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Short communication

Preparation of drying powder inorganic polymer cement based on alkali-activated slag technology

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ABSTRACT

In the alkali activated inorganic polymer preparation process, liquid water glass is used as an alkali-activator to dissolve the solid aluminosilicate precursor and form the cement material. However, liquid water glass is not convenient for storage or transportation. Developing an inorganic polymer-based powder cement (IPBPC) as a one-part mixture ("just add water"), similar to Portland cement, increases its commercial viability. Through compressive strength and heat flow analysis, this paper presents research on the mechanical properties and reaction process of slag with solid or liquid water glass; in contrast, solid water glass has better mechanical performance. The $L_9(3^4)$ orthogonal experiment method is used to optimize the experimental formula. According to the range analysis and SEM, this result indicates that the level of significance of factors are as follows: m(SL)/m(WG) > fly ash > water > retarder; m(SL)/m(WG) has the most significant impact on the 7-day compressive strength of the IPBPC sample. The ^{29}Si solid-state NMR patterns demonstrate that in the IPBPC sample with m(SL)/m(WG) = 5:1, primarily Q_2 and $Q_2(1Al)$, the Al can incorporate into the gel. The optimum formula is as follows: m(SL)/m(WG) = 5:1, retarder 7%, fly ash 10%, and water 28%. The 7-day compressive strength of the optimum composition is 67.38 MPa, the initial setting time is 150 min, and the final setting time is 230 min and meets the construction conditions.

1. Introduction

Currently, cement and concrete are the most widely used man-made building materials [1,2]. It is estimated that cement and concrete are responsible for an average of 5% of the CO_2 emissions in developed countries and can be as high as 10% in developing countries [2–4]. In the last decade or so, extensive research has been undertaken by academics and engineers alike, with the view to find a new type of material as an alternative to Ordinary Portland cement (OPC) concrete for more sustainable construction. This type of new material is called alkali activated cement materials (or inorganic polymer) [2–4].

"Geopolymers" concept was first introduced as a type of inorganic polymer material by Joseph Davidovits in 1978 [5]. Geopolymers are a type of three-dimensional structural aluminum silicate inorganic polymer that is composed of the $[AlO_4]$ and $[SiO_4]$ tetrahedron, which are mainly prepared by the aluminosilicate mineral (such as metakaolin) or industrial waste (such as fly ash and ground granulated blast-furnace slag) [6–8]. Due to their unique, three-dimensional oxide network structure from inorganic polycondensation, inorganic polymers possess favorable features, such as high strength [9,10], corrosion resistance [11–14], water resistance [15], high temperature resistance [16], enclosed metal ion [17], vacuum stability and freeze-thaw cycles [18,19], and many other advantages [20–23]. Inorganic polymer cement materials have broad applications in concrete, mortar, and engineering because of the excellent properties of the material has mentioned above. It is well known that Portland cement is invented, and the cement and concrete industry has been faced with environmental and energy intensity issues [2–4]. Both of these issues are now becoming more serious and a point of international interest as the world begins to move towards the concept of sustainable development [2–4]. Therefore, inorganic polymers could be used as a new material to improve Portland cement.

So far, many studies have been carried out on the behavior of slag-based inorganic polymer paste, mainly using liquid water glass as alkali-activator, while the liquid water glass samples performance is excellent [24,25]. But liquid water glass is corrosive and is greatly influenced by the storage temperature and storage time that is not easy to store and transport, making it difficult to use for bulk production. In this study, we mainly use solid water glass and slag as raw materials to prepare a slag-based inorganic polymer paste. Because steel production increases year to year, the production of slag has increased. The preparation of inorganic polymer concrete using slag as the raw material not only reduces the impact on the environment but also can reduce

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CO_2 emissions [26–28]. Meanwhile, solid water glass is easy to store and transport, it provides more convenience for this large-scale production and the application of slag-based inorganic polymer paste in the future.

Despite these advantages, there are also disadvantages. This fast solidification property would not benefit the inorganic polymer paste storage after mixing inorganic polymeric precursors with an alkali-activator. In this paper, by adding fly ash [29], water [30] and retarder [31] to control the inorganic polymer powder cement setting time, we can obtain better construction conditions [32]. Most of the inorganic polymer cement material is thus two-part product ("liquid activator + solid composition"), and it is not easy to package and transport. Developing an inorganic polymer-based powder cement (IPBPC) as a one-part mixture ("just add water"), similar to Portland cement, increases its commercial viability.

2. Experimental procedure

2.1. Materials

The primary raw materials used in this study were provided by local suppliers. The chemical compositions of slag and fly ash (Chengde Group Company, Beihai Guangxi, P.R. China) given by X-ray Fluorescence are summarized in Table 1 and Table 2. The specific surface area of the slag and fly ash were 1.44 m^2/g and 4.5 m^2/g, respectively. The slag and fly ash used in these experiments were dried by the electro-thermal constant-temperature dry box at 105 °C for 3 h and were subsequently cooled to room temperature. The alkali-activators used were solid water glass that was obtained from industrial-grade water glass (Zhongfa Water Glass Factory, Foshan Guangdong, P.R. China) comprising 50.0–52.0% SiO_2, 26.0–28.0% Na_2O, and 20.0–24.0% H_2O; molar ratio: $SiO_2/Na_2O = 2$. The specific surface area of the water glass was 1.24 m^2/g, and the liquid water glass (Chunxu Chemical Company, Nanning Guangxi, P.R. China) had a solid content of 37.3% and molar ratio of $SiO_2/Na_2O = 3.30$. The retarder was hydrophosphate (AR, Guangdong Xilong Chemical Company, China).

2.2. Preparation of IPBPC samples

Because of slag, water glass and water mixture are an exothermic reaction, the slurry would rapidly solidify. First, the slag, sodium silicate and water used in these experiments were cooling in a refrigerator at 0 °C for 3 h. Secondly, the IPBPC was quickly mixed with water, the fresh mixtures were prepared with intensive mixing for 2 min using an electrical mixer at 2000 rpm. Thirdly, the samples were cast in alloy molds, 20 × 20 × 20 mm-edge cubes. A thin layer of oil was sprayed into the alloy molds prior to filling to aid in the removal of the hardened paste upon curing. The alloy molds were vibrated on a vibration table for 2 min to remove any air bubbles and sealed immediately afterwards. A total of all mixtures were prepared, and 6 samples were cast from each mixture.

2.3. Single-factor and orthogonal experimental design

In this paper, single-factor and orthogonal experiments were designed. The single factor mainly includes the water glass content (A), fly ash content (B), retarder content (C) and water content (D). Experimental formula A: the mixed proportions of samples were mass ratio: slag/water glass = 3:1, 4:1 and 5:1. Experimental formula B: the mixed proportions of samples were mass ratio: fly ash/(fly ash + slag) = 10%, 20% and 30%. Experimental formula C: the mixed proportions of samples were mass ratio: retarder/water glass = 6%, 7% and 8%. Experimental formula D: the amount of water was 28%, 29% and 30% of the total mass of slag and water glass. Here, an orthogonal experimental design method was applied to discuss the impact degrees of A, B, C and D to the 7-day compressive strength of IPBPC for selecting the optimum formula. A, B, C and D were determined as four factors of the orthogonal experiment, and each factor had three levels. The three levels were selected from the better content in the single-factor experiment. It was assumed that any two factors did not interact with each other. The orthogonal array of the 9 IPBPC samples is shown in Table 3, designed according to the orthogonal design table $L_9(3^4)$. The three compressive strengths of each factor at the same level i were summed, and the corresponding average value K_i and range R were calculated, respectively, as follows:

$$R = K_{max} - K_{min} \quad (1)$$

K_i represents the impact of the level i of each factor to the compressive strength of the IPBPC ($i = 1, 2, 3$). The higher the K_i, the better the compressive strength of the IPBPC. K_{max} is the maximum among the three K_i values of each factor, and K_{min} is the minimum. R reflects the impact degrees of the factors to the compressive strength of the IPBPC. The factor having the higher R suggests a stronger impact on the formation of the compressive strength of the IPBPC.

Table 1
Chemical composition of slag/wt%.

Component	CaO	SiO_2	Al_2O_3	MgO	SO_3	TiO_2	MnO	Fe_2O_3	K_2O	Na_2O
Content	36.91	33.98	15.22	9.27	1.81	0.80	0.59	0.62	0.41	0.39

Table 2
Oxide composition of fly ash/wt%.

Component	SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	TiO_2	CaO	SO_3	LOI[a]
Content	57.67	28.50	5.22	4.00	1.25	1.15	0.91	0.53	0.67

[a] Loss on ignition.

2.4. Setting time testing

The setting time of the IPBPC at 25 °C was measured using a Vicat apparatus (RV-300B, China).

2.5. Compressive strength testing

Compressive strength was determined according to standard ASTM C 733 using a DNS100 (Changchun institute of testing machine) universal testing machine. The displacement rate used was 0.5 mm/min. To obtain the average compressive strength, six samples from each mixture were tested.

Table 3
Results and range analysis data of $L_9(3^4)$ orthogonal matrix test.

Factor number	m(SL)/m(WG)	Retarder/%	Fly ash/%	Water/%	Compressive strength for 7 d/MPa
	A	B	C	D	
N1	4:1	6%	10%	28%	57.64
N2	4:1	7%	20%	29%	51.56
N3	4:1	8%	30%	30%	38.02
N4	5:1	6%	20%	30%	50.55
N5	5:1	7%	30%	28%	56.42
N6	5:1	8%	10%	29%	57.83
N7	6:1	6%	30%	29%	26.52
N8	6:1	7%	10%	30%	38.50
N9	6:1	8%	20%	28%	37.98
K_1	47.08	44.91	51.32	50.69	
K_2	54.93	48.82	46.70	45.30	
K_3	34.33	44.61	40.32	42.36	
R	20.60	4.21	11.00	8.33	