### Delayed Ettringite Formation (DEF) in Precast Concrete Fear, Facts and Risks

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Paper prepared for presentation at the Session:

Structures

of the 2016 Annual Conference of the Transportation Association of Canada Toronto, ON

## ABSTRACT

Precast structural concrete units are commonly used in bridge construction. The quality of concrete used in the production of girders, deck elements and other bridge elements is expected to be high given that a service life of greater than 75 years is specified for most of bridge structures. During production the internal temperature of large concrete members is high due to the heat of hydration of Portland cement and SCMs such as silica fume. In addition, many specifications for bridge construction specify steam curing of prestressed concrete units at 95% to 100% relative humidity and 40°C to 60°C ambient temperature. The maximum allowable temperature of concrete interior during hydration is most commonly specified at 70°C in the assumption that this is the only factor mitigating the risk of excessive expansion and cracking of concrete elements due to DEF. The mechanism of DEF in concrete is complex and a number of comprehensive reviews on the DEF phenomenon have been recently published. However, controlling the maximum concrete temperature is often used as the criteria for concrete acceptance or rejection.

This paper will discuss the basics of DEF, the mechanism of deterioration and the properties of cementing materials and concrete that are known to minimize the risk of premature durability failure of the precast concrete elements. The suggested process for determining the potential for DEF will be outlined. Scanning electron microscope analysis of concrete cores obtained from suspect structures will be discussed.

#### CURING OF HIGH PERFORMANCE PRECAST CONCRETE

Precast structural concrete units are commonly used in bridge construction. The long service life expected for bridge structures dictates high expectations for durability and

compressive strength. Additional demands on concrete mix design comes from the need to achieve an adequate strength of the concrete element prior to releasing form the forms, prestressing, and moving. Thus the strength of concrete is typically higher than the minimum specified for the concrete member. All these parameters are satisfied by high cement content, low water to cementing materials ratio (w/cm), proper aggregate blend and gradation and adequate moist curing. Curing is achieved by fogging or spraying, wet coverings, utilizing membranes, and using curing compounds. The most common method, used at precast plants, is accelerated curing where early strength gain is achieved by the use of live steam, radiant heat or heated beds. Live low-pressure steam curing combined with tarping or covering provides the heat necessary to accelerate the hydration process and ensures moisture retention in the product.

A typical accelerated curing consists of four parts; preheating, ramping, holding period and cooling period. Under this curing regime, the 28 day strength of concrete is achieved at stripping times of 16 hours or less depending on the concrete mix design.

The rate of the temperature increase, target curing temperature and the rate of cooling are specified. The maximum curing temperature is typically specified between 60 °C and 70 °C.

There are many challenges for precasters to meet these temperature parameters, especially for large concrete members such as bridge girders. Type of enclosure, actual temperature and humidity of live steam, air temperature in the enclosure, ambient temperature at the plant, seasonal temperature variations, the actual concrete mix design, concrete temperature at the time of placement in the forms, and many other influences that may have a profound impact on the outcome. In addition, the placement of the temperature gauges in the concrete will influence the reading of the probes; probes placed closer to the surface, in a small cross section or in the core of a large piece of concrete will be either more influenced by the steam, ambient temperature, or by the heat of hydration, which is high in high performance precast concrete mixes.

Both the producers and the owners/specifiers assume that good production practices are followed until there is a non-conforming maximum curing temperature reported by the probes inserted in the concrete elements. The fear of high temperatures results in a rejection of the concrete elements, with sometimes limited understanding of the full impact on the long term performance. The reason for fear is DEF and concrete premature deterioration.

The American Concrete Institute (ACI) provides the following definition: *DEF is a form of sulphate attack by which mature hardened concrete is damaged by internal expansion during exposure to cyclic wetting and drying in service and caused by the late formation of ettringite, not because of excessive sulphate; not likely to occur unless the concrete has been exposed to temperatures during curing of 70 °C or greater and less likely to occur in concrete made with pozzolan or slag cement.* For concrete products exposed to infrequent wetting, and those that are continually dry for their service lives, a maximum curing temperature of 82 °C has been shown to be effective in ensuring their long-term durability.

There is a lot of information in this definition but commonly the only criteria to accept or reject concrete members is the temperature of 70 °C.

This paper provides more detailed mechanisms leading to DEF and criteria, other than temperature alone, to determine the level of risk of premature failure of precast concrete elements.

### **MECHANISM OF DEF**

The sulphate present in Portland cement under normal conditions will react to produce hydrated sulphoaluminate compounds (ettringite), and these form in plastic concrete or just after the concrete has begun its initial set. This early ettringite formation does not cause any significant disruptive action (Collepardi 2003). The sulphates are added to Portland cement to avoid flash set of concrete.

DEF can occur in concretes that have undergone heating when the mix design has certain characteristics and the element is exposed to a moist environment (Heinz and Ludwig 1987 and Taylor 2001). The early age heating of concrete alters the normal hydration process of Portland cement and, in the presence of water, ettringite decomposes to calcium monosulphoaluminate. When the concrete cools down, the calcium, the aluminates and the sulphates of the monosulphoaluminate react with the sulphates that are in solution, leading to formation of ettringite. The crystallization of ettringite generates significant stresses in the concrete leading to microcracking. Subsequently, the small ettringite crystals tend to dissolve and recrystallize in the cracks forming larger crystals that are more stable.

There is hardly any consensus on the temperatures and other parameters triggering DEF and the vast research provides often conflicting conclusions. However, there is a general agreement that internal sulphate attack had not been an issue until the early 1980s, when cement manufacturers increased the sulphate content of clinkers and cements, and the fineness of cements, in response to the increased demand for accelerated early strength development of concrete.

In mid-1990s it was believed that damage due to DEF is not a common phenomenon in concrete (Day 1992, Hime 1996) but since that time DEF has been implicated as a cause of deterioration of numerous concrete structures. As the frequency of DEF damage increases, it is becoming more important to understand all triggers of the detrimental reactions.

#### TEMPERATURE

There is a considerable diversity of research that quotes various temperatures at which ettringite decomposes. It has been postulated that up to approximately 70 °C, ettringite is the stable component in concrete but some research indicates that ettringite can exist in equilibrium with its aqueous solution in the range of 75 °C to 85 °C and as high as 90 °C to 95 °C. However, a maximum curing temperature of 70 °C is commonly accepted around the world. The research conducted by Collepardi,et.al. (2004) indicated that, in concrete with up to 4% sulphate content (expressed as SO<sub>3</sub>), the thermal decomposition and reformation of ettringite can occur only in concrete elements steam-cured at high temperatures of 80 °C to 90 °C. From a practical point of view, with a curing temperature lower than 80 °C there is no thermal decomposition of ettringite and then no risk of DEF damage independently of the SO<sub>3</sub> content in Portland cement.

Temperature and duration of thermal treatment strongly influence the expansion due to DEF. There is a pessimum effect that mostly depends on the coupling between temperature and duration; the risk of expansion is elevated at any temperature over extended period of time (Leklou, et. al. 2013).

In this context, a firm specified limit of 70 °C curing temperature of concrete as an acceptance/rejection criteria raises some questions.

- Do we reject concrete element when the temperature recorded by the probes is 70.5 °C?
- Why is it that before 1990s DEF related deterioration did not occur in concrete structures despite the fact that many manufacturers were curing their products at very high temperatures in excess of 70 °C and as high as 90 °C, in order to "double pour" their sections within a 24 hour period?

These questions can only be explained if other conditions contributing to DEF are considered.

# **EXPOSURE TO WETTING**

The structures that have failed due to DEF were also exposed to frequent wetting for extended periods of time. In the presence of water, ettringite decomposes rapidly and in unsaturated conditions a different decomposition processes take place (Baquerizo, et. al. 20016). It has been recognized that DEF is driven by availability of water and that the reaction is halted if the relative humidity (RH) is low. At 100% RH the expansion occurs very quickly. At 98% RH the expansion commence after 600 days and no expansion was confirmed when RH was below 98% (Sharmaa et.al. 2015). The impact of the moisture regime is also recognized in the ACI definition of DEF.

Therefore, moisture availability is a critical factor in assessing risk of DEF and the actual exposure of the elements to moisture should be taken into consideration. Other measures available, in the cases of higher than specified curing temperatures, include surface treatments such as silanes/siloxanes application, which render surfaces hydrophobic.

## ALKALI AGGREGATE REACTIONS (AAR) AND DEF

AAR and DEF are both integral swelling processes that can affect concrete. They lead to expansion and influence cracking and degradation. There is evidence that some of the DEF damage is also combined with AAR as both mechanisms require similar conditions to activate reactions such as increased alkali content, elevated temperature, aggregate mineralogy and concrete porosity (Martin and Bazin 2012). The differences between AAR and DEF could be explained by the difference in viscosity of the chemical products formed by their reactions. AAR products can migrate into the pores and cracks after their formation and expand in free directions. DEF products are crystallized and cannot move easily in the cracks and therefore, the place where reaction products are formed influences the damage process (Bouzabata et.al. 2012). Diamond (1994) postulated that AAR works to develop the initial microcracks inside the concrete elements and DEF propagates the cracks.

### AGGREGATE MINERALOGY

The composition of the aggregate plays an important role in the expansions caused by DEF. Concrete with siliceous aggregates tend to exhibit higher expansions when compared with limestone aggregates. In addition, the expansions are higher when the aggregate particle size is smaller.

### **CEMENT COMPOSITION**

Excessive heat curing may be a factor only in cements with certain ratio of SO<sub>3</sub> to Al<sub>2</sub>O<sub>3</sub>. There is no consensus as to what the critical ratio of sulphates to aluminates is; the critical ratio ranges from 0.6 to 0.8 and as high as 1.1. It should be noted that the sulphate and aluminate content may meet the specified limits for individual components but the ratio may indicate high risk of developing DEF damage. Since the demand for higher early strength drives the sulphate content of cement up, limiting sulphate content to 3%, as suggested to limit the potential for DEF, will not gain much support. Most research is focused on developing a better understanding of ettringite formation at a range of temperatures and the impact of a threshold sulphate content in cement of 4%.

## EFFECT OF NATURAL POZZOLANS ON DEF

There is little evidence that concrete containing fly ash or other pozzolanic materials experiences DEF damage upon exposure to elevated temperatures. Pozzolanic reactions result in limiting the amount of calcium hydroxide in the system and improve sulphate resistance. The fineness of the fly ash affects the expansion rate; the use of finer fly ash as partial replacement of cement may reduce DEF expansion, coarser fly ash is less effective (Nguyen 2013). The use of silica fume do not appear to control DEF expansion but the onset of expansion is delayed significantly due to the low permeability of mortars and concrete with silica fume. More research is needed to properly evaluate the impact of pozzolans on DEF.

## EFFECT OF W/CM RATIO

The increase of w/cm ratio results in an accelerated onset of expansion and this acceleration is more pronounced when the w/cm ratio is high. Since the high w/cm results in a weaker cement paste, the resistance to cracking is reduced. The increase of w/cm ratio leads to an increase in porosity and promotes the mobility of aluminate and sulphate ions and promotes the formation of ettringite.

#### AIR ENTRAINMENT

There is limited evidence that air-entrained concrete structures have been affected by damage due to DEF. It is likely that air voids are acting as relief for pressures induced by ettringite formation.

## PETROGRAPHIC EXAMINATION OF DEF

The identification of DEF related ettringite deposits in concrete is challenging and requires an experienced petrographer. It is not enough to show ettringite crystals in consolidation voids and conclude that DEF is present without a proper understanding of the differences in the morphology of the early ettringite formation, which forms in the process of initial hydration of Portland cement minerals, and late ettringite formation, which is detrimental. As DEF may also be accompanied by AAR, distinct features of both reactions have to be identified. An example is the presence of ring cracks around the aggregates in cases of AAR and DEF can only be confirmed if the texture of the ettringite is consistent with DEF related ettringite.

Examination of thin sections under the optical microscope or polished sections under the scanning electron microscope (SEM) provides a lot of information about texture and morphology. DEF is likely identified if fissures surrounding the aggregate are infilled with ettringite and the orientation of the needles is at right angles to the smooth margins of the cement paste surrounding the fissures. The ettringite crystals appear to grow from the surface of the paste and fill the existing space. Development of late ettringite is also found in the cracks in the cement paste where the crystals are deposited perpendicular to the walls. The texture of ettringite may vary from large crystals to more fibrous filling and to dense formations. SEM images are supplemented by the energy dispersive spectrum (EDS) to determine the elemental composition of DEF features, if any.

## DEF RISK ASSESSMENT

The proposed checklist of concrete element properties is to identify the potential for DEF formation in the future. The risk evaluation is initiated only when the reported temperatures are above the maximum specified for curing conditions. The critical limits are proposed based on the literature review and the most commonly reported values. It is not intended for the evaluation of existing structures in service, which should be evidence-based upon detailed petrographic examination. In cases where the product evaluation and the determination of risk is not persuasive, cores from the elements may be cycled through a thermal regime to accelerate DEF and examined for late ettringite presence.

Type of DEF Condition	Critical Limit for DEF	Level of DEF Risk
Highest curing temperature	<80 °C	Low
SO <sub>3</sub> content of cement	<4%	Low
SO <sub>3</sub> / Al <sub>2</sub> O <sub>3</sub> ratio of cement	<0.7	Low
Fly ash, slag or silica fume	No limit	Low, if present
w/cm ratio	<0.40	Low
Air entrainment	No limit	Low, if air entrained
Exposure to wetting	Frequent or continuous	Low, if moisture limited

### CONCLUSIONS

It is not the intent of this paper to influence or relax current specifications for the curing temperatures of precast concrete elements. The risk of DEF is real and should be considered but the conflicting research does not provide clear direction on how to assess the potential for late ettringite damage in concrete. The focus on temperature alone may unnecessarily penalize concrete that, based on other known risk factors, is sound.

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