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## Comprehensive performances of carbon nanotube reinforced foam concrete with tetraethyl orthosilicate impregnation



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#### HIGHLIGHTS

• MCNT improves the pore structure of the foamed concrete.

• MCNT addition influences the polar corrosion durability of the foam concrete.

TEOS impregnation contributes to the hydrophobic feature of MCNTFC surface.

#### ARTICLE INFO

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#### ABSTRACT

Foam concrete plays an important role on the roof, non load bearing walls and thermal pipe insulation layer application because of its special porous structure and excellent insulation performance. In this paper, the effect of multi-walled carbon nanotube (MCNT) loading ( $w_{MCNT}$ ) on the physical properties, water absorption, and AC impedance (Z) of MCNT reinforced foam concrete (MCNTFC) before and after tetraethyl orthosilicate (TEOS) impregnation were studied. Results reveal, MCNT can improve the pore structure and decrease the average pore diameter of the foamed concrete. When  $w_{MCNT}$  alters in the range of 0%–0.1%, the dry density of MCNTFC smoothly changes between 290 and 320 kg/m<sup>3</sup>, the maximal compressive strength reaches 0.302 MPa, and has the increment of above 27% with respect to the plain; The *Z* with 0.05%  $w_{MCNT}$  is the lowest, and  $w_{MCNT}$  has almost no influence on the polar corrosion durability seen from electrochemical Nyquist curves. After TEOS impregnation, and its surface contact angle of MCNTFC is much larger than 90°, and the corresponding maximal water absorption rate is decreased by more than 40%. The surface hydrophobic feature of porous MCNTFC product is good for developing it to a kind of anti-permeable light-weight thermal insulation layer.

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#### 1. Introduction

Foam concrete, with introducing large quantity of fine air foam into concrete, plays an important role on the roof, nonload bearing walls and thermal pipe insulation layer application because of its special porous structure, superior anti-seismic, nonflammability, and heat insulation performances [1–5]. Notwithstanding, there exist some defeats, such as poor stability of the cementitious slurry, weak bond with reinforcement, the relatively low strength, liability to cracking, which restrict its wider applications [6–9].

Thanon et al. [9], Chen et al. [10–12], Nambiar et al. [13], Yakovlev et al. [14], Luo et al. [15] introduced glass, polypropylene, PVA microfiber, or carbon nanotube (CNT) nanofiber into foam concrete in order to improve mechanical strength, anti-cracking, and thermal properties. It's documented that microsize, esp. CNT nanosize fiber can act as the nucleating agent, and effectively reshape the micropores size in foam concrete, obtained round 20% increase in thermal insulation, and 70% increase in strength only with 0.05% CNT reinforcement [14]. As reported in [15], in order to disperse CNT in water homogeneously, and increase the interfacial interaction between CNT and foam concrete matrix, the polycarboxylatebased cement superplasticizer was simultaneously used as the dispersant combined with sufficient ultrasonic treatment.

However, when these fibers reinforced foam concrete products came into contact with water, or put in the place with high humidity, their pore structure, hardened strength, and anti-cracking property frequently deteriorated attributing to water absorption quickly went up through large quantity of transport pathways of porous surfaces [16]. Tetraethyl orthosilicate (TEOS) is a widelyused penetrating agent, and it can be easily hydrolyzed into SiO<sub>2</sub>-·xH<sub>2</sub>O gels after getting contact with alkaline concrete, which is

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helpful to fill capillary pores and improve compact of the concrete surface [17–19]. It is noting that, the anti-permeable and anticorrosion durability of conductive fiber reinforced concrete products are also related to their electrical properties [20]. In fact, AC Impedance responses were effectively used as indicators of the hydration process and electrochemical durability of cementbased materials and distribution state of conductive CNT nanofibers in cement-based matrix [21,22].

In this paper, CNT was firstly dispersed in aqueous system employing surfactant sonication method; CNT dispersion was secondly incorporated into cement slurry, and the compound foaming agents were added to produce CNT reinforced foam concrete; Thirdly, the physical properties (dry density, strength), antipermeable properties (contact angle, water absorption), and AC impedances of the hardened specimens before and after impregnated by TEOS were explored. These consequences are helpful to develop a kind of high-durability foam concrete product.

#### 2. Experiment details

#### 2.1. Raw materials

Multi-walled CNT (MCNT), its main physical index are shown in Table 1. Sulphoaluminate cement, its chemical compositions, characterizing with X-ray fluorescence (XRF) (XRF-1800, Shimadzu Co. Ltd., Japan), are revealed in Table 2. Fly ash (Class I). Hydrogen peroxide ( $H_2O_2$ ), with mass fraction of more than 30%. Superficial active agent (SAA), a kind of polycarboxylic graft copolymer. Thickening agent carboxymethylcellulose (CMC, AR). Tetraethylorthosilicate (TEOS, AR). Protein foaming agent. Distilled water (DSW).

#### 2.2. Mix design and preparation of MCNT modified foam concrete

Three groups of MCNT modified foam concrete specimens were prepared with different loading to binder (including 0.92 cement and 0.08 fly ash) of MCNT ( $w_{MCNT} = 0$ , 0.05% and 0.1%), fixed water-binder ratio (W/B) at 0.7, and foamer-binder ratio (the H<sub>2</sub>O<sub>2</sub>: protein foaming agent was 3.5:1) at 0.045. The detailed preparing procedure was as follows.

A certain amount of MCNT was firstly added into DSW dissolved with 1.0% polycarboxylic-based SAA and 0.4% CMC. Then the MCNT suspension was sonicated at 100 W for 30 min (KQ2200DB type bath ultrasonicator, Suzhou, China) to prepare black uniformed MCNT dispersion with 0.2% concentration. Cement, a trace of alkaline activator, and fly ash was dry mixed with for 2 min employing a mixer with a shear blade, and blended into cement slurry with adding 30% amount of total water at 300 rpm medium speed. MCNT dispersion with predesigned diluting water was then added into the slurry, and mixed for 90 s at 450 rpm high speed. Thirdly, protein foaming agent was dissolved and mixed into the MCNTcement slurry at 300 rpm medium speed for 1 min, H<sub>2</sub>O<sub>2</sub> was dissolved in rest amount of DSW, and subsequently mixed with the mixture slurry for about 10 s at 450 rpm high speed. Fourthly, the well-blended slurry was poured into the oiled mould in three with the size of  $70 \times 70 \times 70$  mm<sup>3</sup> and swell forming MCNT modified foam concretes (MCNTFCs). It is worth to noting, the MCNTFCs used for electrochemical performance measuring were symmetrically pre-inserted a pair of stainless steel meshes at 60 mm intervals throughout the cross section of the MCNTFC. After sufficient swelling and final setting time of the concrete, a saw was used to cut the top of the MCNTFC vertical to the swelling direction. It's noting that, this type saw is small but flexible, and the sawteeth are sharply edged, and the electrodes used for electrochemical performance measuring are mesh grids, benefiting for adequate interface bonding. After curing for overnight, MCNTFC specimens were carefully squeezed out from their moulds by an air compressor, and transferred to a standard curing room (temperature =  $22 \pm 3$  °C, relative humidity  $\ge 95\%$ ) to a predesigned age, as the code GB/T 11969–2008 (China) (similar to BS EN 12390–1:2000) specified.

#### 2.3. Characterization of physical properties of MCNTFCs

A video optical contact angle tester (LS150 type, Shanghai Suolun Information Scientific Co. Ltd., China) was employed to measure the wetting contact angles ( $\theta_c$ ) of each group MCNTFC specimens before and after TEOS impregnation. Single direction saturated water absorption  $(w_{sa})$  before and after TEOS impregnation, and dry density  $(\rho_d)$  of the specimen surrounding with wax sealing was measured by weighting method in 3d, respectively. A universal material testing machine with 40 kN range (DYW-40 type, Wuxi Dongyi Manufacture Scientific Co. Ltd., China) was used to test the compressive strength  $(f_c)$  at a crosshead loading rate of 2.0 mm/min. An LCR digital bridge (Changzhou Tonghui Electronic Co. Ltd., China) was used to measure AC impedance  $(Z_i)$  of the MCNTFCs after oven dried for 12 h at 55 ± 3 °C under different frequency from 50 Hz to 100 kHz employing two-electrode method. A multi-channel electrochemical workstation (Ametek Inc., USA) was used to obtain electrochemical impedance spectrum (EIS) of the cured MCNTFCs after soaking in 3.5% NaCl solution for 30 min employing three-electrode system method, in which the auxiliary, reference electrode was inert Platinum, saturated calomel electrode, respectively. Finally, Some crushed specimens after testing were crushed into tiny samples and oven dried for 12 h at  $55 \pm 3$  °C, and the microstructures of the corresponding samples were observed by scanning electronic microscopic (SEM) (S-5500, Hitachi Inc., Japan) after top surface metal sprayed.

#### 3. Results and discussions

The  $\rho_{ds}$ ,  $f_{cs}$  and the corresponding deviations of MCNTFCs with varied  $w_{MCNT}$  are shown in Table 3 and Fig. 1.

As revealed in Table 3 and Fig. 1, the  $\rho_d$  steadily increases from 292.3 kg/m<sup>3</sup> up to 319.3 kg/m<sup>3</sup>, and the corresponding error gradually decreases along with  $w_{MCNT}$ , which indicates there exists some negative effect on the light-weight property of MCNTFC with MCNT incorporation. Although the  $\rho_d$ s are mostly much lower than those documented in [14], the trend of  $\rho_d$ - $w_{MCNT}$  relation is somewhat contrary to that reported in [14]. Indeed, the nucleating and reshaping micropores effect of MCNT nanofiber may simultaneously render the pore size of the composite smaller and the texture denser, two above counterpoise effects balance the outcomes.

As shown in Table 3 and Fig. 1, the  $f_c$  of MCNTFC arrives at 0.302 ± 0.029 MPa, and has the maximal increment of 27.4% at  $w_{\text{MCNT}}$  of 0.05% with respect to the plain, which implies that a trace of MCNT nanofiber can exert superior nucleating and bridging effect if well dispersed in foam concrete [15,24–26]. The corresponding pore structure is improved, and the average pore diame-

Table 1Main physical properties of MCNT.

Diameter D (nm)	Length $L(\mu m)$	Mass purity	Ash (wt%)	BET surface area (m <sup>2</sup> /g)	Electrical conductivity $\sigma$ (S/cm)
20-40	10–30	>85%	<1.5	>100	>10 <sup>2</sup>

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