Systematic investigation into mechanical strength, pore structure and microstructure of high performance concrete incorporating nano-hybrids

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Abstract

In this study, high performance concrete (HPC) containing two types of nanoparticles: carbon nano tubes (CNTs) and nano silica (NS) and hybrid nanoparticles (CNTs + NS) were prepared. The compressive strength, phase composition, pore size distribution, surface area, surface morphology and microstructural characteristics were investigated at 7 and 28 days of hydration. In addition, the combined effect of CNTs and silica fume (SF) on the properties of concrete has been investigated in this study for comparison reasons. The results indicated that, all kinds of the utilized nanoparticles provided an enhancement in the compressive strength of the hardened concrete at early and later ages of hydration. Furthermore, Thermogravimetric analysis (TGA) patterns revealed no great difference in the hydration degree of cement with the addition of CNTs. However, Due to its large specific surface area and the pozzolanic activity, CNTs+NS nanohybrid showed the best acceleration efficiency. The concrete containing CNTs+NS nanohybrid exhibited the highest improvement in compressive strength. The pozzolanic reaction induced by NS and silica fume (SF) led to remarkable increase in the surface area and the nanoporosity/gel pores assigned to calcium silicate hydrate (CSH) phase. The SEM analysis showed that, CNTs resulting in bridging nano-size and micro-size pores or gaps within cement hydrates; however, SF and NS are more efficient in filling the pores and gaps between the hydration products and providing more compact microstructure.

Keywords: High performance concrete; Nano-hybrid; Compressive strength; Pore structure; Surface area; Microstructure; TGA
1. Introduction

The production of high-performance concrete can be considered as one of the most important applications of nano materials in construction industry [1]. Nano materials can modify the nano and micro structure of concrete and leads to enhanced mechanical strength at normal curing temperature. Beside the leading role of nano materials in enhancing the mechanical performance, the nano materials considerably improve volume stability, durability, and sustainability of cement and concrete structures [2]. Application of nano technology in concrete has been implemented by using a wide range of nano materials with various chemical and physical characteristics including: Spherical nano particles (like; nano silica, nano alumina, calcium carbonate nano particles, etc.); nanotubes (like; CNTs) or as nano platelets (like; nano clay and nano graphene) [3]. The role of nano pozzolan like NS in improving concrete performance differs from the role of nano fibers like CNTs. NS possesses chemical and physical effects: (1) pozzolanic reaction; i.e., it reacts with the calcium hydroxide which liberates upon hydration of cement and produces supplemental calcium silicate hydrate which contributes to strength development; (2) Due to its ultra-fineness, NS will realize significant reduction in the amount of micro pores by inducing physical packing, providing a more dense concrete [4,5]. CNTs don’t have any chemical affinity with the cement hydrates [6] thereby CNTs are used to reinforce cementitious matrix and attain resistance to micro-crack propagation [7-10]. The effect of NS with mean particle size of 120 nm on the hydration and microstructure characteristics of ultra-high performance concrete (UHPC) with a low cement content has been studied by R. Yu et al. [11] an enhancement by about 28 % was achieved for the 28th day compressive strength of concrete incorporating 4 wt.% of NS. A.M. Said et al. [4] examined the characteristics of concrete containing smaller size NS particles of about 35 nm. The results revealed an enhancement in the 28th day compressive strength by about 36% with the loading of NS up to 6%. Nazari and Riahi [12] investigated the influence of extremely smaller/ultrafine NS of mean particle size of about 15 nm on the compressive strength of concrete, 70% improvement can be achieved with the addition of NS up to 5 wt.%. From the literature survey conducted as a part of the current study, it was concluded that the enhancement ratios in the compressive strength in addition to the optimum NS content are strongly related to the mean particle size of the used NS. The feasibility of CNTs as reinforcement component is not only due to their exceptional high modulus, but also to their high length to diameter aspect ratio. Indeed, the higher the aspect ratio of CNT the higher the contribution to the reinforcement efficiency [13]. Madhavi et al. [1] presented a study on the nano-modification of concrete by multi wall carbon nano tubes (MWCNTs), concrete samples
have been prepared in which MWCNTs with diameter < 40 nm and length < 10 µm were added with the aid of sonication at various ratios ranging from 0.015 and 0.045 wt. %. The highest enhancements obtained for the compressive and splitting tensile strengths were 26.69% and 66.3%, respectively at 0.045% MWCNTs addition. Due to the modest contribution of CNTs to compressive strength as reported in many studies [1, 14, 15] a limited number of researchers tried to add/utilize another micro or nano-pozzolanic materials and investigate their hybrid effect with CNTs to attain additional enhancements. Kim et al. [16] tried to increase the 14 days compressive strength of cement mortars incorporating 0.15 and 0.3 wt. % MWCNTs by adding 10, 20 and 30 wt. % SF, The addition of SF showed promising enhancement in the compressive strength. The concrete mix incorporating 10 % SF and 0.15 % MWCNTs showed the highest compressive strength enhancement i.e., this mix attained 1.29 times higher strength than that of the plain concrete (without SF). Gurumurthy et al. [17] added 1 wt. % nano alumina (NA) into cement composites incorporating 0.75 wt. % MWCNTs, The NA provided additional enhancements in the 28th day compressive strength, flexural strength and toughness by about 27.53%, 30.22% and 98.69%, respectively. Hunashyal et al. [18] added 0.5 wt. % NS to cement pastes containing 0.75 wt. % MWCNTs. The 28th day compressive strength, flexural strength, toughness index and ductility index have been improved by about 12.6%, 51.15%, 72.6% and 5.4%, respectively. Most recent studies on supplementary cementitious materials (SCMs) has concentrated on: examining new materials, changing replacement ratios, adapting new test methods and treating or activating materials to improve performance; nevertheless, limited studies dealt with advances in understanding the mechanisms of improving concrete performance by SCMs. The objective of the current study is to investigate more deeply the role of micro- and nano-supplementary cementitious materials (SF & NS) in enhancing properties of CNTs- modified concrete.

2. Experimental details:

2.1. Materials

2.1.1. Cement

Ordinary Portland cement (OPC: CEM I 42.5 R) was obtained from Suez- Factory in Egypt.

2.1.2. Coarse aggregate

The Corse aggregate was crushed dolomite with nominal maximum size of 20 mm obtained from Attaka quarry.

2.1.3 Fine aggregate
Siliceous sand was used as a fine aggregate with particle size ranging from 0.06 to 4.76 mm.

2.1.4. Water reducer Superplasticiser

Sikament 163M was used as water reducer. Sikamaint 163 has been added as plasticizer with dosage 1.5% of cement weight was incorporated into all mixes to ensure the good workability and no segregation would occur.

2.1.5. Silica fume (SF)

SF with specific surface area of 17 m²/g was supplied from an Egyptian company making ferrosilicon alloys (EDFO). The SF particles are spherical in shape with average diameter of between 1–5 µm.

2.1.6. Nano silica (NS)

NS synthesized by sol-gel procedure were obtained from housing and building National research center (HBRC). The average primary particle size is of about 20-30 nm with specific surface area of about 230 m²/g.

2.1.7. Multiwall carbon nano tubes (MWCNTs)

MWCNTs synthesized by atmospheric pressure chemical vapor deposition (APCVD) were obtained from housing and building National research center (HBRC). The outer diameter is about 10-25 nm; however, the length is of about 25µm.
2.2. Samples preparation

Concrete cubes were prepared with a binder content of 350 kg/m$^3$. The blended concrete samples were prepared in which SF and NS replacing 7.5 % and 2 % by weight of cement respectively. In order to develop the nano hybrid-modified concrete mixes, MWCNTs were added by 0.03 wt. %. All mixes were prepared with a constant water/binder (w/b) ratio. Table 1 illustrates the mix design of the plain and nano-modified concrete. During the preparation of nano modified concrete, the sonication method has been considered to avoid the particle fusion/segregation and to attain high degree of dispersion of nano additives in the blended concrete. NS and CNTs were first added to the mixing water dosed with superplasticiser, the mixtures were stirred with an ultrasonic emitter for 30 min to form an aqueous solution. The aqueous solution with the dispersed/suspended nano particles was mixed with the cement and aggregates using a laboratory electric mixer to prepare the blended concrete. Along with the concrete samples; cement pastes incorporating the nano hybrids have been prepared, especially for the TGA, BET and XRD analyses in order to diminish the effect of aggregates on the accuracy of analyses.

Table (1): Concrete mix proportions (kg/m$^3$)

| Mix ID  | OPC  | SF (replacement) | NS (replacement) | CNTs (addition) | Gravel | Fine Sand | Water | Superplasticizer |
|---------|------|------------------|------------------|-----------------|--------|-----------|-------|-----------------|
| Control | 350  | --               | --               | --              | 1470   | 435       | 125   | 6               |
| CNT     | 350  | --               | --               | 0.105           | 1470   | 435       | 125   | 6               |
| CNT + NS| 343  | 7                | --               | 0.105           | 1470   | 435       | 125   | 6               |
| CNT+SF  | 323.75 | 26.25           | --               | 0.105           | 1470   | 435       | 125   | 6               |

2.3. Test methods

2.3.1. Compressive strength

The compressive strength test was conducted on 15 cm cubic concrete specimens at the 7$^{th}$ and 28$^{th}$ days of curing in accordance with B.S. 1881-116, [19], using 2000 KN Italy Matest pressing machine. The loading rate of 6.5 KN/min was considered.

Three specimens were tested from each mix and the average was calculated.

The pressure resistance of each sample was calculated as follows:

\[
\text{Compressive strength} = \frac{\text{load in N}}{\text{Area in } \text{mm}^2} \text{ MPa} \quad (1)
\]
2.3.2. Surface area and pore structure analysis

Brunauer-Emmett-Teller (BET) method was used to assess the specific surface area (SSA) of the hardened pastes and the Nitrogen adsorption–desorption isotherms were collected using a NOVA2000e (Quantachrome, USA) instrument. Barrett–Joyner–Halenda (BJH) method was adapted for the pore size distribution patterns.

2.3.3. X-ray diffraction analysis

The XRD analysis was carried out to identify the phase composition using a PW 1050/70 Diffractometer (Philips, Holland), equipped with monochromatic Cu-Kα radiation. XRD patterns were recorded in the 2θ range from 5° to 50° at a scanning rate of 2°/min. This test was performed on fine grinded samples passing 75µm sieve to ensure homogenous phase characterization.

2.3.4. Thermogravimetric analyses (TGA)

DSC-TGA Thermogravimetric analyzer (STD-Q600, USA) was used to explore the phase composition of the nano-hybrid modified concrete.

2.3.5. Scanning Electron Microscope (SEM)

The microstructure characteristics were investigated by environmental scanning electron microscope (ESEM- Inspect S, FEI Company, Holland). The 3d-surface topography analysis was performed by the image J software.
3. Results and discussions

3.1. Compressive strength

Figure (1) presents the development in compressive strength for the concrete containing CNTs and the hybrid systems (CNT+NS) and (CNT+SF) at 7 and 28 days of hydration.

As it was expected, the blended concrete showed improved compressive strength at even at early or later ages of hydration. Table (2) summarizes the enhancement ratios achieved after 7 and 28 days for the developed concrete blends.

Table (2): Results for compressive strength tests and the enhancement ratios at 7 and 28 days of hydration

| Mix       | Compressive strength, MPa | Enhancement ratio, % |
|-----------|----------------------------|----------------------|
|           | 7 d | 28 d | 7 d | 28 d |
| Control   | 26.1 | 36.78 | -- | -- |
| CNTs      | 34.93 | 41.80 | 33.83 | 13.37 |
| CNTs+NS   | 36.47 | 50.23 | 39.73 | 36.23 |
| CNTs+SF   | 32.77 | 46.80 | 25.55 | 26.93 |

Although, the CNTs showed a significant enhancement at 7 days of curing, the concrete incorporating CNTs + NS showed the highest enhancement ratios. Actually, the high aspect ratio of CNTs is a very important advantage that makes CNTs contribute physically in improving the mechanical performance and microstructure of concrete, thus serve as a fibers forming crosslinks between cement hydrates, resulting in arresting and bridging micro cracks inside the hardened structure and therefore could suppress crack propagation in the matrix at nanoscale before being pulled out or stressed to rupture [21, 22]. The integration of NS with CNTs, improves the hydration acceleration efficiency and contributes to the early age strength, this is attributed to its nanosize which allows NS to actively act as an active filler thereby providing nucleation/seed centers for the cement hydrates [23]. The combination between CNTs and SF is found to be not effective at early ages and reduces the gain of strength in the CNTs-modified concrete; this is may be assigned to the agglomeration of SF around CNTs within the cementitious structure skeleton which adversely reduces the acceleration efficiency of the cement hydration. After 28 days of hydration, CNTs provided 13.37 % an enhancement in compressive strength relative to the normal/plain concrete; nevertheless, the coupling with SF and NS provided an additional enhancement by about 14% and 23% respectively. These additional enhancements are resulted from the pozzolanic activities of SF
and NS. However, the variation in the provided enhancement ratios is related to the grain size distribution, specific surface area and chemical activity of SF and NS. While NS being nanostructured and composed mainly of active amorphous SiO$_2$ (i.e. more active than SF), these features allow it to play a vital important role in improving mechanical strength of concrete; physically through filling the pores, and chemically through its high pozzolanic activity. In this process SiO$_2$ reacts with the harmful hydration product- Ca(OH)$_2$- producing more calcium silicate hydrate phase in the concrete skeleton and providing a quite compact, homogenous and more dense structure [24,25].

![Bar graph showing compressive strength results of concrete reinforced with CNTs, CNTs + NS and CNTs + SF in comparison with normal concrete.](image)

Fig. 1: Compressive strength results of concrete reinforced with CNTs, CNTs + NS and CNTs + SF in comparison with normal concrete.

**3.2. Surface area and pore structure**

$N_2$-adsorption/desorption isotherms and BJH pore size distribution of hardened cement pastes incorporating CNTs and the hybrid systems (CNTs + NS) and (CNTs + SF) compared to the plain ordinary cement after 28d of curing is presented in fig.2.

As it is clear from the adsorption/desorption isotherms, the examined pastes showed considerable variation in the quantity of the adsorbed nitrogen, depending dramatically on the pore size characteristics and specific surface area (SSA). The four pastes exhibited a physisorption
isotherm of type IV (i.e. porous materials), as evidenced by IUPAC classification [26]. Both the control paste and the paste containing only CNTs adsorbed approximately the same quantity of N₂ manifesting that, they are exhibiting the same surface area. The pastes incorporating the nano hybrids adsorbed more nitrogen due to their significant higher SSA. The SSA of the control, CNTs, (CNTs+SF) and (CNTs+NS) modified pastes are 6.78, 7.06, 13.65 and 17.46 m²/g respectively. The increase in the SSA with nano hybrids addition is logical as the possibly reactive area is increased due to the addition of extra reactive surfaces. This finding conform with the results of previous study which assigned the role of fine minerals including SF, FA, and fine quartz in improving the hydration acceleration efficiency of cement to the increased surface area for CSH nucleation/growth [27]. Furthermore, this may explains the additional enhancements achieved in the mechanical strength of the concrete incorporating hybrid systems and testifies the hydration acceleration provided by NS due to its large SSA and the pozzolanic activity [28]. According to their sizes, pores are categorized into four groups; large pores (macropores) of size >1000 nm, capillary pores have sizes range from 100 to 1000 nm, while transitional or meso-pores are of size between 10 and 100nm, and finally gel pores or nanopores which is considered to be the pores between the (CSH) gel layers have sizes <10 nm [29]. The pore size of the test samples is mainly around 1.5 nm, As it can be observed from the BJH curves, the pores in our prepared mixes are mainly around 1.5 nm; i.e., located in the range of gel pores. It is also clear that, CNTs have not significant effect on the pore volume as it is chemically inert with the cement hydrates. The hybrid systems have a great effect on modifying the pore structure, where the total volume of gel pores was markedly increased. Although the hybrid system (CNTs + SF) showed a considerable increase in the pore volume, the nano hybrid (CNTs + NS) showed the highest intensity due to the higher pozzolanic activity of the nano scale SiO₂. The pore volume of the control, CNTs, (CNTs + SF) and (CNTs + NS) modified pastes are 0.014, 0.018, 0.024 and 0.03 cm³/g respectively. The increased volume of gel pores with the addition of SF and NS is mainly assigned to the filling effect and the formation of additional C–S–H through C2S hydration and pozzolanic reaction [30–32].
3.3. Phase composition analysis by XRD

The XRD patterns of the cement pastes incorporating CNTs, (CNTs + SF) and (CNTs + NS) as compared with the control reference paste at 7 and 28 days of curing are shown in fig. 3. Tricalcium silicate (C3S), dicalcium silicate (C2S), calcium hydroxide (CH), CSH, and calcium carbonate (CC) are the main phases detected. Evidently, the intensities of cement hydrates including CSH and CH increases with the curing time, whereas the peaks of the un-hydrated OPC phases (C3S and C2S) decrease with curing time due to the progress of hydration process. By comparing the CH peaks of the control paste and the paste incorporating only CNTs even at 7 and 28 days of hydration, it is clear that the intensity of CH peaks did not change due to the addition of CNTs. This behavior confirms the very limited role of CNTs in accelerating the hydration kinetics. These results comply with that of TGA and BET. While for cement modified with combination of (CNTs + NS) and (CNTs + SF); the CSH peaks were observed to increase at the expense of CH peaks as a result of the pozzolanic reaction between SiO$_2$ contained in NS and SF with CH, and this explains the remarkably enhanced mechanical performance of (CNTs + NS) and (CNTs + SF) reinforced.
concretes compared to the ordinary concrete. Reducing the CH content in concrete contributes to enhanced strength and improved durability characteristics of concrete structures [33].

Fig. 3. XRD patterns of cement pastes modified with nano hybrid systems hydrated for (a) 7 days and (b) 28 days.
3.4. Thermo gravimetric analysis (TGA)

The TGA results of the cement pastes incorporating CNTs, (CNTs + SF) and (CNTs + NS) as compared with the control reference paste hydrated for 7 and 28 days are displayed in figures (4 & 5) respectively. The decomposition temperatures of the hydration products are the same at both 7 and 28 days of hydration, there were a mass loss at about 200 °C which is due to the CSH phase decomposition, while the reduction in mass at about 470 °C is assigned to the decomposition of CH phase, furthermore, the mass loss appeared at about 700°C is attributed to crystalline CaCO₃ decomposition [3]. From the obtained thermograms, it can be noted that; at 7 days of curing, both control concrete and concrete reinforced with CNTs have almost identical behavior and values, this means that, they have the same CSH and CH contents, where CNTs have no role in promoting the hydration process of cement. On the other hand, the addition of a pozzolanic material to concrete has modified the hydration process resulting in an increase in CSH content and the CH content decreased for concrete reinforced with combinations of (CNTs + NS) and (CNTS + SF), respectively. Furthermore at 28 days of curing; the hydration reaction is almost completed and the hydration phases are formed. Again as it was observed at 7 days, both normal concrete and concrete reinforced with only CNTs have the same hydration products content because of, there is no pozzolanic reaction took place, where CNTs has no chemical contribution in the hydration reaction. They have the highest CH content and the lowest CSH content.

While the lowest CH content and the highest CSH content was found in the concrete-CNTs/NS mix followed by concrete-CNTs/SF mix., which confirms the high pozzolanic activity of both NS and SF, which in turn reflected on the mechanical performance of concrete. This explained behavior was also proofed by the XRD patterns.
Fig. 4: TGA Thermograms of concrete reinforced with CNTs, CNTs/NS and CNTs/SF compared to normal concrete, at 7 days of curing.

Fig. 5: TGA thermograms of concrete reinforced with CNTs, CNTs/NS and CNTs/SF compared to normal concrete, at 28 days of curing.
3.5. Microstructure characteristics

Figure 6 presents the SEM micrographs along with the surface topography/roughness analysis of the control, CNTs, (CNTs + SF) and (CNTs + NS)-modified pastes at 28 days of curing. The microstructure of both the control and CNTs-blended pastes showed the presence of hydration products including; gel-like CSH, hexagonal CH crystals with micro cracks and micro pores. The crack bridging provided by CNTs can be clearly seen. With the inclusion of SF and NS, the gel-like CSH is the predominant phase with the absence of large CH crystals; furthermore, the actions of micro filler and physical packing are evident. Although the surface topography analysis for all cement based mixtures are classified as rough and irregular surfaces. The mixture containing (CNTs + NS) exhibited relatively smoother surface, this is attributed to the unique filler and pozzolanic actions of the nano hybrids and points for the increased gel-like CSH content. This result is compatible with the findings of a previous study, which assigned the smooth surface characteristic of UHPC containing nano metakaolin to the pozzolanic reaction caused by metakaolin [34].
Fig. 6: SEM micrographs and surface topography analysis of cement pastes modified with nano hybrid systems hydrated for 28 days.
4. Conclusions

In this work the combined/hybrid effect of micro- and nano-supplementary cementitious materials including (SF & NS) along with CNTs on the HPC have been investigated. The compressive strength, phase composition, BJH pore size distribution, BET surface area and microstructural characteristics have been determined; the experimental results testify the following conclusions:

- Owing to their fiber like morphologies with very high aspect ratios, CNTs contribute in enhancing the mechanical performance by improving the microstructure of concrete. CNTs are helpful in forming crosslinks between cement hydrates leading to arresting and bridging of micro cracks in the matrix at nanoscale.
- CNTs don't provide significant acceleration effect on hydration degree of concrete.
- The integration of NS with CNTs enhances the hydration acceleration efficiency and contributes to the early age strength. This is attributed to its nano spherical morphology which allows NS to actively act as active filler thereby providing nucleation centers for the cement hydrates.
- The integration of nano hybrids into the concrete led to significant increase in the BET surface area this is ascribed to the addition of extra reactive surfaces.
- The hybrid systems (CNTs + NS) and (CNTs + SF) are effective in providing additional enhancement in the compressive strength and contribute to improved microstructure. This is attributed to the unique filler and pozzolanic actions of the nano hybrids that result in the increased gel-like CSH content.
- As it is chemically inert with the cement hydrates, CNTs have insignificant effect on the pore volume; however, the hybrid systems have a great effect on modifying the pore structure.
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