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## TECHNICAL NOTE

# DELAYED ETTRINGITE FORMATION AND HEAT CURING—IMPLICATIONS OF THE WORK OF KELHAM

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#### Introduction

It has been suggested that the primary cause of delayed ettringite formation (DEF) is "excessive" heat curing, resulting in the decomposition of ettringite (calcium sulfoaluminate hydrate or C<sub>3</sub>A·3CaSO4·32H2O) that is initially produced in Portland cement systems during the early period of hydration. Subsequently, if the concrete is exposed to substantial amounts of water, the ettringite reforms, leading to destructive expansive forces that crack the concrete members. The conditions for the decomposition of the normal ettringite in concrete have not been well established. Curing temperatures as low as 70°C were found by Heinz and Ludwig (1) to be sufficient for certain compositions of Portland cement, primarily as relating to its sulfate and tricalcium aluminate contents. Others have suggested the minimum temperature may be even lower, and German specifications limit the maximum temperature to 60°C. It is difficult for high-performance concretes to be kept below 60°C during curing. During manufacture of U-beams in Texas with high-performance concrete, the concrete temperatures approached 90°C, suggesting that these beams may be at risk from deterioration due to DEF.

## Research by Kelham

In 1996, Kelham (2) published a paper that considered the potential for expansion of mortars cured at various temperatures. The paper discusses work conducted on five different production clinkers that were modified by adding  $SO_3$  and grinding to different levels. Mortar prisms were cast and cured at temperatures ranging from 20 to 90°C. Expansions were measured over a period of approximately 1800 days. Kelham found no significant expansion for most of the mortars cured at 20, 70, or 80°C, but found significant expansion for certain mortars cured at 90°C.

From this work on 70 different cement compositions, Kelham developed an equation that related the amount of expansion of mortars cured at  $90^{\circ}$ C to the surface area of the cement, and the quantities of  $SO_3$ ,  $C_3A$ ,  $C_2S$ , MgO. and  $Na_2O_{(eq)}$  in the cement. The equation

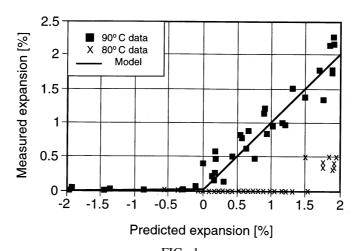


FIG. 1. Predicted and measured expansions of mortars at 80 and  $90^{\circ}$ C.

developed by Kelham to predict the expansion of mortars at 90°C is shown in Eq. 1 and the predicted and measured expansions are shown in Figure 1.

Expansion at 90°C = 
$$0.00474*SSA + 0.0768*MgO + 0.217*C_3A$$
  
  $+ 0.0942*C_3S + 1.267*Na_2O_{eq} - 0.737*abs(SO_3 - 3.7$   
  $- 1.02*Na_2O_{eq}) - 10.1$ 

Values greater than zero are considered to be expansive when concrete made with the cement cured at  $90^{\circ}$ C.

# **ASTM Cement Databases**

A survey of Portland cements marketed in North America was conducted in 1994 under the sponsorship of ASTM Committee C-1 on Cement (3). The primary purpose of the survey was

TABLE 1
Number of cements from the 1953 survey that may be listed as expansive based upon Kelham's equation.

Number of pansive cements	Total number of cements	Percent of expansive cements
0	97	0
0	50	0
1	19	5.2
0	12	0
1	178	0.56
	Number of pansive cements  0 0 1 0 1	pansive cements         of cements           0         97           0         50           1         19           0         12

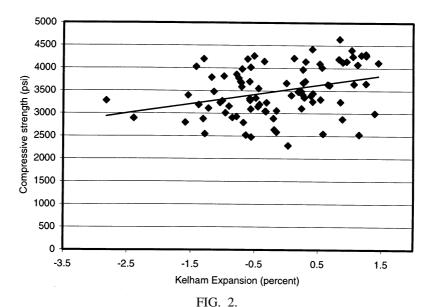
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TABLE 2 Number of 1994 cements that may be listed as expansive based upon Kelham's equation.

Туре	Number of expansive cements	Total number of cements	Percent of expansive cements
I	1	71	1.4
II	0	163	0
III	40	90	44.4
V	0	35	0
Total	41	359	11.4

to provide modern data on cement characteristics and to aid C-1 subcommittees in evaluating standard development needs. The last previous survey of similar scope was done by the National Bureau of Standards on cements procured in 1953 and 1954, with the data published during the period from 1965 through 1971. These data are available to be downloaded on the Internet from the National Institute of Science and Technology (4). The 1994 survey includes 387 different cements, with cements from 136 of the 140 cement-producing facilities in the United States and Canada plus several foreign cements from import terminals.

These databases contain information suitable for use in conjunction with the equation predicting expansions from curing at 90°C presented by Kelham. Results from this study are shown in Tables 1 and 2.



Predicted expansions plotted against the 1-day compressive strength.

It appears that a vastly greater percentage of type III cements being produced today are more susceptible to DEF at 90°C than for those produced in the 1950s, which is one explanation for the increased incidence of DEF being observed in the United States.

Forty-four percent of the type III cements studied in 1994 is considered expansive after curing at 90°C based upon Kelham's equation; thus, 66% is not considered expansive. A review was conducted to determine if the expansion was related to compressive strengths of the cements, as it has been suggested that this may be a factor in the increased incidence of DEF. Figure 2 shows the predicted expansions shown plotted against the 1-day compressive strength. Although there is a slight trend toward higher-strength cements having higher expansions, it is possible to obtain high strengths without obtaining Kelham values greater than zero.

#### **Conclusions**

The work of Kelham and the ASTM Cement databases may be used to explain the increased incidence of DEF being observed throughout the United States. It was determined that type III cements manufactured in 1994 have an increased risk of DEF compared with other cement types. From the 1953 database, only 5% of the type III cements were at risk from DEF when cured at 90°C, whereas from the 1994 database 44% of the type III cements were at risk when cured at 90°C. No simple correlation was found between compressive strength and the expansion calculated using the equation of Kelham. The Kelham equation may be suitable for determining the susceptibility of cements to DEF. Further work determining the risk of DEF on concretes with proper preset periods should be conducted.

## References

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- 2. S. Kelham, Cem. Concr. Comp. 18, 171 (1996).
- 3. R.F. Gebhardt, Survey of North American Portland Cements: 1994, ASTM Cement Concrete and Aggregates, Vol. 17, No. 2, pp. 145–189, December 1995.
- 4. Website: http://ciks.cbt.nist.gov/conmatl.htm