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Electromagnetic shielding performance of graphite in cement matrix for applied application

V. K. Sachdev¹*, S. K. Sharma¹, S. Bhattacharya¹, K. Patel², N. C. Mehra¹, V. Gupta¹, R. P. Tandon¹

¹Department of Physics and Astrophysics, University of Delhi, Delhi, India ²Department of Electronic Science, University of Delhi, South Campus, New Delhi, India

*Corresponding author. E-mail: vk_sachdev@yahoo.com

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ABSTRACT

Growing electromagnetic pollution caused by the rapid proliferation of sophisticated electronic devices has increasingly invited concerns. Cement based composites are commercially established in constructions, also capable of electromagnetic interference shielding besides other multifunctional utilities. Cement matrix a key ingredient in mixes is considered to be the best adhesive that holds together the aggregates. In this work these composites filled with conductive graphite particles were prepared using dry tumble mixing modus operandi followed by compression and subsequent curing with water. Prolong tumble mixing ensures even dispersion of constituents while compression develops a stronger bond to the aggregates besides patterning of graphite particles in cement matrix. This patterning of conductive graphite particles is vital for producing enhanced electrical and dielectric properties. Effects of increasing graphite concentration on electrical properties and electromagnetic interference shielding effectiveness in X band were investigated. The cement with 20 wt% graphite shows an enormous electrical conductivity 2.74×10^{-1} S/cm with a significant shielding effectiveness of 76 dB. Both are ascribe to graphite network formation in the cement matrix. Reflection and absorption of EM radiation increases with the rise of graphite level in cement composites. The contribution of absorption to the total SE of the system is found to be larger than that of reflection. Permittivity of compressed graphite/cement composite > compressed cement > uncompressed cement has been ascertained. Studies revealed that compression pressure and inclusion of graphite, both affect the porosity of cement by reducing the pore volume. Copyright © 2015 VBRI Press.

Introduction

Electromagnetic interference (EMI) has become a major problem as a consequence of rising sensitivity of electronic products and telecommunication equipment. Electromagnetic (EM) radiations interfere with digital devices (particularly in radio frequency range) and directly affect public health due to their adverse consequences on human beings [1-3]. There are guidelines from International Commission on Non-Ionizing Radiation Protection and IEEE for limiting exposure of EM radiations [4, 5]. For reducing or eliminating the effect of EM waves, there are two common methods. The first method uses a shielding barrier, which reflects the EM waves, while in the second method the shielding barrier absorbs the incident waves [6]. EM wave absorbing material is known to be safer for solving such problems, since the reflecting material can act as an antenna [7] that causes malfunction in electronic equipments. Additionally, wave-absorbing materials have added application in reducing radar signature, i.e. stealth technology. EM shielding has been considered necessary for underground vaults containing transformers and other electronic paraphernalia related to telecommunication and electric power generation [8]. Shielding and wave absorbing compartments can be built on devices for averting EM interference and direct

exposure to human beings. Cement is one of the most commonly used structural materials, has rich resources and good ecological compliance. Additionally, it is inexpensive and a superb adhesive which can unite other building materials together. Cement when combined with water, set and harden as a result of chemical reactions. After hardening it retains strength and stability even under water. Cement is faintly conductive and its EM shielding/waveabsorbing properties are inadequate [9]. The simple and practical method to enhance its shielding capability is through inclusion of some conductive filler. Guan et al. [10] have referred some investigations on fillers in cement matrix. Majority of the fillers are metal powders, fibers or ferrites. Nearly all are either expensive or have relatively complex processing technology. Metal powder has a disadvantage of high density. For better performance, its increased content is at the cost of raised material density. In cement with 5 wt% nano TiO₂, a reflection loss of 8-16 dB is reported in 8-18 GHz frequency range. But its further addition depreciated the reflection loss. Through steel fiber in concrete, EM reflection loss of 9.8 dB in 2-18 GHz frequency range by a 31 mm thick sample is pretty low. Ferrite materials have shown good reflection loss at lower frequency but they also suffers from the disadvantage of high bulk density and their EM absorbing performance deteriorates considerably in higher frequency bands [11]. Stainless steel fibers in cement paste have shown shielding effectiveness (SE) ~70 dB with 4.47 mm thick sample [12] and coke powder in cement paste has given ~ 52 dB with 4.77 mm thick sample [8]. These values are inspiring, but are confined to 1.5 GHz frequencies.

Graphite is inexpensive conductive material and commercially available in large quantities for any product application. Investigations on graphite particles as an admixture in cement-matrix composites are related to their low electrical resistivity, elevation in thermoelectric power, high thermal conductivity, electricity generation, fuel cell bipolar plates, EMI shielding and thermal storage [13-16]. SE values of 22 dB at 1 GHz and ~26 dB at 1.5 GHz for 4.40 mm thick sample of graphite/cement have been reported [17]. These shielding are primarily by reflection. The method of processing graphite in cement seems less effective for SE compared to their addition of stainless steel fibers or coke powder in cement paste.

Addition of graphene oxide (30 wt %) along with ferrofluid in the cement have shown SE of 46 dB in 8.2-12.4 GHz frequency range with 3.25 mm thick sample of hardness ~ 38 [18]. The graphene oxide has been synthesized by way of laborious oxidation and exfoliation of natural graphite, besides the composites prepared show the apathetic mechanical properties for application in large building structures.

Attention has been paid to the direct incorporation of exfoliated graphite in cement [19, 20]. The method of processing for exfoliated graphite/cement composites consist of dry mixing of exfoliated graphite and cement, followed by compression and subsequent curing results in a exfoliated graphite network. Composites with networked exfoliated graphite are found to be much superior to distributed exfoliated graphite (wet cement mixing) in terms of lower electrical resistivity, higher compressive strength and superior vibration damping performance [21]. Fabrication of intercalated graphite is a time-consuming chemical indulgence. Additionally worm shape exfoliated graphite heating at ~ 1050°C.

In present work the graphite used in processing for cement composites is cost-effective and easily available in large amounts. Additionally processing method is similar to cement-matrix composites with exfoliated graphite [19-21] for creation of graphite network. The composites were prepared through simple tumble mixing of dry powders of graphite and cement with succeeding compaction under pressure, then treated and cured with water. This technique also results in a graphite network in cement matrix. On compaction under pressure in absence of any shear, the conductive graphite particles are set in amid cement matrix. The specific arrangement of graphite particles within the fairly insulating cement matrix contributes towards improvement of electrical and dielectric properties of the composite. This procedure is looked to be more effective for such composites. Both reflection and absorption of EM radiation had been anticipated to go up with increase of graphite filler [15]. In present work the best of SE obtained is ~ 76 dB at 11.5 GHz, with only 2.29 mm thick sample of graphite/cement composite. In case of such composites, the electrical conductivity is originated due to formation of an interconnected network of graphite particles in the cement

matrix. The conductivity is often found to increase with the increase of filler content and saturate or randomize beyond percolation threshold [12, 22]. The reason of saturation of conductivity can be attributed to the formation of a near-perfect network of conducting pathways through the composite. Addition of more graphite is expected to increase only the dimension of the pathways as in polymer composite [23], whereas the number of pathways remains the same. The conducting particles in the pathways were assumed to be touching each other [24]. But in real systems, energy barriers always separate the filler particles and electrons need to hop/tunnel through the barriers for conduction [25]. Hence, the conductivity of the composite can never be achieved equal to or even nearly equal to the conductivity of the filler.

Since cement has a very high resistivity compared to the graphite, permittivity here is related to Maxwell-Wagner type of polarization that occurs in heterogeneous dielectrics. Water treatment to such composite binds the different constituents together and form lumps [26]. These lumps are believed to be consisting of cement matrix embedded with conducting graphite particles. Scanning electron microscopy (SEM) of such composites has imaged the placement of graphite within the interfacial places [26]. Structural changes with increasing graphite are obvious. Cement pellets are porous in structure. The compaction pressure is expected to decrease the pore size volume, which in turn increases the permittivity of composites. Addition of graphite anticipated to place the graphite particles in pores, reduces the pore volume further thus results in more increase in permittivity.

Experimental

Materials

White cement was procured from Birla White India. It is available commercially, used for prestigious construction projects and decorative work. White cement is similar to ordinary gray cement almost in all respects except color. The chemical composition of good ordinary cement is normally lime, silica, alumina, calcium sulphate, iron oxide, magnesia, sulphur and alkalies. The characteristic gray color of ordinary cement derives from a number of transitional elements such as manganese and iron in its chemical composition. The amount of these ingredients probably has been minimized in white cement as far as possible. True reason for white color is a trade secret. Perhaps it's processing made it exceptional compared to gray cement. Graphite India Limited supplied the graphite powder.

Sample preparation

In conventional method of sample preparation, homogeneous dispersion of the graphite particles in the cement paste is difficult. When mixed in cement paste, the graphite floats onto the surface of the paste and hence causes segregation. While by means of method of preparation [15] used here, graphite and cement powders were tumbled mix for 200 minutes for meaningful dispersion. Cement and graphite particles gather into clusters. SEM micrograph of 3 wt% graphite in cement is

showing their clusters in Fig. 3. The resulting cement/graphite composite powder was compacted to requisite rectangular waveguide shape of X band in a piston-cylinder assembly at 75 MPa for 15 minutes and subsequently treated with water. The pellets of all compositions were cured for about 100 hrs in 100% humidity. After curing, the pellets were left for natural drying at atmospheric temperature (~ 25 °C) for 28 days. A series of specimen pellets were produced by varying the filler concentration from 0 - 20 wt %. For each composition five pellets were prepared. The area of each sample was $22.86 \times 10.16 \text{ mm}^2$ (size of X band waveguide). Table 1 illustrate the thicknesses of five samples of each cement composite with 0, 2, 3, 4, 5, 6, 10, 15 and 20 wt % graphite content. Here 0, 2, 3, 4, 5, 6, 10, 15 and 20 wt % graphite in terms of volume fraction refer to 0, 2.74, 4.10, 5.45, 6.78, 8.11, 13.32, 19.62, 25.69 volume %, respectively.

Table 1. Thicknesses of five samples of each composition.

Gr (wt%)	t (mm)				
0	1.89	2.42	2.84	2.49	2.97
2	3.99	2.43	2.64	2.14	2.75
3	2.67	2.35	2.66	2.72	1.73
4	2.22	1.83	2.40	2.70	2.76
5	2.63	1.74	2.62	3.20	2.79
6	3.21	2.93	2.74	1.64	2.98
10	3.21	3.12	1.74	1.94	2.94
15	2.30	2.91	2.52	1.84	2.59
20	1.80	1.73	3.00	3.10	2.29

Material characterization

The density and conductivity of graphite were 1.75 g cm^{-3} and 1.33×10^4 S cm⁻¹ respectively as quoted by producer. SEM micrograph [23] of graphite particles has revealed their flake like shape. Particles with ~1 µm size are noticeable in micrograph. Fig. 1 showing a SEM micrograph of cement powder illustrates its size and shape, observed estimated particle size is $>2 \mu m$.



Fig. 1. SEM micrograph showing the size and shape of white cement particles.

EDX analysis on the cement powder gives the elemental composition of 64.89 at% O, 23.39 at% Ca, 6.89 at% Si,

2.1 at% Al, 1.01 at% S, 1.55 at% Mg and 0.16 at% Fe elements (Fig. 2). Traces of transitional elements Mg and Fe confirm the conjecture for its white color.

The morphology of cement with 3 wt% graphite additive is shown in Fig. 3.



Fig. 2. EDX analysis of cement powder.

The SEM micrograph displays cluster or aggregate, which is different from pure cement morphology by its flaky appearance.



Fig. 3. SEM micrograph showing the clusters of cement- graphite mixture.

Elemental composition in EDX study on a cement cluster with 15 wt% graphite additive reveals the presence of carbon in the selected area (inset Fig. 4) of same order as of graphite. This endorses the uniform dispersion of graphite particles with in the cement matrix.

Electrical and dielectric measurements

These measurements of samples were carried out in sandwich geometry. Fig. 5 shows the schematic setup for measurements. A purposely-designed sample holder cell was used. Spring loaded copper probes were finely coated with silver.



Fig. 4. EDX analysis of cement powder with 15 wt% graphite content.

Both surfaces of the specimen were layered evenly with SEM grade silver paint. For measurements of electrical resistance lower than 200 M Ω , a digital multimeter and for greater than 200 M Ω , a Keithley electrometer was employed. The resistance of each pellet at a given temperature was measured within first 10 seconds, since polarization of samples supposed to be negligible during this initial period. All values of conductivity of composites reported in this work were of direct current. Programmable automatic RCL meter PM 6306 Fluke having frequency range 50 Hz - 10⁶ Hz was employed for dielectric measurements. The measurements of extra conductive composites were beyond the limit of this equipment.



Fig. 5. Schematic diagram for Electrical and dielectric measurements.

Electromagnetic shielding measurements

For shielding measurements, Vector Network Analyzer (VNA) Wiltron 37247B was used. A schematic diagram for VNA based measurements is shown in Fig. 6. An X-band waveguide sample holder was connected to Port 2 of VNA using coax to waveguide adaptor. VNA was calibrated using X-band waveguide calibration kit for Offset Short-Short-Load technique. Insertion loss (IL) and return loss (RL) of the components between the test ports were measured in terms of scattering parameters. The initial reading of IL and RL were noted which were less than 0.01 dB range. Then the specimen sample was inserted into the sample holder and the final values of IL and RL were recorded. The IL of the specimen sample was obtained from the difference between initial and final values. This was considered as EMI SE of the sample. Similarly, the measured RL was obtained by means of difference between

initial and final values of RL. The observations were repeated 5 times to compute the random components of uncertainty i.e. standard deviation of mean value. Similar measurements were performed for each sample of cement composites with 0, 5, 10, 15 and 20 wt % graphite content. The composites were accurately inserted in the wave guide to fill the entire cross-section, so as to avoid any leakage of EM radiation. The SE measurements were executed on five specimens for each type. During insertion of the samples inside the holder, some samples were cracked. The estimated total uncertainty covers the dispersion/spread of the IL and RL values as obtained by remaining samples for each type. Two types of uncertainties were considered to estimate the total uncertainty, standard deviation of mean value for 5 readings of same sample and uncertainty of VNA on measuring the insertion loss value.



Fig. 6. Schematic set up for VNA based measurement.

Porosity measurements

Gas sorption (both adsorption and desorption) is the most popular method for determination of porosity of dry solid materials. Nova High Speed Gas Sorption Analyzer 2200 interfaced with Nova-Win software was employed for determination of porosity of uncompressed, compressed cement samples and compressed sample of 3 wt% graphite cement. Nitrogen (N₂) gas was used as adsorptive. N₂ adsorption at 77 K and at sub-atmospheric pressures is usually best and can be utilized for investigation of new materials. Sample holder stem cell of 12 mm outer diameter which can comfortably admit large sample pieces, was chosen for carrying out tests. The cell sets supplied with the NOVA Analyzer were pre-calibrated.

Uncompressed sample was prepared by molding cement paste at atmospheric pressure, while the pellets of compressed cement and tumble mixed 3 wt % graphitecement powders were compacted at optimized pressure of 75 MPa for 15 minutes and then treated with water. These sample pellets were cured under similar conditions in presence of 100% relative humidity for 100 hrs and dried at atmospheric temperature for 28 days alike other graphite/cement composites. The pellets were freeze fractured and a small fragment of the sample measuring ~ 1 mm dimension was taken and inserted into sample holder for pore size analysis. Three samples of each uncompressed cement, compressed cement and compressed cement having 3 wt % graphite concentrations were studied.

Results and discussion

Effect of graphite loading on conductivity of shielding composites

The variation of dc electrical conductivity σ of cement composites (at room temperature) as a function of volume fraction of graphite content is illustrated in **Fig. 7**. Small data scattering in values of σ of five pellets of each composition demonstrate even dispersion of graphite into the cement. σ increases rapidly by ~ 4 orders for the specimen with 2 wt% graphite content followed by an overall increase of ~ 7 orders with 5 wt% graphite, suggesting percolation threshold at 2 wt%.



Fig. 7. Variation of conductivity as a function of graphite content in cement composites.

The drastic change in the σ by several orders is believed to have originated due to formation of network of conducting pathways of graphite particles across the composite. At low weight percentage of graphite (<2 wt %), graphite particles embedded in the cement matrix are secluded from each other by the insulating cement surrounding them. Hence, the graphite particles do not contribute significantly towards the overall σ of graphite/cement composites, and resulting σ of the composites shall remain almost the same as that of the constituent cement phase. Near percolation threshold, the graphite particles are close enough to allow the electrons to conduct across gaps between them [27]. At a certain level of graphite (>2 wt %), the sudden large value of σ clearly expresses the formation of a conductive network of such graphite particles in the matrix. Existence of this network allows a very high percentage of electrons to flow through the interconnecting conductive paths in the matrix. For composition higher than that of the graphite fraction ~10 wt %, the conductivity levels off. Away from the percolation limit, only the dimension of conductive paths increases which does not improve the overall conductivity appreciably [23-25]. Hence, at 20 wt % concentration, the flattened conductivity facilitates the unrestricted movement of electrons. This composite has exhibited electrical conductivity of 2.74 x 10⁻¹ S/cm.

The conductivity behavior in these composites has already been made known by power law model [15]. The higher value of critical index "t" was attributed to the possibility of composites utilizing a multiple percolation or "percolation-within-percolation" approach to achieve conductivity because of the composition of cement. Percolation-within-percolation approach has been suggested for materials, which exhibit critical exponents greater than in single percolation systems [28].

Effect of graphite concentration on EMI SE

SE is a measure of attenuation of EM signals after a shield is introduced. Table 2 and Table 3 illustrate the precisely measured values of SE and RL over the frequency range of 8-12 GHz for cement composites with graphite content between 0 - 20 wt percent. SE rises with increasing graphite concentration in composites. It increases sharply beyond graphite loading of 5 wt% at all frequencies. Composite containing 5 wt% of graphite shows a SE of ~10 dB while ~76 dB is noticed for 20 wt % of graphite at 11.5 GHz frequency. This can be explained by noting that the graphite particles become closer to each other with its increase in content. When more graphite particles are dispersed in the matrix, a high value of SE is observed due to the formation of network of graphite pathways with exceptionally close gaps. EM waves are likely to encounter with more graphite particles in the cement matrix at higher concentration. These particles reflect or absorb more radiation as compared to cement rich areas, resulting in an increase in SE with increasing graphite concentration. SE for all compositions of graphite exhibits a marginal frequency dependency. A similar behavior of graphite in polymer composites has also been observed [29].

Table 2. SE for graphite/cement composites.

Gr (wt %)→ Freq (GHz)↓	0 SE(-dB)	5 SE(-dB)	10 SE(-dB)	15 SE(-dB)	20 SE(-dB)
8.0	6.21	11.63	41.18	49.80	59.99
8.5	7.29	12.47	46.35	52.70	67.06
9.0	5.86	10.91	47.38	52.62	65.71
9.5	6.58	11.07	49.57	55.32	70.69
10.0	5.37	09.76	50.84	55.24	74.71
10.5	5.28	09.43	50.79	54.60	69.52
11.0	6.67	10.45	54.54	54.15	68.12
11.5	5.91	09.53	57.25	55.10	75.71
12.0	7.96	10.65	59.49	54.37	70.79
Measurement ->	0.06	0.12	0.58	0.72	1.40
uncertainty (±dB)					
Thickness (mm) -	2.97	2.79	2.94	2.59	2.29

The variation of RL as a function of frequency is listed in **Table 3**. These variations are small and not more than 3 dB over the whole X-band, for all compositions. It is noticeable that cement composites that have a high value of SE yield a lower value of RL. The minor frequency dependence of RL may be due to some structural effects, such as the geometrical distribution of the filler along with interaction of EM waves with graphite. Parameter RL is related to the conductivity of composites, which is linked to reflection of EM energy. Contribution due to reflection of EM energy in SE can be calculated using RL.

The radiated EM energy is mainly attenuated by absorption and reflection. As a consequence,

$$SE = SE_{ref} + SE_{abs}$$
(1)

where SE _{ref} with reflectance is expressed as [30],

$$SE_{ref} = 10\log_{10} (1-R)$$
(2)

R stands for reflectance, and is the ratio of reflected power density (Pr) to incident power density (Pi). In case of normal incidence using Fresnel equation,

$$\mathbf{R} = \mathbf{Pr} / \mathbf{Pi} = \mathbf{Anti} \log_{10} (-\mathbf{RL}/10)$$
(3)

Table 3. RL for graphite/cement composites.

Gr (wt %)→ Freq (GHz)♦	0 RL(-dB)	5 RL(-dB)	10 RL(-dB)	15 RL(-dB)	20 RL(-dB)
8.0	3.52	3.80	2.68	2.04	2.12
8.5	1.64	2.37	1.47	0.99	1.01
9.0	2.23	3.68	2.01	1.32	1.17
9.5	2.23	3.69	1.97	1.38	1.32
10.0	3.62	4.75	2.16	1.70	1.47
10.5	3.90	5.10	2.15	1.64	1.45
11.0	3.29	3.87	1.54	1.19	1.07
11.5	4.89	5.40	2.23	1.72	1.41
12.0	3.04	3.80	1.87	1.56	1.40
Measurement -	• 0.04	0.06	0.08	0.08	0.08
uncertainty (±dB)					
Thickness (mm) -	→ 2.97	2.79	2.94	2.59	2.29

Effect of graphite concentration on SE ref and SE abs

Evaluated values of SE _{ref} as a function of frequency in cement/graphite composites are displayed in **Fig. 8**. Curves exhibit frequency dependency of less than 3 dB for different filler concentrations. Contribution of SE _{ref} in total SE increases with graphite content in the composites. For 20 wt% graphite, upper limit of assessed value is 6.6 dB at 11 GHz frequency. Small values of SE _{ref} indicate its small contribution to the total SE of the system. **Fig. 9** illustrates the calculated values for SE _{abs} for cement composites as a function of frequency dependence can be seen in SE_{abs} of all composition of composites, except in 20 wt% graphite.

SE_{abs} enhances with rising graphite content. Near the percolation threshold, SE abs is only ~ 8dB, which is followed by a significant value of 53 dB for 10 wt% of graphite content. Maximum SE abs of ~ 70 dB at 11.5 GHz has been noticed for 20 wt% graphite composite. This analysis clearly establishes that SE ref and SE abs both increases with increasing graphite and the input of SE abs is more than SE_{ref} in total SE. During sample processing, graphite particles get surrounded in cement matrix and assumed to show dielectric property as reported in case of carbon black [7, 31]. EM wave absorption here is related to interfacial or Maxwell-Wagner type of polarization, which occur in heterogeneous dielectrics where one component has very high conductivity than the other [32]. Graphite/cement composites have shown high dielectric constant that increases with increase of graphite content [15]. This is an important finding that determines the EM wave absorption. SEM micrographs [26] of such composites has revealed the presence of graphite particles within the interfacial places and also in lumps of particles

surrounded by somewhat insulating cement background, probably inducing the dielectric property by generating space charge polarization at the interfaces [31]. Absorption in such composite most probably occurs due to interactive loss processes of the interfacial polarization of the filler particles.



Fig. 8. $SE_{\rm ref}$ as a function of frequency in cement/graphite composites.



Fig. 9. SE_{abs} composites as function of frequency in cement/graphite.

Effect of porosity on SE_{abs}

The high value of dielectric constant of the cement/graphite composites has been influenced both by heterogeneity and porosity of the cement matrix [15] assumed earlier. Fig. 10 illustrates investigational outcome. The dielectric constant of compressed cement pellet is more than the uncompressed pellet, which increases further by addition of graphite (results here are confined to graphite concentration of 3 wt % and frequency range of 10^6 Hz due to limitation of equipment). The dielectric constant of compressed 3 wt % graphite composite is comparatively higher than the compressed and the uncompressed plain cement pellet.

The pores present within the matrix are expected to decrease the dielectric constant [33]. Cement is a micro-

porous material with electrical properties responsive to the volume of pores [**34**]. This implies that during processing, pore volume reduced on compaction that decreases further on addition of graphite. Thus, the large values of dielectric constant of cement/graphite composite can be attributed to the reduction of pore volume along with particle interactions stemming from the heterogeneous nature of the composites.



Fig. 10. Variation of ϵ $_r$ with frequency for pellet of uncompressed cement, compressed cement and compressed 3 wt % graphite/ cement composite.

The reduction in pore volume has been confirmed experimentally. Fig. 11 illustrates N_2 adsorption and desorption isotherms for pellets of uncompressed, compressed cement and compressed cement composite of 3 wt% graphite. It can be seen that there is unambiguous hysteresis loop in N₂ absorption and desorption isotherm plots, which suggests that the pore size obtained are in mesoporous range. Barret-Joyner-Halenda (BJH) adsorption summary from Nova-Win software (attached with Gas Sorption Analyzer) established that the preparation of samples under pressure has actually reduced the pore volume. (BJH) adsorption summary has revealed that total pore volume for uncompressed cement pellet is $0.062 \text{ cm}^3/\text{g}$, for compressed cement pellet $0.006 \text{ cm}^3/\text{g}$ and 0.003 cm³/g for compressed 3 wt% graphite/cement composite. This shows that the pore volume for uncompressed cement pellet > compressed cement pellet > compressed 3 wt% graphite/cement composite pellet. This means on compression the pore size volume of cement has reduced. This reduces further on addition of 3 wt% graphite to cement indicating the presence of graphite in pores.

Fig. 6 and **Fig. 7** have revealed the smaller contribution of SE_{ref} compared to SE_{abs} for all compositions of composites when EM wave transmits through them. Large absorption of wave is assigned to high value of permittivity of composites. Addition of graphite in cement enhanced the permittivity and reduced the pore volume, which is confirmed through analysis of **Fig. 10** and **Fig. 11**. This indicates the presence of graphite in pores. Graphite inside the pores probably have some more influence on its wave absorbing property, thus raising the EM absorbing performance of the composite. The pores filled with graphite can be considered as sort of tiny compartments. When the incident wave refracts through the pores, it will be reflected and scattered by the graphite inside it. As the incident wave pass on from one pore to another, the phase shift will make the incident wave interfere with the reflected wave, which also adds to the EM attenuation. Similar possibility has been referred in cement filled with expanded polystyrene composites [10].

Furthermore, incorporation of graphite has not had a significant negative impact on the inherited mechanical properties of cement [15]. Shore-D hardness had indicated their reasonable strength for product application. On inclusion of 20 wt% graphite in cement, only a slight decrease in bulk density from 2.42 to 2.30 g/cm³ has been noticed. The bulk density of the pellets referred here defines the ratio of its weight to volume, in spite of the pores inside. It was calculated precisely from accurate measurements of sample volume and its weight.



Fig. 11. N_2 absorption and desorption isotherm plots for pellet of uncompressed cement, compressed cement and compressed 3 wt % graphite/ cement composite.

Conclusion

Current work describes some important conclusions for graphite/cement composites: Reasonably high value of SE can be achieved through present processing technique compared to conventional. SE of all composites is almost independent of frequency and absorption dominated in the frequency range of this study. Earlier inference related to increase of both reflection and absorption of EM radiation with increase of graphite content has been confirmed. Large absorption of EM radiation in these composites is related to dielectric constant. Large dielectric constant is assigned to heterogeneity and porosity of the composites. The conjecture about the influence of porosity has also been confirmed through dielectric measurements and pore volume determination of uncompressed, compressed cement pellets and compressed cement composite pellet of 3 wt % graphite. In brief, investigations substantiate the prospective potential of these composites as an effective material for construction of EM shielding compartments for housing electronic equipment and deterrence of direct exposure to human being.

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Reference

- Tong, C; Advanced Materials and Design for Electromagnetic Interference Shielding; Taylor & Francais: UK, 2009. DOI: 10.1201/9781420073591.ch1
- Geetha, S.; Satheesh Kumar K. K.; Rao, Chepuri R. K.; Vijayan, M.; Trivedi, D. C. J. Appl. Polym. Sci., 2009, 112, 2073. DOI: 10.1002/app.29812
- Riadh, W. H.; Brodsky, L. M. Y., Leiss, W, Krewski, D, Repacholi, M. H. *Crit. Rev. Biomed. Eng.*, 2003, *31*, 219. DOI: 10.1615/CritRevBiomedEng.v31.i3.20
- Ahlbom, A.; Bergqvist, U.; Bernhardt, J. H.; Cesarini, J. P.; Grandolfo, M.; Hietanen, M.; Mckinlay, A. F. ICNIRP, *Health Phys.*, 1998, 74, 494.
- IEEE standard for safety levels with respect to human exposure to radio frequency electromagnetic fields, 3 kHz to 300 GHz, IEEE Std, 2005, C95.1.
- 6. Chung, D.D.L. Carbon, 2001, 39, 279.
- Kwon, S.K.; Ahn, J.M.; Kim, G.H.; Chun, C.H.; Hwang, J.S.; Lee, J.H.; *Polym. Eng. Sci.* 2002, 42, 2165. DOI: <u>10.1002/pen.11106</u>
- 8. Cao, J.Y.; Chung, D.D.L.; *Carbon* **2003**, *41*, 2433. **DOI:** <u>10.1016/S0008-6223(03)00289-6</u>
- Chung, D.D.L.; J. Mater. Eng. Perform. 2000, 9, 350. DOI: 10.1361/105994900770346042
- 10. Guan, H.T.; Liu, S.H.; Duan, Y.P.; Zhao, Y.B.; Cem. Concr. Compos. 2007, 29, 49. DOI: 10.1016/j.cemconcomp.2006.08.001
- Emerson, W.H.; IEEE Trans. Antennas Propag. 1973, 21, 484. DOI: <u>10.1109/TAP.1973.1140517</u>
- Wen, S.; Chung, D.D.L.; Cem. Concr. Res. 2004, 34, 329. DOI: <u>10.1016/j.cemconres.2003.08.014</u>
- Wen, S.; Chung, D.D.L.; Carbon 2002, 40, 2495. DOI: <u>10.1016/S0008-6223(02)00142-2</u>
- Shen, C.; Pan, M.; Hua, Z.; Yuan, R.; J. Power Sources 2007, 166, 419.
 - **DOI:** <u>10.1016/j.jpowsour.2007.01.082</u>
- Bhattacharya, S.; Sachdev, V.K.; Chatterjee, R.; Tandon, R.P.; *Appl. Phys. A: Mater. Sci. Process.* 2008, 92, 417. DOI: <u>10.1007/s00339-008-4544-9</u>
- Yuan, H.W.; Lu, C.H.; Xu, Z.Z.; Ni, Y.R.; Lan, X.H.; Sol. Energy 2012, 86, 3227. DOI: <u>10.1016/j.solener.2012.08.011</u>
- Cao, J.; Chung, D.D.L.; Cem. Concr. Res. 2003, 33, 1737. DOI: <u>10.1016/S0008-8846(03)00152-2</u>
- Singh, A.P.; Mishra, M.; Chandra, A.; Dhawan, S.K.; *Nanotechnol.* 2011, 22. DOI: 10.1088/0957-4484/22/46/465701
- Muthusamy, S.; Wang, S.; Chung, D.D.L.; Carbon 2010, 48, 1457. DOI: <u>10.1016/j.carbon.2009.12.040</u>
- 20. Chung, D.D.L.; Muthusami, S.; U.S. Patent, 8, 211, 2012.
- 21. Chen, P.H.; Chung, D.D.L.; *Carbon* **2013**, *63*, 446. **DOI:** <u>10.1016/j.carbon.2013.06.097</u>
- 22. Wang, X.; Wang, Y.; Jin, Z.; J. Mater. Sci. 2002, 37, 223. DOI: 10.1023/A%3A1013107623281
- 23. Sachdev, V.K.; Mehra, N.C.; Mehra, R.M.; *Phys. Status Solidi (A) Appl. Res.* **2004**, *201*, 2089. **DOI:** <u>10.1002/pssa.200306803</u>
- Sachdev, V.K.; Panwar, V.; Singh, H.; Mehra, N.C.; Mehra, R.M.; *Phys. Status Solidi (A) Appl.Mater. Sci.* 2006, 203, 386. DOI: 10.1002/pssa.200521101
- 25. Panwar, V.; Sachdev, V.K.; Mehra, R.M.; *Eur. Polym. J.* 2007, 43, 573.

DOI: <u>10.1016/j.eurpolymj.2006.11.017</u>

 Sachdev, V.K.; Chatterjee, R.; Singh, R.; Proc. Symp. P Int. conf. mater. adv. Technol. Singap.: Mater. Res. Soc., 33, 2007. DOI: <u>10.1142/9789812709547_0005</u>

- 27. Strümpler, R.; Glatz-Reichenbach, J.; J. *Electroceramics* 1999, *3*, 329.
 DOI: 10.1023/A%3A1009909812823
- Levon, K.; Margolina, A.; Patashinsky, A. Z.; *Macromol.* 1993, 26, 4061.
 DOI: <u>10.1021/ma00067a054</u>
- Sachdev, V.K.; Srivastava, N.K.; Kumar, K.; Mehra, R.M.; *Mater. Sci. Pol.* 2005, 23, 269.
- Hong, Y.K.; Lee, C.Y.; Jeong, C.K.; Lee, D.E.; Kim, K.; Joo, J.; *Rev. Sci. Instrum.* 2003, 74, 1098.
 DOI: 10.1063/1.1532540
- Moon, K.S.; Choi, H.D.; Lee, A.K.; Cho, K.Y.; Yoon, H.G.; Suh, K.S.; J. Appl. Polym. Sci. 2000, 77, 1294.
- 32. Paul, A.; Thomas, S.; J. Appl. Polym. Sci. 1997, 63, 247.
- Wen S.; Chung, D.D.L.; Cem. Concr. Res. 2001, 31, 673. DOI: <u>10.1016/S0008-8846(01)00475-6</u>
- Beaudoin, J.J.; Feldman, R.F.; Tumidajski, P.J.; Adv. Cem. Mater. 1994, 1, 224.
 DOI: 10.1016/1065-7355(94)90028-0

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