, non-combustible and thermal insulation characteristics), making it more and more popular in construction. It is used as an effective structural and insulating material for roofing, non-bearing walls and thermal insulation of heat pipes [39, 40]. The use of foamed concrete in buildings provides a reduction in construction costs, makes the construction process simpler. In addition, foamed concrete is a relatively "environmentally friendly" building material [41].

This study proposes an innovative method for effectively integrating carbon nanotubes (CNTs) into cement matrices without the need for additives, surfactants, or intricate chemical procedures. The approach involves pre-growing CNTs on sand particles before introducing them into the solution. The synthesis of CNTs on sand grains utilizes the (CVD) method, involving impregnating the sand with a catalyst for nanotube growth and employing a propane-butane mixture as a carbon source. This method offers a significant advantage by enabling the industrial production of nanomodified cement materials with enhanced physical, mechanical, and operational characteristics. Importantly, it eliminates the need for labor-intensive and costly stages of chemical functionalization of CNTs, as well as the use of surfactants and other functional additives.

2. Materials and Methods

2.1. Growing CNTs on the surface of sand particles

To create a layer of (CNTs) on the sand, an aqueous catalyst solution was prepared using precursors such as (NH4)6Mo7O24•4H2O, Al(NO3)3•9H2O, Co(NO3)2•6H2O, and Mg(NO3)2•6H2O. Citric acid (C6H8O7) served as a complexing agent. The resulting solution was continuously agitated using a magnetic stirrer at 60°C until complete dissolution of the initial components. Subsequently, quartz sand was introduced into the catalyst, and the impregnation process was carried out at 140°C within a fume hood. This step resulted in the formation of a spatial fractal gel structure containing chelate complexes with active catalyst metals, including Mo, Co, Mg, and Al, on the surface of the sand particles [42].

The sand have catalyst was subsequently heat-treated in an inert argon atmosphere within a CNT synthesis reactor at a temp of 500°C for a duration of one hour (conducted by NanoTechCenter Ltd., Tambov, Russia). This thermal treatment resulted in the creation of active sites for the growth of carbon nanotubes (CNTs) on the sand, particularly involving metal oxides like Mo, Co, Mg, and Al. After this stage, a carbon-containing gas, specifically a mixture of propane and butane, was introduced into the reactor, and the temp was raised to 600°C. The synthesis process lasted approximately 30 minutes. Once the CNT synthesis was completed, the modified sand was subjected to crushing and subsequent sieving.

2.2 Foamed Concrete Materials

The lightweight concrete was made with Portland cement type I (Bazyan, Iraq), ASTM C150, and gradient quartz sand (Dwekhla, Alanbar, Iraq) from zone II. The sand had a specific gravity of 2.7 (Table 1). The sieve analysis shown in table 1 conforms to Iraqi Standard 45/1984. The foaming agent under the trademark MAXPN (Russia) was added.

Table 1. Granulometric composition of sand.

Partial residues on sieves, %						Specific gravity
2.5	1.25	0.63	0.315	0.16	<0.16	
15	16	16	30	21	2	2.73

2.3. Preparing Samples of Lightweight Foamed Concrete

For mixing, tap water was utilized, with a (water/cement) ratio of 0.5. The modified sand was employed with a sand weight ratio of (0.1, 0.2, 0.5, and 1%). After sieving the sand, three different particle sizes (0.63, 0.315, and 0.16 mm) were chosen. Then the mortar was prepared by adding water. After that, the lightweight-foamed concrete was created by adding the foam to the mixture—1.6 kg per m3. A prism mold was cast in the concrete mixture (40 x 40 x 160 mm). Samples were removed after 24 hours from the mold and water immersion to cure at 23±2°C according to ASTM C31. The compressive and flexural strengths were calculated according to Russian Standards 310.4-76 after 28 days.

The testing machine was single axle with a capacity of 2000 kN and an applied load of 0.4 MPa/s. The density was (1600–1750 Kg/m3), and the combination was calculated by volume ratios. The mix ratios are shown in Table 2.

Table 2. Preparation of the concrete mixture

Mix Proportion	W/C	Cement, Kg/ m^3	Sand, Kg/m^3	Water, Kg/ m^3	Voids %
(1:1)	0.5	650	650	280	25-30

2.4. Material Characterization

Using scanning electron microscopy (SEM) with an Inspect S50 FEI ranging from 1 mm to 500 µm, the morphological and structural properties of the nanomodified sand were investigated. Additionally, the surface of foamed concrete specimens was examined using SEM analysis. (Thermo Fisher Scientific, Czech Republic), and transmission electron microscopy (TEM) using an FEI Tecnai Spirit M3000 (Zurich, Switzerland). A Bruker D8 ADVANCE X-ray diffractometer (Bruker, Germany) was used to determine the qualitative and quantitative phase composition of the nanomodified sand. Raman spectra were obtained on a DXR™ Raman microscope (Thermo Scientific Instruments Group, Waltham, MA, USA).

3. Results and Discussion

3.1. Assessing the Effect of CNTs Synthesis Time on The Strength Characteristics of Lightweight Concrete

To assess the effect of CNTs synthesis time on the sand surface, an experiment was carried out in which 1% of nanomodified sand with a fraction of 0.16 mm was added to the composition of the foamed concrete mixture. During the experimental investigations, the duration of carbon nanotube (CNT) growth was altered, with durations of 5, 15, 30, and finally 60 minutes being tested. The outcomes of these experiments are illustrated in Figures 1 and 2.

As indicated by Figures 1 and 2, the optimum synthesis time appears to be 30 minutes, during which a consistent and uniform structure of carbon nanotubes (CNTs) is established on the surface of the sand particles. Prolonging the synthesis beyond this point is a waste of time and may lead to the catalyst used for CNT growth becoming deactivated. Consequently, an extended synthesis period can result in a reduction in the proportion of well-ordered CNTs, an increase in structural defects, and the formation of a notable quantity of disordered carbon.

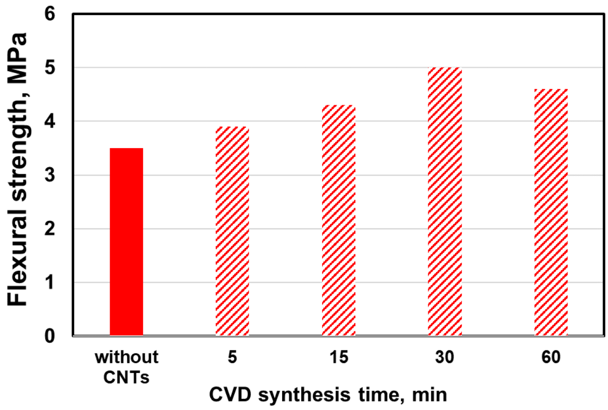


Fig. 1. Effect of CNTs synthesis time on sand surface on flexural strength

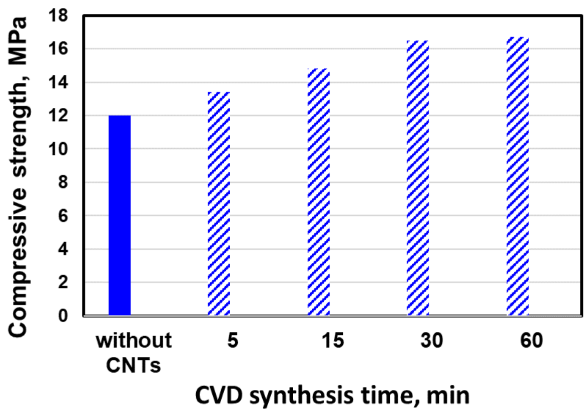


Fig. 2. Effect of CNTs synthesis time on sand surface on compressive strength

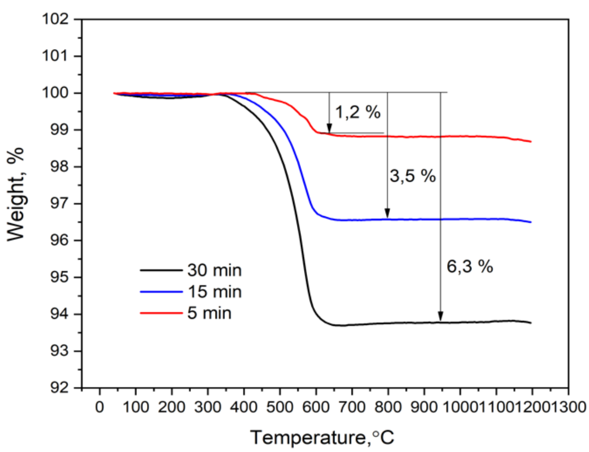


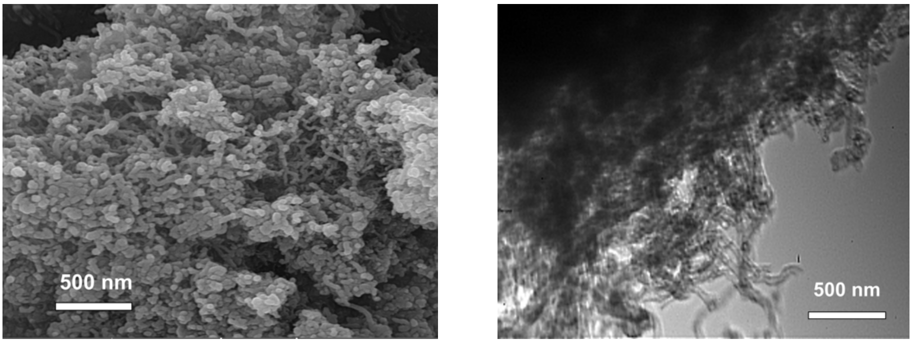
Fig. 3. TGA of nanomodified sand samples with different CNTs synthesis times

Additionally, it's worth noting the consistent progression in the strength detiles of foamed concrete containing modified sand. This can be attributed to the growing concentration of CNTs on the surface of sand over time, a phenomenon substantiated by thermogravimetric analysis, as depicted in Figure 3.

Considering to Figure 3, all the curves exhibit a notable decline in sample mass starting at 400°C. Specifically, at 600°C, it was observed that for a 5-minute synthesis, there was a weight loss of 1.2%, while for 15 minutes, the loss was 3.5%, and for 30 minutes, it increased to 6.3%. These weight loss percentages can be attributed to the CNTs grown on the surface of sand, as it is at 600°C that CNT destructurization occurs. Consequently, the 30-minute synthesis yielded the most substantial increase in CNT mass.

3.2 Characterization of Nanomodified Sand Samples

According to the SEM and TEM, the sand surface is covered with a layer of CNTs (Fig. 4, a). The nanotubes have an average diameter of approximately 30-50 nm, and the material also includes catalyst particles with diameters ranging from 30 to 70 nm. It can be seen in the TEM-image (Fig. 4, b) that the CNTs have an internal channel with a diameter of 10–20 nm.



(a) (b)

Fig. 4. SEM- (a) and TEM-image (b) of the nanomodified sand

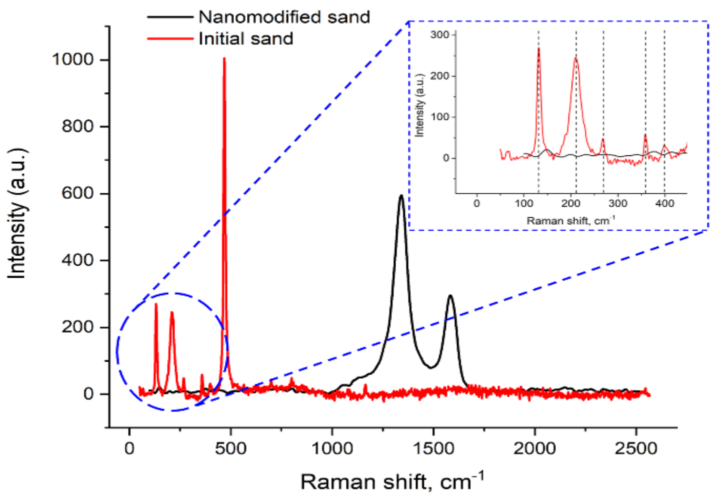


Fig. 5. Raman spectra of nanomodified sand filler and original sand

The Raman spectroscopy is considered to be an effective tool for the characterization of carbon nanomaterials because it provides information, in particular, on the degree of order and on the purity of the material. The existence of carbon forms in the material can be tested from the characteristic bands in the spectrum (Fig. 5).

D-band (approximately 1340 cm-1) indicates the presence of structural defects in the sample. G-band (approximately 1550–1600 cm-1) informs about the degree of ordering of the graphene structure in the material. The third visible 2D signal (defined as the mode D overtone) is visible at about 2700 cm-1 [43].

As can be seen from Fig. 5, the spectral pattern of the original and nanomodified sand is significantly different. The original sand has high intensity peaks at 130, 210, 467 cm-1, as well as weak peaks at 270, 360, 400 cm-1. The resulting spectrum is characteristic of the β-SiO2 crystal lattice [44]. The following characteristic lines were identified in the nanomodified sample: D ~ 1345 cm-1 band, G ~ 1571 cm-1 band, and 2D ~ 2694 cm-1 band. This observation verifies the existence of imperfect CNTs on the sand, since the D band is much more intense than the G band. The diffraction pattern of the original sand (Fig. 6) contains quartz (β-SiO2) as the main mineral [45, 46].

The XRD pattern (Fig. 3) shows several bands with peaks (0 0 2), (1 0 0), and (1 1 0), which correspond to carbon structures [43]. However, only the (0 0 2) reflection has a high intensity, while the other peaks are relatively small. The peak (0 0 2) is located at 2 θ = 26°. Reflection (1 0 0) at 2 θ = 42° corresponds to the CNT interlayer distance. The remaining peaks are attributed to quartz.

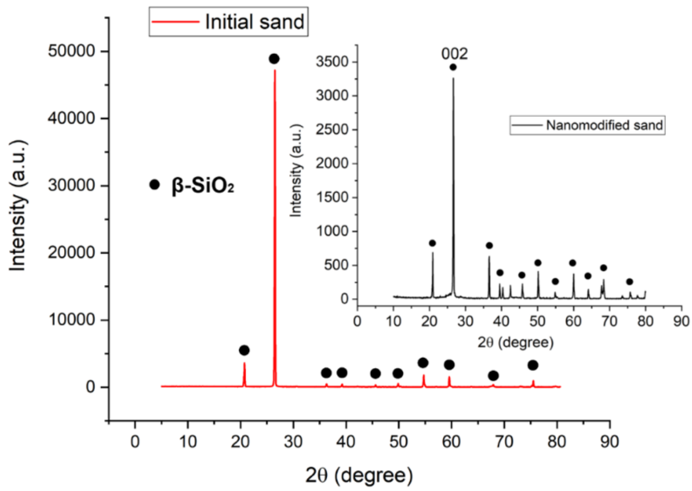


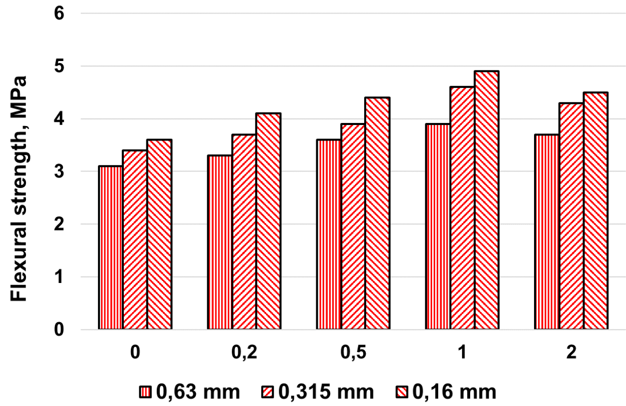
Fig. 6. X-ray diffraction pattern of nanomodified CNT sand

3.3. Assessing The Effect of The Percentage of Introduced Nanomodified Sand and Its Fraction on Strength Characteristics and Water Absorption

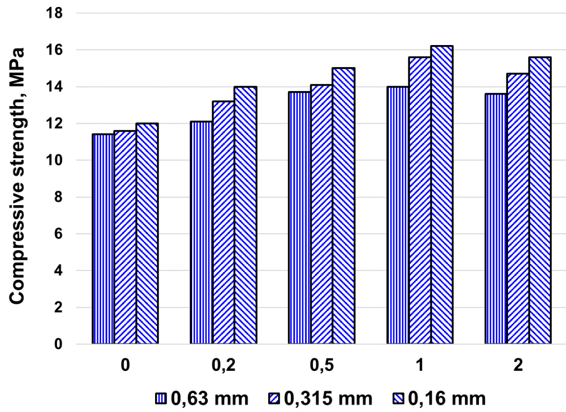
In this paper, the introduction of nanomodified sand additives of three fractions of 0.16, 0.315 and 0.63 mm was studied. The percentage of introduced nanomodified sand was: 0.2; 0.5; 1; 2 wt. %. The results are shown in Figs. 7, 8.

The maximum increase in compressive strength (~ 35%) is observed when adding 1% wt. nanomodified sand filler with a size of 0.16 mm, an increase in flexural strength of ~ 32%.

It is assumed that the smaller the size of the sand particles, the greater the specific surface area they have, which will allow the synthesis of large quantities of CNTs. In addition, with the same weight of different fractions, the 0.16 mm fraction will contain a larger number of particles, therefore, the contact area of CNTs with cements will be increased.



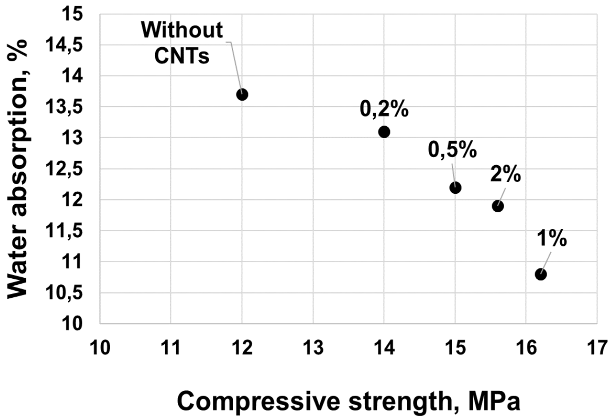
(a)



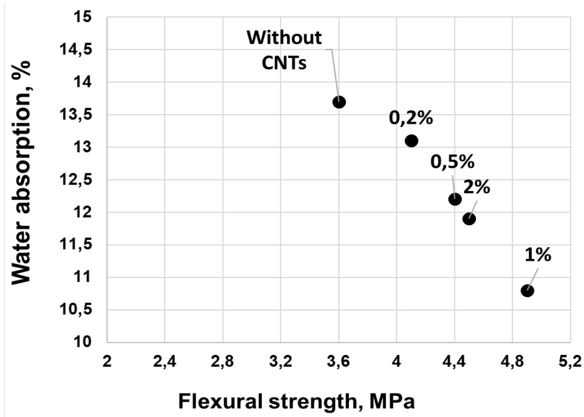
(b)

Fig. 7. The flexural (a) and compressive (b) strength test

Thus, it was found that when making 1% wt. nanomodified sand filler with a size of 0.16 mm, the minimum value of water absorption is achieved (27% less than the control sample without CNTs). The reduction in the percentage of water absorption may be due to the hydrophobic nature of the CNT surface. Thus, by adding CNTs to the cement mixture, the interpore walls of the concrete are compacted and, as a consequence, the access of water to the volume of concrete is reduced. During the hydration of Portland cement, a number of chemical transformations occur, due to which needle-shaped crystals appear on the surface of cement grains when interacting with water. In a short time, the number of these neoplasms increases, and a so-called "reinforcing" gel is formed, which forms a developed spatial network. Then, after 8-10 hours, the gelation process continues and the entire volume is filled with calcium hydro silicates and their modifications, depending on the composition of the cement composition. The remaining voids are filled with clinker minerals. After the hydration process is completed, the cement stone hardens and becomes durable. Considering the above, in order to influence the chemical transformations during the hydration of cement, it is necessary to introduce additional chemically active reagents into the cement mixture. In this work, the authors suggest that CNTs synthesized on the surface of sand, in addition to accelerating the hydration process due to the active functional surface - the CNT shell creates an increased surface area of the sand. Therefore, nanomodified sand creates additional centers for crystal formation, thereby intensifying and qualitatively improving the cement hardening process.



(a)



(b)

Fig. 8. The effect of the percentage of introduced nanomodified sand on the water absorption of lightweight concrete

3.4. Substantiation of the Effect of Improving the Mechanical and Operational Characteristics of Concrete

Using carbon nanotubes to modify different types of concrete materials offers significant advantages over using unmodified concrete; they have a positive effect on the strength properties of samples, Improves the strength of concrete by reducing crack growth and propagation, thus improving the strength properties of concrete [47].

According to the literature review, carbon nanotubes act as crystallization centers for calcium silicate in hardened foam concrete and stimulate the structure formation of hardened calcium silicate binders with high crystallinity, observed in the pore walls compared to conventional control samples Compared to traditional control samples [48,49].

There are multiple interaction mechanisms between CNTs and cement mortar, the most important of which is the crack filling provided by carbon nanotubes [50]. The microstructure of concrete samples can change in the presence of carbon nanotubes for hydration products. The area adjacent to the surface of the CNTs (which affects the properties of the composite) is affected by the kind of the interaction of the CNTs with the cement (e.g., chemical bonds between surfaces, van der Waals forces). , and interwoven).

These mechanisms also count on the chemical bonds between CNTs and hydrates [51]. The study also found that introducing carbon nanotubes into cementitious materials through nanoindentation will lead to the make of a large amount of high-hardness C-S-H in the composite. [52, 53]. The hydration products formed near the CNT surface fill the free space between the particles in the pore structure of the cement stone, while the pores in the sample without the nano modifier remain empty. As a result, the total porosity decreases and the concrete density increases, thus increasing the mechanical properties of the sample [53].

However, in order to improve the strength and technical properties of concretes, it is necessary to obtain a uniform dispersion of CNTs so that they act as effective structure formers and hydration accelerators, as well as connecting bridges between the boundaries of cracks and voids in a porous cement-based matrix. The development of new methods and equipment to control the problems of agglomeration and uneven distribution of CNTs in the structure of a concrete sample is very relevant. The synthesis of CNTs directly on the surface of the sand aggregate using the CVD method to create concrete compositions with improved properties contributes to the solution of the above problems when using CNTs in a concrete mixture. Figure 9 shows a possible mechanism of effect of the developed additive (nano-modified sand) on the structure of lightweight concrete.

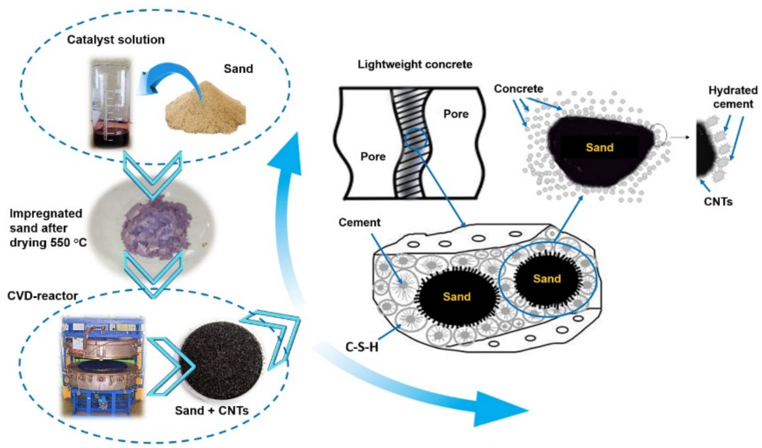


Fig. 9. Illustration of the effect of modified sand on the structure of a foamed concrete sample

The mechanism illustrated in Figure 9 provides insights into the enhancement of the physical and mechanical properties of the samples. This improvement is a consequence of the expedited hydration processes induced by the presence of carbon nanotubes (CNTs), leading to the formation of additional crystallization sites and an increased volume of new structures, such as C-S-H, tobermorite, and gel phases. These newly formed structures occupy the intergranular spaces between the larger calcium hydroxide (CH) and ettringite grains.

The overall increase in strength can be attributed to the creation of a pseudo-framework formed by nanomodified sand particles encased in a shell of hydrated cement grains. This pseudo-framework enhances the regularity and packing density at the 'cement stone-aggregate' interface, resulting in a reduction in the size of the newly formed structures in this area. Consequently, this leads to a decrease in the number of sized pores. Furthermore, a denser cement stone structure results in reduced water absorption, which, in turn, mitigates deformation caused by water crystallization at low temperatures. This improvement augments the frost resistance of the final construction products made from modified foamed concrete.

3.5. Comparative Analysis Of Strength Tests of Carbon Nanotube Concrete Compositions

As a result of the literature review, no studies were found aimed at the synthesis and use of nanomodified sand to improve the properties of foamed concrete. However, the results of this study were compared with publications on the use of CNTs as foamed concrete Nano modifiers. The results are shown in Table. 3.

Table 3. Comparative results of mechanical testing of foamed concrete containing CNTs for compressive strength.

No.	Concrete composition	Compressive strength, MPa	CNTs concentration, %	Reference
1.	CNTs + with lightweight filler based on silica aerogel	5.4	0	[54]
		6.1	0.04	
		6.4	0.1	
		6.7	0.15	
		7.2	0.30	
		7.4	0.45	
		7.6	0.60	
2.	Foamed concrete + CNTs	19.77	0.08	[55]
3.	Foamed concrete + CNTs + nano silica (NS)	7.51	0	[56]
		9.23	3% NS	
		8.58	0.5% CNTs + 2.5% NS	
		8.64	1% CNTs + 2% NS	
		7.71	1.5% CNTs + 1.5% NS	
		10.42	2% CNTs +1% NS	
		9.22	2.5% CNTs + 0.5% NS	
		6.32	3% CNTs	
4.	Foamed concrete + CNTs + recycled polystyrene	7	0	[57]
		9	0.05	
		10	0.1	
5.	Foamed concrete + CNTs + superplasticizer	5.4	0	[58]
		5.91	0.02	
		5.53	0.04	
		5.52	0.06	
6.	Foamed concrete + nanomodified sand	16.2	0.006	This paper

Thus, it can be argued that, in comparison with the results of publications of international research teams in the field of modification of CNT foamed concrete, samples containing nanomodified sand in their composition have a significant advantage in mechanical characteristics. At the same time, the percentage of introduced CNTs is much less than in the compared works. All the above proves the relevance, a promising outlook of the study goal and objectives, and its undoubted scientific novelty.

4. Conclusion

The study's experimental results pointed to the following:

- The using of CNTs are intentionally formed on the surface of quartz sand particles used as the filler in lightweight foamed concrete, to improve the qualities of the material. Complex diagnostics were performed on samples of nanomodified sand utilizing a variety of analytical techniques. It was established that a uniform CNT layer was created on the sand surface. Based on the sand filler's particle size and the mass % of the nano modifying additive, a thorough analysis of the performance characteristics of foamed concrete mixtures of various compositions was conducted.
- Adding 1% weight of nanomodified sand filler with a fraction of 0.16 mm has been found to yield the highest increases in sample strength, with 35% and 32% increases in compression and flexural strength, respectively.
- The water absorption in the modified sample is 27% lower than that of the control sample when sand modified with carbon nanotubes is added.
- A possible mechanism for how the developed additive (nanomodified sand) affects the foamed concrete's composition is suggested by the study. Accelerated hydration processes, the appearance of additional crystallization centers in the presence of CNTs, and an increase in the volume of neoplasms like C-S-H, tobermorite, and gel phases that fill the intergranular spaces are all described as improving the samples' mechanical, physical, and operational properties.

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