Construction and Building Materials 91 (2015) 138–144

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Influence of sodium gluconate on the performance and hydration of Portland cement

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HIGHLIGHTS

- The optimum dosage of Na-gluconate was 0.03% for the compressive strength.
- Na-gluconate reduces the amount of water required, and prolongs setting times.
- Na-gluconate delays the hydration of C_3S, prolonging the induction period.
- Less than 0.03% Na-gluconate promotes the formation of AFt.
- 1.0% Na-gluconate inhibites the dissolution of $CaSO_4{\cdot}2H_2O$ and formation of AFt.

ARTICLE INFO

Article history:
Received 9 September 2014
Received in revised form 31 March 2015
Accepted 1 May 2015
Available online 16 May 2015

Keywords:
Sodium gluconate
Portland cement
Physical properties
Structure development

ABSTRACT

Sodium gluconate is commonly used to delay the setting of cement and concrete. This study investigated the effects of sodium gluconate on the physical properties and structure of Portland cement. Sodium gluconate improved the compressive strength at 3 and 28 days, delayed cement setting and increased the fluidity of the Portland cement mortar. Less than 0.03% sodium gluconate promoted the formation of ettringite (AFt) at early age. A dosage of 1.0% sodium gluconate significantly inhibited the reaction between C_3A and $CaSO_4{\cdot}2H_2O$. Sodium gluconate delayed the hydration reaction of C_3S, which increased the duration of the induction period. Sodium gluconate had only a slight effect on the hydration reaction of the ferrite phase. The pore distribution and porosity of the cement paste were not improved due to the decrease in hydration.

1. Introduction

Sodium gluconate is a crystalline powder that can be produced under properly controlled conditions. It is widely used in industry. It can be utilized as a chelating agent in buildings, textile printing and dyeing, metal surface treatment and water treatment, as a cleaning agent for steel surfaces and glass bottles and as a retarder for concrete. Concrete is the largest class of manufactured materials. It comprises cement, aggregates, admixture and water. One of the most common types of admixture in concrete is a superplasticizer admixture. Many studies [1–3] have shown that the combination of sodium gluconate with a superplasticizer has a significant effect on the slump loss and fluidity of concrete because sodium gluconate has a good retardation action [4]. With respect to the retardation mechanism of sodium gluconate, there are several popular explanations as followed: (1) the formation of AFt is inhibited by the adsorption of gluconate or the complex between gluconate and calcium ions (Ca^{2+}) on the surface of C_3A [5–8]; (2) low concentrations of gluconate can block the nucleation and/or growth of hydration products [9–10]; (3) the retardation action of gluconate may occur via adsorption on the surface of the silicate phase [11]. However, the improper use of sodium gluconate often causes abnormal setting and large slump losses [12], which results in great economic loss. It was reported that there was a threshold dosage of gluconate needed to obtain cement fluidity after five minutes of hydration [11]. However, the threshold dosage has not been precisely determined. The work described in this paper aims to answer the following questions:

– What is the threshold dosage of sodium gluconate necessary to increase fluidity?
– How do hydrate products develop in the cement paste with sodium gluconate?

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http://dx.doi.org/10.1016/j.conbuildmat.2015.05.068
0950-0618/

– What on earth is the retardation mechanism of sodium gluconate?

2. Experimental

2.1. Materials

2.1.1. Raw materials

The materials used in the experiments included conventional Portland cement, standard sand and sodium gluconate. The chemical compositions of the cement, which was obtained from the Yangzhou Yadong Cement Co. Ltd., are shown in Table 1. The surface area of the cement was 360 m^2/kg measured by Blaine method. The cement was a mixture of the clinker, gypsum and fly ash. The mineral compositions of the clinker are shown in Table 2. The standard sand (Chinese Standard GB/T17671) was obtained from China ISO Standard Sand Co. Ltd. Sodium gluconate was from Sinopharm Chemical Reagent Co. Ltd., and its purity was 99%.

2.2. Experimental methods

2.2.1. Mechanical property

The compressive strengths of the mortars were measured according to Chinese Standards GB/T17671-1999. Mortars were prepared with standard sand in accordance with Chinese Standard GB/T17671 for compressive strength. Cement/sand/water weight ratios of 1:3.0:0.5, respectively, were employed.

2.2.2. Physical properties

The fluidities of the mortars were measured according to Chinese Standards GB/T 2419-2005. The preparation protocol for the mortars is the same as the above samples for compressive strength. The normal consistency and setting times of the cement pastes were determined in accordance with Chinese Standard GB/T 1346-2001 using a Vicat apparatus.

2.2.3. Hydration heat flow

A conduction calorimeter (TAM Air from Thermometric AB, Sweden) operating at 20 °C was used to measure the hydration heat flow of the cements. At first, sodium gluconate was dissolved in water. The mass ratio of water to cement is 0.5. Four grams of cement was weight into the sealed plastic ampule. After the solution was added into the cement, they were mixed for 2 min by hands. Finally, the paste was put into the calorimeter as soon as possible and measured over a period of 3 days.

2.2.4. Analysis of the solid phases assemblage

The pastes were made from the cement and aqueous solution containing different dosages of sodium gluconate. The mass ratio of water to cement is 0.5. After the aqueous solution was added into the cement, they were mixed for 2 min by using mixer with speed of about 1600 r/min. The fresh paste was poured into plastic bottles. The bottles were sealed, and then stored in a curing box with the temperature of 20 °C ± 1 °C and humidity of 95% ± 4%. Prior to the X-ray diffraction (XRD) and Differential Scanning Calorimetry (DSC), hydration was stopped after 30 min, 1 h, 2 h, 5 h, 8 h, 1 d, 3 d and 28 d by submerging small pieces for 7 d in alcohol. And then they were dried in a oven. Some pieces were ground and stored in the desiccator for XRD and DSC.

X-ray diffraction (XRD) analysis was used for phase identification. Finely ground clinker was back loaded into sample holders to minimize preferred orientation. XRD patterns were recorded on the Rigaku SmartLab 3000 A diffractometer with CuKα 1,2 radiation (λ = 0.154 nm). The X-ray tube was operated at 35 kV and 30 mA. The optics configuration was a fixed divergence slit (1/2°) and a D/teX Ultra detector. Measurements were made in a θ–θ reflection geometry. Data were collected from 10° to 70° in continuous made.

The thermal analysis was performed on a NETZSCH STA-449C equipment with simultaneous TG and DSC system. Some parts of the above powder was carried out at a heating rate of 10 °C/min from 30 °C to 1000 °C under N2 atmosphere.

2.2.5. Analysis of pore size distribution

The study on porosity and pore size distribution was carried out on a Quanta chrome PoreMaster GT60 mercury intrusion porosimeter capable of generating high pressure in the range of 20 psia to 20,000 psia and low pressure in the range of 1.1 psia to 20 psia. It can be used to measure pore size in the range of 0.0035 μm to 400 μm. The pore radius was calculated according to the Washburn equation, $r = -2\gamma\cos\theta/P$, where r is the pore entry radius in which mercury is being intruded, γ is surface tension, θ and P are contact angles of mercury with the solid and applied pressure, respectively.

Table 2
The mineral compositions of the cement clinker calculated by Bogue.

C_3S	C_2S	C_3A	C_4AF	f-CaO
56.6	20.5	7.3	9.6	1.36

3. Results

3.1. Compressive strength

Fig. 1 shows the compressive strength and rate of strength development for cement mortars. Sodium gluconate enhances both the early strength and late strength, with the exception of the 0.05% dosage (see Fig. 1(a)). The optimum dosage of sodium gluconate is 0.03% for conventional Portland cement, as assessed by the strength of the cement mortar. The compressive strengths are increased by approximately 10% and 6% respectively at 3 and 28 days, compared to the blank cement mortar. The compressive strength began to decrease when the dosage of sodium gluconate is greater than 0.03%. Two separate domains are identified for the rate of strength development between 3 d and 28 d (see Fig. 1(b)). In the first one, the rate of strength development decreases gradually for extremely low dosages. In the second one, the rate of strength development increases with increasing the dosage of sodium gluconate. The above results illustrates that sodium gluconate has a more significant effect on the early strength at less than 0.03%, relative to the late strength. Above this dosage, the effect is mainly on the late strength.

3.2. Normal consistency and setting time

The effects of sodium gluconate on the normal consistency and setting times of the conventional Portland cement are shown in Fig. 2. Sodium gluconate had a significant effect on the setting times. Both the initial setting time and final setting time are prolonged with an increase in the dosage of sodium gluconate. When the dosage of sodium gluconate is less than 0.01% by weight of cement, it has a more significant effect on the finial setting time, compared to the initial setting time. Beyond this dosage, it has a greater affect on the initial setting time. Hence, the difference between the initial setting time and finial setting time is decreased with the increasement of sodium gluconate. On the other hand, sodium gluconate significantly reduces the amount of water required, except for the dosage of 0.01% (see Fig. 2). Thus, it is not only a strong set retarder but also an effective water reducing agent. Sodium gluconate is commonly used as a major component of commercial water reducing admixture to improve the flow properties of concrete.

3.3. Fluidity of cement mortars

The mortar flow is expressed as a spreading measurement after different hydration ages. Fig. 3 depicts the fluidity and fluidity loss of the cement mortars. At any hydration age, sodium gluconate improves the fluidity of the blank cement mortar. It shows further

Table 1
The chemical compositions of cement measured by X Ray Fluorescence (XRF) wt./%.

LOI	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	K_2O	Na_2O	TiO_2	P_2O_5	MnO	Sum
2.67	56.37	24.38	7.20	3.48	1.21	2.82	0.81	0.12	0.32	0.09	0.07	99.54