

## **A State-of-the-Art Review on Delayed Ettringite Attack on Concrete**

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**Abstract:** The paper presents a critical review of the sulphate attack related to ettringite formation. This process is associated with expansion. However, not necessarily any ettringite-related expansion is related to the sulphate attack. For example, early ettringite formation which occurs *immediately* (within hours) in a plastic fresh mixture does not produce any damaging expansion and is associated with the regulation of setting time of Portland cement paste. Expansion after the hardening of cement paste can be advantageously used for the development of chemical prestress in expansive cements. Delayed ettringite formation (DEF) occurs at *late* ages and the related *heterogeneous* expansion in a hardened concrete can produce cracking and spalling. Two different types of DEF are examined depending on the sulphate source: DEF caused by external sulphate attack or internal sulphate attack.

**Keywords:** Delayed ettringite formation; Expansion; Sulphate attack.

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## 1. Introduction

Ettringite formation is considered to be the cause of most of the expansion and disruption of concrete structures involved in the sulphate attack [1]. However, not necessarily any sulphate attack is caused by ettringite formation [2]. Moreover ettringite formation can be advantageously used without any sulphate attack for the development of shrinkage-compensating concrete by using expansive cements.

The purpose of this review is to present critical analysis of the relationship between ettringite formation and sulphate attack including some special cases of this attack such as those related to thaumasite formation or "delayed ettringite formation (DEF)" as intended according to the terminology currently used [3, 4].

The term "sulphate attack", as used here, means deterioration of concrete involving any type of sulphate interactions with cement paste independently of the curing temperature and sulphate source. In other words, both concrete structures cured at ordinary temperatures and those steam-cured will be examined irrespective of whether the source of sulphate is external or internal. This paper does not cover damage which is specifically attributed to the physical sulphate attack such as crystallization of water-soluble sulphate salts [5].

## 2. Ettringite Formation

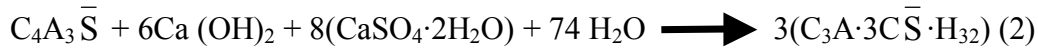
Ettringite formation is associated with expansion and many hypotheses of ettringite related expansion have been advanced [2-4]. However, not necessarily any ettringite-related expansion produces damaging disruption of concrete structures.

When ettringite occurs *homogeneously* and *immediately* (within hours), it does not cause any significant localized disruptive action (early ettringite formation, EEF). This type of harmless ettringite formation happens, for instance, when gypsum reacts with anhydrous calcium aluminate in a through-solution reaction and acts as a set retarder in Portland cement mixtures, as elaborated in Table 1:



The retardation has been attributed to the early formation of an ettringite layer which acts as a coating over the surfaces of the cement grains soon after mixing [3]. This type of ettringite may also be called "primary" ettringite as opposite to "secondary" ettringite which will be defined later. The formation of "primary" ettringite occurs in the plastic stage of the cement mixture and does not produce any significant harmful stress. Another example of harmless and useful EEF occurs when, under proper restraint, a calcium aluminate sulphate

( $C_4A_3\bar{S}$ ) hydrates within few days producing ettringite uniformly distributed and then homogeneous expansion throughout the hardened concrete (expansive cements):



In such a case, the restrained expansion is advantageously transformed into a rather useful stress (0.2 - 0.7 MPa in shrinkage-compensated concrete and 3 to 8 MPa in self-stressed reinforced concretes) provided that a certain strength level and a corresponding modulus of elasticity are reached within few days.

On the other hand, when ettringite forms *later* (after several months or years) - DEF - the related *heterogeneous* expansion in a very rigid hardened concrete can produce cracking and spalling. The disruptive effect is due to the non-uniform expansion localized only in the area of the concrete structure where ettringite forms. Therefore DEF, and not EEF, is associated with a damaging sulphate attack, as explained in Table 2. There are two different types of DEF-related damage depending on the sulphate source, as shown in Table 3: external or internal sulphate attack. External sulphate attack (ESA) occurs when environmental sulphate (from water or soil) penetrates concrete structures. Internal sulphate attack (ISA) occurs in a sulphate-free environment when the sulphate source is inside the concrete and comes from either cement with high sulphate content or gypsum-contaminated aggregate.

According to the terminology currently used [3,4], "DEF" or "secondary ettringite" [6] is related only to the ISA, particularly in heat-cured concrete structures where "primary" ettringite, responsible for setting regulation, is thermally decomposed and then forms again in an atmosphere that is saturated intermittently or continuously. However, correctly speaking the term *delayed* ettringite formation only means that ettringite forms *later* and therefore it should include both ESA and ISA as shown in Table 3. The following terms are used in the present paper: "ESA" or "ISA" to indicate DEF related to *external* or *internal* sulphate attack, respectively.

### 3. External sulphate attack

The ESA-induced damage, which is the traditional sulphate attack, is determined by the chemical interaction of a sulphate-rich soil or water with the cement paste. Soils containing sodium, potassium, magnesium, and calcium sulphate are the main sources of sulphate ions in groundwater. For ESA to occur, the following three conditions must be fulfilled:

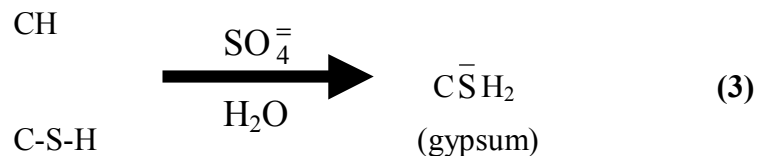
- high permeability of concrete;
- sulphate-rich environment;
- presence of water.

A diagrammatic representation of the holistic approach [7] to the ESA-related damage can be illustrated through the help of Fig. 1 [8] where each

contributory element corresponds to a circle. Each circle corresponds to only one of the three elements, and this situation does not present any risk at all for damage to a concrete structure. The area in the middle, where the three circles overlap, corresponds to situations of serious risk for the ESA-related damage since all the above-mentioned three elements are present. In the absence of one of these elements, the ESA-related damage cannot occur. For instance, in porous and/or micro-cracked concrete not exposed to water, the ESA-related damage does not happen, even if sulphate ions are present in the environment because, in the absence of water, these ions — for instance in dry soil — cannot migrate through the interconnected pores of the concrete.

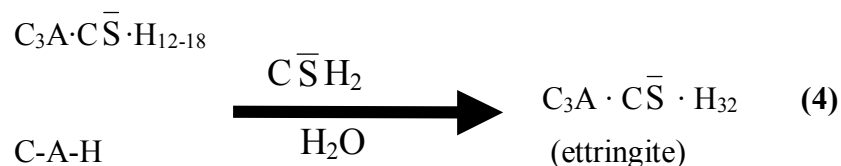
The ESA-related damage manifests itself in several forms including cracking and spalling. The specific manifestation of the ESA-related damage depends on which one of the following three chemical processes is predominant:

- i) Sulphate attack on CH and C-S-H to form gypsum:



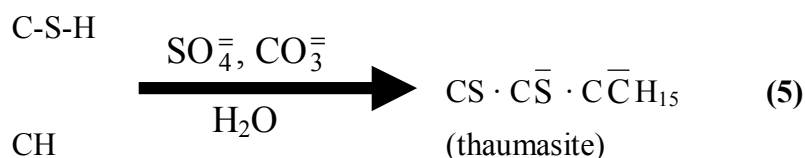
This process may cause expansion and spalling. However, its most important feature is the loss of strength and adhesion of the cement paste due to decalcification of *C-S-H* which is responsible for the binding capacity of the cement paste [2]. This process may occur with all the sulphate salts (containing  $\text{Na}^+$ ,  $\text{K}^+$ , etc.) except calcium or magnesium sulphate.

- ii) Sulphate attack on calcium aluminate hydrates (C-A-H) and monosulfate hydrate ( $\text{C}_3\text{A} \cdot \text{C}\bar{\text{S}} \cdot \text{H}_{12-18}$ ) to form ettringite:



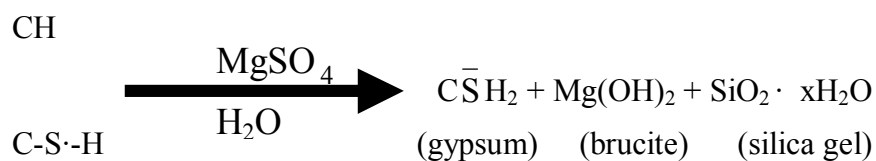
This process is mainly responsible for cracking and spalling as a result of expansion produced by delayed ettringite formation. This process may occur with all the sulphate salts (except  $\text{MgSO}_4$ ) including calcium sulphate, produced according to the reaction (3) which acts directly on C-A-H and/or monosulfate hydrate.

- iii) Sulphate attack on C-S-H and CH in the presence of carbonate ions to form thaumasite:



The thaumasite formation is accompanied by the most severe loss of strength and adhesion, which is able to transform hardened concrete into a pulpy mass, since a significant part of C-S-H can be destroyed according to reaction (5). This process may occur with every type of sulphate salts and is favoured by humid atmospheres and low temperature (<10°C) [9].

- iv) Sulphate attack on C-S-H attack by magnesium sulphate (Mg SO<sub>4</sub>) wich is not directly related to ettringite formation:



Even in this type of attack, without ettringite formation, there is loss of strength and adhesion of the cement paste due to decalcification of C-S-H as in the process (3).

#### 4. Internal sulphate attack

The ISA-related damage is relatively "new" with respect to the "traditional" sulphate attack (ESA) since it was detected in the middle of 1980's in prestressed concrete railway ties [10]. Two different mechanisms of DEF caused by ISA well be examined in this paper:

- Thermal decomposition and re-formation of ettringite in a saturated atmosphere at room temperature;
- Holistic approach based on micro-cracking, late sulphate release, and exposure to water.

##### 4.1 Thermal decomposition mechanism

According to one school of thought [3,4,6], "primary" ettringite, formed at early age is destroyed by high-temperatures (> 70°C) steam curing. Therefore, ettringite develops again at later ages in concrete structures exposed to water (either intermittently or permanently) and causes expansion or cracking. According to this school of thought [3,4,] alkali silica reaction (ASR) has a particularly important effect in promoting expansion from DEF related to ISA. In the presence of reactive aggregates, ASR-related expansion would begin during the heat treatment and cracking would immediately occur. Then ettringite forms in these cracks or other pre-existing weakness and is considered responsible for the subsequent additional expansion and cracking, if any. The following questions cannot find convincing answers from the above hypothesis [3,4]:

- Why are prestressed concrete railway ties much more vulnerable to ISA-induced distress than other precast steam-treated concrete structures?

- Why before the 1980's ISA-related deterioration did not occur in concrete structures, particularly in prestressed concrete ties [10,11], even if steam curing at temperatures as high as 70°C was adopted at that time?
- Why is ISA-related distress so erratic [11] in the sense that, with the steam curing treatment, sometimes the damage occurs and sometimes it does not?
- Why ISA-induced distress does occur even in cast-in-place concrete structures cured at ordinary temperatures [10-14], although it is fair to admit that a higher frequency of this type of damage occurs in precast steam-cured concretes?

In relationship to the last question, according to Taylor [3] and Lawrence [4], DEF related to ISA may also occur in large sections and mass pours for non-steam-treated concrete, where the heat of hydration of the cement or an external heat source during service, causes an early temperature rise to 70°C. Accordingly, ISA-related DEF could happen even in non-steamed-treated concrete provided that thermal decomposition of ettringite at or above 70°C had occurred.

According to the field experience of other researchers [10-14], *ISA*-related *DEF* would have occurred even in non-steam-treated concrete at temperatures much lower than 70°C that are insufficient to cause thermal decomposition of the "primary" ettringite. This would have happened, for instance, in non-steam-treated "Friday concrete" ties which are not mass concrete structures. These concrete ties are manufactured on Friday and cured at room temperature because by Monday they would attain adequate strength to allow for cutting the prestressing strands. *ISA*-related *DEF* would have occurred even in concrete ties non-steam-cured according to the available informations given by the producer, and this would not conform to the hypothesis [3,4] that *ISA*-related *DEF* is necessarily caused by thermal decomposition of ettringite at high temperatures (>70°C).

According to unpublished results by Ogoumah Olagot and the author of this paper [15] temperature measurements in concrete elements cured at room temperature or steam cured up to 50-90°C, with different sulphur content in the clinker phase (1-2% as SO<sub>3</sub>), and different sulphate content in the Portland cement (2-4% as SO<sub>3</sub>), definitely indicate that the thermal decomposition and reformation of ettringite can occur only in concrete elements steam-cured at high temperatures (80-90°C). The expansion of the concrete elements accompanying the reformation of ettringite was favoured by pre-existing cracks, high sulphur content in the clinker phase (2% as SO<sub>3</sub>), and high sulphate content (4% as SO<sub>3</sub>) in Portland cement.

These results confirm some laboratory studies [6, 16] on specimens cured at room temperatures: these were not subjected to *ISA*-induced *DEF* in contrast to what occurred in the correspondig overheated steam cured specimens.

#### 4.2 Holistic approach mechanism

A revised hypothesis exposed by the author [11], which satisfactorily addresses the above four questions, is based on the role played by the *late sulphate release* (from thermal decomposition of ettringite or gypsum-contaminated aggregates) on delayed deposition of ettringite in the *pre-existing micro-cracks*. *Exposure to environmental water or saturated air* causes swelling or crystal growth of ettringite leading to subsequent damage of the concrete elements.

The holistic approach according to this mechanism for the ISA-related DEF is based on the presence of three essential elements:

- micro-cracking
- late sulphate release
- exposure to water

This model can be represented in Fig. 2, where each element corresponds to a circle. The area in the middle, where the three circles overlap, corresponds to situations of serious risk of ISA-related damage since all the three needed elements of the system are present. In the absence of one of these elements, ISA-related damage cannot occur.

#### 4.3 The role of micro-cracking in ISA

Each of the three needed elements of the system can be related to numerous causes. For instance, concrete micro-cracking can be promoted by one or more of the following causes:

- Curing at high temperatures, excessive heating/cooling rate or too short preliminary curing at room temperature
- *ASR* with micro-cracks around aggregate particles
- Weathering effects cycles including wetting/drying and heating/cooling changes
- Dynamic loads in service
- Plastic shrinkage in poorly cured slab structures
- Freezing/thawing cycles
- Localized high stress in prestressed structures.

According to the holistic approach, *ASR* is just one of the possible causes of micro-cracking promoting the ettringite deposition into pre-existing micro-cracks. DEF related to ISA was detected even in a paste cement system where micro-cracks could not be caused by *ASR* due to the absence of any reactive form of aggregate [11].

On the basis of this model, one can explain why prestressed concrete railway ties are more vulnerable to DEF-related damage induced by ISA than any other precast or cast-in-situ concrete structures. The higher frequency of ISA-related

DEF occurrence in concrete ties was related to the excessive micro-cracking caused by localized stress induced by the cutting of the prestressing strands in concrete ties manufactured according to the "long line method" [11]. On the other hand, lower and more uniform stress distribution has been recently adopted - such as in the "anchored steel plate method" – and now the frequency occurrence of the ISA-related DEF is much lower in the prestressed concrete ties produced in this manner [11]. The different behaviour in terms of DEF-related damage as a function of the manufacturing process is a special feature which can be understood through the holistic model related to the whole experiential knowledge rather than laboratory studies alone.

#### 4.4 The role of delayed sulphate release in ISA

Modern cements, manufactured in kilns that burn sulphur-rich fuels or organic residues - such as rubber tires - can incorporate large amounts of sulphate (up to about 2.5%) in the clinker phase [17]. Some of this sulphur, according to Hime [14], is present as relatively slowly soluble sulphate. It would be not available, for the EEF according to the reaction (1), but would be available *later* to produce DEF.

However, the last results obtained by the author of this paper [15] do not confirm this hypothesis since, in sulphur-rich clinker phase ( $\text{SO}_3 = 2\%$ ), EEF with regular setting times occurred even in the absence of gypsum. According to the holistic model, *late sulphate release* can be attributed to these possible causes:

- Sulphate release from gypsum-contaminated aggregates, for the relatively coarse size of the calcium sulphate particles, is not immediately available for EEF and then can feed DEF.
- Thermal decomposition [3,4,6,15,18] of "primary" ettringite in overheated concrete is potentially another internal source of late sulphate release.
- Sulphate ions are slowly released from that adsorbed on the C-S-H phase in high temperature steam-cured concretes [19].

The last two potential sources of late sulphate release are included in the holistic model of the present paper (Fig. 2) as other potential sulphate releases in addition to those considered in a previous paper [11].

In agreement with the school of thought [3, 4] based on thermal decomposition of "primary" ettringite ( $>70^\circ\text{C}$ ) as responsible for ISA-related DEF, some researchers [17, 20-26] think that the sulphate content of the clinker phase can be ruled out as a source for feeding the delayed ettringite formation at ambient temperature. According to these researchers, even for clinkers with high sulphur levels ( $\text{SO}_3 = 2.5\text{-}3\%$ ), all the clinker sulphate would be rapidly available to form "primary" ettringite within several minutes [25] or few days at most [26].

There is another reason why the important role played by *late sulphate release* from the clinker phase is denied by the researchers in favour of the



thermal decomposition of "primary" ettringite ( $> 70^{\circ}\text{C}$ ) as responsible for ISA-related DEF [3, 4, 17, 20-24]: according to this school of thought, laboratory studies indicate that even with sulphur-rich clinkers there is no risk of ISA-related DEF at ambient temperature.

According to the unpublished results of the author of the present paper [15] the *late sulphate release* from sulphur-rich clinker phase ( $\text{SO}_3 = 2\%$ ) can aggravate the ISA-induced DEF distress provided that steam-curing at high temperature ( $80\text{-}90^{\circ}\text{C}$ ) is adopted. Therefore, in the absence of this high temperature treatment, high sulphur-rich clinker phase cannot by itself cause ISA-related distress.

#### 4.5 The role of exposure to water in ISA

The third needed element for the ISA-induced damage is the intermittent or continuous exposure to water or humid air (Fig.2). The presence of water filling the pore system of concrete is essential for the migration of reactant ions —  $\text{SO}_4^{-2}$ ,  $\text{Al}(\text{OH})_4^-$  and  $\text{Ca}^{+2}$  — to the micro-cracks where ettringite deposition can occur. Field experience indicates that concrete ties exposed to the rain and sun alternate actions (on the sides and especially at the tops of outdoor storage stacks) are more severely damaged by ISA than those exposed to rain but in a permanent shadow condition [11,27]. This different behaviour could be attributed to the important role played by the degree of super saturation in favouring the deposition of ettringite. On the other hand, there is experiential knowledge [10, 28] that micro-cracked concrete ties, with the same cement source and concrete composition causing ISA-induced damage in other wet concrete structures, do not show such type of deterioration when they are protected from contact with water (concrete products protected by hydrophobic treatments or stock-piled ties below the outdoor storage stacks).

### 5. Concluding remarks and recommendations

Ettringite formation by reaction of internal or external sulphate with anhydrous or hydrated calcium aluminates has an expansive character. However, not necessarily the ettringite formation produces damaging expansion. When it occurs *immediately* (within several hours) in a fresh mixture (EEF), there is no expansion. EEF is advantageously used for setting time retardation of Portland cement.

On the other hand, when ettringite forms *heterogeneously at late ages* (after months or years), — DEF — in a rigid hardened concrete, it can produce cracking and spalling.

There are two different types of DEF-related damages depending on the sulphate source: external or internal sulphate attack. ESA usually occurs when *environmental sulphate* (from water or soil) penetrates into a concrete structure. It can be prevented or reduced by using impermeable concrete.

ISA occurs in a sulphate free-environment by the *late sulphate ions release* from either cement or gypsum-contaminated aggregates. Of these two internal

sulphate sources, the latter is relatively rare because it can be prevented by controlling the sulphate content of the aggregates. The ISA-induced damage caused by the late sulphate release from the cement can be related to thermal decomposition of “primary” ettringite and sulphate release from C-S-H. Moreover, cements with relatively high sulphate content both as clinker phase (2%) and cement (4) for the gypsum addition, can aggravate the distress in form of higher expansion related to DEF when over heating (80-90°C) occurs.

Independently of the mechanism, the risk of ISA induced damage can be prevented provided that one of the following essential elements is precluded:

- micro-cracking
- late sulphate release
- exposure to water

Apparently the most easy preclusion would relate to water exposure. However, in practice, permanent protection of concrete structures from exposure to water is very expensive by using the present day available impermeable coatings or hydrophobizing products.

Micro-cracking can be significantly reduced to harmless level by adopting adequate measurements during the design and the execution phases. For instance, precast concrete ties with lower and more homogeneous stress distribution, deriving from the prestressing process itself, should be taken into careful consideration by design engineers. On the other hand, producers of precast concrete elements should take into account that steam-curing with lower thermal gradients, from controlled heating and cooling rates, can also reduce micro-cracking and consequently the ISA-induced damage risk.

Finally, cement producers should investigate why certain cements are more prone to ISA-damage than others. Moreover, according to Lawrence [29], blended cements — particularly pozzolanic and blast furnace slag cements — should be preferred instead of high early strength Portland cements, in concrete elements, such as prestressed concrete ties, which are more vulnerable by this distress.

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## DEF RELATED EXTERNAL SULPHATE ATTACK (ESA)

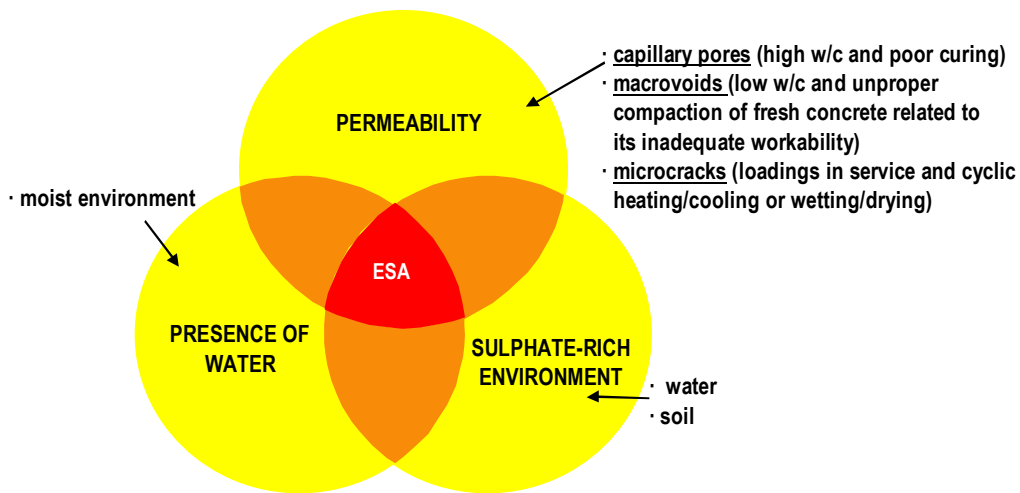


Fig. 1 – Ternary representation of the DEF related to ESA.

## DEF RELATED TO INTERNAL SULPHATE ATTACK (ISA)

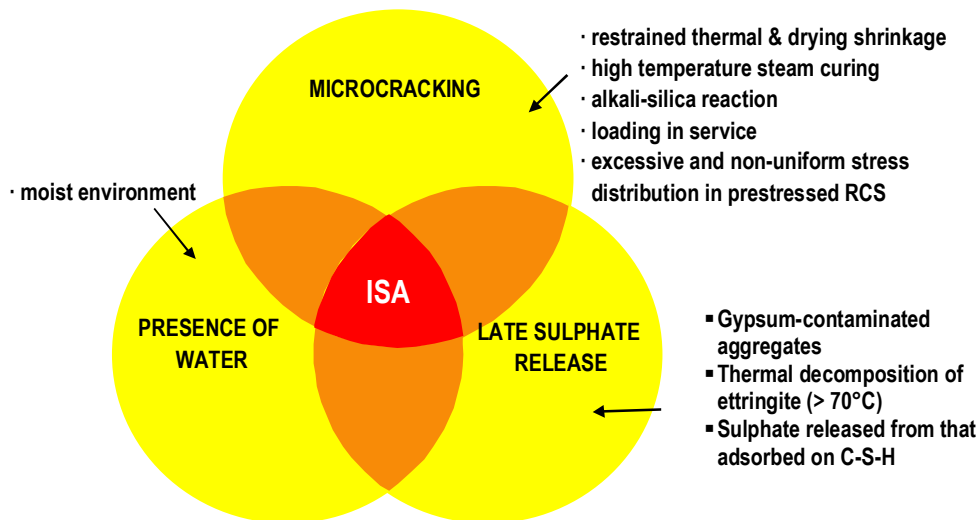


Fig. 2 – Ternary representation of the DEF related to ISA.

**Table 1 - Early Ettringite Formation (EEF) "Primary" Ettringite.**

- It occurs *homogeneously* and *immediately* (within hrs or days).
- The related expansion, if any, does not cause a localized and disruptive action since it occurs in a deformable concrete.
- It is due to ground gypsum which reacts with calcium aluminates of the Portland clinker phase (set regulation) or to sulphate-based expansive agents in shrinkage compensating concretes.

**Table 2 - Delayed Ettringite Formation (DEF) "Secondary" Ettringite.**

- It occurs *heterogeneously* and *later* (after months or years).
- The related expansion produces cracking and spalling, loss since it occurs in a rigid, stiff, hardened concrete.
- Its damaging effect is related to environmental or internal sulphate source (sulphate attack).

**Table 3 - Delayed ettringite formation by external and internal sulphate attack.**

