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Characterization and synthesis of Nano Zeolite by ball mill and survey of effects on concrete

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ABSTRACT: This project is engineering the properties of concrete containing natural nano zeolite as supplementary cementitious material in the blended Portland-cement based binder in amounts of 5, 7 and 10% by mass. Crashing of clinoptilolite zeolite is performed by means of planetary ball mill. Two types of concrete along with water to cementitious material ratio (W/(C + P)) in 0.45 and 0.4 at the ages of 7, 28 and 90 days and were compared with each other. The effect of these additives on mechanical properties (compressive and tensile strength) and durability has been investigated by Electrical Resistivity (ER) and Rapid Chloride Penetration Test (RCPT) at the ages 28 and 90 days. Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) revealed that nano particles of natural clinoptilolite could improve quality of concrete. As a result of the tests, decrease in penetration of chloride ion and increase electrical resistivity significantly that are appropriate option for controlling of corrosion in reinforced concrete structures but increase of mechanical characteristics is not considerable.

Keywords: *Ball mill; Durability; Mechanical properties; Nano Zeolite; SEM; XRD*

INTRODUCTION

Concrete is most important construction material in the world and cement is major component of concrete, in cement industry CO_2 emissions generated during the production of Portland cement has serious environmental threatens. For reducing the consumption of cement and enhancing the properties of concrete, we need the pozzolanic materials (Kratz, 2007, Wacker, *et al.*, 2011, Longley, *et al.*, 2003, Pinedo & Peters, 1988, Alter, *et al.*, 2006). Mines of zeolite in Iran are abundant and have a lot of potential for replacement with Portland cement. Natural zeolite, a hydrated aluminosilicate of alkali and alkaline earth cations with a three dimensional frame structure with nano size (Wigmore, *et al.*, 2010, Fadeian, *et al.*, 2015, Gheisari, *et al.*, 2015, Gheisari & Karamian, 2014), By replacing zeolite, consume hydroxide calcium in matrix cement and produce silica gel in the cement paste and combine with the cations to form strong bonds and reduce the porosity of concrete and nano particle of the zeolite has tremendous improvement in durability factors of concrete (Iwata, *et al.*, 2004).

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Table 1. Chemical composition of zeolite, cement and silica.

Compound	Nano Zeolite	Cement
SiO_2	69.28	20.26
Al_2O_3	10.43	5.2
Fe_2O_3	0.49	3.9
CaO	3.56	60.12
Na_2O	0.73	0.34
K_2O	1.27	0.75
MgO	0.5	2.55
TiO_2	0.166	-
MnO	0.001	-
P_2O_5	0.004	-
SO_3	0.005	2.6
L.O.I	12.97	0.95
IR	-	3.23

Materials and Methods

The utilized zeolite is a clinoptilolite type of natural zeolite manufactured by Semnan-Negin Powder Company that has been micronized and the average size is 5 micron. Afterwards the nano zeolite manufactured by planetary ball mill (PM100; Retsch Corporation) and prepared the 30-100 nano particle of zeolite, and type II Portland cement from Fars-Nov cement factory and fine (0-4.75 mm) and coarse (4.75-19 mm) aggregates meeting the requirements of ASTM C33 were used in this investigation, The chemical composition and mineralogical compounds used in this study are presented in Table 1.

In addition, its chemical properties are compared with the requirements of ASTM C618 and are reported in Table 2. As it can be seen, the summation of silica, alumina and iron oxide, which are considered to be responsible for pozzolanic activity, is 80.2% for this natural zeolite, exceeding the 70% minimum level for class N raw and calcined natural pozzolans specified in ASTM C618.

(a) Planetary Ball Mill

(b) Instruments and Nano Zeolite

Fig. 1. Ball mill and nano powder is depicted. (a) Planetary Ball Mill & (b) Instruments and Nano Zeolite

Milling the Zeolite

Based on the previous experimental studies (Karamian, *et al.*, 2015) the optimized condition for wet material has been suggested. The best magnitudes of that for carry out with, PM100- Retsch Corporation, model

Table 2. Chemical properties of natural zeolite according to ASTM C618

Requirements		Class N, ASTM C618
Chemical requirements	$SiO_2 + Al_2O_3 + Fe_2O_3$ (%)	Min, 70.0
	Sulfur trioxide (SO_3) (%)	Max, 4.0
	Moisture content (%)	Max, 3.0
	Loss on ignition (%)	Max, 10.0

Table 3. Optimized condition for ball milling

milling speed (rpm)	ball to powder weight ratio	water to powder ratio	milling time (hours)
500	4.5	1	3

Table 4. Mixture proportions

Mixtures	(W/C)	Fine Aggregates (Kg/m^3)	Cement (Kg/m^3)	Nano Zeolite (%)
NZ-0-0.45	0.45	899	378	-
NZ-0-0.4	0.4	881	430	-
NZ-5-0.45	0.45	899	378	5
NZ-7-0.45	0.45	899	378	7
NZ-10-0.45	0.45	899	378	10
NZ-5-0.4	0.4	881	430	5
NZ-7-0.4	0.4	881	430	7
NZ-10-0.4	0.4	881	430	10

of ball mill mentioned in the Table 3 and make the finest particle of the nano zeolite (Karamian, *et al.*, 2015).

Specimen Preparation and Testing

The dry materials were mixed first, followed by the addition of water into the mixer. Then, the superplasticizer was added to the mixture to achieve a desirable workability. Immediately after mixing, the concrete mixtures were molded and consolidated by a vibrating table. The specimens were covered in the casting room for 24 h. The test samples were then demolded and moist cured at 23±1°C until the specified age for each test.

Table 3 summarizes the concrete mixture proportions. The control concrete without additive (NZ0), three mixtures were made by replacing 5,7and 10% nano zeolite instead of Portland cement; hereafter named as NZ-5, NZ-7 and NZ-10 respectively. The extension of 0.45 and 0.4 show water/cement ratio. By use of natural zeolite decreased the workability of concrete which was compensated by using a commercially available Polycarboxilate Superplasticizer. In this regard, by addition of that, the slump of the mixture was maintained in the range of 80-100 mm. As shown in Table 4, by increasing the replacement level from 5% to 10%, the superplasticizer consumption increased.

The compressive and tensile strength of the mixtures was measured on 300×150 mm cylindrical specimens by ASTM C39-86 and ASTM C496-90 respectively. Compressive tests have done for ages 7, 28 and 90

Table 5. Results of mechanical and durability tests (W/C= 0.45)

Test	Mixture ID / Ages	NZ-0-0.45	NZ-5-0.45	NZ-7-0.45	NZ-10-0.45
Compressive Strength (MPa)	7	29.3	23.6	24.2	26.9
	28	41	41.4	36.1	39.4
	90	44.4	51	47	53
Tensile Strength (MPa)	28	2.83	3.35	2.42	2.83
	90	3.4	3.85	3.61	3.7
Electrical Resistivity (kΩ.cm)	28	11.8	11.8	14	14.2
	90	15.9	16.2	22.6	21.1
Rapid Chloride Penetration (Coulomb)	28	2953	2100	1627	1510
	90	1704	1720	1570	1220

Table 6. Results of mechanical and durability tests (W/C= 0.4)

Test	Mixture ID / Ages	NZ-0-0.4	NZ-5-0.4	NZ-7-0.4	NZ-10-0.4
Compressive Strength (MPa)	7	37.2	24.4	30.5	24.5
	28	53.3	41	35.3	45.4
	90	56	43	41	50.3
Tensile Strength (MPa)	28	3.1	2.92	3.4	3.24
	90	3.7	3.87	3.57	3.4
Electrical Resistivity (kΩ.cm)	28	8.9	11.4	13.7	13.9
	90	9.2	16.3	17.8	16.5
Rapid Chloride Penetration (Coulomb)	28	2181	1760	1773	2497
	90	1960	716	791	1025

days also 28 and 90 days specimens were tested for tensile, electrical resistivity and rapid chloride penetration tests. The average of the results is presented in Tables 5 and 6.

RESULTS AND DISCUSIONS

As it can be seen in (Fig. 2), the compressive strength of specimens containing nano zeolite, have been de-

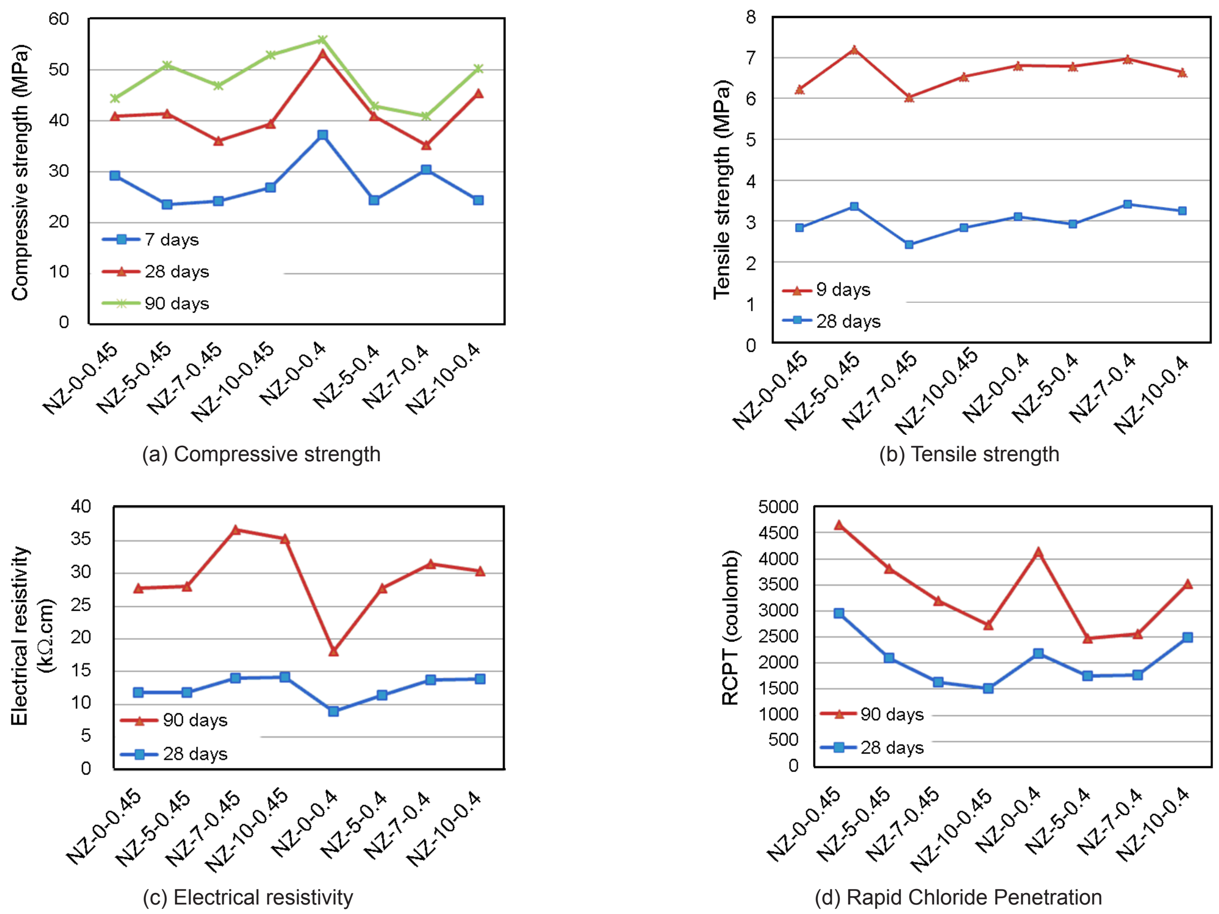

Fig. 2. Effects of nano zeolite as supplementary of cement in concrete

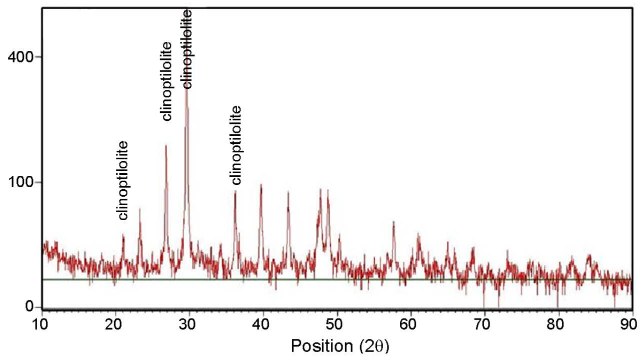

Fig. 3. XRD pattern

creased and maximum decrease refer to NZ-7 specimen and is same for both 0.45 and 0.4 W/C ratio. In term of compressive strength, it seems that natural zeolite performs better in blended cement composites with lower W/C ratios. For NZ-5 tensile strength has increase but is not significantly. The factors of durability were measured by electrical resistivity and rapid chloride penetration. Results show that incorporation of these mineral admixtures as pozzolanic materials have significant effects.

At the result of replacement, nano zeolite is not effective for mechanical characteristic. The XRD pattern (Fig. 3) Confirm that specimens contains micro zeolite have high index of durability. Fig. 4 shows

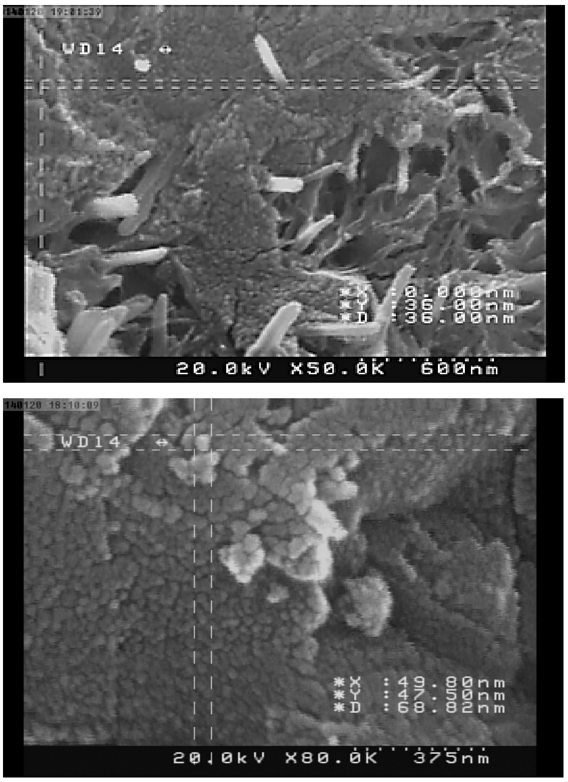

Fig. 4. SEM image transition zone

SEM image from specimens in 28 days that illustrate nano particle in cement paste in transition zone.

CONCLUSIONS

Based on the obtained results, the following conclusions can be drawn from this investigation:

1. The compressive strength of concretes containing additives was lower than the concrete without supplementary replacement at all ages. However, the percentages of reduction were lower at later ages, which can be attributed to lump in transition zone.

2. Decrease rate of tensile strength is less than compressive strength in all ages. Also effectiveness of additives on strength in 0.45 (W/C) is less than 0.4 (W/C), probably because of the lack of water, strength have been decreased.

3. The optimized mixture for achieve high quality of durability is incorporation 7% nano zeolite. This derive that, concrete contained 7% nano zeolite has high resistance in penetration chloride ion.

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