



Hydration and properties of expansive additive treated high temperature carbonation



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ABSTRACT

An expansive additive (CSA) containing lime (CaO), ye'elimite ($3\text{CaO}\cdot 3\text{Al}_2\text{O}_3\cdot \text{CaSO}_4$) and anhydrite (CaSO_4) was treated with high-temperature carbonation, and used to make mortar samples for expansion tests. After 1 day, the mortar made with the CSA which underwent high-temperature carbonation (treated CSA) expanded to a lesser degree than the mortar made with the untreated expansive additive (CSA). However, from 1 to 3 days significant expansion occurred in the treated-CSA mortar, and by day 7, the sample had expanded ~23% more than the CSA mortar. The hydration of the CSA was suppressed until 6 h after it came into contact with water; in particular, reaction of free lime was suppressed. Loss of expansion performance during the accelerated weathering test was significantly reduced by high-temperature carbonation, and the weathering rate was reduced by a factor of ~7. The CO_2 and calcite contents increased with high-temperature carbonation, and a 0.2- μm -thick calcite film formed on the lime surface. This film reduces the extent of lime reaction before formation of the cement matrix, resulting in increased expansion, and improvement in the stability towards weathering.

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1. Introduction

Expansive additives are widely used for reducing cracking in concrete, for example by compensating for the drying shrinkage strain of concrete or providing chemical pre-stress. Strength development of the cement matrix is closely associated with reaction of the expansive additive [1–17]. Fig. 1 shows the relationship between mortar strength development and mortar expansion with expansive additives [1]. Expansion is not useful during period I in the fresh state, while expansive strain becomes small in period IV once sufficient strength has developed, and, in some cases, the risk of expansion failure may increase. To provide expansion effectively, the expansive additive should react in the effective expansion periods which are periods II and/or III. Large expansion can be achieved if reaction occurs in period II where plastic properties are dominant [1]. This means that it is desirable for the additive to be hydrated together with the Portland cement in the concrete. We have developed a new type of expansive additive containing lime, ye'elimite and anhydrite which shows similar expansion [1]. However, free lime, one of the mineral constituents of the expansive additives [5], is quick to react, and this early reaction may reduce the effectiveness of the expansion.

Because the expansive additives weather (or lose their properties when stored in a moist or humid environment), care must be taken in

their storage [17]. Measures that have been taken to minimize the degradation of expansive additives include using packing materials with very low moisture diffusion capacity, or storing additives in a container not exposed to air circulation, such as a silo. However, the amount of expansive additive used overseas has been increasing, as more countries seek to minimize concrete cracking. This means that transportation to a site takes longer, and that the materials may be stored in a region of high temperature and humidity. Thus, enhanced resistance to weathering is required so that the quality-assured period can be increased. Free lime, one of the constituents of the expansive additive, reacts with carbon dioxide gas at high temperature and generates calcium carbonate, which restricts absorption of moisture [18–20]. If high-temperature carbonation treatment is applied to the expansive additive, initial hydration of the expansive additive is suppressed, and the potential loss may be reduced; this could also slow down the progress of weathering. This paper describes the stability during storage and the properties and hydration of high-temperature carbonation-treated expansive additive.

2. Experiments

2.1. Materials used

Table 1 shows the chemical compositions of the materials. Ordinary Portland Cement (OPC), expansive additive containing lime, ye'elimite and anhydrite (CSA), JIS standard sand, and tap water were used. Table 2 shows the composition of the CSA. The CSA was treated with

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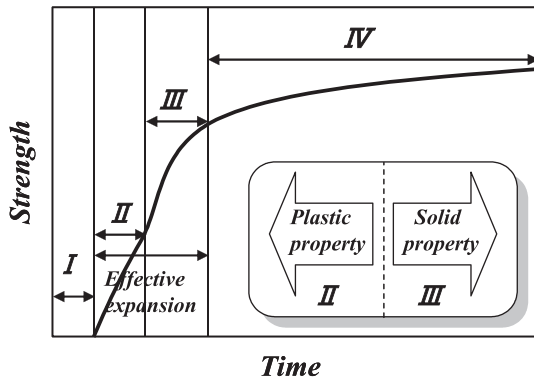


Fig. 1. Schematic illustration of the relationship between mortar strength and expansion with expansive additives.

CO₂, 100% at 600 °C for 60 min to give the treated expansive additive (treated CSA). The expansion properties of treated-CSA mortar and CSA mortar were compared.

2.2. Methods

(1) Mortar tests

Expansion of mortar was evaluated in accordance with JIS A 6202, Attachment-1. Seven mass% of the OPC was replaced with additive; the water/powder ratio = 0.5 and sand/powder ratio = 3. Tests were conducted inside at 20 °C. Specimens (4 cm × 4 cm × 16 cm), for uniaxially restrained tests were demolded after 1 day and cured in water at 20 °C. Compressive strength of mortar was evaluated in accordance with JIS R 5201. Mix proportions were the same as for the mortar expansion test. Tests were conducted at 1, 3, 7, 28, and 56.

(2) Accelerated storing stability test of expansive additive

Both CSA and treated-CSA samples were tested; 31.5 g of CSA was measured into a 100-mL polyethylene cup, and exposed (cap open) to 20 °C, 60% R.H. Samples were analyzed and tested after 8, 15, and 29 days of exposure.

(3) Hydration analysis of expansive additive

(a) Heat of hydration

Heat of hydration was measured to investigate the effects of high-temperature carbonation treatment on hydration of the expansive additives. Equal masses of water and expansive additive were mixed, and the temperature was monitored for 24 h after water addition using a multi-micro-calorimeter (MMC-511C6 made by Tokyo Riko-Sha Co. Ltd.). The ambient temperature for this measurement was 20 °C.

(b) Measurement of hydration ratio of expansive additive

The expansive additive was mixed at a water/powder mass ratio = 0.5 at 20 °C, put into polystyrene bottles and cured under sealed conditions. The reaction times were 30 min, 1, 3, 6, and 12 h, and 1, 3, and 7 days. Hydration of the samples was stopped with acetone flushing. Vacuum drying was applied for 3 h using an aspirator. Samples were prepared for X-ray diffraction (XRD, CuK α , tube voltage 40 kV, tube current 40 mA, scan range of 5–65° 2 θ in 0.02° steps) measurements by adding

Table 2
Composition of CSA.

Material	Composition of expansive additive (mass%)					
	f-CaO	Ye'elimite	CaSO ₄	C ₂ S	C ₄ AF	Insol
CSA	45.4	9.2	31.4	8.0	3.3	0.3

reagent-grade magnesium oxide (MgO) as an internal reference at a concentration of 10 mass% using a planetary ball mill. For Rietveld analysis, SIROQUANT Ver. 3.0 was used. The reaction ratios of the minerals contained in the expansive additive were calculated based on the ratio of mineral contents in the unhydrated samples by taking the quantity of each mineral derived from the internal reference method and correcting it for the LOI. Measurement of LOI was conducted after 30 min burning at 1000 °C. The amounts of calcium hydroxide and ettringite were determined for the prepared samples to quantify the reaction rate. Calcium hydroxide was determined from the weight loss during heating from 350 to 500 °C using the TG-DTA apparatus. The contents of calcium hydroxide and ettringite generated were also determined by Rietveld analysis of the XRD patterns. Both contents were corrected for the LOI.

(4) Characterization of expansive additive

The CO₂ content in the expansive additive was measured by an inorganic carbon analyzer. The mineral composition of the expansive additive was measured by Rietveld analysis of XRD. To identify where the substances generated by high-temperature carbonation treatment were located, back-scattered electron images were acquired and elemental analysis was conducted. Each expansive additive was mixed with epoxy resin, cast into a mold, and heated at 80 °C to harden. The surfaces were then polished using an Argon Ion Beam Section Processing Apparatus (SM-09010, made by Nippon Electric Co. Ltd.) with an acceleration voltage of 6 kV, and the polished surface was coated with osmium (Os). The specimen was observed by a scanning electron microscope (SEM:SU6600, made by Hitachi High-Technology Co. Ltd.). A particle of treated CSA was sliced with a focused ion beam (FIB) system, and the thin specimen was observed using transmission electron microscopy (TEM:H-9000NAR, Hitachi High-Technology Co. Ltd.) at an acceleration voltage of 200 kV. A 0.75- μ m area of the lime surface was analyzed by electron diffraction (acceleration voltage 200 kV, camera length 0.76 m).

3. Results and discussion

3.1. Properties of mortar

Fig. 2 shows the expansion properties. Although the expansion of the treated-CSA mortar was smaller than that of the CSA mortar during the first day, the treated-CSA mortar expanded significantly from days 1 to 3, and by day 7 showed ~23% more expansion than the CSA mortar. Both samples did not expand further after 28 days. Compressive strength development is shown in Fig. 3. Both the treated-CSA mortar and the CSA mortar showed similar strength increases with age.

Table 1
Chemical composition and physical properties of materials.

Material	Chemical composition (mass%)									Density (cm ³ /g)	Blain (cm ² /g)
	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	MgO	SO ₃	LOI	Insol		
OPC	64.4	5.3	20.1	2.9	0.2	0.8	2.2	3.0	0.1	3.13	3500
CSA	67.0	5.4	1.8	0.9	0.1	0.9	19.6	2.9	0.3	3.04	4090

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