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

1Saied Izadpanah, *1,2 Issa Shooshpasha, 1Alborz Hajianni

1Department of Civil Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran
2Faculty of Civil Engineering, Babol Noshirvani University of Technology

Abstract

The main objective of this research was to study the impact of zeolite on mineralogical changes led to the development of compressive strength of cemented-sand mixtures. The mixtures consisted of Portland cement type II, natural zeolite (Clinoptilolite) and Babolsar sand. The cement content was 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 percent zeolite along with the amount of the 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, 90 days). The microstructural properties were analyzed using X-Ray Diffractometer (XRD) tests and Scanning Electron Microscope (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 Interface 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 was clearly shown suitable relations between compressive strength and the intensity of the C-S-H phase.

Keywords: Microstructure, Compressive Strength, Interfacial Transition Zone, Cement, Zeolite.

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 was 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]. 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 the geology [6,7]. It should be mentioned that 7% of the \( \text{CO}_2 \) emissions devoted to the world cement industry [6,8]. Among the various pozzolanic fillers available in place of cement, zeolite is a suitable substitute for cement because of its improved performance and environmentally friendly treatment [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 the 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 the large surface area, the molecular structure of the sieve type and the high absorption capacity cause to unique physical and chemical properties compared to other pozzolanic materials which have also been studied by geotechnical engineers. Aksoy [15] has shown that the shear strength of zeolite is significantly high.

The main aim of this research was 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 \( \text{C}-\text{S}-\text{H} \) gel and the compressive strength.

Several studies have been carried out to demonstrate the excellent physical and chemical properties of zeolites, including zeolite-sand, 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 the oil-in-water emulsion. Their findings showed the remarkable removal of cationic surfactant molecules both from the continuous phase and from the oil-water interface through the addition of zeolite particles. Joanna and Kazimierz [17] investigated the mixture particularly in the vicinity of old landfills. Foo and Hameed [18] have 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 depending 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 the base layer, slope of the protected earth dams and as a support layer for flat foundations [31–35]. The cement content, porosity and moisture are the 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 the UCS results. Salamatpour et al. [41] have studied 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 deviating stress (q_{max}) of the grouted sand samples increased.
In previous studies, however, the relationship between microstructure analyzes such as XRD and SEM tests, which were equipped with an EDX measurement according to the strength development behavior of zeolite-cement-sand mixtures, was neglected. Since the microstructural properties play a key role in the assessment of the strength development in cemented soil mixtures, the aforementioned tests were taken into account in this investigation together with an unconfined compression test in order to reveal the microstructural effectiveness of zeolite-cement-sand mixtures in different curing times and zeolite Percentages. Due to the small size of the ITZ; the weakest part of the mixture acts as a bridge between 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 analyzes 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 analyzes are consistent with unconfined compression test results.

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

2. Experimental Program

The plan of experimental program was scheduled as follow:

12 unconfined compression tests, 13 SEM tests and 4 SEM tests equipped with EDX system and 4 XRD tests were performed to assess the relation between microstructural properties and compressive strength of zeolite-cement-sand mixtures.

2.1 Materials

The materials were used in this research, containing of Sand, Portland cement type II and natural Clinoptilolite Zeolite. Sand belonged to Persian coastline located in Babolsar sea side zone and cement was supplied from Neka cement companies in north of Iran. Clinoptilolite was exploited from the mines of Semnan province of Iran.
As it can be seen from Figure 1, the component analysis of natural zeolite was performed by EDX and also chemical analyses of sand and cement have made by means of an ARL 8680+ X-Ray Fluorescence spectrometry (XRF) as shown in Table 1. Also physical analysis of zeolite and cement are shown in Tables 2, 3 respectively. As it is evident in Table 1, the amount of free lime (CaO) of cement composition is very high in comparisons to zeolite. This composition participates to pozzolanic reaction with zeolite.

(Fig.1)
Table 1
Table 2
Table 3

The Scanning Electron Microscope (SEM) images of zeolite, cement powder and sand with sub-rounded particles and grain size distribution of sand are shown in Figures 2, 3 respectively. As is evident in Figure 3, Babolsar sand was classified as poorly graded sand (SP) according to the Unified Soil Classification System (USCS). Mean grain size of the sand ($D_{50}$) is 0.22 mm, 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.

(Fig.2)
(Fig.3)

2.2 Preparation of Specimens

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

Mechanical behavior of zeolite-cement-sand mixtures was accomplished by measuring unconfined compressive strength of samples in accordance with the ASTM D2166 test method with 7, 28 and 90 days of curing time [47]. Unconfined compression machine has load ring of 3 KN and 5 KN capacities and carried out at a constant strain rate of 0.3 mm/min due to brittleness behavior of cemented sand mixtures. Due to repeatability of test result response, three identical specimens for each mixture were tested. The average value of obtained results was used in reports. Amount of water and dry density of the mixture were adapted based on dry weight of materials mixed together. The standard proctor compaction method was repeated on three identical specimens for each mixture. 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 Philips PW 3710 diffractometer [42]. XRD diagrams were obtained using a Cu Kα radiation with a voltage of 40kV and current settings of 30 mA. All specimens were oven-dried (80c, 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. Mineralogy of zeolite-cement-sand mixtures, were investigated on scanning electron microscope (LEO 435VP) by secondary electron image method. EDX analysis was performed with backscattered method in 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 is illustrated the impact of zeolite on optimal water content and maximum dry unit weight of sandy soil in. Larger specific surface of zeolite in comparison with cement led to water retaining properties and occupying pores between sand and cement matrix. As it can be seen in Figure 4a, the optimal water content of the mixtures was decreased by increasing zeolite in all rates. Maximum dry density of sandy soil increases as a result of increasing zeolite replacement, obviously shown in Figure 4b. In fact, fine particles of zeolite within sand and cement matrix was occupied pores and produced dense structure leading to increase the maximum dry density.

(Fig.4)
3.2. Uniaxial Compression test

Figure 5 is shown the impact of zeolite replacements on compressive strength in different days curing. As it is evident in Figure 5, zeolite addition has a noticeable impact on development of compressive strength of samples especially the most efficient of strength magnitude was obtained for 35% replacement in 90 days curing time. Since zeolite particles are finer than cement, hence they act as filler, occupy pores between sand, and cement matrix. Therefore, replacement by up to 35% led to reduce amount of total pores and developing compressive strength of mixture. Obviously, adhesion forces of sand particles to cement matrix have a twofold nature. On one hand, these are physical forces whose magnitude depends on the topography of 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 interface in presence of zeolite. Increasing zeolite more than 35% in cement treated sand mixture, has negative impact on mixture strength and reduce the UCS. This phenomenon was explained by microstructure analyses and vivid the connection between micro and macro evidence was demonstrated in section 3.3 & 3.4 respectively.

(Fig.5)

3.3. Microstructural analyses

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

(Fig.6)

Ca (OH)₂ phase has been liberated from hydration of cement and water. Immediately after reacting cement with water, primary ettringite needle-like was formed. Ettringite, found in this benign state as large needle-like crystals, should not be interpreted as causing the expansion of deteriorating mixture. Increasing zeolite
replacement by up to 35% led to reduce the intensity of calcium hydroxide and ettringite phase. C-S-H peak has broad and diffuse shape and formation of C-S-H phase was raised due to 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 cement matrix and sand. Hence, C-S-H gel ameliorates the ITZ. Replacing beyond of 35% reduces growth rate of calcium hydroxide crystal. Reduction of formed calcium hydroxide causes to lessen pozzolanic reaction and crystal-compact C-S-H phase.

3.3.1. Scanning Electron Microscope test and Energy Dispersive X-ray analysis

The SEM tests were accomplished to investigate 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 is shown the images of zeolite-free mixture and with 35% zeolite replacement at 28 days of curing time. In absence of zeolite, discernible micro cracks are visible while propagation and thickness of cracks reduced for the one with 35% zeolite.

(Fig.7)

Figure 8 is depicted the morphology of mixtures with zeolite and zeolite-free at 90 days of curing time. Many pores are shown in Figure 8a, and hydration product mostly formed as calcium hydroxide Ca (OH)₂ hexagonal plate shape which was released from hydration of cement and water are evident in absence of zeolite. As shown in Figure 8b for the one with 35% zeolite replacement, dense-compact C-S-H gel occupies the pores between 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 presence of zeolite, large flat plate Ca (OH)₂ was decomposed to C-S-H phase with smaller sizes and enhanced the chemical bond between cement matrix and sand particles. In contrast with Figure 8b, this dense-compact C-S-H phase was changed with a delayed formation of ettringite needle-like as it is shown in Figures 8c, 8d. This delayed needle-like crystallization phase of ettringite was agglomerated matrix of mixture and led to induce tensile stress and crack development in matrix of mixture, which is undesirable. Consequently, weak adhesion was formed between cement matrix and sand in the ITZ. Strong interfacial adhesion in the ITZ and development of dense-compact morphology promotes UCS in presence of 35% zeolite.

(Fig.8)
Figure 9 is shown the structure of Ca (OH) \(_2\) with oriented needle-like hexagonal plate shape crystals which is perpendicular to the surface of sand grains for specimen with zero percent zeolite at 90 days curing.

(Fig.9)

Figure 10 is depicted the effect of curing time on specimens in at ages of 7, 28 and 90 days. Here, Figures 10a, 10c and 10e are represented for zeolite-free specimens. As is evident in Figure 10a, 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 sand grains (also see Figure 8a and Figure 9) which results in the formation of a highly porous structure as well as large ITZ between sand and cement matrix. Figure 10c shows the start of the formation of C-S-H phase at 28 days, and the ITZ between sand and cement matrix become smaller than Figure10a. A gradual growth of C-S-H gel due to chemical process of Ca (OH) \(_2\) which is obvious in Figure 10e for specimen at 90 days of curing time. In contrast to Figure10a, Ca (OH) \(_2\) crystals were strongly produced in Figure 10b. Specimen with 35% zeolite replacement at 28 days is depicted in Figure 10d. Here, C-S-H formations are more than Figure 10b and pores in the ITZ become smaller. As is evident shown in Figure 10f, dense and compact structure of specimen was formed at 90 days of curing time. Vast distribution of compacted C-S-H gel led to cover pores and finally form strong interfacial adhesion in the ITZ. In comparison with Figure 10e for specimen with zero percent zeolite and the same curing time, more accumulation of C-S-H gel was produced and covered the ITZ space showed in Figure 10f. One could conclude that strengthening the ITZ is key component to develop compressive strength of 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 produce new cracks developing with increasing stress. Reaching developed cracks under stress concentration to ITZ cause to a continuous crack and consequently failure of mixture. In contrast with Ca (OH) \(_2\), C-S-H gels enhance interfacial adhesion in the ITZ and also develop compressive strength of mixture.

(Fig.10)

90-day hardened cement-sand mixture with zeolite and zeolite-free were scanned with backscattered method by T-SCAN equipped with EDX system.

As is evident shown in Figure 11, Atomic compositions of point EDX were represented. From Figure 11a, as it can be seen analysis spectrum of zeolite-cement-sand mixture in specimen with zeolite-free 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 zeolite replacement more Si than Ca are observed in Figures 11c and 11d. The analyses prone which existence of enough zeolite to complete pozzolanic reactions could enhance compressive strength of mixture while extra zeolite remains excessive Si and lessen compressive strength.

(Fig.11)

3.3.2. 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 it is evident in Figure 12 the intensive peaks of C-S-H phase is shown a logarithmic behavior along of curing time.

(Fig.12)

Figure13 is shown the connection between compressive strength and intensive of C-S-H phase. Increasing compressive strength with intensity of C-S-H phase, along in different zeolite replacement proven a good relation. In the other words, since the growth rate of C-S-H phase increases with enhancing the zeolite replacement by up to 35% has noticeable impact on compressive strength. This connection was demonstrated a logarithmic behavior between aforementioned parameters. The XRD peak intensities identified as "C-S-H" illustrate logarithmic behavior along of curing time in blended samples witch were exploited from the integral values.

(Fig.13)

4. Conclusion

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

Acknowledgement
The authors gratefully acknowledge the RAZI metallurgical research center with ISO/IEC 17025 qualification confirmation certificate from NACI (national qualification confirmation center of Iran), Islamic Azad university of Iran, NajafAbad branch and Kohan Khak Parsian consulting engineers for utilizing laboratory facilities as non-financial aids without receiving any funds.

Compliance with ethical standards
In case of Funding, this research have not received any fund.

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Biographies

Saied Izadpanah is a PhD graduate in the Department of Civil Engineering in Najafabad Azad University. He received his BSc and MSc Degrees at Gorgan and Arak Azad University branches, respectively. He is the head of Kohan Khak Parsian consulting engineers. His research interest are mainly in the area of soil improvement technics, deep excavations and Non-destructive testing methods.

Issa Shooshpasha is an Associate Professor in the Faculty of Civil Engineering in Babol Noshirvani University of Technology. He received his BSc degree in Tabriz University in 1987, his MSc and PhD degrees at McGill University in 1993 and 1996, respectively. His research interests are mainly in the area of bearing capacity of shallow and deep foundations, slope stability, liquefaction, and soil improvement.

Alborz Hajiannia is an Assistant Professor in the Department of Civil Engineering in Najafabad Azad University. He received his MSc and PhD at Amirkabir University. His research interests are mainly in the area of soil bearing capacity, Investigation of granular soil improvement.
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Table 1. Chemical analysis of cement and zeolite

<table>
<thead>
<tr>
<th>Chemical composition</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>K₂O</th>
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<th>MgO</th>
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<td>Cement</td>
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Table 2. Physical properties of Portland cement [48]

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<td>Initial setting time (min)</td>
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Table 3. Physical properties of zeolite [48]

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<td>Water absorption</td>
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<td>Specific gravity (Gs)</td>
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