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LONG TERM DURABILITY OF CONCRETES PREPARED WITH CRYSTALLINE ADMIXTURES

Summary: Concrete is the most used construction material worldwide and material with the second greater consumption globally, just after water. However, durability of concrete is a major problem that minimizes the service life of structures. Concrete deterioration is almost always caused by reactants that penetrate the concrete matrix with water. Therefore, almost all concrete structures require protection from water. One of the most effective ways to protect concrete from the ingress of water for a lifetime interval is the use of crystalline admixtures. Their effectiveness in watertight concrete is well proven in several projects around the world. However, their influence on the long-term durability of concrete under different durability environments is not well understood. A long-term experimental program was carried out at the Building Materials Laboratory (BML) at Democritus University of Thrace to investigate the effect of crystalline admixtures in prolonging the service life of concretes. A total of 12 concrete mixtures were produced and crystalline admixtures were added in six of them. All specimens were used for the estimation of the carbonation resistance factor and the chloride migration coefficients according to the relevant European standards. These indicators were used to calculate service life of all mixtures. Results indicate that addition of crystalline admixtures enhances durability of concrete mixtures and prolongs the service life of reinforced concrete structures up to 80%.

Keywords: crystalline admixtures, durability, service life, protection

TRAJNOST BETONA SA MINERALNIM DODACIMA NA BAZI KRISTALA

Rezime: Beton je najkorišćeniji građevinski materijal na svetu i drugi materijal po potrošnji globalno, odmah iza vode. Ipak, trajnost betona je ključni problem koji limitira radni vek armiranobetonskih konstrukcija. U većini slučajeva štetne materije odgovorne za propadanje betona prodiru u betonsku matricu zajedno sa vodom, te je skoro svim betonskim konstrukcijama potrebna zaštita od vode. Jedan od najefikasnijih načina za doživotnu zaštitu betona od prodora vode je upotreba kristališućih dodataka što je dokazano u mnogim projektima širom sveta. Ipak, njihov uticaj na trajnost betona u različitim agresivnim sredinama nije dovoljno razjašnjen. U Laboratoriji za građevinske materijale na Demokritovom univerzitetu u Trakiji sproveden je eksperimentalni program radi istraživanja efekta kristališućih dodataka na radni vek betona. U šest od ukupno 12 pripremljenih betonskih mešavina dodati su kristališući dodaci. Svi uzorci su korišćeni za procenu faktora otpornosti na karbonatizaciju i koeficijenta migracije hlorida u skladu sa evropskim standardima. Ovi indikatori iskorišćeni su za proračun radnog veka svih mešavina. Rezultati pokazuju da kristališući dodaci poboljšavaju trajnost betonskih mešavina i produžavaju radni vek armiranobetonskih konstrukcija do 80%.

Ključne reči: kristališući dodaci, trajnost, radni vek, zaštita

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1. INTRODUCTION

Concrete is the most used construction material worldwide and the material with the second greater consumption globally, just after water. One can say that it was concrete that was used for the major infrastructures built during the last century all over the world and therefore is the material strongly connected with the development of our society and the improvement of our way of living. However concrete structures do not last forever; deleterious substances penetrate from the surrounding environment into concrete and react with its components resulting to its deterioration and/or to the corrosion of the embedded steel. Almost in all cases the reactants responsible for concrete deterioration are penetrating into the concrete matrix with water. Since durability of concrete is of major concern, both for financial as well as for sustainability issues, reducing or even better minimizing the penetration of water should be of major priority. Water can enter concrete though the network of pores and capillaries that forms during cement hydration, or through cracks and other voids in the concrete. Therefore, almost all concrete structures require protection from water.

In European level the prescriptive approach method is currently used. EN 206 Standard [1] is using the deemed-to satisfy approach. The environmental action a concrete structure is about to face during its service life is classified as exposure classes as defined in EN 1992.

Requirements for the method of specification to resist environmental actions are given in this standard in terms of established concrete properties and limiting values of composition. Depending on the environmental class the concrete is subjected to, it should fulfill the requirements of the Table F1 of EN206. Thus EN 206 is trying to ensure the durability of concrete by reducing the water permeability of the material i.e. by increasing the cement content and by lowering the w/c ratio.

1.1. WATER PERMEABILITY ADMIXTURES

It is well known that a carefully designed concrete mixture which is properly produced and executed (transferred on site, casted, vibrated and cured) using a low w/cm ratio will result to a final product with low permeability and enhanced durability. However, no concrete is totally waterproof [2]. Concrete is a porous material and water can penetrate its mass through pores and microcracks (formed initially or during the service life of the structure) due to capillary absorption, or due to hydrostatic pressure. Capillary absorption is the movement of water through the small pores in concrete in the absence of an externally applied hydraulic pressure. This is the result of surface tension between the water and the pore walls [3]. Permeability of concrete is the movement of water due to a pressure gradient, such as water in contact with a concrete structure installed underground [3]. A well-known and widely accepted method for reducing the permeability of concrete mixtures and therefore increase their durability is the addition of supplementary cementing materials (SCMs) into concrete mixtures [4,5]. As it is stated in ACI212.3R-16, a class of materials referred to as permeability-reducing admixtures (PRAs) have been developed to improve concrete durability through controlling water and moisture movement [6] as well as by reducing chloride ion ingress [7,8] and permeability [4]. According to ACI212.3R-16 [3], admixtures intended to reduce water ingress split into two subcategories: PRAs for concrete exposed to nonhydrostatic conditions (PRAN) and PRAs for concrete exposed to hydrostatic conditions (PRAH) [3].

Permeability-reducing admixtures (PRAs) typically include, but are not limited to, the following categories [3]:

a) Hydrophobic water repellants,
b) Polymer products,
c) Finely divided solids,
d) Hydrophobic pore blockers,

e) Crystalline products.

PRANs consist of either hydrophobic or water-repellent chemicals (soaps and long-chain fatty acid derivatives, vegetable oils and petroleum), finely divided solids (talc, bentonite, silicious powders, clay, hydrocarbon resins, and coal tar pitches) or chemically active fillers (lime, silicates, and colloidal silica). They are most widely used for damp proofing protection under non-hydrostatic conditions [3,9].

PRAHs include finely divided solids (such as colloidal silica), hydrophobic pore blockers and crystalline admixtures. However, finely divided solids, including colloidal silica, are typically used under non-hydrostatic conditions and only some of the polymer materials can be categorized as PRAHs. Hydrophobic pore-blocking materials are used only under non-hydrostatic conditions. Crystalline hydrophilic polymers (latex, water-soluble, or liquid polymer) are only used in hydrostatic conditions [3,9]. The crystalline admixture consists of Portland cement, specially treated quartz sand and a compound of "active chemicals". The chemical composition of active chemicals in the crystalline material is kept confidential by all producers. The crystalline material's waterproofing effect in concrete is achieved by the reaction of various chemical components, which are contained in the solution, when combined within the concrete matrix [10]. PRAHs are primarily intended for use in concrete that is exposed to water under pressure and are sometimes called waterproofing admixtures. PRAHs use a pore blocking mechanism that is stable even under high hydrostatic pressure [11]. One can distinguish two different time periods: the first period is called the "hydrophilic period" when crystalline chemicals react with cement and water to grow pore blocking deposits that pack into pores under pressure. After this occurs the second period named "hydrophobic period" starts. During this period pores are filled with crystalline products that seal them against the ingress of water. In the case of crystalline admixtures, the admixture can reactivate in the presence of incoming water (such as though a crack) and generate new deposits that increase the self-sealing capacity of cracked, leaking concrete.

Concrete is a porous material. Water enters the concrete matrix through micro-pores and the capillaries. If crystalline materials are added in concrete, the active compounds react with water, calcium hydroxide and aluminum as well as various other metal oxides and salts contained in the concrete (hydrophilic period). Insoluble crystal materials are forming that fill the pores and the cracks. Water is unable to pass through these crystal formations, and as a result the concrete becomes impermeable (hydrophobic period). If new cracks appear during the service life of the concrete, the unreactive active compounds react with water and form additional crystals that appear in these cracks and fill them, thus sealing the newly formed cracks.

It is essential to notice that crystalline admixtures need water to react and form crystalline compounds. If the concrete is dry or the humidity level of the porous network is low, active compounds will remain dormant. However, when moisture increases, the compounds will start to react (hydrophilic period) and form crystals that will grow and fill the pores, thus inhibiting the further ingress of water (hydrophobic period).

2. EXPERIMENTAL PROGRAM

In the laboratory of Building Materials in Xanthi twelve different concrete mixtures were produced following the restrictions of EN 206 Standard for the exposure classes XC3, XC4, XS2, XS3, XA1, XA2 and XA3. All concretes were produced with the minimum cement content and the maximum w/c ratio described for each class as indicating in Table F of EN 206.Only a CEM I 42.5N cement was used. Both reference concretes (X--) and alternative concretes (X-- CA), prepared for each class., were produced with the same mix design. In the case of alternative concretes (X-- CA), only crystalline admixtures were added at a dosage of 0,8 per weight of cement.

Compressive strength at the ages of 2, 7, 28,180 and 1825 days (five years) were measured on all mixtures. The following durability properties were estimated in all mixtures:

Natural and accelerated carbonation, resistance to chloride induced corrosion, resistance against freeze-thaw attack and resistance against sulfate attack.

3. RESULTS AND DISCUSSION

3.1. COMPRESSIVE STRENGTH DEVELOPMENT

The compressive strength was measured on 150mm cubes according to the procedure described in 12390-3 standard [12]. The results for all mixtures rested are presented in Table 1.

Table 1: Compressive strength (MPa) of reference and alternative concretes produced with the addition of Crystalline Admixture

	XC3	XC3 CA	XC4	XC4 CA	XS2	XS2 CA	XS3	XS3 CA
2 d	18.3	17.6	30.5	23.8	25.2	25	41.8	40.4
7 d	28.5	25.4	40.6	39.6	34.6	42.0	55.5	52,3
28 d	35.1	34.1	45.2	48.2	45.8	53.3	62.9	61.9
180 d	42.7	40.3	48.0	55.2	55.5	59.9	69.0	73.2
1825 d	47.10	53.80	62.60	64.90	64.60	68.10	72.80	76.90
	XA2	XA2 CA	XA3	XA3 CA				
2 d	42.0	35.0	39.0	45.0				
7 d	51.0	54.0	51.4	52.0				
28 d	63.8	67.8	66.2	70.2				
180 d	70.8	73.4	73.1	78.2				
1825 d	75.3	77.9	76.8	80.2				

3.2. CARBONATION RESISTANCE

3.2.1. Accelerated carbonation resistance

The procedure described in EN 13295 [14] was used to assess the resistance of concrete mixtures against accelerated carbonation. All mixtures were cured in water for 3 days and then remain in the internal room of the laboratory till the age of 28 days. After this age all specimens inserted in the accelerated carbonation chamber (CO2 = 1%, R.H. = 65%, T = 21±2°C). After 56 days specimens were splitted and sprayed with phenopthalein indicator. All results are presented in Table 2.

Table 2: Accelerated carbonation depth (mm) of prepared concretes

	XC3	XC3 CA	XC4	XC4 CA	XS2	XS2 CA	XS3	XS3 CA
(mm)	4,3	14,5	6,34	6,26	11,3	9,1	4,5	4,1
	XA2	XA2 CA	XA3	XA3 CA				
(mm)	6.2	6.0	3.31	3.25				

3.2.2. Natural exposure resistance

Additional specimens were prepared for assessing the carbonation after natural exposure. These specimens were water cured for 3 days as in 3.1 and then exposed to the open air in Xanthi, Greece. This exposure class corresponds to the class XC4 of EN 206 (unsheltered air exposure). Carbonation depth was assessed at regular intervals up to the age of 900 days with a phenopthalein indicator. Carbonation depts of all concretes as estimated at the age of 900 days are presented in Table 3.

Table 3: Carbonation depth (mm) of concrete mixtures after 900 days of natural exposure

	XC3	XC3 CA	XC4	XC4 CA	XS2	XS2 CA	XS3	XS3 CA
(mm)	8.9	6.5	4.4	3.7	5.7	4.6	3.4	2.9
	XA2	XA2 CA	XA3	XA3 CA				
(mm)	3.7	3.4	3.3	3.2				

Based on the results up to the age of 900 days the carbonation resistance factor k was assessed for all produced mixtures, according to *fib* Bulletin No 34 [15]. In the next table the carbonation factor k was used for estimating the service life of reinforced concrete structures cast with the prepared concretes with and without Crystaline Admixture.

Table 4: Service life against carbonation induced corrosion or reinforced concrete structures. Environmental class typical of Mediterranean countries.

Mixture	Carbonation resistance factor k (mm/√years)	Cmin,dur (%)	Service life (years)	Increase in service life (%)
XC3	6,22	35	32	
XC3 CA	4,65	35	57	78,13%
XC4	3,13	35	125	
XC4 CA	2,59	35	182	45,60%
XS2	4,47	50	125	
XS2 CA	3,41	50	215	72,00%
XS3	2,35	50	450	
XS3 CA	2,06	50	560	24,44%
XA2	2,55	35	188	
XA2 CA	2,39	35	215	14,36%
XA3	2,24	35	244	
XA3 CA	2,17	35	260	6,5%

4. CHLORIDE INDUCED CORROSION RESISTANCE

The chloride migration coefficient was estimated for all mixtures according to the procedure described in EN 12390-18 Standard [16]. All results are presented in Table 5.

Table 5: Chloride migration coefficient of concrete mixtures according to EN 12390-18

	XC3	XC3 CA	XC4	XC4 CA	XS2	XS2 CA	XS3	XS3 CA
Dnssm	12.10	11.60	9.40	6.90	8.10	7.00	5.30	4.30
	XA2	XA2 CA	XA3	XA3 CA				
Dnssm	5.80	5.10	4.90	4.10				

Additional concrete specimens were prepared for concrete mixtures XC3, XC4, XS2 and XS3 produced with and without Crystalline Admixture. These mixtures were immersed in a chloride solution and used for estimation of the aging factor α according to the procedure described in fib Bulletin 76 [17]. It was revealed that addition of the specific Crystalline Admixture resulted to an increase of the aging factor α ranging between 0.05 and 0.09. Thus, it was supposed that Crystalline Admixture increased the aging factor of all concretes by 0.07. This conclusion was used for calculating the service life of reinforced concrete structures against chloride corrosion.

Table 6: Service life against chloride induced corrosion or reinforced concrete structures. Salinity, and mean annual temperature typical of Mediterranean countries

Mixture	Chloride migration coefficient Dnssm ($x10^{-12}$ m^2/sec)	Cmin,dur (%)	Service life (years)	Increase in service life (%)
XC3	12,10	50	25	
XC3 CA	11,60	50	31	40,00%
XC4	9,40	50	29	
XC4 CA	6,90	50	46	89,47%
XS2	8,10	50	38	
XS2 CA	7,00	50	54	57,14%
XS3	5,30	50	50	
XS3 CA	4,30	50	78	70,00%
XA2	5,80	50	47	
XA2 CA	5,10	50	68	56,76%
XA3	4,90	50	53	
XA3 CA	4,10	50	81	65,12%

5. RESISTANCES AGAINST FREEZING AND THAWING

The procedure described in EN 12390-09 [18] was used for assessing the resistance of concrete mixtures against freezing and thawing. The mass loss of all mixtures after 15 freezing and thawing cycles is presented in Table 7.

Table 7: Mass loss (%) of prepared concrete mixtures after 15 freezing and thawing cycles.

	XC3	XC3 CA	XC4	XC4 CA	XS2	XS2 CA	XS3	XS3 CA
loss (%)	90	19	13,9	6,1	4,9	4,6	4,3	3,8
	XA2	XA2 CA	XA3	XA3CA				
loss (%)	5,8	4,1	2,84	2,02				

Figure 1: Final view of XC3 concrete specimens after completion of 15 cycles

6. SULFATE RESISTANCE OF CONCRETE MIXTURES

The sulfate resistance of XA concrete mixtures produced with and without Crystalline Admixture was assessed by following the procedure described in ASTM C 1012 standard. Prismatic specimens were immersed in a 5% Na2SO4 solution for a period of 180 days. The length change of concrete specimens was measured at regular intervals. The results are plotted in Figure 2.

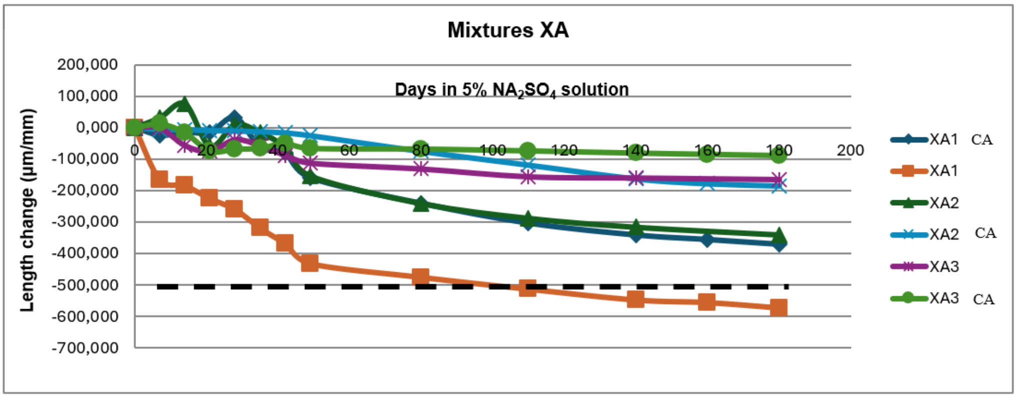

Figure 2: Length change of XA concrete mixtures

Among the concrete mixtures tested Mix XA1 fails to pass the test, since the length change measured after the age of 6 months was higher than the limit value of the standard. It is noticeable that this mixture performed equal to the upper-class concrete mixture XA2 when Crystalline Admixture was added in a dosage of 0.8% per weight of cement. This is considered as a significant enhancement of Crystalline Admixture's addition in the performance of concrete mixtures against sulfate attack, since mixture XA2 was produced with lower w/c ratio, higher cement content and sulphate resistant cement (CEM I 42.5 SR5).

All alternate concretes produced with the addition of Crystalline Admixture have better resistance than the reference concretes of the same environmental class, while they perform equal with the reference concretes of the upper class (XA1CA equal with XA2, XA2CA equal with XA3).

7. APPLICATION OF THE EQUAL DURABILITY PERFORMANCE CONCEPT

According to the CEN/TR 16563 Principles of the Equal Durability Procedure standard [20] a candidate concrete can be confirmed as being of equivalent performance with reference concrete where testing and other appropriate assessments are made to demonstrate equivalent performance with a reference value or reference concrete. The determination of reference value is based on concretes that satisfy fully the limited value specification valid in the place of use and are representative of concrete that are successfully used in the local environment as providing a satisfactory service-life. To reconsider a viable alternative, the proposed candidate concrete need to have a test performance that equals, or better is better than, the reference value when tested by the same method and at the same age as used to establish the reference performance. The Equivalent Durability Procedure (EDP) is to determine the equivalence of a candidate concrete used with the same minimum cover in the same exposure classes and intended working life as those appropriate for the reference concrete.The procedure is applicable to any exposure class, but is limited to the exposure classes where there are agreed standardized test methods:

Table 8: Performance related test methods that are standardized at the European level.

EN 12390-9	Testing hardened concrete – Part 9: Freeze-thaw resistance-Scaling
EN 12390-10	Testing hardened concrete – Part 10: determination of the relative carbonation resistance of concrete
EN 12390-11	Testing hardened concrete – Part 11: Determination of the chloride resistance of concrete, unidirectional diffusion
EN 12390-18	Testing hardened concrete – Part 18: Determination of the chloride migration coefficient

As indicating in Table 4, all candidate concretes produced with Crystalline Admixture perform better carbonation resistance factors k than their reference concretes. This results in enhanced service life of the reinforced concrete structures cast with these alternate concrete mixtures. It is noticeable that alternate concretes XC4CA and XS3CA perform better carbonation values than the reference concretes XS2 and XA2 respectively, produced with higher cement content.

Chloride migration coefficients of concrete mixtures prepared are presented in Tables 5 and 6. Tests were performed according to EN 12390-18 [16]. All candidate concretes produced with Crystalline Admixture perform better service life than their reference concretes. As mentioned above, the aging factor α was determined for all reference and alternate concretes. It was revealed that alternate concretes perform increased aging factors as compared to reference concretes of any environmental class. This increased aging factor values are responsible for the enhanced service life against chloride induced corrosion of the alternate mixtures produced with Crystalline Admixture. As indicated in Table 6, the service life of mixtures produced with Crystalline Admixture is longer than the service life of the reference concretes of the same and the more severe environmental class (XS2CA and XA2CA perform longer service life compared to XS2 and XS3, XA2 and XA3 respectively).

Addition of the specific Crystalline Admixture also improved the resistance of concrete mixtures against freezing and thawing attack, as indicated in Table 7. The protection offered by the addition of Crystalline Admixture is higher in concrete mixtures produced with higher w/c ratios: mixture XC3CA performed a mass loss of 21% compared with the relevant mass loss (of the reference mixture XC3, while mixture XC4CA performed a mass loss of 44% compared with the relevant mass loss of the reference mixture XC4. The protection offered by the addition of Crystalline Admixture in alternate concretes is reducing, as the w/c ratio of the concretes is also reduced: concrete mixture XA3CA, produced with w/c=0.45, performs a mass loss equal to 71% compared with the mass loss of the reference concrete mixture XA3.

8. CONCLUSIONS

All durability indicators assessed in this research were significantly improved on concrete mixtures produced with Crystalline Admixture. Moreover, the aging factor a was measured for the first time in mixtures containing the specific Crystalline Admixture. It was revealed that addition of Crystalline Admixture increases the aging factor a between 0.05 and 0.09 more than the value measured in reference concrete mixtures. This conclusion reflects a significant increase in the service life of reinforced concrete structures. Service life against carbonation induced corrosion was increased by almost up to 80%, while the relevant increase against chloride induced corrosion was up to 90%.

The resistance against freezing was also significantly improved on concrete mixtures produced with Crystalline Admixture. For this durability indicator there is a clear trend that addition of Crystalline Admixture is more effective in the most permeable concrete mixtures i.e. the mixtures produced with the highest w/c ratio (mixtures XC3-w/c = 0.55).

Sulfate resistance of concrete mixtures was also significantly improved after addition of Crystalline Admixture. It should be emphasized that concrete mixture XA1 was produced with a common blended cement –CEM II (A-M)42.5N- and the highest w/c ratio equal to 0.55. After addition of Crystalline Admixture this mixture performed the same as concrete mixture XA2, which was produced with a sulfate resistant cement CEM I/SR5 and a lower w/c ratio equal to 0.50.

The procedure described in EN 16563 "Principles of the Equivalent Durability Procedure" was applied on the results of this research. It is clearly shown that, in some cases, mixtures with the addition of Crystalline Admixture may replace reference concrete produced with higher cement content and lower w/c ratio without any compromission in the long-term durability and the service life of the casted reinforced concrete structures.

It could be concluded therefore that addition of Crystalline Admixture at a dosage of 0.8% per weight of cement significantly enhances the durability of concrete mixtures produced with w/c ratios in the range of 0.45 – 0.55, increasing this way the service life of reinforced concrete structures casted with these mixtures. This results in more economical, sustainable and environmentally friendly concrete mixtures.

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