

A Much Better Concrete with Zeolite Additions- State of the Art Review

Juan Carlos de la Cruz, Jose María del Campo, and David Colorado

Abstract— The main aim of this study is the use of additives that can be incorporated into concrete for new added benefits in order to gain understanding into the characterization of their influence and into their specific behavior in linear structures. With the use of special additives the number of benefits of concrete during its useful life increases, both in terms of mechanical properties and durability.

Zeolites are widely used in the construction industry and in the production of construction materials. In fact, more than 60% of the zeolite world production is consumed by China's construction industry for the production of light and high quality concrete.

Zeolite addition contributes to substantially increasing the stability of mixtures, even if added in small quantities during the preparation of cement suspensions, whether it be for subterranean watertight walls, injection tasks, or concreting of micropiles, and also during preparation of injection mixtures for cabling cavities.

The use of zeolites in mixture designs, both of mortar and concrete, significantly decreases the permeability of structures, thereby shutting out aggressive species such as sulfates.

The use of zeolites in mortar and concrete can mitigate, and even completely avoid, certain phenomena that are very significant because of their destructive nature, such as the alkali-silica and the alkali-carbonate reactions, both of which are responsible for the cracking and expansion of concreted structures.

A study of the most usual applications in the construction industry and of their future projection is carried out, concluding with the most suitable types of zeolites, their optimal proportions, and their beneficial effects on concrete.

Keywords—Zeolites, concrete, compressive strength, pozzolanic additions.

I. INTRODUCTION

“CONCRETE, as it is known today, is a construction material consisting of primarily rocks of limited maximum size that meet certain characteristics related to their mechanical, chemical, and granulometric properties and which are merged by a binding paste formed by a binder (cement) and water.” [1]

The durability of concrete can be defined as its ability to resist actions from the service environment, physical,

chemical, and biological attacks, and any process that tends to deteriorate it. Therefore, a healthy and compact concrete refers to a concrete that maintains its original form and its resistance under service in time [1]. Generally, a healthy and compact concrete presents good durability when it is subjected to normal conditions of environment and wear, and, in the case of reinforced concrete, it also offers high resistance to steel corrosion.

Zeolites are aluminosilicates of alkaline and alkaline earth cations (potassium, calcium, and sodium) [2]. They make up the largest and most varied mineral group of the earth's crust. Their main features are an open structure and their ability to accommodate water and cations within its structure and give them up without changing its structure significantly [3]. As part of the tectosilicate group, they are sedimentary rocks.

Structurally they are considered hydrated aluminum tectosilicates where aluminum is replacing silicon (Si) at the center of the structural tetrahedrons, with alkaline and alkaline earth cations forming open structures. The general chemical formula of zeolites is [4]:



Where,

X = Na, Ca, K, Ba, Sr

Y = Si and Al

The Si to Al ratio is < 1

n is a variable number which depends on the type of zeolite

The Si+Al to O ratio is 0.5

The framework contains interconnected channels occupied by cations of sodium, calcium, potassium, and magnesium, among others cations that balance the negative charge of the anionic framework, and by water molecules. These cations are mobile and can be exchanged in varying degrees for other cations. The abundance of natural zeolites is given by the large number of combinations of cations in nature and arrangement [5].

Zeolites can be considered a novel product in the construction industry due to the great number of advantages they offer, including ease of removal, wide availability of deposits, lack of complexity of their manufacturing process and a reduction of environmental pollution. Several research studies have evidenced the reduction of environmental pollution caused by the use of zeolites [6]. Specifically, zeolite application in cementitious materials such as mortar and concrete contributes to enhancing the mechanical properties of these materials as well to a noticeable increase of their

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resistance chloride ion diffusion, their freeze-thaw resistance, and overall permeability. It also increases their sulfate resistance, enabling them to be used in aggressive environments, such as special foundations or injection mortars.

The International Zeolite Association (IZA) has recognized more than 50 types of natural zeolites. Nonetheless, synthetic zeolites are being studied, characterized, and used in various industrial processes. According to IZA, “a zeolite is characterized by a structure of linked tetrahedrons that contains cavities in the form of channels and boxes commonly occupied by water molecules and cations” (Fig. 1) [7].

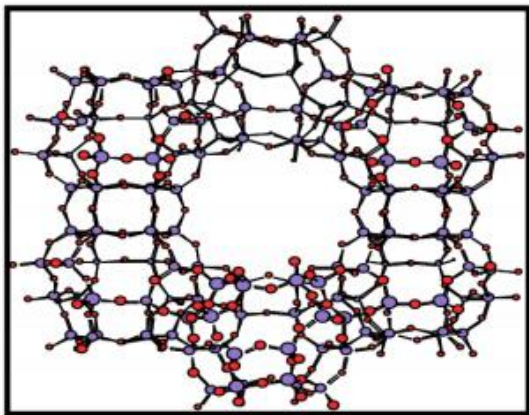


Fig. 1 Structure of natural zeolite

The usual structure of zeolite can be modified by hydroxyle groups (OH-) or fluoride (F-), which would occupy the vertices of the tetrahedrons that are not shared between adjacent tetrahedrons. This modification is accompanied by a lengthening of the channel, which allows host species to gain access into them. The hydration and dehydration process of zeolites is readily reversible, with dehydration occurring at moderate temperatures, i.e., temperatures lower than 400 °C [8].

Synthetic zeolites have a high added value and thus they are being widely studied and applied for the preparation of catalysts, as adsorbents, as well as for numerous industrial process, e.g. food industry processes, production of detergents, etc (Fig. 2). [9]

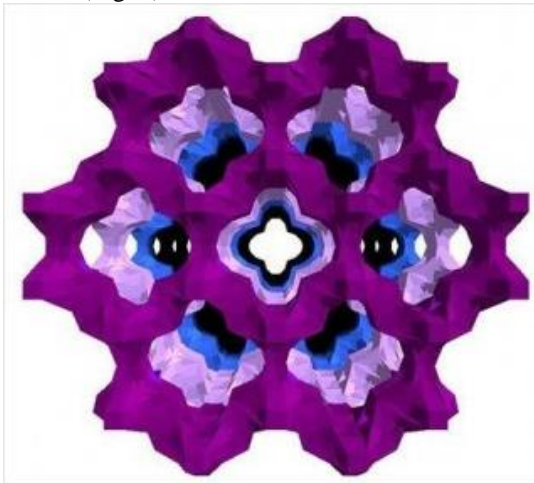


Fig. 2 Mesopores of zeolite ITQ-43

II. METHODOLOGY

A. Mortars and concrete

Mortars and concrete of high mechanical strength can be produced with the use of a wide range of zeolites. Due to the fact that they have greater cationic exchange ability than other natural pozzolans, they hinder the alkali-silica reaction and decrease the permeability of the structure, closing the access of sulfates from the outside. The presence of these minerals in the cement paste helps to curb the harmful action of chlorides on the metal armor of the armed concrete.

B. Sulfate attack

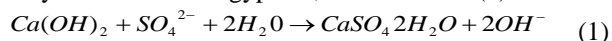
Sulfate attack occurs when concrete is exposed to groundwater, lakes, wells or soils containing sulfate ions. When sulfate ions penetrate the concrete, they react with components in the cementitious matrix causing expansive chemical reactions.

The cementitious concrete attack by sulfates can happen through three different mechanisms:

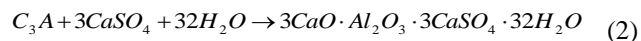
- Calcium hydroxide transformation into crystalline hydrated calcium sulfate, with an increase in concrete volume and consequential rupture of the concrete.
- Conversion of hydrated calcium aluminate and ferrite aluminate into calcium sulfoaluminate and sulfoferrite, producing swelling and rupture of concrete.
- Decomposition of hydrated calcium silicates with a noticeable decrease in the strength of concrete. This process is different from the above and typical occurs with magnesium sulfate.

The main manifestation of sulfate attack is a progressive loss of cement paste strength due to the decrease in adhesion between the hydration products. In addition, expansion causes the development of cracks, thereby increasing the permeability of concrete and allowing other aggressive species to penetrate, which causes further deterioration [10].

The most vulnerable species to sulfate attack are calcium hydroxide and aluminosilicates. Sulfate ions can react with calcium hydroxide to form gypsum, as in reaction (1):



and with calcium aluminates to form hydrate tricalcium sulfoaluminate, known as “Candlot salt”, “ettringite” or “cement bacillus” [Error! Bookmark not defined.], which is more harmful as it causes the expansive effects in concrete (2), increasing the volume by two times and a half due to the large amount of water that crystallizes:



The severity of the attack depends mainly on the concentration of sulfate ions in the soil or water that is in contact with the concrete.

Sulfates can react with the calcium hydroxide and calcium

silicate hydrate (C-S-H), producing tumatiste, under particular conditions, such as high relative humidity (above 95%), presence of carbon dioxide (CO₂) and low temperatures, generating a complete loss of concrete strength [11].

A water-cement low ratio decreases permeability and hinders the access of sulfate ions. Zeolites, too, increases sulfate resistance [12].

A zeolite addition of 15 weight % acting as replacement in the usual amount of ordinary Portland cement produces a sulfate resistance equal to that of the Portland cement specifically manufactured for protection against sulfate attack [Error! Bookmark not defined.].

C. Compressive strength

Several research studies [13]-[15] demonstrate the excellent pozzolanic properties of zeolites in the production of cements and binding materials of high standards for use in the construction industry. These studies report increases in compressive strength after 28 days of curing by the addition of zeolites into mortar and cement mixes.

By adding natural zeolites to cement Portland, its reactive character increases as well as its degree of fineness (< 63μ). The main characteristic zeolite pozzolanic consists of Ca(OH)₂ and decrease the free lime in the pasta with the passage of time [15].

Table I lists varied additions of zeolite into Portland cement, where different percentages and types of zeolites have been used. Compressive strengths are shown as a function of curing time of the test tube.

Comparing the results above with those obtained in a compressive test on the specimen of concrete type [23]:

3 days	7 days	28 days	90 days
16.1 MPa	34.1 MPa	38 MPa	40.8 MPa

Results demonstrate the increase of concrete strength in proportional amount to the quantity of zeolite added. After 28 days of curing, concretes exhibit a high increase in strength, with a greater divergence of results among them than after 7 days. Strength values eventually display a dramatic increase after long curing times.

Sammy Y.N. Chan, Xihuang Ji [19] observed that a zeolite addition of 30% did not improve the results of a 20% addition. Moreover, when the zeolite content was increased further to 40%, the compressive strength of the concrete decreased. Therefore, there is an optimal addition of zeolite which needs to be determined.

D. Setting regulator

Due to the ionic exchanging properties of zeolites by which they can quickly saturate with water, they regulate the presence of water and they neutralize the reaction of C₃A with Ca(OH)₂. Thus the setting time is increased, leading to an optimization of silicate hydration, in a greater extent than that of the dicalcium ones [24]. Zeolites are good ionic exchanges and this enables them to have a great internal balance and to

regulate variables such as humidity [25].

Setting is a process in which cement undergoes a hardening and initial traces of strength with time. The initial setting time lasts from 1 to 4 hours, while there is a subsequent setting time lasting from 3 to 7 hours [16]. The exact duration of each stage depends on the type of zeolite.

The use of zeolite as a setting regulator is an alternative to the conventional use of gypsum during the production process of clinker. In this process the temperature increases considerably, which may cause the removal of water molecules from gypsum. However, water loss with zeolite use does not take place unless temperatures are very high (90°C - 450°C).

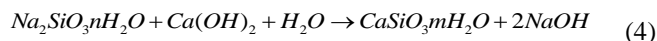
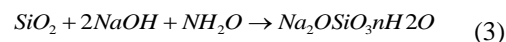
If a type of zeolite not composed of sulfates is used, the incorporation of salt in the clinker is avoided, and hence in the paste too, and this prevents the appearance of a false setting and anomalous incorporation of sulfate.

Zeolite at the cement-pozzolan interface promotes the formation of tobermorite and stimulates the crystallization of the secondary variety of this mineral as a reaction product of zeolite-portlandite phase. Thus, the slowing effect caused by the natural zeolite can be considered a positive process that enables C-S-H to coagulate and crystallize in optimal physico-chemical conditions. The abundant formation of tobermorite provides great stability and strength to mortar and to cement-based structures [24].

E. Alkali-silica reaction

The group of reactive silicious aggregates includes opaline rocks, SiO₂H₂O. The alkali-silica reaction is produced by the attack of the aggregate silicious minerals by alkaline hydroxides Na₂O and K₂O that the cement contains. The reaction produces a gel which absorbs water and thus tends to an expansion of the gel. The inhibited of such expansion by the hardened cement paste provokes strong stresses that destroy the paste and crack the concrete. The expansion could be due to the adsorption of water which causes the gel to grow and leads to hydraulic pressure effect generated by an osmosis phenomenon. The withdrawal of the gel inside the concrete through cracks can cause it to appear at the surface in the form of drops [Error! Bookmark not defined.].

This type of attacks provokes the following reactions (3), (4):



Dolomitic aggregates, CaMg(CO₃)₂. In this case “dedolomitization” by the reaction with alkaline solutions favors the formation of brucite, Mg(OH)₂, and the regeneration of alkaline hydroxide can make the process continuous (5) [10]:

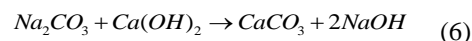
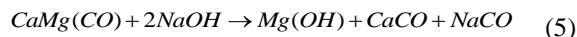


TABLE I
VARIED ADDITIONS OF ZEOLITE TESTED

Cement	Type of addition	Compressive strength, MPa			
		3 days	7 days	28 days	90 days
Mariel Cement [15]	Zeolite from Tasajera	16.9	25.8	46.5	-
	Zeolite from Mexico	-	27.9	50.4	59.1
Portland Cement [16]	Zeolite from Cuba	-	28.5	50.9	62.3
	Zeolite from Spain	-	30.3	43.7	55.5
Portland Cement P-350 [17]	10%	32.5	36	45	-
	20%	30	31.5	38.5	-
	30%	23.5	29	38.5	-
	40%	12.5	21	25.8	-
	Clinoptilolite-heulandite		27.9	50.4	62.4
	Mordenite-clinoptilolite		28.5	50.9	66.5
Portland Cement [Error! Bookmark not defined.]	Zeolite natural en un 25%	Mordenite-esmectite	29.8	47.8	70.1
		Quartz-stilbite	22.9	43.3	55.5
		Tuff-zeolitized	26.8	44.9	61.5
		5%	90.5	103.8	
Ordinary Portland cement (OPC) complying with BS4550: Part 2. [18]	Zeolite: Quarried in Du Shi Kou, Heibei Province and ground with a vibration grinder to obtain high specific surface.	10%	88.1	104.8	
		15%	96.0	110.2	
		30%	75.3	101.6	
		Normal temperature mixing	35.4	42.3	
Portland cement [19]	Zeolite 5.39 885,500 2.16	Hogh temperature mixing	43.2	45.3	
		5%	19	29	37.5 50.9
Portland cement Type II [20]	Zeolite: Karaj River region.	10%	17	29.8	38.2 52.5
		15%	16	25.7	41.7 54
		20%	16.4	28	42 50
		5%	28.2	56.8	
Portland cement [21]	Natural zeolite				

The appearance of brucite, $\text{Mg}(\text{OH})_2$, can occur in the areas where the aggregate contacts the paste, or in the aggregate itself, reacting with silica to form a magnesium silicate that is incoherent and does not conglomerate. The porous area created around the aggregate by removal of Mg^{2+} ions weakens the aggregate-paste union and no expansive gels are formed.

The expansive and destructive effect of the reaction varies with the alkaline content in the cement, their shape and type of adhesion with the cement, cement type, the mineralogical characteristics, amount and maximum size of the aggregates, the concrete porosity, and the presence of water. Other variables than may influence the process are the alkaline content in the paste water, additives, temperature and relative humidity of the environment, and the mechanical stress the concrete is subjected to [26].

The alkali-aggregate reaction inflicts damage manifested in the appearance of small, irregular cracks in the surface that look like "espuela de Gaucho" [1], or in the form of localized craters with gel droplets mentioned above.

The reactivity of the aggregate must be initially determined by a petrographic characterization to identify the type of rock with standard ASTM C295 [27]. A classification of the types of reactive rocks can be found in RILEM AAR-1 [28].

Puzzolanic type additions help mitigate this attack and thereby reduce the volumetric expansion of the alkali-silica reaction. According to the European standard EN179-1 [27], the zeolite is identified as a natural pozzolan.

The alkali-silica and alkali-silicate potential reactivities of two different aggregates were studied using the standard UNE 146508 EX99 [29]. The former had a zeolite addition of 5%, the latter had 10% content, and a third aggregate was used as reference.

The test tubes were immersed in a 1 N NaOH dissolution at 80 °C during two weeks, as the standard establishes. The measurements of the test tube were made with a refractometer and a standard bar. The results indicated in Fig. 3 are expansion averages of the three samples.

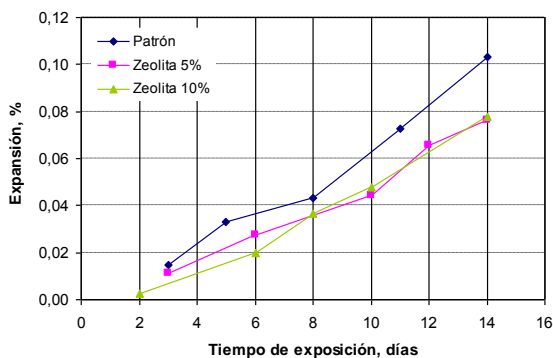


Fig. 3 Observed expansions in zeolite concrete

According to the standard specifications, an aggregate is potentially dangerous when its expansion is greater than 0.2% of the initial length of the specimen. In neither of the mortars containing zeolite expansions greater than 0.08% were

observed. The expansions were lower than that of the reference specimen, which is not considered reactive either. This indicates that the zeolite definitely improves the properties of the silicious aggregate and makes it less reactive [29].

F. Free-thaw resistance

When ambient temperature decreases below 0 °C, the water occluded in concrete pores freezes and this leads to a volume increase of about 9% which in turn triggers tensile stresses that cause cracks, concrete delamination, and in more severe cases, complete disintegration [30]. Freezing occurs mostly in the water that is inside the capillary pores. The interlaminar spaces will be affected when the temperature is below -35 °C [11]. The presence of air gaps may decrease the effect caused by ice formation in the concrete.

The evaluation of the behavior of concrete with additives under the action of water is done through an internal procedure based on a comparative study with conventional concrete and under the standard Natural Stone UNE-EN 12371:2011 "Test methods for natural stone. Determination of resistance to freezing."

Mercury intrusion porosimetry (MIP) can be used to compare and predict the freeze-thaw resistance of concretes through a model based on a statistical analysis of many building materials made by many authors from which a freeze-thaw resistance parameter named F was defined which can be calculated easily with the following empirical equation (7):

$$F = \frac{6,2}{V_{TOTAL}} + 2,1V_{15nm} \quad (7)$$

In this equation V_{Total} corresponds to the total volume of intruded mercury, V_{15nm} is the volume fraction of intruded pore, expressed as a percentage of the total volume, equivalent to a pore radius larger than 15 nm. According to the author, a correlation exists between the value of F and the freeze-thaw resistance of concrete; when $F > 310$, the concrete is considered resistant, when $F < 280$ it does not offer adequate resistance, while for values of F between 310 and 280 its resistance cannot be accurately predicted.

The content of occluded air is a factor that plays an important role in the freeze-thaw resistance of concrete [31]. The MIP technique cannot determine the volume of pores whose size exceeds 360 nm, which are formed by air entrapment, and thus these pores are not taken into account in the calculation of the resistance factor F.

The existence of pores with occluded air created by the addition of zeolite improves the freeze-thaw resistance and produces a microstructure that is more resistant to sudden changes in temperature.

G. Resistance to chlorine diffusion

Chloride attack can occur for two reasons: the chloride ions can either be present within the concrete mix or enter from the outside. In the former case, the aggregates and the water may

be contaminated; cement, additives or both may have high chloride content. In the latter case, thawing salts, exposure to marine environments, and the presence of chemicals containing chloride can attack the concrete [29], [30], [32].

The result of the penetration of chloride ions, Cl^- , is the appearance of a chloride concentration profile. There is a high concentration of chloride near the exterior and the concentration decreases as concrete depth increases. In order to obtain an estimation of the concentration profile the second law of Fick can be applied [33] (8):

$$C(x, t) = C_s \left(1 - \operatorname{erf} \frac{x}{2\sqrt{D_{app}t}} \right) \quad (8)$$

$C(x, t)$ represents the chloride concentration at depth x in a time t ; the chloride concentration at the surface is (kg/m^3) is C_s ; and erf is the error function [32], [35], [36].

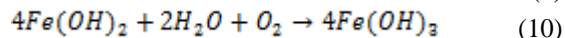
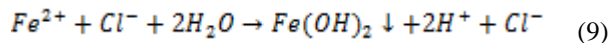
When chloride ions penetrate concrete they distribute in two different ways, either as bonded chlorides or as free chlorides. Bonded chlorides do so by reacting with tricalcium aluminate C_3A , which is a component in the paste, and yielding calcium chloroaluminates, "Friedel Salt", without producing any expansion [29], [34]. Free chlorides diffuse up to the reinforcing steel; when they reach a critical concentration, the passive layer of steel is destroyed and the process of corrosion starts [37].

The European Standard EN206 [36] classifies the types of corrosion severity other than that of marine origin (XD1, XD2, XD3) and the corrosion by chlorides from sea water (XS1, XS2, XS3). According to this standard, the maximum content of ion chlorides for these steel reinforced structures ranges from 0.2 to 0.4 weight % by cement, while for prestressed concrete structures it varies between 0.1 and 0.2%.

The origin of chloride ions (Cl^-) in concrete can reside in the concrete components themselves or be external. Chloride attack in concrete depends on the temperature, the humidity to which they are exposed, and the porosity of the concrete. The attack is often aided by carbonation attack, but not vice versa.

The consequence of chloride attack is the pitting corrosion of the steel reinforcement. The attack on the reinforcement depends mainly on the physical resistance of the concrete to chloride diffusion, the ability to chemically bond chlorides, and the type of steel employed.

The pitting corrosion process in steel is produced by the following reactions (9)(10):



Chloride attack is not uniform, since the content of chlorides and their penetration in the structure are a function of the environmental conditions to which the concrete is exposed, the water to cement ratio, the cement type, the reinforcement, aggregate size, and curing time [23].

The presence of pozzolanic additions, in particular zeolites, modifies concrete porosity and reduces permeability, thus improving the resistance to chloride penetration [37]. The

mean pore radius and the critical pore size can control the diffusion process. This pore can lead to a more or less interconnected network that can promote or slow down the diffusion process and the corresponding rate of entry of ions.

III. CONCLUSIONS

Zeolites can be used to manufacture mortars and concretes of high mechanical strength. Their high cation exchange capacity prevents the alkali-silica reaction and decreases the permeability of the structures and the sulfate access from outside. They also slow down the chloride attack on the armor. For all these reasons the possibility of using zeolite as addition as defined by EHE should be studied. The definition under section 29.2 of the EHE is the following: "Additions are inorganic, pozzolanic or latently hydraulic materials that can be added in finely divided form into concrete in order to improve some of its properties or to confer special characteristics onto it. The present Instruction covers only the use of fly ash and silica fume as additions to concrete at the time of manufacture" [37].

Structures work not only under compressive stresses, which is why the behavior of concrete with zeolite addition (of 5 and 10 weight %) should be studied under tension and flexion.

In particular, the effects related to conferring greater impermeability and resistance to aggression could be key in techniques that today require, in some regulations, application of stainless steel armor as protection against predictable attacks (piles, screens, reinforced concrete caissons in ports, ...) and where, therefore, the higher cost of zeolite addition would be compensated by the savings these induced.

ACKNOWLEDGMENT

The authors would like to thank Mercedes Castro Rodríguez, researcher of the Technical University of Madrid, for her work and contribution to this paper.

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