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Study The Effect of Micro $CaCO_3$ and SiO_2 and their Mixture on Properties of High Strength Concrete

Abstract- *This paper investigated the effect of incorporating two types of micro particles micro $CaCO_3$ and micro SiO_2 on mechanical properties and durability of concrete. Micro materials were added in four different dosages of 1%, 2%, 3% and 4% by weight as partial replacement of cement in concrete mixture. Mechanical properties of hardened concrete (compressive strength, flexural strength and split tensile strength) have been done after 28 days of water curing. In addition, water absorption test was carrying out for obtaining the durability properties of concrete specimen. Binary combination of micro $CaCO_3$ + micro SiO_2 were also studied the combined effect of the micro particles. Micro-structural characteristic of modified concrete was done through the scanning electron microscope. The results showed that incorporation of micro $CaCO_3$ and micro SiO_2 particles lead to increase the packing and enhance the mechanical properties and durability of concrete. A significant performance was observed in case of micro silica addition to the concrete in comparing with other micro particles.*

Keywords- *High strength concrete- Durability of concrete- Micro silica- Micro $CaCO_3$- Mechanical properties of concrete.*

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1. Introduction

Concrete is a newer construction material compared to steel and stone. The usage of concrete in constructions have begun less than a century ago. In recent century, wide and effective research has seen on improving the mechanical properties and durability of concrete by incorporating wide range of cementing materials such as pozzolans and nanoparticles due to increasing the use of concrete permanently [1]. High Strength Concrete is a comparatively recent development in concrete technology made by introduction of efficient water-reducing admixtures and high strength cementitious materials simple definition would be ‘concrete with a compressive strength greater than that covered by current codes and standards’ [2]. Generally, Micro-silica is used for most of high performance concrete around the world. During the manufacturing process of Ferro-silicon and silicon metal in electric arc furnace, Micro-silica is obtained as by-product contains 85 to 98 percent silicon dioxide [3]. Micro silica can classify as one of highly pozzolanic materials because of it consists essentially of silica in non-crystalline form with a high specific surface, and thus develops a great pozzolanic activity [4]. Calcium carbonate ($CaCO_3$), however, has shown an ability to accelerate the hydration process of Portland cement in addition to its filling effect. $CaCO_3$ usually has no chlorides and can readily be found, for instance, in chalk and marble, or produced artificially by combining calcium and carbon dioxide [5]. Verma Ajay et al. found that usage of micro silica with concrete enhance the compressive strength and reducing in the environment pollution also decrease the porosity of concrete, reduces capillary and absorption because fine particles of micro silica reacts with lime present in cement [6]. Baid et al. were found that using up to 12% replacement of cement with micro silica an increasing in the compressive strength of concrete with increasing dose of micro silica would be recognized and then decreased slightly, while the absorption and workability decreased [7]. Camiletti et al. shown that $CaCO_3$ accelerates the hydration process by acting as a nucleation site on which cement hydration product form. This microphysical influence leads to higher improvement rate of mechanical properties; the gain in strength can be due to the formation of calcium silicate hydrate CSH gel, which is stronger than the normal calcium hydrate CH gel [5]. Mohammed et al. studied the durability of micro silica and nano silica contained concrete and found that water absorption test, for mixes incorporated micro and nano silica, except for %5

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micro silica mix, showed lower absorption than control mixes [8].

2. Experimental Work

I. Materials

Commercially available ordinary Portland cement Type I, named Karasta, is used in this study. Chemical and physical properties of cement, which are indicated that the cement is conformed to Iraqi specifications (I.Q.S.) No. 5/1984 [9], are shown in Table 1 and Table 2 respectively. Natural sand (from Al-Akhedher in Karbala) was used. Table 3 shows the grading of the fine aggregate and their sulfate content according to the Iraqi Specification No. 45 /1984 [10]. To obtained high strength concrete crushed gravel used throughout this work of maximum size (14) mm. Table 4 shows the grading of the coarse aggregate and sulfate content according to the Iraqi Specification No. 45 /1984[10]. Micro silica purchased from Fluka Company, which is used as pozzolanic admixture, Figure 1, showed the particle size analysis and Figure 2 give the XRD spectra. Micro $CaCO_3$ purchased from Himedia Company is used as admixture; Figure 3 indicated the particle size analysis while Figure 4 showed the XRD spectra. Glenium 54 (G54) high range water reducing admixture, purchased from BASF Company, was used as workability adjusting material for concrete mixtures.

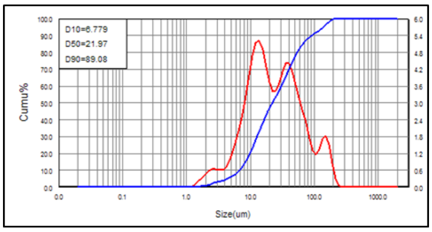


Figure1: Particle size analysis of micro silica

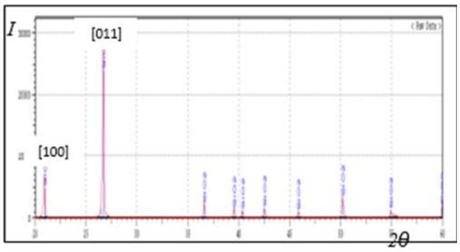


Figure: 2 XRD spectra of micro silica

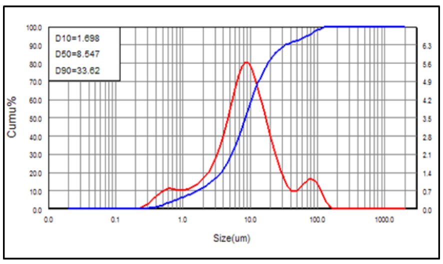


Figure3: Particle size analysis of micro $CaCO_3$.

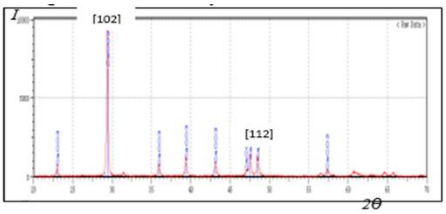


Figure 4: XRD spectra of micro $CaCO_3$.

Table 1: Chemical analysis of the cement test

Oxide	%
CaO	66.11
SiO_2	21.93
Al_2O_3	4.98
Fe_2O_3	3.10
MgO	2.0
K_2O	0.75
Na_2O	0.35
SO_3	2.25
Loss On Ignition (L.O.I)	2.39
Lime Saturation Factor (L.S.F)	0.93
Insoluble residue (I.R)	1.29
Free lime (F.L)	0.67
Compound Composition	**%**
C_3S	50
C_2S	20.48
C_3A	4.0
C_4AF	13.17

Table 2: Physical Properties of Cement test

Physical Properties	Test Results
Fineness , Blaine , cm^2/gm	3300
Setting Time :	
Initial hrs. ; min	2;05
Final hrs. ; min	4;00
Compressive Strength MPa	
3-days	20,0
7-days	25,0

Table 3: Sieve analysis and sulfate content of fine aggregate.

Sieve opening (mm)	Accumulative passing, %
10	100
4.75	94
2.36	85.6
1.18	76.9
0.60	46.3
0.3	10.8
0.15	1.1
0.075	0.5
Property	Result
SO_3, %	0.4

Table 4: Sieve analysis and sulfate content of gravel

Sieve opening (mm)	Accumulative passing, %
14	97
10	62
5	10
0.075	0.037
Property	Result
SO_3, %	0.09

II. Mix Proportion

Target design strength of 50, 70 MPa was designed according to British mix design method BS5328. Part 2:1991[11], thirteen types of concrete mixes are implemented in this study. The fixed parameters for all mixes are: water/cementitious, coarse and fine aggregate fractions, and superplasticizer contents. Mixes details and symbols can be seen in Table 5.

III. Specimens

The types and dimensions of specimens that used were cubic with 150x150x150mm for compressive strength test according to BS 1881-Part 101 [12], split tensile strength specimens for concrete were cylinder (100x200)mm according to ASTM C31/C31M [12], flexural strength test specimens were prism (100x100x400)mm according to ASTM C31/C31M [13]. In addition, cylindrical 100x50 mm for absorption test according to ASTM C 642 [14] as shown in Figure 5.

Table 5: Mixes symbols, content and quantity

Mix symbol	Cement, kg /m^3	Sand, kg /m^3	Gravel, kg /m^3	w/b%	G54 kg /m^3	Micro silica kg /m^3 (rep.%)	Micro CaCO3 kg/m^3 (rep.%)
Control	515	721	1030	0.32	6.43	---	---
1MS	509.85	721	1030	0.32	6.43	5.15 (1%)	---
2MS	504.7	721	1030	0.32	6.43	10.3 (2%)	
3MS	499.55	721	1030	0.32	6.43	15.45 (3%)	
4MS	494.4	721	1030	0.32	6.43	20.6 (4%)	
1MC	509.85	721	1030	0.32	6.43		5.15(1%)
2MC	504.7	721	1030	0.32	6.43		10.3 (2%)
3MC	499.55	721	1030	0.32	6.43		15.45 (3%)
4MC	494.4	721	1030	0.32	6.43		20.6 (4%)
1MS+MC	509.85	721	1030	0.32	6.43	2.575(0.5%)	2.575(0.5%)
2MS+MC	504.7	721	1030	0.32	6.43	5.15 (1%)	5.15(1%)
3MS+MC	499.55	721	1030	0.32	6.43	7.725(1.5%)	7.725(1.5%)
4MS+MC	494.4	721	1030	0.32	6.43	10.3(2%)	10.3(2%)

(a) (b) (c) (d)

Figure 5: Specimens of test
(a)Specimens of compression test. (b) Specimens of split tensile strength test.
(c) Specimens of flexural strength test. (d) Specimens of water absorption test.

3. Tests

I. Compressive Strength Test

This test was carried out according to BS 1881-Part 116 **[15]**. The curing age was 28 days. Three cubes were made for each mix at the specified age.

II. Split Tensile Strength Test

This test done according to ASTM C 496/C 496M – 04[16]. The curing age was 28 days. Three cylinders were made for each mix at the specified age. The value of splitting tensile strength of the specimen was calculated as follows:

$\sigma = 2F/\pi DL$ (1) [16]

where:
σ= splitting tensile strength, [MPa],
F = maximum app.lied load. [N],
L = length, [mm],
and D = diameter, [mm].

III. Flexural Strength

This test is carried out according to ASTM C293-02 [17]. Duplicate beam specimens were tested and the average results were considered. The flexural strength determined by using equation (2) [17].

$$\sigma = 3\,Fl/2bd^2 \quad (2)$$

Where
σ = flexural strength, (MPa).
F = maximum app.lied load, (N).
l = span length, (mm).
d = depth of the specimens, (mm).
b = width of the specimens, (mm).

IV. Water absorption test

The water absorption was conducted according to ASTM C642 [14], the water absorption test is carried out using (50x100mm) cylinder specimens, and the average water absorption of two samples were recorded and considered. Water absorption determine by using equation (3) [14].

$$\text{Absorption after immersion, } \% = \frac{Ws-Wd}{Wd}x100 \quad (3)$$

Where:
Wd = mass of oven dried sample in oven (gm).
Ws = mass of saturated surface-dry sample in air after immersion (gm).

4. Results

I. Results of Compressive Strength Tests

Compressive strength test was performed after 28 days of curing. The results shown in Figure 6 were the average of three specimens, for concrete mixes with micro silica the results showed increasing in compressive strength with the content of micro silica because micro silica enhance the properties of concrete through two mechanisms, first: pozzolonic effect when water is added to cement hydration occurs forming two products, CSH(Calcium Silicate Hydroxide gel) and CH (Calcium Hydroxide), in the presence of micro silica, SiO_2 will react with the calcium hydroxide to produce more aggregate binding CSH gel , second: micro filler effect of micro silica reduces permeability and improves paste to aggregate bond in concrete compared to conventional concrete [6]. Concrete mixes with micro $CaCO_3$ did not show the improvement in compressive strength up to 3%, but at 4% of $CaCO_3$ showed increasing about 7.5% in compressive strength**.** Studies have shown that CaCO3 accelerates the hydration process by acting as a nucleation site on which cement hydration products form, this microphysical effect results in a higher enhancement rate of mechanical properties. A higher accelerating effect occurs when more CaCO3 is added [5,18]. Binary concrete mixes showed moderate enhancement in compressive strength about 9.4% increment for concrete mix with 4% of (micro silica+ micro $CaCO_3$) due to the dual effect of micro silica and micro $CaCO_3$ on the hydration process which reflected on the properties of concrete. Table 6 indicated the values of compressive strength for all concrete mixes in this work.

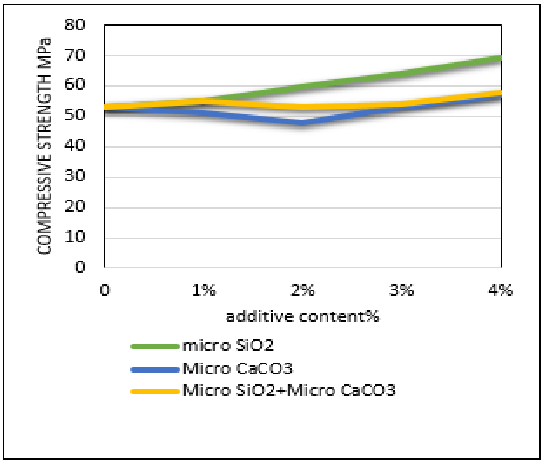


Figure 6: The results of compressive strength.

Table 6: Compressive Strength Results.

Concrete mixes type	Compressive strength (MPa)				
	0	1%	2%	3%	4%
Micro silica	53	55	59.6	63.9	69.3
Micro $CaCO_3$	53	51.2	48	53	57
Micro silica+ micro $CaCO_3$	53	55	53	54	58

II. Results of Split Tensile Strength Test

Figure 7 shows the indirect tensile strength results. Similar behavior was recognized in splitting tensile strength as in case of compressive strength results. About 10%, 17.5%, 21.25% and 33.75% enhancement in splitting tensile strength founded in specimen contain 1, 2, 3 and 4 % of micro silica, respectively, while for mixes with 1, 2, 3 and 4% micro $CaCO_3$ the strength enhancements were 8.75%, 12, 87%, 7.5% and 16.2 % respectively. Binary systems showed the also the same trend, about 0%, 22.5%, 23.75% and 30% strength improvement were achieved using 1, 2, 3 and 4% of (micro silica + micro $CaCO_3$). This can be attributed to additional CSH gel formation due to the effect of fine particles presence in concrete, which lead to denser packing of material at their interfacial transition zone [19]. Table 7 indicated the values of splitting tensile strength for all concrete mixes in this study.

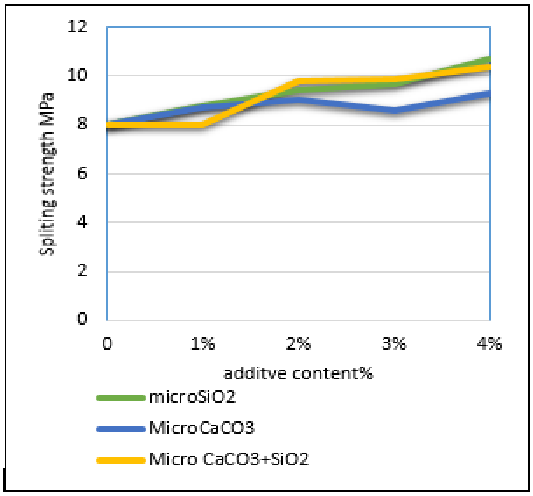


Figure 7: The results of splitting tensile strength.

Table 7: Split Tensile Strength Results.

Concrete mixes type	Splitting tensile strength (MPa)				
	0	1%	2%	3%	4%
Micro silica	8	8.8	9.4	9.7	10.7
Micro $CaCO_3$	8	8.7	9.1	8.6	9.3
Microsilica+ Micro $CaCO_3$	8	8	9.8	9.9	10.4

III. Results of Flexural Strength Test

Figure 8 showed the results of flexural strength after 28 days of curing, similar trend in the test result values were observed in case of compressive strength values and splitting tensile strength. About 25.4%, 30.9%, 32.7 and 45.45% for mixes with 1,2,3 and 4% micro silica, while for mixes with 1,2,3 and 4% micro $CaCO_3$ the increasing in strength were 10%, 12.36%, 13.6% and 9% respectively, finally the increasing in strength for 1, 2, 3 and 4% of binary mixes were 4.5%, 7.27%, 16.36% and 21.8% respectively, the same explications for results of compressive and splitting tensile strength can be app.lied here [20,7]. Table 8 indicated the values of flexural strength for all concrete mixes in this study.

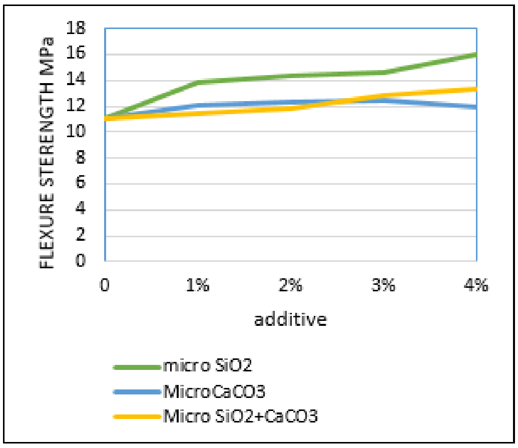


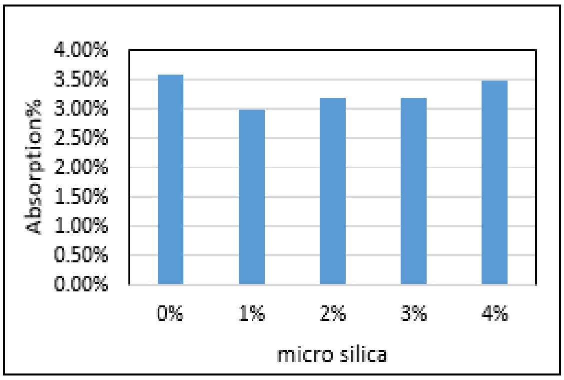
Figure 8: The results of flexural strength

Table 8: The results of flexural strength test.

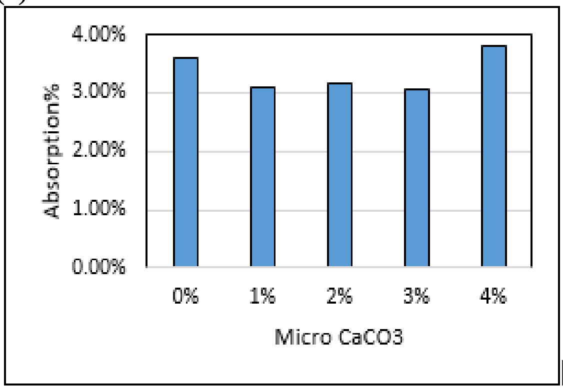
Concrete mixes type	Flexural strength (MPa)				
	0	1%	2%	3%	4%
Micro silica	11	13.8	14.4	14.6	16
Micro $CaCO_3$	11	12.1	12.4	12.5	12
Micro silica+ Micro $CaCO_3$	11	11.5	11.8	12.8	13.4

IV. Results of Water Absorption Test

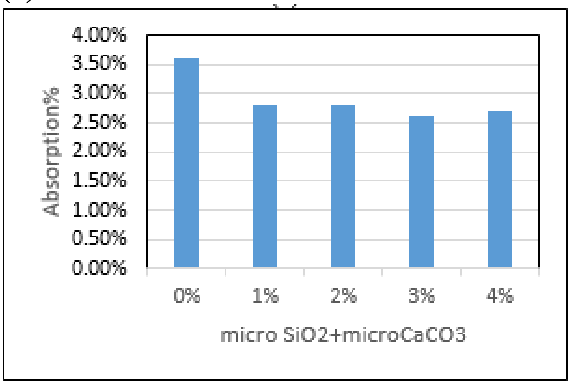
All mixes improved the durability of concrete through the reduction of water absorption. As shown in Figure 9, micro silica implemented in this work generally exhibits a reduction in the water absorption potential of concrete the reduction, recognized at 1%, while at 2, 3 and 4% that showed higher water absorption, however, it's still lower than control mix this result agrees with [7] who reported an improvement when using micro silica, while [21] indicate that the combined effect of micro and nanosilica enhance the durability of concrete by decreasing of the water absorption. The reduction in water absorption values in micro silica mixes were attributed due to the higher pozzolanic effect of micro silica, which made the concrete more compact and denser than conventional concrete. In mixes with $CaCO_3$ recognize less reduction in water absorption cab when comprised with micro silica mixes. Better enhancement was found in binary mixes due to the dual effect of micro silica and micro $CaCO_3$ by accelerate the hydration process and formation additional amount of C-S-H gel which develop the pore structure and mechanical properties of concrete [20,22,23].



(a)



(b)



(c)

Figure 9: The results of water absorption test. (a)For concrete mixes with micro silica. (b)Forconcrete mixes with micro $CaCO_3$. (c)For concrete mixes with (micro silica + micro $CaCO_3$).

V. Microstructure Characteristics Study

The study of the microstructure for concrete specimens in case of addition micro SiO_2 and $CaCO_3$ SEM analysis was adopted. The samples were taken from the broken pieces obtained after testing for the compressive strength of the specimens. The samples were dried at room temperature and then examined by SEM analysis with higher magnification. Figures 10 and 11 shows the micrographs of specimens for the concrete mixes containing micro silica particles and micro $CaCO_3$ particles respectively. From the images, it could be seen that the concrete specimens with addition of micro particles were homogenous matrix filled up by the micro particles due to rapid processing of CSH gel in presence of desperation micro particles. Agglomerate of particles were also observed in this micrograph.

5. Conclusion

Based on experimental results of this work, the following conclusions are reported:

1.The addition of micro silica leads to important increase in compressive strength, splitting tensile strength and flexural strength for all tested proportions, were, max increasing ratios found at concrete mix with 4% micro silica. In addition, a remarkable reduction in water absorption was obtained when using micro silica as replacement of cement.

2. $CaCO_3$ particles have no obvious effect on mechanical properties until reaching to 3% weight replacement. Slight effect on water absorption found when adding micro $CaCO_3$ to concrete mixes.

3. Binary mixes showed good improvement in both of mechanical and durability properties of high strength concrete.

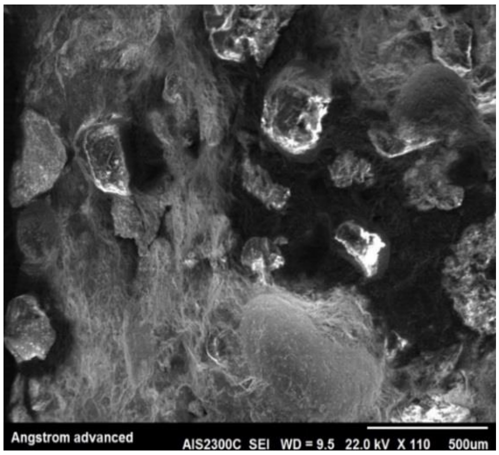


Figure 10: SEM for concrete with micro silica

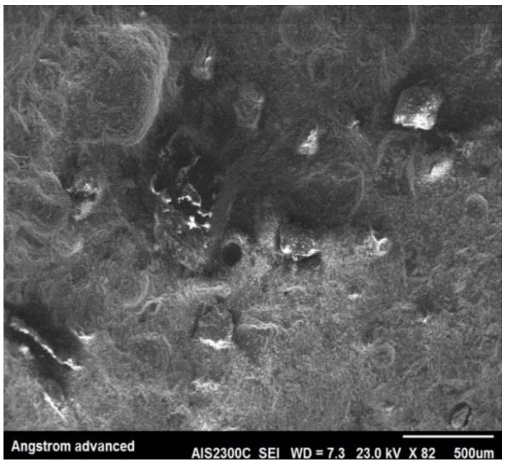


Figure 11: SEM for concrete with micro $CaCO_3$.

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