

Investigation on electromagnetic and microwave absorption properties of copper slag-filled cement mortar



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ABSTRACT

Ordinary cement mortar is a widely used structural material. However, its microwave absorbing ability is very weak due to the lack of magnetic and electrical conductive components. Copper slag is a by-product produced from copper smelting, which possesses excellent magnetic properties. In this paper, ordinary cement mortar specimens incorporated with copper slag were prepared. The electromagnetic (EM) parameters were determined by a network analyzer in 8.2–12.4 GHz frequency ranges. The reflectivity of the mortars against EM wave was evaluated through an arch reflectivity testing system in the same frequency ranges. The skin depth and compressive strength of mortars were calculated and tested, respectively. Results show that both the real and imaginary parts of permittivity increase with the increase of copper slag content and over 30% copper slag contents can evidently improve the permittivity of the mortars. Meanwhile, permeability of the mortars is evidently improved in the frequency ranges with the addition of the copper slag. The reflectivity values of the copper slag-filled mortars tend to decrease and then increase with the increase of copper slag contents. The minimum reflectivity reaches -13.4 dB and the reflectivity below -10.0 dB can be obtained in the frequency range of 8.2–8.4 and 10.4–10.9 GHz representing 90% absorption when the copper slag content is 50%. Over copper slag content can make the mortar not absorb but reflect the EM microwave. Skin depth decreases with the increase of microwave frequency and copper slag content. The mortars with 50% copper slag content exhibit higher compressive strength value, as well as better microwave absorption performance.

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1. Introduction

Nowadays, the EM environment pollution has been paid more and more attentions [1,2]. EM waves may interfere with electrical equipments. In addition, the different EM radiations do harms on human health [3,4]. It is urgently required for the microwave absorbers, which can attenuate the radiation energy. The microwave with 8.2–12.4 GHz frequency bands and 3.75–2.4 cm wave length is usually used in high frequency communication devices [5]. Therefore, it is particularly needed for microwave absorbers within this frequency bands to prevent approximately EM interference of the devices [6].

Cement based composites are widely used as structural materials. However, their microwave absorbing ability and electrical

conductivity are very weak. Although some researchers use carbon fibers to reinforce cement based composites and to improve their mechanical properties and microwave absorbing ability [7–15], the cost of the composites is enhanced enormously. Therefore, it is urgent to find out materials with better electrical or magnetic conductivity and lower price to substitute carbon fibers in cement based composites. The effective way to improve the EM absorbing performance is the use of high magnetic or conductive materials [16,17].

Copper slag is a by-product obtained by copper smelting [18]. Production of one ton of copper produces about three tons of copper slag [19]. However, copper slag is endowed with outstanding mechanical properties and can be used in concrete as a substitute for fine aggregates [20]. On the other hand, the copper slag can reduce the mixing water content and is used to produce high-strength concrete [21]. In addition, the study of Goñ, et al. also shows that the copper slag can improve the durability of the hardened Portland cement pastes by enhancing mechanical property of the pastes [22]. Therefore, copper slag is a kind of

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sustainable and useful materials for cement based composites applications [23].

However, despite the promising facts [24–28], further investigations are greatly urgent in order to produce copper slag/cement based materials with microwave absorption function. Therefore, this study aims to investigate the feasibility of using copper slag as substitute for natural sand in improving the EM absorbing performance of ordinary cement mortar. Four different volume percentages of copper slag were used to study the effects of copper slag content on the permittivity, permeability and microwave reflectivity of cement mortar. The primary scope of this study is to interpret how copper slag affects the EM performance to put forward theoretical basis for improving microwave resistivity of the ordinary cement mortar in EM shielding engineering.

2. Experimental

2.1. Raw materials

Ordinary Portland cement was used and its properties are shown in Table 1. The fineness modulus of natural sand was 2.51 and density was 2.570 g/cm³. Tap water was used for mixing. In this study, the waste from refining of copper slag with the particle size of less than 4.75 mm was used. Its density and fineness modulus were 3.300 g/cm³ and 2.49, respectively.

2.2. Preparation of specimens

Copper slag was adopted in 10%, 30%, 50% and 70% by volume of natural sand. The mass ratio of cement: water: sand was 1:0.5:3 in the control group. Mixture proportions of the mortar are shown in Table 2. A cement mortar mixer was used for mixing. Water was first added into the mixer and then the cement. The mixer rotated with 62 ± 5 r/min speed for 30 s. Sand and copper slag were added and stirred with the same speed for 30 s. After being placed statically for 90 s, the mixtures were stirred with 125 ± 10 r/min speed for 60s. At last, the stirred mixtures were poured into 25 mm × 12 mm × 3 mm (EM parameter tests), 180 mm × 180 mm × 10 mm (reflectivity test) and 160 mm × 40 mm × 40 mm (compressive strength test) steel molds. The vibrating time of the steel molds in a vibration platform was 30 s.

Then, the steel molds were placed in a cement maintainer with

Table 1
Properties of the ordinary Portland cement.

Properties	Values
<i>Physical properties</i>	
Density/(g/cm ³)	2.917
Mean diameter size/μm	20.786
3 day compression strength/MPa	20.5
28 day compression strength/MPa	35.8
<i>Chemical properties</i>	
CaO content by weight/%	56.40
SiO ₂ content by weight/%	26.41

Table 2
Mixture proportions of the mortars.

Percentage (%)	Cement (g)	Sand (g)	Water (g)	Copper slag (g)
0	450	1350	225	0
10	450	1215	225	173
30	450	945	225	520
50	450	675	225	867
70	450	405	225	1213

the temperature of 20 ± 2 °C and the relative humidity of up to 90%. After 24 h, the specimens were remolded and dropped into 20 °C water for different curing ages (EM parameter tests and reflectivity test, 28 days; and compressive strength test, 3, 7 and 28 days). Afterwards, the specimens for EM parameter tests were ground into rectangular blocks with dimensions of 22.86 mm × 10.16 mm × 2 mm by sanding paper. Finally, all specimens for EM parameter tests and reflectivity test were dried to constant weight in an oven at 40 °C before the measurement, in order to decrease the influence of moisture contained in the specimens on the testing results.

2.3. Characterization

2.3.1. Analysis measurements

The morphology of the copper slag and the mortar were analyzed with S4800 Scanning Electron Microscope (SEM). The SEM resolution was 3.5 nm; the test condition was in vacuum and the test voltage was 15 kV. Energy Dispersive Spectrometer (EDS) was adopted to quantify the chemical compositions of the copper slag.

The copper slag and the mortar specimens were milled into fillers and were investigated with X-ray Diffraction (XRD, D/MAX 2400 diffractometer, Cu-Kα radiation), which was used in quantitative phase analysis of the crystals in the specimens. The test voltage was 40 kV, the electrical current was 100 mA and the XRD scan speed was 4°/min.

2.3.2. Compressive strength test

Three specimens of each group with 3, 7 and 28-day curing ages were prepared for the compressive strength test in accordance with a Chinese specification "Method of Testing Cements-determination of Strength (ISO Act)" (GB/T 17671-1999) [29]. Three specimens of each group were prepared and the average testing results were used.

2.3.3. EM parameter tests

In this study, the EM parameters, including permittivity ($\epsilon = \epsilon' - i\epsilon''$) and permeability ($\mu = \mu' - i\mu''$), were investigated. The real parts (ϵ' and μ') and the imaginary parts (ϵ'' and μ'') of the permittivity and permeability of the specimens with 22.86 mm × 10.16 mm × 2 mm dimensions were measured by a network analyzer (Agilent technologies E8362B) with rectangular wave-guide method [30] in 8.2–12.4 GHz frequency ranges at the temperature of 20 °C. Three specimens for each group were prepared and the average results were used.

2.3.4. Reflectivity measurement

The reflectivity of the specimens with 180 mm × 180 mm × 10 mm sizes was measured in the frequency ranges of 8.2–12.4 GHz by the arch reflectivity measurement system using a network analyzer (Agilent technologies E8362B) at the temperature of 20 °C. Test procedure can be referenced in the reported literature by Wang et al. [1]. One side of specimen was tested 3 times and the calculated average results were adopted. If the materials possess the reflectivity being lower than −10.0 dB, they can be used as excellent microwave absorbing materials [31].

2.3.5. Electrical conductivity test

The electrical resistance of the specimens with 22.86 mm × 10.16 mm × 2 mm dimensions was tested by a LCR meter (Wayne Kerr 4100) at the temperature of 20 °C. Four connectors, two current contacts and two voltage contacts were used as probes. The measurement accuracy was 0.1%. Three specimens of each group were tested to ensure data reliability and the average results were adopted. The electrical conductivity (σ) was calculated

by Eq. (1).

$$\sigma = \frac{1}{\rho} = \frac{L}{RS} \quad (1)$$

Where, σ - electrical conductivity, S/m; ρ - electrical resistivity, $\Omega \cdot m$; R - electrical resistance of specimen, Ω ; S - area of specimen, m^2 ; L - length of specimen, m.

3. Results and discussion

3.1. Microscopic analysis and mechanical property

The SEM morphology of copper slag is shown in Fig. 1. Its chemical compositions were quantified and shown in Fig. 2 and Table 3 using EDS. As can be seen in Fig. 1, the copper slag has irregular appearance. As shown in Fig. 2 and Table 3, the total mass fraction of silica, alumina, iron and oxide elements in the copper slag is 94.5% whereas the contents of other elements are 5.5 wt%. In addition, The XRD pattern of the copper slag as shown in Fig. 3

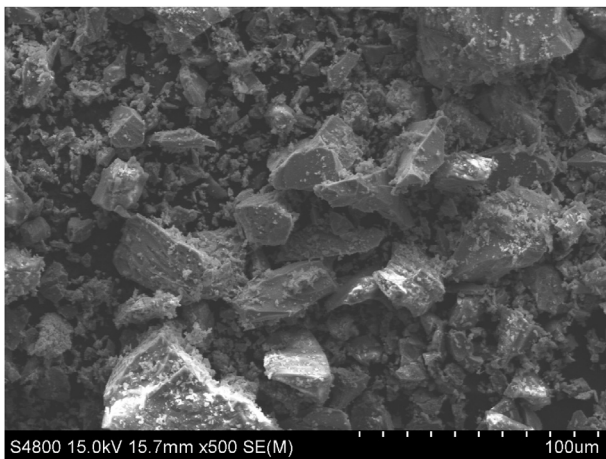


Fig. 1. SEM image of the copper slag.

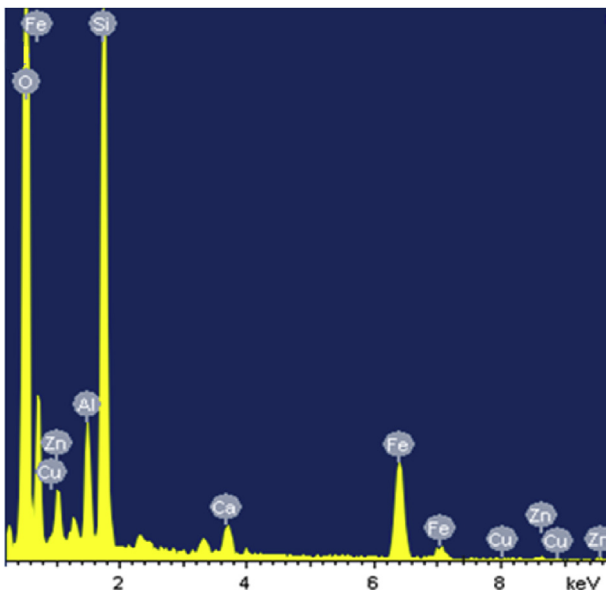


Fig. 2. EDS spectrum of copper slag.

Table 3
Element percentage in copper slag.

Elements	O	Fe	Si	Al	Zn	Ca	Cu	Total
Mass per.-%	49.94	21.73	18.40	4.46	2.33	1.63	1.51	100.00
Atom per.-%	70.83	8.42	14.87	2.91	1.51	0.92	0.54	100.00

reveals the presence of fayalite, anorthite and magnetite. Among them, magnetite has been paid great attention due to its superior magnetic property [32].

SEM image of the mortar incorporated 50% copper slag and its XRD pattern is shown in Figs. 4 and 5, respectively. After adding amounts of copper slag, ordinary Portland cement can produce hydrates as normal and a large number of clusters of hydrates and calcium hydroxide exist as shown in Fig. 4. Therefore, the strength of the mortar would not be significantly affected. Fig. 5 shows the compressive strength of the mortars under the different curing ages. It can be seen that the compressive strength firstly increases with the increase of copper slag content and then slightly decreases when the copper slag content is higher than 50%. However, compressive strength of mortars with 70% copper slag content is evidently higher than that of the control group. It indicates that the suitable addition of copper slag can enhance the mechanical property of mortars. Moreover, the study results also show that copper slag with suitable substitution in natural sand mass can improve the compressive strength of cement mortars [33,34].

Fig. 6 shows that in addition to quartz mainly coming from natural sand and calcium hydroxide, one of cement hydration products, there still exist fayalite, anorthite and magnetite in the cement mortar incorporated with copper slag. It indicates that magnetite in copper slag does not participate in the hydration of cement, and the microwave absorption ability of copper slag will not be affected by the cement hydration in the mortars.

3.2. The EM properties of the copper slag-filled mortars

3.2.1. Effects of copper slag contents on the dielectric properties

Fig. 7 shows the real (ϵ') and imaginary (ϵ'') parts of permittivity for the mortars with different copper slag contents over the frequency of EM wave. The ϵ' values of the specimens with copper slag are clearly higher than that without copper slag all over the frequency ranges as shown in Fig. 7 (a). Although the ϵ' changes little only from 7.0 to 7.6 for the specimens with over 30% copper slag contents, the ϵ' values are evidently higher than those of the mortars with 10% copper slag contents. It means that 30% copper slag contents can be considered as a threshold for improving the permittivity of the mortars. Thus, the microwave absorbing ability of the specimen would be improved greatly.

As to the ϵ'' values as shown in Fig. 7(b), the ϵ'' values of the specimens without copper slag are smaller than 0.4. And, the ϵ'' values still do not exceed over 0.5 even when 10% copper slag is incorporated. However, when the copper slag content is over 30%, the ϵ'' values are all improved greatly. Furthermore, one evident peak at about 9.5 GHz can be found in the ϵ'' curves, which is attributed to the dielectric change of the mortars with higher copper slag contents. In addition, when the copper slag content is 50% and 70%, the ϵ'' values are higher and the curves are similar. In a word, permittivity increases with the increase of copper slag content, and over 30% copper slag contents evidently improves the permittivity because of the enhanced electrical conductivity of the mortars.

Fig. 7 also shows that the values range of the ϵ' and the ϵ'' are 6.19–6.32 and 0.33–0.40, respectively, for the specimen without copper slag. It indicates that the specimen without copper slag can

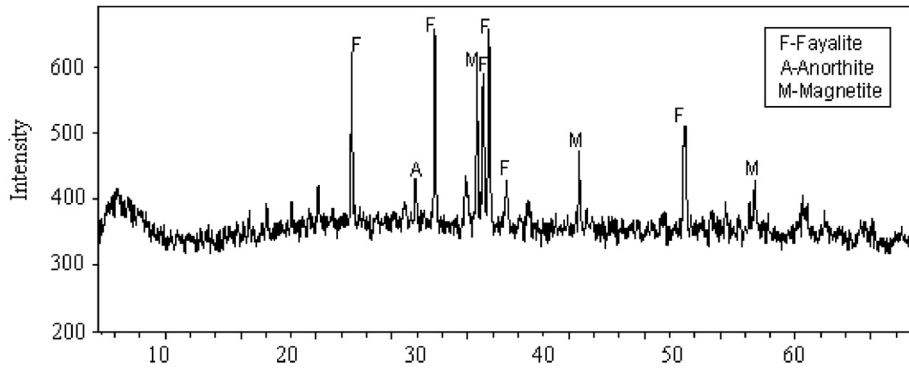


Fig. 3. XRD spectrum of copper slag.

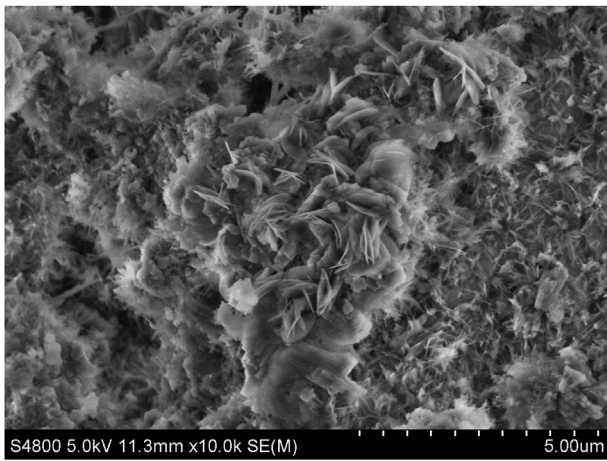


Fig. 4. Morphology of the mortar with 50% copper slag.

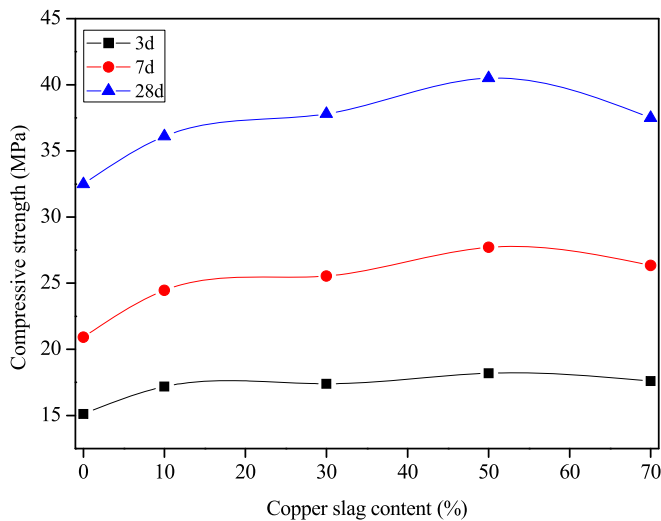


Fig. 5. Changes of compressive strength with copper slag contents.

also cause a little EM wave loss because of the small amount of metal oxides in cement clinker and the existence of hydration water in the hardened mortar. Besides, it can be observed that the ϵ' values decrease with increasing frequency, while the ϵ'' values increase with the increase of frequency, as shown in Fig. 7(b). Based on the Debye theory [2,35], Eq. (2) and Eq. (3) can be used to illustrate the relationship between permittivity and frequency,

which can be expressed as:

$$\epsilon' = \epsilon_{\infty} + \frac{\epsilon_s - \epsilon_{\infty}}{1 + \omega^2 \tau(T)^2} \quad (2)$$

$$\epsilon'' = \epsilon''_{ac} + \frac{\sigma_{dc}}{\omega \epsilon_0} \quad (3)$$

Where, ω - angular frequency; ϵ_s - static (zero frequency) permittivity; ϵ_{∞} - relative dielectric permittivity at high frequency limit; σ_{dc} - direct current conductivity; $\tau(T)$ - polarization relaxation time; and ϵ''_{ac} - alternating current loss contribution at the high frequency. From Eq. (2), the $\omega\tau$ values increase with the increase of the angular frequency. Therefore, the ϵ' values show a decrease with increasing frequency as shown in Fig. 7(a). In addition, for the mortar with certain copper slag content, the increase of frequency would result to the decrease of the $\sigma_{dc}/\omega\epsilon_0$ values and the increase of the ϵ''_{ac} values. Thus, according to the Eq. (3), the relaxation polarization loss resulted from interfacial polarization plays a dominating factor in enhancing ϵ'' in the specimen. And, the ϵ'' values would increase with increasing the frequency as a whole.

Generally speaking, the dielectric properties of conductive particle filled insulation composites depends on the EM wave frequency, the property and volume fraction of the filler and the matrix characteristics. For the copper slag/cement mortars, interfacial polarization resulted from the moving charge with alternating electrical field is an important polarization mechanism. Meanwhile, the associated relaxation loss induced by interfacial polarization will give rise to dielectric loss of the mortars. Therefore, it is considered that interfacial polarization plays roles in the permittivity of the specimen. It is reasonable that higher ϵ' and ϵ'' values can be obtained when the mortars are incorporated with a higher copper slag content.

3.2.2. Effects of copper slag contents on the magnetic properties

Fig. 8 shows the real (μ') and imaginary (μ'') parts of permeability for the mortars with different copper slag contents. For the specimens without copper slag, due to the lack of magnetic materials in ordinary Portland cement, the μ' and μ'' value is about 1.0 and 0 with all the frequency ranges. After the copper slag is incorporated, both μ' and μ'' values of the specimens are evidently enhanced in the frequency ranges of 8.2–12.4 GHz. In general, the complex permeability of the magnetic particles filled composites depended on the content of magnetic fillers. It is reasonable that the higher values of μ' and μ'' can be obtained when the composite filled with higher copper slag content. Thus, the permeability increases with the increase of the copper slag content, which would decrease the reflectivity and improve the microwave absorbing ability of the mortars. Besides, it is also observed that the μ' value

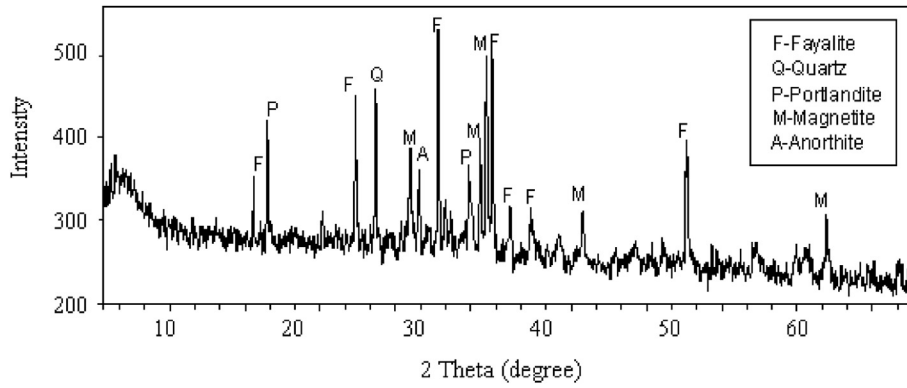


Fig. 6. XRD spectrum of the mortar with 50% copper slag.

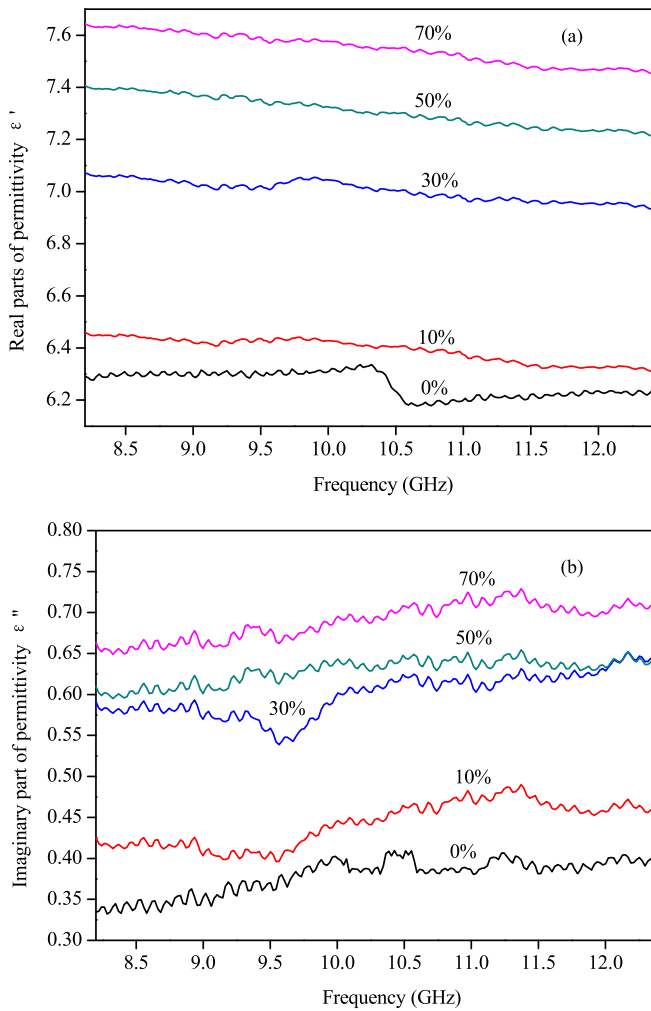


Fig. 7. Permittivity versus frequency curves of the specimens with different copper slag content: (a) ϵ' and (b) ϵ''

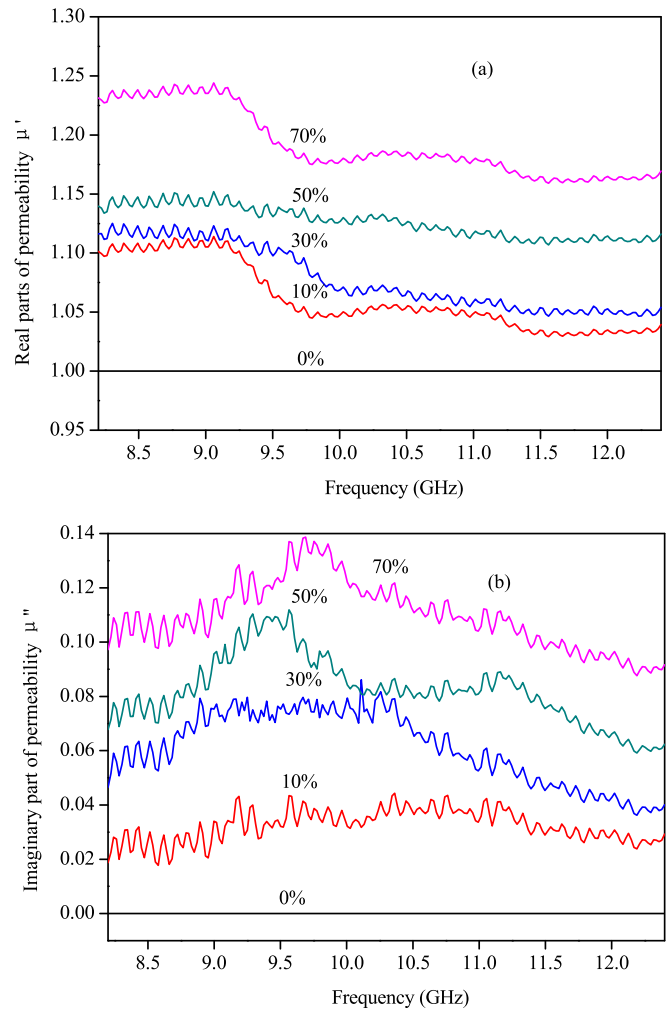


Fig. 8. Permeability versus frequency curves of the specimens for different copper slag contents: (a) μ' and (b) μ''

decreases with the increase of frequency and the visible peaks can be observed when the specimens with certain copper slag content. The magnetic loss of these results in such composites may be attributed to the natural resonance and exchange resonance.

Electromagnetic radiation at high frequencies penetrates only the near surface region of an electrical conductor [6,36], which is known as the skin effect. The electric field of a plane wave

penetrating a conductor drops exponentially with increasing depth into the conductor. The depth at which the field drops to $1/e$ of the incident value is called the skin depth (d), which is given by

$$d = \frac{1}{\sqrt{\pi f \mu \sigma}} \quad (4)$$

Table 4
Electrical conductivity of mortars with different copper slag contents.

Copper slag content (%)	0	10	30	50	70
Electrical conductivity ($\times 10^{-3}$, S/m)	14.29	20.83	44.47	99.56	288.46

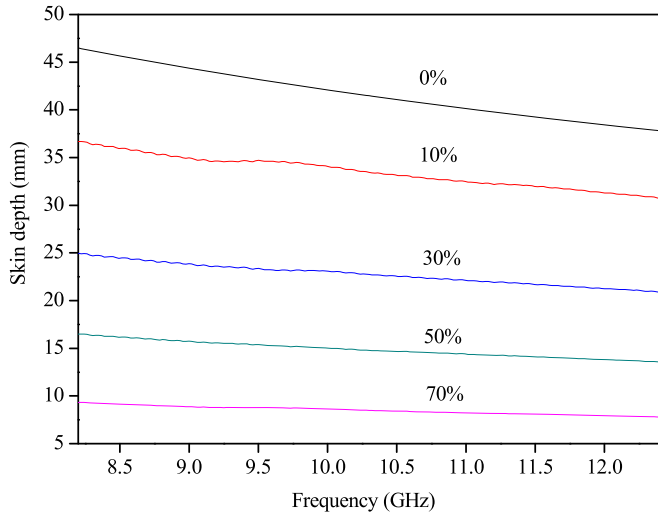


Fig. 9. Changes of skin depth with frequency and copper slag contents.

where, σ - electrical conductivity, S/m; d - skin depth, m; f - frequency, Hz; μ - magnetic permeability, $\mu = \mu_0\mu_r$; $\mu_0 = 4\pi \times 10^{-7}$ H m⁻¹; μ_r - relative magnetic permeability. The electrical conductivity of the specimen is listed in Table 4. According to the Eq. (4), The skin depth (d) of each specimens was calculated and the results are shown in Fig. 9. Hence, the skin depth values decrease with the increase of microwave frequency and copper slag content.

3.3. The microwave absorption properties of the copper slag-filled mortars

Fig. 10 shows the reflectivity of the mortar with different copper slag contents in the frequency ranges of 8.2–12.4 GHz. For the specimen without copper slag, it is shown that the reflectivity in

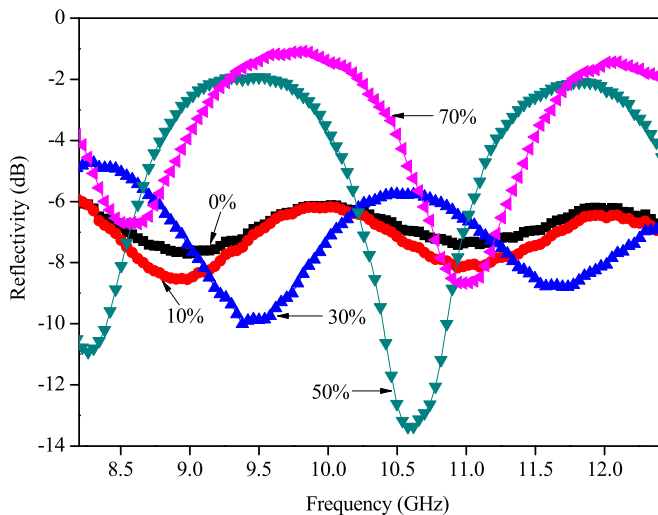


Fig. 10. Reflectivity curves of the specimens with different copper slag contents.

the frequency ranges is higher than -5.0 dB, which indicates that the specimen possess 60% microwave absorption property. However, when the copper slag content is 10%, two peaks with the reflectivity of -8.5 dB and -8.2 dB occur at 8.9 GHz and 11.0 GHz, respectively. When the copper slag content increases to 30%, one evident absorbing peak appears. The reflectivity value is -10.1 dB around 9.4 GHz. However, other values are greater than -10.0 dB. As the copper slag content reaches 50%, there exist two frequency bands with lower than -10.0 dB, responding to 90% microwave absorption. The reflectivity values are -10.9 dB at 8.3 GHz and -13.4 dB at 10.6 GHz, which indicate the strong microwave absorbing properties. However, when the copper slag content reaches 70%, the reflectivity is within a range of -8.3 to -1.0 dB. The reflectivity values are greater than -10.0 dB and the microwave absorbing ability of this specimen begins to decline.

The minimum reflectivity of the specimens with different copper slag contents is shown in Table 5. It can also be seen that the minimum reflectivity values tend to decrease with the increase of copper slag contents below 50%. The absorbing band width (reflectivity < -10.0 dB) for the specimen with 50% copper slag content is 0.7 GHz over the frequency ranges of 8.2–8.4 and 10.4–10.9 GHz, and the minimum reflectivity values reaches -13.4 dB at 10.6 GHz. However, the reflectivity below -10.0 dB does not appear for the specimen with 30% and 70% copper slag content and the microwave reflecting property becomes dominant.

The ideal absorbing materials should absorb EM waves as much as possible, which requires that the materials to possess large dielectric loss or magnetic loss [37]. The loss is weak if the copper slag content is low because the loss of the cement based material itself is low. If the copper slag content is too high, although the ability of dielectric loss or magnetic loss ability is high, the impedance matching is poor, which is not conducive to the effective absorption of EM wave. Therefore, there is an optimum content of copper slag in the cement mortar for acquiring high EM wave absorbing effect.

Therefore, the utilization of copper slag in cement mortars used in EM shielding engineering can consider the microwave absorption properties under the satisfactions of the structural requests. The optimum copper slag can promote the decrease of microwave reflection and the improvement of mechanical performance. Based on the results of this work, 50% copper slag contents are beneficial for the enhancement of both mechanical and microwave absorption performances of the mortars.

4. Conclusions

This is a preliminary study on EM parameter analyses and reflectivity evaluations of the copper slag-filled cement mortar. However, there are following results worth noting.

- a) Both the ϵ' and ϵ'' of the specimens with copper slag are clearly higher than those without copper slag all over the frequency range. Permittivity increases with the increase of copper slag content and over 30% copper slag contents can evidently improve the permittivity of the mortar.
- b) Both the μ' and μ'' of the specimens without copper slag are about 1.0 and 0. The complex permeability of the mortar depends on copper slag content. After the copper slag is incorporated, both the μ' and μ'' values are evidently enhanced in the frequency range.
- c) The electrical conductivity of the mortars evidently increases with the increase of copper slag content. In addition, the skin depth decreases with the increase of microwave frequency and copper slag content.

Table 5
The minimum reflectivity of the specimens.

Volume percentage of copper slag (%)	Minimum reflectivity (dB)	Frequency (GHz)	Band width below –10 dB (GHz) (frequency rang)
0	–7.7	8.8	Not appear
10	–8.5	8.9	Not appear
30	–10.1	9.4	0.2 (9.3–9.5)
50	–13.4	10.6	0.7 (8.2–8.4, 10.4–10.9)
70	–8.4	11.1	Not appear

- d) The mortar exhibits mainly reflection performances in the frequency range of 8.2–12.4 GHz below the copper slag volume mass fraction of 30%. The minimum reflectivity reaches –13.4 dB at the matching frequency of 10.6 GHz when the copper slag content is 50%. Whereas, when the copper slag content reaches 70%, the reflectivity values are greater than –10.0 dB and the mortar begins to reflect the wave.
- e) Optimum copper slag content is the key to achieve high EM wave absorption and compressive strength for the copper slag-filled cement mortar. The mortars with 50% copper slag content exhibit higher compressive strength value, as well as better microwave absorption performance.

Future researches

The results from this study indicate that copper slag can be used as one of components to prepare cement mortar as a potential sustainable microwave absorber for shielding applications. However, the following future studies are recommended. The EM wave frequency bands need to be extended to suit more electrical devices. The other is to study the internal structures of the mortar to interpret the mechanism of the EM and the reflectivity changes of the mortar.

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