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Performance of copper slag contained mortars after exposure to elevated temperatures

Qianmin Ma [a,*], Haiyun Du [b], Xintao Zhou [c], Kecheng He [d], Zhiwei Lin [a], Feng Yan [a], Liping Huang [a], Rongxin Guo [a]

[a] Faculty of Civil Engineering and Mechanics, Kunming University of Science and Technology; Yunnan Provincial Key Laboratory of Civil Engineering Disaster Prevention, Jingming South Road, 650500 Kunming, China
[b] China Railway Fifth Survey and Design Institute Group CO., LTD, 9 Kangzhuang Road, 102600 Beijing, China
[c] Faculty of Chemical Engineering, Kunming University of Science and Technology, Jingming South Road, 650500 Kunming, China
[d] Roads and Traffic Authority of Yangxi County, Guangdong Province, 89 Qiaopingyi Road, 529800 Yangxi, China

HIGHLIGHTS

- Mechanical properties of copper slag cement mortars after exposure to high temperature.
- Mechanical properties of alkali activated copper slag mortars after exposure to high temperature.
- Chemical and microstructural evolutions of copper slag cement and alkali activated copper slag with temperature.

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ABSTRACT

Performance of copper slag contained mortars after exposure to elevated temperatures is studied in this paper. Portland cement (PC) based mortar with copper slag replacements of 0, 5%, 10% and 15%, as well as NaOH activated copper slag mortar with NaOH concentrations of 6 M, 8 M, 10 M and 12 M were manufactured. Compressive strength and flexural strength of the mortar specimens were tested at room temperature and after exposure to 200 °C, 400 °C, 600 °C, 800 °C, 1000 °C and 1200 °C, respectively. Paste specimens were further prepared to measure their chemical and physical changes with temperature by using X-ray diffraction (XRD), thermal gravity (TG)/differential scanning calorimeter (DSC) and scanning electron microscopy (SEM)/SEM-energy dispersive spectrometer (EDS) techniques. The results show that the strength of PC mortars containing copper slag increased within 200 °C, but after that their strength declined continuously. While the strength of alkali activated copper slag mortars increased dramatically both within 200 °C and after 800 °C. Amorphous $Fe(OH)_3$/$Fe(OH)_2$ gels could be formed in the hydration products of PC containing copper slag at room temperature and of alkali activated copper slag at a higher temperature. For alkali activated copper slag, after 600 °C, Fe_2O_3 was formed as a result of the oxidation of Fe_2SiO_4 and/or the decomposition of $Fe(OH)_3$/$Fe(OH)_2$. With the further increase of temperature, Fe_2O_3 could have melted in a Na-rich environment. The melted matters could sufficiently flow through the pores and cracks in the matrix. When the matters cooled down, they could fill in the pores and cracks and then make the matrix much dense to provide high strength.

1. Introduction

Copper slag, which is a kind of by-product from copper smelting industry [1], has been used as supplementary cementitious materials to manufacture mortars and concretes [2]. Further, alkali activated copper slag is also successfully prepared [3–5]. Mechanical properties and durability of the copper slag contained mortars and concretes have been investigated by studies [3–14].

More and more attentions have been paid on the performance of concrete at high temperature worldwide in recent years due to fire on buildings is seriously threatening personal and property safety [15]. Deterioration of binding materials is one of the most important reasons causing damage of concrete at high temperature [15]. Copper slag contained materials have exhibited potential to be used as cementing binder and have considerable mechanical

* Corresponding author.
E-mail address: maqianmin666@163.com (Q. Ma).

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properties and durability. However, performance of such materials at high temperature needs to be studied further before their wider application.

2. Experimental programme

Performance of copper slag contained mortars after exposure to elevated temperatures is studied in this paper. Portland cement (PC) based mortars with copper slag replacements of 0, 5%, 10% and 15%, as well as NaOH activated copper slag mortars with NaOH concentrations of 6 M, 8 M, 10 M and 12 M were manufactured. Compressive strength and flexural strength of the mortar specimens were tested at room temperature and after exposure to 200 °C, 400 °C, 600 °C, 800 °C, 1000 °C and 1200 °C, respectively. Paste specimens were further prepared to measure their chemical and physical changes with temperature by using X-ray diffraction (XRD), thermal gravity (TG)/differential scanning calorimeter (DSC) and scanning electron microscopy (SEM)/SEM-energy dispersive spectrometer (EDS) techniques.

2.1. Materials

The cement used in this paper is P·O 42.5 Portland cement from Hongshi Cement Ltd., Yiliang, Yunnan, China. Its chemical composition and physical properties are given in Table 1. Copper slag from Yunnan Copper (Group) Co., Ltd., China, was used in this paper. Its chemical composition and physical properties are also given in Table 1, and Fig. 1 shows its XRD pattern. From Fig. 1 it can be seen that the main minerals of copper slag are fayalite (Fe_2SiO_4) and magnetite (Fe_3O_4). Pozzolanic activity of the copper slag is 76%, which was tested by using the method specified in GB/T 12597-2005 [16]. NaOH pellet with purity of 99.99% was used to prepare 6 M, 8 M, 10 M and 12 M NaOH solution to activate copper slag for the manufacture of alkali activated copper slag mortars and pastes.

China ISO standard sand manufactured by Xiamen ISO Standard Sand Co., Ltd., China, was used in this paper to prepare mortar specimens. Its chemical and physical properties meet the requirement of GSB 08-1337-2017 [17].

Water from tap was used for specimens mixing and curing.

2.2. Preparation of specimens

2.2.1. Mix proportions

Copper slag cement mortar (CCM) and alkali activated copper slag mortar (AACM) specimens were prepared for the mass loss and mechanical properties tests to be described in Sections 2.4.1 and 2.4.2. Copper slag replacements in the former were 0%, 5%, 10% and 15%, and concentrations of NaOH of 6 M, 8 M, 10 M and 12 M were adapted in the latter. Water-to-binder ratio of 0.5 was used for CCM according to Chinese standard GB/T 17671-1999 [18], while NaOH solution-to-binder ratio of 0.4 was used for AACM to minimise any possible bleeding. The proportions of the mortar mixes are given in Table 2. Further, corresponding paste specimens (CCP and AACP) were also prepared for the chemical and physical tests to be described in Section 2.4.3. Water (NaOH solution)-to-binder ratio of 0.3 was used for the paste specimens.

Table 1
Chemical composition (%) and physical properties of cement and copper slag.

	Cement	Copper slag
CaO	56.48	5.86
SiO_2	17.29	17.88
Al_2O_3	5.80	6.64
Fe_2O_3	2.94	56.16
MgO	2.92	4.66
SO_3	1.96	0.16
Na_2O	0.62	0.70
K_2O	1.61	0.90
P_2O_5	0.32	0.18
Fineness (m^2/kg)	293	256
Specific gravity	3.10	3.89

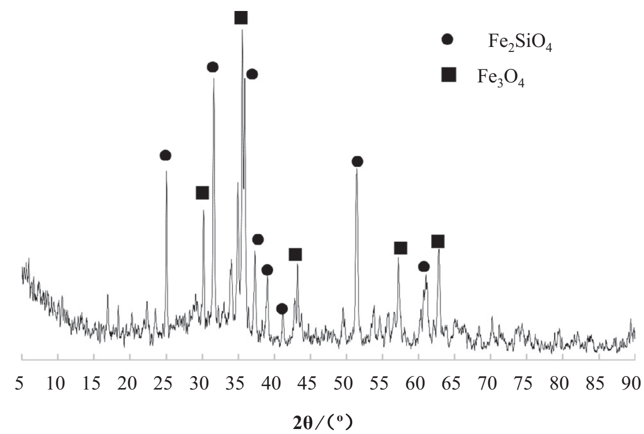

Fig. 1. XRD pattern of copper slag.

2.2.2. Preparation of specimens

Mortar specimens with size of 40 × 40 × 160 mm were manufactured according to Chinese standard GB/T 17671-1999 [18]. The size for paste specimens was 25 × 25 × 25 mm. After casting, the specimens with moulds were cured in a room with temperature of 20 ± 1 °C and RH > 95%. One day later, the specimens were demoulded and then kept in the same room up to 28 days.

2.3. Heating regime

Mortar and paste specimens were heated by using an electric heating furnace. Specimens were placed in the furnace in an unstressed condition and were then heated to 200 °C, 400 °C, 600 °C, 800 °C, 1000 °C and 1200 °C, respectively, with an increasing rate of temperature of 5 °C/min. When the targeted temperatures were achieved, the temperatures were maintained constant for 1 h to allow the specimens reaching a thermal steady state. After that, the heated specimens were cooled down slowly to room temperature in the furnace. Fig. 2 presents the heating regime described above.

2.4. Tests

2.4.1. Mass loss

After curing for 28 days, mortar specimens were taken out from curing room and the extra water on the surface of the specimens was wrapped off followed by weighing the specimens. After heating at the different temperatures, the specimens were weighed again, and a mass loss was calculated by using the mass difference before and after heating divided by the mass before heating.

2.4.2. Flexural and compressive strengths

According to the requirement specified in Chinese standard GB/T 17671-1999 [18], flexural and compressive strengths of the mortar specimens were tested at room temperature and after exposure to the high temperatures. For each mix, three duplicated specimens were tested at each temperature.

2.4.3. XRD/TG/DSC/SEM/SEM-EDS

Simultaneously with the preparation of the mortar specimens, paste correspondence specimens were also manufactured by using