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Performance of Concrete Containing Zeolite As a Supplementary Cementitious Material

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Abstract - *Pozzolans and supplementary cementitious materials (SCMs), either natural or artificial, are often used as an enhancer material replacement for cement with different percentages in concrete. Due to cost reasons, effective alternative and innovative green material must be used. Silicon dioxide in zeolite is expected to increase compressive strength of concrete through reaction with the calcium hydroxide at the hydration process of cement. This present study provides a comprehensive laboratory investigation of the effects of incorporation of zeolite (untreated and calcined) as a pozzolan at different levels of substitutions of Portland cement (from 0 to 40% by weight of cementitious materials content) on the properties of fresh and hardened concrete and on concrete durability (e.g., absorption and elevated temperatures). Fresh concrete properties (e.g., workability and unit weight) were apparently affected by zeolite incorporation the main effect, however, was in significant reduction in workability, especially at percentages greater than 10% level, which are required adding of mid-range and high-range water reducing admixtures in concrete mixture, also a noticeable reduction in the unit weight of concrete, therefore, calcined zeolite is the suitable raw material to produce light weight concrete. Compared to the control mixture, zeolite-blended concrete mixtures (untreated and calcined) showed an improvement in compressive strength at 10% cement replacement level at (7, 28 and 56 days), where as 25 to 40% replacements caused a modest reduction in strength at the same ages. Pozzolanic action enhances the strength at lately ages more than early ages. Pozzolanic action of zeolite improved the compressive strength of concrete.*

Key Words: Supplementary Cementitious Materials (SCMs), Zeolite, and Physical and Mechanical Properties of Concrete.

1. INTRODUCTION

Concrete belongs to the most frequently used materials in the civil engineering industry for many years. It's have an energetically consumption component like cement. Production of cement is not only energy intensive but it also, presents one of the important CO_2 emission sources in the world. Due to safe environmental condition, a lot of attempts were took place to use different supplementary cementitious materials (SCMs), which can replace at least a part of cement in concrete by more environmental friendly materials **[1]**. Natural pozzolan give a solution of cement replacement in concrete in case of availability near to the cement production field. After mining they do not need any special treatment except for grinding which is their main advantage. There are three main categories of natural pozzolan, namely the materials of volcanic origin, sedimentary origin and mixed origin (hybrid rocks). Zeolite, as probably the most often used natural SCM, belong to the volcanic pozzolan **[2]**.

Clinoptilolite is one of zeolite contents, an aluminosilicate with micro porous framework structure, Concerning illustration the fundamental intensify. The structure of clinoptilolite is based on a three dimensional skeleton made of silicon tetrahedrons unified by oxygen atoms with a part of silicon atoms replaced by aluminum atoms. Zeolite contains silicon oxide with almost 70% and there is usually almost 12% of aluminum oxide. In civil engineering (construction), their usage as pozzolan dates back to ancient times when the mixture of zeolite containing lime and tuff were used as hydraulic binder. Recently, in building construction, zeolite are used mostly as concrete additives **[3]**.

Mechanical properties of zeolite concrete were investigated in many studies as well. **Chan and Ji [4]** noted that, concrete with 10% Portland cement replacement with zeolite gave compressive strength after 28 days within a range of 58–116 MPa, depending on the water to cementitious material ratio. **Najimi et al. [5]** conduct that, incorporation of 15% zeolite in the blended binder improved compressive strength of concrete but for concrete with 30% zeolite content they obtained a 25% strength reduction even with adding a superplasticizers which was not used in the control mixture. **Ahmadi and Shekarchi [6]** observed that, an increase in compressive strength of concrete for up to 20% of zeolite used as Portland cement replacement but this was achieved with an increasing amount of superplasticizers in the mixtures containing zeolite. **Uzal and Turanlı [7]** reported similar compressive strength of mortar with 55% zeolite in the binder to that of 100%Portland cement mortar but once again, this could only be achieved using superplasticizers. **Karakurt and Topcu [8]** found 30% replacement of Portland cement by zeolite in their blended cement mortars recorded the optimum; the compressive strength was similar to the control mortar. **Valipour et al. [9]** observed a fast decrease in the compressive strength of concrete with the increasing amount of zeolite (10–30% of the weight of Portland cement) in the mixture, even with an increasing superplasticizers dosage.

Hence, the present study provides a comprehensive laboratory investigation of the effects of incorporation of zeolite (untreated and calcined) as a pozzolan at various levels of substitutions of Portland cement (from 0 to 40% by weight of total cementitious materials) on the properties of fresh and hardened concrete and on concrete durability (e.g., elevated temperatures).

2. UESED MATERIALS AND MIXTURE PROPORTIONS

The used cement was Ordinary Portland Cement type CEM I – 42.5N complies with the **Egyptian Standard (ES: 4756-1/2009) [10]**. The chemical analysis as well as the physical properties of the used cement as determined by laboratory tests **(as per E.S.S No. 2421/2005) [11]**, showed its suitability for concrete work. The properties of used cement in this study are shown in **Table -1**. Zeolite were delivered in 25-kg package with particle size 50μm and **Table -2** shows chemical composition of it. Crushed stone from natural sources with a nominal maximum size of 12 mm is used in the experimental work. The properties of crushed stone are complying with **E.S.S No. 1109/2002 [12]**. The physical properties are presented in **Table -3**. Fine aggregate used in this work is natural siliceous sand. The sand is clean free from impurities. Testing of fine aggregate (sand) was carried out according to the Egyptian standard specifications **E.S.S No. 1109/2002 [12]**. The physical properties of the sand are shown in **Tables -4**. Clean tap drinking water is used in this work. It is clean and free from impurities. The stated materials were used as the basic solid ingredients in all concrete mixtures.

Seventeen different concrete mixtures were prepared divided into two groups, the first group (untreated) incorporating 5, 10, 15, 20, 25, 30, 35 and 40% zeolite by weight of total cementitious materials (as a replacement), the second group (calcined) also, incorporating 5, 10, 15, 20, 25, 30, 35 and 40% zeolite by weight of total cementitious materials (as a replacement), where the zeolite used in this group was calcined at temperature (500˚C) for 2 hours, in additional to control mixture as presented in **Table -5**. All mixtures were prepared by using a fixed total cementitious materials content of 350 kg/m^3, a water cementitious materials ratio (w/c) of 0.5, also, a high range water reducer and set retarding admixture of modified synthetic dispersion basis **(complies with ASTM C 494 Type G and BS 5075 Part 3) [13]** were used in the designed concrete mixture for reducing the amount of the mixing water. Incorporation of zeolite makes concrete unworkable and sticky due to loss of mixing water (because it's have a very high surface area "open crystal structure" and will appear significantly in microstructure study), which required addition of high range water reducer to maintain the optimum slump at a given w/c. The quantities of coarse aggregate and fine aggregate are 1260 kg/m^3, 635 kg/m^3, respectively. Constant amount of admixture was also added with (2% of cement).

Table -1: Properties of Cement Type (CEM I 42.5 N)

Properties		Measured Values	Limits of the E.S.S*
Fineness (cm^2/gm)		3290	-
Specific Gravity		3.15	-
Expansion (mm)		1.2	Not more than 10
Initial Setting Time (min)		180	Not less than 60 min
Final Setting Time (min)		230	-
Compressive Strength (N/mm^2)	2 days	22.4	Not less than 10
	7 days	33.7	-
	28 days	56.8	Not less than 42.5 and not more than 62.5
Chemical Compositions	SiO_2	20.36 %	-
	Al_2O_3	5.12 %	-
	Fe_2O_3	3.64 %	-
	CaO	63.39 %	-
	MgO	1.03 %	-
	SO_3	2.21 %	-
	Loss Ignition %	1.3 %	-

* Egyptian Standard No: 4756-1/2009.

Table -2: Chemical Composition of Zeolite

Chemical Composition	Percentage (%)
SiO_2	63.72
Al_2O_3	11.40
Fe_2O_3	2.73
CaO	3.29
MgO	0.05
SO_3	0.13
Na_2O	1.02
K_2O	2.83
TiO_2	0.29
P_2O_5	0.03
L.O.I	14.20
Total	**99.69**

Table -3: Physical Properties of the Used Crushed Stone

Test	Results	Acceptable Limit
Specific gravity	2.61	-
Unit Weight (t/m^3)	1.65	-
Materials Finer than no 200 Sieve	1.38	Less than 3%
Absorption %	2.15	Less than 2.5%
Abrasion (Los Anglos)	14.84	Less than 30%
Crushing Value	17.55	Less than 30%
Impact	9.20	Less than 45%

Table -4: Physical Properties of the Used Sand

Test	Results	Acceptable Limit
Specific Gravity	2.63	-
Unit Weight (t/m^3)	1.70	-
Materials Finer than No. 200 Sieve	1.36	Less than 3%

Table -5: Mixtures Proportions for Untreated and Calcined Zeolite

Mixture	Cement (Kg/m3)	Zeolite (Kg/m^3)	*Treatment of Zeolite
M0	350	-	-
M5	332.5	17.5	*Untreated
M10	315	35	*Untreated
M15	297.5	52.5	*Untreated
M20	280	70	*Untreated
M25	262.5	87.5	*Untreated
M30	245	105	*Untreated
M35	227.5	122.5	*Untreated
M40	210	140	*Untreated
M5	332.5	17.5	*Calcined
M10	315	35	*Calcined
M15	297.5	52.5	*Calcined
M20	280	70	*Calcined
M25	262.5	87.5	*Calcined
M30	245	105	*Calcined
M35	227.5	122.5	*Calcined
M40	210	140	*Calcined

*Untreated zeolite which in its natural composition.
*Calcined zeolite which is calcined at temperature (500˚C) for 2 hour.

3.1 CONCRETE TESTING

3.2.1 FRESH CONCRETE

3.2.1.1 WORKABILITY

The workability was tested using slump cone test on the control mixture and combination with zeolite powder (untreated and calcined). **Table -6**, shows the slump test results for different mixtures. It is found that from **Figure -1** a clear reduction in workability, which required addition of a mid-range water reducer, as mentioned before, by increasing the amount of zeolite in the mixture. By appropriate dosages of the high-range water reducer, slump values of all mixtures were kept within 50 to 120 mm for untreated mixtures, while the in the calcined mixtures ranged from 40 to 120mm.

Table -6: Slump Values for Untreated and Calcined Zeolite Mixtures

Mixture	Slump (mm) untreated zeolite	Slump (mm) calcined zeolite
M0	120	120
M5	110	115
M10	95	85
M15	80	70
M20	65	70
M25	55	50
M30	55	45
M35	50	45
M40	50	40

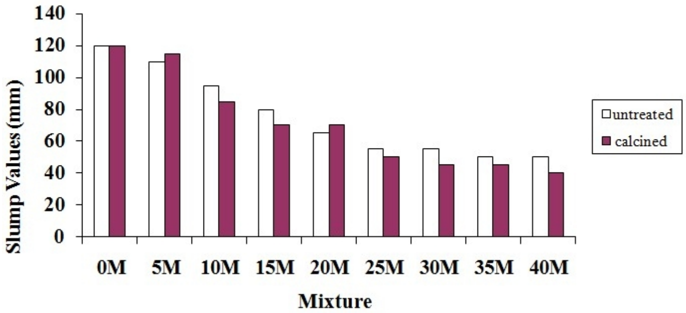

Fig -1: Slump Values for Untreated and Calcined Zeolite Mixtures

3.2.2 HARDENED CONCRETE

3.2.2.1 UNIT WEIGHT

Table -7, presented the unit weight values for all mixtures, it can be seem that, the calcined mixtures shows lower unit weight comparing with the untreated mixtures, this may attributed to zeolite is a porous silicoaluminate mineral. It has generally a very large surface area greater than 20 m^2/g. It's solid surface can be activated to have high surface energy after calcinations at temperature greater than 400°C. When zeolite immersed in water, the surface generates a large amount of bubbles in addition to a amount of heat due to adsorption process. The generated heat increases the temperature of the bubbles in pores or stick on the surface of zeolite particles. The expansion of the bubbles volume lead to foaming and volume expansion of fresh concrete during the mixing and pre-storage periods **[14].** Based on the listed results it is conduct that, the usage of zeolite is suitable for production of lightweight concrete.

Table -7: Unit Weight Values for Untreated and Calcined Zeolite Mixtures

Mixture	Unit Weight (Kg/m^3) untreated	Unit Weight (Kg/m^3) calcined
M0	2426	2426
M5	2428	1890

M10	2429	1824
M15	2422	1798
M20	2419	1742
M25	2412	1712
M30	2406	1698
M35	2385	1675
M40	2377	1643

3.2.2.2 COMPREWSSIVE STRENGTH

Table -8 and **Figures -2 and -3**, provide results of compressive strength tests and rate of strength development in different concrete mixtures. For untreated concrete mixtures, zeolite incorporation at 10 % level showed 7, 28 and 56 days high compressive strengths nearly about 26% comparable to the control mixture. Therefore, at 10 % cement replacement of zeolite incorporation showed a significant enhancement in compressive strength of concrete in compared to the control mixture. While the compressive strengths for calcined concrete mixture at different ages (7, 28 and 56 days decreased comparable to the control mixture by (3.7, 8.4 and 12.4%) respectively, but at this level of incorporation it showed a higher increase in compressive strength comparing with other levels of incorporation.

At higher levels of zeolite incorporations (e.g., from 25 to 40%), however, compressive strengths show a modest reduction ranged from 4 to 16% (untreated) and 34.7 to 47.3% (calcined) respectively from the control mixture at 28 and 56 days. It is apparent that the great reduction in strength of calcined concrete mixtures are attributed to that zeolite is a porous silicoaluminate mineral. It has a very large surface area generally greater than 20 m^2/g. Its solid surface can be activated to have high surface energy after calcinations at temperature greater than 400°C. When zeolite immersed in water, the surface generates a large amount of bubbles and a amount of heat due to adsorption. This heat increases the temperature of the bubbles in pores or stick on the surface of zeolite particles. The expansion of the bubbles volume results in foaming and volume expansion of fresh concrete during the mixing and pre-storage periods. On the other hand, zeolite can react with calcareous materials to form calcium silicoaluminate hydrates which contribute to the strength of the concrete, so that, zeolite is suitable material to use as a suitable raw material to produce light weight concrete by foaming function **[14]**.

Other literature studies on mechanical properties like compressive strengths of zeolite blended Portland cement concrete showed strength enhancement, comparable strength, and modest reductions compared to the strength of control mixture. Such variations in strength results are due to the differences in chemical composition, fineness, reactivity, and the percentage of zeolite levels in the blended cement.

Table -8: Compressive Strength Values for Untreated and Calcined Zeolite Mixtures

Mixture	**Untreated Zeolite Compressive strength (N/mm²)**			**Calcined Zeolite Compressive strength (N/mm²)**		
	7 days	**28 days**	**56 days**	**7 days**	**28 days**	**56 days**
M0	24.1	31.0	33.8	24.1	31.0	33.8
M5	28.8	34.5	36.7	22	26.8	28.6
M10	30.5	38.9	42.5	23.2	28.4	29.6
M15	27.0	35.6	41.2	22.5	27.2	29.8
M20	26.2	32.4	36.5	20.4	24.5	26.5
M25	23.7	29.8	32.4	15.5	20.2	22.9
M30	21.5	28.8	31.2	15.6	18.6	19.2
M35	20.2	26.5	29.8	14.6	18.2	19.3
M40	20.5	26.0	29.0	13.6	17.2	17.8

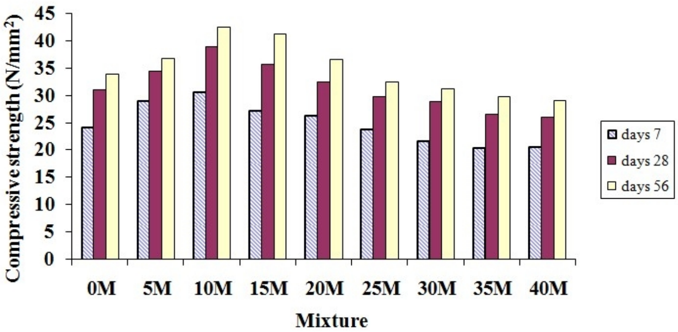

Fig -2: Compressive Strength for Untreated Zeolite Mixtures

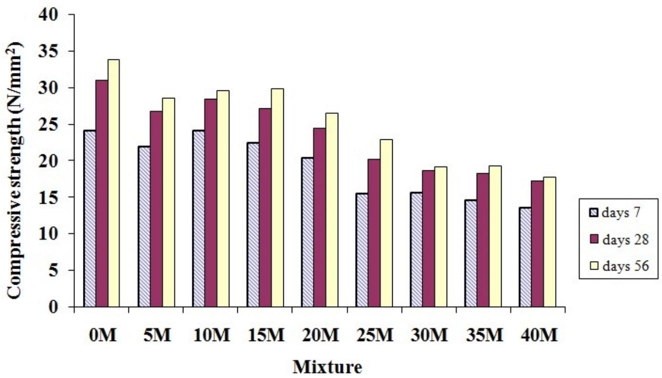

Fig -3: Compressive Strength of Calcined Zeolite Mixtures

3.2.2.3 ELEVATED TEMPERATURE

Table -9 and **Figures -4 and -5** provide results of compressive strength tests and the rate of strength development of different concrete mixtures at 28 days after subjecting to elevated temperatures. For untreated concrete mixtures, the compressive strength of concrete mixtures showed a reduction ranged from (11 to 18.4%) at 300˚Cfor percentage of zeolite incorporation ranged from (0 to 40%), respectively, while it was ranged from (22 to 28.5%) at 600˚C for percentages of zeolite incorporation ranged from (0 to 40%), respectively.

For calcined concrete mixtures, the compressive strength of concrete mixtures showed a reduction ranged from (8.2 to 14%) at 300˚C at percentage of zeolite incorporation ranged from (0 to 40%), respectively, while it was ranged from (13.8 to 23.2%) at 600˚C at percentage of zeolite incorporation ranged from (0 to 40%), respectively. It is apparent that the use of calcined zeolite decrease the rate of reduction of compressive strength which attributed to heat treatment of zeolite which activate the surface of zeolite and result in high surface energy. Heat treated zeolite release large amounts of bubbles from their surface after re-wetting as water is re-adsorbed on the zeolite surface. Heat generation during bubbles desorption and water adsorption increases bubbles formation in the mixture. Heat treated zeolite can be use as an air-generation agents or as a pozzolanic binding materials to produce lightweight concrete **[14]**.

Table -9: Compressive Strength Values for Untreated and calcined Zeolite Mixtures after Subjecting to Elevated Temperatures

Mixture	Untreated Zeolite Compressive strength (N/mm^2)			Calcined Zeolite Compressive strength (N/mm^2)		
	At room temp.	300˚C	600˚C	At room temp.	300˚C	600˚C
M0	31.0	27.6	23.8	31.0	27.6	23.8
M5	34.5	30.7	26.6	26.8	24.6	22.7
M10	38.9	33.6	30.1	28.4	25.5	23.4
M15	35.6	30.6	27.5	27.2	24.2	22.6
M20	32.4	27.8	25.2	24.5	22.3	19.7
M25	29.8	26.7	21.8	20.2	18	15.6
M30	28.8	24.6	22.3	18.6	16.5	15
M35	26.5	22.6	20.2	18.2	16.2	14.5
M40	26.0	21.2	18.6	17.2	14.8	13.2

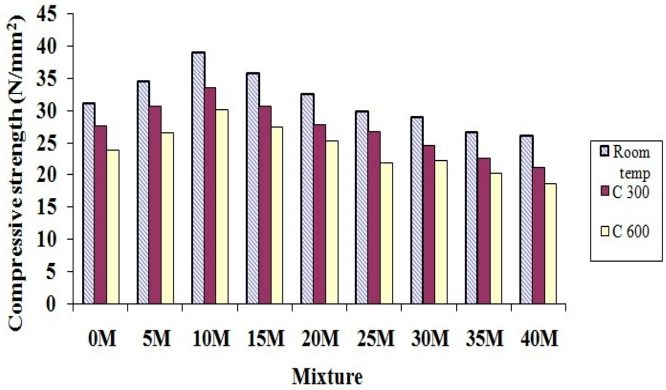

Fig -4: Compressive Strength for Untreated Zeolite Mixtures at 28 Days

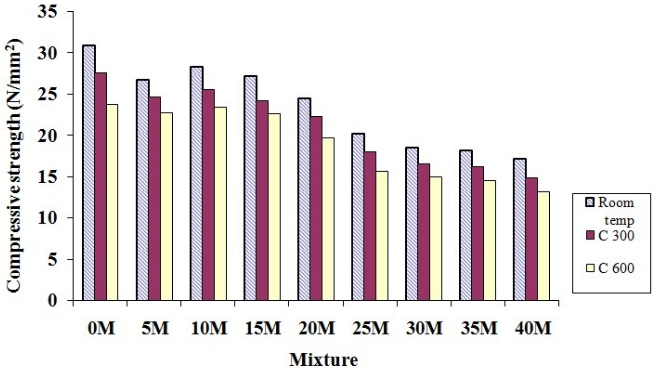

Fig -5: Compressive Strength of Calcined Zeolite Mixtures at 28 Days

3.2.2.4 ABSORPTION

Table -10 and **Figure -6** provide results of percentage of absorption test of different concrete mixtures at 28 days. For untreated concrete mixtures, the percentage of absorption was decreased as the percentage of zeolite incorporation increased from (0 to 40%) ranged from (1.02 to 0.313%) respectively, and these results agree with other investigators. **Chan and Ji [4]** found water initial surface absorption decreasing up to 15% cement replacement level and increasing above that limit. **Najimi et al. [5]** reported a decrease in water penetration depth up to 30% of zeolite content in the blended binder. While for calcined concrete mixtures, the percentage of absorption was increased as the percentage of zeolite incorporation increased from (0 to 40%) ranged from (1.02 to 1.724%), respectively and this may attributed to the calcinations of zeolite at high temperature (500˚C) before used in the concrete mixtures, This heat increasing the temperature of the bubbles in pores or adsorbed on the surface of zeolite particles. The expansion of the bubbles volume results in foaming and volume expansion of fresh concrete during the mixing, and subsequently increase the ability of concrete to absorb water in the hardened state.

Table -10: Percentage of Absorption Values for Untreated and Calcined Zeolite Mixtures

Mixture	Untreated Zeolite % of Absorption	Calcined zeolite % of Absorption
M0	1.02	1.02
M5	0.942	1.257
M10	0.845	1.324
M15	0.754	1.405
M20	0.578	1.488
M25	0.502	1.564
M30	0.425	1.601
M35	0.389	1.689
M40	0.313	1.724

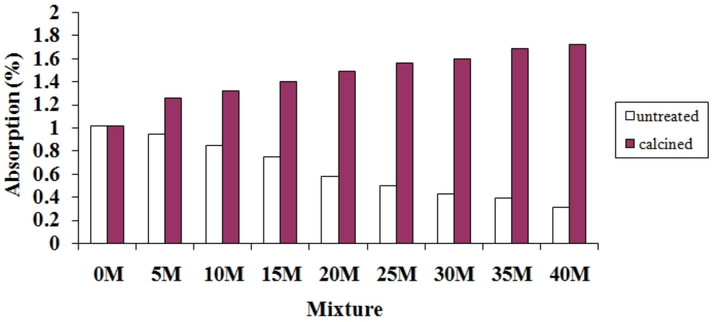

Fig -6: Percentage of Absorption for Untreated and Calcined Zeolite Mixtures

3.2.2.5 MICROSTRUCTURE

Figure -(7a) shows, the scanning electron microscopy (SEM) slide of the ordinary Portland cement concrete without Zeolite. It has independently formed a C-S-H gel, mutually linked needle-shaped hydrates (ettringite), and many $Ca(OH)_2$ crystals, showing a sparse internal structure with non-crystal hydrates. **Figure -(7b)** Incorporation of zeolite (untreated) causes the following important micro structural modifications, which are responsible for the overall improved durability of concrete: (a) by the pozzolanic reaction, a decrease in calcium hydroxide component of Portland cement hydration in the cement paste, (b) concretion of the cement paste microstructure by combined pore size and grain size refinements, which cause an increased volume of very fine pores at the expense of the large pores, and (c) improvement of aggregate paste interfacial zone by a combination of filler effects and pozzolanic reaction effects. Zeolite can be utilized not only as a pozzolan but also as a micro filler in the concrete for the overall densification of micro structure. **Figure -(7c)** shows the presence of small pores on the surface of the specimen due to the incorporation of calcined zeolite as heat treatment of zeolite will activate the surface of zeolite and result in high surface energy. Where heat-treated zeolite release large amounts of bubbles from their surface.

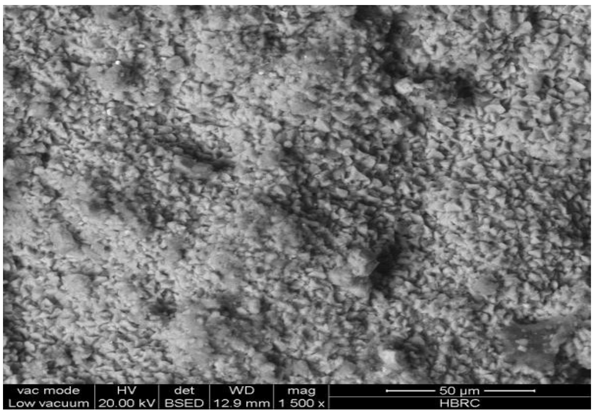

Fig -(7a): SEM of Control Concrete Specimen without Incorporation of Zeolite

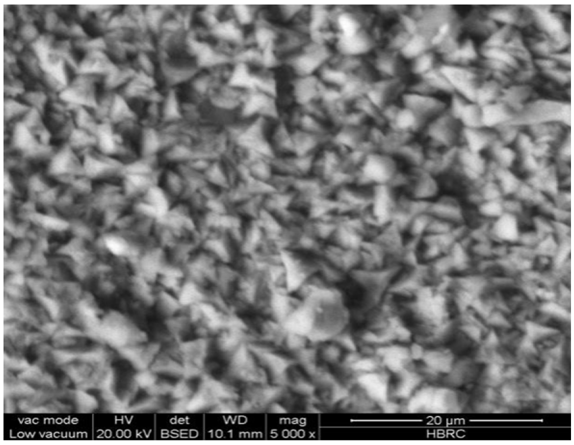

Fig -(7b): SEM of Concrete Specimen 10% Incorporation of Zeolite (Untreated)

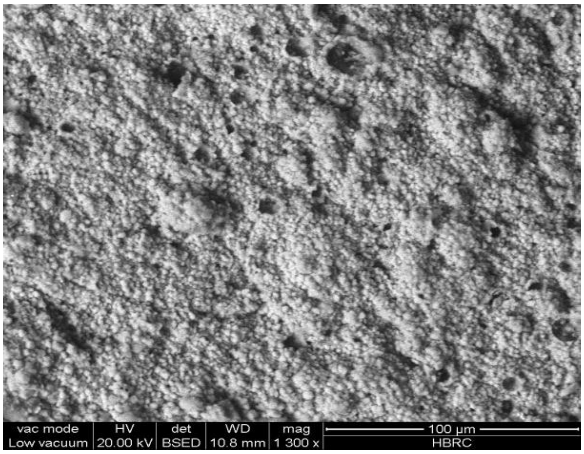

Fig -(7c): SEM of Concrete Specimen 10% Incorporation of Zeolite (Calcined)

4. CONCLUSIONS

In summary, based on the present study and others, zeolite incorporation may show a modest strength benefit or a reduction compared to that in a non-zeolite control mixture. The results can be conducted as follow:

1. Incorporation of zeolite in concrete mixtures up to 40 % by weight of the show a significant reduction in workability of the fresh mixture (due to the high surface area of zeolite) requiring addition of both high-range water reducers.

2. Based on unit weight results the calcined mixtures shows lower unit weight comparing with the untreated mixture, which recommended the application of zeolite as a major component in production of lightweight concrete products.

3. Compared to the control mixture, zeolite blended mixtures (untreated)shows an improvement in compressive strength of concrete at 10% cement replacement level by 26%, whereas 25 to 40% replacements have caused a modest reduction in

strength at the same age ranged from 4 to 16% (untreated) and 34.7 to 47.3% (calcined) respectively at 28 and 56 days.

4. It is apparent that the use of calcined zeolite decrease the rate of reduction of compressive strength.

5. For untreated concrete mixtures, the percentage of absorption was decreased as the percentage of zeolite incorporation increased, while for calcined concrete mixtures, the percentage of absorption was increased as the percentage of zeolite incorporation increased.

6. Incorporation of zeolite (untreated) causes important micro structural modifications, which are responsible for the overall improved strength of concrete.

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