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Effect of MnO_2 additive on the dielectric and electromagnetic interference shielding properties of sintered cement-based ceramics

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Abstract

Cement-based ceramic pellets were prepared and their properties were studied for electromagnetic interference (EMI) shielding applications. The shielding materials were made of Portland cement with the addition of different concentrations of manganese oxide (MnO_2) up to 10 wt%. The pellets were sintered at 850 °C for 5 h and then polished prior to characterizations of density, porosity, microstructures, dielectric properties, and EMI shielding effectiveness (SE). Results show that the MnO_2–cement pellets have good dielectric properties, i.e. high dielectric constant (~300) and low dielectric loss (<0.3). The dielectric constant increased with increasing MnO_2 content in the cement matrix. The SE values of the MnO_2–cements fluctuated between 2 dB and 9 dB in the frequency range of 8–13 GHz. The sample with 10 wt% MnO_2 additive had SE values of up to 9 dB. Most of the samples with high additive concentrations produced SE exceeding 7 dB.

Keywords: A. Sintering; B. Composites; C. Dielectric properties; E. Functional applications; Electromagnetic interference shielding; Manganese oxide

1. Introduction

An electromagnetic interference (EMI) is created by electromagnetic energy which is transmitted from one electronic device to another via radiated or conducted paths or both [1,2]. For example, the power systems of laptops and computers generate broadband EMI energy. This radiated energy is propagated and picked up by television antenna, wireless remote control units, and any other power units that might cause irregular performance. In general, EMI problems can be caused by electromagnetic energy at any frequency in the electromagnetic spectrum. The electromagnetic (EM) spectrum is the range of all potential electromagnetic radiation frequencies, and generally, the electromagnetic spectrum of an object is the quality distribution of electromagnetic radiation from that particular object. Most problems are caused by energy in the radio frequency range, which extends from about 100 kHz to 1 GHz. Undesired energies which come from cellular phones are a specific form of EMI known as radio frequency interference (RFI) [1]. Besides that, researchers also report the importance of EMI shielding in relation to the high demand of the technology industry today, especially on the reliability of electronics and the rapid growth of radio frequency radiation sources. EMI prevention is in critical demand due to the interference of these radio frequency devices with digital devices and also the increasing sensitivity and importance of electronic devices [1–3].

The main mechanism for EMI shielding using conductive materials is reflection [4]. The loss (attenuation) due to reflection increases with decreasing frequency. Shielding effectiveness (SE) can be broken into three terms: reflection loss (R_{dB}), absorption loss (A_{dB}), and multi-reflections (M_{dB}). The SE of an EMI shielding material is defined in decibels (dB) and its magnitude can be written as follows [5]:

$$\mathrm{SE\ (dB)} = 20\ \log\left|\frac{E_i}{E_t}\right| = R_{dB} + A_{dB} + M_{dB} \quad (1)$$

where E_i is electric fields that are incident on the shielding material and E_t is electric fields that are transmitted through the

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shielding material

$$R_{\mathrm{dB}} = 106 + \log\left(\frac{\sigma_r}{f\mu_r}\right) \quad (2)$$

is the reflection loss caused by the reflection at the surface of the shielding material,

$$A_{\mathrm{dB}} = 20\ \log\left(\exp\left(\frac{-t}{\delta}\right)\right) \quad (3)$$

is the absorption loss of the waves as it proceeds through the shielding material,

$$M_{\mathrm{dB}} = 20\ \log\left(1 - \exp\left(\frac{-2t}{\delta}\right)\right) \quad (4)$$

is the additional effect of multiple reflections and transmissions in the interior of the shield material,

$$\delta = \frac{1}{\sqrt{\pi\mu\sigma f}} \quad (5)$$

is the skin depth of the shielding material, a distance into the shield that the incident wave propagates when the wave amplitude decays by a factor of e^{-1}; where f = frequency, μ = magnetic permeability = $\mu_o\mu_r$, μ_r = relative magnetic permeability, $\mu_o = 4\pi \times 10^{-7}\ \mathrm{H\ m^{-1}}$, and σ = electrical conductivity in $\Omega^{-1}\ \mathrm{m}^{-1}$ [6,7].

With the rise of more problems associated with electromagnetic environment pollution, the study on cement materials capable of preventing EMI has attracted great attention. This is due to the fact that a cement-based building material is not only a structural material, but may also possess EMI shielding effectiveness properties. The composites can absorb or reflect the electromagnetic waves to decrease interference phenomenon.

Cement is a material of rich resource and good environmental flexibility and is one of the most common structural materials used in engineering constructions. Cement is slightly conductive, but its EMI shielding effectiveness and wave absorbing properties are very low. Thus, a simple and practical method that can increase its EMI shielding effectiveness is by adding conductive fillings and loadings into the cement [6]. Eq. (1) shows that the shielding effectiveness of a material is linked closely to the electric conductivity and the electromagnetic parameters of the material. In contrast to a polymer matrix which is electrically insulating, the cement matrix is slightly conductive and its shielding effectiveness is closely related to its conductivity [6]. Due to its special performance properties, Portland cement was used as the cementation starting material in this study.

The cement matrix is only slightly conductive with an electrical resistivity of 10^5–$10^6\ \Omega\ \mathrm{cm}$ [4]. With the use of electrically conductive admixtures in the form of particles or short fibres, the resistivity of a cement-based material can be greatly decreased. Continuous fibres can also be used to reduce the resistivity but they cannot be integrated in a cement mix. Thus, the making of a continuous fibre cement-based material is much more complicated than that of a short fibre cement-based material.

Based on previous research, the ratio of water to the total cementation material was 0.35 [8]. A water-reducing agent such as sodium salt of a condensed naphthalenesulfonic acid can be used in the amount of 1.00% by mass of cement without aggregate.

Due to the high demand for low cost EMI shielding material and long-term compatibility with the chemical environment in a cement-based material, the electrically conductive admixtures used are mainly either steel or carbon. Previously, steel was used because it is more conductive than carbon. However, it is less available in the form of fine particles or fibres thus contributing to uneasy processing [4]. Generally, three main kinds of conductive fillers have been used in cement matrix materials: conductive polymers, carbon materials and metal materials. The most commonly used fillings are carbon materials (including graphite, carbon black and carbon fibre) which have relatively high conductivity and EMI shielding effectiveness [6]. It is desirable for EMI shielding materials to attain a low resistivity at just a low volume fraction of an admixture. This is because the workability and compressive strength decreases with increasing volume fraction of the admixture (due to the increase in air void content) and the cost increases with the admixture volume fraction. As a consequence, a small particle size, a small fibre diameter and a large aspect ratio are usually attractive properties of the admixture. However, an admixture unit size that is too small can be a disadvantage for the conductivity [4]. This may be due to the electrical contact resistance at the interface between the adjacent admixture units and the large number of such interfaces when the filler unit size is small.

In order for a conductive filler to be highly effective for shielding, it should preferably have a small unit size, high conductivity and a high aspect ratio. As to improving the conductive ability and shielding effectiveness of cement matrix composites, carbon fibres are more effective than particles such as carbon black and coke due to their large aspect ratio which can help to make more conductive networks through intercalating [9,10]. With the decrease in carbon fibre cost and the increase of demand for cement-based composites with high structure and multiple functions, carbon fibre cement matrix composites are gaining importance quite rapidly [6].

Many other conductive fillers such as nickel-plated carbon fibres, nickel coated mica and conductive papers have been used in EMI shielding applications [11–13]. However, these fillers are considered expensive and complicated to process as well so they are not commonly used in cement matrix composites. Metal powder has a disadvantage of high density, which produces lower shielding effectiveness with a small introduction. When the loading is increased to improve the shielding effectiveness, the density of the EMI shielding material is increased, resulting in a decrease of the mechanical strength of the composite material [6]. On the other hand, a material which is superior in conductivity is not necessarily superior in shielding, as proven by comparing carbon nanofibre cement and coke cement. The conductivity of carbon nanofibre cement is better than coke cement, but their shielding effectiveness have opposite values. A material which is excellent in shielding is not necessarily excellent in conductivity too. This can be exemplified by comparing graphite powder cement and carbon fibre cement, where the former has much lower conductivity but better shielding behavior [4].

Using Portland cement as the base matrix material and fly ash as the admixture, the fabricated composite can be used as an EMI shielding material. Studies show that a 4.3 mm sample thickness can have a shielding effectiveness of 4 dB in the frequency range 1–1.5 GHz. It was also shown that the shielding effectiveness is increased monotonically by increasing the fly ash composition, while the attenuation upon reflection is decreased slightly and monotonically. That means the fly ash can enhance the shielding by improving the reflectivity [6].

In this work, the sintered cement-based ceramic samples were prepared by using a casting-like method. The starting raw material was Portland cement as the matrix while manganese dioxide (MnO_2) was used as an additive in order to improve their EMI shielding properties. The samples were prepared with additions of 0.1, 0.5, 1, 5 and 10 wt% of MnO_2 into the cement powder. The effects of the additive composition on the dielectric and EMI shielding properties of the prepared samples were then investigated.

2. Materials and methods

Cement-based ceramic materials were prepared from the starting raw materials of Portland cement (PHOENIX by Lafarge) as matrix and manganese oxide (MnO_2) (Merck) as additive. The samples were prepared with the addition of 0.1, 0.5, 1.0, 5.0 and 10.0 wt% of MnO_2 into the cement matrix. The samples were mixed using the wet mixing method with a water-to-cement ratio of 0.35. No water-reducing agents, such as sodium salt of a condensed naphthalenesulfonic acid or aggregate, were used [8]. Wet mixing was performed by ball milling process using a porcelain jar filled with alumina balls for 2 h. The ratio of the mixed powder to alumina balls is 1:10.

Thermal analysis was performed to a selected sample of the mixed powder of 5 wt% manganese dioxide and Portland cement using Differential Thermal Analysis Linseis (DTA-PT1600) machine. Sample was heated from room temperature to 1000 °C at heating and cooling rate of 5 °C/min.

The mixed powder or paste samples were then poured into PVC pipe moulds. The size of each PVC mould is 15 mm in inner diameter and 10 mm in height. Prior to use, the PVC moulds were immersed in glycerin in order to produce unsticky mould walls. The casted samples were demolded after 1 day and cured in air at room temperature for another 2 days. Green pellets were subjected to a sintering process using an electrical furnace at 850 °C for 5 h with a heating and cooling rate of 5 °C/min. The sintered pellets were then grained and polished to produce a smooth sample surface with a final thickness of 2.0 mm for sample characterizations.

The samples were characterized using a field-emission scanning electron microscopy (FESEM) (Zeiss SUPRA 35VP) for microstructure observation. Dielectric properties were investigated using an HP 16453A Dielectric Material Test Fixture and HP 4291B RF impedance/material analyzer. This equipment provides a high-accuracy and easy measurement of dielectric/magnetic materials. In this work, an internal synthesizer sweep frequency from 1 MHz to 1 GHz was used.

The equipment used to measure EMI shielding effectiveness (SE) was arranged by having the electromagnetic wave directed at the center of the test fixture hole with the receiving antenna located directly behind the plate, as shown in Fig. 1. The appropriate signal generator (Agilent 83620B Signal Generator) was attached to the transmitting antenna while the spectrum analyzer (Agilent 8565E Spectrum Analyzer) was attached to the receiving antenna. After the SE test equipments are set up, the open reference (the hole is opened without a sample pellet) measurement was performed. Then, a closed reference is set up with the sintered sample pellet placed on the test fixture hole. The measurement frequency of SE was in the range of 8–13 GHz. The SE was obtained by taking the difference between the power level from the open reference and the power level recorded in the closed reference (Eq. (6)).

$$\begin{aligned}\text{SE (dB)} &= \text{open reference} - \text{closed reference}\\ &= \text{signal strength without sample}\\ &\quad - \text{signal strength with sample}\end{aligned} \tag{6}$$

The bulk density as well as porosity testing of sintered pellets was performed using Digital Density Meter (Micromeritics AccuPyc 1340 Series Pycnometer). A set of three specimens of each composition were prepared for the density, porosity, dielectric and EMI shielding testing.

3. Results and discussion

3.1. Thermal analysis

Differential thermal analysis (DTA) testing was done to estimate the optimum heat treatment temperature in the

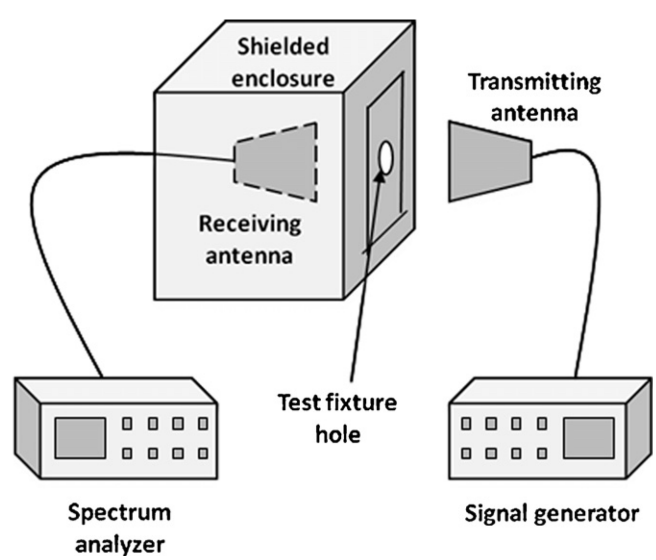

Fig. 1. Schematic diagram of EMI shielding effectiveness measurement setup.

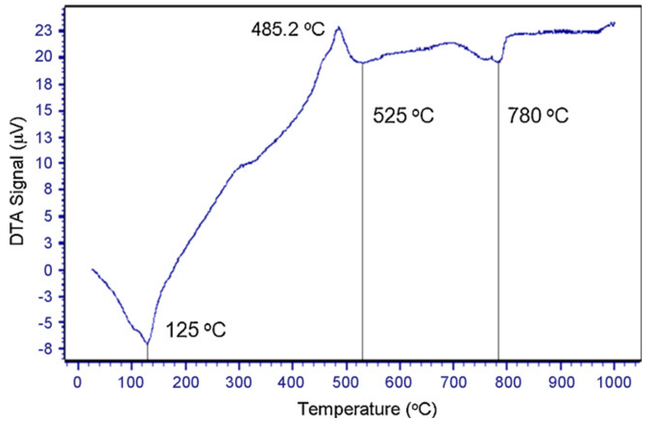

Fig. 2. DTA curve of mixture of 5 wt% MnO_2 and Portland cement powder.

preparation of cement-based EMI shielding materials. Fig. 2 shows the typical DTA curve of the mixture with 5 wt% manganese dioxide and Portland cement powder. At the first stage, an endothermic peak at around 125 °C is dehydration reactions due to the loss of water from calcium silicate hydrate (C–S–H) [14,15]. A rapid change toward ΔT positive in the DTA curve was observed from 120 to 485 °C. The exothermic peak at around 485 °C is due to the combustion of the organic components in the sample [15]. The endothermic peak at around 525 °C is dehydroxylation temperature of calcium hydroxide ($Ca(OH)_2$). Meanwhile, an endothermic peak at 780 °C is the de-carbonation temperature of $CaCO_3$, together with possible solid–solid phase transformations [14,15]. A plateau curve above 800 °C indicates the stable formation of cement phases. Based on these results, the heat treatment temperature for the green pellets was decided to be 850 °C for 5 h.

3.2. Density and porosity

Density and porosity were measured using the Archimedes principle for each sample. Fig. 3 shows the bulk density and

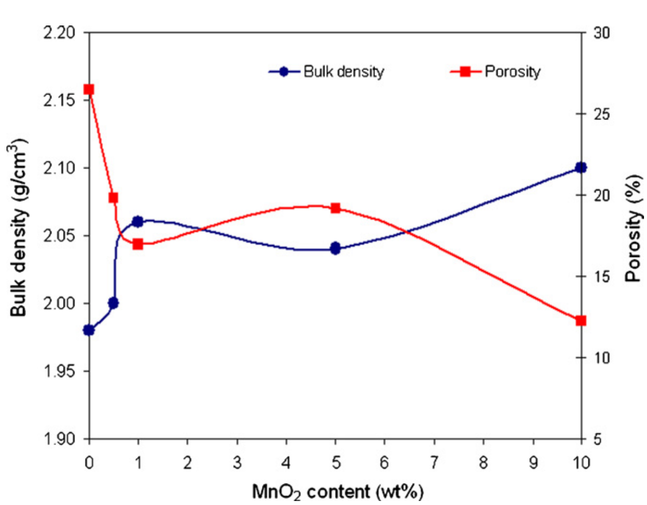

Fig. 3. Effect of manganese oxide additive concentration on bulk density and porosity of sintered MnO_2–cement pellets.

porosity for sintered MnO_2–cement pellets. As a general observation, the density increased with increased concentrations of MnO_2 additive. The opposite trend was observed for porosity as it decreased with increased concentrations of MnO_2 additive. The bulk density of sintered MnO_2–cement pellets was in the range of 1.98–2.11 g/cm^3 while the porosity range is from 13 to 27%.

3.3. Microstructures

Fig. 4 is a SEM micrograph of sintered cement pellets without and with additive. The pellet without MnO_2 additive shows particles in clustered or aggregated forms (Fig. 4(a)). The morphology of the sintered cement with MnO_2 additive is shown in Fig. 4(b)–(d). Fig. 4(b) is the SEM micrograph for 0.5 wt% MnO_2 that shows cluster or aggregate which is almost similar to the sample without additive. This may be due to the fact that extremely low concentrations of MnO_2 in cement do not significantly affect its morphology. Increasing MnO_2 concentrations to 5 wt% and 10 wt% show that the morphology of these sintered pellets are definitely different from pure cement morphology by its flaky appearance (Fig. 4(c) and (d)). EDX analysis was performed on a selected sample (sintered cement pellet with 10 wt% MnO_2 additive) give the elemental composition of 56.07 at% O, 17.04 at% Ca, 3.59 at% Si, 3.22 at% Al, and traces of C, S, Mn and Fe elements (Fig. 5).

3.4. Dielectric properties

Portland cement is a complicated hydrated material which is consists of four major compounds: tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate (C_3A), and tetracalcium aluminoferrite (C_4AF). Both C_3S and C_2S react with water (H) to form calcium silicate hydrate (C–S–H) and calcium hydroxide (CH) as their principal hydration products [14]. The XRD analysis was performed on the sintered cement pellets in order to investigate the phase formation after heat treated at 850 °C for 5 h. It was found the predominant phases of sintered cement are orthorhombic calcium silicate (γ-$Ca_2(SiO_4)$) and triclinic calcium silicate (Ca_3SiO_5) as shown in Fig. 6. Besides, the traces of serendibite ($CaMg_3O(Si_3O_9)$) and calcium manganate ($Ca_3Mn_2O_7$) have been detected co-exist in the sintered cement pellet.

The free water (pore solution) inside the material has a great influence on dielectric properties of cement [16–18], thus affecting the electromagnetic absorbing performance of the composite material [19]. Previous research has found that this material shows giant permittivity or dielectric constant at lower frequency ranges (below 1 MHz) and its curve presents a more stable plateau in a broad frequency range. The density of a material also plays an important role in the variation of dielectric constant. High porosity and low density results in low dielectric constant and high dielectric losses [20]. Dielectric constant and dielectric loss are important properties for EMI shielding.

In Fig. 7, it is shown that the dielectric constant of sintered MnO_2–cement pellets is reduced as frequency increased. At a

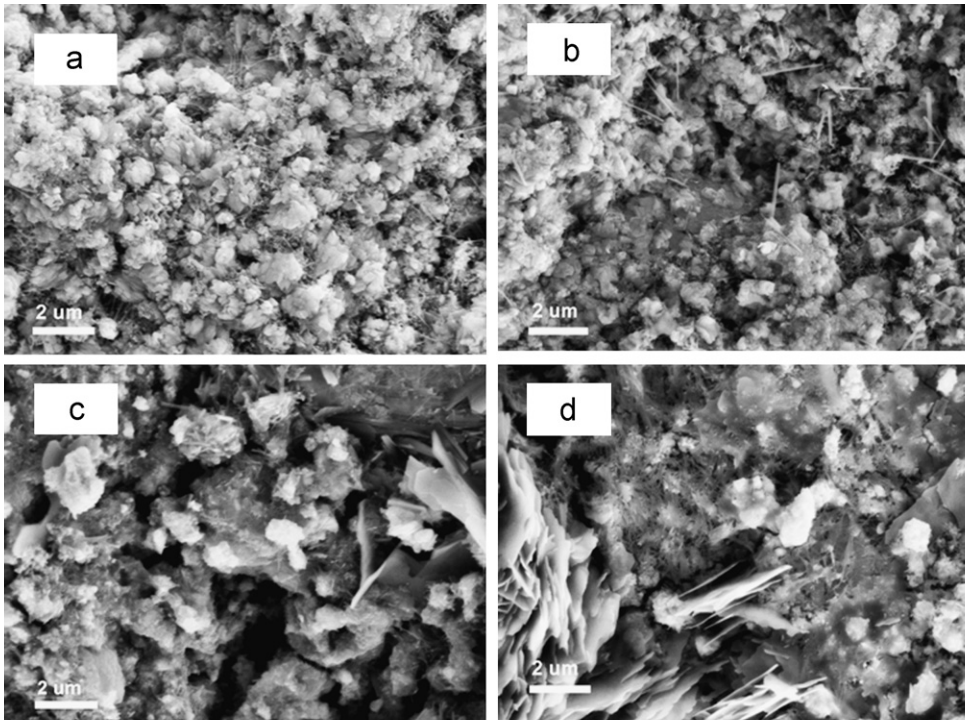

Fig. 4. SEM micrographs of sintered cement pellets with MnO_2 additive: (a) 0 wt%, (b) 0.5 wt%, (c) 5.0 wt%, and (d) 10.0 wt%.

lower frequency (1 MHz), the dielectric constant was about 300 and decreased to about 25 when the frequency increased to 200 MHz or higher. It was also observed that the dielectric constant value increased with the addition of the MnO_2 in cement matrix. The increase in the dielectric constant with increased manganese dioxide concentrations is due to the presence of high resistance in the internal barrier layer with the addition of manganese dioxide. The Mn ions can enter both the grain and grain boundary regions, and results in important changes in the electric properties of either the grains and/or grain boundaries with increasing Mn concentrations [21].

The incorporation of a magnetic material like MnO_2 as an additive reduces the dielectric loss of sintered cement pellets. Furthermore, the values of the dielectric constant can be altered to achieve maximum absorption of the electromagnetic energy. This means that the dielectric properties will be closely related to the EMI shielding behavior of materials. Fig. 8 shows the dielectric loss against frequency for sintered MnO_2–cement pellets. The dielectric loss was found to decrease from 0.7 to 0.3 as frequency increased.

Dielectric properties determine the electrical characteristics of a material. The dielectric constant and dielectric loss for different additive concentrations of sintered MnO_2–cement

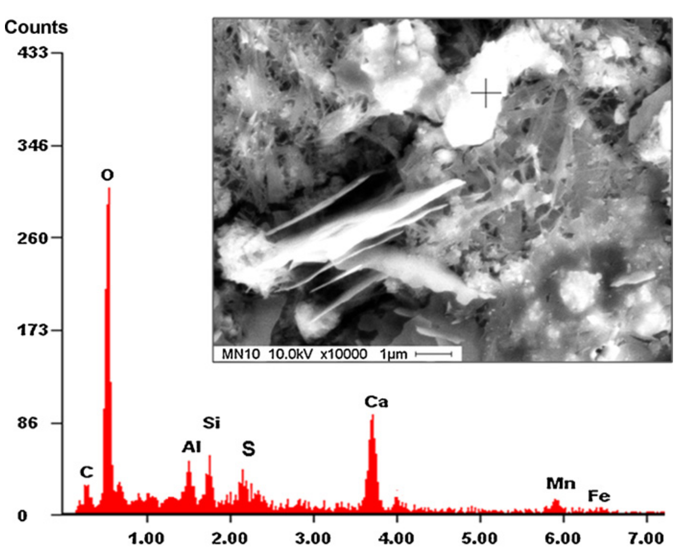

Fig. 5. EDX analysis of sintered cement pellet with 10 wt% MnO_2 additive give the elemental composition of 56.07 at% O, 17.04 at% Ca, 3.59 at% Si, 3.22 at% Al, and traces of C, S, Mn and Fe elements.

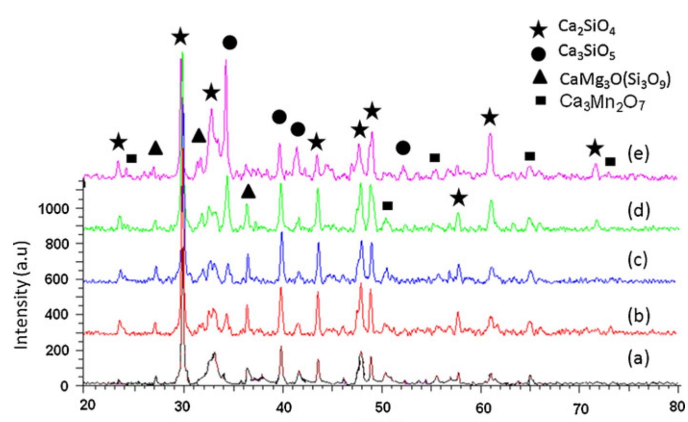

Fig. 6. XRD analysis results of sintered cement pellets with different Mn additive concentration: (a) 0.1 wt%, (b) 0.5 wt%, (c) 1 wt%, (d) 5 wt%, and (e) 10 wt%.

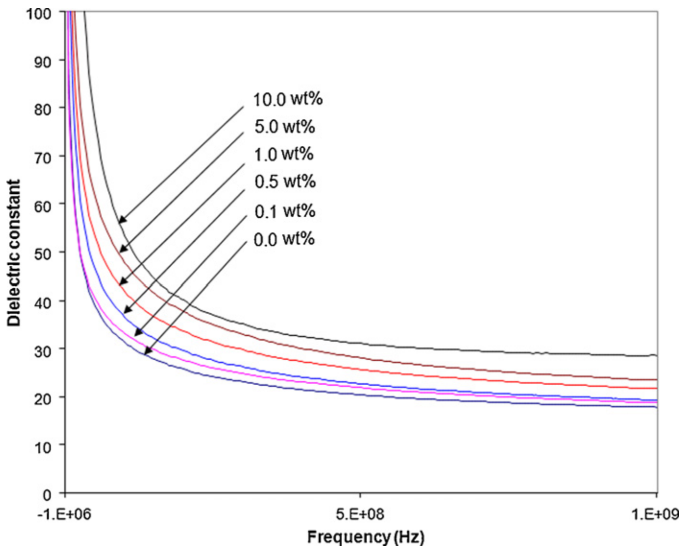

Fig. 7. Dielectric constant versus frequency for different concentration of sintered MnO_2–cement pellets.

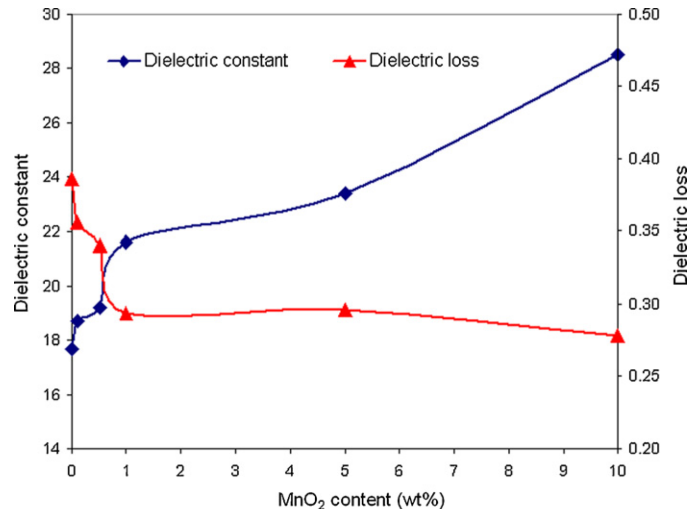

Fig. 9. Effect of additive concentration on dielectric constant and dielectric loss of sintered MnO_2–cement pellets measured at 1 GHz.

pellets were measured at 1 GHz, as plotted in Fig. 9. In Fig. 9, it can be observed that the dielectric constant of sintered MnO_2–cement pellets increased with increasing concentrations of additive. This may be due to the magnetic properties of the MnO_2 additive which contribute to more polarization for better dielectric constant value. Improvement of density and porosity through the increase in additive concentration were also expected to contribute in enhancing the dielectric constant. Results show that the dielectric loss of sintered MnO_2–cement pellets lie in the range of 0.28–0.35. These dielectric loss values are acceptable for most applications in electronic and microwave devices. A near ideal dielectric behavior with a high dielectric constant and a low dielectric loss was obtained. A slightly higher dielectric loss for sintered MnO_2–cement pellet with 5 wt% may be due to porosity inside the pellet sample. However, the low overall values of dielectric loss (~0.3) should be taken into consideration when applying these cement-based ceramics as dielectric material generally used in capacitors and transmission lines in filtering EMI.

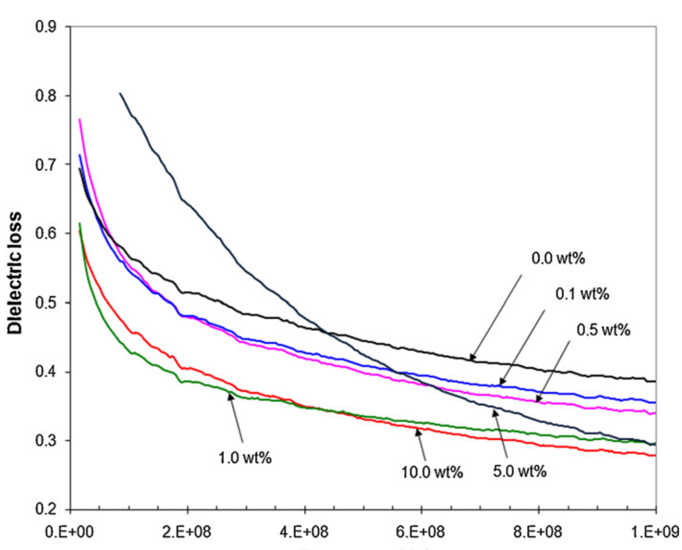

Fig. 8. Dielectric loss versus frequency for different concentration of sintered MnO_2–cement pellets.

3.5. *Shielding effectiveness (SE)*

With the rapid increase in the use of telecommunications, digital systems, and fast processors, the induced electromagnetic interference (EMI) has become a serious problem. EMI problems have impacted almost all electrical and electronic systems in daily life, in military activity and even space exploration. Thus, an ideal material with certain desired properties is needed to minimize these problems.

It is known that EMI shielding effectiveness is a combination of the reflection from the material surface, the absorption of electromagnetic energy and the multiple internal reflections of electromagnetic radiation. Based on literature, the shielding effectiveness (SE) for most cement-based ceramics is measured in the frequency range of 1–2 GHz or in the radio frequency range (1 kHz to 1 GHz) [6,8,22–24]. However, in this work, SE of sintered MnO_2–cement pellets were measured in the microwave frequency range of 8–13 GHz using a signal generator and spectrum analyzer. The microwave frequency is usually used for higher engineering applications such as antenna for weather monitoring, vehicular detection, air traffic control, and defense tracking [2].

The thickness of the sintered MnO_2–cement pellets used for the SE measurement is 2.0 mm. Fig. 10 shows the effects of additive concentration and frequency on the SE values of sintered MnO_2–cement pellets. As shown in Fig. 10, the SE values for sintered MnO_2–cement pellets fluctuated between 2 dB and 9 dB in the frequency range of 8–13 GHz. The sample with the highest additive content (10 wt% MnO_2) had better frequency stability of SE values, in the range of 4–9 dB. At high frequency, most of the samples with higher concentrations of additive gave SE exceeding 7 dB as shown in Fig. 11. These

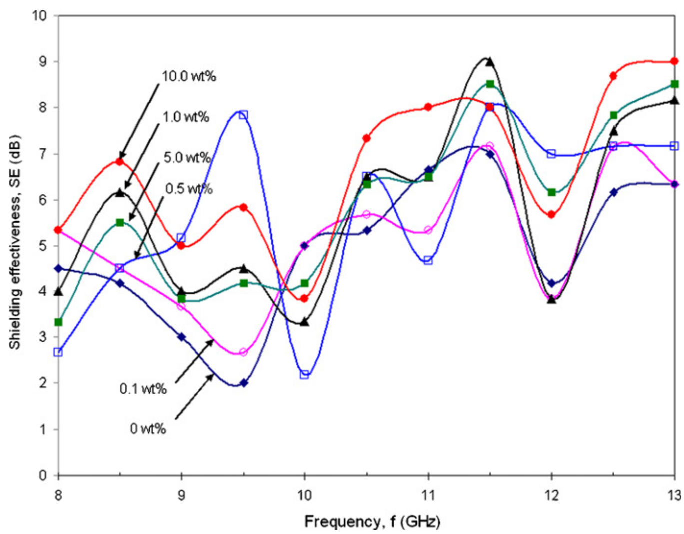

Fig. 10. Shielding effectiveness (SE) against frequency for different concentration of sintered MnO_2–cement pellets.

results indicate that the MnO_2 additive concentration plays an important role in improving the SE of cement-based EMI shielding materials.

From the literature survey, no published reports on MnO_2–cement materials for EMI shielding applications were found. However, some of the soft ferrites like manganese zinc ferrites (MZFO) have been studied for radio frequency applications such as inductors for resonant circuits, AC/DC converters, wide-band transformers, antennas, and electromagnetic interference (EMI) suppression. Composite materials possessing both conducting and ferromagnetic properties are extremely useful due to their application potential in electrochemical display devices, sensors, broadband microwave absorbers and electromagnetic shields. Feng et al. [25] reported that a double-layer microwave absorber with reflection loss <-8 dB can be obtained using carbonyl iron and barium ferrite powder ($BaZn_{1.5}Co_{0.5}Fe_{16}O_{27}$) and measured over the frequency range of 2–18 GHz. Meshram et al. [26] reported broadband characteristics with a minimum absorption of 8 dB from 8 to 12 GHz for a coating thickness of 2 mm using the hexagonal ferrite powder ($BaCo_{0.5\delta}Ti_{0.5\delta}Mn_{0.1}$-$Fe_{(11.87-\delta)}O_{19}$) and ($Ba(MnTi)_{\delta}Fe_{(12-2\delta)}O_{19}$) at $\delta = 1.6$. Reflection loss exceeding -5 dB was obtained between 5 and 18 GHz for coating containing 1.3 wt% FeCo-filled CNTs [27]. Li and et al. found a 15–20 dB EMI shielding effectiveness on single-walled carbon nanotube (SWNT)–polymer composite measured in the range of 500 MHz to 1.5 GHz [28]. As a conclusion, the EMI shielding results on sintered MnO_2–cement pellets written in this work concurs with the other shielding materials such as carbonyl iron, barium ferrite powder and CNT–polymer composite, and is even better than FeCo-filled CNTs.

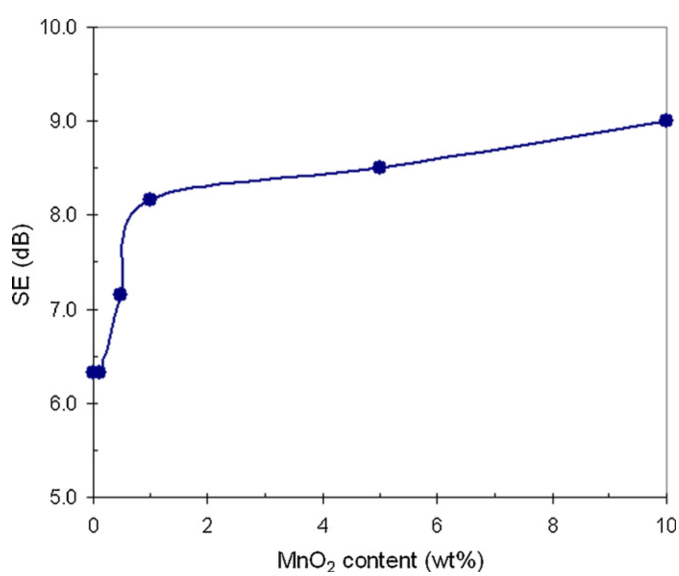

Fig. 11. Effect of additive concentration on shielding effectiveness (SE) of sintered MnO_2–cement pellets measured at 13 GHz.

4. Conclusion

Electrical properties such as dielectric constant and dielectric loss determine the EMI shielding behavior of materials. In this work, it was found that sintered MnO_2–cement pellets have good dielectric properties, i.e. high dielectric constant and low dielectric loss. The dielectric constant was increased by increasing the MnO_2-additive content in the cement matrix. The SE values of sintered MnO_2–cement pellets fluctuated between 2 dB and 9 dB measured in the frequency range of 8–13 GHz. The sample with the highest additive content (10 wt% MnO_2) had the most stable SE values, i.e. in the range of 4–9 dB. Most of the samples, especially ones with high concentrations of MnO_2-additive, gave SE exceeding 7 dB at the frequencies above 12 GHz. These results indicate that the manganese oxide filler concentration plays an important role in improving the SE of cement-based EMI shielding material.

Acknowledgments

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