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# Effect of Micro and Nano silica as supplementary cementitious materials on mechanical properties of cement mortar

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***Abstract*- In recent decades, the usage of different additives has been developed to enhance the properties of cement-based materials. Cement mortar is a basic cementitious material with a composite structure that, creates diverse properties by utilizing various admixtures. Due to this fact, the present study aim is to evaluate the effect of micro and nano-silica on the mechanical properties of cement mortar. For this purpose, 16 mix designs were considered with various replacement percentages of nano silica (0, 1.4, 2.8, and 4.2%) and micro silica (0, 4, 9 and 13%) in forms of alone and together at the age of 3, 7 and 28 days. So, a comparative probe was carried out to evaluate the effect of different percentages of micro and nano-silica on the mechanical properties of cement mortar. In this regard, different macroscopic (porosity, compressive and flexural strength) tests were conducted on compressive and flexural specimens. The results show that the simultaneous addition of micro and nano-silica has a considerable effect on improving the cement mortar properties by decreasing the porosity and following that enhancing the strength properties of cement mortar in comparison with adding micro or nano-silica alone.**

***Keywords* –**Cement mortar, Micro silica, Nano silica, Mechanical properties

## 1. INTRODUCTION

Cement mortar as a cement-based material is a non-homogeneous material with complex behaviors, which various proportions of its constituents make different characteristics. Hence, the physical and mechanical properties are basically don't depend on only one parameter but, under the influence of relationships between various parameters such as, water to cement ratio (W/C) [1], sand to cement ratio(S/C) [2], curing age [3], and admixture [4] of specimens. These parameters have been examined in some previous literature. For instance, the previous studies by Haach et al. [1] have been revealed that an increasing W/C ration would lead to a decrease in the mechanical strength of cement-based materials. Singh and Dhillon [5] investigated the compressive strengths of high strength concrete containing Ns and Ms, the results showed that the best compressive strength refers to the combined addition of Ms and Ns. Moreover, the effect of an increase in the specimens' curing ages was examined in the latest research of De Vargas et al. [6], so their results showed that an increase in the age of specimens improved the compressive and flexural strength of cementitious materials.

During the last decade, with the development of concrete technology, the utilization of alternative cementitious materials have become widespread to improve the physical and mechanical properties of cementitious materials. [2, 7, 8]. These nanomaterials have attracted notable research interest. The supplementary cementitious materials dramatically are used to decrease the consumption of cement and enhance the mechanical properties of concrete or mortar owing to their pozzolanic reactivity [9, 10]. Among these materials that are finer than cement, the Ms has been recognized as the most effective supplementary cementitious materials, not only because of their large surface area which exhibits great pozzolanic activity, but also because of their fine size which lead to an increase in the packing density with fill into the voids between cementitious materials [11, 12]. Recently, the advent of nanotechnology has brought in the various types of nano-materials (mainly silica, titanium, aluminum, and iron oxides) for possible uses in cement-based materials, especially concrete production, owing to their four major effects: size, surface, quantum -sized pores, and interface effect [13-16]. It is worth noting that, the Ns would react with the calcium hydroxide $Ca(OH)_2$ or (CH) crystals between the aggregates and hardened cement paste in the interfacial transition zone (ITZ) and leads to an increase in produced calcium-silicate-hydrate (C-S-H ) gel at this critical bonding zone [17, 18]. In this regard, owing to the well-known relation between porosity and mechanical properties of cement mortar, porosity evaluation and the role of it, is a priority in evaluating the properties of cementitious materials.

As previously mentioned, the potential of utilizing Ms and Ns to improve the mechanical properties of cement mortar is remarkable and requires further examination, particularly in ternary mixtures with the presence of Ms, Ns, and cement. The absence of available experimental data in the performance of concrete or mortar containing Ms, Ns, and combined of Ms+Ns is a key role that justifies this research. The aim of the present study is evaluating the effect of different percentages of micro and nano-silica as the supplementary cementitious materials on porosity values and following that, on the compressive and flexural strength of cement mortar.

## 2. Experimental Program

### *2.1 Materials*

In this study, an ordinary Portland cement (OPC-type I 52.5R) and supplementary cementitious materials such as micro and nano-silica in different percentages of 0%, 4%, 9%, 13% and 0%, 1.4%, 2.8%, and 4.2%, were utilized respectively. The physicochemical properties of cement, Ms and Ns are reported in Table 1.

High range water reducing (HRWR) is a high-performance superplasticizer based on the carboxylic ether polymer which induces increased early and ultimate strengths as well as improve workability [19]. The maximum size distribution of sand aggregate equal to 4.75 mm and conducted in accordance with the ASTM C778 [20] limit, represented in Fig. 1.

**Table 1.** Chemical composition and physical properties of cementitious materials.

| cementations materials | Chemical Analysis (wt %) | | | | | | | | | | | | Physical Analysis | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | LOI | F.CaO | $C_3A$ | $C_3S$ | Specific Gravity (ton/m$^3$) | Blaine Test (cm$^2$/gr) |
| C 52.5 | 21 | 4.7 | 3.52 | 64.18 | 1.93 | 2.53 | 0.32 | 0.65 | 1.2 | 1.2 | 6.5 | 57.85 | 3.15 | 3600 |
| Ns | 23.6 | 0.13 | 0.07 | 0.07 | 0.05 | 0.1 | 9.3 | 0.12 | - | - | - | - | 1.2 | - |
| Ms | 89.6 | - | - | - | - | - | 0.11 | 0.3 | 3.8 | - | - | - | 1.9 | 220000 |

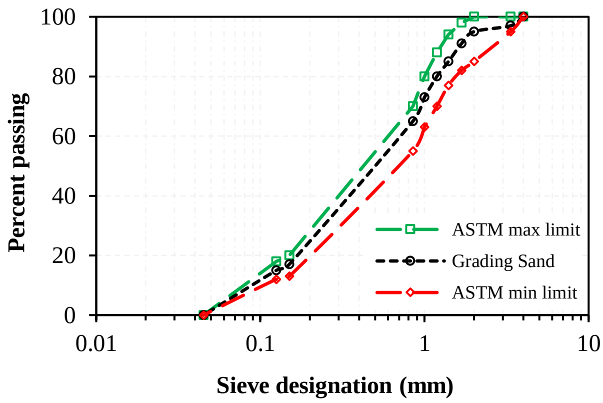


**Fig 1.** Grading curve of sand aggregate.

### *2.2 Mixture proportions*

In the present study, 16 different mix design group with fixed water to binder ratio of 0.50 including 1 control mix, 3 Ms-containing mixes, 3 Ns-containing mixes, and 9 Ms and Ns-containing mixes were reported. The details of all 16 cement mortar mix proportions are given in Table 2. Additionally, the various percentages of Ms and Ns as supplementary cementitious materials were utilized in the cement mixtures to achieving the optimal range of Ms and Ns percentages in accordance with previous findings [7, 21]. For each mix design, the amount of HRWR with a solid content of $47 \pm 2\%$ was different in mortar mixture that is determined during the mixing by incremental adding to achieve at least a flow of $110 \pm 5\%$ in 25 drops of the flow table. The size of the cubic compressive and prismatic flexural specimens which has been used for compressive and flexural strength tests were $50\times50\times50$ mm$^3$ and $40\times40\times160$ mm$^3$, respectively. The cubic and prismatic specimens were cast from a homogeneous fresh cement mortar which mixed with a planetary mixer in accordance with ASTM C305 [22]. The cubic and prismatic specimens were cured 24h in the mold with a controlled temperature of $22\pm2$ °C and RH> 95%, then immersed in the water tank at $25\pm1$ °C till the time of testing. Note that the cubic specimens were tested after 3, 7, and 28 days curing, and the prismatic specimens were tested after 28 days curing.

**Table 2.** Mix properties of all cement mortar mix.

| Mix NO. | C (gr) | W/C | S/C | Ns/B | Ms/B | HRWR (mL) |
|---|---|---|---|---|---|---|
| 1 | 1200 | 0.500 | 2.667 | 0 | 0 | 4.5 |
| 2 | 1152 | 0.521 | 2.778 | 0 | 4 | 9 |
| 3 | 1092 | 0.549 | 2.930 | 0 | 9 | 12 |
| 4 | 1044 | 0.575 | 3.065 | 0 | 13 | 15 |
| 5 | 1183.2 | 0.507 | 2.705 | 1.4 | 0 | 6.5 |
| 6 | 1166.4 | 0.514 | 2.743 | 2.8 | 0 | 8 |
| 7 | 1149.3 | 0.522 | 2.784 | 4.2 | 0 | 9.5 |
| 8 | 1135.2 | 0.529 | 2.819 | 1.4 | 4 | 10 |
| 9 | 1118.4 | 0.536 | 2.861 | 2.8 | 4 | 11.5 |
| 10 | 1101.3 | 0.545 | 2.906 | 4.2 | 4 | 12.5 |
| 11 | 1075.2 | 0.558 | 2.976 | 1.4 | 9 | 13 |
| 12 | 1058.4 | 0.567 | 3.023 | 2.8 | 9 | 14 |
| 13 | 1041.3 | 0.576 | 3.073 | 4.2 | 9 | 15.5 |
| 14 | 1027.2 | 0.584 | 3.115 | 1.4 | 13 | 16.5 |
| 15 | 1010.4 | 0.594 | 3.167 | 2.8 | 13 | 17.5 |
| 16 | 993.3 | 0.604 | 3.222 | 4.2 | 13 | 20 |

*C = Cement, W/C = Water to Cement ratio, S/C = Sand to Cement ratio, Ns/B = Nano silica to Binder ratio, Ms/B = Micro silica to Binder ratio, and HRWR= High range water reducer.

*2.3 Test procedure*

To minimize the testing error, the reported cubic and prismatic strength and porosity values were the averages of three specimens obtained during the test procedures.

*2.3.1 Porosity test*

To this aim, the porosity values of hardened specimens were calculated at different ages (3, 7, and 28). At first, the whole of specimens was entirely dried in the oven at $105\pm5$ °C to obtain a stable weight ($W_d$). Then, the weight of specimens was gathered at the different conditions of underwater ($W_w$) and, and the saturated surface dry weight ($W_{ssd}$). Finally, the percentage of the specimens' porosity (P) was determined according to the following formula:

$$P=\frac{(W_{ssd}-W_d)}{(W_{ssd}-W_w)}\times100\% \tag{1}$$

*2.3.2 Compressive strength test*

The compression strength test was applied to the hardened cubic specimens according to ASTM C109 [23] by the hydraulic testing machine. The compressive strength ($F_c$) of cement mortar specimens can be determined in MegaPascals (MPa) by using the following formula:

$$F_c=\frac{F}{l\times b} \tag{1}$$

Where,

$F$= Maximum compression load in Newton.
$l$ and $b$ = length and width of specimens in millimeter (50 mm for both of them)

*2.3.3 Flexural strength test*

The flexural strength test of prismatic cement mortar specimens was determined with the three-point testing setup in accordance with ASTM C 348 [24] limits. The testing was conducted with a Universal Testing Machine (UTM) with a maximum load capacity of 5 kN at 0.3 kN/min constant rate, as shown in Fig. 2. The flexural strength ($F_f$) of cement mortar specimens can be determined in MegaPascals (MPa) by using the following formula:

$$F_f = \frac{3}{2} \times \frac{F \times l}{b \times h^2} \tag{2}$$

Where,
$F$= Maximum load in Newton.
$l$= Distance between the supports in millimeter (120 mm).
b= Width of the specimen in millimeter.
h= Height of the specimen in millimeter.

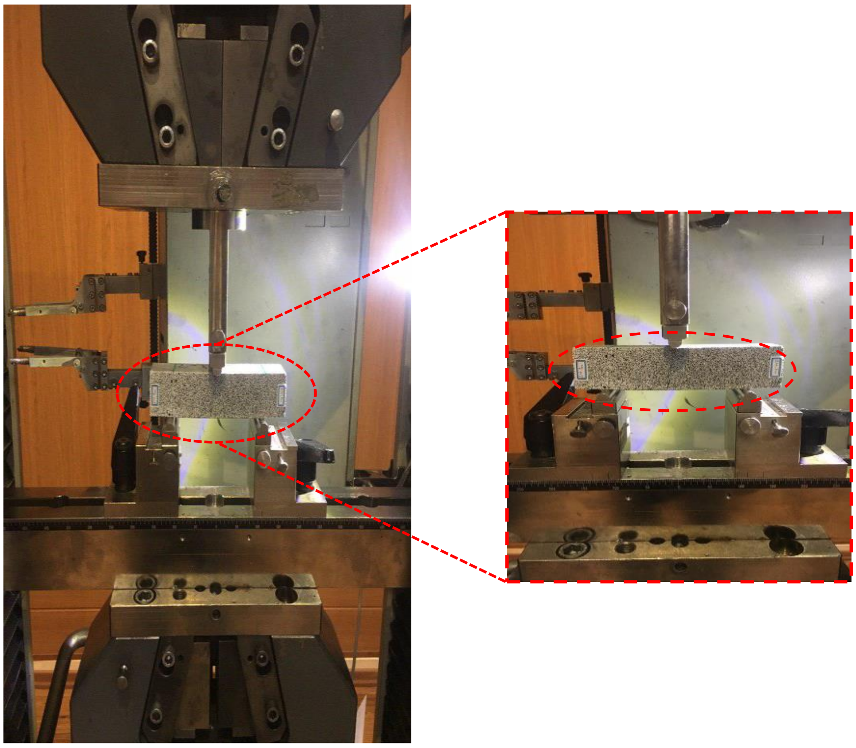

**Fig 2.** Experimental setup of flexural prismatic specimens.

## 3. Results

*3.1 Porosity*

The variations of cement mortar specimens' porosity at different ages of 3, 7, and 28 days versus respective percentages of Ms and Ns for 16 mixtures are represented in Fig. 3. The variation of porosity ranges' is limited to 7-20%. As shown in Fig. 3, the 3-days porosity of the control mortar (0% Ms + 0% Ns) has the highest porosity among all mixtures, and it altered from 15 to 20% by gradually increasing in Ns values up to 4.2%. The simultaneous presence of Ms and Ns in mortar mixtures leads to a reduction in porosity values compared to the mixtures containing only Ns values. For example, the porosity values of cement mortar mixture contain 4% Ms+ 1.4% Ns and 9% Ms+ 1.4% Ns compared to mixture contains 1.4% Ns were reduced from 18.5 to 16 (8.1% porosity reduction) and 18.5 to 15.5 (16.21% porosity reduction) respectively. It can be indicated in Fig. 3a that the lowest porosity among all 16 mix design is13.5% which has appeared in mixtures containing 9% Ms+ 2.8% Ns. The lowest 3-days porosity value is 13.5%, which is obtained in a mixture with 9% Ms+ 2.8% Ns, as shown in Fig 3a. Moreover, it can be found in Fig. 3a-c that increasing the age of specimens from 3 to 28 days, the porosity values are reduced, owing to the completion of the hydration process with the remain specimens' water throughout the curing time of specimens. Therefore, the spaces belonging to the water and pores have been filled with more hydration products such as calcium silicate hydrate (C-S-H), and, finally leads to a reduction in porosity value and appearing the dense texture. In fact, as confirmed by previous studies, the increasing age of specimens containing Ms and Ns lead to form a denser structure, owing to the better hydration activity in the microstructure of cement-based materials. [3, 25]. For example, the 3, 7 and 28-days porosity of the mixtures contain 9% Ms + 1.4% Ns and 13% Ms + 1.4% Ns are (15.5%, 13%, and 7.5%) and (16%, 13.5%, and 8.5%) respectively. The lowest porosity value among all mixtures is 7% which appeared in the mixtures with 9% Ms + 2.8% at the age of 28 days. It worth noting that, the current result is in the agreement with the previous studies [26-28].

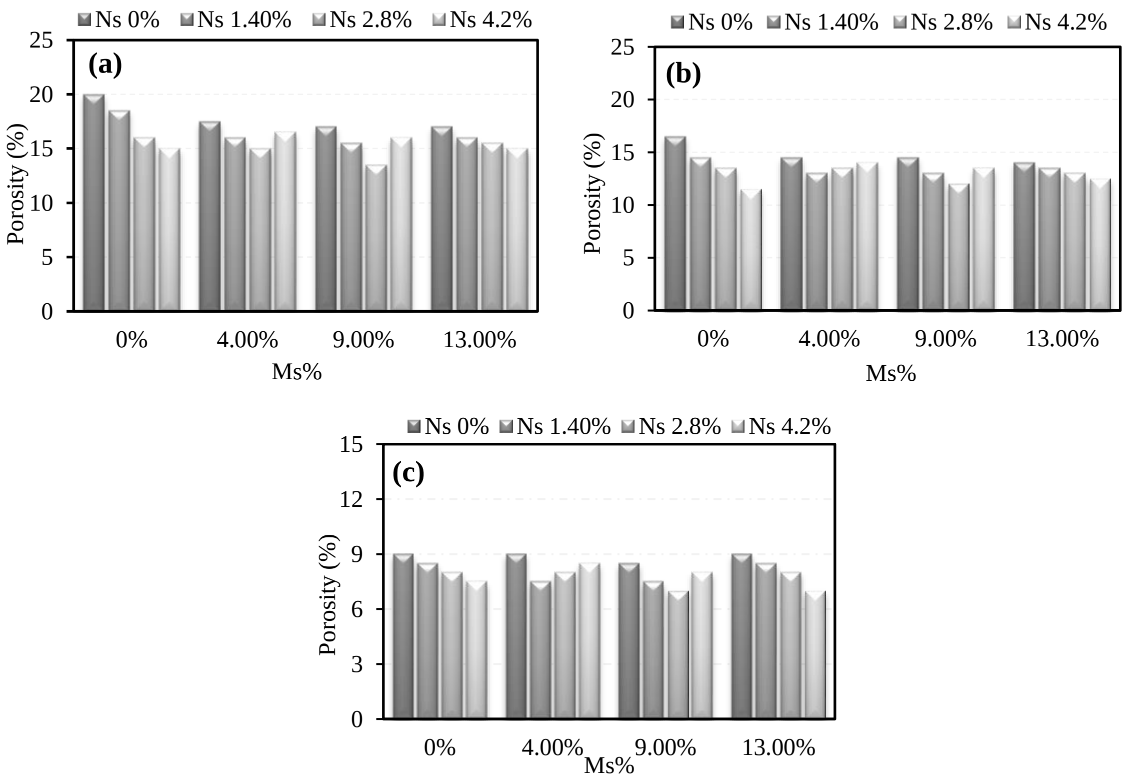


**Fig 3.** Comparison of porosity versus Ms and Ns percentages at different ages of: (a) 3, (b) 7, and (c) 28 days.

*3.1 Compressive strength*

The relevance of compressive strength ($F_c$) of cement mortar hardened specimens versus the different percentages of Ms (0%, 4%, 9%, and 13%) and Ns (0%, 1.42%, 2.8%, and 4.2%), considering the age of specimens (3, 7, and 28 days) are depicted in Fig. 4. As shown in Fig. 4, the lowest compressive strength content emerged in control mortar (0% Ms + 0% Ns), owing to the absence of pozzolanic materials that leads to the maximum porosity values. The variation of $F_c$ content in control mortar specimens at the ages of 3 to 28 days is from 18 MPa to 50 MPa. It is obvious that the improvement in the compressive strength of mortar specimens affected by some factors, such as decreasing the porosity, increasing the age of specimens curing ,and eventually the completion of the hydration process. In this regard, the addition of Ns values alone up to 4.2% leads to increases in the $F_c$ of cement mortar hardened specimens, due to fills the pores and reduces the porosity values. For instance, the compressive strength of specimens while the Ns was altered from 0% to 4.2% were increased from 27 to 33 and in a similar trend, the porosity values of the respective mixtures also reduced from 18.5% to 15%. It is observed from Fig. 4a–c that the combined increase in Ms and Ns percentages have a higher effect on each early age (3 and 7 days) specimens. For instance, the

3, 7, and 28-days compressive strength of the mortar mixture contains 9% Ms + 2.8% Ns compared to respective the control mortar (0% Ms + 0% Ns) were growing up about 144%, 107%, and 54% respectively, while in the similar trend the porosity values were reduced about 32.5%, 27%, and 22% respectively. In this regard, Jalal et al. [29] studied the influence of pozzolanic material include Ms and Ns on cement-based materials. Their result show that the combined usage of Ms and Ns had better influences on cementitious materials. Moreover, the current studies' results reveal that the optimum values of $F_c$ appear in the mixture containing 9% Ms + 2.8% Ns, and the further values would reduce the $F_c$ content. The reason for this fact might be owing to the high fineness of the pozzolanic particles and, also their large surface area could separate particles non-uniformly and whereby agglomerate the particles, which is in agreement with the latest finding of Haruehansapong et al. [12] and Mohamed [30].

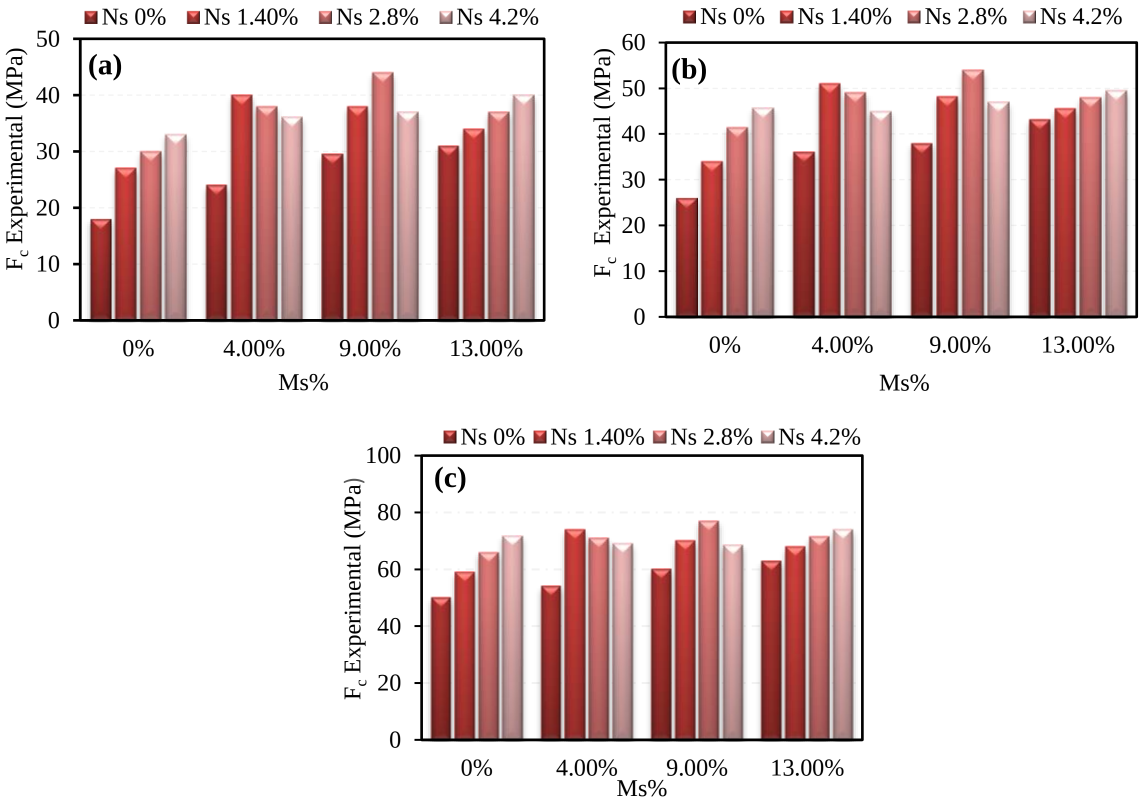


**Fig 4.** Comparison of compressive strength versus Ms and Ns percentages at different ages of: (a) 3, (b) 7, and (c) 28 days.

*3.1 Flexural strength*

Fig. 5 illustrate the relevance of the flexural strength ($F_f$) of cement mortar hardened specimens at different ages (3, 7, and 28-days) versus different percentages of Ms (0%, 4%, 9%, and 13%) and Ns (0%, 1.4%, 2.8%, and 4.2%). The absence of Ms and Ns values leads to appear the lowest flexural strength content in control mortar (0% Ms + 0% Ns), as shown in Fig. 5. The flexural strength content of cement mortar specimens with only the addition of Ns values up to 4.2% is increased, owing to the reduction in porosity values. The combined usage of Ms and Ns values in mortar mixture leads to significant improvement in flexural strength content compared to mixtures contain only Ns values. For instance, the flexural strength content of the cement mortar mixtures contains 1.4% Ns, 4 Ms+1.4% Ns, and 9% Ms+ 1.4% Ns were 8.9 MPa, 11.1 MPa, and 10.5 MPa respectively, and also in similar trend the flexural strength content for the mortar mixtures with 2.8% Ns, 4 Ms+2.8% Ns, and 9% Ms+ 2.8% Ns were 9.9 MPa, 10.7 MPa, and 11.6 MPa respectively. It worth noting that, the highest flexural strength content was 11.6 MPa which was appeared in the cement mortar mixture contains 9% Ms + 2.8% Ns. It is generally obtained from Fig.5 that the combined usage of Ms and Ns values led to a reduction in porosity values, and has improved the ultimate flexural strength of cement mortar hardened specimens, which is in agreement with the previous studies [3].

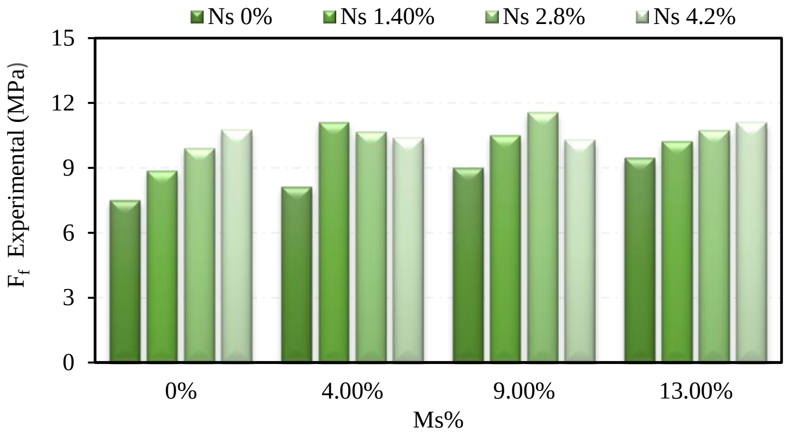


**Fig 5.** Comparison of flexural strength versus Ms and Ns percentages at the age of 28 days.

## 4. Conclusion

In this research, the macro-structure properties of cement mortar containing micro and nano-silica were investigated. We particularly concentrate on the porosity parameter as the influential parameter which affected the other cement mortar mechanical properties. The concluding remarks can be drawn as follow:

1. The addition of Ms or Ns contents in cement mortars would decrease the porosity values through an increase in the compressive and flexural strength of cement mortar, while the porosity reduction effect is higher at the age of 28 days specimens, especially when Ns and Ms are combined together. The combined addition of the Ms and Ns has a more remarkable influence on the porosity, compressive strength, and flexural strength.
2. The optimum dose of cementitious replacement materials (Ms and Ns values) with cement is 2.8% and 9% by weight of cement, respectively.
3. The effectiveness of the combined presence of Ns and Ms on mechanical properties of cement mortar comparing separately presence of Ms or Ns is higher. Such higher effectiveness in the presence of the other form of silica implies that there is certain synergy between the Ms and Ns.
4. The addition of Ms and Ns would significantly increase the 3, 7, and 28-day cube, and 28-days prismatic strengths. Comparatively, the presence of Ns values in cement mortar mixture had more efficient influence in increasing the compressive and flexural strength than the presence of Ms Values. Fundamentally, the presence of only 1% Ns would provide almost the same improvements in cubic and prismatic strengths as the addition of 10% Ms.
5. Consequently, the porosity value has an important undeniable, and effective factor in determination cement mortar mechanical properties, so, the effect of porosity as an influential parameter should be considered the experimental investigations.

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