

Effects of superplasticizers on the stability and morphology of ettringite



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HIGHLIGHTS

- The stability of hydration products of C₃A with gypsum was investigated.
- The changes of ettringite crystallization were investigated by XRD and SEM.
- The bonding state was investigated via XPS.
- The changes of stabilities of ettringite vary in the presence of superplasticizers because of the formation of morphologies.
- The superplasticizers and the formed ettringite exhibited certain interactions.

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ABSTRACT

Stability and morphology of ettringite with and without four different superplasticizers, namely, polycarboxylic acid, naphthalene sulfonate, amino sulfonate, and aliphatic superplasticizers, were investigated on hydration and synthetic samples. Thermogravimetric analysis, X-ray diffraction, scanning electron microscopy, and X-ray photoelectron spectroscopy were used for analysis. The stability of ettringite increased when naphthalene sulfonate superplasticizer was used, whereas the stability decreased when the three other superplasticizers were applied. The stability further increased because of the morphology of the formed ettringite crystals. In addition, certain chemical reactions occurred between ettringite and the superplasticizers, particularly the polycarboxylic acid. Some calcium complex chelate compounds were also produced, and the hydration products obtained with polycarboxylic acid and naphthalene sulfonate were more compact and stable than those obtained with the two other superplasticizers.

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1. Introduction

Ettringite is formed by tricalcium aluminate (C₃A) with gypsum or hydrated sulfoaluminate cements; its formation is an important reaction associated with retardation or rapid hardening of fresh portland cements, sulfate attack on hardened Portland cements, high early strength of supersulfated cements, expansion properties, and self-stressing cements [1,2]. Ettringite is an unstable phase and it decomposes to form the monosulfate hydrate at curing temperatures higher than 60 °C; sulfate ions are released by the decomposition [3]. During service time, the reformation of ettringite (delayed ettringite) in the hardened concrete causes expansion and cracking when sulfate ions are desorbed [4].

Superplasticizers are widely used to produce flowable, strong, and durable Portland cement concretes and mortars. Superplasticizers can adhere to certain faces of hydration product

crystals and prevent or slow the addition of new growth layers on those faces [5]. Different superplasticizers have different adsorption properties on cement minerals; aluminate minerals have considerably better adsorption properties than silicate minerals [6]. Ettringite is the main hydration product of aluminate minerals. The effects of superplasticizers on the morphology and stability of ettringite are related to the thermal stability of self-stressing cement, as well as fluidity, setting time, strength development, and durability of concrete [7,8]. Therefore, investigating the effects of superplasticizers on the stability and morphology of ettringite is important.

2. Experimental methods

2.1. Materials

Ettringite was prepared by hydrating the mixture of C₃A and 3% gypsum with water to a binding material ratio of 0.29 at 28 days of age; this mineral can also be synthesized in the Ca(OH)₂ and Al₂(SO₄)₃·18H₂O solution with a molar ratio of 6:1 and water to solid ratio of 4:1, subsequently dried using a vacuum drying oven.

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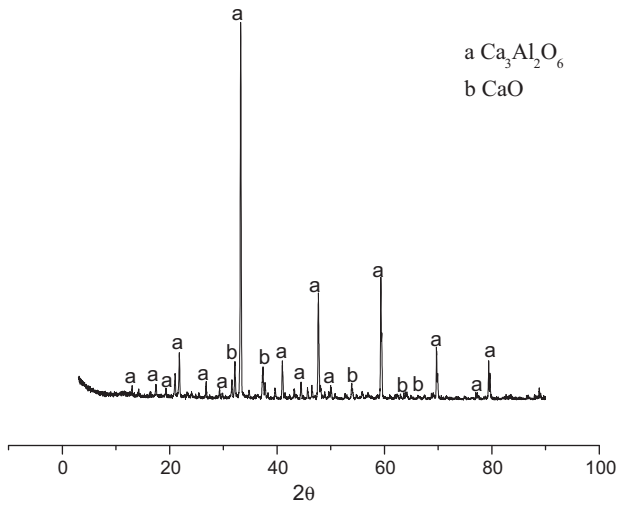


Fig. 1. X-ray diffraction pattern of synthetic C_3A powder.

C_3A was synthesized from $CaCO_3$ and Al_2O_3 via rapid cooling after reacting raw materials at $1350\text{ }^\circ\text{C}$. The mineralogical components of these materials were identified using X-ray diffraction (XRD). XRD result showed that the f -CaO component was less than 1% (Fig. 1).

Four types of superplasticizer, namely, polycarboxylic acid (PC), naphthalene sulfonate (NS), amino sulfonate (AS), and aliphatic (AH) superplasticizers, were used in this study. The solid contents of these superplasticizers are as follows: PA, 30%; NS, 35%; AS, 32%; and AH, 35%. Fig. 2 provides the molecular structures of the superplasticizers.

2.2. Measurements

Thermal stability and composition of the hydrated materials were examined using thermogravimetric analysis (Model TGA/DSC 1 STAR System; Switzerland Mettler Toledo Company, the sample is heated at a uniform rate of $10\text{ }^\circ\text{C min}^{-1}$

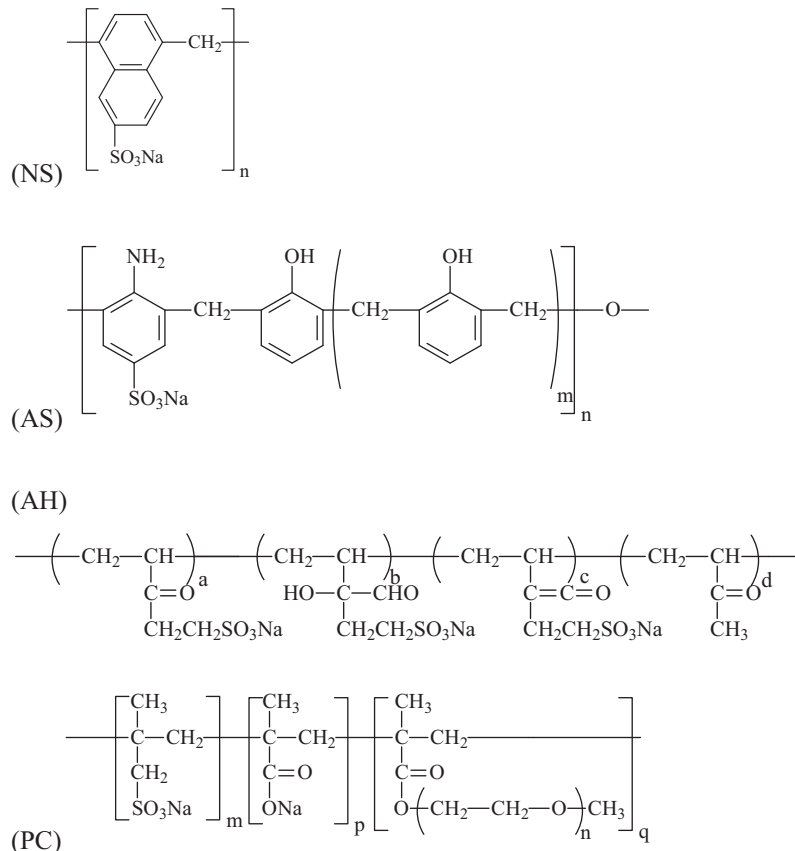


Fig. 2. Molecular structure of the superplasticizer.

from 25 to $1150\text{ }^\circ\text{C}$ in a gas flow of nitrogen N_2) and using XRD (Model D/max-2400; Japan Rigaku Corporation), respectively, to estimate the crystallization and stability. The microstructure of the hydration products was examined using scanning electron microscopy (SEM) (Model S-4800; Japan Hitachi Company, operated at 20 kV). The bonding state was investigated via X-ray photoelectron spectroscopy (XPS) (Model XSAM800; England Kratos Company) with a $MgK\alpha$ ($h\nu = 1253.6\text{ eV}$) X-ray source operating at 260 W (13 kV , 20 mA) by measuring the bond energy of calcium on the sawn and polished surface from each specimen.

The amounts of superplasticizers for the TGA test are as follows: PC (0.18%, 0.27%, 0.36%), NS (0.21%, 0.32%, 0.42%), AS (0.29%, 0.38%, 0.48%), and AH (0.32%, 0.42%, 0.52%). Specimens were prepared for XRD, SEM, and XPS test using the middle amount of each superplasticizer, such as 0.27% PC (0.27PC), 0.32% NS (0.32NS), 0.38% AS (0.38AS), and 0.42% AH (0.42AH).

3. Result and discussion

3.1. The effects of superplasticizers on the stability of ettringite

The basic formula of ettringite is $[Ca_3Al(OH)_6]_2 \cdot 24H_2O \cdot (SO_4)_3 \cdot 2H_2O$, and its crystallization has a high water content of about 46% by weight [9]. Ettringite is unstable at temperatures higher than $60\text{ }^\circ\text{C}$; this mineral decomposes to form the monosulfate hydrate, and the sulfate ions released by the decomposition are absorbed by calcium-silicate hydrate. Later, when sulfate ions are desorbed, the ettringite is reformed. The secondary ettringite produces expansive forces, which lead to cracking, strength loss, and concrete disintegration. To know the effects of these superplasticizers on the stability of ettringite is important.

Fig. 3 shows the TGA analyses of ettringite formed by hydrating C_3A and gypsum with and without the different superplasticizers. According to the water weight loss, the content of C_3A and gypsum hydration products can be generalized.

The dehydration below $100\text{ }^\circ\text{C}$ has no effect on the stability of ettringite because calcium is not completely dehydrated. The dehydration within $100\text{--}300\text{ }^\circ\text{C}$ will significantly reduce the

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