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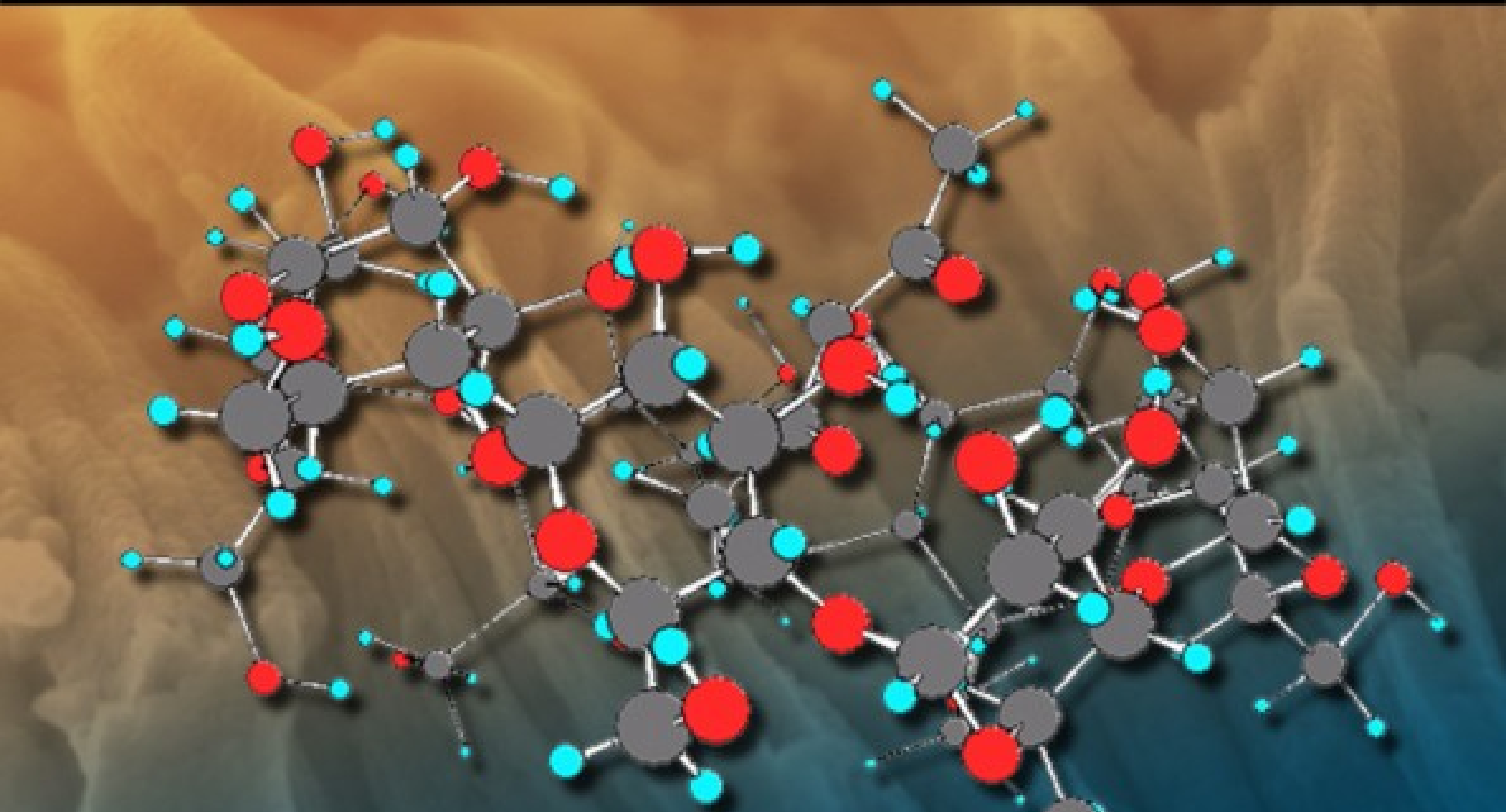

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Contents

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Multiwalled Carbon Nanotubes Reinforced Cement Composite Based Room Temperature Sensor for Smoke Detection

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Abstract: In this paper, smoke sensing property of pellets, fabricated from multiwalled carbon nanotubes (MWCNTs) (w: w) reinforced with Portland cement has been demonstrated; so as to explore their feasibility as an embedded smoke sensor for civil structures. DC transient response depicted increase in their electrical conductivity when exposed to smoke. The increase in responsivity of the pellets under smoke was found in the range 26-46 % depending upon the MWCNTs content. Ac impedance spectroscopy performed at room temperature under ambient and smoky environments over a frequency range 100 Hz - 2 MHz has demonstrated a gradual increase in the ionic conductivity with frequency for all composites. This increase in ionic conduction was also found to be assisted with an increase in the percentage of MWCNTs. The sensing mechanism has been explained on the basis of electrical conduction due to a combination of flow of ions present and the conductive carbon fibers in series present in the porous matrix of the cement. *Copyright © 2012 IFSA.*

Keywords: Multiwalled carbon nanotubes, Portland cement, Composite, Sensor, Smoke detection.

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1. Introduction

Recent advances in the area of smart materials have promoted the development of smart sensing composites that could improve the way stress/strain; crack and damage in structural components are detected for structural health monitoring (SHM). While several smart materials are under investigation, researchers have shown a particular interest in developing cement and concrete based nanocomposites [1-5]. Cement being the basic material for all construction works, has been considered as the most appropriate candidate for developing civil SHM sensors. Both the chemistry that forms cement hydration products and the physical behaviour of these products can be manipulated through nanotechnology. One of the goals of research in the area of cement/nanocomposites is to fabricate stronger and tougher concrete products by reinforcing with nano-particles/nano-fibers/nanotubes [6, 7].

Carbon nanotubes (CNTs) are being considered as one of the most promising fillers for fabricating smart and multifunctional composites due to their excellent mechanical, electrical, electromechanical and other physical properties. In addition to their high strength and elastic constant, CNTs have extremely high aspect ratios with values typically higher than 1000:1 and reaching as high as 2,500,000:1 [8]. The size and aspect ratios of CNTs provide reinforcement at a much finer scale than commonly used reinforcing fibers. These attractive properties have led to considerable research on CNT composites for developing low cost, low operating voltage and smart sensitive composites/sensors as candidates for aerospace structures, biomedical and environmental engineering applications and especially for SHM [9-16]. CNT/cement based nanocomposites display piezoresistive properties and therefore can be used to sense stress/strain or deformations by monitoring their electrical resistivity [17]. Thus, CNT/cement composite sensors are also known as intrinsic smart (self-monitoring) structural materials for monitoring structural health and can be used as a part of these structures. Here, we present a new approach for fire safety monitoring of infrastructures by sensing smoke with multiwalled carbon nanotube reinforced cement nanocomposite pellets.

Within the fire safety community, it is clearly recognized that two smoke alarm technologies with distinct operating characteristics exists: a photoelectric type smoke alarm and an ionization smoke alarm. Some of the recent inventions have related use of carbon-based nanostructures in detecting smoke. In a patent by John Edward [18] it has been described that carbon nanotubes based sensor, indicative of a potential fire, responds to the presence of a predetermined gas. Whereas, in another smoke detector patented by Richard Lee [19], the invention describes that a field emission device using carbon nanotubes in turn replaces the americium source of alpha particles of a standard ionization-type smoke detector.

In this paper we discuss a new application of MWCNTs reinforced Portland cement i.e. smoke detection. Cement is well known for its ionic conductivity because of the ions (such as Na^+, Ca^{2+}, OH^-, etc.) present within the pores of cementious material. However, MWCNTs are electronic conductor (with the charge carriers being electrons and/or holes) [20, 21] rather than an ionic conductor. The charge carrier mechanisms that are responsible for electrical conduction in carbon fiber/nanotube reinforced matrices have been explained by S. Wen [22], McCarter [23, 24], S. Wansome [25] and Xie [26]. These fibers create electrical conductivity and piezoresistivity in the material that is of major importance in the development of cement-based sensors. The addition of even a small amount of carbon fibers/nanotube to cement paste significantly reduces the resistivity of the material. Conductivity boosts by several orders of magnitude till the volume fraction of carbon fiber reaches a certain critical value, referred to as the percolation threshold. The electrical conduction is due to a combination of flow of ions present in the cement and the conductive carbon fibers in series with the cementitious material.

Based on these theories, we have developed a sensor for sensing smoke with pellets of nanocomposite of MWCNTs reinforced Portland cement. To better understand how material composition and microstructural modifications participate in the sensing mechanism, characterization techniques such as scanning electron microscopy (SEM) and Fourier Transform Infrared (FT-IR) spectroscopy were performed. DC transient studies and AC impedance spectroscopy under ambient and smoky environment were performed to establish conductivity mechanism in the pellets. The paper also develops a theory for correlating the sensitivity of the pellets toward smoke with existing theories. The MWCNTs/cement nanocomposite pellets provide a simple, durable and cost effective alternative to conventional smoke sensors. By surface mounting or embedding a network of these sensors in a structure and continuously acquiring electrical response, the performance of the structure can be accurately monitored for smoke detection.

2. Experimental

2.1. Sample Preparation

The cementitious material used in this study was ordinary Portland cement purchased from Ashoka Group Ltd. India. The MWCNTs (outer diameter=10-15 nm, inner diameter=2-6 nm, length = 0.1-10 m, purity > 90 % & density=1.7-2.1 gm/cm^3) were procured from Sigma-Aldrich, USA. The surfactant used for dispersing the MWCNTs, sodium dodecyl sulfate (SDS) was procured from Merck India Ltd. Commercial grade conductive silver paste was used for fabricating electrical connections.

For the fabrication of MWCNTs/cement nanocomposites, mg quantities of SDS were stirred for 30 min with sufficient distilled water to achieve effective dispersion of MWCNTs. MWCNTs (maintaining different concentrations of MWCNTs by weight of cement i.e. 0 %, 0.17 %, 0.34 % and 0.68 %) were added to the aqueous solution and again agitated for 30 min to suspend MWCNTs into the aqueous solution. 20 gm of Portland cement (maintaining water/cement ratio (w:w) as 0.6:1) was added into the aqueous solution of SDS and MWCNTs. The mixture was then ultrasonicated and stirred magnetically alternatively for 2 hr to achieve a uniform dispersion of the components. Pellets of the composite (MWCNTs/cement) were casted using acrylic plastic moulds into cylindrical shapes (outer diameter = 3.5 cm, inner diameter = 3 cm and height = 1.5 cm). All specimens were de-moulded after 96 hrs of casting. The as cast pellets were kept for curing at room temperature under 100 % relative humidity conditions for 2 weeks. The final dimensions of the solid cylindrical pellets obtained were the following: diameter=1.5 cm, height=1.5 cm, volume =10.6 cm^3 and surface area=28.26 cm^2. Thereafter, they were cured at an ambient temperature of 30°C for a day before testing. For electrical ohmic contacts, thin copper strips were pasted on the circular surfaces of the cylindrical pellets using high grade conductive silver paste. Two insulated wires were soldered onto copper strips for external electrical connections. The final structure of the obtained pellets is shown in Fig. 1.

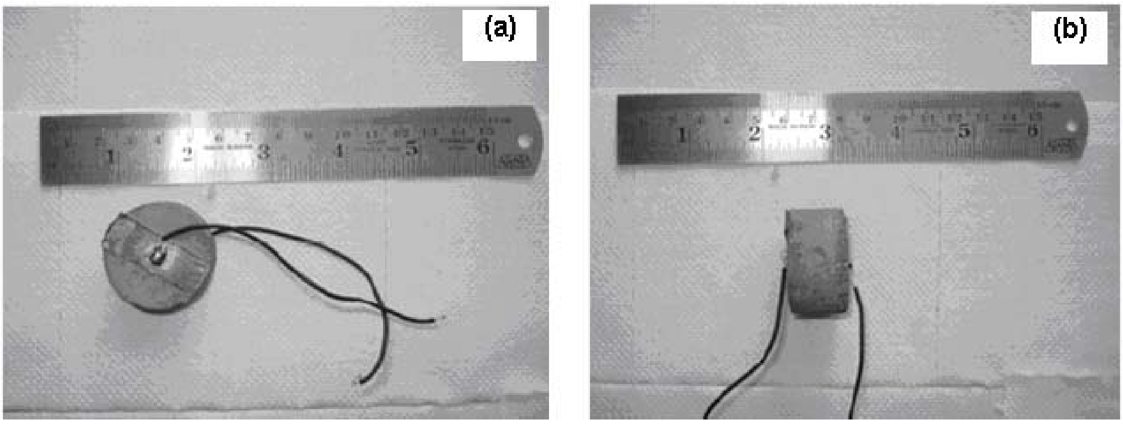

Fig. 1. Top (a), and side (b) views of MWCNTs reinforced cement pellet.

2.2. Fourier Transform Infrared (FT-IR) Spectroscopic Characterization

FT-IR studies were performed to analyze the chemical interactions between MWCNTs and cement. Bruker, ALPHA FT-IR spectrometer was used to measure the FT-IR-spectral response of the pristine cement as well as MWCNTs/cement composites in the range of 800-3100 cm^{-1}.

2.3. Scanning Electron Microscopic Characterization

Morphological and micro-structural studies were performed with Scanning Electron Microscope (SEM) model Carl Zeiss EVO40 at 20 kV. Powdered samples prepared with different concentrations of MWCNTs with cement slurry were coated onto conductive carbon tape and gold-coated before examination.

2.4. Smoke Generation Method

For the detection of smoke using MWCNTs/cement composite pellets, a vast number of combustible materials were selected to generate smoke such as paper, cloth, wood, cotton, organic solvents, vehicular emissions etc. The present study reports the data obtained by the combustion of paraffin oil of molecular weight 325 gm/mol and density 0.8 gm/cc. In a 500 ml closed beaker, 30 µl of paraffin oil was heated to 200 °C for 10 min. The paraffin oil was burnt completely and the smoke produced by its complete combustion was collected in the same closed beaker. Thereafter, the smoke was released into a testing chamber. It was observed that the testing chamber was uniformly filled with smoke before recording the real-time data.

2.5. Experimental Set-up

2.5.1. DC Transient Electrical Measurements with Smoke

The principle of detection was based on measuring changes in the electrical resistance of the composite pellets when exposed to smoke in a two-pole format. The electrical resistance of the composite pellets was determined using Keithley 6514 Electrometer interfaced with a computer, as illustrated in Fig. 2. The experiments were performed several times to establish the reproducibility of the data. Similar responses were obtained with ±3 % variation over the several test runs.

The Responsivity S, of the MWCNTs/cement composites to smoke was calculated in percentage using the following equation [27]

$$S = \left[{\left(R_f - R_0 \right)} \big/ {R_0} \right] \times 100, \tag{1}$$

where R_0 is the initial resistance value of the composite pellet, and R_f is the steady state resistance value achieved after exposure to smoke.

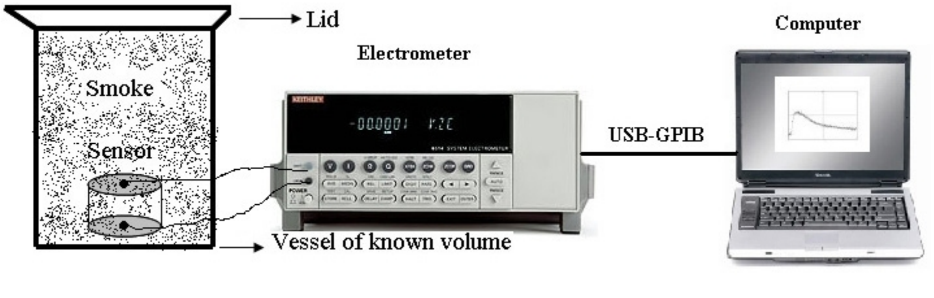

Fig. 2. Experimental setup for sensing smoke using digital electrometer interfaced with computer.

2.5.2. Impedance Measurements

The impedance characterization of the composites in the presence and absence of smoke was carried out by impedance spectroscopy technique. The measurements were performed in the frequency range 100 Hz - 2 MHz, with an applied potential of 1 V, using computer interfaced high precision Agilent E4890A impedance analyzer and high precision Agilent 16048A test lead at room temperature. The experimental arrangement is shown in Fig. 3.

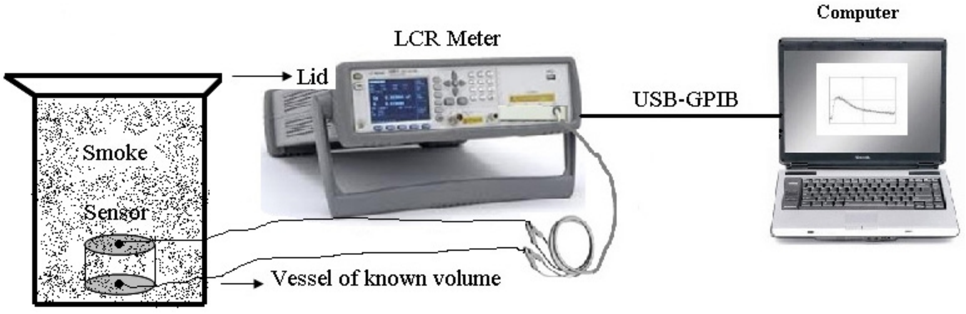

Fig. 3. Experimental setup for Impedance spectroscopy measurements under ambient and smoky environment using computer interfaced high precision impedance analyzer.

3. Results and Discussions

3.1. FT-IR Characterization of Cement and MWCNTs/cement Composite

The FT-IR spectra of cement slurry and MWCNTs reinforced cement slurry in water are shown in Fig. 4 (a) and Fig. 4 (b) respectively in the region 500-3500 cm^{-1}. The spectrum of cement slurry (slurry of Portland cement in water) exhibited the presence of bands at 3373, 1681, 1658, 1407, 1107, 997, 900 and peaks at 870 and less cm^{-1}. The bands at 870 cm^{-1} and less arose due to CO_3^{2-} ν^2 modes of calcium carbonate. Bands at 900 and 997 cm^{-1} were due to stretching and absorption bands of Si-O of C-Si-H phase respectively present in the cement. Bands at 1107 and 1407 cm^{-1} depicted the presence of sulfate (SO_4^{2-}) and carbonate (CO_3^{2-}) phases in the cement respectively. The band at 1658 cm^{-1} was due to the presence of calcium sulfate in the cement. The stretching bands due to (OH) of water and carbon hydroxyl C-OH appeared 1681 and 3373 cm^{-1} [28].

The spectrum of MWCNTs reinforced cement slurry showed appearance of bands at 3373, 2366, 1683, 1666, 1645, 1407, 1107, 991, 900 and peaks at 870 and less cm^{-1}. The appearance of new bands at 2366 and 1645 were due to C-H_x stretching and stretching of unsaturated C=C bonds respectively. The unsaturated C=C bonds stretching was associated with the stretching of the backbone of MWCNTs in the cement matrix. Covalent interactions between the MWCNTs with cement were not evident [6, 27].

3.2. Microstructural Studies

Fig. 5 (a) and Fig. 5 (b) show typical SEM micrographs of cement and MWCNTs/cement respectively. It was observed that the cementitious material in general provided a porous composition of several materials such as carbonates and silicates. SEM analysis of MWCNTs reinforced cement at low magnification, as shown in Fig. 5 (b), demonstrated that carbon nanotubes were uniformly dispersed in the matrix of cementitious material. At higher magnification it was revealed that MWCNTs acted as bridges across pores of the cementitious material. Similar observations have been reported by Li [6].

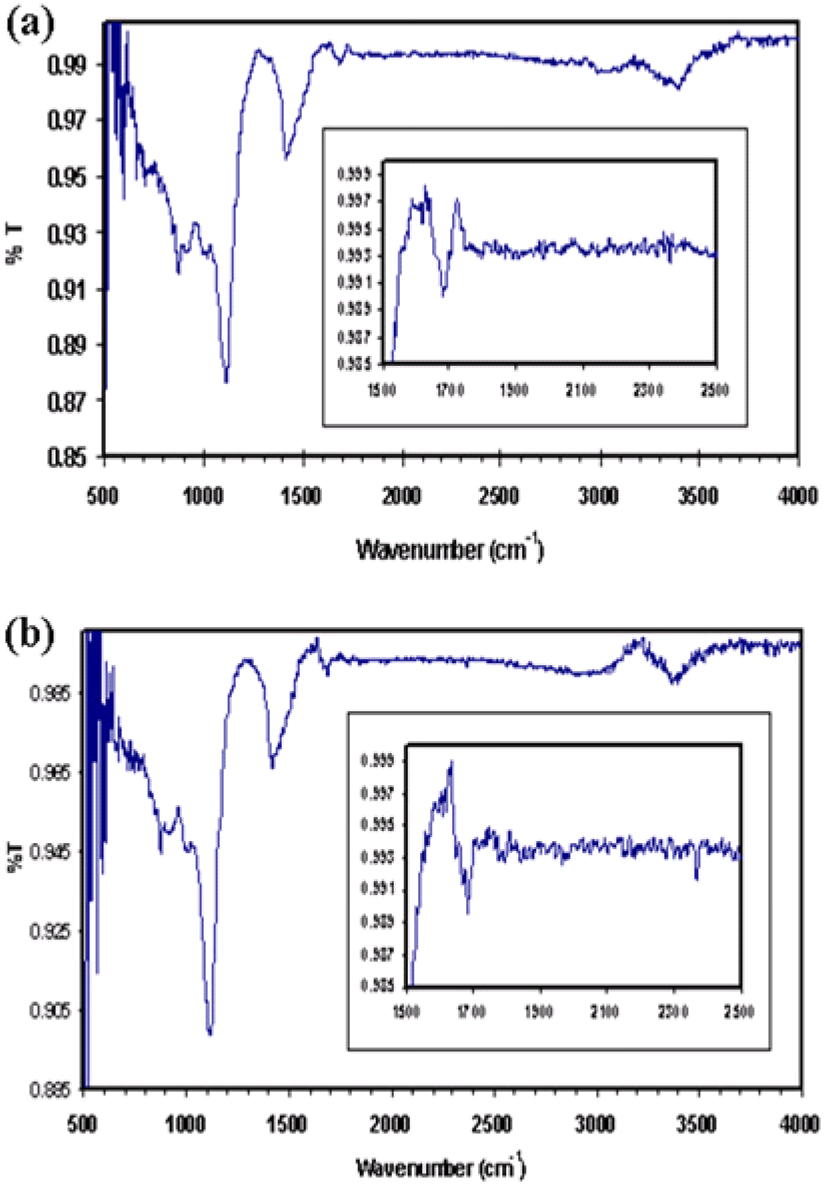

Fig. 4. FT-IR spectra of (a) cement slurry and (b) MWCNTs reinforced cement slurry in water.

3.3. DC Transient and AC Impedance Spectroscopic Characterizations

Fig. 6 illustrates typical DC transient percentage responsivity, %S, curves in smoky environment. During these measurements, it was observed that the conductivity of all the pellets, having different concentrations of MWCNTs increased. The conductivity reached to a maximum steady-state value within ~500 sec depending upon % MWCNTs in the pellets.

It can clearly be seen from the curves that initial responsivity of 10-15 % was achieved within 25-50 sec. Responsivity up to 90 % was achieved in ~ 300 sec depending on the % MWCNTs present in the composites, since the time of exposure to smoke. As the density of smoke decreased in the environment, a decrease in conductivity was observed indicating self-desorption nature of the composites. Experiments were performed several times with different lots of pellets to establish the reproducibility of the data. Similar responses were obtained with different lots, with slight variation in sensitivity; over several test runs.

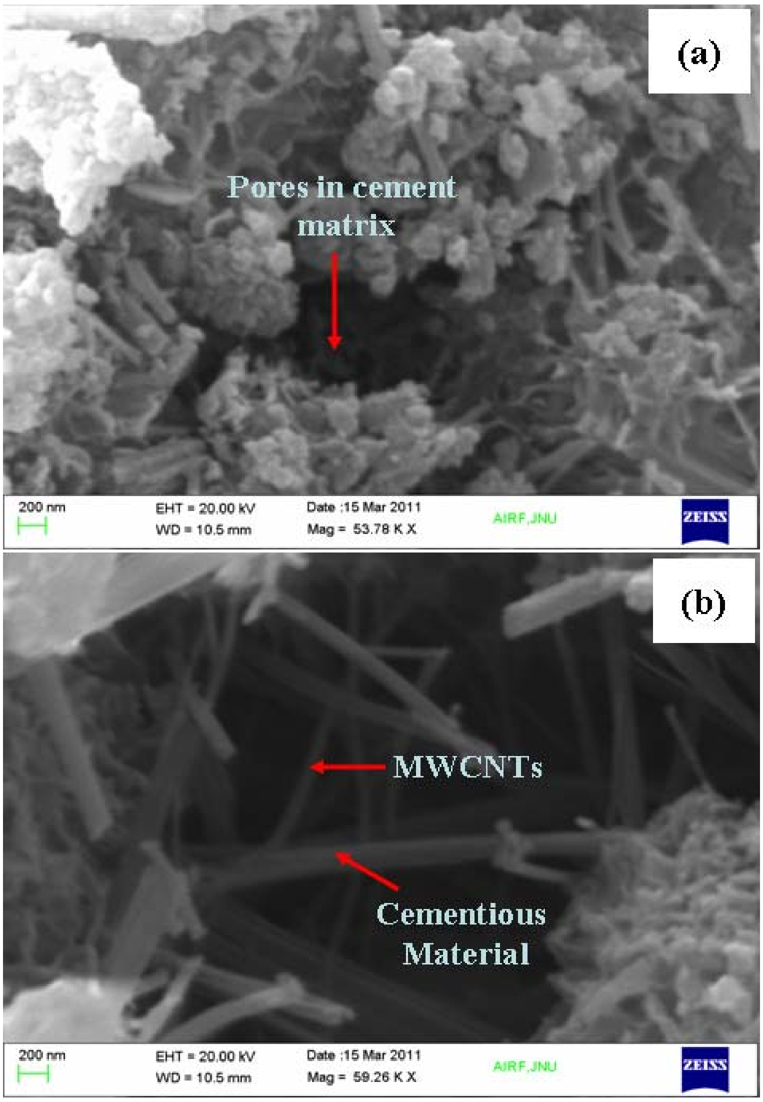

Fig. 5. SEM micrographs of (a) cement and (b) cement reinforced with MWCNTs.

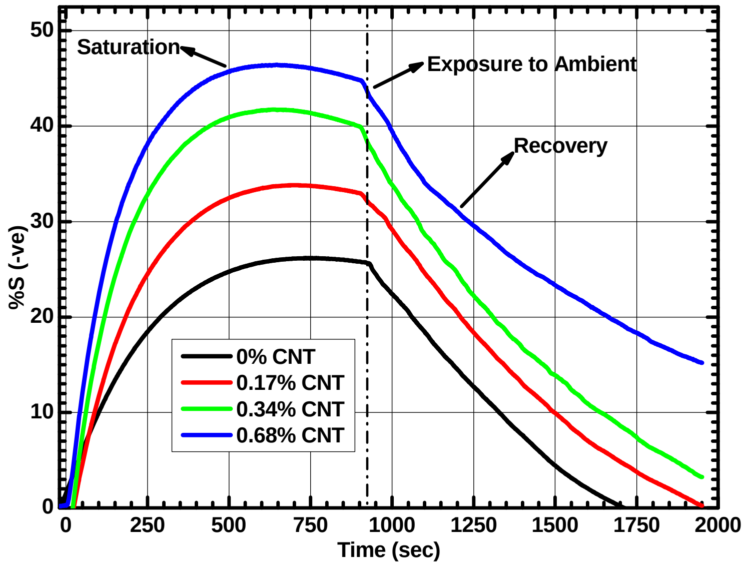

Fig. 6. Percentage responsivity of the MWCNTs reinforced cement pellets of different concentrations of MWCNTs under smoky environments.

Table 1 reports average response time, τ_{90} (sec), and average decay time, τ_{decay} (sec), for the pellets. τ_{90} is the time in which the pellets response was enhanced by 90 % of their initial value in the smoky environment and the τ_{decay} is the time in which the pellets were able to retrieve back to 90 % of their initial resistance value. It can be established from the Table 1 that although the response time for the pellets with higher % of MWCNTs is less, the recovery of these pellets was found to be slow. This observation suggests that the charged carbon particles and several ions present in the smoke interact with the MWCNTs present in the pores of the composites. Thus, as % MWCNTs increases in the composite, polar interaction becomes stronger and hence slower desorption of smoke [29]. Thus it is being concluded based on this observation and the percolation limit suggested by Xie [26]; the % MWCNTs in the composite can not be increased beyond 0.68 %, otherwise it is expected that the desorption time will increase manifolds; making such pellets impractical for real time applications.

Table 1. The average percentage responsivity, average response time τ_{90} (sec), and the average decay time τ_{decay} (sec) of different MWCNTs/cement composite pellets.

S.No.	% CNT	Average %S	Average τ_{90} (sec)	Average τ_{decay} (sec)
1.	0	26.23	373.48	788.07
2.	0.17	33.81	345.87	995.36
3.	0.34	41.72	304.99	1182.01
4.	0.68	46.46	276.31	1523.87

Fig. 7 shows the frequency response of ionic conductivity of MWCNTs reinforced cement pellets under smoky and ambient environments for different concentrations of MWCNTs. A gradual increase in the ionic conductivity with frequency was observed for all the samples. This increase in ionic conduction was also found to be assisted by the increase in concentration of MWCNT in the composite. The universal dynamic response of ionic conductivity of materials is generally described by a power law given by Jonscher [30, 31] as $\sigma(\omega) = \sigma_{dc} + A\omega^s$; where σ_{dc} is the limiting zero frequency conductivity, A is a pre exponential constant and s is the dimensionless frequency exponent parameter in the range $0 \le s \le 1$. For an ideal Debye dielectric 'n' is approximately 1 and for ionic-type materials 's' tends to 0.

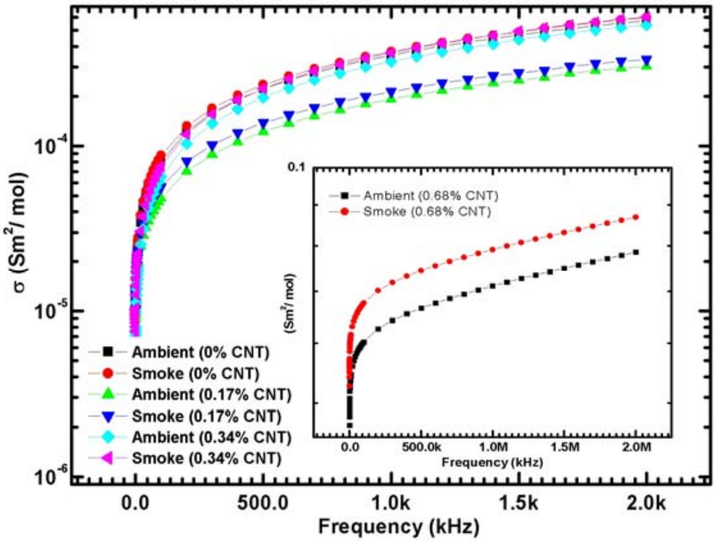

Fig. 7. Frequency dependent ionic conductivity curves for MWCNTs reinforced cement pellets under smoky and ambient environments for different concentrations of MWCNTs.

Further, the dc conductivity is due to the long-range movement of free charges present in the sample and the frequency dependence of the ionic conductivity is generally explained by a hopping conduction mechanism. Fitting of curves using the above equation to the experimental data provided the values of 's' in the range 0.28-0.14. A decrease in the value of 's' suggested an increase in the ionic nature of the composite with an increasing concentration of MWCNTs in the composite. Further, the enhancement in ionic conductivity, when the samples were exposed to smoke was also found to increase with increasing MWCNTs concentration in the samples suggested an increase in the sensitivity towards smoke with increase in the concentration of MWCNTs in the composites.

The above discussed Ac and DC electrical characteristics can be explained based on the following theory. In a cementitious material electrical current results from the flow of free ions such as Ca^{2+}, Na^+, K^+ or OH^- through the porosity of the material. In conductive fibers and MWCNTs, conduction is through the motion of free electrons. Thus, in the composites of the cementitious material containing CNTs, the electrical conduction is mainly due to a combination of flow of ions and electrons. Depending on the CNTs content and various other internal parameters, this combination may include three possible conduction pathways in CNTs reinforced cement composites [22-26] as: (a) via the capillary pore water within the cementitious composite; (b) through a continuous network of carbon fibers; and (c) through the cement paste and the conductive carbon fibers in series.

Thus, within the percolation zone, where a network of MWCNTs within the porosity of the cementitious material has formed, the two latter paths dominate the conduction route and hence overall electrical conductivity is increased under ambient conditions. Under smoky environment that in general contains charged particles of carbon in combination with several ions further assists ionic and DC conductivity by providing enhanced ionic conduction through polar interaction with MWCNTs with the desorption limit.

5. Conclusions

Composites of MWCNTs reinforced cement were prepared with different concentration of MWCNTs. The FT-IR analysis confirmed the presence of various ionic species as well as MWCNTs bonds in the composite without any indication of chemical bonding among themselves. In addition, the SEM micrographs revealed a continuous network of conductive carbon nanotubes within the pores of the cementitious material. The electrical conduction was due to a combination of flow of ions present in the cementitious material and the conductive carbon fibers present in the pores in series. The conduction was found to enhance further under smoky environment, with responsivity higher for higher concentration of MWCNTs, due to ions presents in smoke. Further, mounting or embedding these smoke sensitive pellets in civil structures would certainly be proved to be an asset for smoke sensing applications.

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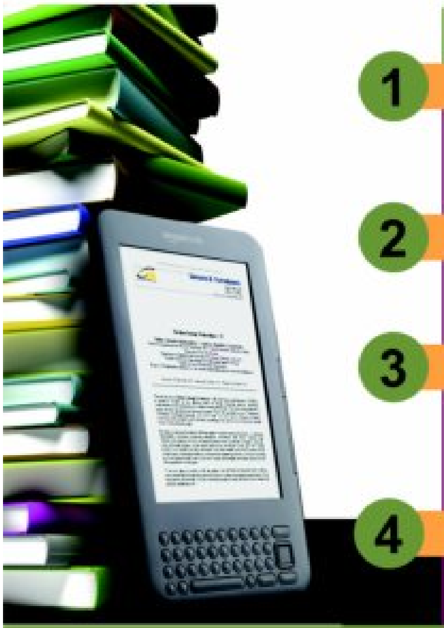

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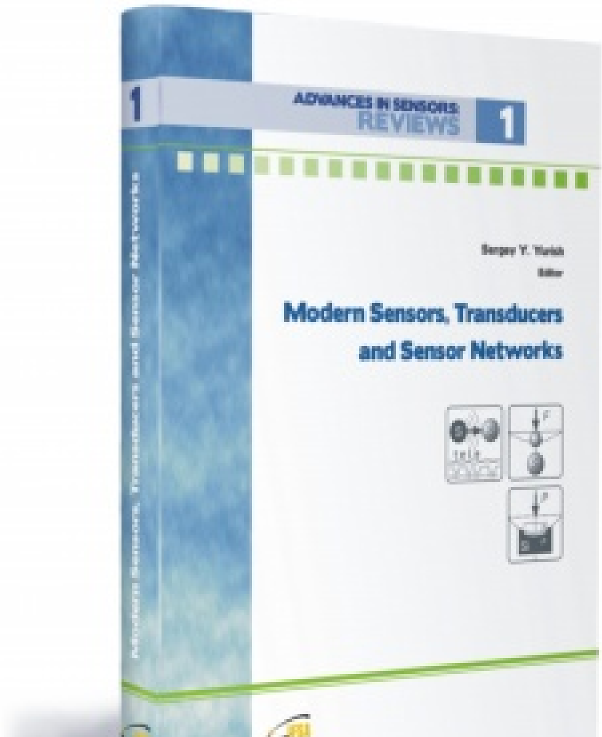
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