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ORIGINAL PAPER

Effect of Natural Zeolite and Cement Additive on the Strength of Sand

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Abstract It is widely known and well emphasized that the cemented sand is one of economic and environmental topics in soil stabilization. In some instances, a blend of sand, cement and other materials such as fiber, glass, nano particle and zeolite can commercially be available and effectively used in soil stabilization especially in road construction. In regard to zeolite, its influence and effectiveness on the properties of cemented sands systems has not been completely explored. Hence, in this study, based on an experimental program, it has been tried to investigate the potential of a zeolite stabilizer known as additive material to improve the properties of cemented sands. A total number of 216 unconfined compression tests were carried out on cured samples in 7, 28 and 90 days. Results show unconfined compression strength and failure properties improvements of cement sand specimens when cement replaced by zeolite at optimum proportions of 30 % after 28 days due to pozzolanic reaction. The rate of strength improvement is approximately 20–78 and 20–60 % for 28 and 90 days curing times respectively. The efficiency of using zeolite has been enhanced by increasing the cement content and porosity of the compacted mixture. The replacement of cement by natural zeolite led to an increase of the pH after 14 days. Chemical oxygen demand (COD) tests demonstrate that the materials with the zeolite mixture reveal stronger adsorptive capacity of COD in compare to cemented mixture. Scanning electron microscope images show that adding zeolite in cemented sand changes the microstructure (filling large porosity and pozzolanic reaction) that results in increasing strength.

Keywords Zeolite · Cemented sand · Strength · Unconfined compression · Microstructures

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1 Introduction

Soil stabilization with cement has for many years been a ground improvement approach in geotechnical engineering. Using cemented soil is a versatile and reliable technique among others to increase shear strength parameters. The major advantages of cemented soils are avoiding borrowing materials from elsewhere, economy, simple and rapid performances of them. The cemented technique is particularly suited for stabilization of problematic soils such as loose sand. Cementation of sand outcomes in increasing brittle behavior of the material.

The unconfined compression test is one of the major and rapid laboratory tests in order to evaluate the effectiveness of the stabilization with cement or other additives. The compressive strength of artificially cemented soils has been studied in the past by several investigators (e.g. Clough et al. 1981; Coop and Atkinson 1993; Huang and Airey 1998; Consoli et al. 2000, 2006, 2007, 2009, 2013a, 2014; Thomé et al. 2005; Dalla Rosa et al. 2008; Horpibulsuk et al. 2014; Yilmaz et al. 2015).

A number of studies have been done to assess the mechanical behavior and compression strength increase of cemented sands using additive fiber, glass, fly ash, silica fume and nano particle (Choobasti et al. 2015; Arabani et al. 2015; Pino and Baudet 2015; Consoli et al. 1998, 2013b). Using these additive materials may reduce the cost and provide less brittle behaviours. However, there has been a little effort devoted to the research on the use of pozzolans such as natural zeolite as an additive material to the cemented sands. Natural zeolite, an extender, has been investigated for using as cement and concrete improver by some researchers (Poon et al. 1999; Perraki et al. 2003). Natural zeolite contains large quantities of reactive SiO_2 and Al_2O_3 (Poon et al. 1999). Similar to other pozzolanic materials, zeolite substitution can improve the strength of cement by pozzolanic reaction with $Ca(OH)_2$ and prevent undesirable expansion due to alkali-aggregate reaction. Moreover, zeolite can reduce the porosity of the blended cement paste and improve the interfacial microstructure properties between the blended cement paste (Feng et al. 1990; Poon et al. 1999; Canpolat et al. 2004). Poon et al. (1999) stated that pozzolanic activity of natural zeolite is higher than fly ash but lower than silica fume. Yilmaz et al. (2007) concluded that the clinoptilolite blend decreases the specific gravity of cements.

This study aims to quantify the influence of the amount of zeolite, cement, porosity and curing time on the strength parameters of artificially cemented sandy soils via unconfined compression tests. In this paper, first experimental program is presented and discussed in details. Then the chemical, environmental and microstructure properties of zeolite cemented sand mixture are described.

Table 1 Physical characteristics of the sand soil

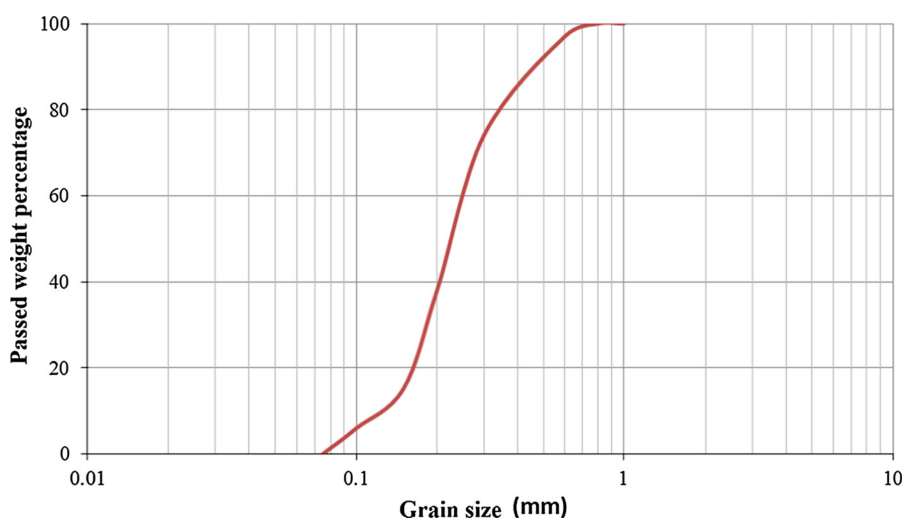
Parameter	Value
Soil name	SP
C_u	1.75
C_c	0.89
$\gamma_{d,max}$ (kN/m^3)	17.7
$\gamma_{d,min}$ (kN/m^3)	14.9
G_s	2.74

2 Experimental Program

2.1 Materials

The base sandy soil used in present study was obtained from Babolsar City located on the southern shorelines of the Caspian Sea. The specifications and gradation of the sand are presented in Table 1 and Fig. 1 respectively.

Fig. 1 Grain size distribution of Babolsar sand



Portland cement type II (according to ASTM C150) was applied in this research. The most important physical characteristics of the cement are presented in Table 2.

The zeolite is of Natural clinoptilolite kind and particles smaller than 75 µm (No. 200 sieve) are referred to as fine aggregates. It's physical and chemical terms are given in Table 3.

2.2 Experimental Program, Sample Preparation and Test Process

Cement content (C), replacement of cement by zeolite (Z), relative density (D_r) and curing time (day) are the variable parameters in the testing program to identify the effect of cement and zeolite additives on sand strength. The variables considered in sample preparation are presented in Table 4. Of note, increasing strength of pozzolanic reaction is time consuming, therefore the curing time of 28 and 90 days are selected which are appropriate times for subsoil improvement.

For the unconfined compression tests, cylindrical specimens, 38 mm in diameter and 76 mm height, were used. Once established a given voids ratio (e), the target dry unit weight (γ_d) was calculated according to Eq. (1).

$$\gamma_d = \frac{G_s \gamma_w}{1 + e} \quad (1)$$

where G_s, a composite specific gravity (due to the specific gravity of the cement grains 3.11 is greater than the specific gravity of the sand and zeolite grains 2.74, 2.2 respectively) based on the zeolite, cement and sand percentages in the specimens. This procedures also used for the precise calculation of void ratio and porosity. Sand, cement and zeolite (based on their relative density and mixture plan as Table 4) were dry mixed uniformly, and then water (10 % of dry weight soil) was added continuously to the soil cement mixture. For the unconfined compression tests, cylindrical specimens, 38 mm in diameter and 76 mm height, are used. The specimens were tamping in three identical layers to reach $D_r = 50$, 70 and 85 % while considering under compaction (Ladd 1978). Additionally, the specimens were wrapped in plastic bags and cured in a humid room at 24 °C and >90 % relative humidity for 7, 28 and 90 days. Total numbers of 216 unconfined compression tests were performed in accordance with ASTM D 2166 (2000).

Table 2 Physical properties of Portland cement

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

Table 3 The physical and chemical properties of zeolite

Parameters	Value
Specific weight (N/m^3)	11,900
Specific surface (m^2/N)	10
Water absorption	60 % volumetric
Cation exchange capacity (N/meq)	26
G_s	2.2

3 Results

The stress–strain curve of specimens stabilized with 4 and 8 % cement and different zeolite substitution cured for 7 and 90 days and $D_r = 85$ % are illustrated in Fig. 2. It is evident that the maximum axial stress increases considerably, due to cement stabilization, and the strain corresponding to peak axial stress decreases. By increasing zeolite replacement of cement, the strain matching to maximum axial stress increases in comparison with cemented samples. In other words, utilizing zeolite in cemented sand increases displacement at failure, and reduce brittle behavior.

As shown in Fig. 2, UCS of zeolite cemented sand mixture increases by curing time. The effect of curing time on maximum UCS is more pronounced for higher cement content. Also, the effect of curing time on maximum UCS of soil cement with 30 % zeolite is more than that of soil cement with other percentage of zeolite. In the other word, the optimum value of zeolite for all cement contents is 30 %. The strain at failure was generally in the range between 0.6 and 2 %. It should be reminded that trend of the stress–strain curves for the different amounts of zeolite cemented sand are similar, apart from the difference in peak stress and strain. Since the main objective of this paper is estimation of the UCS and hence, less attention has been paid to strain and failure types.

Table 4 Description of parameters

Variable	Description of samples
Soil type	Poorly graded sand from Babolsar City (Shores of Caspian Sea)
Cement agent	Portland cement (type II)
Cement contents	2, 4, 6 and 8 % dry unit weight of base soil
Type of zeolite	Natural cilnopiolite zeolite
Zeolite contents (replacement by cement)	0, 10, 30, 50, 70 and 90 % of cement
Relative density (D_r)	50, 70 and 85 %
Water content	10 % weight of base soil
Sample size	38 mm diameter and 76 mm height, compacted in three layers
Curing condition	Cured for 7, 28 and 90 days in humid room

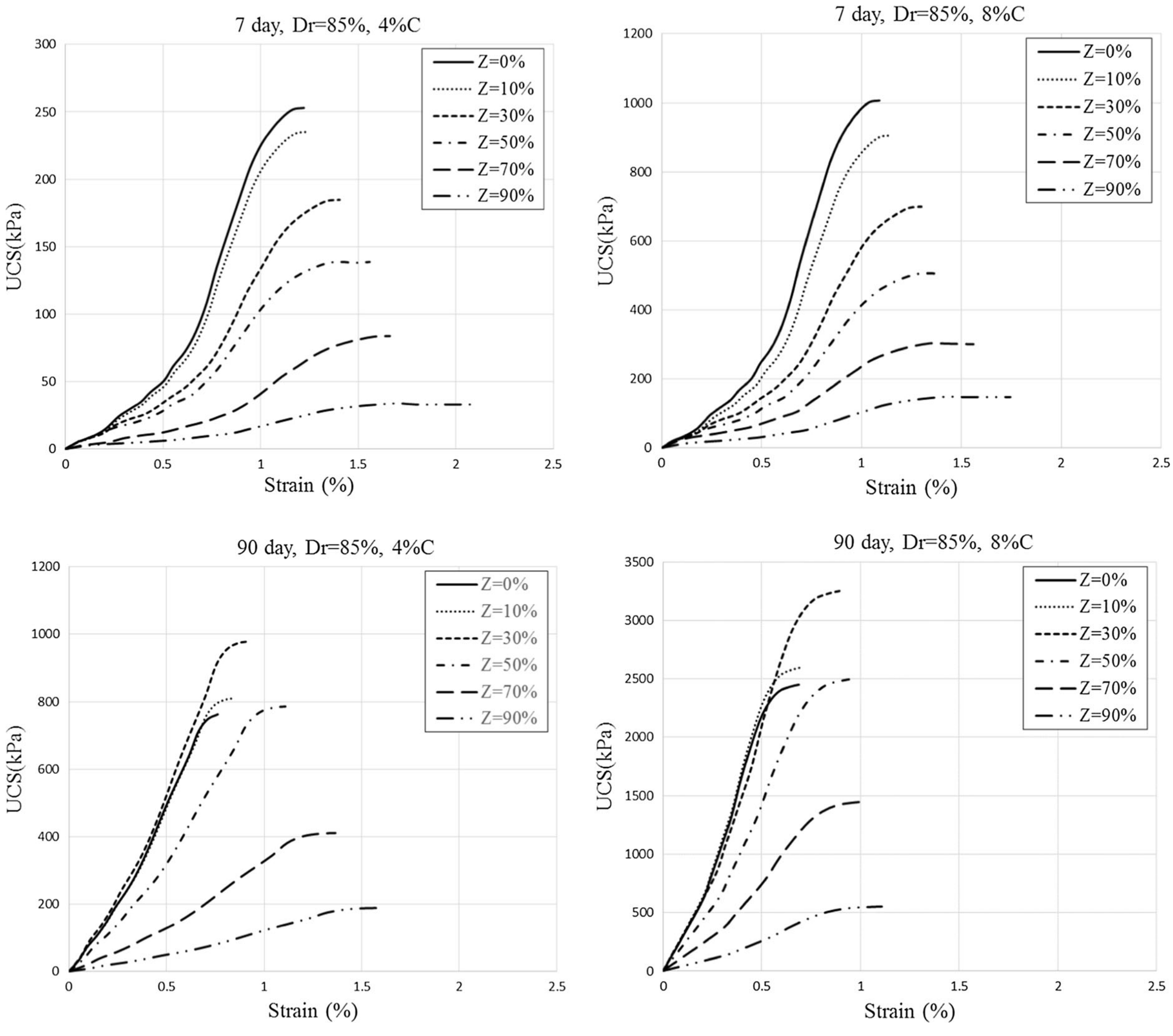


Fig. 2 Stress-strain behavior of zeolite cemented sand

3.1 Cement and Zeolite Content and Relative Density Effects

The UCS results for specimens with different relative density cured for 7, 28 and 90 days are shown in Fig. 3. The figure indicates the effect of additive materials to sand on UCS.

For 7 days cured specimens, UCS decrease with increasing cement replacement by zeolite. This may be attributed to the time consuming pozzolanic reaction that is not complete in 7 days. As shown in Fig. 3, for 28 and 90 days specimens (Shi 2012; Napia et al. 2012), cement replacement by zeolite in the cemented sand (for the whole range of cement studied) causes an increase and decreases in UCS.

To explain differences in the results when using zeolite (up to 30 % and after 30 %) instead of cement for 28 and 90 days of curing time, it is important to point out that the zeolite is formed by amorphous minerals without definable crystalline structure. Chemically, both cement and zeolite are mainly formed of silica and alumina. After 28 days of curing, time-dependent chemical reactions between cement and zeolite particles, namely, pozzolanic reactions,

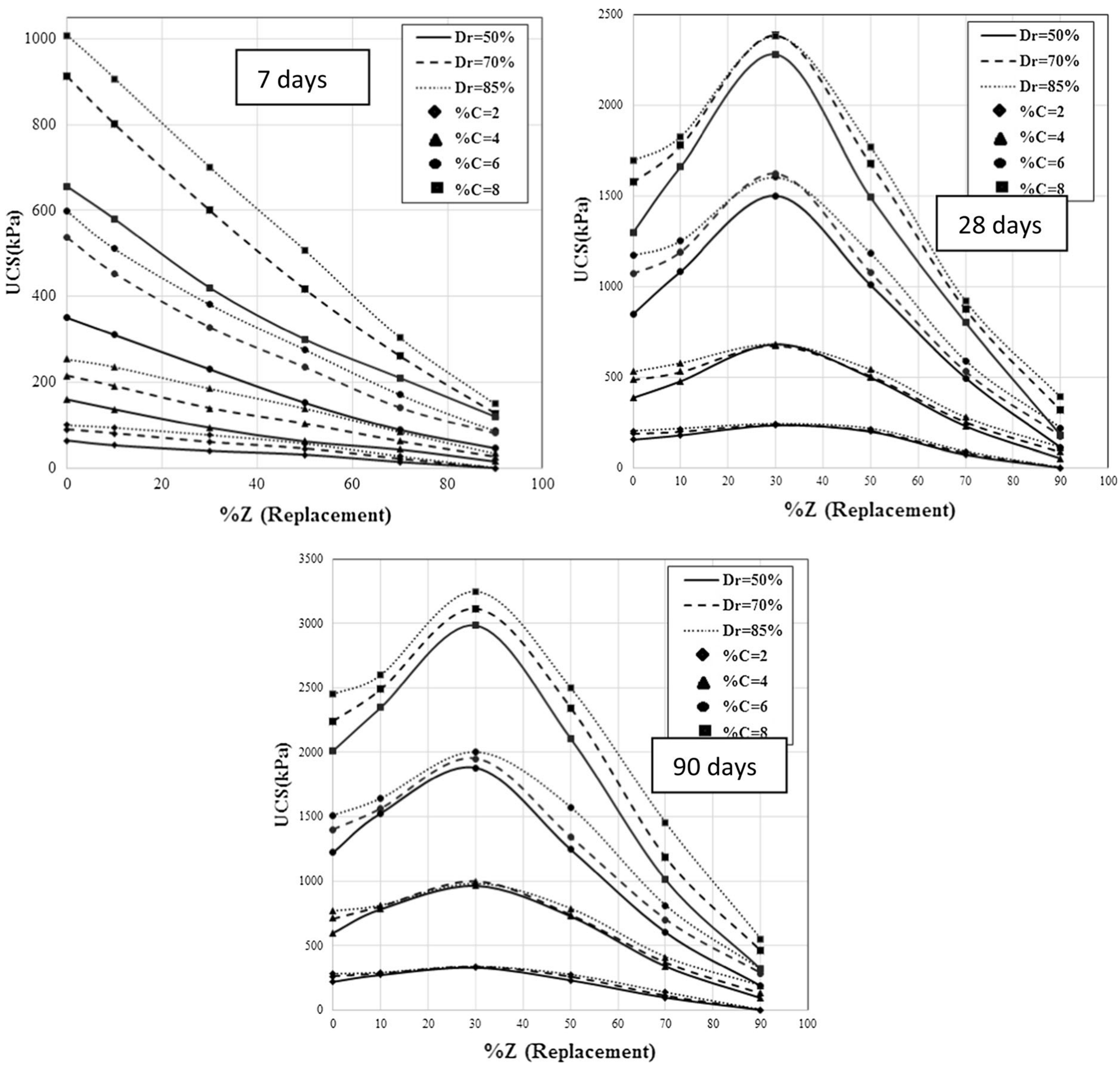


Fig. 3 Effect of additive materials to sand on UCS

have also occurred. Such reactions occur because silica and alumina within the zeolite structure react with water and cement to form calcium silicate hydrate and calcium aluminate hydrate gels, which subsequently crystallize to bind the structure together. Insertion of zeolite into the mixture increases the availability of alumina and silica from amorphous minerals, growing reactions with cement, and consequently increasing strength. Since, more than 30 % the amount of zeolite replaced, calcium silicate hydrate and calcium aluminate hydrate gels reduces because of cement content reduction.

Based on these results, the optimum value of cement replacement by zeolite achieved at 30 %. Therefore, zeolite has a great effect on the strength of zeolite cement sand. Substitution of 30 % is enough to generate a significant gain in strength. The rate of increases in UCS of optimum zeolite cemented sand samples in comparison cemented ones ($[UCS_{zeolite\ cemented\ sand} - UCS_{cemented\ sand}]/UCS_{cemented\ sand}$) is demonstrated in Fig. 4. Strength development for 28 days samples evaluated much more higher than 90 days cured. These rates for higher cement content and lower densities

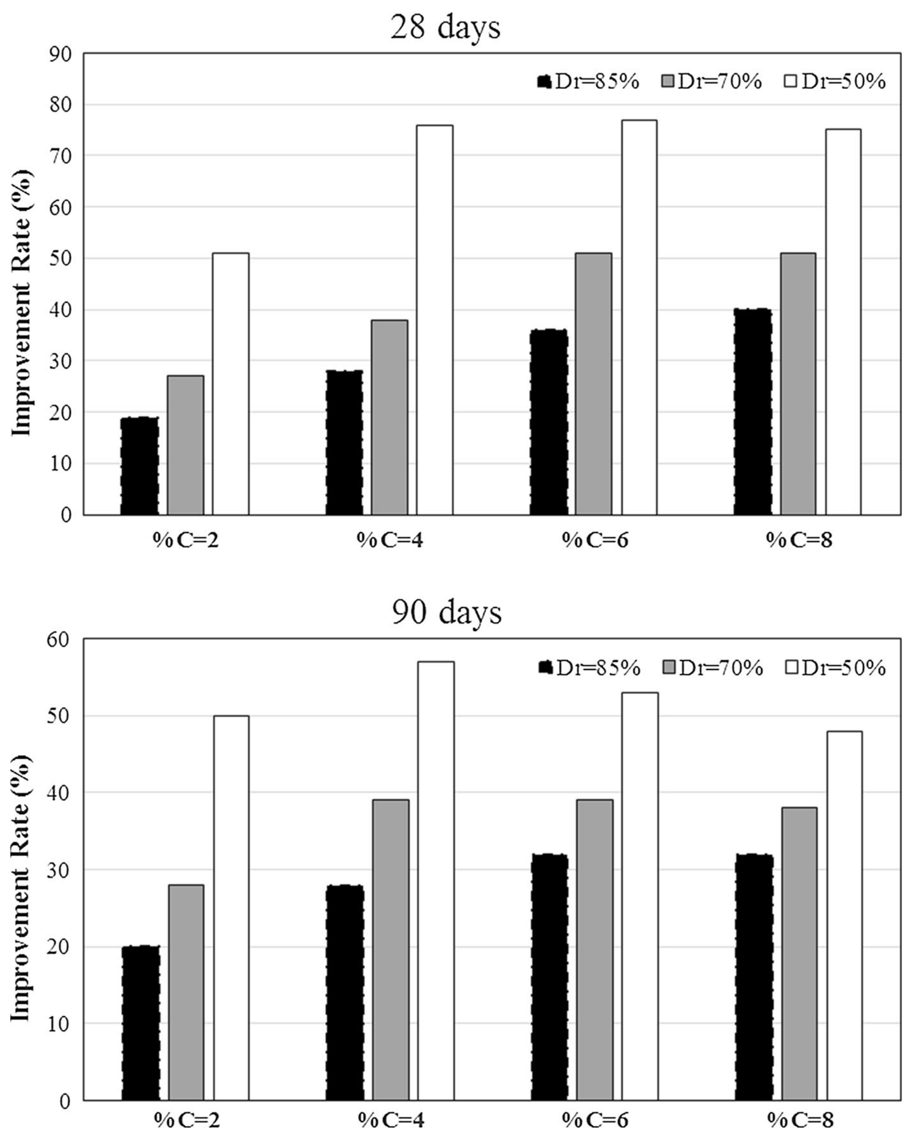


Fig. 4 UCS improvement of cemented sand replaced by optimum value of zeolite

mixtures are grater due to higher amounts of zeolite-cement hydration products in lower density.

3.2 Porosity Effects

Figure 5 shows the effects of porosity, *n*, on the peak strength of zeolite cemented sand (up to 50 % cement replacement).

UCS reduced with the increases in porosity of both zeolite and cement samples. The losses strength rate is more for cemented mixtures and samples that cured in 7 days which for 28 and 90 days curing times of zeolite cemented samples are less. In other words, when cement replaced by optimum zeolite (30 %), the variation of UCS is approximately constant by increasing porosity. Therefore the effectiveness of

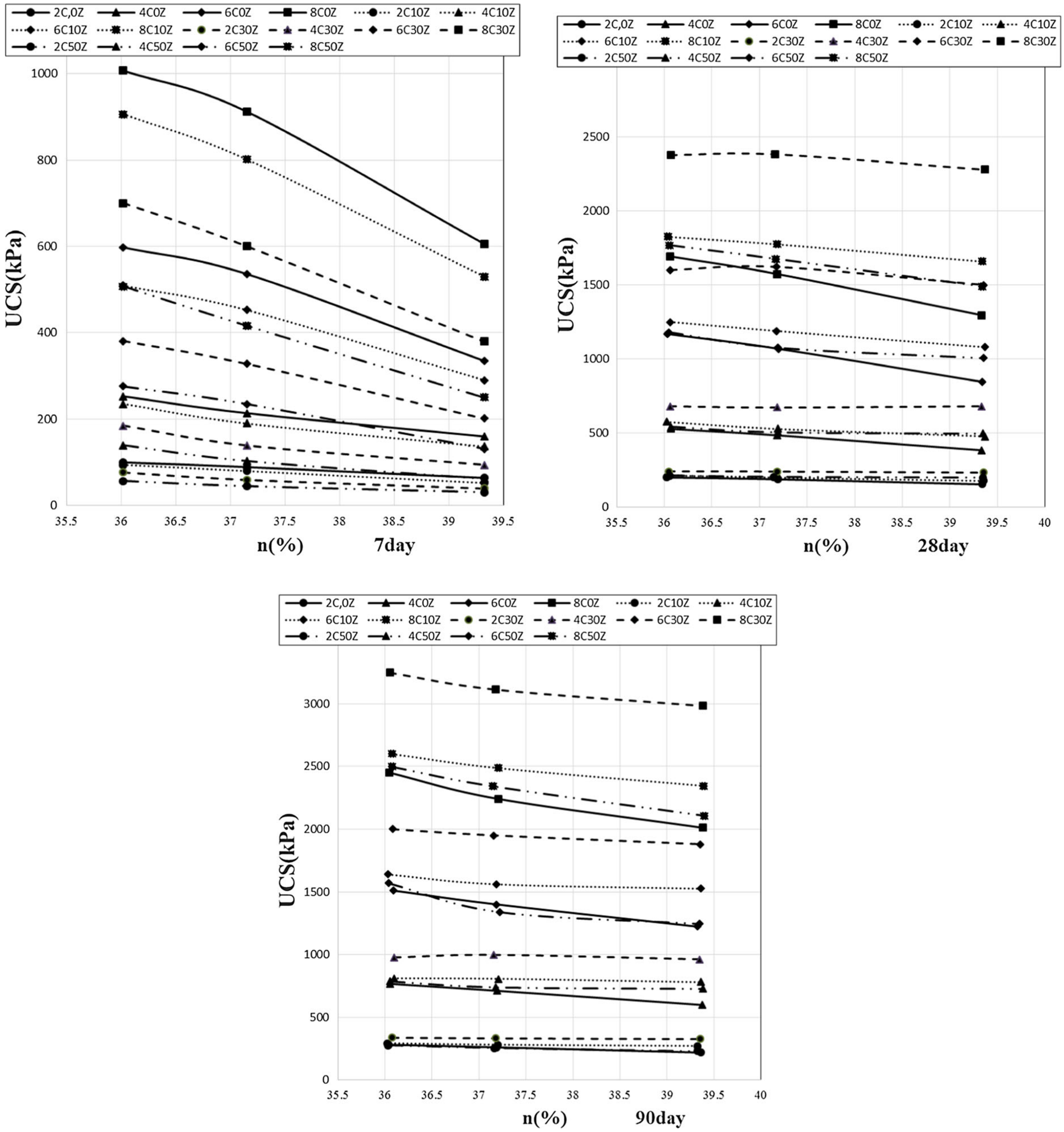


Fig. 5 UCS variations through porosity for cement-zeolite samples for 7, 28 and 90 days

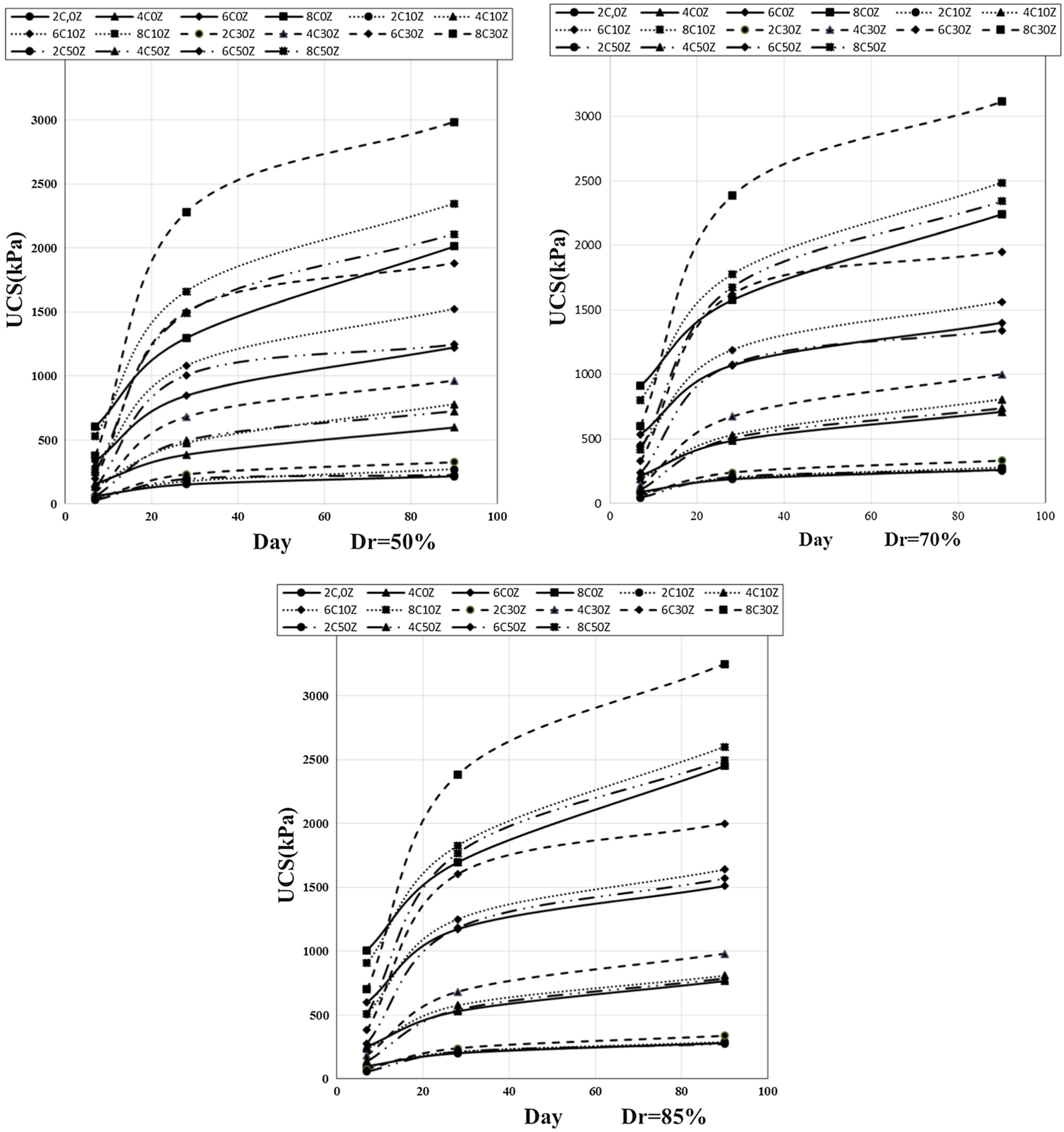


Fig. 6 UCS variations with curing time for cement-zeolite samples with different relative densities

using zeolite instead of cement is more in more porosity blends.

Moreover, the engineer can choose the less amount of cement and the compaction energy to provide a mixture that meets the strength required by the project. Once a poor compaction has been identified, it can be readily taken into account in the design, through this study results, and adopting corrective measures accordingly such as the zeolite of the treated layer or the reduction in the load transmitted. It is important to make clear that the trends observed herein are relevant for the soil, cement and zeolite type and content used

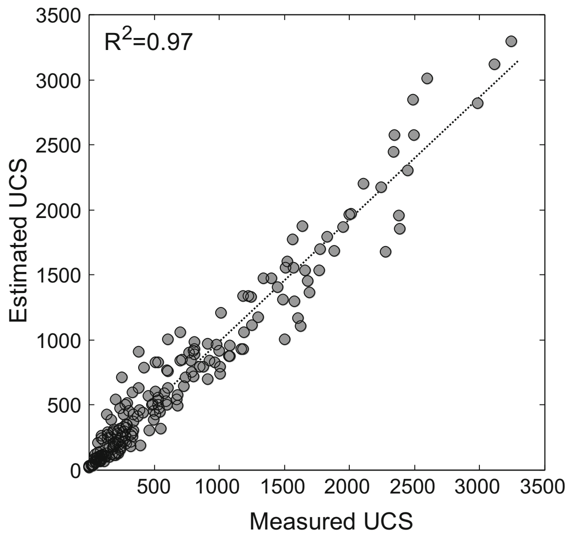


Fig. 7 Comparison between the measured and predicted UCS using Eq. (2)

in the present research and that further studies are necessary to generalize such findings.

3.3 Effect of Curing Time

The variation of curing time affects the UCS of zeolite cemented sand mixtures are presented in Fig. 6 (up to 50 % cement replacement). This figure shows that by increasing curing time, UCS increased with decreasing rate.

3.4 Empirical Correlations

In present study, multiple regression analysis using curing time (t, days), porosity (n), replacement of cement by zeolite (Z) and cement content (C) as input variables was performed to predict UCS (q_u). The developed model is proposed as:

$$q_u = 20.21t^{0.446}n^{-1.78}(533.64 + 362.46Z - 28.34Z^2)C^{1.357} \quad (2)$$

Figure 7 shows scattergram for the estimated UCS from the application of the proposed equation (Eq. 2) and the measured values from the UCS tests. The model shows very good correlation and the proposed equation results in points closely located around the 1:1 line. It is clearly evident that the evolved equation could successfully predict the UCS.

3.5 Scanning Electron Microscope Analysis

Scanning electron microscope (SEM) images of specimens with 8 % cemented sand and 8 % cemenets and with 30 % zeolite replacement after 90 days of curing are shown in Fig. 8. The zeolite cemented sand specimen shows a less open, porous matrix in comparison to cemented sand. Adding zeolite in cemented sand changes the microstructure that result in increasing strength which this could be due to:

(a) Decreasing hydrated $Ca(OH)_2$ of cement past due to pozzelanic reaction,
(b) Compacting the structure of cement paste because of filling large porosity liberated during the hydration of cement,
(c) Leading to the formation of calcium silicate hydrate (C–S–H) gels and aluminates.

3.6 pH Tests

pH tests have been done on zeolite and cement mixtures which the results are shown in Fig. 9. It can be observed that after an hour of mixing materials, pH of cemented samples are higher than zeolite cemented mixture. It can also be seen that by passing time, pH of zeolite cemented samples increased more than cemented samples. Therefore, based on these results, 42 days of curing time is the maximum day to obtain maximum hydration reaction chosen for all mixtures. Such reactions occur due to zeolite and cement minerals.

Portland cement is a system composed of numerous minerals that react with water at different rates, giving hydration products of different composition and crystallinity, and influence the engineering properties of the final product. When a cement–water mixture comes in contact with a zeolite mineral, the alumino silicate framework of the zeolite starts decomposing, under the attack of OH^- in a high pH solution. Depolymerized species, such as $[SiO(OH)_3]^-$ and $[Al(OH)_4]^-$, enter the solution and react with Ca_2^+, forming hydrated calcium silicate and calcium aluminate compounds, very similar to those formed during the hydration of cement (Shi and Day 2000). The pozzolanic activity of zeolites depends on their chemical and

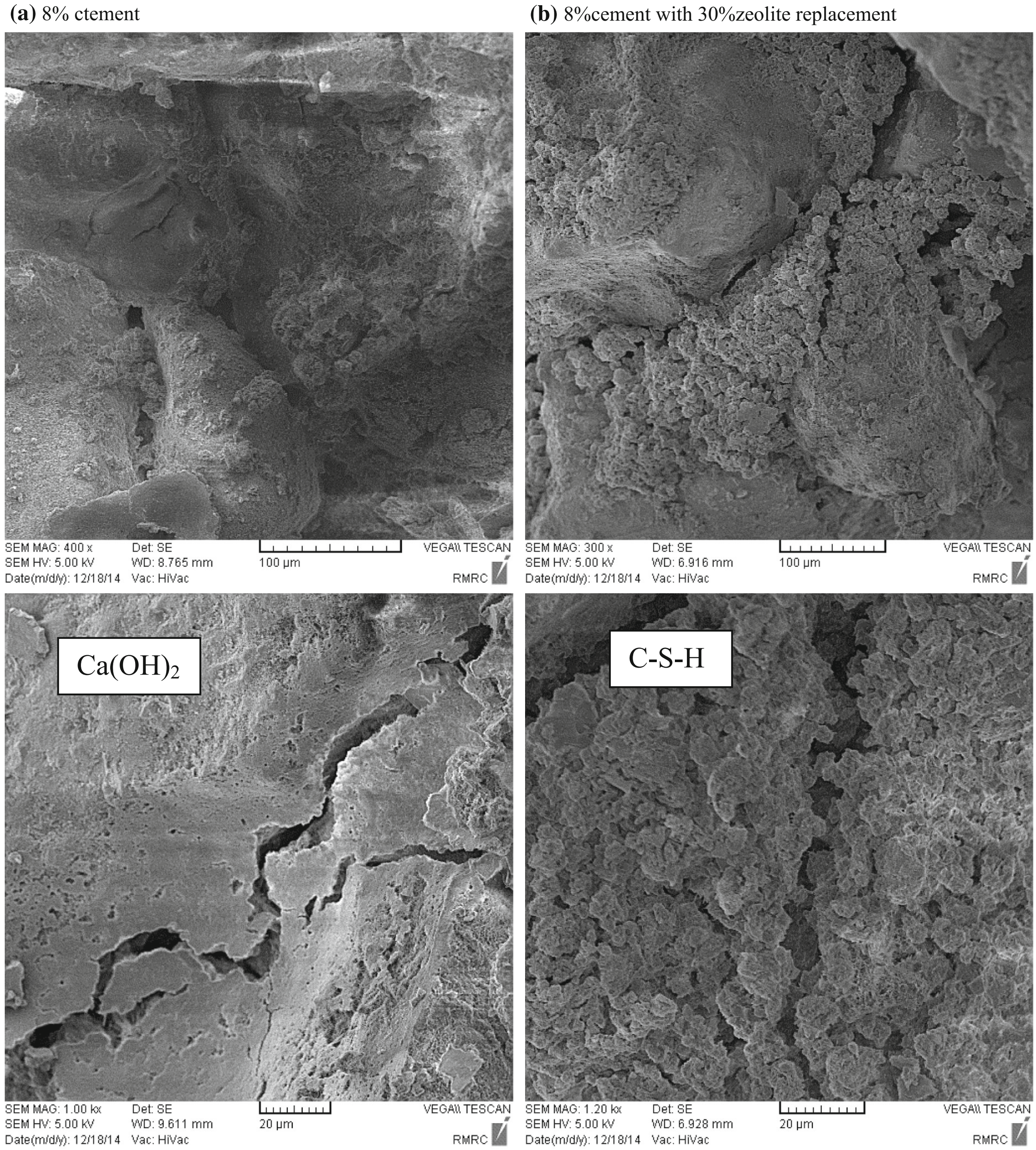


Fig. 8 SEM analysis of zeolite cemented and cemented sand

mineralogical composition. As a result, the microstructure of hardened cement is improved when 30 % cement replaced by zeolite (the optimum value of SiO_2 and Al_2O_3 of zeolite react with the $Ca(OH)_2$ of cement) and becomes more impervious.

3.7 Chemical Oxygen Demand (COD) Test

The chemical oxygen demand (COD) measures the oxygen-depletion capacity of a water sample contaminated with organic waste matter. Specifically, it

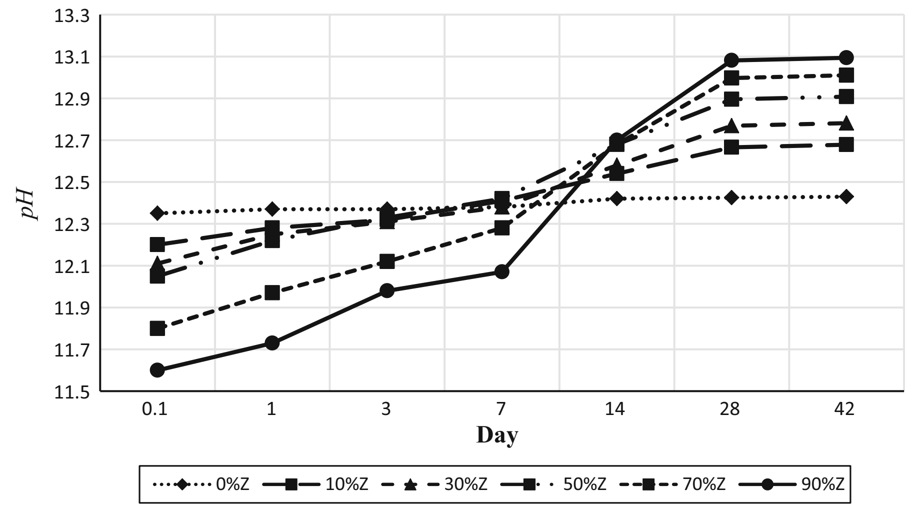
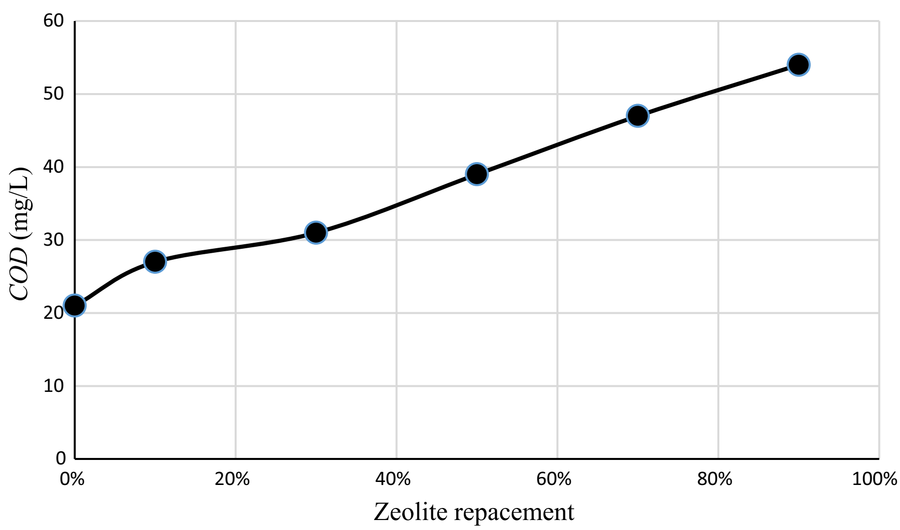


Fig. 9 pH variations with curing time for materials with different zeolite replacement

Fig. 10 COD variation for different zeolite replacement of cement



measures the equivalent amount of oxygen required to chemically oxidize organic compounds in water. COD is used as a general indicator of water quality and is an integral part of all water quality management programs. For environmental studies, it is often calculate the percentage removal of COD to determine the efficiency of the treatment process. The variation of COD of cemented with zeolite replacement has been shown in Fig. 10. As shown in Fig. 10 by increasing zeolite amount in cemented sands, the COD improved. It means that the mixture of zeolite and cement absorbed an abundance of bacteria and organic materials, the bacteria will take in oxygen in order to breakdown these molecules. Bacteria are taking in large amounts of oxygen and will have a detrimental effect on the surrounding ecosystem. On the contrary, when there are high levels of organic waste in the water, there are more bacteria present, the COD will be higher and the dissolved oxygen levels lower (Bhatmagar and Minocha 2006). Therefore, using zeolite in cemented sands results in absorbing bacteria and organic materials to improved environmental aspects.

4 Conclusions

Using zeolite instead of cement causes an increase in unconfined compression strength in the cemented soil (for the whole range of cement studied).

- The addition of cement, even in small amounts, improves greatly the soil strength of zeolite cement and cemented soils. For cement replaced by zeolite samples, UCS increase and decrease by zeolite replacement after 28 days of curing time.
- The effect of curing time on maximum UCS of soil cement with 30 % zeolite is more than that of soil cement with other percentage of zeolite. In the other word, the optimum value of zeolite for all cement contents is 30 % which improve UCS 20–78 and 20–60 % for 28 and 90 days samples respectively
- The rate of strength gain increase by density reduction as well as increase in cement content. It indicates that the effectiveness of the zeolite is larger for higher cemented and less compacted mixtures.
- Decrease in the porosity of the compacted mixture improves greatly the strength for the cemented soils and less for zeolite cement mixtures.
- Materials with the zeolite mixture reveals stronger adsorptive capacity of COD in compare to cemented mixture.
- The replacement of cement by natural zeolite led to an increase of the pH after 14 days.
- Adding zeolite in cemented sand changed the microstructure (filling large porosity and pozzolanic reaction) that result in increasing strength by SEM analysis.

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