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Using incinerated sewage sludge ash to improve the water resistance of magnesium oxychloride cement (MOC)



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MIS

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HIGHLIGHTS

• More phase 3 were generated when adding incineration sewage sludge ash (ISSA) in magnesium oxychloride cement (MOC).

• ISSA could react with MOC and magnesium chloride silicate alumina hydrate gel was generated.

• ISSA could increase the volume stability of MOC in water.

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ABSTRACT

This paper presents an experimental investigation on the water resistance of magnesium oxychloride cement (MOC) incorporating incinerated sewage sludge ash (ISSA). Cement pastes were prepared to evaluate the compressive strength and microstructure of the blended cements. Besides, the volume stability of cement mixture during air curing and water immersion was tested by measuring the length change of mortar bar specimens. In addition to cement paste and mortar, Na₂SiO₃·9H₂O and Al(NO₃)₃·9H₂O were used to react with MOC as the replacement of ISSA to synthesize pure hydration products. Paste studies revealed that ISSA additions of 10–30% significantly improve the water resistance of MOC. Mortar studies showed that ISSA significantly reduced expansion of mortar bars immersed in water. The improved water resistance and reduced expansion is directly related to the decrease in MgO content of pastes and an improved stability of Phase 3 (3Mg(OH)₂·MgCl₂·8H₂O) and Phase 5 (5Mg(OH)₂·MgCl₂·8H₂O) in water. Results from sol-gel experiments suggest that ISSA can act as a source of soluble Al and Si which results in the formation of an amorphous M-Cl-A-S-H type cementitious gel. The gel formed could help improve Phase 3 and Phase 5.

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1. Introduction

Magnesium oxychloride cement (MOC) [1] is an attractive cementitious material, which is used for many applications in the construction such as industrial flooring [2–4], fire protection [5], and grinding wheels due to its ability to gain strength rapidly and to withstand abrasion and high temperatures [6,7]. However, this cement exhibits poor water resistance as the decomposition of the main hydration products $3Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (Phase 3) and $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ (Phase 5) leading to the sharp decrease of compressive strength [8]. Previous results indicate that the compressive strength of MOC paste decreased by about 90% when soaked in water for 28 days. As a result, the common application of this cement is only limited to indoor environments. In order to

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http://dx.doi.org/10.1016/j.conbuildmat.2017.04.187 0950-0618/© 2017 Elsevier Ltd. All rights reserved. widely utilize MOC, different approaches have been adopted to improve the water resistance, among which adding additives and supplementary cementitious materials are the most effective methods [9,10]. It was reported that the compressive strength of MOC immersed in water for 28 days was decreased by only 10% and 15% when adding 1% soluble phosphates [11] and 30% fly ash respectively [6]. Some researcher concluded that the phosphates could react with magnesium to produce insoluble hydrated products such as magnesium phosphate that protected the magnesium cement crystal from decomposing [12]. However, it was argued that the quantity of the insoluble phosphates was not high enough to produce a layer of the insoluble phosphates [11]. The mechanism of the improvement needs further research. On the other hand, it was reported that fly ash could improve the water resistance of MOC due to formation of amorphous gel formed by the reaction between fly ash and MOC [13].

As a consequence of waste water treatment, a large amount of sewage sludge is produced every day in urban municipalities in Hong Kong. Traditional landfilling method is not acceptable to manage this waste as it adversely impacts human health and the environment [14,15]. An alternative for sewage sludge disposal is incineration, which can achieve significant reductions of material volume [16] and leaving only a small quantity of incinerated sewage sludge ash (ISSA) which is particularly important for a small city like Hong Kong. The incineration process at T-Park reduces the volume of the waste in Hong Kong. But the residual ISSA still amounts to about 100-200 tonnes/day which is currently disposed of at landfill. Many green approaches have been developed to recycle the ISSA. Test results indicated that the major elements in ISSA are Si, Al, Ca, Fe and P [17,18]. ISSA can be used to manufacture bricks [19–21], tiles [22], and lightweight aggregates [23,24], etc. Additionally, a number of investigations have evaluated the potential of ISSA as a supplementary cementitious material due to a presumed moderate pozzolanic activity [25,26]. However, it should be noted that factors such as fineness [27], acid pre-treatment [28] and test method used [29] will significantly influence any assessment of the pozzolanic activity of ISSA.

Considering the moderate pozzolanic nature and its high phosphate content, ISSA may be useful to improve the water resistance of MOC. However, no previous study was conducted before on this. The aim of this study is to evaluate the water resistance of MOC incorporating ISSA. The mechanical properties of MOC pastes prepared with different percentages of ISSA (0, 10, 20 or 30%) were studied. Mortar specimens were used to evaluate the volume stability of MOC during air curing and after water immersion. Due to the chemical complexity of ISSA-MOC systems, a series of simplified sol gel experiments were conducted where ISSA was substituted for sources of soluble silicate ($Na_2SiO_3.9H_2O$) and soluble aluminate ($Al(NO_3)_3.9H_2O$) in order to assess what type of cementitious gel phases may form. The compositions and microstructure of the sample were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

The MOC specimens were prepared by mixing various amounts of light-burned magnesia powder (MgO) and a bischofite (MgCl₂·GH₂O), which were obtained from Liaoning and Qianghai provinces in China, respectively. The ISSA was obtained from the sewage sludge incineration center-T park, HK. Table 1 shows the mineralogical composition of ISSA. The main crystalline phases determined through X- ray diffraction were quartz, magnetite and albite. The relatively high amount of amorphous phase detected by quantitative XRD suggests that there are glassy phases in ISSA that could act as sources of soluble Si and Al, resulting in some pozzolanic behavior of the ash. The chemical analysis of MgO and ISSA are shown in Table 2, and the particle size distribution curve of ISSA is shown in Fig. 1 measured by the particle-sizing instrument (The Coulter LS230). The fine aggregate used was a standard quartz sand with particle size ranging from 0.5 mm to 1 mm. The pure chemical reagents Na₂SiO₃·9H₂O and Al(NO₃)₃·9H₂O were used to react with MOC as a replacement of ISSA.

2.2. Sample preparation

2.2.1. Cement paste and mortar

Cement paste specimens of size $20 \times 20 \times 20$ mm were produced with a MgO/MgCl₂ molar ratio of 9 and a water/MgCl₂ molar ratio of 10. Different dosages of ISSA (10%, 20%, 30% by mass of MgO) were added to the paste and the mortar specimens as the replacement of MgO. The mix proportions of the paste are shown in Table 3. Cement mortar prisms specimens of size $25 \times 25 \times 25 \times 285$ mm were prepared to record the volume stability of MOC during air curing and water soaking. The mix proportions of the mortar were similar to that of the pastes except that the sand to powder weight ratio was 1.5 to obtain good workability. The fresh cement paste and mortar samples were covered with polyethylene sheet to prevent evaporation. After 24 h, the specimens were removed from the mould and cure in air curing chamber (25 °C, RH = 50%) followed by water immersion at room temperature for different days.

XRD-Rietveld analyses of the ISSA (wt.%).

	%
Quartz(SiO ₂)	28.17
Magnetite(Fe ₃ O ₄)	14.31
Albite(NaAlSi ₃ O ₈)	8.82
Leucite(K(AlSi ₂ O ₆))	2.46
Hematite(Fe ₂ O ₃)	1.22
Amorphous	45.03

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Elemental	composition	of MgO	and ISS	A (wt%)
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	MgO	ISSA
MgO	94.86	3.21
SiO ₂	2.75	28.34
AlO ₃	-	12.44
Fe ₂ O ₃	0.45	18.60
CaO	1.60	10.63
Na ₂ O	-	7.43
K ₂ O	-	1.92
SO ₄	0.24	6.22
Others	0.10	11.21
Total	100.00	100.00



Fig. 1. Granulometric distribution curve for ISSA.

Table 3Mix proportions of MOC paste.

Mixtures	Mix proportion in mass (g)			Molar ratios		
Notation	MgO	MgCl ₂ ·6H ₂ O	H_2O	ISSA	MgO/MgCl ₂	H ₂ O/MgCl ₂
SA0	360	202	72	0	9	10
SA1	324	202	72	36	8.1	10
SA2	288	202	72	72	7.2	10
SA3	252	202	72	108	6.3	10

2.2.2. Sol-gel experiments to study pure hydration products Three sets of solution were prepared as follows:

Solution A: Magnesium oxide (20 g) was dissolved in 200 ml of 2.5 M $\rm MgCl_2$ aqueous solution

Solution B: Magnesium oxide (20 g) was dissolved in 200 ml of 2.5 M $MgCl_2$ aqueous solution followed by adding 0.5 mol of $Na_2SiO_3.9H_2O$ and 0.5 mol of $Al(NO_3)_3.9H_2O$

Solution C: Magnesium oxide (20 g) was dissolved in 200 ml of deionized water followed by adding 0.5 mol of Na_2SiO_3 ·9H₂O and 0.5 mol of $Al(NO_3)_3$ ·9H₂O

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