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Electromagnetic wave absorbing cement-based composite using Nano-Fe_3O_4 magnetic fluid as absorber

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ABSTRACT

The idea of preparing electromagnetic wave absorption cement-based composite using nano-Fe_3O_4 magnetic fluid as electromagnetic wave absorber is proposed. Nano-Fe_3O_4 magnetic fluid with obvious superparamagnetism is synthesized based on the co-precipitation method. When 5% nano-Fe_3O_4 magnetic fluid is added into cement, the composite prepared shows excellent electromagnetic wave absorption properties, e.g. the absorption bandwidth with reflection loss lower than −10 dB and lower than −15 dB is about 9.5 GHz and 6.3 GHz respectively, much better than that of the composite prepared with nano-Fe_3O_4 powder and bulk Fe_3O_4 powder. As well, nano-Fe_3O_4 magnetic fluid accelerates the early hydration of cement and improves its early age compressive strength obviously. Due to the advantages of easy processing, cheap cost, non-toxic and high electromagnetic wave absorption, cement-based composite prepared with nano-Fe_3O_4 magnetic fluid shows the huge potential application in construction of electromagnetic wave interference shielding buildings.

1. Introduction

Electromagnetic waves (EMW) are widely used in industrial production, wireless communication, military applications and daily life. However, electromagnetic radiation causes environmental pollution and is potentially harmful to human health [1], information safety as well as electromagnetic compatibility [2]. In recent years, the negative effects of electromagnetic radiation have been a big concern of the society, and developing electromagnetic wave absorbing materials is of great significance in military and civil applications such as stealth, microwave interference protection and microwave darkrooms [3]. Generally, these materials are sorted into three types, magnetic loss type such as Fe_3O_4 and Mn-Zn ferrite, dielectric loss type such as TiO_2, and resistive loss type such as carbon black. Some researchers attempted to add these materials into cement to improve the electromagnetic wave absorption of cement-based composite building materials [4–6]. However, commonly the absorbers were added in the form of fine powders, and they tend to agglomerate together, which is unfavorable for the homogenous distribution of the absorbers in the cement paste, further obviously affecting the EMW absorption. So, to reach the threshold for effective absorption, the mass content of absorber might be as high as 10%–30% of cement, which is harmful for the workability of fresh cement paste, and inevitably damage the mechanical properties of cement-based composite.

In this paper, the idea of using nano-Fe_3O_4 magnetic fluid as the EMW absorber in preparation of EMWA cement-based composite is proposed. Fe_3O_4 is a kind of widely studied and used material, which has exhibited unique electric and magnetic properties based on the transfer of electrons between Fe^{2+} and Fe^{3+} in the octahedral sites [7]. Nano Fe_3O_4 has high magnetic performance, high saturation magnetization and it shows great advantages of being used as microwave absorber. For example, D. Wang et al. prepared $Fe_3O_4@SiO_2$ nanoparticles and used them as absorbent in cement-based microwave absorbing material [8]. Nano Fe_3O_4 can exist in stable suspension in the form of magnetic fluid. Recently nano-Fe_3O_4 magnetic fluid has been reported for the applications in various fields such as physics, medicine, biology and materials science due to its multifunctional properties such as small size, superparamagnetism and low toxicity [9–12]. However, to the best of our knowledge, there has no report on the application of nano-Fe_3O_4 magnetic fluid as the EMW absorber in cement-based composite. Herein, effects of nano-Fe_3O_4 magnetic fluid on EMW absorption performances are investigated.

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2. Experimental section

2.1. Materials

Ferrous sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$, AR), ferric chloride hexahydrate ($FeCl_3{\cdot}6H_2O$, AR), ammonium hydroxide (0.3 mol L $NH_3{\cdot}H_2O$, AR), and sodium oleate ($C_{18}H_{33}NaO_2$, AR) were obtained commercially (Shanghai Hushi Chemical Co., Ltd.). Ordinary Portland cement used was from Huaxin Cement Co. Ltd. ISO 679–1989 standard quartz sand was used as the fine aggregate to prepare the mortar. Expanded perlite with apparent density of 180 kg m^{-3} and diameter of 1–3 mm was used in the preparation of electromagnetic impedance matching layer.

2.2. Processing

The nano-Fe_3O_4 magnetic fluid was prepared based on the co-precipitation method. $FeCl_3{\cdot}6H_2O$ and $FeSO_4{\cdot}7H_2O$ were dissolved into deionized water at a total iron concentration of 0.15 mol L^{-1} under the protection of N_2 atmosphere. After vigorous stirring for 5 min, aqueous ammonia was added dropwise until the pH value was titrated to 10.0. The mixture was then heated to 80 °C by water bath and kept reacting under vigorous stirring for 60 min. Afterwards, the products were washed repeatedly with deionized water for 4–5 times until the filtrate was neutral. The filtered nano-Fe_3O_4 particles were then added into 15 g·L^{-1} sodium oleate, heated to 80 °C and stirred vigorously under N_2 atmosphere protection for 60 min. Sodium oleate solution was used here as surfactant, which is widely used in the preparation of magnetic fluid for improving the dispersion and stability of the nano-particles [13,14].

The prepared nano-Fe_3O_4 magnetic fluid was measured and mixed with cement and sand to produce the EMWA cement-based composite, according to the mixture of cement:Fe_3O_4:sand:water = 100:3/5/7:300:40. Cement and sand were mixed first for 1 min, and then the nano-Fe_3O_4 magnetic fluid and the additional water was added slowly and mixed for 3 min. In the process, small amount of polycarboxylic superplasticizer (0.1 wt% of cement) and hydroxypropyl methylcellulose (0.05 wt% of cement) were used to modify the workability of fresh mortar.

The fresh mortar was cast into 180 mm × 180 mm × 20 mm plate molds, flattened and compacted on a vibrating table for 10 s. After initial setting, matching layer mortar with the thickness of 10 mm was cast onto the top surface of the plate specimen; the mortar was prepared by mixing 70 vol% expanded perlite and 30 vol% cement paste (W/C = 0.4) together. After curing in room environment for 1 day, the specimens were demolded and cured in 20 °C and 90% relative humidity environment until 28 days. One mortar specimen is displayed in Fig. 1; a pen was placed on the specimen to show its size.

2.3. Characterization

The morphologies of the specimens were observed on a FEI/Quanta450 FEG scanning electron microscope (SEM) in secondary electron imaging mode, and backscattered electron-energy dispersive spectrometer (BSE-EDS) was used to determine the distribution of the Fe element in the hardened cement paste. The mineral phases of the specimens were determined by X-ray powder diffraction (XRD). The XRD patterns were collected on a Bruker D8 Advance diffractometer using Cu Ka radiation (k = 1.54184 Å). Before testing, the nano-Fe_3O_4 particles were separated from the magnetic fluid by magnet and dried in a vacuum oven at 40 °C for 6 h. Core part of the hardened mortar or cement paste was taken out from the sample using sharp tool for SEM testing, and part of it was dried and ground to fine powder seived through 75 μm sieve for XRD and non-evaporable water content testes. The ignition loss of the powder sample between 105 °C and 1000 °C was acquired on a NETZSCH STA449F3 thermal analyzer in N_2 atmosphere, and then non-evaporable water content was calculated based on the ignition loss and the net mass of cement.

Fig. 1. Mortar specimen of EMWA cement-based composite.

The magnetic properties of nano-Fe_3O_4 particles were evaluated on a vibrating sample magnetometer (VSM-PPMS-9). The complex permittivity and complex permeability of nano-Fe_3O_4 were analyzed using a network analyzer (Agilent technologies N5230A) in classic coaxial transmission/reflection mode in the frequency range of 8–18 GHz. The samples used for measurement were made by uniformly dispersing the nano-Fe_3O_4 powder and paraffin wax in a mass ratio of 7:3, and then the mixtures were pressed into toroids. The reflection loss of the mortar specimens was measured in an anechoic chamber using the arched testing method.

3. Results and discussion

3.1. Morphology and crystalline phase of nano-Fe_3O_4

Fig. 2 shows the XRD pattern of the prepared nano-Fe_3O_4. Compared with the data in PDF card (No. 75–33), all the main peaks can be identified as face centered cubic Fe_3O_4. Fig. 3 shows the SEM image of Fe_3O_4 particles with the particle size of tens of nanometers.

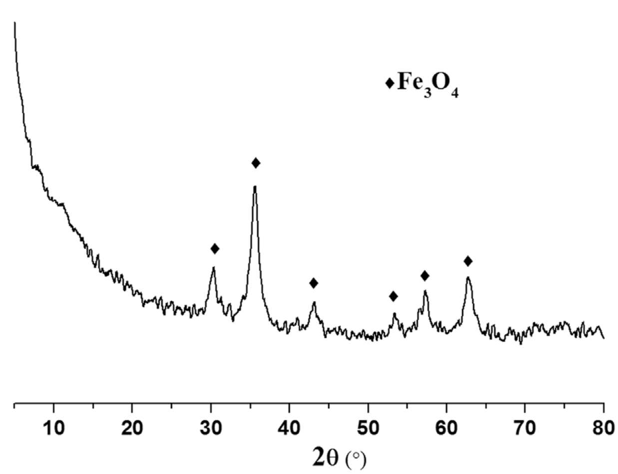

Fig. 2. XRD pattern of nano-Fe_3O_4 particles.