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The impact of zeolite on mineralogy changes and compressive strength development of cement-treated sand mixtures through microstructure analysis

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Zeolite.

Abstract. The main objective of this research is to study the impact of zeolite on mineralogical changes leading to development of the compressive strength of cemented-sand mixtures. The mixtures consist of Portland cement type II, natural zeolite (Clinoptilolite) and Babolsar sand, and the cement content is chosen to be 8% based on the dry weight of the sand. The experimental program consists of a cement substitute with 0, 35, 60, and 90% zeolite along with the amount of optimal water content obtained from the standard compaction test. The samples were made by an under-compaction process and cured at room temperature for various periods of time (7, 28, and 90 days). The microstructural properties were analyzed using X-Ray Diffractometer (XRD) tests and Scanning Electron Microscopy (SEM) tests equipped with an Energy Dispersive X-ray analysis system (EDX). In addition, an unconfined compression test was carried out for various zeolite percentages in the same curing time. Strong adhesion in the Interfacial Transition Zone (ITZ) resulted in densely compacted mineralogy in the presence of 35% zeolite, which promoted the Unconfined Compressive Strength (UCS). The connection between microstructure and macrostructure clearly showed suitable relations between compressive strength and the intensity of the C-S-H phase.

1. Introduction

Sandy soils exist all over the world and many geotechnical engineers face these problematic soils. Further considerations about large pores, lower bearing capacity, and large deposits relate to these soils. Particular attention has been paid to improving the strength behavior of sandy soils over cement and pozzolan. Cement as a conventional modifier has been used in the last few decades to improve the mechanical behavior of sand [1–5]. However, economic and ecological considerations for stabilizing sandy soils with cement must be carefully considered. The high cost of cement production compared to the cost of raw materials leads to the production of more expensive materials than natural pozzolanic materials such as zeolite. In addition, the pollution caused by cement plants has a negative impact on geological conditions [6,7]. It should be mentioned that 7% of CO_2 emissions are

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due to the global cement industry [6,8]. Among the various pozzolanic fillers available in place of cement, zeolite is a suitable substitute because of its improved performance and environmentally friendly composition [9,10]. In contrast to cement considerations, zeolites are natural cation exchangers and also have a molecular sieve structure; thus, they tend to remove toxic cations [11,12]. Clinoptilolite seems to be the most efficient ion exchanger and ion selective material among natural zeolites [13,14].

Today, zeolite is widely used in many parts of our lives as adsorbents, detergents for gas and petroleum processing, mining and paper products. Among other things, the excellent properties of zeolites such as their large surface area, the molecular structure of the sieve type, and the high absorption capacity cause unique physical and chemical properties compared to other pozzolanic materials that have also been studied by geotechnical engineers. Yukselen-Aksoy [15] has shown that the shear strength of zeolite is significantly high. The main aim of this research is to investigate the impact of zeolite addition on the compressive strength development of cement-treated sand mixes, due to their inherent positive mechanical performance. In addition, a suitable relationship was established between the intensity of the C-S-H gel and the compressive strength.

Several studies have been carried out to demonstrate the excellent physical and chemical properties of zeolites, including zeolite-sand and zeolite-cement mixtures. Zeolite-sand mixtures have been examined in numerous studies for environmental applications, since they have little or no cement-like properties in the absence of water and cementitious materials. The zeolite-sand mixture as a reactive material is suitable for groundwater remediation. Dinari and Eslami [16] studied the effect of Clinoptilolite on destabilizing oil-in-water emulsion. Their findings showed the remarkable removal of cationic surfactant molecules both from the continuous phase and from the oil-water interfacial through the addition of zeolite particles. Joanna and Kazimierz [17] investigated the mixture particularly in the vicinity of old landfills. Foo and Hameed [18] investigated the environmental applications of activated zeolite composites such as wastewater treatment, gas cleaning and several other uses, and reported that the enhancement of activated zeolite composite is a potentially viable and powerful tool that leads to a plausible improvement in environmental protection.

Adding zeolite promotes the properties of the interfacial microstructure and also reduces the porosity of the mixed cement matrix [5,9,19–25]. Iswarya and Beulah [1] studied zeolite and industrial waste in high strength concrete. They observed that the partial replacement of cement by zeolite, metakaolin, fly ash, and silica vapor increases the properties of concrete to a certain extent compared to conventional concrete. The strength development of mixed cement with the addition of natural zeolite depends on the zeolite content [6,26]. Mehta [27] has reported that mixed portland cements with 10%, 20% and 30% natural pozzolan produced a compressive strength similar to or higher than the reference portland cement, and were much more resistant to alkali-silica reaction and the sulphate effect. The reaction of silica from zeolite with Ca $(OH)_2$ due to the addition of zeolite to cement is responsible for the production of C-S-H gel and leads to the development of strength [28–30].

Much effort has been made to study the compressive strength of cemented sand and its multiple uses in many geotechnical projects, e.g. pavement of base layers, the slope of protected earth dams, and as a support layer for flat foundations [31–35]. Cement content, porosity, and moisture are key parameters that have been taken into account to control the strength of cemented sand in order to study the strength development of cemented sand [36–39].

Sand stabilization with cement and zeolite has been applied in various constructions such as substrates, embankments and foundation structures. The impact of zeolite substitution on the mechanical properties of cement-treated sand has been investigated in recent years.

Mola-Abasi and Shooshpasha [40] used artificial neural networks to obtain a polynomial model to predict UCS results. Their model has shown that the cement and zeolite content are the most effective parameters for UCS results. Salamatpoor et al. [41] have studied the physical and mechanical properties of sand stabilized by cement and natural zeolite. Their results showed that the zeolite was introduced as an effective cement substitute in improving sandy soils. Jafarpour et al. [42] performed a series of consolidated non-drained tri-axial tests to investigate the impact of cementation on yield strength from shear stress. They found that the effective internal friction angle, which corresponds to the yield, always increases with a slight gradient. Jafarpour et al. [43] investigated the behavior of sand grouted with zeolite-cement under a tri-axial compression test. Their results showed that under all pressure limits at which the zeolite increased to a certain level, the maximum deviatoric stress ($q_{\max}$) of the grouted sand samples increased.

In previous studies, however, the relationship between microstructure analyses, such as XRD and Scanning Electron Microscopy (SEM) tests, which were equipped with an EDX measurement according to the strength development behavior of zeolite-cement-sand mixtures, was neglected. Since microstructural properties play a key role in assessment of strength development in cemented soil mixtures, the aforementioned tests were taken into account in this investigation,

Table 1. Chemical analysis of cement and zeolite.

Chemical composition	SiO_2	Al_2O_3	Fe_2O_3	K_2O	CaO	MgO	SO_3	NaO
Percent (%)								
Cement	22.1	4.37	3.1	0.54	64.27	1.1	2.09	3.02
Zeolite	69.32	10.64	1.1	0.41	2.37	1.3	0.11	1.35

together with an unconfined compression test, in order to reveal the microstructural effectiveness of zeolite-cement-sand mixtures at different curing times and zeolite percentages. Due to the small size of the Interfacial Transition Zone (ITZ), the weakest part of the mixture acts as a bridge between the matrix and sand, which plays an important role in mechanical behavior [44,45].

In order to determine the effects of the ITZ on the mechanical properties, microstructure analyses were carried out in detail. In the current research, the results obtained have been confirmed by UCS, resulting in a deep understanding of the microstructural properties, and demonstration of the mechanical behavior of the mixtures. The results showed how the compressive strength of the zeolite-cement-sand mixture decreased after a peak with the addition of additional zeolite powder. In summary, it can be said that a high cement replacement by zeolite over 35% led to poor adhesion between the cement matrix and sand in the ITZ. It should be noted that microstructure analyses are consistent with unconfined compression test results.

The remainder of the paper is divided into four sections. Section 2 illustrates a test program with material definitions, sample preparation, and test procedures. Section 3 is devoted to experimental results and discussions, and the conclusion was given in Section 4.

2. Experimental program

The plan of the experimental program was scheduled as follows: 12 unconfined compression tests, 13 SEM tests, and 4 SEM tests equipped with an EDX system and 4 XRD tests were performed to assess the relation between the microstructural properties and compressive strength of zeolite-cement-sand mixtures.

2.1. Materials

The materials used in this research were sand, portland cement type II, and natural Clinoptilolite Zeolite. The sand came from Babolsar located on the Persian coastline and the cement was supplied by Neka cement companies in the north of Iran. Clinoptilolite was taken from the mines of the Semnan province, Iran.

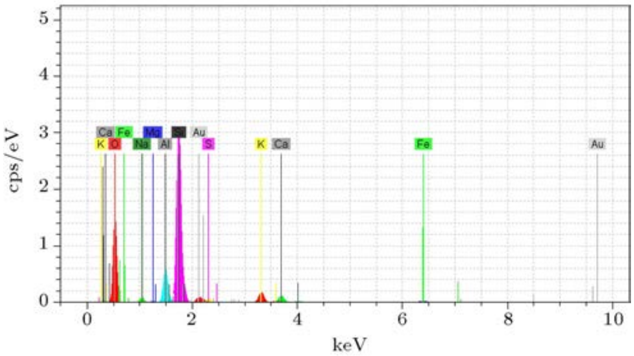


Figure 1. Component analysis of zeolite using EDX.

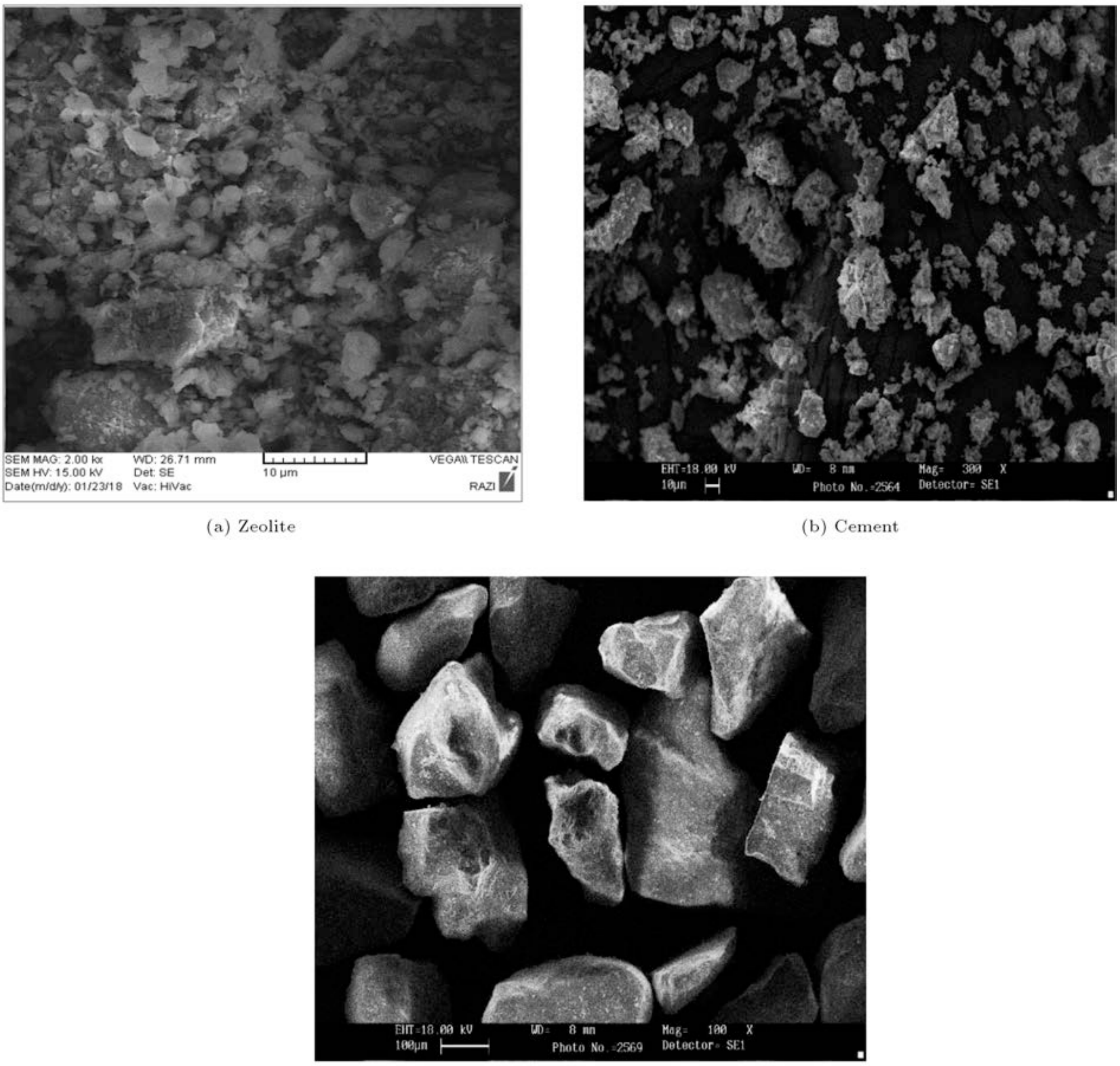
As seen from Figure 1, the component analysis of natural zeolite was performed by EDX and the chemical analyses of sand and cement have been undertaken using ARL 8680+ X-Ray Fluorescence spectrometry (XRF), as shown in Table 1. The physical analysis of zeolite and cement are shown in Tables 2 and 3, respectively. As evident in Table 1, the amount of free lime (CaO) in the cement composition is very high in comparison to zeolite. This composition participates in a pozzolanic reaction with zeolite.

Table 2. Physical properties of Portland cement [48].

Parameters	**Value**
Specific surface (m^2/N)	> 30
Autoclave expansion (%)	< 0.05
Initial setting time (min)	> 75
Specific gravity (G_s)	3.11

Table 3. Physical properties of zeolite [48].

Parameters	**Value**
Specific weight (N/m^3)	11900
Specific surface (m^2/N)	10
Water absorption	60% volumetric
Cation exchange capacity (N/meq)	26
Specific gravity (G_s)	2.2



(a) Zeolite

(b) Cement

(c) Sand

Figure 2. SEM images: (a) Zeolite particles magnified with 2 kx by second electron method–TESCAN model, (b) cement particles magnified with 300× by second electron method–LEO 435VP model, and (c) Babolsar sand magnified with 100× by second electron method–LEO 435VP model.

The SEM images of zeolite, cement powder and sand with sub-rounded particles and grain size distribution of sand are shown in Figures 2 and 3, respectively. As evident in Figure 3, Babolsar sand was classified as poorly graded sand (SP) according to the Unified Soil Classification System (USCS). The mean grain size of the sand (D_{50}) is 0.22 mm, and the coefficient of uniformity and the coefficient of curvature are 1.76 and 1.06, respectively. The specific gravity of the sand is 2.74.

2.2. Preparation of specimens

The samples were prepared roughly uniform based on the optimal water content and dry weight of materials.

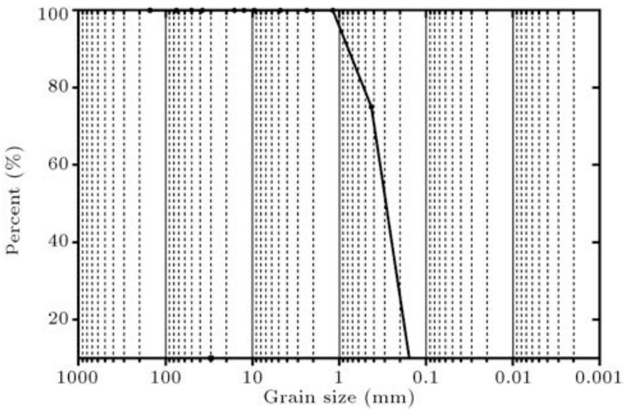


Figure 3. Grain size analysis of sand.

Ladd [46] described preparation of specimens using a compaction method. In this method, the kiln-dried sand was mixed with the required percent of cement based on the dry weight of sand and the cement was replaced by zeolite. The mixing of dry materials continued until a uniform appearance of zeolite-cement-sand mixtures was obtained. Subsequently, water was added continuously to the optimal water content of the material. Further mixing was continued until a homogeneous appearance of moist zeolite-cement-sand mixtures was achieved. The mixture was divided into three portions and stored in an airtight container to avoid any moisture loss before subsequent preparation. Prior to the placement of the mixture, the mold was lubricated and provided with a "thin transparency sheet" to avoid cement materials sticking to the wall of the mold. The samples were prepared using a split mold, 38 mm in diameter. Samples were prepared at a height of 76 mm and compacted in three layers. Each layer was poured into a mold and compacted using a metal hammer until the desired height was reached. The top of each layer was scratched before adding the next layer to promote suitable bonding.

2.3. Test procedure

The mechanical behavior of zeolite-cement-sand mixtures was accomplished by measuring the unconfined compressive strength of samples in accordance with the ASTM D2166 test method, with 7, 28, and 90 days of curing time [47]. The unconfined compression machine has a load ring capacity of 3 kN and 5 kN the loading of which was carried out at a constant strain rate of 0.3 mm/min due to the brittleness behavior of the cemented sand mixtures. Due to the repeatability of the test result response, three identical specimens for each mixture were tested. The average value of the obtained results was used in reports. The amount of water and the dry density of the mixture were adapted based on the dry weight of materials mixed together. The standard proctor compaction method was repeated on three identical specimens for each mixture. An average value of obtained results was used. The XRD tests of zeolite-cement-sand mixtures at 90 days of curing time were accomplished, in accordance with the BS EN 13925-1, via a Philips PW 3710 diffractometer [42]. XRD diagrams were obtained using a Cu Kα radiation with a voltage of 40 kV and current settings of 30 mA. All specimens were oven-dried (80°C, 48 hours), crushed, and sieved. The scan step size is 2θ in the range from 30 to 99. The XRD measurements were conducted with a step size and counting time of 0.02 and 0.5 sec, respectively. The mineralogy of zeolite-cement-sand mixtures was investigated using a scanning electron microscopy (LEO 435VP) via a secondary electron image method. EDX analysis was performed using the backscattered method in a T-SCAN device. Before photographing, specimens were stored on aluminum stumps coated with gold for better conductivity.

3. Results and discussion

3.1. Compaction

Figure 4 illustrates the impact of zeolite on optimal water content and maximum dry unit weight of sandy soil. The larger specific surface of zeolite in comparison with cement led to water retaining properties and the occupation of pores between the sand and cement matrix. As seen in Figure 4(a), the optimal water content of the mixtures was decreased by increasing zeolite in all rates. The maximum dry density of sandy soil increases as a result of increasing zeolite replacement, as clearly shown in Figure 4(b). In fact, fine particles of zeolite within the sand and cement matrix occupied the pores and produced a dense structure leading to an increase in maximum dry density.

3.2. Uniaxial compression test

Figure 5 shows the impact of zeolite replacements on compressive strength with different numbers of

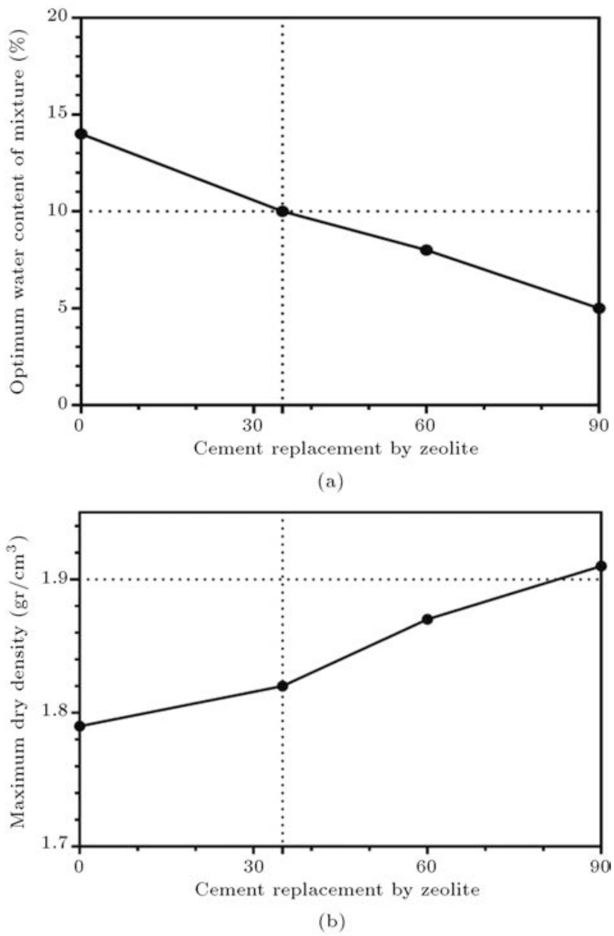


Figure 4. Impact of zeolite replacement percentage on (a) optimal water content and (b) maximum dry density.

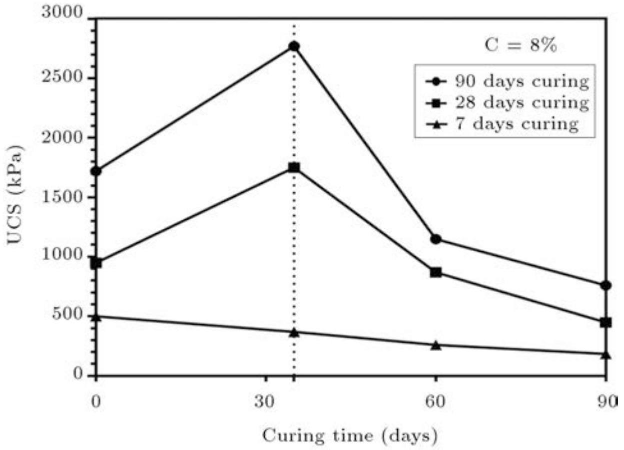


Figure 5. Impacts of zeolite replacement on unconfined compressive strength: (a) 7 days, (b) 28 days, and (c) 90 days.

curing days. As evident in Figure 5, zeolite addition has a noticeable impact on the development of the compressive strength of samples. The most efficient strength magnitude was obtained for 35% replacement in 90 days curing time. Since zeolite particles are finer than cement, they act as filler and occupy the pores between the sand and the cement matrix. Therefore, replacement of up to 35% led to a reduced amount of total pores and a development in the compressive strength of the mixture. Obviously, the adhesion forces of sand particles to the cement matrix have a twofold nature. On one hand, these are physical forces whose magnitude depends on the topography of the sand grain surface and on the grain shape, and on the other hand, they are the adhesion forces of chemical bonds created at the sand–cement paste interfacial in the presence of zeolite. Increasing zeolite by more than 35% in the cement treated sand mixture has a negative impact on mixture strength and reduces the UCS. This phenomenon was explained by microstructure analyses and a vivid connection between micro and macro evidence was demonstrated in Section 3.3.

3.3. Microstructural analyses

Microstructural properties play a fundamental role in the assessment of strength development in zeolite-cement-sand mixtures. To this end, the XRD, SEM, and EDX analyses were performed and presented in the following sections.

3.3.1. X-ray diffractometer tests

The XRD tests were accomplished for evaluating the presence of different crystalline phases and the intensive peaks of hydration products. The relation between zeolite augmentation and consumption of calcium hydroxide Ca $(OH)_2$ is depicted in Figure 6.

The Ca $(OH)_2$ phase has been liberated from the hydration of cement and water. Immediately after the reaction of the cement with water, primary ettringite was formed. Ettringite is found in this benign state as large needle-like crystals, and should not be interpreted as causing the expansion of the deteriorating mixture. Increasing zeolite replacement by up to 35% led to a reduction in the intensity of the calcium hydroxide and

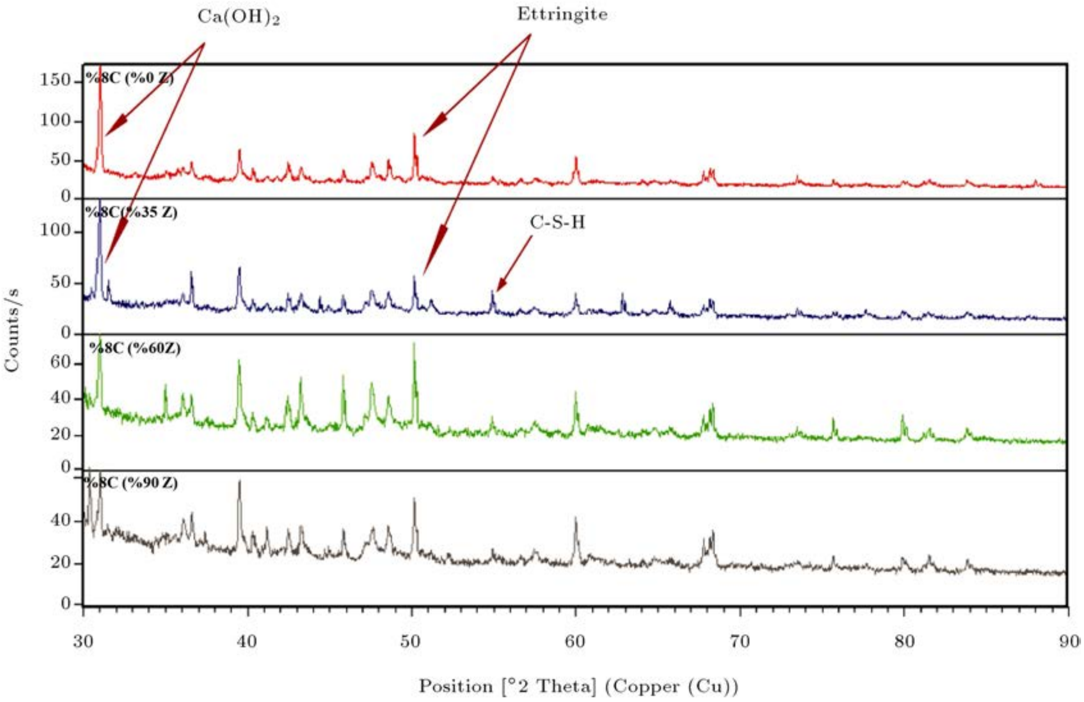


Figure 6. Effect of cement replacement by zeolite on XRD graph of zeolite-cement-sand mixtures.

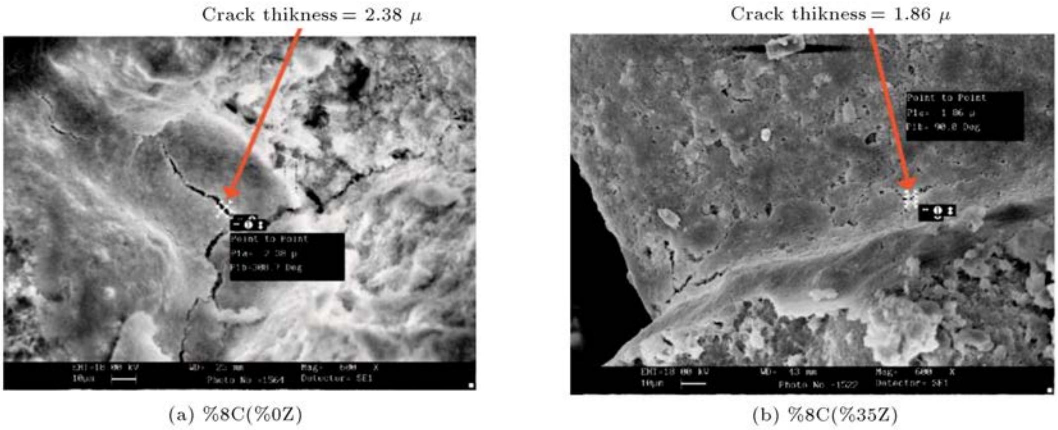


Figure 7. Impact of zeolite replacement on crack propagation and amelioration of the ITZ at 28 days curing of time.

ettringite phase. The C-S-H peak has a broad and diffuse shape and the formation of the C-S-H phase was raised due to the reaction of calcium hydroxide with silica from zeolite. Since this phase fills and covers the ITZ, the interfacial adhesion was strengthened through chemical reactions between the cement matrix and sand. Hence, C-S-H gel ameliorates the ITZ. Replacing beyond 35% reduces the growth rate of calcium hydroxide crystals. The reduction in formed calcium hydroxide causes a lower pozzolanic reaction and crystal-compact C-S-H phase.

3.3.2. Scanning electron microscopy test and energy dispersive X-ray analysis

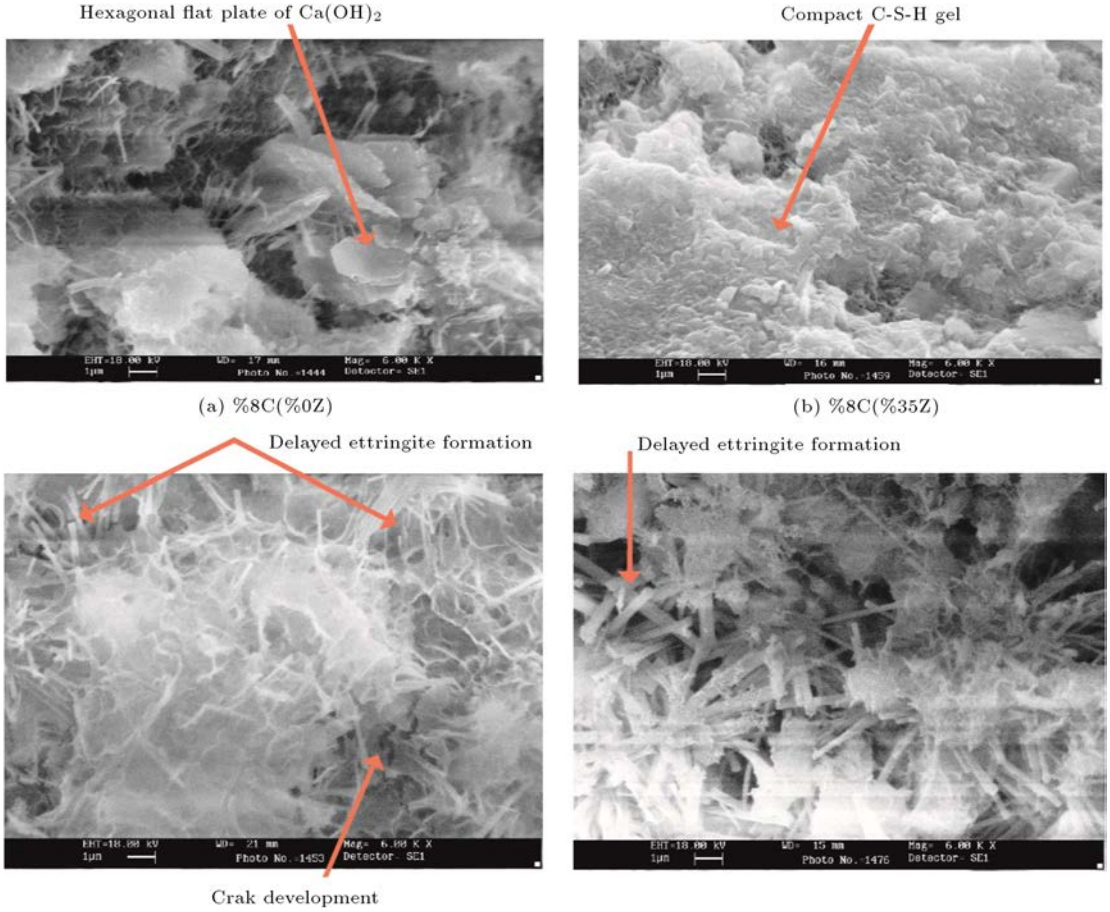
The SEM tests were accomplished to investigate the morphological properties of zeolite-cement-sand mixtures with different zeolite percentages and various durations of curing time. Images were taken by (LEO 435VP) via the secondary electron image method.

Figure 7 shows the images of the zeolite-free mixture with 35% zeolite replacement at 28 days of curing time. In the absence of zeolite, discernible micro cracks are visible, while the propagation and thickness of cracks reduced for the mixture with 35% zeolite.

Figure 8 depicts the morphology of mixtures with and without zeolite at 90 days of curing time. Many pores are shown in Figure 8(a), and the hydration product mostly formed as a calcium hydroxide Ca $(OH)_2$ hexagonal plate shape which was released from the hydration of cement and water, and which is evident in the absence of zeolite. As shown in Figure 8(b), for the one with 35% zeolite replacement, dense-compact C-S-H gel occupies the pores between the sand and cement matrix in the ITZ. This formation of C-S-H gel is a product of Ca $(OH)_2$ consumption during its reaction with silica from zeolite. Hence, in the presence of zeolite, the large flat plate Ca $(OH)_2$ was decomposed to the C-S-H phase with smaller sizes, and enhanced the chemical bond between the cement matrix and sand particles. In contrast with Figure 8(b), this dense-compact C-S-H phase was changed with a delayed formation of needle-like ettringite, as shown in Figure 8(c) and (d). This delayed needle-like crystallization phase of ettringite was an agglomerated matrix of the mixture and led to induce tensile stress and crack development in the matrix of the mixture, which is undesirable. Consequently, weak adhesion was formed between the cement matrix and sand in the ITZ. Strong interfacial adhesion in the ITZ and the development of dense-compact morphology promotes UCS in the presence of 35% zeolite.

Figure 9 shows the structure of Ca $(OH)_2$ with oriented needle-like hexagonal plate shape crystals which is perpendicular to the surface of the sand grains for the specimen with zero percent zeolite at 90 days curing.

Figure 10 depicts the effect of curing time on specimens 7, 28, and 90 days. Here, Figure 10(a), (c) and (e) are illustrated for zeolite-free specimens. As evident in Figure 10(a), a shortage of silica in the absence of zeolite prevents the development of a pozzolanic reaction in which Ca $(OH)_2$ crystals accumulate. These crystals are perpendicular to the surface of the sand grains (also see Figures 8(a) and 9), which results in the formation of a highly porous structure as well as large ITZ between the sand and cement matrix. Figure 10(c) shows the start of the formation of the C-S-H phase at 28 days, and the ITZ between the sand and cement matrix become smaller than Figure 10(a). A gradual growth of C-S-H gel is due to the chemical process of Ca $(OH)_2$ which is obvious in Figure 10(e) for the specimen at 90 days of curing time. In contrast to Figure 10(a), Ca $(OH)_2$ crystals were strongly produced in Figure 10(b). The specimen with 35% zeolite replacement at 28 days is depicted in Figure 10(d). Here, C-S-H formations are more than Figure 10(b) and pores in the ITZ become smaller. As evident in Figure 10(f), a dense and compact structure of the specimen was formed at 90 days of curing time. Vast distribution of compacted C-S-H gel led to cover



(a) %8C(%0Z) (b) %8C(%35Z) (c) %8C(%60Z) (d) %8C(%90Z)

Figure 8. Impact of zeolite replacement on morphology of cement treated sand at 90 days curing of time.

pores and, finally, form strong interfacial adhesion in the ITZ. In comparison with Figure 10(e) for the specimen with zero percent zeolite and the same curing time, more accumulation of C-S-H gel was produced and covered the ITZ space, as shown in Figure 10(f). One could conclude that strengthening the ITZ is a key component in developing the compressive strength of the mixture. It should be noted that Ca $(OH)_2$ contents are oriented needle-like crystals which make ITZ susceptible to crack development. Besides, stress concentration in large pores led to the production of new cracks developing with increasing stress. Reaching developed cracks under stress concentration to ITZ causes a continuous crack and, consequently, failure of the mixture. In contrast with Ca $(OH)_2$, C-S-H gels enhance interfacial adhesion in the ITZ and also develop the compressive strength of the mixture.

A 90-day hardened cement-sand mixture with and without zeolite were scanned using a backscattered method via a T-SCAN equipped with an EDX system. As evident in Figure 11, atomic compositions of point EDX were represented.

From Figure 11(a), it can be seen that the analysis

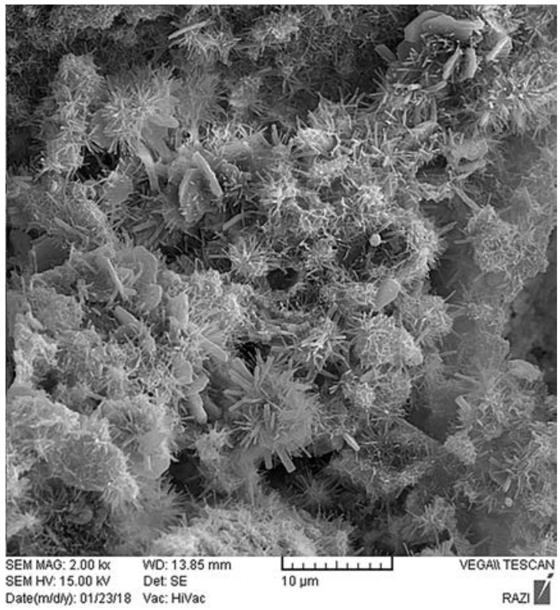


Figure 9. Oriented needle-like with hexagonal plate shape crystals of Ca $(OH)_2$ with perpendicular aspect to the surface of sand grains for specimen with zero percent zeolite at 90 days curing of time.

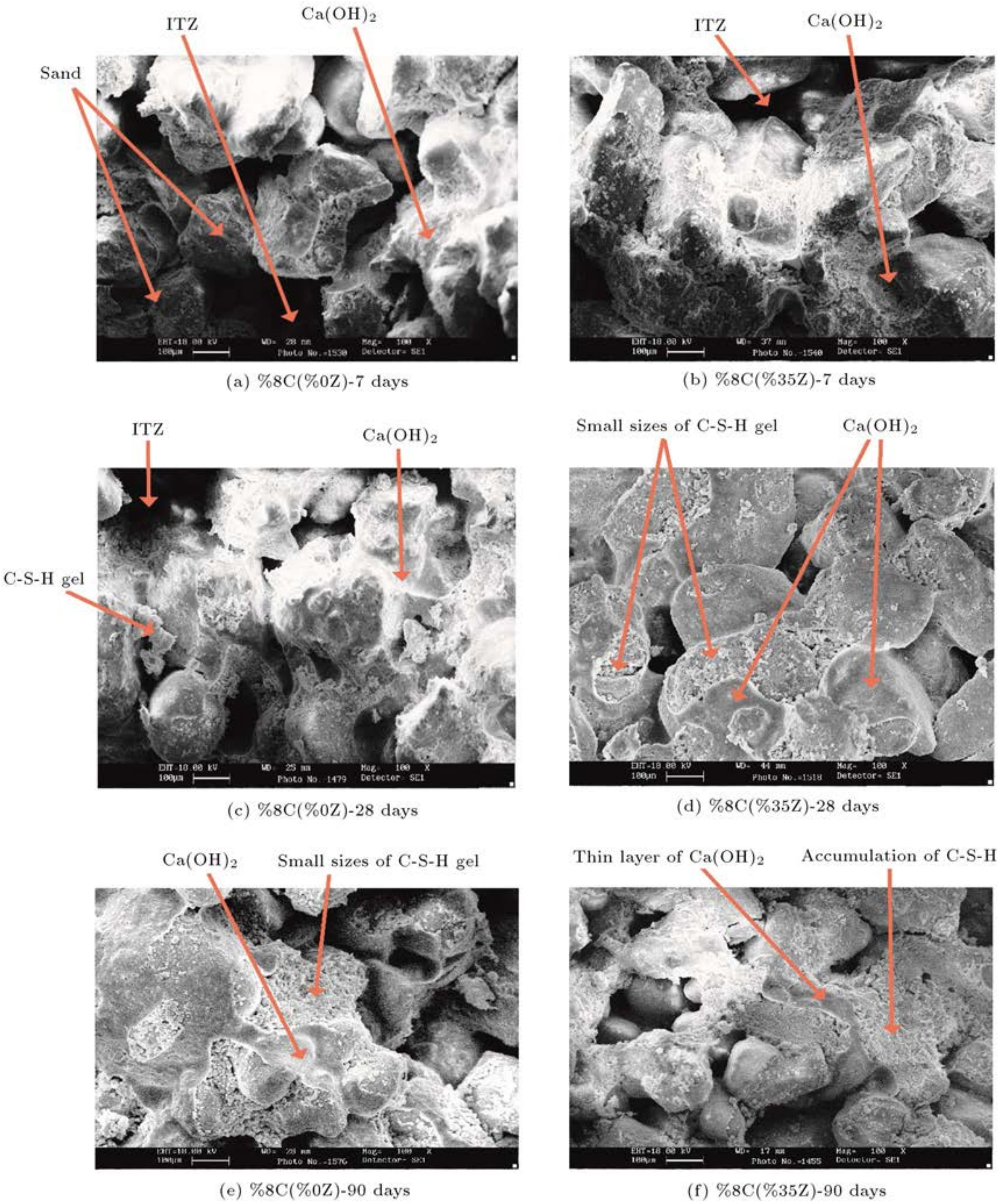


Figure 10. Effect of curing time on the specimens: (a) Zeolite-free specimen at 7 days, (b) specimen with 35% zeolite replacement at 7 days, (c) zeolite-free specimen at 28 days, (d) specimen with 35% zeolite replacement at 28 days, (e) zeolite-free specimen at 90 days, and (f) specimen with 35% zeolite replacement at 90 days.

spectrum of the zeolite-cement-sand mixture in the zeolite-free specimen was presented, and the Si content is low and the Ca content is high. In Figure 11(b), Si and Ca atomic contents are in equilibrium states, while, by increasing the zeolite replacement, more Si than Ca is observed in Figures 11(c) and (d). The analyses of the prone with the existence of enough zeolite to complete pozzolanic reactions could enhance the compressive strength of the mixture, while extra zeolite creates excessive Si resulted in decreasing the cement content and free lime, therefore compressive strength was deminished.

3.3.3. Relationship between compressive strength and mineralogy

The connection between compressive strength and mineralogy in relation with time is demonstrated in Figures 12 and 13, respectively. As evident in Figure 12, the intensive peaks of the C-S-H phase show a logarithmic behavior, along with the curing time.

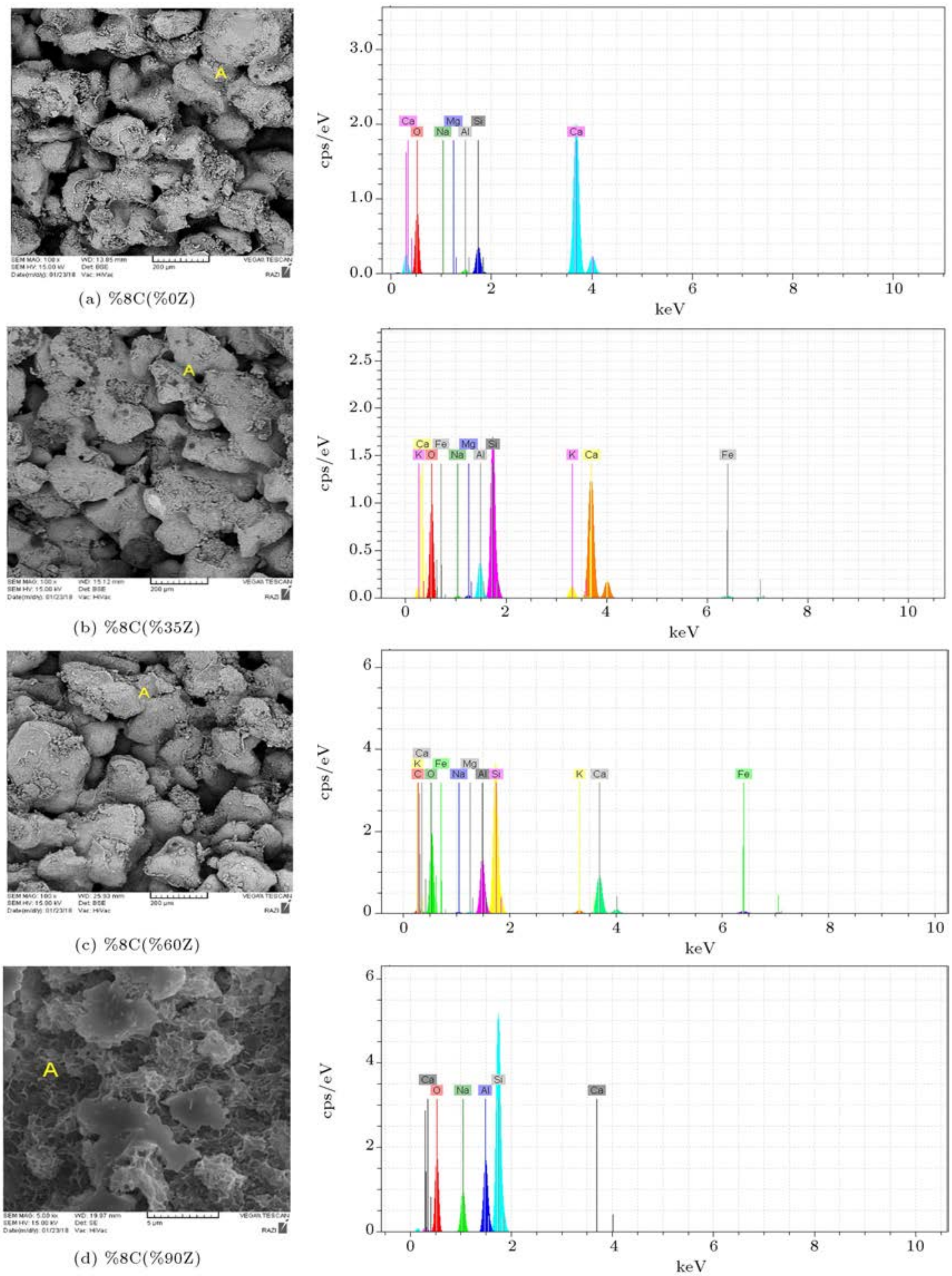


Figure 11. EDX analysis spectrum of zeolite-cement-sand mixture at 90 days curing of time.

Figure 13 shows the connection between compressive strength and the intensity of the C-S-H phase. Increasing compressive strength with the intensity of the C-S-H phase, along with different zeolite replacements provide a good relation. In other words, since the growth rate of the C-S-H phase increases with enhancing the zeolite replacement by up to 35%, it has a noticeable impact on compressive strength. This connection demonstrated a logarithmic behavior between the aforementioned parameters. The XRD peak intensities identified as "C-S-H" illustrate the logarithmic behavior, along with curing time, in blended samples which were exploited from the integral values.

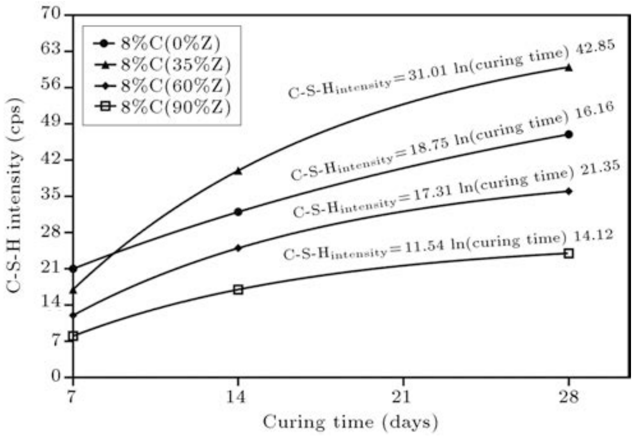


Figure 12. The relationship between the peak intensity of C-S-H phase and curing time.

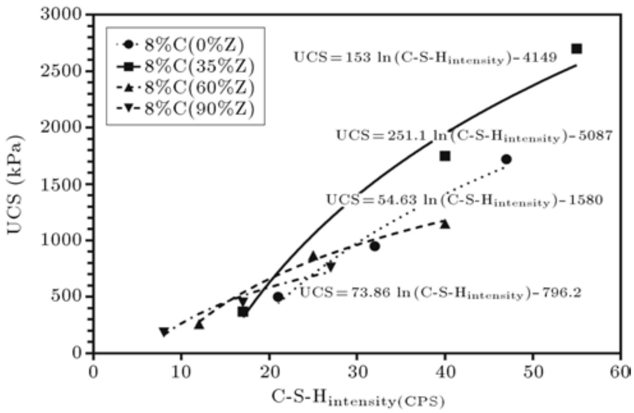


Figure 13. The relationship between the intensity of C-S-H phase in the matrix and the compressive strength for the samples with 0, 35, 60, and 90% of zeolite replacement.

4. Conclusion

In this research, the impact of zeolite on the compressive strength development of cement-treated sand mixtures was investigated through microstructure analysis. Also, the connection between the microstructure and macrostructure was considered, and achieved a suitable relation between compressive strength and the intensity of the C-S-H phase. Specimens were composed of Portland cement type II, natural Clinoptilolite zeolite and Babolsar sand. The amount of cement was selected as 8%, based on the dry weight of sand, and replaced with different percentages of zeolite. Since microstructural analyses are crucial to comprehensive assessment of strength development in zeolite-cement-sand mixtures, these analyses were accomplished and results presented. Among the cement matrix, sand particles, and ITZ, the latter is the weakest part of the mixture. Therefore, strengthening the ITZ as the bridge between sand particles and the cement matrix is a key component in developing the compressive strength of the mixture. Based on presented results, strong interfacial adhesion in the ITZ and development of dense-compact morphology in the presence of 35% zeolite promotes UCS. Further increase in zeolite replacement has a negative effect on mixture strength, decreasing the cement content and free lime in the hydration product. In fact, extra zeolite reduced compressive strength due to remaining excessive Si weakening adhesion, which formed between the cement matrix and sand in the ITZ. On the other hand, extra zeolite could decrease the crystallization speed of hydrates and the intensity of the C-S-H phase. As a result, weak interfacial adhesion was formed between the cement matrix and sand in the ITZ. This research demonstrated evidence of the formation of new phases in the presence of zeolite and its impact on the compressive strength, which led to strength increases. In addition, the connection between the intensity of the produced C-S-H phase and compressive strength was established via a logarithmic equation. As shown, the formation of new phases in the presence of zeolite and its impact on the compressive strength of the mixture was proven by a good relation. Therefore, the impact seen at the lab could be replicated in a full structure. Improvement of soil can easily be applied using zeolite as a suitable alternative to cement in future construction, especially in coastline applications.

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