Smart Structures and Systems, *Vol. 28, No. 2 (2021) 181-193*
https://doi.org/10.12989/sss.2021.28.2.181

Performance of cement composite embeddable sensors for strain-based health monitoring of in-service structures

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(*Received February 8, 2020, Revised April 11, 2021, Accepted May 29, 2021*)

Abstract. There is a growing need to develop sensors which can be embedded into the structures during the construction stage itself for developing smart structures. It is preferred to develop these kinds of sensors with the material same as that of material used in construction for the sake of compatibility and better capturing the actual state of distress in the structure. Towards this, in this study cement based piezo-resistive sensors are developed with the help of conductive nano-fillers (Carbon Nanotubes (CNTs)). Since the sensors are cement based, and porous in nature, the characteristics of the sensor will vary due to water penetration into the sensor. As the structures with such embedded sensors have to perform for years, understanding the variations in the characteristics of the sensor due to pore structure is very important. In this regard, the conductivity of the sensor is assessed where the effect of dosage of CNTs, functionalization of CNTs, type of electrical conductivity measurement (both DC and AC) and pore water are the parameters. The strain sensitivity of the sensors under cyclic stress is also investigated and reported in the present study. The findings of this study will help in developing continuous health monitoring strategies using highly sensitive embeddable cement-based nanocomposites.

Keywords: carbon nanotubes; cement-based sensors; electrical resistivity; impedance; long-term performance; SHM; tunnelling length

1. Introduction

Development of smart structures is gaining rapid attention in the construction industry due to its ability of long term, continuous and real-time structural health monitoring (SHM). Development and implementation of smart sensors for robust health monitoring of structures has attracted many researchers (Nagayama *et al*. 2007, Rice *et al*. 2010, Hannan *et al*. 2018). In the last few decades, sensor technology has become an integral part of SHM system and demand for the development of new sensors and monitoring techniques is steadily increasing. Many conventional sensors including metallic/semiconductor-based strain gauge, fiber optic sensor (FOS), linear variable differential transducer (LVDT), piezo-electric transducer (PZT), ultrasonic sensor, acoustic sensor, and accelerometer has been used for evaluating the integrity and stability of various existing and developing structures (Sun *et al*. 2010, Güemes *et al*. 2020, Rao and Sasmal 2019). In recent years, wireless health monitoring techniques have also emerged as the mainstream in SHM of large-scale structures due to the problems associated with wired technology, easy installation and low cost (Noel *et al*. 2017, Wan *et al*. 2021, Luo *et al*. 2021, Ma *et al*. 2021). Smart structures can be developed by embedding sensors at identified critical locations at time of construction itself. Data obtained from these sensors will aid in detecting the initiation of damage and monitoring its growth which will, in turn enable in assessing the state of the structure. Thus, it will help in optimising the repair and maintenance activities and also increase the safety of the structures. Another advantage of embedded sensors is its ability to assess the hardened state of the construction material (concrete) which will aid in removal of formwork at the time of construction. Since embedded sensors are integral part of the structure, it will be advantageous if it is made of the same material as that of the structure.

Towards this, various types of cement based smart sensors are being developed by incorporating conductive fillers (e.g., carbon fibers, steel fibers, nickel conductive powder, carbon nanotubes (CNTs), carbon nanofibers (CNFs), graphite nanofibers (GNFs), graphene (G), ZnO nanofibers, etc.) into highly resistive cement composite to impart conductivity and piezo-resistivity. The piezoresistive property of cement-based composite can be used to detect crack initiation in the concrete members (Li and Li 2019), to function as stress/strain sensor (Yoo *et al*. 2018a), Rao *et al*. 2020) and acceleration sensors (Ubertini *et al*. 2014, Tohidi *et al*. 2018). It was also illustrated that the piezo-resistive cement composite can be utilized in traffic monitoring activity such as detection of traffic flow, vehicle speed and weigh-in-motion measurement (Han *et al*. 2009, Yu and Kwon 2009). An innovative strain sensing concept for large structures was proposed by Ryu *et al*. (2011). For health monitoring of massive structures, an innovative idea was proposed by Lezgy-Nazargah *et al*. (2019) by

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ISSN: 1738-1584 (Print), 1738-1991 (Online)

developing a cement-resin and fiber composite which is effective under both static and dynamic conditions.

Cement based composites have a porous, heterogenous microstructure, where the dissolved ions in pore water are mobilized to generate the electric current under induced electric field (electrolytic conduction). When conductive nano-fillers are incorporated into cement-based composites, the conduction mechanism takes place due to electronic (movement of free electrons in conductive fillers) mechanism in addition to electrolytic mechanism (Chung 2004, Chen *et al*. 2004). Therefore, the electrical properties of composite are influenced by the concentration of nano-fillers in the cement matrix and the adopted mixing procedure for incorporating the same in the cement matrix. Chung (2004) conducted experimental study for determining the electrical conduction mechanism within the cement-based composites containing conductive fibers and brought out a relation between the electrical conductivity of cementitious composites and concentration of carbon fibers.

However, few conductive fillers have some specific drawbacks and thereby, not beneficial to use. For instance, steel fibers are prone to corrosion and therefore, a thin passive film forms on the surface leading to an increase in electrical resistivity of cement-based composites (Solgaard *et al*. 2014). Similarly, the incorporation of graphite nanofibers (GNFs) can improve the electrical conductivity of cement-based composites, but it requires a large quantity (minimum of 10% by weight of required cement) to achieve the desired electrical conductivity (Rovnaník *et al*. 2019). With the advancements in nanotechnology, researchers started incorporating conductive nanomaterials like CNTs and CNFs which have superior mechanical, electrical, thermal properties and are extremely light in weight compared to micro fibers to impart conductivity to cement-based composites (Wang and Aslani 2019).

However, dispersing nanoparticles into cement-based materials remains a challenging task due to the high specific surface area of nanomaterials and presence of strong van der Waals force between the particles (Jang *et al*. 2016). To overcome these problems, different methods such as mechanical method, chemical method (covalent surface modification) or physical (non-covalent surface modification by surfactants) have been employed to disperse CNTs (Han *et al*. 2011). Ultrasonication is one of the most promising and commonly used physical methods to disperse CNTs. The use of dispersing agent in combination with sonication process can help in achieving a good quality CNT dispersion by reducing the hydrophobic interactions on the surface of CNT (Reales *et al*. 2018). To enhance solubilization/dispersion of MWCNT in aqueous solution and cement matrix, Luo *et al*. (2009) used five different surfactants, and reported that the efficiency of dispersion is found to decrease in order as SDBS & TX10, SDBS, NaDC&TX10, NaDC, AG, TX10 and CTAB. Apart from the type of surfactant, choosing right sonication parameters are also very crucial. Sasmal *et al*. (2017a) used the SDBS surfactant and have reported optimum sonication parameters (duration, energy and frequency) to improve the dispersion and reduce the agglomeration of CNTs. Surface modification of MWCNTs using H_2SO_4 and HNO_3 has also proven to improve the interfacial interactions between surface-modified nanotubes and cement constituents and hydration. This will produce a strong bonding and increase the load-transfer mechanism from cement matrix to the reinforcement (Li *et al*. 2005). The concentration of CNTs incorporated into cement based material also dictates its conductivity. The conductivity of the composite increases with the increase in the dosage of CNTs. However, beyond the critical value of CNTs, known as percolation threshold, not much improvement in electrical conductivity was observed (Han *et al*. 2009, 2011).

Coppola *et al*. (2011) found that 0.1wt.% CNTs is sufficient to reduce the electrical resistivity of cement composites. Luo *et al*. (2011) used MWCNTs in cement composite with 0.1 wt.% and 0.5 wt.% for measurement of electrical resistance and found that 0.5 wt.% exhibits the least electrical resistivity and shows the good piezo-resistive behaviour, while Konsta-Gdoutos and Aza (2014) reported that 0.1 wt.% CNTs showed the lowest electrical resistivity of cement-based composites by forming stronger bond between CNTs and cement particle. Jang *et al*. (2016) reported that 0.5 wt.% of MWCNTs can reduce formation of clusters and subsequently reduce the electrical resistivity of cement-based composites. Jiang *et al*. (2018) observed that the cementitious composites containing CFs and CNFs have a percolation threshold less than 0.5 wt.% whereas for cementitious composites with CNTs, it is around 1 wt.%. Musso *et al*. (2009) explained that the incorporation of CNTs higher than that of the percolation threshold value (> 0.5 wt.%) leads to agglomeration of CNTs, and thus it deteriorates the mechanical and electrical properties. However, Kim *et al*. (2016) observed the insensitiveness of addition of CNTs beyond the percolation threshold. D'Alessandro *et al*. (2016) carried out studies on the electrical properties of cement-based paste, mortar and concrete incorporated with CNTs. The percolation threshold of cementitious composites with CNTs was found to be 1 wt.% of CNT which is around 2 times higher than the percolation threshold reported by Kim *et al*. (2016). Another major issue experienced during assessment of electrical conductivity of cement-based composites is polarization effect which takes place when the charge accumulates over time in the opposite electrodes, as the centre of positive and negative charge fails to coincide. Due to this, the resistivity of cement-based composites incorporated with CNTs increases as time increases. This phenomenon was more noticeable during direct current (DC) measurement, while by alternative current (AC) measurement, this problem is effectively overcome.

Apart from the influence of CNTs concentration, the electrolytic and electronic conduction mechanisms of cement-based composites containing CNTs are significantly influenced by water content available in matrix (Han *et al*. 2010, Chen and Wu 2013). This becomes a major concern in embedded type of sensors since exposure to moisture to a structure may fill in the pores of the cement-based sensor and affects its conductive property. Many researchers observed that the increase in water content reduces the connectivity among CNT particles in cement matrix and therefore, electrical conductivity of cement-based composites decreases (Han *et al*. 2010, Luo *et al*. 2011, Chen and Wu 2013). Additionally, the increase in water content increases the impact of electrolytic pore arrangement on the electrical conductivity of cementitious composites and sequentially, increase the instability of the electrical conductivity (Chen and Wu 2013, Zhang *et al*. 2019). It was reported that the degree of cement hydration and test time ionic conduction dominates the electrical resistivity of the cement-based nanocomposites.

The conductive fillers form a three-dimensional conductive network inside the cement matrix and reduces the potential barrier for continuous flow of current inside the cement-matrix which help to increase the electrical conductivity. However, the homogeneity of the developed electrically conductive network comprising of CNTs can be compromised by the pore structures in cement-based composites with the progress of hydration (Nochaiya and Chaipanich 2011). In this condition, the pores behave like insulation cap in the cement-based nanocomposites and can increase the electrical resistivity of composites. Sasmal *et al*. (2017b) reported that during the progress of hydration, electrical resistivity increments with time, which is due to the reduction in continuity of capillary pores, evolving micro-structure and modification in level of ionic fluid present in pore.

From the above literature, it is found that many studies are reported on evaluation of electrical conductivity of cement-based nanocomposites and their conductive mechanism inside the cement matrix which are primarily focussed to develop smart structures and systems. However, for successful implementation of the cement based sensors for continuous health monitoring of structures, role of concentration of CNTs, type of electrical conductivity measurement (direct and alternating current), effect of pore water, etc. on the electrical conductivity of the cement-based nanocomposite is extremely important. Since the smart system has to function in the structure without false alarms, it is required to evaluate the change in resistance caused due to reasons other than structural health (due to crack). In view of this, in the present work, an elaborate study has been carried out to determine the optimum dosage of MWCNTs and type of electrodes on the response measurement. Further, the electrical resistivity of cement-based composites incorporated with well dispersed multi-walled carbon nanotubes were investigated. The electrical response was measured by using two-electrode-alternating current (AC) and two-electrode direct current (DC). To understand the effect of pore water and microstructure of the sensing system (porous cement matrix abridged through discrete CNTs), different stages of hydrated cement system was considered as it typically provides a wide range of porous structure. Further, to obtain the extreme condition of least/zero pore water system, mechanical heating of the cement based sensors was carried out. From the initiation of hydration to the externally heated samples, represents almost all the possible cases of microstructure with pores and water in the cement based sensor when it is embedded in structure. Further, the efficacy of developed sensors for self-sensing capacity was explored under long cyclic load. The studies carried out, observations made, and inferences drawn from the present study are described in the following sections.

2. Experimental studies

2.1 Raw materials

Ordinary Portland Cement of grade 53 (density-3159 kg/m^3) was used for fabrication of cement-based nanocomposites. Three different types of CNTs, viz., one non-functionalized pristine MWCNT (P-MWCNT) and two chemically functionalized MWCNT (carboxyl acid (-COOH) and hydroxyl (-OH) group) were incorporated into cement matrix. CNTs were procured from Nanostructured and Amorphous Materials Inc., Houston, Texas. The properties of pristine and functionalized MWCNTs, as provided by the manufacturer, are listed in Table 1. Different MWCNTs of similar physical properties were chosen so that the measured electrical properties can be reasonably comparable. Water to cement ratio was kept constant at 0.4 for all the mixtures. To ensure the dispersion of the MWCNTs in the cement matrix, Sodium Dodecyl Benzene Sulfonate (SDBS, 348.48 MW, 80% assay) was used as dispersing agent, while tributyl phosphate was used as defoamer to decrease the formation of air bubbles. The surfactant SDBS was selected based on its capability to act as the better superficial active agents (SAAs) for dispersing MWCNTs as reported in various studies (Luo *et al*. 2009, Blanch *et al*. 2010). The surfactant to nanotube ratio (S/C) was selected to be 0.55 based on the experimental studies conducted by Sasmal *et al*. (2017a).

2.2 Dispersion of CNTs

High aspect ratio and hydrophobic nature of CNTs increase the possibility of entanglement and close packing of CNTs, and hence, lead to certain difficulties for incorporation in any medium. Also, due to strong van der Waals force of attraction, pristine CNTs have low dispersibility and high tendency to agglomeration. Therefore, to reduce the agglomeration and ensure the uniform dispersion of CNTs, both the physical- and chemical- methods were used. Chemical method involves using the chemical solvent to improve the dispersibility of CNTs. The solvent used in this study was synthesized by mixing surfactant power SDBS with distilled water for 5 minutes followed by magnetic stirring for 10 minutes at a speed of 250 ± 5 rpm at a concentration of 0.02 g/ml. Then,

Table 1 Properties of Pristine MWCNTs, OH and COOH-MWCNTs

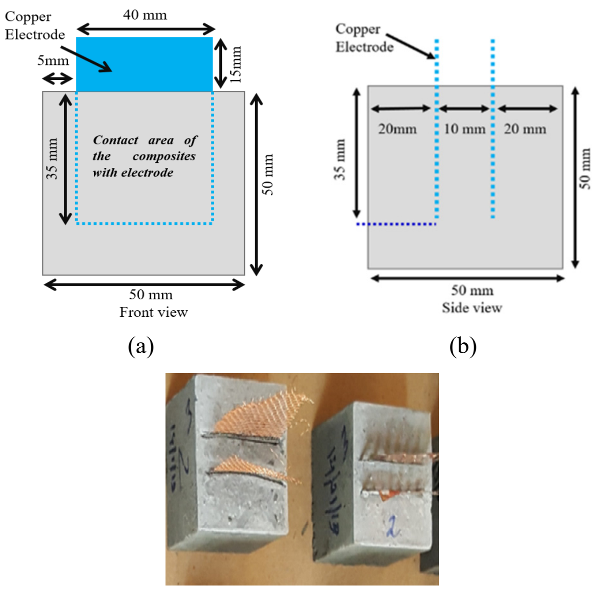
OD	ID	L	ρ	Purity	SSA
(nm)	(nm)	(μm)	$\left(\frac{g}{cm^3}\right)$	(%)	$\left(\frac{m^2}{g}\right)$
50-80	5-15	10-20	0.18	> 95	> 40

*OD = Outer diameter; ID = Inner diameter; L = Length; ρ = Bulk density; SSA = Specific surface area the CNT-water-surfactant solution was subjected to sonication (physical method) using probe ultrasonicator (Hielscher, ultrasound technology) for 45 minutes.

2.3 Specimen preparation

Four different types of smart cement composites (containing 0.1, 0.25, 0.5, and 0.75 wt.% by cement weight) were synthesized from each group of CNTs (P-MWCNT, OH-MWCNT and COOH-MWCNT). For preparing the specimens, the sonicated CNTs suspension was added to the measured quantity of cement and mixed thoroughly. The remaining water, to achieve w/c ratio of 0.4, was then added (the amount of water used while preparing the aqueous solution is taken into consideration) and mixed. At last, tributyl phosphate defoamer of 0.25 vol.% was added into the cement/CNT paste and mixed properly for 3 minutes to reduce the formation of air bubbles.

The well mixed fresh composite was then poured into oiled cubic molds of size 50 mm × 50 mm × 50 mm and compacted on a vibrating table to ensure desired compaction. For measuring the electrical resistivity, copper wire mesh electrode (1.6 mm × 1.6 mm) with dimension of 40 mm × 50 mm was utilized, which was embedded into the specimens immediately after casting. The electrodes were embedded in parallel plane of specimens to a depth of 35 mm from top surface as shown in Fig. 1. The distance between two electrodes was 10 mm and distance from outer side of specimen was 20 mm. Three samples of each type were fabricated following the same preparation method. After 24 hours, the samples were demoulded and allowed to cure for 28 days under the laboratory condition 20 ± 2°C. Finally, few identified samples were kept in an oven at 60°C for 3 days to dispense with abundance water content.

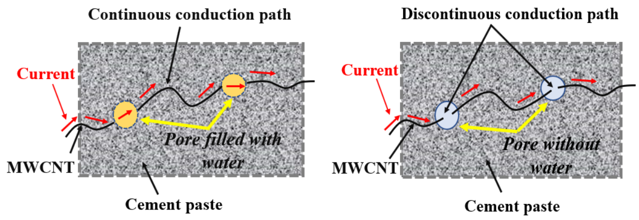


(c)

Fig. 1 Schematic diagram of two probe sample (a) front view arrangement; (b) side view arrangement; and (c) cured sample

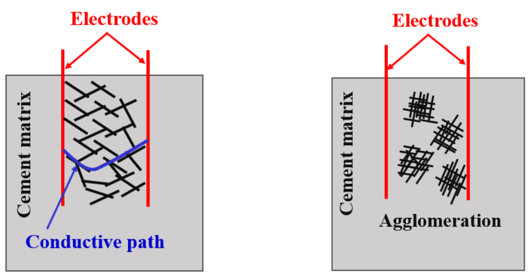
3. Electrical conductivity measurement

Unlike polymer matrix or polymer composite, which is highly non-conductive in nature, the cement matrix exhibits some electrical properties due to presence of different ions. Due to conductive nature of cement matrix, incorporation of even a meagre amount of conductive fillers into the cement matrix can enormously enhance the electrical conductivity. The electrical conductivity of cement-based composites incorporated with conductive nanomaterials is attributed to two types of conduction mechanism: electrolytic and electronic (Chung 2004, Chen *et al.* 2004). As discussed in the previous section, the electrolytic conduction mechanism is attributed to the cement matrix due to the presence of ions like Na^{+}, Ca^{2+}, K^{+} *or* OH^{-} in the evaporable water present in the porous cement paste and the movement towards oppositely charged electrodes. On the other hand, the electronic conduction mechanism involves the movement of free electrons in the conductive fillers, i.e., CNTs in this study. Thus, in the cement-based composite containing conductive nanotubes, the electrical conduction will be through a combination of ion and electron flow and both play an important role in the electrical conductivity of whole composite system (cement paste+conductive fillers). Therefore, in cement-based composites with conductive fillers, there will be three possible conduction pathways to determine the electrical conductivity or resistivity of the entire composites: (a) through capillary pore water inside the cement-based composites (as shown in Fig. 2); (b) through a conductive network of carbon nanotubes (as shown in Fig. 3); and (c) through the CNT-cement matrix-CNT in series.



(a) (b)

Fig. 2 Electrically conductive measurement (a) pores filled with water; (b) dry pores



(a) (b)

Fig. 3 Conduction path in CNT incorporated system; (a) optimum content and well-dispersed; (b) excessive content and agglomerated

The electrical conductivity between two pathways (b and c) depends on the concentration of nanotubes. When the dosage of CNT is close to percolation threshold and a network of carbon nanotubes is formed, the distinct pathways provide the conduction route and consequently, in general, the electrical conductivity increases. Path b) brings around ohmic behaviour entirely through the flow of free electrons, whereas in path c), a conduction mechanism named as electron tunnelling takes place which is due to the fact that the microstructure of the cement-based matrix network hinders the formation of a continuous path of nanotubes, causing the electrons to tunnel from one nanotube to another (Cao and Chung 2004).

Due to the heterogeneous microstructure, a cement-based composite materials cannot be treated as an ideal resistor and the research conducted on cement-based composites containing conductive nanoparticle indicates that the electrical conductivity of the nanotube-cement matrix interface is frequency dependent (Hou 2008). The investigation of electrical resistivity/conductivity can be realized by using direct current (DC) or alternating current (AC). The DC method is easier for measurement and implementation, therefore sensing by AC impedance measurement is less popular than DC resistance measurement (Li 2013). In this study both AC and DC techniques are used for electrical conductivity measurement of the nano engineered cement composites to quantitatively show their respective efficacies.

3.1 DC test method

In the present study, two probe method was adopted to assess the electrical characteristics of cement-based composites. The DC electrical resistance was measured using an Agilent 34401A Digital Multimeter, and the measured resistance was converted into the resistivity using following equation

$$\rho = \frac{RA}{L} \tag{1}$$

Where, R and ρ denote the resistance (Ω) and resistivity (Ω-cm) of cement-based nanocomposites respectively. A is the cross-sectional area of the specimen and L is the distance between the two electrodes. For each experiment, a set of three specimens was cast and measurements were taken to check the consistency of the results and finally, the average value from the consistent results is reported. During electrical resistivity measurement, due to the presence of an electric field, polarization will occur in the material. The polarization affects the electrical resistance measurement by giving a nonlinear response during the initial phase. In this study, all the measurements were carried out after the system has reached its stable state (i.e., after polarization). The experimental set-up for DC electrical resistivity measurement is shown in Fig. 4(a).

3.2 AC test method

In order to avoid the polarization problem, an alternating current (AC) is often used for determining the resistivity of cement-based composites. In the present study, the AC electrical resistivity of cement-based nanocomposites was measured using Agilent 4294A impedance analyser (Fig. 4(b)). The amplitude of the sinusoidal voltage was chosen to be 500 mV rms. The resistance (real part of impedance) measurement frequency was swept from 40 Hz to 100 kHz and whole range was swept by setting 801 number of points. Finally, resistivity of composites was calculated using Eq. (1).

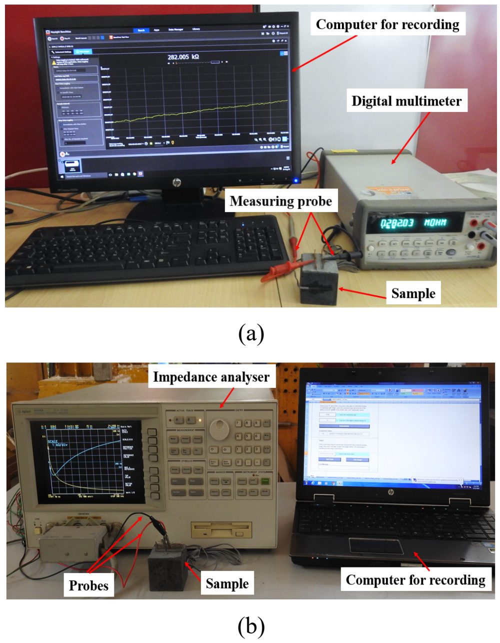


Fig. 4 Electrical resistance measurement set-up (a) DC measurement; (b) AC measurement

4. Results and discussion

4.1 DC electrical resistivity

(a) Electrical resistivity during hydration.

Cement based composites are chemically reactive in nature. They undergo hydration process during which compounds in cement react with water to form hydration products. This process takes place either till all the pozzolanic compounds or free water is fully consumed. Due to the change in microstructure and water content during the course of hydration (28 days in general) in cement-based composites, its electrical resistivity also changes. During initial stage of hydration, more capillary water is available which forms a continuous conductive pathway. As hydration progresses, the capillary water gets consumed for the formation of hydration products leading to the damage/interruption in the conductive pathway which may be one of the major reasons for increase in resistivity of cement-based composites with hydration. The electrical resistivity increases with age for all samples (with different dosages and without MWCNTs) due to hydration process. The rate of increase in electrical resistivity of plain cement paste with curing age is much higher than other samples (Fig. 5(a)). This may be due to the reason that capillary

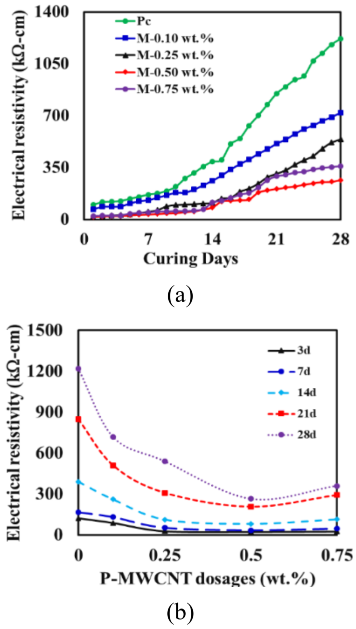


Fig. 5 Change in DC electrical resistivity with (a) hydration time; (b) weight fraction of MWCNTs

water is the major source of conductivity in plain concrete paste which gets reduced as hydration progresses and hence the resistivity increases, whereas in cement composite with nano materials, some conductive fillers are also present in addition to water which is responsible for conductivity of the composites. Hence, the rate of increase in resistivity in plain cement paste is much higher compared to the cement composites with conductive fillers during hydration. Another important phenomenon was observed after 14 days of hydration i.e., a drastic increase in electrical resistivity. This can be related with the densification of the cement paste with non-conductive phases, particularly considering calcium silicate hydrate and calcium hydroxide (Yoo *et al.* 2018b, Tyson *et al.* 2011).

(b) Electrical resistivity vs. CNTs concentration

In this study, the effect of four different concentrations (0.1, 0.25, 0.5, and 0.75 wt.%) of MWCNTs on the electrical resistivity of composite is investigated. The influence of the dosage (weight percentage w.r.t cement) of MWCNTs on the electrical resistivity of cement-based composites can be visualized in Figs. 5(a) and(b). At low concentration (0.1 wt.%), the resistivity remains very close to the resistivity of pure cement matrix of cement-based composites. However, in Fig. 5(b), a drastic reduction in resistivity (or increase of conductivity) is observed from 0.25% of MWCNTs at 3rd day of hydration process. This demonstrates that the threshold of the cement composites in this study is 0.25 wt.% at 3 days of hydration. With the increase in the hydration stages, the effect of CNTs was more noticeable and the decrease in electrical resistivity is found to increase with hydration time. Similar observation can be made up to 14 days of hydration process (Table 2). Beyond that (at 21 and 28 days) the drastic increase of conductivity occurs only after 0.5 wt.% which states that

Table 2 Decrease in electrical resistivity (%) of cement composites incorporated with P-MWCNT

Before heating	0.10 wt.%	0.25 wt.%	0.50 wt.%	0.75 wt.%
3 d	27.44	59.27	66.07	68.54
7 d	21.34	58.68	65.42	58.22
14 d	32.78	70.95	74.86	70.51
21 d	39.75	63.59	75.71	65.60
28 d	40.91	55.63	78.24	70.42

percolation threshold varies from 0.25 wt.% to 0.50 wt.% in cement composites based on the degree of hydration.

The addition of 0.5 wt.% of P-MWCNTs is able to decrease the resistivity of cement-based composites up to 78.24% at 28 days of curing. From Table 2, it can be noticed that addition of P-MWCNTs above 0.50 wt.%, the electrical resistivity started to increase due to poor dispersion and agglomeration of P-MWCNTs inside the cement matrix. This was even evident from UV-Vis spectroscopy and particle size analysis studies carried out in author's previous investigations (Sasmal *et al.* 2017a, b). The electrical resistivity of cement-based composites (after 28 days of hydration) can be decreased by 40.91%, 55.63%, 78.24% and 70.42% with the introduction of 0.10 wt.%, 0.25 wt.%, 0.50 wt.% and 0.75 wt.% of P-MWCNTs respectively (Table 2).

(c) Influence of functionalization of CNTs on electrical resistivity

Functionalized MWCNTs with carboxyl group (COOH-MWCNTs) and hydroxyl group (OH-MWCNTs) have distinct physical properties and are more hydrophilic in nature in comparison with non-functionalized MWCNTs (pristine MWCNTs), due to which their effect on properties of cement-based composite can be different. The introduction of both the types of functionalized MWCNTs into cement-matrix, can decrease the electrical resistivity of cement-based composites as shown in Figs. 6 and 7. Compared to the composites with P-MWCNTs, the cement-based composites with COOH-MWCNT display lesser electrical resistivity, while the composites with OH-MWCNTs display little higher electrical resistivity during initial days of hydration (3, 7 days) and at low concentration of MWCNTs (0.1 wt%). The lowest rate of decrease in resistivity was observed at 0.10 wt.% of both functionalized MWCNTs during every stage of hydration (Tables 3 and 4). However, in comparison, the rate of decrease in resistivity with COOH-MWCNT is higher than that of the rate of decrease in resistivity with OH-MWCNTs. The presence of functionalized carbon nanotubes with the COOH group in the hydration process acts as a bridge between the polar hydrophilic cement matrix and non-polar carbon nanotubes, and it causes nanotubes to get better dispersed in the cement matrix. Furthermore, the COOH-MWCNTs penetrate well inside the cement products and by bridging, they form a good conductive path inside the cement matrix with CNTs and

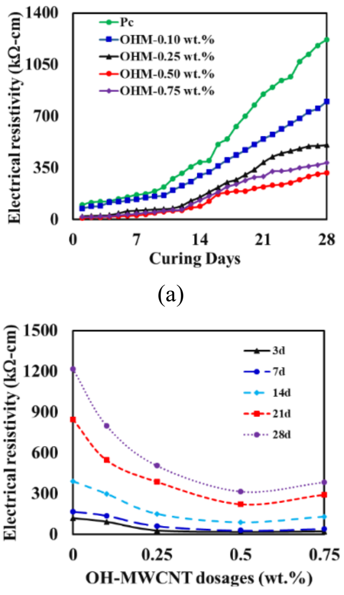


Fig. 6 Change in DC electrical resistivity with (a) hydration time; (b) weight fraction of OH-MWCNTs

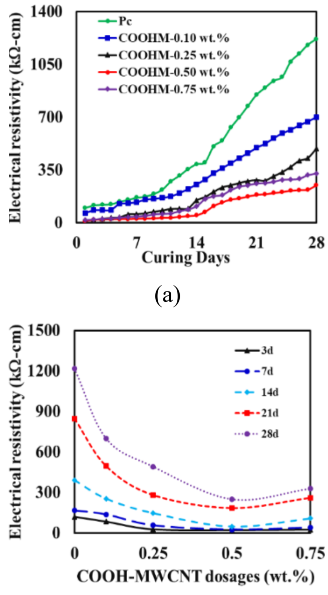


Fig. 7 Change in DC electrical resistivity with (a) hydration time; (b) weight fraction of COOH-MWCNTs

Table 3 Decrease in electrical resistivity (%) of cement composites incorporated with OH-MWCNT

Before heating	0.1 wt.%	0.25 wt.%	0.5 wt.%	0.75 wt.%
3 d	22.13	46.15	52.97	60.48
7 d	17.94	48.12	56.29	63.54
14 d	23.22	60.87	72.29	66.20
21 d	35.48	54.23	73.79	65.48
28 d	34.25	58.34	74.03	68.43

Table 4 Decrease in electrical resistivity (%) of cement composites incorporated with COOH-MWCNT

Before heating	0.1 wt.%	0.25 wt.%	0.5 wt.%	0.75 wt.%
3 d	29.34	54.25	63.97	63.80
7 d	17.57	56.91	69.05	63.70
14 d	34.54	61.88	74.54	72.05
21 d	41.32	66.72	78.06	69.13
28 d	42.44	59.68	79.53	73.05

hence the electrical resistivity decreases in comparison with P-MWCNTs.

(d) Effect of electrode types on electrical resistivity

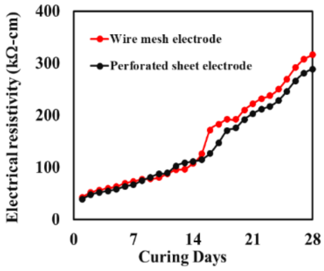
By observing the results obtained from the experimental studies, it is clear that the composites with 0.5 wt.% CNTs shows the considerable decrease in electrical resistivity in all cases. However, the observations were from one type of electrode, i.e., copper wire mesh. Since the type of electrode can also affect the electrical resistivity of composite materials, it is worthy to consider more than one type of electrode. Therefore, for comparison purpose, the cement-based composites incorporated with 0.5 wt.% CNTs (all three types of MWCNT) were fabricated by embedding copper perforated sheet as the electrode. The electrode properties and fixing style is listed in Table 5. The types of electrode are shown in Figs. 7 and 8.

The electrical resistivity of the composites with wire mesh electrode and perforated sheets electrode is shown in Fig. 8. The experimental results show that the electrical resistivity of the composites exhibit almost similar behaviour as the composites with wire mesh electrodes. The resistivity increases with increase in hydration progress. From Fig. 8, it is observed that the electrical resistivity of the material with perforated sheet electrodes is almost same as the electrical resistivity of material with wire mesh electrode upto 14th day of hydration process. However, after 14 days of hydration process, the resistance measured using perforated sheet electrode shows little lower value in comparison with wire mesh electrode. Due to this, the self-sensing capacity of the nanocomposites fabricated with different types of electrodes vary, which is presented in detail in Section 5.0.

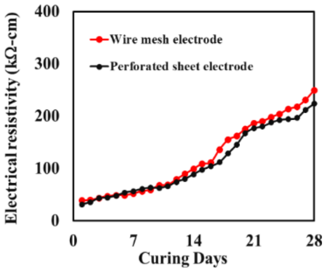
Table 5 Electrode properties and fixing style

Types	A	B	t	Fixing style
WME	4×5	1.6×1.6	0.3	E
PSE	4×5	4 (hole dia.)	0.3	E

*(WME- Wire Mesh Electrode; PSE- Perforated Sheet Electrode; A- Electrode dimension (cm); B- Mesh size (cm); t- thickness of electrode (mm); E- Embedded)



(a)



(b)

Fig. 8 Comparison in DC electrical resistivity with different electrodes at 0.5 wt.%: (a) OH-MWCNT; (b) COOH-MWCNT

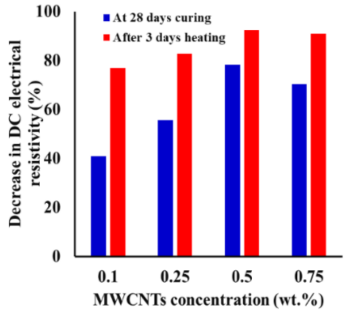
(e) Influence of external heating on the electrical resistivity

After 28 days of hydration, the samples were kept in oven for 3 days at 60°C to find out the effect of external heating and drying on the electrical resistivity of cement-based nanocomposites. The electrical resistivity of nanocomposites increases after heating in comparison with the resistivity measured at 28 days of curing without heating. The results, as shown in Fig. 9, clearly indicate that the presence of water inside the composites even after 28 days of hydration, while, after heating, all available water gets evaporated and the resistivity drastically increases. Another important observation is that the decrease of resistivity with respect to plain cement paste is higher in samples after heating in comparison to samples before heating (Fig. 9). This is due to the fact that before heating, there is contribution both from ionic water and CNTs connectivity, in overall conductivity of the composite, whereas, after heating, the contribution from ionic water is lost.

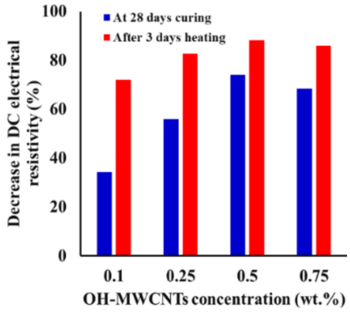
4.2 AC electrical resistivity

The alternating current (AC) electrical resistivity of the cement-based composites incorporated with nanomaterials was measured using a high precision impedance analyser (Agilent 4294A) over the frequency range from 40 Hz to 100 kHz at room temperature after 28 days of hydration. Fig. 10 shows the frequency dependence of the AC electrical resistivity of composites containing different weight fraction of the carbon nanotubes.

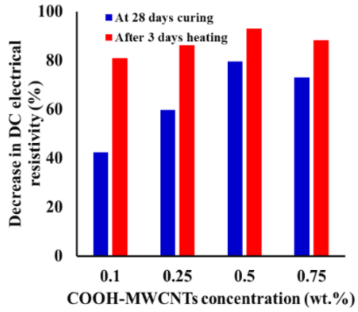
A strong frequency dependence, i.e., a slow decrease in AC electrical resistivity with an increase in frequency is observed for plain cement paste, which indicates an



(a)



(b)



(c)

Fig. 9 Decrease in DC electrical resistivity of cement-based composites before and after heating with respect to the plain cement paste incorporated with (a) MWCNTs; (b) OH-MWCNTs; (c) COOH-MWCNTs

electrical insulator or high electrical resistivity characteristic of composites. Incorporation of small amount of the conductive fillers (0.10 wt.% of MWCNTs) into cement matrix also shows the same behaviour as plain cement paste with slow linear decrease in resistivity as frequency increase from 40 Hz to 100 kHz. This indicates that the resistivity is strongly influenced by the measuring frequencies. In this situation, the exhibited typical dielectric behaviour of cement-based composite indicates the absence of strong and stable conductive path in the composites due to insufficient amount of CNTs (Hou 2008).

While further addition of conductive fillers (0.25 wt.%) the electrical resistance significantly decreases within measured frequency range. At a higher concentration (0.50 wt.% and 0.75 wt.%) of CNTs the electrical resistivity values are almost independent of the change of frequency (as shown in Fig. 10). In this situation, the conductive fillers are very close to each other, and the conductivity is dominated by Ohmic behavior, as a result of charge carriers

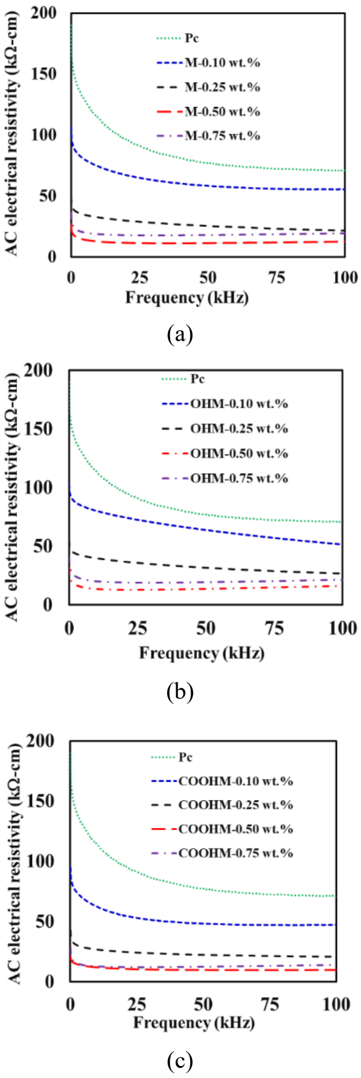


Fig. 10 AC electrical resistivity of cement-based composite with change in frequency incorporated with (a) MWCNTs; (b) OH-MWCNTs; (c) COOH-MWCNTs

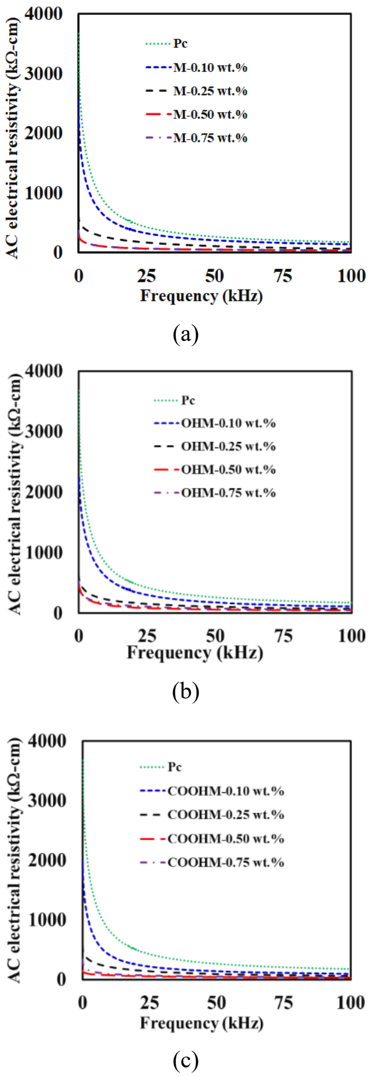


Fig. 11 AC electrical resistivity with frequency after heating incorporated with (a) MWCNTs; (b) OH-MWCNTs; (c) COOH-MWCNTs

traveling through the conductive pathways (Lazarenko *et al.* 2009). One important observation is that around percolation threshold, the AC electrical resistivity becomes nearly frequency independent.

The resistivity of cement composite decreased by 21.47% by incorporation of 0.1 wt.% of MWCNTs while a significant reduction of 69.82% and 82.35% in electrical resistivity was observed at a frequency of 100 kHz after incorporation of MWCNTs of 0.25 wt.% and 0.50 wt.% respectively. The decrease in resistivity of cement composites is very less with 0.1 wt.% P-MWCNTs, in comparison to decrease in resistivity of cement paste with 0.25 wt.% and 0.50% P-MWCNTs. This reduction in electrical resistivity indicates that the percolation threshold is around 0.25 wt.%. However, when MWCNT is added beyond 0.5 wt.%, an increase in electrical resistivity is observed.

The same frequency dependence behaviour was observed in case of functionalized MWCNTs. The incorporation of functionalized MWCNTs decreases the electrical resistivity of cement-based composites as similar to P-MWCNTs. At 0.10 wt.%, the decrease in electrical resistivity is observed to be very less in both the functionalized MWCNTs (OH and COOH). However, comparatively, decrease in electrical resistivity was more in COOH-MWCNT (33.36%) than that of 0.1 wt.% OH-MWCNTs (27.57%) at 100 kHz frequency. Similarly, highest decrease in electrical resistivity was observed due to addition of 0.5 wt.% in both types of functionalized MWCNTs. The decrease in electrical resistivity of cement paste incorporated with 0.5 wt.% of COOH-MWCNTs and OH-MWCNTs was found to be 86.27% and 77.14% respectively, while in P-MWCNT the decrease in electrical resistivity was 82.35% at the same weight fraction at 100 kHz frequency.

The measurements were taken again after subjecting the specimens to heating in oven at 60°C for 3 days in order to nullify the effect of trapped or unconsumed water in the cement matrix. Fig. 11 shows the electrical resistivity of cement composites with different types of MWCNTs at different concentration after heating (AH). After heating, the AC electrical resistivity of plain cement paste increases drastically. The increase in resistivity was observed in all cases as it was observed during DC resistance measurement. Increase in MWCNTs concentration decrease the resistivity. However, the rate of decrease in resistivity with respect to plain cement composites at 40 Hz frequency after heating is more in comparison with the rate of decrease in resistivity at 40 Hz frequency at 28 days hydration before heating (BH). At 100 kHz frequency there was not much influence of heating and resistivity of the samples was observed almost similar before heating (BH) and after heating (AH).

5. Self-sensing of strain during stress development

For demonstrating the performance of the smart cement-based composite under cyclic loading, cyclic stress was applied through a compression testing machine. The change in electrical resistance in the cement composite due to the change in internal path was measured online using a high accuracy voltmeter, as shown in Fig. 12. From this, the fractional change in resistance is calculated using the expression as given below

$$FCR\ (\%) = \frac{R_t - R_0}{R_0} \times 100 \tag{2}$$

Where, R_t is the resistance at time, t and R_0 is the initial resistance before loading. Accordingly, gauge factor is determined from the fractional change in resistivity per unit strain which is expressed as

$$\text{Gauge factor } = \frac{FCR}{strain} = \frac{R_t - R_0}{R_0}/\varepsilon \tag{3}$$

Where, ε is the strain in the composite measured from strain gauge.

The experimental set up for piezoresistive test is shown in Fig. 12. The response in terms of fractional change in resistivity with the change in strain for all types of sensors (cement with pristine CNT, OH-CNT, and COOH-CNT) using wire mesh electrode and perforated sheet electrode are shown in Figs. 13 and 14, respectively. It is important to mention that, though a range of CNT dosage was

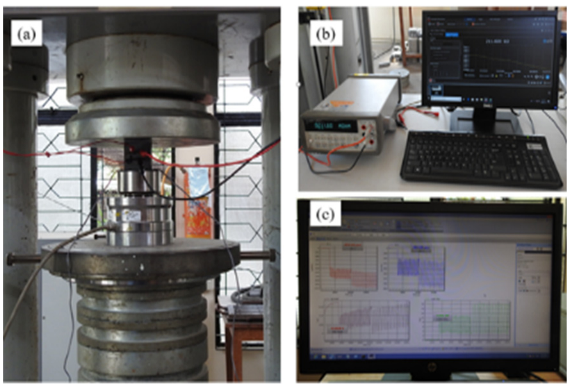


Fig. 12 Instrumentation set up for piezoresistive test

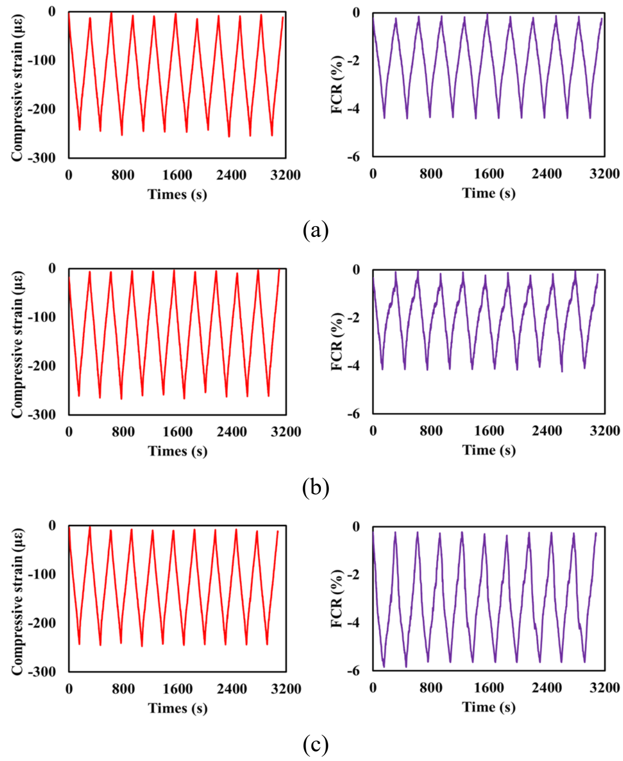


Fig. 13 Piezo-resistive response of cement composite: (a) 0.50 wt.% MWCNTs; (b) 0.50 wt.% OH-MWCNTs; and (c) 0.50 wt.% COOH-MWCNTs measured from wire mesh electrode

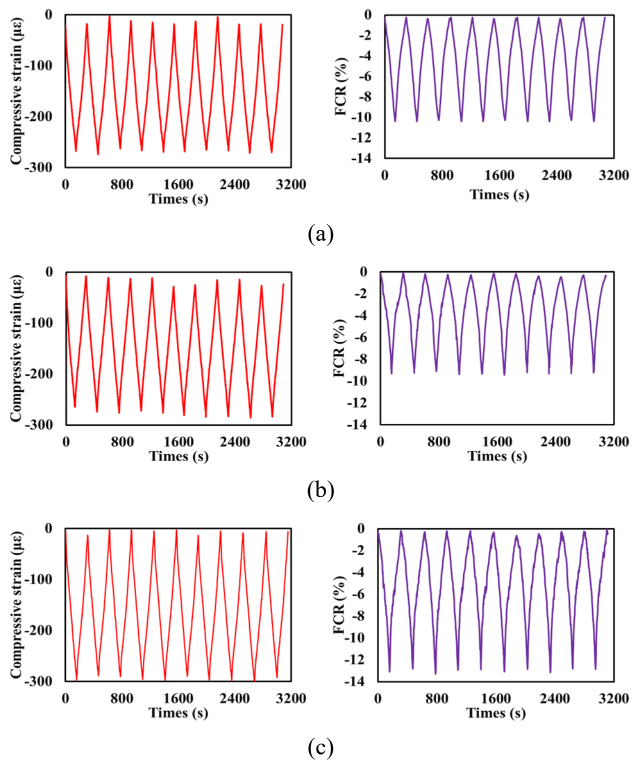


Fig. 14 Piezo-resistive response of cement composite: (a) 0.50 wt.% MWCNTs; (b) 0.50 wt.% OH-MWCNTs; and (c) 0.50 wt.% COOH-MWCNTs measured from perforated sheet electrode

Table 6 Gauge factor of developed sensors at 0.50 wt.% MWCNT

MWCNT	WME	PSE
Pristine-	172.06	407.16
OH-	151.98	367.73
COOH-	222.31	451.26

investigated, the reported results here are typical for 0.5% CNT dosage. It is found that the developed sensors are very effective in sensing the change in strain. The calculated gauge factor for COOH-CNT based cement sensor was above 450, which can be treated to be the best among the reported results. The calculated gauge factor for both type of electrode is listed in Table 6.

6. Conclusions

A detailed study on microstructure-dependant conductive (electrical) properties of cement-based composites is presented. In the present study, pristine and two types of functionalised CNTs are used to study their efficiencies to develop the tunnelling mechanism in the microstructure of the cement-based composite and the effect of percolation threshold thereon. The microstructural variation in cement-based composite during hydration and heating were considered. Polarization effect of the developed composite due to AC and DC current measurements were also studied. The major remarks are:

- The electrical resistivity of plain cement paste was significantly low with the incorporation of conductive MWCNTs. With increase in the concentration of MWCNTs, resistivity of the composite was found to be reduced. Functionalization of MWCNTs has considerable effect on the electrical resistivity of developed composite. The rate of decrease in electrical resistivity with 0.5 wt.% of COOH-MWCNTs was found to be 79.53%, which is higher than P-MWCNTs (78.24%) and OH-MWCNTs (74.03%).
- The rate of decrease in electrical resistivity of the composites was greatly influenced by external heating after 28 days of normal curing. The rate of decrease in resistivity increases after heating in comparison with without heating.
- The investigations related to AC measurement reveal that input frequency of AC has significant role in reduction of electrical resistivity of cement-based nanocomposites. The influence of microstructure/pore water on the resistivity is significantly dependent on the operating frequency range during the AC measurement. It is also observed that the frequency dependency is more pronounced with less concentration of CNTs (< 0.5 wt.%).
- The type of electrode (copper wire mesh and copper perforated sheet electrodes) has shown very less effect on the overall measured resistivity of cement-based nanocomposites and has considerable effect on the self-sensing capacity. The gauge factor of the developed sensor with 0.5 wt.% of CNTs fabricated with perforated sheet electrode was about 450, which is the better than the available/reported cement based sensors for embedded sensing.
- The continuous cyclic tests on the sensors show that the effectiveness and sensitivity of the cement-based sensors are extremely promising for usage as embedded sensors in the structures. It is also important that the functionalization of CNT improves the gauge factor (reflecting the sensitivity in resistance change due to change in strain).
- The present study emphasizes that cement-based sensors are extremely promising for strain sensing, however also dependent on the microstructure which has a significant influence due to the presence of pore water. Thus, a proper water proofing coating with appropriate arrangement is to be applied before embedding the sensor in structure for consistent output, otherwise the received data is to be analysed appropriately considering the site consideration.

Acknowledgments

The contribution from the staff members of SMSL of CSIR-SERC during preparation of specimens and carrying out the experimental studies is acknowledged. The first author would like to thank CSIR-Senior Research Fellowship for the financial support.

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