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Microwave pre-curing of Portland cement-steel slag powder composite for its hydration properties

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HIGHLIGHTS

- Microwave pre-curing improves the cement-steel slag mortar's compressive strength.
- Microwave pre-curing decreases the porosity of cement-steel slag mortar.
- RO or Fe_2O_3 has little effect on the microstructure under microwave pre-curing.

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ABSTRACT

A comparative study was carried out between normal curing, 40 °C steam curing for 10 h, 80 °C steam curing for 4 h and microwave pre-curing for 45 min to investigate the effect of microwave pre-curing on the hydration of Portland cement-steel slag powder composite. The compressive strength of mortar prepared with composite binder was tested. The hydration and microstructure of the composite were also studied by XRD, TG-DSC, MIP, Water absorption and SEM-EDS methods. The results show that although the hydration degree of composite binder microwave treated is lower than that cured with steam, the porosity, especially the pores larger than 50 nm, is reduced significantly by microwave pre-curing. Therefore, a small amount of hydrated products can connect each phase into a whole to improve the compressive strength of mortar treated with microwave when compared with other three regimes. In addition, as for the inert RO or Fe_2O_3 in the steel slag, it does not have an adverse effect on the microstructure as it can be tightly connected with the hydrated gel. But microwave pre-curing cannot accelerate the hydration of C_2F, which is different from 80 °C steam curing.

1. Introduction

Steel slag is the by-product of steelmaking. It is often used as the grain material of road base [1] and the aggregate of pavement asphalt mortar [2] for its good wear resistance. The chemical composition of steel slag is mainly CaO, MgO, SiO_2 and FeO, and their content is about 88% to 92% of steel slag [3]. The main mineral composition of steel slag is dicalcium silicate (C_2S), tricalcium silicate (C_3S), RO phase (solid solution of CaO, MgO, FeO and MnO) and a small amount of free calcium oxide (*f*-CaO). It is precisely because of the cementitious phase such as C_2S and C_3S that steel slag can be used in cement production [4]. But RO phase is hardly grinded, and its activity is very low [5]. Therefore, the increase of the proportion of the silicate phase and the inert phase (RO) can improve the activity of the steel slag powder to a certain extent. However, its own activity of the cementitious phase mainly γ-C_2S in the steel slag is low. So the overall activity of steel slag is always lower than that of cement. Another way to improve the activity of steel slag is to add modified material such as slag powder to increase the content of CaO and SiO_2 [6–9]. However, with the increase of SiO_2 content, the alkalinity of steel slag is reduced, thus reducing the activity of the steel slag [10]. Therefore, the alkalinity should also be considered when adding the modified material. Research has shown that C_3S can promote the hydration of C_2S [11]. So the alkalinity can be improved and the hydration of steel slag can be promoted by C_3S if it is added into the steel slag powder. In the process of steel production, an excess of CaO is added to adjust the alkalinity to change the mineral composition of steel slag, which has the same effect.

In addition to improve the cementitious properties of the steel slag itself, three main methods are used to stimulate the hydration activity of steel slag, namely, grinding, chemical excitation and thermal excitation. Grinding is to improve the fineness of steel slag. It is widely believed that the active index of steel slag powder

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increases with the increase of specific surface area of slag powder [12,13]. Although grinding can stimulate the hydration activity of steel slag to a certain extent, the activity of ultra-fine steel slag is still obviously lower than that of cement clinker [14]. Chemical stimulation is to promote the hydration of active minerals in steel slag by increasing the alkalinity of the solution [15]. When the $[AlO_6]$ tetrahedron is deactivated in steel slag, $[AlO_6]$ is dissolved in its original position in the form of $Al(OH)^{2+}$, forming water soluble ions and reacting with $H_3SiO_4^-$, OH^-, Ca^{2+} and Na^+ existing in the solution to form zeolite hydrates. Thermal excitation, that is, increase the hydration activity of steel slag by increasing the hydration temperature of steel slag. The excitation mechanism is that under high temperature the Si—O bonds and Al—O bonds in the structure of steel slag vitreous network are more easily fractured under thermal stress, which is conducive to depolymerization of the vitreous and thus improving the hydration activity of steel slag. Wang and others' studies show that RO, Fe_3O_4 and dicalcium ferrite (C_2F) remain inert by simple alkali excitation, but in the high temperature environment, C_2F is mostly hydrated in 1 d. But the high temperature cannot stimulate the reaction of RO and Fe_3O_4. Under the compound excitation of alkaline environment and high temperature, the latter plays the leading role [16,17].

Watson began to study microwave curing of concrete as early as 1960s, but its compressive strength was only half of that under standard curing [18]. Because the detailed data is unknown, according to Lee's speculations, this may be caused by the unequal temperature of the microwave curing system [19]. Experiments have proved that optimization of parameters such as microwave output power and operation time of microwave curing can effectively improve the strength of concrete and shorten the curing period [20,21]. In addition, Li et al. thinks that water to cement ratio is the key to the strength development of microwave curing concrete [22]. When the water to cement ratio of microwave curing concrete is reduced to about 0.38 (microwave radiation 45 min), the strength is the highest and the porosity is the lowest. When the microwave curing time continues to increase, the water to cement ratio decreases continuously, resulting in a slight increase of total porosity of concrete. However, the study of Sohn et al. believes that temperature is the key to control the 28-d strength of microwave curing mortar [23]. The results show that the 28-d strength of the mortar prepared with composite binder at 40 °C and 60 °C is basically the same as that of the standard curing, while the strength of mortar at 80 °C is significantly reduced.

In fact, some problems are still unknown so that the microwave processing is still in the stage of theoretical exploration. Although microwave can quickly heat the concrete, the uneven temperature distribution in the microwave curing chamber restricts the application of microwave in the field. Rattanadecho and Makul are working together to solve this problem and have developed a multi-mode cavity microwave curing equipment in 2016 [24]. In addition, microwave is an electromagnetic wave, so electric arc or spark will be produced when it meets metal. Mangat et al. recently developed a microwave pre-curing model equipment, and used it to study the effect of microwave on the temperature change of repair mortar [25]. The results show that microwave curing can be used for ordinary CEM II repair mortar. And the equipment is also suitable for mortar with embedded bar. Even if the steel bar is exposed to the outside of mortar, there is no arc or spark on the surface of steel bar. Lau, Ong and Tan et al. have used microwave to produce ferrocement slabs. The results show that compared with standard curing, microwave curing will not reduce the strength and durability of slabs, but the bonding performance of steel and mortar needs further confirmation [26]. Wu et al. found another phenomenon that microwave increases the brittleness of mortar [27]. This is because the increase of compressive strength of microwave curing mortar is greater than that of flexural strength. The effects of microwave curing on the mechanical properties and microstructure of glass fiber reinforced cement composites were investigated by Pera et al. [28]. The results show that the crystallization of stratlingite and monosulphate will gather on the surface of glass fiber. They are tightly combined with glass fiber to prevent it from pulling out, thereby reducing the composites' ductility.

Although microwave pre-curing of cement-based materials has the above disadvantages, it also has more advantages, which drives researchers to push the microwave application. First of all, microwave is a clean energy. The use of microwave can save fossil fuels. And more importantly, microwave can quickly improve the strength of concrete. This can be explained by the following reasons. Firstly, the damage of concrete caused by temperature rising rate can be ignored as the concrete is heated from inside to outside by microwave. According to the Rattanadecho's study, the microwave curing has no effect on the compressive strength and the skin quality of specimens when the curing temperature rises from 20 °C/h to 40 °C/h [29]. Mak et al also demonstrated that compared with steam curing, the temperature gradient of microwave curing concrete is low, and the heating rate of microwave curing does not cause the deterioration of concrete surface quality [30]. Secondly, microwave accelerates the cement hydration. Hutchison et al. has studied the microwave pre-curing of type I Portland cement and mortar. The results indicate that microwave curing shortens the induction period of cement hydration, increases the hydration degree of cement in 1 d, improves the early strength of mortar, and has no effect on the hydration degree of cement and strength of mortar in the later stages [31]. Wu et al. got the similar result that microwave accelerates the hydration of C_3S and C_2S of cement [27]. Thirdly, microwave pre-curing reduces the porosity significantly at early ages, making the structure more homogeneous [32]. This is attributed to the plastic compaction of mortar resulted from water evaporation during microwave pre-curing [27]. The last but not the least, microwave curing is beneficial to the hydration of supplementary cementitious materials. Pera et al. studied the effect of microwave curing on the hydration activity of metakaolin. The results show that after the hydration of 28 d, the consumption of Calcium hydroxide (CH) in the 15% metakaolin system after microwave curing was completed, while 30% metakaolin was required for the completely consumption of CH under 20 °C environment [33]. Korpa et al. used P.I 52.5R cement, silica fume, fly ash and nanometer volcanic ash as cementitious materials to prepare super high strength concrete by microwave pre-curing. The results show that the superposition of the microwave action and the active effect of the volcanic ash material makes the 1-d compressive strength of concrete up to 420 MPa [34]. At present, some progress has also been made in the study of microwave curing of alkali activated cementitious materials. Chindaprasirt and Inada et al. tried to stimulate the high calcium fly ash by microwave and heat curing to improve the physical and mechanical properties of the cementitious material [35–37]. The results show that the hydrogen bond of water molecules is broken by the microwave stimulation to form active water, which can interact with Al—O and Si—O alone to promote the dissolution of fly ash. At the same time, the temperature of the pore solution increases rapidly, which is beneficial to the dissolution of Si and Al to form the hydrated product zeolite. Microwave and high temperature composite curing is conducive to the formation of more hydration products, making the structure denser, thereby improving the early strength, thermal stability and dimensional stability of geopolymer. Shi et al. also has studied the microwave curing of alkali activated fly ash. The results show that hydroxysodalite was formed under thermal oven curing and chabazite-Na was produced under microwave curing. And higher polymerized aluminosilicate was formed in the sample under microwave curing [38]. The effect of alkali concentration on the