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Expansion of Fly Ash Cement Composites Due to Controlled Delayed Ettringite Formation

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Abstract— ***Delayed Ettringite Formation (DEF) is a chemical reaction with proven damaging effects on the mechanical properties of hydrated concrete. Ettringite crystals can cause cracks and their widening due to pressure on the crack walls caused by the positive volume difference in the reaction. Concrete may show improvements in strength at early ages, but further growth of cracks causes widening and spreading of these cracks through the concrete structure. In Hydrated concrete crystals of ettringite can also cause the disintegration of the concrete. In this paper, we investigated the potential to utilize the positive volume difference in DEF in order to improve the mechanical properties of hydrated fine grained aerated concrete. Finely dispersed crystallization nuclei, achieved by adding air-entraining agent (AEA) and short vibration of specimens, is presented as the main prerequisite for such improvements. Control of expansion and mechanical properties were performed on samples of concrete with and without AEA by inducing DEF. For microstructure examination of fine grained aerated concrete an optical microscope and scanning electron microscope were used. We found that controlled DEF, which is guaranteed by adding AEA and with the formation of uniformly dispersed air bubbles which are crystallization sites for ettringite crystals, improves the mechanical properties. The specimens with induced DEF were measured and found to have a 6,8% increase of compressive strength.***

Index Terms—**Delayed Ettringite Formation (DEF), aerated concrete, microstructure, mechanical properties**

I. INTRODUCTION

Delayed Ettringite Formation (DEF) in cementitious materials is considered as a harmful chemical reaction leading to a variety of damages [1]-[3]. The volume of the formed DEF crystals in the hardened concrete is larger than the volume of reactants and the main results are forces from the growing crystals acting upon the walls of the crack. As a consequence, DEF cracks continue getting wider and spread through the concrete structure [4]-[5]. Considerable recent research has led to a better understanding of the mechanisms of DEF [6]. In general, it is acknowledged that DEF is a result of various factors and conditions including excessive temperatures of above 70°C, the presence of sulphates, existing cracks, moist conditions and so on [7]-[10]. Ekolu et al [11] summarise various control measures that could be used for the prevention of DEF including the use of chemical additives. However, preventative measures and improvements in general durability require further attention. In practice, concrete and mortar mixes are normally based on Portland cement clinker, where the chemical process of hydration of clinker minerals yields hydrates and hydroxides. However, because of the presence of gypsum, the chemical reaction between tricalcium aluminates (C_3A), gypsum ($CaSO_4$ $2H_2O$) and water forms ettringite crystals ($3CaO$ Al_2O_3 $3CaOSO_4$ $32H_2O$). The volume difference in this reaction is positive and ettringite crystals grow fast, growing quickly on the unhydrated cement particles, which can slow down the hydration [12]. The presence of ettringite in a liquid cementitious system is unproblematic but its formation or re-formation in already hydrated concrete can lead to extensive damages[6]. Due to the resulting volume difference, particularly in the presence of sulphates, an expansive force within concrete can cause its disintegration (sulphate corrosion). It is well known that cements with low C_3A content are more resilient to sulphate corrosion, but that also depends on the form of C_3A [13]. For instance, crystalline C_3A is more reactive than its amorphous version. The positive volume difference as a result of early ettringite formation (EEF) in cementitious materials rich with $C_4A_3\bar{S}$ calcium aluminate sulphate (expansive cement) can be used to compensate for the shrinkage during drying [14]. In this case, $C_4A_3\bar{S}$ hydrates within a few hours or days producing uniform distribution of ettringite and homogeneous expansion of hardened concrete at early stages. However, it is less known that ettringite formation and expansion in hardened cementitious materials could also be used for their controlled strengthening. The formation of a new phase characterized by substantial volume expansion for the purpose of strength improvement is well known in the mainstream material science. Such strengthening is based upon the creation of the internal compressive stress on the contact between the existing matrix and the new phase particles and depends on their shape, size and their overall dispersion. It is desirable that the newly formed particles are small, spherical and located sufficiently apart from each other to avoid overlapping stress fields. In the case of metallic materials is known dispersion strengthening of the cooper matrix with ZrO_2 particles [15]-[16]. Strength improvement of the Al_2O_3 ceramics with finely dispersed ZrO_2 particles is one example of this kind of Internal stresses in the Al_2O_3 matrix created by applying the external force trigger polymorphic transformation of a tetragonal crystalline structure of ZrO_2 into a monoclinic crystalline structure. Increased volume creates substantial compressive stresses in the matrix surrounding the transformed particles leading to a several fold increase in compressive strength as well as resistance to the spreading of cracks. Studies that exactly apply this type of mechanism for strength improvement of concrete are rare. However there are a lot of very similar research to improve the properties of concrete with an emphasis on changes in the microstructure [17]-[22] and the use of innovative technologies and materials [23]-[24].

II. EXPERIMENTAL

A. Description of the experiment

The experimental work included the selection and research of concrete components and investigation of cement paste and testing of samples of fine grained concrete. Experimental work was determined by experimental design research which is shown on **Figure 1**, with the aim of confirming that the DEF in hydrated concrete improved strength properties and also that the addition of an air entraining agent (AEA) creates by volume uniformly distributed nucleation sites for the formation of ettringite crystals and target microstructure.

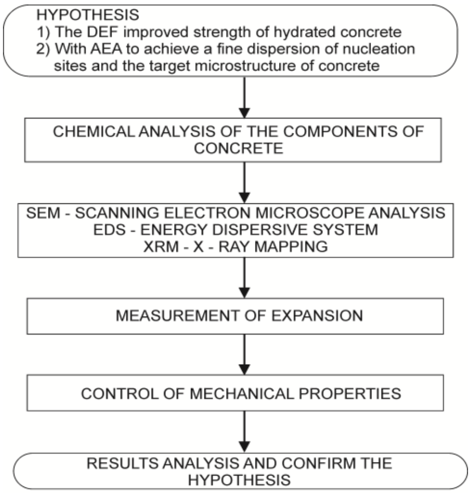

Fig. 1. Experimental design research

B. Materials

For sample preparation was used cement with mark CEM I 42,5 R. In order that we ensure the formation of the AF_m phase, we used fly ash in which we defined the components and specific surface area. The role of the fly ash was twofold – chemical and physical, the chemical as fly ash is involved actively in the Pozzolanic reaction, the physical it works as a nuclei site and as filler. For the aggregate we used chemically neutral standard sand with properties according to EN ISO 196-1 and ISO 697. The purpose of adding a chemical neutral aggregate was to prevent an alkali silica reaction (ASR), which would result from reactive minerals in aggregate. The most important precaution was the use of the petrographic components of aggregates, which will not react with alkali [25]. The used additive – air entraining agent, was the anion type based of abietic acid. The components were mixed with clean drinking water.

C. Characterization techniques

Concrete mixture components were determined on the basis of a standard consistency of cement paste. We prepared three different series of samples. The composition of mixtures is shown in Table 1. Samples were mixed with a laboratory mixer to the requirements of EN 196-1. Fresh concrete was built in standard moulds with dimensions 4/4/16 cm by vibrating table with a vibrate of 5 seconds, frequency 50 HZ and amplitude 0,75 mm. Samples were tended for 28 days in a climate chamber at a temperature of 20 ± 2 °C and a relative humidity of 98 ± 2 %. In all samples we performed measurements of density, compressive and flexural strength at intervals of 7, 14, 28 and 56 days. On the set of samples that contained fly ash, after 28 days of treatment in the climatic chamber we performed the Duggan's test. This test is used to achieve accelerated delayed ettringite formation. The test consists of soaking the samples in dematerialized water at 20 ± 2 °C and drying at a temperature of 81 ± 2 °C. After the Duggan's [26] test the prisms (samples BI-DT and CI-DT) were put through a laboratory conditioning for 48 hours in a desiccator in between each of the above phases, and were once again immersed in the demineralised water for 24 hours in order to fill the capillaries and voids with water. In these samples we controlled the Delayed Ettringite Formation, by measuring the expansion as a result of ettringite crystal growth.

Table 1. The composition of fine grained concrete mixtures

Specimen	A (g)	C (g)	FA (g)	W (g)	AEA (g)
AI	1350	450	-	218,2	6,8
BI	1350	310	140 (type 1)	218,2	6,8
CI	1350	310	140 (type 2)	218,2	6,8

A – aggregate FA – fly ash C - cement
W – water AEA – air entraining agent

At the moment when, in micrometers, expansion was no longer observed, we stopped the measurements. We considered that the Delayed Ettringite Formation had been completed. Measurements of density and strength were then performed on the samples. These measurements were repeated on the samples where the Duggan's test was not carried out, so we could obtain the comparisons of these values.

III. ANALYSIS AND DISCUSSION

A. The results of investigations of concrete components

Cement, standard sand and air entraining agent were used as the benchmark for standard-quality results. Therefore, declared properties are not included. Fly ash contains silicates, carbonates and phosphates of calcium, magnesium, iron and aluminum and other elements. Illite – kaolinite clays, apart from illite and kaolinite minerals, also contain a-quartz, Fe_2O_3 and $CaCO_3$ [27]. Results of the laboratory analysis for fly ash are shown in **Table 2**.

Table 2. Results of the laboratory analysis for fly ash

Component part	Fly Ash Type 1 Content (m%)	Fly Ash Type 2 Content (m%)
Loss on ignition	2,63	0,41
Insoluble residue	10,23	16,67
SiO_2 impure	5,77	13,08
SiO_2 pure	42,82	47,62
SiO_2 soluble	0,48	0,64
SiO_2 total	43,30	48,26
SiO_2 active	37,53	35,18
CaO reactive	8,01	7,56
SO_3	1,88	1,88
CaO free	1,22	2,00

B. Characterization of the macrostructure

With the optical microscope type Olympus SZX we observed significant break areas of the hydrated concrete. Control of the dispersion bubbles of air entraining agent showed that they were essentially distributed uniformly. The bubbles had a diameter between 25 do 50 µm. The average measured distance between the bubbles was 0,1 to 1,2 mm.

C. SEM analysis

With an electron scanning microscope JEOL JSM 5610 and QUANTA 200 3D, we observed the characteristic fields of air entraining bubbles. In these bubbles we discovered ettringite crystals. This proved our assumption that the bubbles of air entraining agents are nucleation sites. The morphology of ettringite crystals are very similar. Ettringite crystals grow in bunches, and all crystals are needle-like and thin. Viewed on a large number of bubbles showed that sample AI has less crystals of ettringite as shown in **Figure 2**. The samples which are labeled BI and CI show many more bunches of ettringite crystals. These crystals are grown mainly on porous sites in air bubbles – see **Figure 3**. On the samples which are labeled BI-DT and CI-DT we noticed more micro cracks in bubbles than for the samples labelled BI and CI. In these micro cracks we also observed the presence of ettringite crystals, which are very thin and needle-like, suggesting their rapid growth – see **Figure 4**.

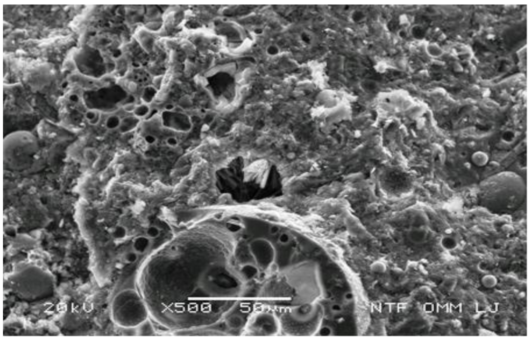

Fig. 2. Micro fractography of sample AI with ettringite crystals, SEM, SEI

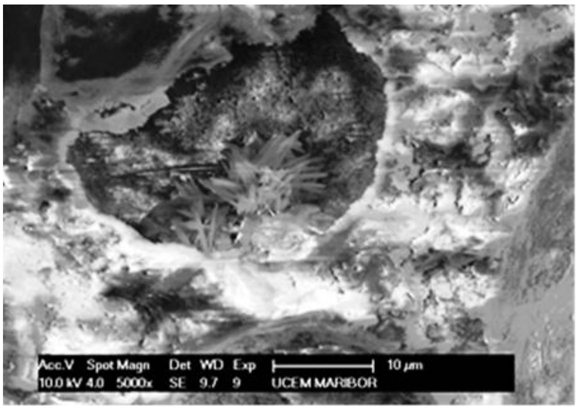

Fig. 3. Micro fractography of sample BI with ettringite crystals; SEM, SEI

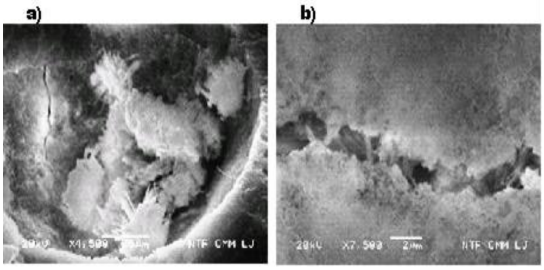

Fig. 4. Micro fractography of sample BI-DT with ettringite crystals; SEM, SEI

Comparing the microstructures of the specimens from fine grained concrete mixes AI, BI and BI-DT presented in **Figures** 2, **3** and **4** clearly demonstrates that, as expected, similar AEA-induced nuclei exist in AI, BI, CI and BI-DT, CI-DT specimens. Although ettringite crystals can be found in concretes produced by using pure Portland cement no visible ettringite crystals were detected in any of the large number of prisms from the mix AI. However, ettringite crystals did appear in specimens of all other fine grained concrete mixes. Microstructures of the specimen from the fine grained concrete mix AI show very little ettringite crystals in AEA nuclei themselves and they were detected in micro cracks. Similar to Myneni et al [28] these crystals have thin, needle-shaped morphology and are approximately 2 µm in length, revealing rapid growth. Fly ash in fine grained concrete mix BI may well be a source of soluble calcium for ettringite formation, as reported by Solem and McCarthy [29], Zhang and Reardon [30] or Chrysochoou and Dermatas [31], because its crystals were found in greater quantities in micro cracks and within the AEA-induced nuclei. **Figures 2** and **3** show that ettringite crystals have thin, needle-shaped morphology, but those found in micro cracks are only approximately 2µm long as opposed to the 10 µm long crystals found in the nuclei. The micro crack that appeared on the surface of a nucleus in **Figure 4** can be associated with the shrinkage of the matrix during hydration [32]. The comparison between various different BI and CI specimens shows that ettringite crystals start growing wherever there is enough space for growth before further damaging concrete which enables further growth. AEA-induced nuclei may, therefore, act as relief reservoirs enabling the growth of substances like ettringite crystals in hardened concrete with minimum or no damaging effects. Hime [33] even recommends air-entrainment as a way to prevent DEF and reports only a single incident where air-entrained concrete suffered from DEF.

D. Energy Dispersive Spectroscopy (EDS) analysis

EDS analysis was made by scanning electron microscope JEOL JSM 5610. Energy-dispersive X-ray spectroscopy (EDX or EDS) enables qualitative and semi quantitative elemental analysis of solids. The purpose of this study was to determine the chemical composition of the ettringite crystals of fine grained concrete mixes AI, BI, CI and in samples BI-DT, CI-DT where Duggan's test was performed. The analysis which lasted 100 second was performed on hydrated fine grained concrete which was 121 days old.

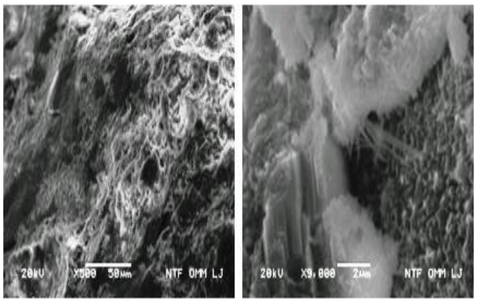

Fig 5. A typical microstructure of the fracture surface of concrete recipes AI at 500 x magnification (5a); ettringite crystals on porous area (5b)

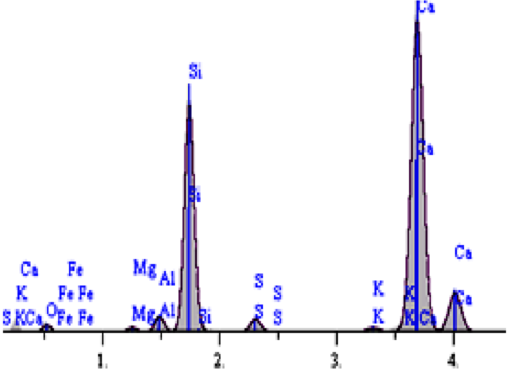

Fig 6. Typical EDS result for a specimen AI

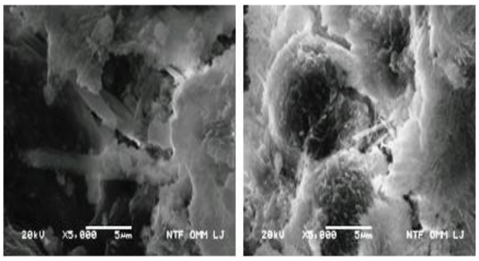

Fig 7. Set of ettringite crystals on a porous place of the fracture surface of concrete recipes BI at 5000 x magnification (7a); area of hydrated concrete with bubble of AEA and micro crack with small tiny ettrringite crystals (7b)

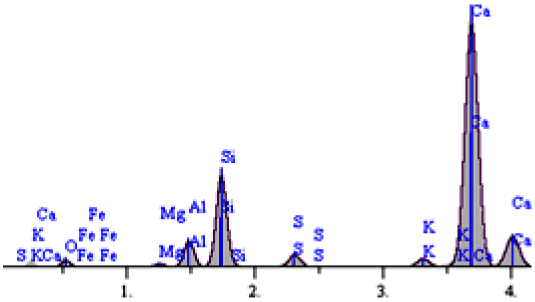

Fig 9. Microstructure of the observed region of specimen CI with bubbles of AEA with a diameter about 50 µm and a porous structure at 500 x magnification (9a); ettringite crystals on porous site

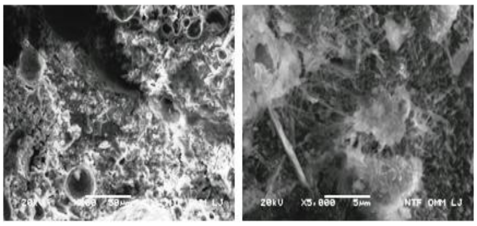

Fig 9. Microstructure of the observed region of specimen CI with bubbles of AEA with a diameter about 50 µm and a porous structure at 500 x magnification (9a); ettringite crystals on porous site

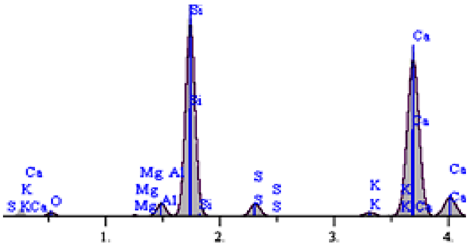

Fig 10. Typical EDS result for a specimen CI

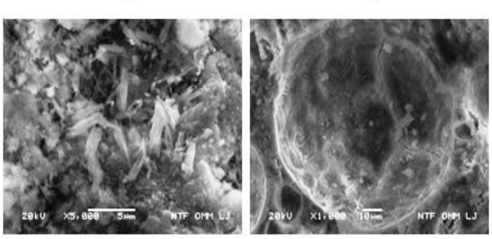

Fig 11. Fracture surface of the sample BI-DT with a porous microstructure and ettringite crystals at 5000 x magnification (11a); microstructure of the area with AEA bubble which has microcracks where

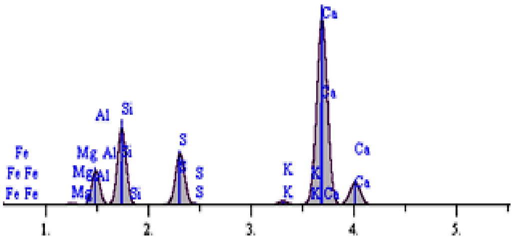

Fig 12. Typical EDS result for a specimen BI-DT

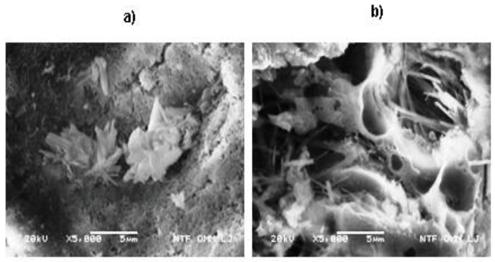

Fig 13. Bouquet of ettringite crystals which grown on a porous area in the wall of AEA bubble – specimen CI-DT at 5000 x magnification (13a); area with a very porous structure, where crystals of ettringite grow in concentrated bouquets

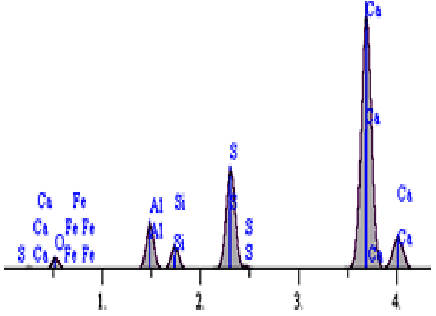

Fig 14. Typical EDS result for a specimen CI-DT

Table 4. The results of quantitative EDS analysis on ettringite crystals given by the atomic ratios of Al/Ca and S/Ca

Specimen	Al/Ca	S/Ca
AI	0,03	0,04
BI	0,11	0,04
CI	0,07	0,07
BI-DT	0,20	0,22
CI-DT	0,15	0,31

The results of quantitative EDS analysis which were made on ettringite crystals are given by comparing the atomic ratios of chemical elements Al/Ca and S/Ca. Calculated values are given in **Table 4**. The ratio Al/Ca and S/Ca is the smallest in samples AI. As expected, the value of this ratio is much higher for samples BI and CI. Enormous increase in both values Al/Ca and S/Ca is evident for samples BI-DT and CI-DT.

E. XRM analysis

X-Ray Mapping (XRM) analyses were performed with electron scanning microscope JEOL JSM 5610 on samples which are fracture sites of hydrated fine grained concrete labelled BI-DT and CI-DT. We analyzed the particular field with crystals of ettringite as shown in **Figure 15**. Based on the results, it was suggested that in areas of air bubbles in the cement matrix the sulphate attack is completed. These results confirm that the process of delayed ettringite formation is completed too. Chemical elements – sulfur and aluminum, which are characterized by chemical reaction, are present only in sites with ettringite crystals.

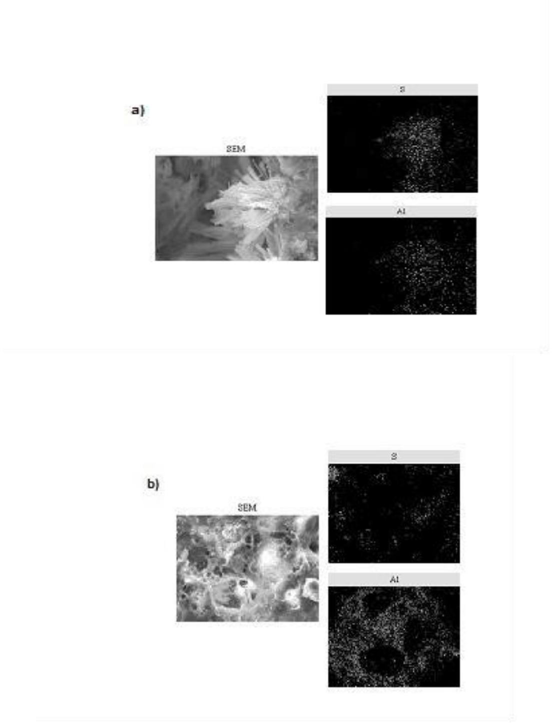

Fig 15. XRM analysis of ettringite crystal in the air bubble of AEA (specimen BI-DT – a); specimen CI-DT – B)

F. Expansion testing

After a required 28-day curing period 6 prisms of the mix BI and CI were exposed to a Duggan's test in order to achieve the accelerated ettringite formation. The prisms were then placed into a standard apparatus for the determination of length change of hardened cement paste, mortar and concrete, constructed according to ASTM C490-86. During these measurements the apparatus itself was placed in a climatic chamber with a constant temperature of 20±2ºC and relative humidity of 98±2 % (see **Figure 16**).

Fig 16. Apparatus for the measurement of expansion of hardened concrete according to ASTM C490-86 placed in a climatic chamber.

Ettringite formation was then monitored by measuring length change (expansion) with a Mahr's MarCator 1080/12.5/0.005 mm digital micrometer. The results were recorded with an analogue/digital converter connected to a workstation. Developing expansion was measured regularly at 15 minute intervals with a measurement accuracy of 0.005 mm, although intervals could well be longer considering the slow pace of DEF. **Figure 17** and **18** shows the change of length for the six fine grained concrete prisms from the mixes BI and CI that were exposed to the Duggan's test and a final 24 hour immersion in demineralised water. The change of length of prisms was measured daily and stopped at prisms for the mix BI after 73 days and for the mix CI after 82 days when measurements did not show any further expansions. The results of measurements of the length' change (expansion) of prismatic samples was evaluated as an average value which is equal to 0.0125%. All samples were inspected visually and no cracks were found.

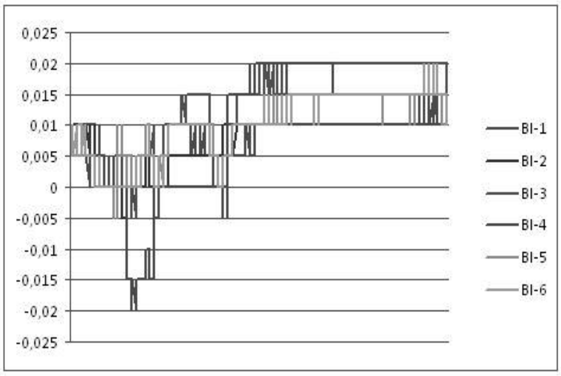

Fig 17. Results of expansion measurements for samples labelled BI-DT after the Duggan's test.

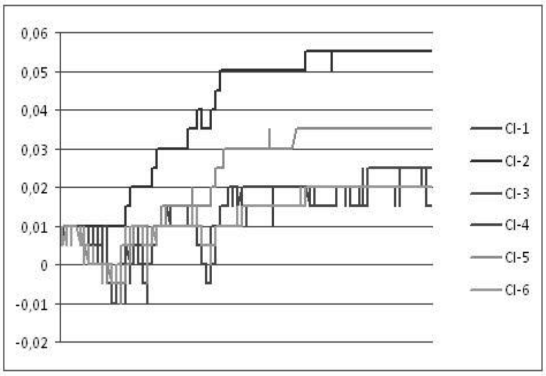

Fig 18. Results of expansion measurements for samples labelled CI-DT after the Duggan's test.

G. Mechanical properties of hardened concrete

The density of hardened fine grained concrete (ρ), its compressive (f_c) and flexural strength (f_m) were measured on 6 additional prisms for each of the three mixes after standard 7, 14 and 28 days, and additionally after 56 and 121 days when compressive and flexural strengths should reach a plateau. The mechanical properties of fine grained concrete were examined with a universal dynamometer Zwick Roell and a method according to EN 196-1. **Tables 5**, **6** and **7** show measured densities (ρ), flexural (f_m) and compressive strength (f_c) of hardened fine grained concrete prisms for mixes AI, BI, CI, BI-DT and CI-DT. The compressive strength of (BI) prisms after 121 days is 6.9% lower than that of prisms (BI-DT) and the. Compressive strength of (CI) prisms after 112 days is 6,8% lower than (CI-DT) prisms. Comparison of results showed that concrete with fly ash has slightly lower early and increased final strength [34].

Table 5. Densities and mechanical properties of hardened fine grained concrete (mix AI)

Time interval (days)	ρ (kg/m^3)	f_m (MPa)	f_c (MPa)	Time interval (days)	ρ (kg/m^3)
7	1806	2,5	18,0	7	1806
14	1804	4,0	14,8	14	1804
28	1883	5,2	17,3	28	1883

Table 6. Densities and mechanical properties of hardened fine grained concrete (mix BI and BI-DT)

Time interval (days)	ρ (kg/m^3)	f_m (MPa)	f_c (MPa)	Mix
7	1801	2,7	11,6	BI
14	1807	4,1	14,3	BI
28	1803	5,2	17,7	BI
56	1817	5,3	18,8	BI
121	1818	5,6	21,0	BI
121	1810	6,0	22,5	BI-DT

Table 7. Densities and mechanical properties of hardened fine grained concrete (mix CI and CI-DT)

Time interval (days)	ρ (kg/m^3)	f_m (MPa)	f_c (MPa)	Mix
7	1819	3,2	13,0	CI
14	1822	3,5	15,3	CI
28	1804	3,9	17,3	CI
56	1823	4,1	18,5	CI
112	1799	5,6	20,5	CI
112	1781	6,0	21,9	CI-DT

IV. CONCLUSION

Controlling DEF by using AEA as a nucleation agent, results in a slight increase of the compressive strength of fine grained concrete. Small and thin crystals of ettringite, resulting from a series of chemical reactions that take place in hydrated concrete, caused swelling of the concrete. Local stress concentration at the nucleation sites, which are air bubbles of air entraining agent, where ettringite crystals grew did not cause an extension of the microcracks which could lower the compressive strength of the concrete. Ettringite crystal growth in porous parts of the walls of the air bubbles caused the change of microstructure of the concrete. This change represents a transformation of the existing porous microstructure in line with tiny crystals condensed in the microstructure. EDS analysis shows an increase of the value Al/Ca and S/Ca ratio on ettringite crystals at these areas. The result of these changes in the microstructure of the nucleation sites is a reinforced cementitious matrix. Strength improvement is a result of hardening of the cementitious matrix, causing an increase in the compressive strength of the concrete.

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