

# Transport properties and strength development of Blended Cement Mortars Containing Nano-Silica

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## ABSTRACT

*In the present study, the mechanical properties and durability of blended cement mortars containing nano-silica and natural pozzolans is investigated. Trass and pumice, two different Iranian natural pozzolan, are used in the experiments. For cement blends preparation, nano-silica replacement levels of 2, 3 and 4% by mass of cement was considered; each mixture contained one of the natural pozzolans with a fixed replacement percentage of 15. The Standard mortar samples were made with w/cm ratio of 0.485; the flow of all mortars ranged from 14 to 16 cm. All prepared mortars' samples were cured in saturated limewater until the test ages. The electrical resistivity, chloride permeability, capillary absorption, and compressive strength tests were performed on mortar samples in accordance with the standards. Generally, the results indicated significant improvement in durability parameters of mortars containing both nano-silica and natural pozzolan. Furthermore, it was observed that nano-silica can improve the strength development of natural pozzolan mortars and compensate the reduction in the initial compressive strength of natural pozzolans mortars. The blend containing 4% nano-silica and 15% Trass, was found to have the best performance from the view point of durability characteristics and compressive strength at all test ages.*

**Keywords:** Nano-Silica; Blended Cement; Natural Pozzolan; Durability.

## 1.0 INTRODUCTION

Today, an important topic of multidisciplinary studies is the environmental sustainability (Chen & Chen, 2007; Song & Chen, 2016). The sustainability of cement production and concrete production due to its considerable contribution to construction industry development and environmental issues has been widely concerned (Zhang *et al.*, 2017).

The recognized solutions for concrete sustainability are partially replacing cement with supplementary cementing materials (SCM), designing concrete mixtures with optimum content of cement, and improving concrete durability (Ramezaniapour, 2014). SCMs are found noticeably effective on reducing cement consumption and increasing the durability of mortar and concrete (Valipour *et al.*, 2013, Mirvalad & Nokken, 2015). They primarily have role in refinement of the concrete pore structure, which leads to higher strength and lower permeability (Habert *et al.*, 2008). Pozzolans, especially the natural ones, are mainly low-priced; therefore, can be

useful and effective when introduced in concrete mixtures (Valipour *et al.*, 2013).

In recent years, many studies have been performed on using nano-materials in blended cements in order to enhance the properties in hardened state. Zahedi *et al.* (2015) used nano-silica (NS) to improve the mechanical properties and durability of cement mortars containing rice husk ash (RHA), and found that nano-silica can improve compressive strength of RHA mortars at all ages; as well, it can effectively improve their durability at ages of 28 and 90 days. Shaikh *et al.* (2014) used nano-silica to enhance compressive strength of high volume fly ash mortars. They reported significant improvement (33% - 48%) in the 28-day compressive strength of high volume fly ash mortars containing 60% and 70% fly ash due to addition of 2% nano-silica; slight improvement was seen at fly ash contents of 40% and 50%. Similar results are also observed by other researchers (Zhang *et al.*, 2012; Hou *et al.*, 2013). Heikal *et al.* (2013) demonstrated that use of blended cements containing 45% granulated blast furnace slag and 3–4% nano-silica by mass had resulted in the highest

improvement of mechanical properties, hydration kinetics, and microstructure of hardened cement pastes and mortars. The mentioned observations have been proved by other authors (Liu *et al.*, 2016) as well. Eskandaria *et al.* (2015) also showed that incorporation of 2% nano-silica and 8% micro zeolite in cement blend resulted in a noticeable durability characteristic.

In summary, the role of the nano-silica in the cement matrix can be summarized as follows:

- Nanoparticles act as fillers in the empty spaces (Li *et al.*, 2004; Ji, 2005).
- Well distributed nanoparticles act as crystallization centers for hydrated products, increasing hydration rate (Shah *et al.*, 2015)
- Nanoparticles assist towards the formation of small in size  $\text{Ca}(\text{OH})_2$  crystals and homogeneous clusters of calcium silicate hydrate matrix (C–S–H) (Jo *et al.*, 2007).
- Nanoparticles improve the transition zone state between aggregates and paste.

This paper presents an experimental study carried out on synergistic effect of nano-silica and natural pozzolans (Jajrood Trass and Khash Pumice) on resistance of mortars against chloride ions permeability.

## 2.0 EXPERIMENTAL PROGRAM

### 2.1 Materials

Portland cement Type I-425, purchased from Tehran cement plant, in accordance with ASTM C150 (2018) was used in all mortar mixtures. In addition to cement, two natural pozzolans; Jajrood Trass and Khash Pumice were obtained. The former was sampled from Tehran cement plant and was ground in the laboratory. The latter was received from Khash cement plant in powder form. The chemical analysis of cement and supplementary cementing materials is presented in Table 1.

A colloidal nano-silica with pureness of 99%, average particle size of 12 nm, special surface area of 200  $\text{m}^2/\text{g}$ , and solid content of 18% was used in the experimental study.

The sand used in preparation of mortar samples was a graded silica sand with particle size distribution in accordance to ASTM C778 (2017) (See Table 2). The silica sand had a fineness modulus of 3.49 and specific gravity of 2350  $\text{kg}/\text{m}^3$ .

A third-generation Poly carboxylate ether-based super-plasticizer with brand name of PS-10, manufactured in "Shimi Sakhteman" factory was used to sustain the desired stability of nano-silica and consistency of mortar mixtures without increasing the water to cement ratio.

**Table 1.** Chemical composition and physical properties of cement and natural pozzolans

%Properties	Cement	Pumice	Trass
$\text{SiO}_2$	20.12	60.85	75.02
$\text{Al}_2\text{O}_3$	3.89	15.02	11.59
$\text{Fe}_2\text{O}_3$	3.46	5.44	2.14
$\text{CaO}$	63.22	9.71	3.7
$\text{MgO}$	3.02	1.94	1.19
$\text{SO}_3$	3.02	0.22	0.19
$\text{Na}_2\text{O}$	1.38	3.08	2.04
$\text{K}_2\text{O}$	0.8	2.2	3.08
$\text{Cl}$	0.074	0.064	0.033
Loss on ignition	0.81	3.15	8.5
Blaine specific surface area ( $\text{cm}^2/\text{g}$ )	3180	5400	3600

### 2.2. Procedures

In order to investigate the effect of addition of nano-silica to blended cements containing natural pozzolans, nine different mortar mixtures were prepared. Two types of natural pozzolan, Jajrood Trass and Khash Pumice, with a fixed replacement level of 15% of cement (by mass) were used. In addition, three cement replacement levels of 2%, 3%, and 4% was considered for nano-silica. All samples had a constant water-cement ratio of 0.485, and a constant sand-cement ratio of 2.75, in accordance with ASTM C109 (2016). Using super-plasticizers, the consistency of mixtures measured with flow table was kept in the range of 14 to 16 cm. Table 3 depicts the mortar mixtures.

**Table 2.** Sieved silica sand particle size distribution

Sieve size	Passing sieve (%)	
	ASTM C778	Experimental
1.18 mm (No. 16)	100	100
600 $\mu\text{m}$ (No. 30)	96-100	99
425 $\mu\text{m}$ (No. 40)	65-75	70
300 $\mu\text{m}$ (No. 50)	20-30	25
150 $\mu\text{m}$ (No. 100)	0-4	2

Mortar samples were produced in accordance with ASTM C109. Cement and pozzolan were manually blended in dry form whenever required. For mixtures containing nano-silica, a nano-silica solution in water was added during the mixing process, right at the time that water is considered to be added to the mixer. Before addition of nano-silica to the mixture, the nano-silica solution and the water were mixed by an ultrasonic probe with frequency of 100 Hz for 15 minutes. The mentioned process resulted in homogeneous distribution of nano-silica in water and prevention of their agglomeration; this was verified with particle size distribution test.

**Table 3.** Proportions of mortar mixtures

Mortar mix	Cement (kg/m <sup>3</sup> )	Pumice (kg/m <sup>3</sup> )	Trass (kg/m <sup>3</sup> )	Nano-silica (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Sand (kg/m <sup>3</sup> )	w/(cm)	Superplasticizer (%)	Flow (cm)
OPC	666	0	0	0	323	1831.5	0.485	0	16-15
P15	566	100		0	323	1831.5	0.485	0	15.5-14
T15	566		100	0	323	1831.5	0.485	0.43%	15.5-14
P15NS2	552.68	100	0	13.32	323	1831.5	0.485	0.41%	15.5-14
P15NS3	546.02	100	0	19.98	323	1831.5	0.485	0.94%	15.5-14
P15NS4	539.36	100	0	26.64	323	1831.5	0.485	1.18%	15.5-14
T15NS2	552.68	0	100	13.32	323	1831.5	0.485	0.84%	15.5-14
T15NS3	546.02	0	100	19.98	323	1831.5	0.485	1.23%	15.5-14
T15NS4	539.36	0	100	26.64	323	1831.5	0.485	1.58%	15.5-14

The prepared mortar was poured into 50 \* 50 \* 50 mm cubic molds and 100 \* 200 mm cylinders in 3 layers. Each layer was compacted by a vibrating table so that the air bubbles escaped out. The resulted samples were covered with a wet sack and a plastic cover and were kept in a humidified room for 24 hours. They were demolded after 24 hours, and cured in saturated limewater at 23 ± 2°C until testing.

### 2.3 Testing methods

#### Compressive strength

At ages of 3, 7, 28 and 90 days, the compressive strength tests were conducted on three 50 mm cubic mortar specimens of every mixture in accordance with ASTM C109 (2016). For each mixture, the average of three compressive strength test results was reported.

#### Rapid chloride migration test (RCMT)

This test investigates the performance of concrete, mortar, and other cement-based repair materials in harsh environments. Based on NT Build492 (1999), in order to determine the chloride resistivity of mortar mixtures at ages of 28 and 90 days, the accelerated RCM test was utilized. In this respect, three 50 mm thick slices were made by cutting a 100 \* 200 mm cylindrical specimen of every mixture from the middle. Then, the accelerated penetration of chloride ions were determined by subjecting mortar disks (diameter of 100 mm and thickness of 50 mm) to the electrical potential for 24 hours. The primary electrical potential value was set based on the initial charge passed within the 50 mm disks. Afterwards, the disks were removed from the test cells and axially divided into halves. In order to determine the depth of chloride penetration, the solution of 0.1 M AgNO<sub>3</sub> was applied on the surface of each split disk for identifying the penetrated area which turns to purple in this process. Finally, the migration coefficient average was calculated according to the equations of NT Build492. Applied voltage, initial and final temperature, and penetration depth of chloride ions and test duration are among the factors influencing the migration coefficient.

#### Surface electrical resistivity

Wenner four electrode method was utilized in order to define the surface resistivity of 100 \* 200 mm cylindrical specimens at ages of 7, 28 and 90 days.

#### Capillary absorption

According to BS EN 480-5 (2005), the capillary absorption was investigated. In order to determine the water absorption in each mortar mixture at ages of 7 and 28 days, three cubic specimens were utilized. The amount of absorbed water was calculated at 3, 6, 24 and 72 hours.

## 3. RESULTS AND DISCUSSION

### 3.1 Compressive strength

The results of average compressive strength (three samples at each age) for various mixtures are given in Table 4. To provide a better comparison, the normalized compressive strength results relative to the control mixture are presented in Fig.1.

**Table 4.** Average compressive strength of mortar mixes

Mortar mix	Compressive strength (MPa)			
	3 days	7 days	28 days	90 days
OPC	27	34.8	47	50.2
P15	17.8	26	35.4	40.6
P15NS2	19.6	27.6	37.4	41
P15NS3	24.8	35	41.6	46.4
P15NS4	26.8	39.8	51.2	56
T15	18.4	28.2	41.4	41.8
T15NS2	21.4	29.2	42	42.6
T15NS3	25.2	36	43.6	50.2
T15NS4	27.5	42.8	52.4	59.2

The addition of natural pozzolans decreased the strength of mortar samples, especially at early ages compared to the control specimen. This can be attributed to reduction of the cement content, and the natural pozzolans low activity at early ages of cement hydration. At later ages, with availability of enough time for formation of secondary C-S-H gel resulted by pozzolanic reactions (Pourkhorshidi *et al.*, 2010), the strength of samples containing natural pozzolans improved compared to the control specimens (OPC). For Pumice, the compressive strength compared to the control sample was enhanced from -26% at 3 days to -16% at 90 days, and for Trass, the

proportions changed from -23% at 3 days to -13% at 90 days.

It was observed that Jajrood Trass pozzolan had superior performance which can be attributed to its better pozzolanic activity compared to Khash Pumice (Ramezani pour *et al.*, 2010; Najimi *et al.*, 2012).

According to Fig. 1, the addition of nano-silica improved the compressive strength of pozzolanic mortars. As a result of increasing nano-silica content from 2 to 4% of cement mass, the ratio of compressive strength was increased from -25.29% to +14.37% for mixtures containing Trass at the age of 7 days. The use of 2% nano-silica showed a slight improvement in the mechanical properties of mortar samples. The greatest effect of nano-silica on samples was observed on ones containing mixed cements at 7 days of age, which is in agreement with previous research (Said *et al.*, 2012).

According to the nucleation theory, high level of lateral surface area in nano-silica particles accelerates and improves hydration reactions (Li *et al.*, 2004; Land & Stephan, 2012). On the other hand, the specific surface area and high roughness of nanoparticles' surface accelerates the rate of reactions of nano-silica significantly, which in turn results in the production of a secondary C-S-H gel (Jo *et al.*, 2007). Additionally, the small size of nanoparticles is effective in filling the cement matrix pores (Ji, 2005). In conclusion, all the mentioned effects lead to a denser cement matrix structure and consequently a remarkable improvement in the mechanical properties of mortars contained nano-silica.

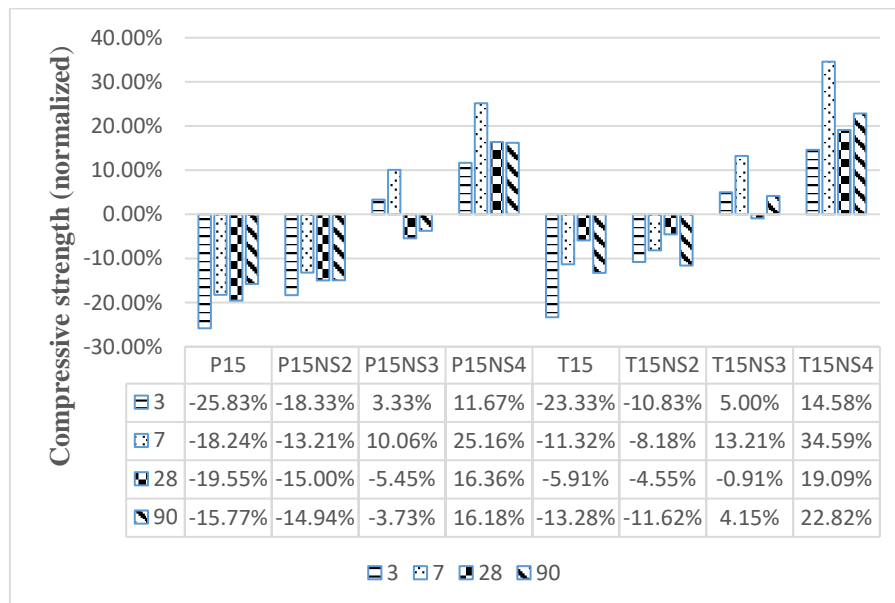
### 3.2 Rapid chloride migration test

The results of RCMT at the ages of 28 and 90 days are presented in Table 5. Normalized migration coefficients relative to the control mixture are also displayed in Fig. 2.

**Table 5.** Migration coefficient value of mortar mixes

Mortar mix	Migration coefficient ( $10^{-12} \text{ m}^2/\text{s}$ )	
	28 days	90 days
OPC	15.10	13.93
P15	13.61	12.67
P15NS2	13.30	12.54
P15NS3	13.17	9.83
P15NS4	10.21	8.01
T15	10.74	8.56
T15NS2	10.27	7.72
T15NS3	9.39	6.46
T15NS4	7.13	5.38

It can be inferred from Table 5 and Fig. 2 that the use of natural pozzolans has reduced the chloride ion penetration of all specimens at all ages. Trass showed a better performance compared to Pumice; similar results have been reported previously as well (Ramezani pour *et al.*, 2010). In other words, the reduction of the effective coefficient of chloride ion migration in comparison with the control specimen for samples containing Trass was 2.9 and 4.3 times greater than samples of Pumice at the age of 28 and 90 days, respectively.



**Fig. 1.** Normalized compressive strength results relative to control mix

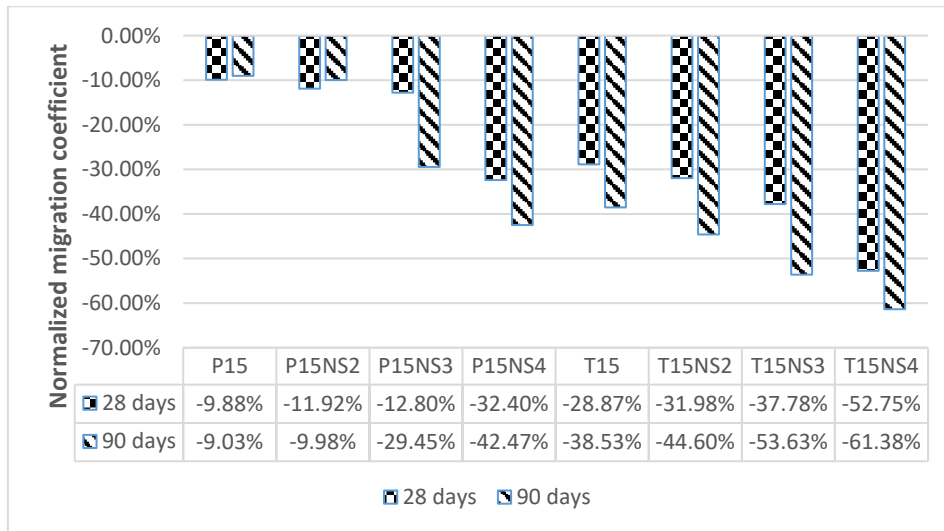


Fig. 2. Normalized migration coefficient value relative to control mix

Addition of nano-silica to the mixtures containing pozzolans has improved the performance of mortars against chloride ion migration at both ages of 28 and 90 days which is in agreement with previous research (Jalal *et al.*, 2015; Sharkawi, Abd-Elaty and Khalifa, 2018). Additionally, with an increase in the replacement percentage of nano-silica from 2 to 4, positive effect on mortar samples is more pronounced, such that adding 4 percent of the nano-silica has strongly reduced the chloride ion migration coefficient. As an instance, at the age of 90 days, T15NS4 and P15NS4, when compared to OPC sample, showed a reduced chloride ion migration coefficient of 61.8% and 42.5%, respectively.

Due to high pozzolanic activity of nano-silica particles, they consume coarse calcium hydroxide crystals that had created highly porous media. So, nano-silica particles create a secondary dense C-S-H gel (Shaikh *et al.* 2014) and diminish the structure of the cavities, pores, and communication paths between them. Moreover, the optimal and denser structure of hydration products due to the nucleation theory (Li *et al.*, 2004), the acceleration of the activity of natural pozzolans used in the mortar along with the nano-silica, and the effect of nanoparticle filling (Ji, 2005) result in significant improvement in the structure of cavities in the cement matrix. This leads to a significant reduction in the chloride ions migration rate.

### 3.3 Electrical resistivity

The results of Electrical resistivity test at the ages of 28 and 90 days are presented in Table 6. Normalized electrical resistivity relative to the control mixture are also displayed in Fig. 3.

Similar to the results of chloride ion migration, the presence of natural pozzolans has increased the electrical resistance of all specimens at all ages. Trass showed a very good performance in improving

the electrical resistance of the samples compared to Pumice, which is in accordance with previous research (June *et al.*, 2010; Pourkhorshidi *et al.*, 2010). The mentioned observation is in accordance with RCMT and compressive strength test results. The ratio of the increase in the electrical resistance compared to the control specimen for samples made with Trass is 5.7 times greater at 28 days and 2.9 times greater at 90 days compared to the Pumice samples.

Table 6. Electrical resistance of mortar mixes

Mortar mix	Resistivity (Ω.m)	
	28 days	90 days
OPC	87	105
P15	96	155
P15NS2	98	163
P15NS3	104	180
P15NS4	133	200
T15	138	250
T15NS2	156	280
T15NS3	167	295
T15NS4	226	322.5

The combined effect of nano-silica along with natural pozzolans has improved the surface electrical resistivity of all specimens at both ages of 28 and 90 days. Also, with increasing the percentage of nano-silica replacement from 2 to 4 %, the effect of nano-silica on improving the electrical resistivity of the mortars has increased. Sample T15NS4, that represents the effect of simultaneous combination 4%-nano-silica and Trass showed the best performance; the electrical resistivity of the mortar has improved by 207.1% compared to the control specimen at 90-day testing. Using 2% nano-silica had a slight effect on improving the mechanical properties of the mortar sample.

### 3.4 Capillary absorption

The results of capillary absorption test at the ages of 28 and 90 days are shown in Table 7. Normalized Capillary absorption to the control mix are also displayed in Fig. 4.

The presence of natural pozzolans due to their filling effect, the pozzolanic reactions, and the production of secondary C-S-H gel, improves the structure of the cement matrix (Najimi *et al.*, 2012). Therefore, it is expected that the absorption rate and the total absorption of pozzolanic mortars decreases compared to the control specimens.

Considering the capillary absorption results, samples made with Trass had improved performance compared to Pumice. Moreover, as the age of samples increased from 28 to 90 days, the absorption

of the samples at all the corresponding hours decreased due to the progress of pozzolanic reactions and the completion of cement hydration.

At 28 days, the addition of nano-silica to the mortars made with pozzolanic cements reduced the absorption during the first hours as well as the 72-hour absorption of samples; this is in agreement with other studies (Najigivi *et al.*, 2012; Sharkawi *et al.* 2018). This indicates a reduction in the volume of the capillary pores, and also the reduction of the connection between them, due to the consumption of coarse calcium hydroxide which creates a highly porous media filled with a C-S-H dense gel (Shaikh, *et al.* 2014). In addition, the effect of nanoparticle filling (Ji, 2005), along with activating the pozzolanic reaction of other pozzolans used in this study should be mentioned.

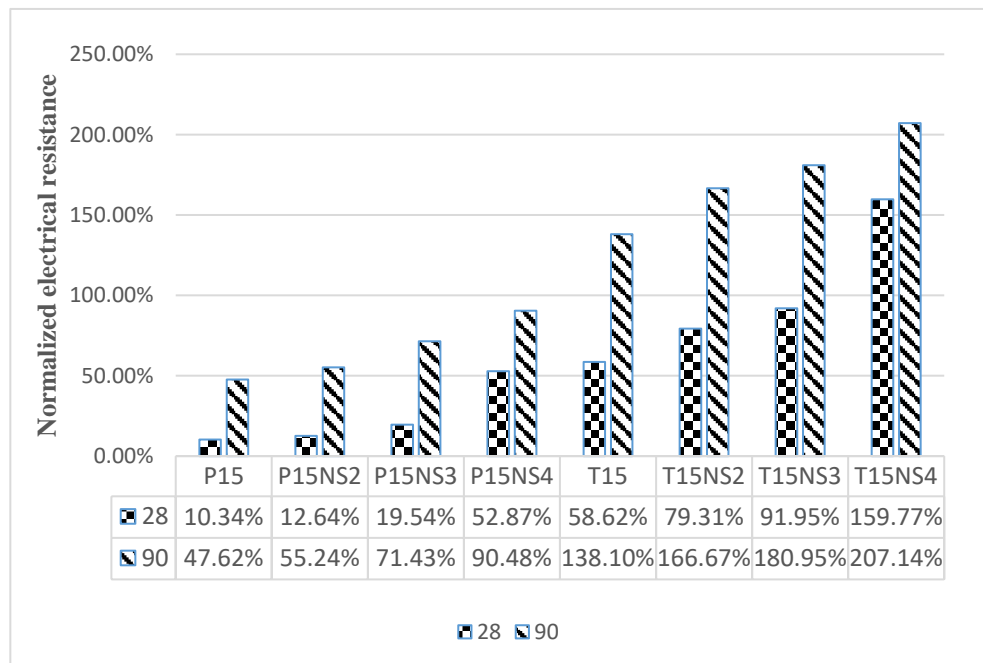
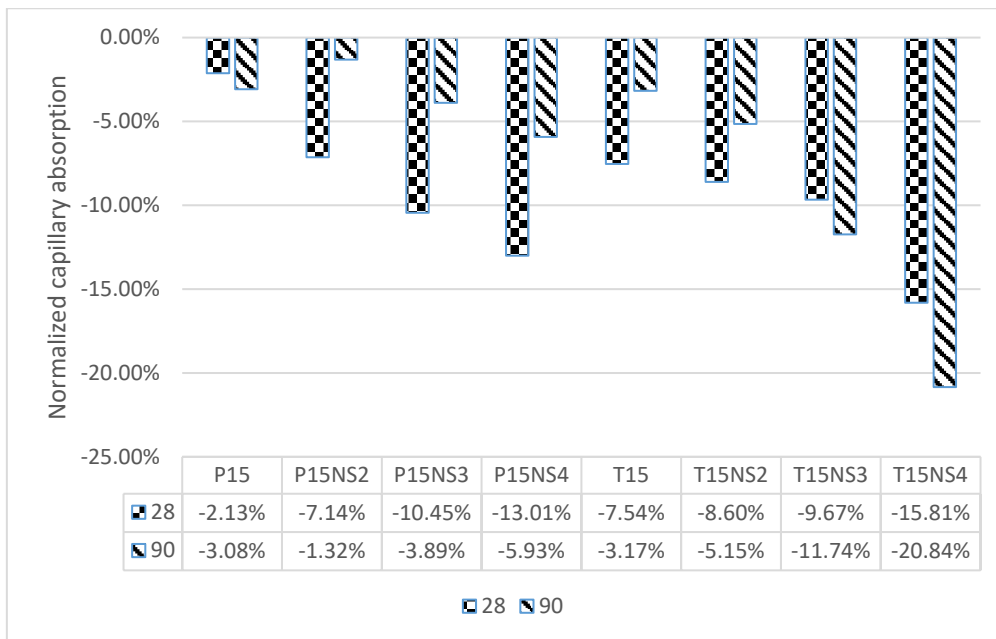


Fig. 3. Normalized electrical resistance relative to control mix

Table 7. Capillary absorption values of mortar mixes

Mortar mix	Water absorption (g)							
	28 days				90 days			
	3h	6h	24h	72h	3h	6h	24h	72h
OPC	10.16	14.28	19.31	19.76	7.24	9.29	15.59	16.70
P15	5.94	8.59	15.19	19.34	5.65	7.76	13.70	16.19
P15NS2	5.54	7.26	13.26	18.35	7.68	10.78	16.12	16.48
P15NS3	5.36	7.27	12.56	17.70	9.19	11.76	15.88	16.05
P15NS4	5.31	7.43	13.28	17.19	6.69	8.92	14.22	15.71
T15	5.48	7.79	13.95	18.27	5.14	7.24	13.53	16.17
T15NS2	5.68	7.91	14.69	18.06	7.93	10.13	14.47	15.84
T15NS3	6.28	8.05	14.58	17.85	6.53	8.09	11.96	14.74
T15NS4	5.94	7.91	13.90	16.64	6.31	8.13	11.50	13.22



**Fig. 4.** Normalized capillary absorption in 72-h, relative to control mix

It was observed that with an increase in the percentage of nano-silica replacement from 2 to 4 percent, the absorption was reduced at all ages (except for the T15NS3 sample). In particular, this is confirmed by the reduction of absorption at the end of 72 hours.

At the age of 90 days, the addition of nano-silica caused more water to be absorbed in the early hours, compared to nano-silica-free pozzolan specimens and similar mixture of nano-silica at the age of 28 days, which can be attributed to the increase in the number of intermediate capillary pores due to the conversion of large capillary pores to average ones (Chithra *et al.* 2016). This conversion is a result of more progress in pozzolanic reactions of nano-silica which in turn increases the speed of capillary absorption due to the shrinking capillary tubes and increasing their number. But, in general, due to the reduction in the total volume of capillary pores because of the microstructural improvement of the mortar and the discontinuity of the pores as a result of using nano-silica, the capillary absorption has decreased in the range of 72 hours. This reduction is more significant if more nano-silica is added. The best performance is attributed to the T15NS4 sample, which had 20.8% lower absorption at 90 days (after 72 hours) compare to the control samples.

#### 4.0 CONCLUSIONS

- i) Even after 90 days, the mortar samples containing natural pozzolans could not achieve the compressive strength of the control sample at the same age.
- ii) The reduction of chloride ion migration coefficient, electrical resistance increase, and capillary water absorption decrease indicated that Jajrood Trass and Eskandan Pumice enhanced the structure of cavities and pores of cementitious matrices

- iii) Jajrood Trass showed more satisfactory performance in mechanical properties and durability characteristics compared to Eskandan Pumice. This was attributed to its greater pozzolanic activity which has been observed in previous research.

- iv) Despite the fact that the performance of both pozzolans with regards to the mechanical properties of mortars was almost identical, the effectiveness of Jajrood Trass from the point of view of durability was more noticeable. For instance, Jajrood Trass improved the surface electric resistivity 5.6 times more than Khash Pumice at the age of 28 days.

- v) Replacement of blended cement with nano-silica resulted in improved mechanical and durability properties of mortar samples at all ages. The reasons can be considered as: 1- The theory of nucleation, which provides large amount of silica surfaces for precipitation of hydration products resulting in enhancement and acceleration of cement hydration. 2- The nano-silica high pozzolanic activity leads to the formation of secondary C-S-H gel by reacting with coarse calcium hydroxide crystals 3- The nano-silica small size and its high adhesion characteristic results in the filling of pores between the structures of the cementitious matrix.

- vi) The replacement of blended cement with 2% nano-silica did not demonstrate a significant effect on improvement of mechanical properties and durability of natural pozzolans specimens. However, increasing the replacement level from 2 to 4%, properly modified the mortar properties.

- vii) Nano-silica incorporated in blended cement showed remarkable effect on the compressive strength of the specimens at the age of 3 and 7 days because of the noticeable effects of nano-silica at the early ages. The reason can be considered as the

theory of nucleation which leads to the cement hydration acceleration and the ability of nano-silica filling pores in cement matrix rapidly.

viii) Finally, the T15NS4 specimen showed the best mechanical properties and the highest durability.

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