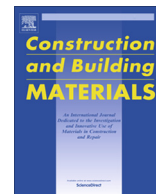




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Comparison of the retarding mechanisms of sodium gluconate and amino trimethylene phosphonic acid on cement hydration and the influence on cement performance

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HIGHLIGHTS

- The retarding mechanisms of the two retarders on cement hydration are different.
- ATMP performs better than SG in terms of cement hydration cumulative heat reduction.
- The long-term mechanism performance is retained even their dosages are less than 0.15%.
- The use cases of the two retarders are extensively studied.

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ABSTRACT

ATMP and SG were effective hydration retarders of cement. The goal of this study was to provide the comparison of the influence of ATMP and SG on the performance and hydration of Portland cement. The setting time at 20 °C and 35 °C, compressive strength and fluidity of cement pastes containing either ATMP or SG at different dosages from 0.02% to 0.10% were tested respectively. The hydration behaviors of Portland cement with ATMP and SG were investigated by the means of isothermal calorimetry measurements, X-ray diffraction (XRD), thermogravimetric analysis (TGA) and scanning electron microscope (SEM). The results showed that the retarding effect of ATMP was much intensive than SG, especially in the case of a higher temperature; the “dispersion loss resistant” of ATMP were superior to SG; The limitation dosage of ATMP and SG was 0.15%; Although SG could reduce cement hydration cumulative heat and hydration evolution rate at some degrees, the effect was much weaker than ATMP. ATMP continued to inhibiting the hydration of C₃S effectively during the whole hydration period. SG was also inhibiting the hydration of C₃S during the hydration of 1d, but it promoted the hydration of C₃S beyond 1d at some degrees.

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1. Introduction

Retarders were required to delay the setting time of concrete and to improve its workability duration without affecting the long-term mechanical properties. Retarders were usually used for long-distance transportation and pouring concrete in hot climates [1], mass concrete construction [2], roller compacted concrete (RCC) construction [3] and oil or gas well cementing operation [4,5].

Organic phosphonic acid was widely used in industrial circulating cooling water system [6], abrasive free slurry for copper polishing [7] and surface modifications of self-assembly thin film [8]. It

performed good chemical stability, strong cationic chelating ability, excellent corrosion inhibitory and scale resisting effect. Ramachandra [9] was first proposed that the retarding effect of organic phosphonic acid on Portland cement was more intensive than conventional retarders. More importantly, the retarding effect of organic phosphonic acid on Portland cement could be maintained in high temperature, therefore organic phosphonic acid was suggested for using in high-temperature oil and gas plugging operation. However, the mechanism of organic phosphorous acid delayed cement hydration was not yet completely understood, two different viewpoints of the retarding mechanism of organic phosphonic acid on Portland cement hydration were presented. Peter V. Coveney [10,11] and J. Billingham [12] proposed that organic phosphonic acid inhibited the process of Aft crystallisation and crystal growth. According Joerg Rickert [13] organic

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phosphonic acid severely delayed the hydration of C_3S , inhibited the formation of portlandite and calcium silicate hydrate gel (C-S-H). But the formation of AFt was less affected, even contributed to the hydration of C_3A at some degrees.

SG was a commonly retarder used in real concrete due to the effective retardation action for cement, dispersion ability, good adaptability with superplasticizer system and economic price [14]. Some studies of SG on dispersion and strength of cement were deeply investigated [15,16]. Meanwhile, some studies suggested that the effect of SG on the performance and hydration of Portland cement was sensitive to its dosages. According Ma et al. [17,18] SG promoted the formation of ettringite (AFt) at early age when the dosage was less than 0.03%, nevertheless, it inhibited the formation of AFt when its dosage more than 0.05%. Perez [19] proposed that at a lower dosage, SG was not completely capable of neutralize all the active dissolution sites of C_3S , resulting in a slight retarding effect on hydration. At a higher dosage, SG could be apparently adsorbed on C_3S surfaces, inducing a great inhibition of the hydration. Likewise, the mechanism of SG retarded the cement hydration was not conclusive. SG mainly delayed the hydration of C_3S , which increased the duration of the induction period. However some different views [20,21] proposed that SG delay the cement induction period, whereas it accelerated the hydration of C_3S in the hydration acceleration period.

ATMP was a kind of organic phosphonic acid, the influence of ATMP on the performance and hydration of Portland cement had been less investigated in previous studies. Furthermore, the comparison of the influence of ATMP and SG on the performance and hydration of Portland cement had been never investigated. This study provided a better understanding of the performance and mechanisms by which ATMP and SG retarded cement hydration, leading to a better prediction of the retarder dosage for cement and laying the foundation for how to choosing the two different retarders applications in real concrete construction projects.

The comparison of ATMP and SG on cement setting time at 20 °C and 35 °C, cement paste compressive strength and cement paste fluidity were discussed in this paper. The effects of ATMP and SG on hydration behaviors of Portland cement were investigated by the means of isothermal calorimetry measurements, X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscope (SEM).

2. Experimental

2.1. Materials

The chemical compositions of the cement were shown in Table 1. The ATMP (commercially available) was slightly yellow

Table 1
Chemical composition of cement (wt%).

SiO ₂	CaO	f-CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	Na ₂ O	Loss [*]
21.02	61.41	0.97	4.80	3.68	1.03	2.02	0.60	0.02	3.78

^{*} Loss on ignition of cement.

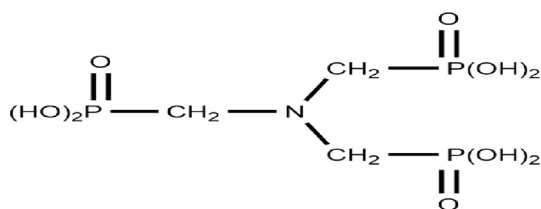


Fig. 1. Structural formula of ATMP.

Table 2
Parameters of ATMP (wt%).

Active Component	PO ₄ ³⁻	PO ₃ ³⁻	Cl ⁻	Fe ³⁺
50.84	0.77	2.47	1.61	8.38×10^{-3}

transparent liquid, the density was 1.37 g/cm³, the molecular formula of was N(CH₂PO₃H₂)₃ and the structural formula of ATMP was shown in Fig. 1. The parameters of ATMP were shown in Table 2. The purity of SG (commercially available) was 99%. The solid content polycarboxylate superplasticizer (commercially available) of was 40%.

2.2. Experimental methods

2.2.1. Physical and mechanical properties

The measurements of cement pastes setting time were tested using vicat apparatus according to Chinese Standards GB/T 1346-2011. The curing conditions of cement pastes were the humidity of 95%, the temperature was 20 °C and 35 °C respectively. The addition of ATMP and SG were 0.02%, 0.04%, 0.06%, 0.08%, 0.10%, 0.15%, 0.20%, 0.30% and 0.40% respectively by the mass of cement. The retention percentage of cement setting time at 35 °C was characterized by following (1):

$$S_r(\%) = 100 \times \frac{T_{(35)}}{T_{(20)}} (\%) \quad (1)$$

$S_r(\%)$ was percentage of retention of cement setting time at 35 °C, $T_{(35)}$ was cement setting time at 35 °C, $T_{(20)}$ was cement setting time at 20 °C.

Cement pastes were prepared for compressive strength using paste mixer. The water to cement ratio was 0.27 and the size of samples were 40 mm × 40 mm × 40 mm. The compressive strength of 3d, 7d and 28d were tested. The addition of ATMP and SG were 0.02%, 0.04%, 0.06%, 0.08%, 0.10%, 0.15% and 0.20% respectively by the mass of cement.

The measurements of cement pastes fluidity were prepared by mixing 0.34% (by the mass of cement) superplasticizer and different additions of ATMP, SG according to Chinese Standards GB/T 8077-2000. The addition of ATMP, SG were 0.06% and 0.10% respectively by the mass of cement. The water to cement ratio was 0.29, the cement and water were 300 g and 87 g respectively. The fluidity of cement pastes were measured at 0, 30, 60 and 120 min after mixing, the curing condition was under humidity of 95% and temperature of 20 °C.

2.2.2. Hydration heat flow

The cement hydration heat flow was measured by means of the microcalorimeter with 8 channels (TAM air from TA instruments, United States) at 20 ± 1 °C. All samples were 2.000 g and the ratio of water to cement was 0.30. The heat flow was recording by computer and the period of measurement during 0–168 h.

2.2.3. Micro analyses of the hardened cement pastes

Cement pastes were prepared for X-ray diffraction (XRD), thermogravimetric analysis (TGA), scanning electron microscope (SEM) analyses on water to cement ratio 0.30. The hydration of hardened cement pastes were stopped after predetermined curing age by breaking test block into small pieces in absolute ethyl alcohol. The small samples were ground into less than 80 μm powder by agate grinding for XRD and TGA analyses. The samples of size range from 5 mm to 8 mm were selected for SEM analyses. The mix proportions of cement paste were shown in Table 3.

XRD was employed to identify the cement hydration products phase. It was used method of semi-quantitative analysis to study

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