

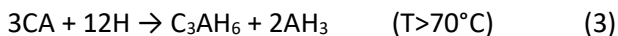
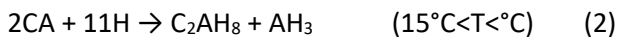
Test Method 2B.6 Analysis of Calcium Aluminate Cement Conversion by Thermal Gravimetric Analysis

Scope

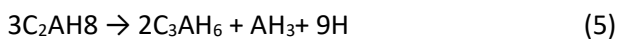
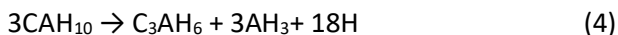
The purpose of this test is to quantify the convertability of calcium aluminate cement mortar (CAC). The determination involves subjecting the cured CAC to conditions to promote conversion followed by quantification of the cement hydrates by thermal gravimetric analysis (TGA).

What is calcium aluminate conversion?

Calcium aluminate cement mortar (CAC) is a specialty cement material that are principally made up of hydraulic calcium aluminate. Calcium aluminate, once hydrated, undergoes a series of chemical reactions that influences both its physical, mechanical and chemical properties. Calcium aluminate cement reacts with water to form metastable (CAH_{10} and C_2AH_8) and stable hydrates (AH_3 , and C_3AH_6) through time and temperature dependent reactions (Scrivener et al. 1999). (The designations for the chemical formulae are C - CaO, A - Al_2O_3 , H - H_2O and S- SiO_2):



Once hydrated CAC undergoes further chemical change referred to as conversion that transforms the metastable phases to the stable phase (Bradbury et al. 1976):



The densities of the metastable hydrates (density (g/cm^3): $CAH_{10} = 1.72$; $C_2AH_8 = 1.95$) are lower compared to the density of the stable hydrates (density (g/cm^3): $C_3AH_6 = 2.52$; $AH_3 = 2.4$) (Fryda et al. 2001). Conversion therefore increases the porosity of the concrete resulting in the reduction of concrete strength and reduced resistance to corrosion. Although the speed of conversion may take years at low temperatures, such as those found in sewers any potential acceleration of corrosion and lack of control over the process is a major concern in protecting major

and critical assets.

Procedures

i) Sample Preparation

- 1) Concrete shall be cast into a 5 cm cube mold, demolded after 24 hours and cured for 28 days at $21^\circ C$ and 99% RH.
- 2) Following curing, the concrete shall be crushed then milled to obtain particle size distribution $< 125\mu m$.
- 3) Sub-sampling from the milled samples shall be performed using a riffle splitter until approximately 2 grams of sample is obtained.
- 4) Two sets of 3 sub-samples samples (6 sub-samples in total) shall be prepared. The first 3 sub-samples (Sample A) shall be analysed by TGA as set out below. The next 3 sub-samples (Sample B) shall be converted followed by TGA analysis.

ii) Conversion

- 1) Sample B shall be transferred into a jar with distilled water (2 grams in 50 ml of water) and placed in heated water bath. The temperature of the water batch shall be adjusted until the slurry temperature is $70^\circ C \pm 2^\circ C$. The slurry shall be heated continuously for 3 hours.
- 2) Sub-samples of the slurry shall be taken every hour and analysed by TGA.

iii) TGA Analysis

- 1) The sample shall be tested by TGA immediately after milling and conversion test.
- 2) The concrete shall be washed with isopropanol (2 g concrete/20 ml isopropanol) for 10-15 minutes followed by di-ethyl ether wash (10-15 ml). Dry the concrete at $40^\circ C$ for 8-10 minutes in air. The concrete shall be stored in a desiccator under N_2 gas or vacuum while waiting for the TGA analysis to minimise carbonation.
- 3) TGA test run shall be performed with 20 mg of the milled sample heated from 30 to $900^\circ C$ at

the rate of 20°C/min in the presence of nitrogen metered at 30-50 ml/min

- 4) Test shall be repeated at least 3 times from 3 different sub-samples

Analysis of Data to Estimate Convertability

The TGA analysis shall follow the procedure set out in [Test Method 2B.5 Analysis of Calcium Aluminate Cement Hydrates by Thermal Gravimetric Analysis](#). The analysis is based on the comparison of the amount of C_3AH_6 and $C_4A_3H_3$ before and after conversion.

The first stage of the analysis shall verify the conversion is complete. The changes in the cement hydrates with heating at various period of time is shown in Figure 1. As shown further heating after 2 hours resulted in no significant change in the stable phases. Conversion after three hours can be presumed to be complete.

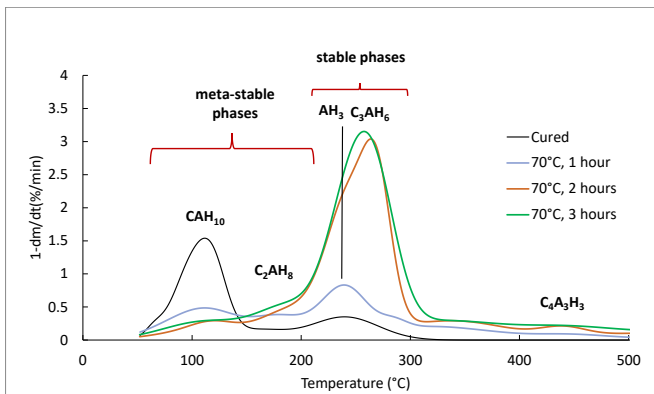


Figure 1. DTG showing the effect of time on conversion at 70°C.

The second stage is to quantify the C_3AH_6 and AH_3 phases following the deconvolution method set out in Test 2B.5 to estimate the areas under each peaks (see Figure 2).

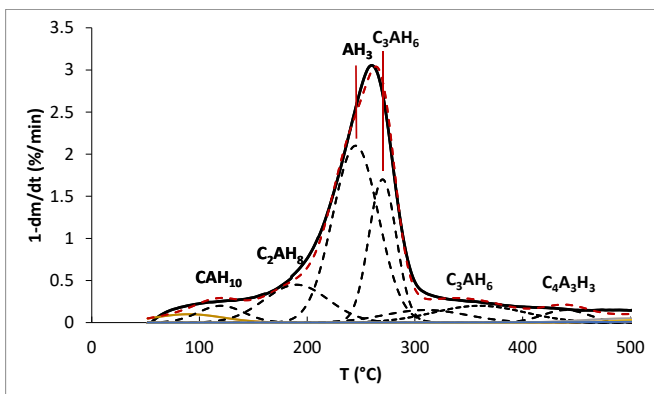


Figure 2. Deconvoluted DTG curve to identify cement phases
Black solid line (raw DTG data) dashed red line (sum of the fitted lines)

Convertability is defined from the difference in the weight % C_3AH_6 (o) before and C_3AH_6 (t) after conversion:

$$\text{Convertability} = C_3AH_6(t) - C_3AH_6(o) \quad (6)$$

How is convertability used?

The convertability is used in assessing the suitability of the coating under the different sewer corrosion classification and for estimating the design thickness to meet the required service life in the Sewer Rehabilitation & Prioritisation Decision Platform (see [fact sheet](#)).

Note the impact of conversion on CAC can also be verified from the increase in porosity and loss of compressive strength. The absence of these changes may be also used to support the non-convertability of the cement.

The 'Test Method 2B.6 Analysis of Calcium Aluminate Cement Conversion by Thermal Gravimetric Analysis' was developed by the University of Sydney.

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