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Microstructural and Strength Characteristics of Cement Treated Clay Stabilized with Zeolite for Road Base Application

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Abstract. The stabilization of clay soils with cement has been identified to be uneconomical, and its production is not environmentally friendly. The addition of pozzolanic material to cut down on the use of cement is very pertinent. Clayey soil was stabilized with 0, 2, 4, and 6% cement admixed with 0, 3, 6, 9, 12, and 15% zeolite. Index properties tests were conducted on the clay, while the unconfined compressive strength (UCS) test was carried out on the clay soil and clay treated with the selected composition of cement and zeolite. Tests including X-ray fluorescence (XRF) were conducted on the clay, cement, and zeolite. X-ray diffraction (XRD) and scanning electron microscopy (SEM) tests were conducted on the clay and clay mixed with the selected composition of cement and zeolite. The objective of this study is to investigate the effect of zeolite on the strength and microstructure of cement stabilized clay soil. Index properties result in the group the clay as A-7-6 and clay of high plasticity (CH) based on AASHTO and Unified Soil Classification Systems, respectively. The (UCS) increased from 62kN/m^2 for untreated clay to a maximum of 1552 kN/m^2 for clay treated with 6% cement and 6% zeolite after 60 days of curing. The (XRF) test revealed major oxide compositions of the clay as silica, alumina, and iron; that of cement was quicklime and silica, while that of zeolite is silica, alumina, and sodium. The (XRD) result showed that the clay contained active montmorillonite along with other minerals. The addition of 6% cement disintegrates the montmorillonite minerals to less active kaolinite minerals with the introduction of calcite minerals. The addition of 6% cement and 6% zeolite further converted the montmorillonite mineral to kaolinite minerals with the introduction of calcite and zeolite. The (SEM) result of the clay mixed with cement and zeolite revealed a smooth, compact structure without pores which justifies the highest UCS value recorded in this mixture. 6% zeolite was observed to be the optimal zeolite that gave the highest UCS for each cement addition.

Keywords: Cement, Zeolite; X-ray fluorescence; X-ray diffraction; scanning electron microscopy; stabilization; unconfined

1. Introduction

Some soil deposits in their natural form are suitable for use for road structures whereas others are unsuitable unless it is stabilized or modified. One of such soils is clayey soil which is usually described as problematic soil. The unsuitable soils are usually excavated and replace with better materials or to treat the deficient soil such that they can sustain the applied wheel loads from vehicles. One of the major problematic soils is the expansive soils, which are frequently encountered due to their existence all over the world, except for the arctic regions [1]. The usual swelling characteristics of expansive soils are mainly resulting from their clay mineral content which is mostly montmorillonite. Due to economic reasons, geotechnical engineers often prefer to stabilize the properties of these problematic soils using different stabilization techniques. Stabilization has been commonly used to improve the engineering properties of soils such as strength, durability, compressibility, hydraulic conductivity, and density. Different additives have been employed to stabilize these soils through cementing, waterproofing, bonding or a combination of both [2]. Puppala and Chittoori [3] classified these additives into traditional, non-traditional, and byproduct stabilizers.

The traditional stabilizers are cement, lime, bituminous materials, and fly ash; non-traditional stabilizers include polymers, enzymes, ammonium chloride, while the byproduct stabilizers comprise of cement kiln dust, iron ore tailings, bagasse ash, etc. Stabilization of clay soils has been carried out by many researchers, such as the work of [4], who combined natural pozzolana and lime to stabilize yellow clay soil. It was concluded that natural pozzolana and lime could be successfully used to stabilize yellow clay. Similarly, the study by [5] who treated expansive clay with up to 10% cement kiln dust (CDK) and used UCS and California bearing ratio (CBR) as evaluation criteria. The research recorded maximum UCS and CBR of 357kN/m^2 and 7% respectively at 10% CDK treatment. Gunturi et al [6] treated expansive clay soil with RBI-81 stabilizer to investigate the CBR and free swell index of the clay. Improvement in strength and reduction in the swell index was recorded. Most stabilization additives are usually added in small percentages. The study by [7], however, considered the use of high fraction class F fly ash to stabilize lime-treated clay soil. The results revealed that the addition of high fraction class F fly ash gave extremely high durability. Active sodium montmorillonite clay was stabilized with CDK using Atterberg limit test, pH value, UCS, stiffness, and stability as evaluation criteria [8]. The CDK was observed to improve the plasticity of the treated clay and hence, its workability.

The study by [9] is also a stabilization but using phosphogypsum (PG) and fly ash (FA). The addition of 6% PG and 5% FA gave the highest increase in strength of above 700kN/m^2. Mir [10] worked to evaluate the effect of FA and lime on the physical and mechanical properties of clay soil. Results revealed that FA and lime improved the strength of the mixture tremendously. Chemical stabilization of bentonite using lime and PG was attempted by [11]. The addition of 8% lime and 8% PG reduced the swelling index of the bentonite. The application of stabilization technics to marine clays was studied [12]. Marine clay was stabilized with cement, and a formula to estimate the UCS of cement-treated marine clay was developed. All these researches proved to have enhanced the strength and durability of clay soils as well as their Atterberg limits. The use of cement to stabilize deficient soils was later discovered to be uneconomical, and its production is environmentally unfriendly. The use of other eco-friendly materials to reduce the use of cement for stabilization has been used by researchers.

Zeolite is one such material that contains large quantities of reactive SiO_2 and Al_2O_3 based on XRF results [13]. Similar to other Pozzolanic materials, zeolite substitution can improve the strength of cement stabilized clay by Pozzolanic reaction with $Ca(OH)_2$, prevent undesirable expansion due to alkali-aggregate reaction, reduce the porosity of the blended cement paste, and improve the interfacial microstructure properties between the blended cement paste [14, 13, 15]. Zeolite has unique characteristics such as high specific surface area and cation exchange capacity, as well as the ability to store heat between hydration and dehydration cycles [16]. The effect of natural zeolite on the unconfined compressive strength of cement-sand mixtures was studied [17]. It was revealed that the unconfined compressive strength (UCS) of cement-soil mixtures increased with an increase in zeolite and then decreased after attaining an optimal level of zeolite. The work of Turkuz and Vural [18] deals with the effect of zeolite on the dispersive and swelling characteristics of some clay soils. The author, who used swelling percentage, swelling pressure, pinhole, crumb, and unconfined compressive strength tests as evaluation criteria, also used 3% as a fixed percentage of cement with the varied composition of zeolite.

The author concluded that cement and zeolite improved both the swelling properties and the strength of the clays. A maximum UCS value of 1200kN/m^2 was recorded in soil 2 at 3% cement and 10% zeolite. Stabilization of sandy soils is observed to be best with cement in comparison with other additives. However, its cost and environmentally unfriendliness of its production have called for concerted effort to reduce its usage by adding other environmentally friendly additives.

The study by Mola-Abasi and Shooshpasha [17] was aimed at stabilizing sandy soil with cement replaced with the varied composition of zeolite. The researcher observed that 30% zeolite gave the optimal zeolite required to increase the strength from 20% to 78%. Shi [19] carry out similar research on the use of cement and zeolite for stabilization of gravelly-sandy soil and clayey-silty soils. A maximum UCS value of 1280kN/m2 was recorded for the clayey-silty sandy soil at 10% binder content. A similar study was carried out by Salamatpoor et al. [20], who treated sandy soil with cement and zeolite. The author recorded 40% replacement as an optimum replacement to give effective strength with a strength – improvement ratio of between 128-209%. It is common knowledge that organic substances inhibit the hydration reaction of cement in soils. Ling et al. [21] have attempted to stabilize organic soils using cement and zeolite. The findings of the study revealed that low lime content resulted to lower strength gain while slight replacement of lime with zeolite performed better after a long curing period.

During the early discovery of zeolite as Pozzolanic material, the issues concerning the reaction of its constituents with lime and other stabilization additives were of serious concern to researchers. The work of Mertens et al. [22] is one of the early attempts to investigate the reaction of zeolites with lime and the parameters that affect their reactions. The result of this study revealed that the external surface area of the materials only affects the short-term reactivity while the cation exchange affects both the short and long-term reactivity. The long-term reactivity was influenced more by the Si/Al ratio of the zeolite. A similar study was carried out to investigate the influence of silica/alumina ratio of Pozzolanic zeolites when used as Pozzolanic material [23]. The researcher aimed at investigating the Pozzolanic abilities of isostructural zeolites with different frame work compositions. It was concluded that zeolite structure affects its Pozzolanic activity. This study is therefore aimed at investigating the effect of zeolite on cement-treated clay soil with justification from its microstructure and possible application for flexible pavement structure.

2. Materials and method of experimentation

2.1 Materials

The materials used for this study are clay soil, Portland cement, and zeolite. The clay soil was collected from Bako village along Gwagwalada-Garki road in Federal Capital Territory, Abuja, Nigeria. It was collected at a depth of between 0.8m to 1.5 m using the method of disturbed sampling to avoid organic matter inclusion. The soil sample collected was then wrapped in multiple polythene bags to avoid loss of moisture and was immediately transferred to the Civil Engineering Laboratory of the Federal University of Technology, Minna, Nigeria. The sample was then air-dried, passed through a sieve 2.00 mm, and prepared based on the method highlighted in BS 1377 [24] before usage. The Portland cement was obtained in Gidan Kwanu village and kept in a dry place to avoid moisture attack. The zeolite was also obtained from commercial dealers in Zaria, Kaduna State, Nigeria. The zeolite was also kept in a dry place to avoid moisture infiltration. The distilled water used for this study was obtained from a medical shop opposite General Hospital, Minna, Nigeria. These materials are shown in Figure 1.

Figure 1. Materials (a) clay, (b) cement, (c) zeolite, (d) distilled water, used for this study.

2.2 Method of experimentation

Index properties tests including natural moisture content, specific gravity, grain size analysis, liquid limit, plastic limit, and plasticity index were carried out on the untreated clay. Compaction test was carried out at standard proctor compaction energy level to determine the maximum dry densities (MDD) and optimum moisture content (OMC) of the clay soil, which will be used to mold specimen for unconfined compressive strength (UCS) test. The clay was dry-mixed thoroughly with 0, 2, 4, and 6% cement which in turn is admixed with 0, 3, 6, 9, 12, and 15% zeolite each according to the method highlighted in [25] until both the chemical and clay becomes homogenous. A predetermined amount of water was then added and mixed to homogeneity. The resultant clay-chemical-water mixture was molded into a mold of 38mm diameter and 84mm length for UCS specimen. The specimen was immediately transferred to a thick nylon leather, sealed appropriately, and immersed in a container filled with moist sand to avoid loss of moisture. The UCS test was started after 24 hours to allow enough time for moisture homogeneity.

The specimen was kept in the laboratory at an average room temperature of 36^0C and tested after 1, 7, 14, 28, and 60 days of curing. Each specimen was treated to the diameter of 38mm and length of 76mm and was loaded at a rate of 1.2N/min during testing. After 28 days of testing, untreated clay and clay treated with a sellected amount of chemical additives were crushed into small rubber bottles for XRD and XRF tests while the same samples were cut out and trimmed into a very small mold, and placed in a small rubber tube for SEM and EDS test. (XRD) and (XRF) tests were conducted in Ithemba Laboratory, Somerset West, South Africa, and Electron Microscope Unit, University of Western Cape, Cape Town, South Africa. The oxide composition of the clay, cement, and zeolite is shown in Table 1. Phase characterization of the minerals and estimate of the average crystallite size of the various synthesized materials were conducted on a Bruker AXS D8 XRD system. The predominant oxides contained in the clay are silica, alumina, and iron oxide, while that of cement is quicklime and silica. The Pozzolanic zeolite consists predominantly of silica, alumina, and sodium oxide. Scanning Electron Microscopy (SEM) test was also carried out by placing 0.05 mg of the synthesized materials, sprinkled on a sample holder, covered with carbon adhesive tape, and wire sputter-coated with Au-Pd using Quorum T15OT for 5 minutes prior to analysis. The sputter-coated samples were characterized using Zeiss Auriga HRSEM. The SEM, which visualizes morphology and microstructure of the synthesized products were analyzed using Zeiss Auriga HRSEM.

Table 1. Oxide composition of clay, cement, and zeolite.

Oxide (%)	Fe_2O_3	TiO_2	CaO	K_2O	SiO_2	Al_2O_3	MgO	Na_2O	LOI
Clay	9.61	1.66	2.06	1.35	56.64	19.07	1.38	0.64	7.14
Cement	4.57	0.44	64.44	0.56	21.60	4.13	1.06	0.11	0.76
Zeolite	0.02	0.03	0.01	0.45	38.70	29.53	0.00	20.19	11.57

3. Results and Discussion

3.1 Physical properties of clay soil

The result of some physical properties of the clay soil is shown in Table 2. From the table, the clay contained high composition of clay-sized particles without gravel. It falls under A-7-6 according to AASHTO soil classification system and clay of high plasticity (CH) based on a unified soil classification system. The clay soil is not suitable to be used in the construction of any component of road structure or support any road structure unless it is stabilized to improve its strength and durability. The low value of MDD and high value of OMC recorded is an indication of poor soil for road construction.

Table 2. Physical properties of the clay soil.

Properties	Quantities	Properties	Quantities
Gravel (%)	0.0	Natural moisture content (%)	35.1
Sand (%)	18.4	AASHTO soil classification	A-7-6
Silt (%)	28.6	Unified soil classification	CH
Clay (%)	53.0	Maximum unit weight (kN/m^3)	16.58
Specific gravity	2.66	Optimum moisture cont. (%)	18.8
Liquid limit (%)	58.0	Color	Greyish brown
Plasticity Index (%)	31.4	-	-

3.2 Effect of zeolite on the ucs of specimen

The most suitable and adaptable method of evaluating the strength of stabilized soil is the unconfined compressive strength which is the basic test recommended for the determination of the required amount and proportion of additive to be used in the stabilization of soil [26]. The values of the UCS recorded from the test conducted on the untreated clay and clay mixed with zeolite for specific cement additions are shown in Figure 2. At 0% cement (Figure 2a), The UCS values were observed to increase from 62 kN/m^2 at 0% zeolite continuously with an increase in zeolite addition to the maximum of 285 kN/m^2 at 15% zeolite addition for all curing days. This result is an indication of the reaction between sodium ions, aluminum ions, and silica from the zeolite and clay. In the absence of significant calcium ions in the solution, sodium silicate hydrate (S-S-H) and sodium aluminate hydrate (S-A-H) may form a cementitious compound that may increase the strength of the soil marginally [27,28]. This probably informs the reason for the continuous increase in UCS with an increase in zeolite addition, as shown in Figure 2a. After curing for 60 days, the UCS values increased from 62kN/m^2 at 0% zeolite addition to 290 kN/m^2 at 15% zeolite addition which represents 3.68 times increase in UCS values.

The addition of a low percentage of cement (2%) recorded an increase in UCS values from 0% zeolite addition to maximum UCS value at 6% zeolite addition, after which the values dropped. After 60 days of curing, the UCS increased from 315 kN/m^2 at 0% zeolite to a maximum UCS value of 465 kN/m^{2,} after which the UCS values dropped to 400kN/m^{2,} representing a 47.6% increase in UCS value. This is expected because of the cementitious calcium silicate hydrate (C-S-H) formed on the addition of water to the mixture of clay, cement, and zeolite. The optimal mixture which gave the highest UCS value was observed at 6% zeolite addition which is just sufficient to react with the lime in cement and the calcium hydroxide, which is the byproduct of cement hydration. Beyond 6% zeolite addition, the UCS values were observed to drop due to the low acid buffering capacity exhibited by the higher composition of zeolite because of its inability to provide a suitable pH environment for the pozzolanic reaction [21]. The UCS values were also observed to increase from 359 kN/m^2 after 1 day of curing to 466 kN/m^2 after 60 days of curing which is an indication of the occurrence of Pozzolanic reaction in the mixture. With the addition of 4% and 6% cement, similar trends were observed but with higher UCS values. At 4% cement addition and 60 days curing, the UCS increased from 752 kN/m^2 at 0% zeolite addition to maximum of 1009 kN/m^2 at 9% zeolite addition after which the value dropped to 834 kN/m^2 at 15% zeolite addition which represents 34% strength increase. For 6% cement addition and 60 days curing. However, the UCS increased from 1350 kN/m^2 at 0% zeolite addition to a maximum of 1552 kN/m^2 at

6% zeolite addition, after which the value reduced to 1009 kN/m^2 representing a 15% increase in strength.

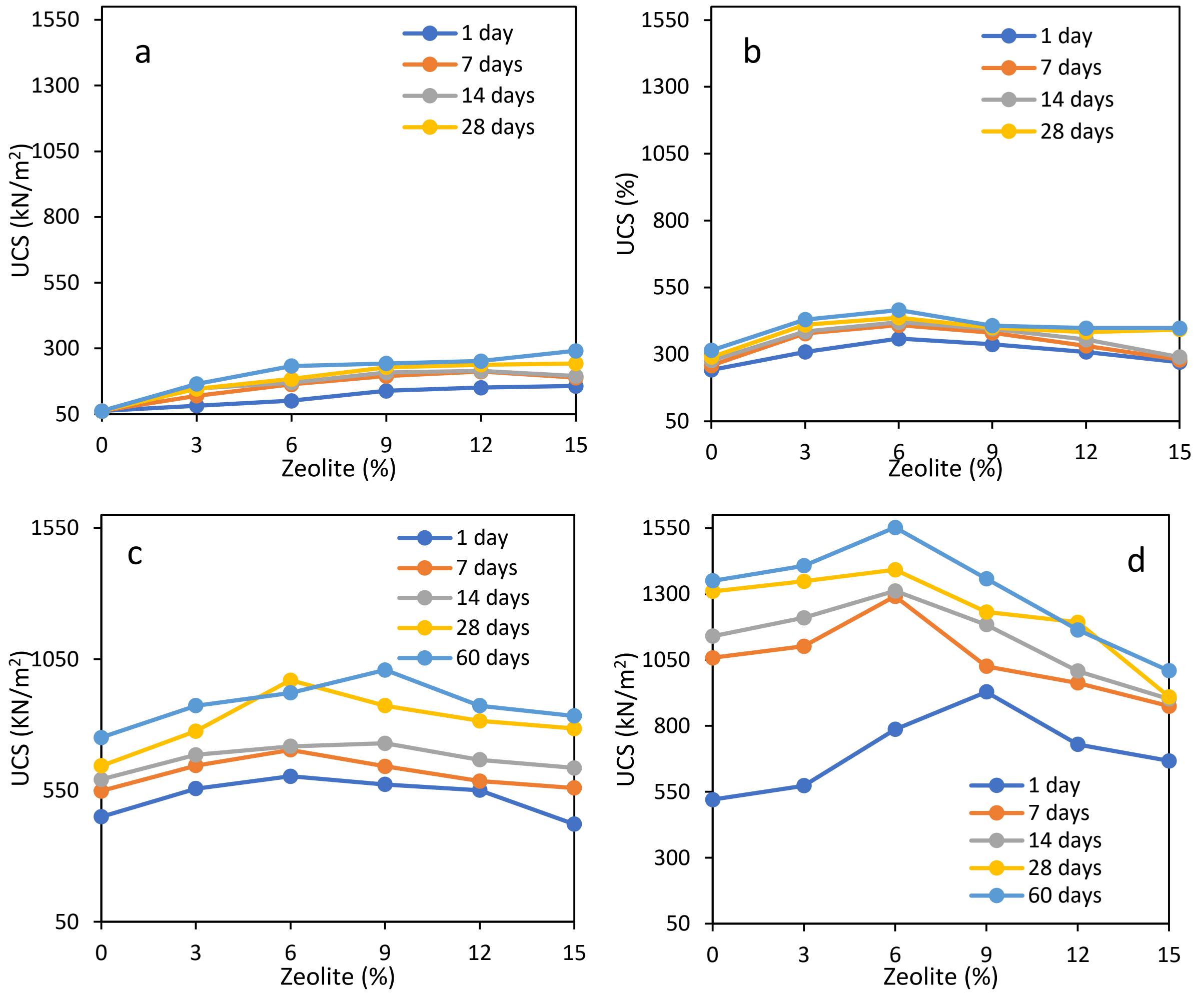

Figure 2. Variation of UCS with varied percent of zeolite at varied curing days for (a) 0% cement, (b) 2% cement, (c) 4% cement (d) 6% cement.

3.3 Effect of zeolite on the stiffness of cement stabilized clay soil

The stress-strain behavior of clay treated with specific cement and zeolite is presented in Figures 3a–3d. The graph of 0% cement addition revealed gradually prolonged failure with higher strain at failure of between 2% and above. At 2% cement addition, the strain at failure was observed to have reduced to between 1.1–1.5% with 0% and 3% zeolite addition having the higher strain values of 1.5% and 6, 9, 12, and 15% zeolite addition having low values of 1.1% strain. These trends probably resulted from the hydration or ion exchange reaction of cement, which formed particle flocculation, thereby converting clay particles to silty and fine sand particles. This will lead to brittleness in the specimen, thus reducing the failure strain. The trend is similar for 4% cement addition, with strain at failure occurring at 0.8%. This trend differs for 6% cement addition, where the strain at failure extended marginally to above 1.25%. This may be attributed to the higher amount of cement and Pozzolanic zeolite, whose reaction is Probably persisting and which must have delayed the agglomeration of clay particles to form brittleness in the mixture. The modulus of elasticity for specimen with 6% zeolite after 28 days of curing is observed to increase from 211 MPa for 0% cement addition through 414 MPa for 2% cement addition to 2850 MPa at 4% cement addition and finally 2857 MPa at 6% cement addition.

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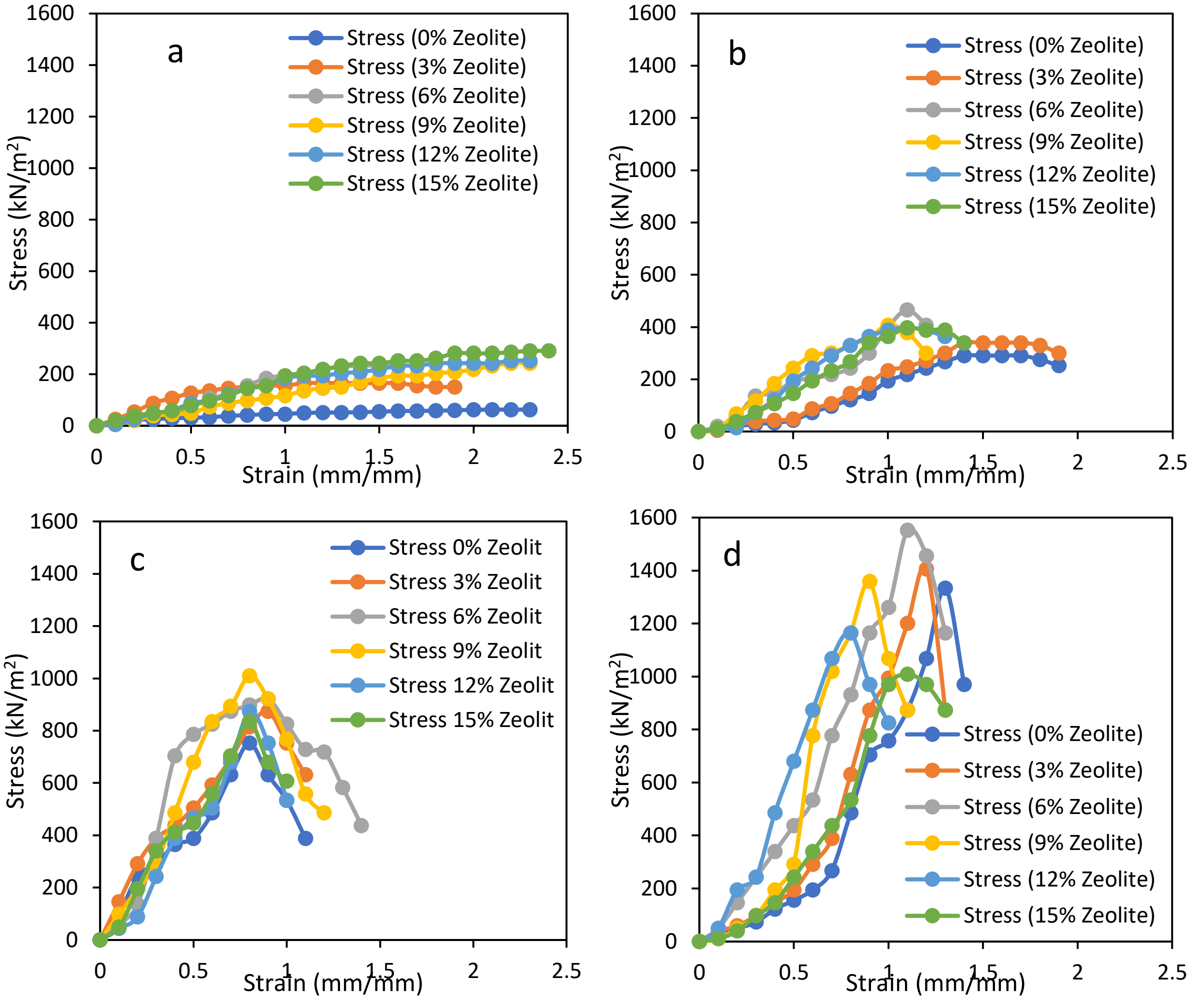

Figure 3. Stress-Strain relation for specimen of varied composition of zeolite for 60 days curing at (a) 0% cement, (b) 2% cement, (c) 4% cement, (d) 6% cement.

4. Microstructural analysis

The clay consists predominantly of silica, alumina, and sodium oxide. Table 2, addition of cement composed of high quick lime content and zeolite containing high composition of silica and alumina to the clay, will result in the formation of both cementitious calcium aluminate hydrate (C-A-H) and calcium silica hydrate (C-S-H). Calcium hydroxide that is formed as a byproduct of cement hydration will continue to react with Pozzolanic zeolite to produce more (C-S-H). The mineralogy tests result in Figure 4 for untreated clay soil and clay soil treated with cement, zeolite, and a combination of cement and zeolite. The untreated clay consists predominantly of albite, montmorillonite, anorthoclase, quartz, ankerite, calcium silicide, and orthoclase, as shown in Figure 4a. The addition of 6% cement disintegrates the montmorillonite mineral in the natural clay to form kaolinite mineral, which is less reactive. This is justified by the disappearance of the montmorillonite peaks recorded in the natural clay at 8.2 and 19.8 (2θ) in Figure 4a. The orthoclase was recorded as microcline; see Figure 4b.

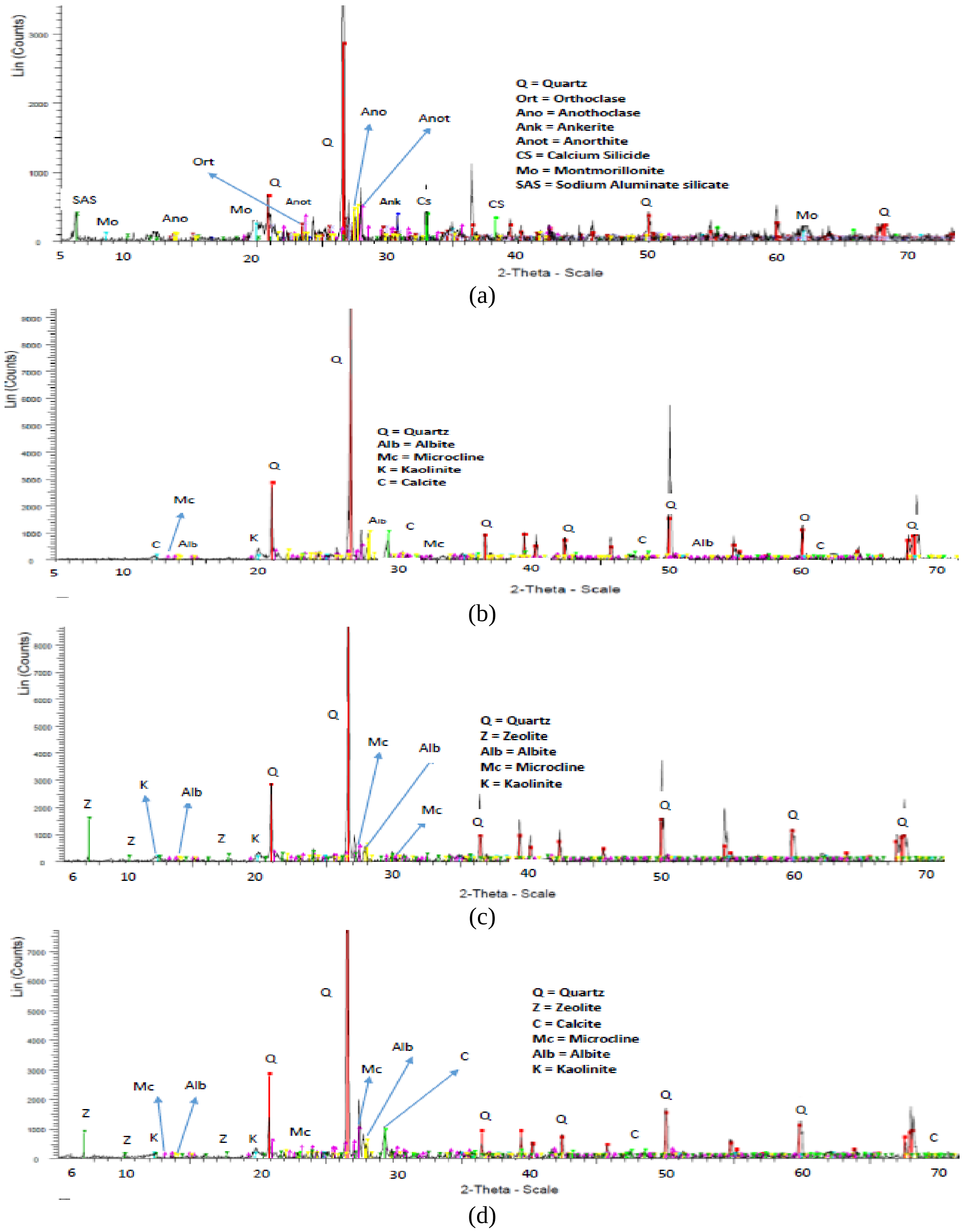

Figure 4. XRD result for (a) untreated clay, (b) clay treated with 6% cement, (c) clay treated with 6% zeolite, and (d) clay treated with 6% cement and 6% zeolite.

This process must have resulted from the cation exchange between the lime contained in the cement and ions around the montmorillonite minerals, which justified the cation exchange reaction of cement and clay. Introduction of calcite among the resulting minerals resulted from the constituent of cement. The addition of zeolite to the clay soil gave a similar reaction to cement, except that zeolite mineral was recorded among the resultant minerals, as shown in Figure 4c. Albite mineral was observed to be a hard mineral that cannot easily be disintegrated into other minerals judging from its appearance in all other mixtures.The sodium ions in the zeolite must have contributed to the disintegration of the montmorillonite minerals due to the exchange of ions. The addition of both 6% cement and 6% zeolite introduced both zeolite and calcite in the resultant minerals. This combined ionic exchange will result in the agglomeration of clay particles into larger particles which will increase the workability and strength gain of the clay soil. The morphology of the untreated clay specimen and clay mixed with cement, zeolite, and a combination of cement and zeolite is presented in Figure 5a -8h.

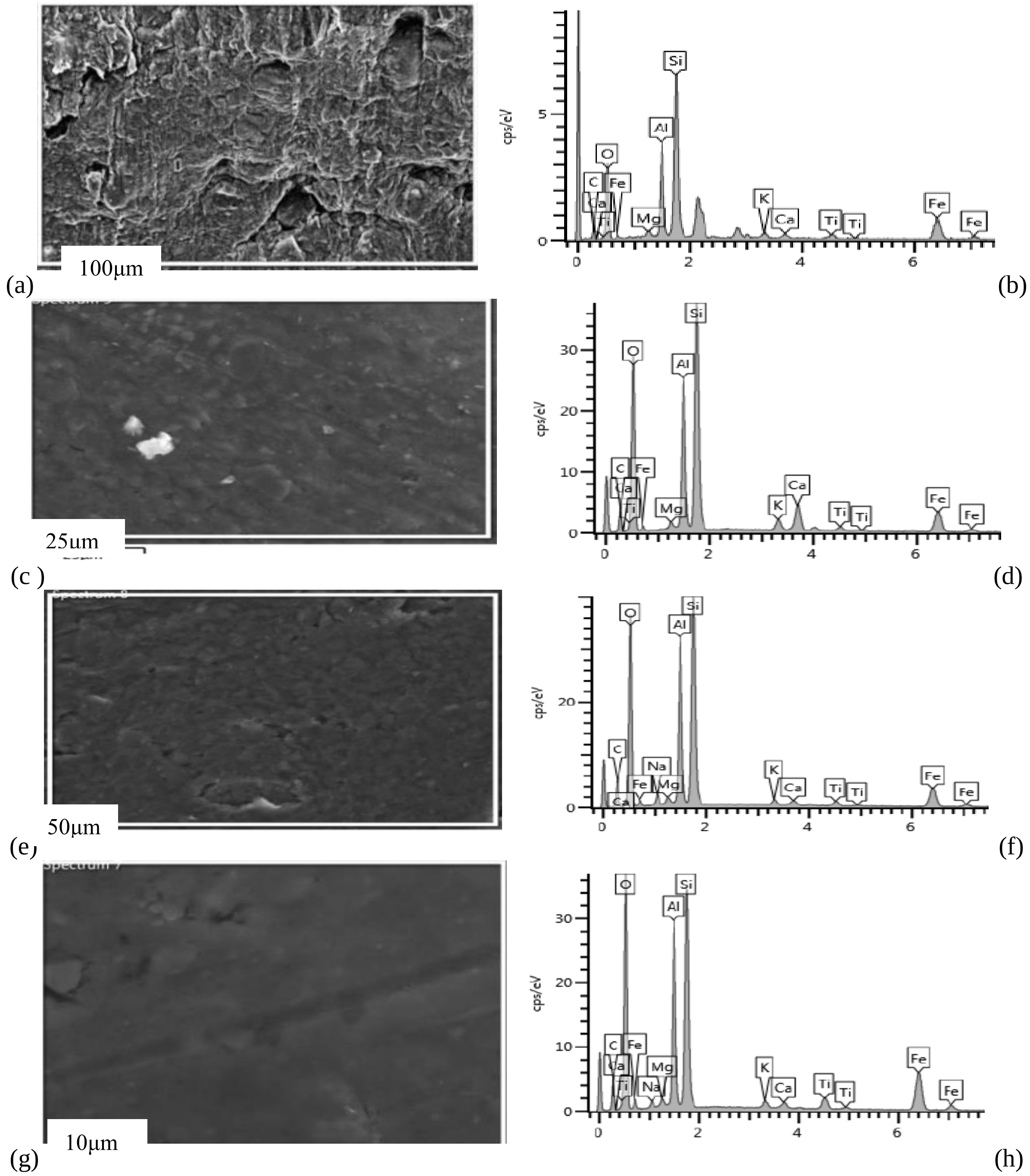

Figure 5. SEM and EDS results for (a), SEM for natural clay, (b), EDS for natural clay, (c), SEM for clay mixed with cement, (d), EDS of clay mixed with cement, (e), SEM of clay mixed with zeolite, (f), EDS of clay mixed with zeolite, (g), SEM of clay mixed with cement and zeolite, (h), EDS of clay mixed with cement and zeolite.

The natural clay shown in Figure 5a revealed whitish flaky clay particles with pore spaces. The EDS recorded a silica to alumina ratio of 1.784, which is close to 2, an indication of the presence of montmorillonite minerals [8]. The addition of 6% cement to the clay presented in Figure 8c, showed a relatively compact, non-porous structure. The silica-alumina ratio of this specimen was 1.558, which is an indication that montmorillonite mineral has begun the process of disintegration. The addition of zeolite to the clay revealed a similar structure of compact but relatively loose with occasional particulate pores. The silica-alumina ratio recorded for the mixture was 1.381. The addition of both the 6% cement and 6% zeolite to clay showed a very compact and smooth structure with very few cracks. These cracks must have resulted from the hydration reaction of cement, which can cause shrinkage stress resulting in cracks. The silica-alumina ratio of 1.216 was recorded, which is the minimal of the four specimens, which justifies the higher UCS value recorded in this mixture.

5. Conclusions

From the discussion presented, the following conclusions can be drawn:

- Addition of zeolite alone to clay after 60 days of curing increased its strength by 3.68 times the initial strength.
- For all the curing days and percentage cement additions reported, the addition of zeolite to cement-treated clay soil revealed an increase in strength to optimal percentage zeolite of 6%, after which the values dropped. Therefore, 6% zeolite is the optimal percentage zeolite required for effective stabilization of cement-treated clay soils.
- The UCS values of 1552 kN/m^2 were recorded at 6% cement and 6% zeolite addition. This value satisfies the minimum requirement for soil to be used as base course material for road structures based on [29].
- Mineralogically, the untreated clay consists of active montmorillonite clay mineral, which dissociated into fewer active minerals on addition of 6% cement and 6% zeolite with the introduction of calcite mineral from cement and zeolite mineral from the zeolite.
- The silica-alumina ratio recorded from EDS results showed a reduction from 1.784 for untreated clay, which indicates active montmorillonite minerals, to 1.216 for clay mixed with both 6% cement and 6% zeolite, which is also an indication of less active clay minerals.
- The SEM result of the specimen of clay mixed with both 6% cement and 6% zeolite showed a smooth but compact structure, which justifies the higher UCS value recorded in this mixture.

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