

Original paper

Evaluation of the Mechanical Properties and Durability of the Mortar Coating Containing Micro silica and Nano silica

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ABSTRACT

Reinforced concrete is vulnerable to corrosion due to chloride attacks. Coating mortars containing pozzolanic materials are available to increase concrete resistance to a corrosive environment. With the utilization of silica in different sizes (micro/nano), more durable concrete can be achieved. The aim of this study is to investigate the mechanical and durability properties of mortars containing micro and nano-silica. In this respect, 0 to 7.5% micro silica and 0 to 5% nano-silica in two different particle sizes were substituted in Portland cement. According to the results, adding micro and nano silica to the concrete mixture enhances the mechanical and durability of the concrete. The optimum mix design consists of 7.5% micro silica with 5% nano-silica of lower fineness. With regard to the nano-silica particle size, lower fineness generally performs better than the higher one due to a greater likelihood of the hydration process developing over time. An increase in shrinkage is the downside of using micro/nano-silica pozzolan. However, no negative influence on concrete durability was observed.



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1. Introduction

In the concrete industry, silica is one of the useful materials which has a key role in binding and filling the pores in high-performance concrete. The common product of silica is micro silica with particle diameter in the range of approximately 0.1 to 1 μ m. Micro silica generally contains more than 85% silicon dioxide. Nano silica consists of particles less than 100 nm in diameter which is provided in dry powder or colloidal suspension. According to investigations, 3 main mechanisms have been proposed for nano silica's effective properties: (1) Filling effect, (2) Nucleation, and (3) Pozzolanic activity. (1) Silica particles are placed among calcium silicate hydrate gel particles due to the significant fineness and superficial adhesion [1]. Concrete microstructure porosity can be reduced when nano silica particles perform as fillers [2]. (2) Nano silica particles form a nucleus that develops cement hydration products in solution. These particles play the role of skeleton-like aggregates against the cement gel. This connection to cement gel plays a major role in supplying strength [3]. (3) The reaction of amorphous silica with calcium hydroxide forms calcium silicate hydrate gel which enhances the interfacial transition zone and reduces the concrete porosity [4-5]. One of the advantages cited for using nano-silica particles is that it reduces the concrete permeability significantly [6].

Experiments show that the speed of colloidal silica reaction with calcium hydroxide is much higher than with micro silica. Therefore, low amounts of nano silica have the same pozzolanic effect as higher amounts of micro silica in a shorter time [7]. The microstructure of concrete containing nano silica is more homogeneous and dense than a control [8].

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Krishnakumar et al. [9] reported that adding 5 to 12.5% micro silica to normal concrete reduces the chloride migration and flux of chloride in Rapid Chloride Migration Test (RCMT) and Rapid Chloride Permeability Test (RCPT) tests respectively. Bagheri et al [10] reported the same results in this respect. Electrical resistivity measurements of concrete indicate that both micro and nano silica increase the resistivity and consequently enhance corrosion resistance. Nano silica is more effective in the early stages [11]. The results of other investigations about the mechanical properties and durability of nano silica specimens with different substitutions are shown in Table 1.

Table 1. Mechanical and durability properties of nano silica concrete based on literature review.

| Researcher(s) | Nano Silica Replacement (%) | Parameters (and Effects) |
|--|-----------------------------|---------------------------------|
| Nematian et al [12] | 0, 2, 4, 6, 8, 10 | |
| Verma [13] | 0, 1.5, 2, 2.5 | |
| Kelagiri et al [14] | 0, 1, 1.5, 2, 2.5 | |
| Lin et al [15], Zapata et al [16], Boshehrian et al [17] | 0, 1, 2, 3 | |
| Riahi et al [18-19], Givi et al [20-22] | 0, 0.5, 1, 1.5, 2 | Compressive Strength (Increase, |
| Jo et al [3-5, 23-25], Esmaeili et al [26] | 0, 3, 6, 9, 12 | [20]: Decrease at 3%) |
| Li et al [3,5], Latifi et al [27] | 0, 3, 5, 10, 20 | |
| Ramezanianpour et al [28-29] | 0, 2.5, 4.5, 6.5 | |
| Ibrahim et al [30] | 0, 2.5, 5, 7.5 | |
| Kontoleontos et al [31] | 0, 2, 4 | |
| Nili et al [32] | 0, 1.5, 3, 4.5 | |
| Madani et al [23] | 0, 1, 2, 3, 5, 7.5 | Electrical Resistivity |
| Ramezanianpour and Firoozmakan [29] | 2.5, 4.5, 6.5, 8.5 | (Increase) |
| Jalal et al [33] | 0, 2 | |
| Ramezanianpour and Firoozmakan [29] | 2.5, 4.5, 6.5, 8.5 | Elux of Chlorido Long (PCPT) |
| Said et al [34] | 0, 6, 12 | (Decrease) |
| Madani et al [23] | 0, 1, 2, 3, 5, 7.5 | (Decrease) |
| Kong et al [35] | 0, 0.25, 0.5, 0.75, 1 | Chlorido Iono Donotration |
| He and Shi [36] | 0, 1 | (Decrease [20], Increase) |
| Madani et al [23] | 0, 1, 2, 3, 5, 7.5 | (Decrease, [29]: Increase) |
| Sadrmomtazi et al [37] | 0, 3, 5, 7 | Water Absorption |
| Firoozmakan [38] | 0, 5 | (Decreace) |
| Jalal and Noorzad [39] | 0, 2 | (Decrease) |

As seen in the above table and other studies, the optimal nano silica replacement reported in investigations is generally in the range of 3 to 10%. Almost all the researchers believe that nano silica enhances the mechanical and durability properties of concrete. However, the side effects of nano silica utilization should not be neglected. For instance, high shrinkage and shortage of long-term strength development are two main side effects of using nano-silica in concrete.

Sadrmomtazi et al. [37] used 3, 5, and 7% nano-silica in mortar specimens and measured the drying shrinkage at 90 days. They indicated that the shrinkage will increase with the increase in nano silica replacement. Yang [24] also reported that although the addition of nano-silica into the cement paste increases the compressive strength, shrinkage is accelerated with the passage of time which causes early-age cracking in concrete. He also expressed that increases in compressive strength with nano silica addition occur at optimal nano silica content. Further increase in nano silica reduces the compressive strength. This issue was observed in other investigations [7]. Oltulu et al [7] believed that the growth of compressive strength of nano specimens is maximum at 7-days and as time goes on, this higher strength will be decreased.

Roa [40] studied the effect of micro silica on drying shrinkage. He finally observed that the presence of micro silica in concrete mixes increases the drying shrinkage at 28-days of age.

Some researchers studied the effects of both micro and nano-silica on the mechanical and durability properties of different types of concretes [41-59]. Li et al [41] studied the combined effect of nano and micro silica on the durability of mortar. They reported that for enhancing the strength, carbonation resistance, and sulfate resistance, 1% nano-silica was almost as good as 10% micro silica. However, for improving the chloride resistance, $1\%{-}2\%$ nano-silica was less effective compared to 10% micro silica maybe because the filling effect of 1%-2% nano-silica was not as good as that of 10% micro silica. The combination of micro and nano silica showed certain synergistic effects in the sense that the combined effects were larger than the respective sums of the individual effects. They finally suggest that nano silica should not be added alone but should be added together with micro silica for the best overall performance [41-42]. It was found that nano silica has much higher superplasticizer demand and cementing efficiency than micro silica. Nevertheless, at the same strength, the superplasticizer demand of micro + nano silica is not higher than that of pure cement [43]. Naddaf et al [44] investigated the effect of nano silica replacing micro silica to reduce the corrosion rate of steel in concrete subjected to a corrosive environment. Various combinations of micro and nano silica weigh a constant 11% in total of the weight of cement used in each sample. The corrosion potential, linear polarization, and impedance spectroscopy were

some of the electrochemical tests carried out. They finally suggested the combined use of 9.4% micro and 1.6% nano-silica for achieving the optimum reduction in the rate of corrosion with cost consideration. Zhang et al [45] used silica fume and nano SiO2 to modify cement mortar as a surface protection material. Protection material was coated on the surface of the concrete and then bond strength, hydration process, shrinkage consistency, chloride penetrability, and compressive strength were investigated. According to the results of this experiment, the matrix coated with surface protection material has good integrity, with excellent interfacial bond strength and little difference in shrinkage, and the chloride diffusion coefficient of the system considerably declined. Emamian et al [46] reported that the use of simultaneous micro and nano silica led to a decrease in the porosity and an increase in the flexural and compressive strengths. This is due to the synergistic effect on the microstructure of cement paste. Sharkawi et al [47] conducted a study to evaluate the synergistic influence of micro-nano silica addition on the durability performance of cementitious materials. They resulted that concrete corrosion protection and mortar sulfate resistance were remarkably enhanced by using 2% nano + 8% micro silica, instead of the optimum micro or nano-silica cement replacement ratios.

Utilization of nano-silica in concrete enables reaching a significant compressive strength higher than the normal or micro-silica concrete in a shorter time. However, this superiority in gaining strength will not be so tangible with time [8].

Nano silica, by enhancing the concrete microstructure through pozzolanic reaction and also filling the pores reduces the concrete permeability significantly. Micro silica has a similar impact on permeability reduction, but due to the slower pozzolanic reaction compared with nano silica, its effect appears at linger times [8]. A combination of nano and micro silica could mitigate the side effect of nano silica. The aim of this study is to evaluate the effect of these pozzolans separately and with each other in different amounts to find an appropriate mix design for coating mortars.

2. Experimental Plan

2.1. Materials

Micro and nano-silica are used as supplementary materials to improve the mechanical and durability properties of concrete. The physical properties and chemical composition of cement and pozzolanic materials used in this investigation are shown in Tables 2 and 3.

Table 2. Cement and micro silica specification.

| Specification | | Cement | Micro Silica |
|---|---|--------|--------------|
| Silicon Dioxide (SiO ₂) | % | 22.52 | 87.5 |
| Aluminum Oxide (Al ₂ O ₃) | % | 5.24 | 0.5 |
| Ferric Oxide (Fe ₂ O ₃) | % | 3.86 | 1.53 |
| Calcium Oxide (CaO) | % | 59.8 | 1.27 |
| Magnesium Oxide (MgO) | % | 4.16 | 1.01 |
| Sulfur TriOxide (SO3) | % | 2.12 | 0.46 |
| Sodium Oxide (Na ₂ O) | % | 0.43 | 0.36 |
| Potassium Oxide (K ₂ O) | % | 0.78 | 1.14 |
| Chlorides (Cl) | % | - | - |
| Loss on Ignition (LOI) | % | 0.85 | 5.92 |
| Insoluble Residue | % | 1.07 | - |
| Titanium Oxide (TiO2) | % | - | 0.02 |
| Phosphorus Oxide (P ₂ O ₅) | % | - | 0.13 |
| Manganese Oxide (MnO) | % | - | 0.086 |

Size distribution(s)

| Specific Surface Area | m^2/g | 0.285 | 21^{*} |
|-----------------------|---------|-------|----------|
| Specific Gravity | - | 3.18 | 2.14 |

| Table 3. Nano silica specification. | | | | | | | | | | |
|-------------------------------------|---------|------|------|--|--|--|--|--|--|--|
| Specification | | L100 | L300 | | | | | | | |
| Silicon Dioxide (SiO ₂) | % | 40 | 25 | | | | | | | |
| Sodium Oxide (Na ₂ O) | % | 0.43 | 0.35 | | | | | | | |
| Loss on Ignition (LOI) | % | < 1 | < 1 | | | | | | | |
| Moisture content | % | 60 | 75 | | | | | | | |
| Specific Surface Area | m^2/g | 100 | 300 | | | | | | | |
| Particle Size | nm | 36.2 | 12.9 | | | | | | | |
| Viscosity | MPa | < 15 | < 7 | | | | | | | |
| | | | | | | | | | | |

A Zetasizer experiment is carried out on nano-silica samples (see Fig. 1). The average particle size for L100 and L300 silica cells are 36.2 and 12.9 nanometers respectively. Zeta potential for both nano silica types is between -40 to -60. According to the ASTM D4187-82, the nano-silica which is used in this study provides appropriate stability.



Fig 1. Zetasizer results for nano silica: L100 (Right) and L300 (Left).

The aggregates used in this investigation are sands. The specific gravity and saturated surface dry (%SSD) are 2.57 and 2.95 respectively.

50 100 Diameter (nm) 500 1000

10

and durability properties of cement mortar, supplementary materials were gradually added to the standard mortar composed with type 1 cement to make 15 mix designs as shown in Table 4.

2.2. Mix designs

%in dass

10

5

In order to investigate the effect of nano-silica with different specific surface areas (100 and 300 m²/g), micro silica and their combination on mechanical

| _ | Table 4. Mix designs of mortar specimens. | | | | | | | | | | | | |
|---|---|------|------|-------|-------|----------------|-----------------|------|------------|--|--|--|--|
| _ | Mix Id | C | W | S | W/C | Nano Silica | Micro Silica | SP/C | Flow Table | | | | |
| | | (gr) | (gr) | (gr) | | (gr) | (gr) | (%) | (mm) | | | | |
| 1 | OPC | 1000 | 485 | 2750 | 0.485 | 0 | 0 | 0.5 | 14.5 | | | | |
| 2 | 5L100 | 950 | 485 | 2750 | 0.485 | 50 | 0 | 1.2 | 13.5 | | | | |
| 3 | 5L300 | 950 | 485 | 2750 | 0.485 | 50 | 0 | 2.4 | 13.8 | | | | |
| 4 | 2.5L100 | 975 | 485 | 2750 | 0.485 | 25 | 0 | 0.8 | 14.5 | | | | |
| 5 | 2.5L300 | 975 | 485 | 2750 | 0.485 | 25 | 0 | 1.4 | 14.8 | | | | |
| 6 | 5M | 950 | 485 | 2750 | 0.485 | 0 | 50 | 0.6 | 14.5 | | | | |
| 7 | 7.5M | 925 | 485 | 2750 | 0.485 | 0 | 75 | 0.8 | 14 | | | | |
| 8 | 5M+2.5L100 | 925 | 485 | 2750 | 0.485 | 25 | 50 | 2.6 | 14.5 | | | | |
| 9 | 5M + 5L100 | 900 | 485 | 2750 | 0.485 | 50 | 50 | 3 | 14.3 | | | | |

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| 10 | 5M+2.5L300 | 925 | 485 | 2750 | 0.485 | 25 | 50 | 3.2 | 15.2 |
|----|----------------|-----|-----|------|-------|----|----|-----|------|
| 11 | 5M+5L300 | 900 | 485 | 2750 | 0.485 | 50 | 50 | 3.5 | 14.1 |
| 12 | 7.5M + 2.5L300 | 900 | 485 | 2750 | 0.485 | 25 | 75 | 3.8 | 14.5 |
| 13 | 7.5M+2.5 L100 | 900 | 485 | 2750 | 0.485 | 25 | 75 | 3.3 | 13.8 |
| 14 | 7.5M + 5L300 | 875 | 485 | 2750 | 0.485 | 50 | 75 | 4.6 | 14 |
| 15 | 7.5M + 5L100 | 875 | 485 | 2750 | 0.485 | 50 | 75 | 4.1 | 14.8 |

As seen in Table 4, 2.5 and 5% nano-silica and 5 and 7.5% micro silica were substituted by ordinary Portland cement (OPC).

to the experimental design, Superplasticizer consumed from 0.5 to 4.6% of cement content for OPC to 7.5M+5L300 mix respectively.

Superplasticizer is used in mixes to satisfy mortar workability. Due to the increase in the specific surface area of nano silica and its content in mixes, higher Superplasticizer content is needed to satisfy workability. According

In mix designs for shrinkage test, the water content is consumed as needed for workability fixed at 100 ∓ 5 mm. Details of mixed designs are shown in Table 5.

| | Mix Id | C (gr) | W (gr) | Micro Silica (gr) | Nano Silica (gr) | W/C | S (gr) |
|----|----------------|-----------|--------|-------------------------|------------------------|------|--------|
| 1 | OPC | 750 | 337.5 | 0 | 0 | 0.45 | 1500 |
| 2 | 5L100 | 731.24 | 375 | 0 | 18.76 | 0.5 | 1500 |
| 3 | 5L300 | 712.5 | 412.5 | 0 | 37.5 | 0.55 | 1500 |
| 4 | 2.5L100 | 731.24 | 390 | 0 | 18.76 | 0.52 | 1500 |
| 5 | 2.5L300 | 712.5 | 427.5 | 0 | 37.5 | 0.57 | 1500 |
| 6 | 5M | 712.5 | 352.5 | 37.5 | 0 | 0.47 | 1500 |
| 7 | 7.5M | 693.74 | 367.5 | 56.26 | 0 | 0.49 | 1500 |
| 8 | 5M+2.5L100 | 693.74 | 375 | 37.5 | 18.76 | 0.5 | 1500 |
| 9 | 5M + 5L100 | 675 | 412.5 | 37.5 | 37.5 | 0.55 | 1500 |
| 10 | 5M+2.5L300 | 693.74 | 390 | 37.5 | 56.26 | 0.52 | 1500 |
| 11 | 5M+5L300 | 675 | 427.5 | 37.5 | 37.5 | 0.57 | 1500 |
| 12 | 7.5M + 2.5L300 | 675 | 390 | 56.26 | 18.76 | 0.52 | 1500 |
| 13 | 7.5M+2.5 L100 | 675 | 375 | 56.26 | 18.76 | 0.5 | 1500 |
| 14 | 7.5M + 5L300 | 656.24 | 442.5 | 56.26 | 37.5 | 0.59 | 1500 |
| 15 | 7.5M + 5L100 | 656.24 | 427.5 | 56.26 | 37.5 | 0.57 | 1500 |

Table 5. Mix designs of mortar specimens for shrinkage experiment.

In addition to the above mixes, another mix design is prepared for based concrete production to perform the optimum mix design which is finally determined as a coating. Table 6 shows the details.

| Table 6 | . Mix | design | of | substrate | concrete. |
|---------|-------|--------|----|-----------|-----------|
|---------|-------|--------|----|-----------|-----------|

| С | W | Agg. | W/C | Slump | |
|-------------|----------------------|----------------------|------|--------|--|
| (kg/m³) | (kg/m ³) | (kg/m ³) | | (mm) | |
| 400 | 180 | 1750 | 0.45 | 70-100 | |

2.3. Preparation of specimens

Here, sample preparation procedures are described for coating mortars and substrate concrete.

2.3.1. Mortar specimens

At first, micro/nano-silica, water, and Superplasticizer were manually mixed. After that cement is added to the mentioned solution and mixing was continued for about 30 sec. Finally, aggregates were added to the paste and the complex was mixed for 30 sec and 60 sec at low and high speed respectively. The workability of specimens was measured by a flow table according to ASTM C230. Eventually, the mortar was poured into cubic and cylinder molds with the help of vibration. After surface finishing, specimens were kept in wet sacks and plastic covers. After 24 hours, the samples were remolded to be cured in lime water until the time of the experiment.

2.3.2. Compound specimens

2.3.2.1. Substrate concrete

At first, fine and coarse aggregates were mixed in dry mode. Subsequently, water was added to reach the aggregates to SSD. Then cement and water were gradually added to make 30 L concrete. Concrete samples were molded in $50 \times 50 \times 100$ cm³ dimensions. After vibration, finishing the surface of the

samples was carried out with a wire brush. After similar covering, substrate concrete samples were kept at 23±2 °C and 50±4% moisture content. After 28 days, based concrete will be repaired by optimum coating mortar.

2.3.2.2. Coating

After more than a month from the time of the production of substrate concrete, coating mortars were prepared. Coatings were made with the control and optimum mix designs.

Before coating is carried out, the surface of the substrate concrete was monitored from the viewpoint of health and contamination of harmful substances. Before coating implementation, a rough surface was prepared on the substrate concrete with a rotary wire brush and then the moisture content of the surface is changed to SSD with water sprinkling. Finally, coatings were implemented on the substrate concrete.

2.4. Description of experiments

In this study, the mechanical and durability properties of pozzolanic mortars were evaluated by different experiments including compressive strength, capillary absorption, electrical resistivity, rapid chloride migration, and shrinkage. Test methods and other details are shown in Table 7.

| Table 7. Experiments specifications. | | | | | | | | |
|--------------------------------------|--------------------|--|----------------------|--|--|--|--|--|
| Experiment | Test method | Specimens | Test duration (days) | | | | | |
| Compressive | ASTM CLOO | Cubic (5×5×5 cm ³) | 7 22 1 00 | | | | | |
| strength | A51M C109 | (3 specimens at each age) | 7, 28 and 90 | | | | | |
| C : 11 1 + | DS EN 490 5, 2005 | Cubic $(5 \times 5 \times 5 \text{ cm}^3)$ | 7 1 20 | | | | | |
| Capillary absorption | D5 EIN 460-5: 2005 | (3 specimens at each age) | 7 and 20 | | | | | |
| Electrical accientation | A ASUTO TD OF | Cylinder ($10 \times 20 \text{ cm}^2$) | 7 28 1 00 | | | | | |
| Electrical resistivity | AASHIO IF 93 | (2 specimens at each age) | 7, 28 and 90 | | | | | |
| Rapid chloride | NT D:14 409 | Cylinder (5×10 cm ²) | 29 1 00 | | | | | |
| migration test | INT Dulla 492 | (3 specimens at each age) | 28 and 90 | | | | | |
| Shrinkage | ASTM CEOC | Prismatic (2.5×2.5×28.5 cm ³) | 4 11 19 1 25 | | | | | |
| | A51M C590 | (2 specimens at each age) | 4, 11, 10 and 25 | | | | | |

3. Test Results

3.1. Compressive strength

Compressive strength results are obtained at the age of 7, 28 and 90 days (see Fig. 2).



Fig 2. Compressive strength results at 7, 28 and 90 days.

As seen in Fig. 2, the following results can be drawn:

- Compressive strength increases with time due to a reduction in porosity and an increase in the cement hydration process.

- OPC specimens had lower strength results compared with mixes containing micro/nano-silica.

- Substitution of 7.5% micro silica in OPC was more effective than 5% substitution in almost all combined mixes, especially at longer times. Of course, this superiority of 50% increase in micro silica content has not been established at an earlier time for the 7.5M+2.5L100 specimens.

- In regard to nano silica content, it should be noted that 5% substitution led to higher compressive strength results compared to 2.5%.

- Although the increase in fineness of nano silica from 100 to 300 m2/g led to a small increase in compressive strength results at earlier times, at later

times better results occurred from the lower fineness. It may be due to the lack of full dispersion of nano-silica with higher fineness in the cement matrix.

- By adding just nano silica in both fineness's and both substitutions, the compressive strength results are less than 40 MPa at 90 days. However, micro silica in this regard is more effective. Adding just micro silica brings the results to a higher level of more than 40 MPa. Therefore, a combination of micro silica and nano-silica maintains superior performance.

3.2. Capillary absorption

The capillary absorption of specimens was measured at 7 and 28 days. Results are shown in Fig. 3.





As seen in Fig. 3, nano silica specimens have a maximum reduction in absorption coefficient at 7-days. Although lower absorption of higher fineness of nano-silica can be observed in a few specimens, the better performance is for lower fineness, especially at 28days.

Nano silica particles absorb lots of water due to their high fineness. At an early age, enough water is more accessible for nano silica reaction, so nano silica specimens performed well in filling the pores and disconnecting the capillary tubes which are important to reduce the capillary absorption coefficient. At later times, nano silica specimens experienced a lack of enough water to complete the hydration process, so that a significant absorption reduction generally could not be observed at 28days. Micro silica specimens have a higher absorption coefficient compared with nano silica due to the greater number of particles that have less ability to react and disconnect the pores in proportion. In combined micro and nano silica specimens, acceptable results can be observed especially at 28days.

3.3. Electrical resistivity

The electrical resistivity was measured at 7, 28 and 90days. Results are shown in Fig. 4.





- As seen in Fig. 4, usage of nano and micro silica increases the electrical resistivity. An increase in the percentage of substitution of silica in cement leads to an increase in electrical resistivity. The best results are found in the 7.5M+5L100 mixture in which 61 kohm.cm resistivity is obtained at 90days. This result is 8.7 times greater than the control mixture.

As seen at 28days, the corrosion rate is promoted from high in the control to low in all specimens with 5% and more cement replacement by micro/nano-silica. At 90days, the corrosion rates of these specimens are generally classified in the negligible category. Filling the pores and making secondary silicate gel that condenses the cement paste and enhances the interfacial transition zone are the effective reasons for using micro/nanosilica in the cement paste. Electrical resistivity depends on microstructure and also penetrant liquid conductivity. Micro/nano-silica improves the cement matrix in these two aspects. mixture with 5% nano-silica replacement, 65% of 90-day age resistivity (\cong ultimate resistivity) is obtained at 7 days, while at this age, only 22% of ultimate resistivity is obtained for 5M mixture with 5% micro silica. According to the results obtained in nano silica specimens, most of the resistivity (more than 50%) is obtained before 7-days age while in micro silica specimens, the resistivity of 7 days is not as high as the resistivity gains in duration between 7 to 28days. The maximum rate of increase in resistivity (kohm.cm/day) in micro silica specimens, occurs between 7 to 28 and then 28 to 90days. In regard to nano silica specimens, it should be noted that a lower fineness (L100) performs better than a higher one (L300). In combined specimens, the behavior in resistivity is generally similar to micro silica. So that earning resistivity at 7 days is generally limited to 22%.

3.4. Rapid Chloride Migration Test (RCMT)

Rapid chloride migration results are represented in Fig. 5.

- Due to the high pozzolanic activity of nano-silica, most of the resistivity is obtained at earlier times, especially at 7 days. For instance, in $5\mathrm{L}300$





- As seen in Fig. 5, the higher impact of nano silica is in early gains of the strength of concrete and its effectiveness decreases with increased time. For instance, 57% reduction is observed in the migration coefficient of 5L100 specimen compared with the control at 28days. However, the rate of this reduction has diminished with time. This issue indicates that most of the pozzolanic activity of nano silica occurs at early ages. Micro silica specimens have less pozzolanic activity compared with nano silica, so a greater proportion of reduction at later times is found with micro silica. As seen in Fig. 5, although about 40% reduction at 28days is observed in 5M specimens, the performance of micro silica is better than the nano-silica at 90days.

- In combined specimens, the migration coefficient reduction is between 45% to 78% at 28days and 63% to 87% at 90days. By utilization of micro or nano-silica separately, achieving a migration coefficient reduction of



3.5. Shrinkage

After the preparation of shrinkage specimens, they were cured in saturated lime water for 48 hours. Afterward, the baseline reading for shrinkage was recorded. Samples were exposed to air for 25 days. Meanwhile, follow-up measurements were made at 4, 11, 18, and 25days. Finally, the amount of shrinkage is obtained by dividing the difference between a primary and secondary reading by the effective length of the gauge. Shrinkage results are shown in Fig. 6.





- As seen in Fig. 6 all the micro/nano-silica specimens had higher shrinkage compared with the control one. An increase in the substitution content of silica pozzolan increases the shrinkage too.

- Results show that micro silica has a significant effect on shrinkage in its early stages. Considerable change at 4 days confirms this issue. Of course, the slope of the shrinkage increment of micro silica specimens significantly decreases with time. This reduction also can be observed in combined micro/nano specimens. The maximum shrinkage is in the 7.5M+5L300 specimen which is 6.1 to 1.7 times more than the control at 4 to 25days respectively.

4. Discussions

For a more thorough investigation, the development of the experimental results compared with the control is shown in Table 8. As seen in Table 8, almost all the micro/nano-silica specimens perform better than the control in compressive strength, capillary absorption, resistivity, and RCMT tests at all ages. However, the weakness of the micro/nano-silica specimens is related to the shrinkage. Shrinkage of these pozzolanic specimens is greater than the control. According to the results, adding nano and micro silica increases the shrinkage at all the investigated times.

Table 8. Increment of the experimental results in comparison with the control.

| | Сотр | ressive str | ength | Capillary | absorption | tion Resistivity | | RC | мт | | Shr | inkage | | |
|-------------------|--------|-------------|------------|-----------|------------|------------------|------------|------------|---------|---------|-----------|------------|------------|---------|
| | 7 days | 28 days | 90 days | 7 days | 28 days | 7 days | 28 days | 90 days | 28 days | 90 days | 4 days | 11 days | 18 days | 25 days |
| 5L100 | 0.335 | 0.325 | 0.233 | -0.378 | -0.583 | 2.421 | 3.231 | 2.571 | -0.567 | -0.679 | 0.611 | 0.154 | 0.110 | 0.113 |
| 5L300 | 0.410 | 0.187 | 0.072 | -0.405 | -0.214 | 1.895 | 1.692 | 1.429 | -0.399 | -0.545 | 1.421 | 0.140 | 0.281 | 0.965 |
| 2.5L100 | 0.252 | 0.189 | 0.141 | -0.324 | -0.250 | 0.842 | 0.731 | 1.000 | -0.291 | -0.495 | 0.842 | 0.053 | 0.203 | -0.341 |
| 2.5L300 | 0.285 | 0.113 | 0.023 | -0.365 | -0.167 | 0.316 | 0.538 | 0.357 | -0.130 | -0.208 | 2.705 | 0.329 | 0.542 | 0.531 |
| 5M | 0.297 | 0.332 | 0.318 | -0.054 | -0.250 | 0.263 | 1.500 | 2.143 | -0.395 | -0.685 | 3.947 | 0.352 | 0.036 | 0.035 |
| 7.5M | 0.340 | 0.384 | 0.476 | -0.135 | -0.333 | 0.053 | 2.077 | 2.500 | -0.564 | -0.683 | 3.400 | 0.480 | 0.146 | 0.158 |
| 5M+ 2.5L100 | 0.483 | 0.479 | 0.371 | -0.162 | -0.250 | 0.447 | 2.462 | 2.750 | -0.584 | -0.728 | 3.695 | 0.510 | 0.170 | 0.192 |
| 5M+ 5L100 | 0.485 | 0.547 | 0.485 | -0.108 | -0.450 | 1.105 | 4.769 | 4.714 | -0.737 | -0.837 | 3.989 | 0.690 | 0.381 | 0.418 |
| 5M+ 2.5L300 | 0.586 | 0.424 | 0.341 | -0.135 | -0.254 | 0.079 | 1.692 | 2.321 | -0.453 | -0.632 | 4.421 | 0.908 | 0.546 | 0.618 |
| 5M+ 5L300 | 0.572 | 0.411 | 0.402 | -0.027 | -0.500 | 0.674 | 2.654 | 3.143 | -0.553 | -0.726 | 4.684 | 0.986 | 0.604 | 0.656 |
| 7.5M+ 2.5L300 | 0.569 | 0.470 | 0.453 | -0.081 | -0.333 | 0.447 | 2.462 | 3.286 | -0.605 | -0.806 | 4.263 | 0.685 | 0.317 | 0.436 |
| 7.5M+ 2.5 L100 | 0.313 | 0.429 | 0.396 | -0.203 | -0.411 | 0.842 | 2.846 | 3.286 | -0.646 | -0.791 | 4.411 | 0.575 | 0.237 | 0.299 |
| 7.5M+ 5L300 | 0.592 | 0.627 | 0.595 | -0.027 | -0.450 | 1.105 | 3.231 | 4.571 | -0.694 | -0.837 | 5.105 | 1.098 | 0.668 | 0.664 |
| 7.5M+ 5L100 | 0.507 | 0.652 | 0.733 | -0.081 | -0.500 | 1.368 | 5.154 | 7.714 | -0.783 | -0.872 | 4.158 | 0.747 | 0.429 | 0.467 |

In order to better simulate the experimental investigation to the site conditions, the selected mix design is made as a coating on substrate concrete (see Table 6). In general, regardless of shrinkage, 7.5M+5L100 specimen yields better results compared with other mix designs for mechanical and durability properties. In the following, compound specimens are evaluated based on shrinkage, RCMT, and capillary

absorption tests. It should be noted that RCMT and capillary absorption tests are carried out on cored samples.

4.1. Shrinkage

Two samples are made in $10 \times 50 \times 50$ cm³ dimensions with similar substrate concrete and different coating mortars. Shrinkage results of the control and selected mix design are shown in Fig. 7.



4.2. RCMT



Fig 8. Chloride ions migration coefficient of the selected mortar coating compared with the control in different curing conditions.

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4.3. Capillary absorption



Fig 9. Capillary absorption of the selected mortar coating compared with the control in different curing conditions.

As seen in Figs. 7 to 9, selected coating mortar which contains 7.5% micro silica and 5% nano silica (L100) performs better than the control one in reducing the chloride migration coefficient and capillary absorption results when implemented on substrate concrete.

As a comment, it should be noted that specimens cured in lime water have lower chloride migration coefficient and lower capillary absorption compared with air-cured specimens. For instance, migration coefficient of the control and pozzolanic specimens cured in lime water are 77% and 36% of those cured in air, respectively.

While pozzolanic specimen performs better than the control in RCMT and capillary absorption tests, shrinkage of this specimen is higher than the control one. However, the higher shrinkage is within the permissible range which does not have a destructive effect on durability.

5. Conclusions

In this study, different experiments were carried out to investigate the effect of micro and nano-silica on the mechanical and durability properties of cement mortars. The following results can be drawn:

1- Micro and nano-silica increase compressive strength. The main effect of nano-silica can be observed at early ages. The maximum compressive strength at 7 days is found for 7.5M+5L300 specimen. However, 7.5M+5L100 has maximum compressive strength at both 28 and 90 days.

2- For the durability experiments, a combination of micro and nano silica especially in 7.5M+5L100 has an excellent performance. The combination of micro silica and nano-silica allows the sample to perform better in the long term. So that the best results of RCMT and resistivity tests are yielded by the 7.5M+5L100 specimen. Capillary absorption results of these pozzolanic specimens are better than the control one. 7.5M+5L100 mixture has an acceptable performance in this respect, especially at 28days. Due to the high water demand of silica fine particles, shrinkage is the only test in which the results of the micro/nano specimens are weaker than the control one.

3- A verifiable approach to achieve better performance of specimens containing pozzolan is to grind them to appropriate fineness. In this investigation, nano silica is prepared in 2 fineness's: L100 and L300. Although the higher fineness of nano silica has relatively contributed to higher compressive strength at 7 days, this development will not last at later times. Because of the high reactivity caused by the higher fineness of particles, the microstructure of concrete will not generally improve over time. The better performance of nano-silica with a lower fineness (L100) in durability experiments, especially for resistivity and RCMT tests at all ages, confirms this.

4- According to the results for compressive strength, electrical resistivity, RCMT, capillary absorption, and shrinkage tests, 7.5M+5L100 is the best mix design. The selected mix design is used as coating mortar on the substrate concrete. The performance of compound micro and nano silica specimens in RCMT and absorption tests is again better than the control one. Although micro/nano-silica increases the shrinkage, the total shrinkage is not high enough to affect concrete durability significantly. Moreover, results show that moist curing has a significant effect on the durability promotion of coating mortars.

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Conflict of interest

The authors declare no conflict of interest.

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