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Strength and Volume Change Characteristics of Clayey Soils: Performance Evaluation of Enzymes

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Received: 1 October 2019; Accepted: 25 December 2019; Published: 6 January 2020



Abstract: This study was conducted to evaluate the strength and volume change characteristics of a sedimentary residual soil mixed with bentonite (S1) when treated with three different enzymes. In addition, three reference clays including bentonite, illite, and kaolinite were also treated with enzymes to study the effect on their strength characteristics. Soil samples prepared at the optimum moisture content (OMC) were sealed and cured for four months. After curing, reference clays were tested for unconfined compressive strength (UCS). For swell tests, the S1 soil samples were placed on porous stones, which were immersed in water to allow capillary soaking of the samples. S1 samples were allowed to dry at ambient temperature for shrinkage test until the rate of reduction in volume became negligible. On completion of swell tests, the samples were tested for UCS to determine the decrease in strength due to saturation. No increase in strength and decrease in volume change were observed for any of the enzymes and dosages. Field Emission Scanning Electron Microscope (FESEM) showed some dense packing of particles for treated samples, whereas X-ray diffraction (XRD) did not reveal any change; in fact, the pattern for untreated and treated soil samples were indistinguishable.

Keywords: soil; enzymes; swell; shrinkage; stabilization

1. Introduction

Soil strength improvement is one of the basic requirements of the construction field. Researchers have been making efforts to reduce the dependence on energy-intensive stabilizers such as cement and lime [1]. In this regard several non-traditional stabilizers have been tried for improving desirable soil characteristics, which include sulfonated oils, lignin derivatives, polymer-based additives, resins, enzymes, silicates, and calcium/sodium chloride geopolymers [2–4]. A newly developed slag-blended cement and fly ash were used to improve the cemented paste backfill as a sustainable solution. It was observed that 1.5% slag-blended cement was alike 4% Portland cement to improve the mechanical properties of backfill. Furthermore, slag-blended cement when used together with fly ash proved better than used alone [5]. A soft clay with two void ratios i.e., 1.46 and 0.64 (termed as soft and stiff) was

treated with Vinyl Alcohol and 1,2,3,4 Butane Tetra Carboxylic Acid together with short polypropylene fibers to enhance its unconfined compressive strength (UCS). The results showed that the combined treatment improved the UCS and ductility of the tested clay significantly. Though, the optimum content of binders and fiber content were linked to the initial void ratio and moisture content of the clay sample [6]. Commercially manufactured sulfonated oil with different concentrations was used to treat an expansive soil. Tests were conducted to evaluate the effect of sulfonated oil on swell, UCS, and cyclic wetting and drying of expansive soil. A concentration of 1.25% yielded in optimal stabilization [7]. Expansive soils stabilized with polymers showed reduction in swell potential [8–10] and improvement in California Bearing Ratio and maximum dry density [9]. Recycled tire rubbers of fine and coarse gradations [11] and rubber powder–polymer [12] were found quite capable of reducing the swell and shrinkage potential of expansive soils.

Enzymes are organic materials which have recently been introduced to improve different pavement layers (sub-base and subgrade). They are supplied in concentrated liquid form and are easily soluble in water, thus can be added in the water used for compacting pavement layers. Enzymes are extracted by the fermentation of vegetables and sugarcane. Thus, enzymes are degradable material which are broken and dissolved with the passage of time. The main advantages of these enzymes over other additives as claimed by suppliers include cost effectiveness, environment friendly behavior, and convenience in use. Due to absence of independent and unbiased testing of these enzymes, engineers must rely only upon the information and reports as generally provided by the manufacturers. Further adding the doubts, enzymes are generally reformulation of other products therefore specific testing for a given enzyme is imperative [13]. Enzymes have actively supported in improving soil health [1,14–20]

A tropical red soil [1] and compressed stabilized earth blocks [17] were treated with TerraZyme and it was observed that the enzyme improved the compressive strength of the red soil and blocks. Soil (from Tilda region of Chhattisgarh, India) treated with enzyme showed considerable increase in cohesion though marginal increase in angle of internal friction was observed [21]. Treatment of enzyme (TerraZyme) with c clayey sand (SC, classified by Indian Standard) resulted in reduction of plasticity index and increase in unconfined compressive strength [1]. A clayey soil classified as CL, was treated with three enzymes but no improvement was observed [14]. Significant improvement in strength was found in soils treated with enzymes [22,23]. Shankar, Rai [24] treated lateritic soil with TerraZyme and found many folds increase in strength. Lacuoture and Gonzalez [20] used TerraZyme to improve different soils but did not find any considerable gain in strength but rural roads treated with TerraZyme showed better performance against monsoon rains which faced severe damage due to heavy rain season [25]. Soil treated with Permazyme 11-X did not reveal any improvement in stiffness, resistance against freeze-thaw and wet-dry cycles [26] whereas Brandon, Ding [27] noticed decrease in plasticity index and some gain in strength when Atterberg's limits and strength tests were conducted on six single source and three blended soils treated with Permazyme 11-X. Mgangira [28] treated soils with Permazyme 11-X and EarthZyme but no improvement was noted when compaction, Atterberg's limits, and strength tests were carried out. Two native soils and three reference clays (illite, kaolinite and montmorillonite) were treated with three liquid stabilizers including one enzyme but no notable improvement was found for different tests conducted [29]. Tingle and Rosa [30] selected two soils with low and high plasticity and treated those with different non-traditional additives including enzymes. Soaked and unsoaked UCS tests were conducted on untreated and treated soil samples and it was found that the enzymes were unable to improve the strength. Two soils (75.2% and 14.5% clay content) and two enzymes were chosen by Velasquez, Marasteanu [31]. Resilient modulus tests were carried out on samples treated with two enzymes, and it was observed that both enzymes increased the resilient modulus considerably of soil with high clay content (75.2%). However, for soil with lower clay content (14.5%) only one enzyme was effective and improved the resilient modulus on average 54%. It was concluded that the clay content played role in effectiveness of enzyme activity.

As already mentioned, there is lack of reliable information and the published results are contradictory about the enzymes effectiveness. It is worth mentioning here that significant improvement in soil

properties (i.e., plasticity index, strength, volume change, and permeability etc.) cannot be achieved without a chemical reaction. Unfortunately, some of the studies mentioned above which report noteworthy or substantial improvement in soil properties did not make any effort to validate their results with any chemical reaction. Thus, this study includes X-ray diffraction (XRD), an analytical test, to explore chemical change, if any, due to enzyme treatment. This study was aimed to evaluate the effect of three enzymes on three reference clays and a soil suitable for their use as recommended by the suppliers. Two very basic but important characteristics of soils related to pavements namely compressive strength and volume change were chosen for this study. Larger sized samples (10 cm × 10 cm) were prepared to minimize the errors in sample preparation and testing. An extended time of four months was selected for curing to offer enough time for slow progressive enzymes activity.

It is assumed that enzymes work as catalyst and speed up the rate of chemical reaction without becoming a part of final product. They generate a reactant mediator by attaching themselves with the big organic molecules. This mediator exchanges ions with the clay structure and break down the clay lattice, resulting in a covering affect which blocks the absorption of water. The enzymes are absorbed by the clay lattice and once the metal cations are exchanged they get free, as illustrated in Figure 1 [32,33] The enzymes support the wetting action of water to produce higher unit weight and the formulation aids cohesion among the soil particles [34].

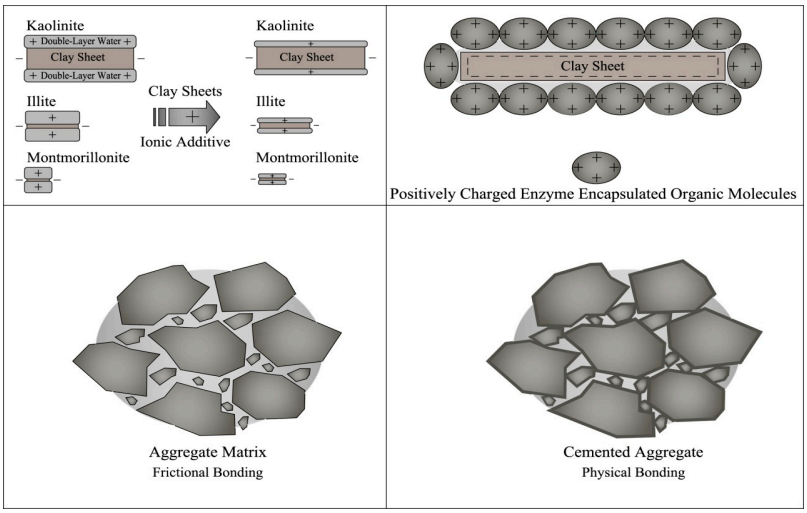


Figure 1. Proposed stabilization mechanism [35].

Enzymes catalyze the reactions between the clay and the organic cations and improve the cat-ionic exchange rate without becoming part of the final product. Enzymes help to produce cementitious compounds by the following, general reaction.

$$H_2O + \text{clay Enzyme} \rightarrow \text{Calcium silicate hydrates} \quad (1)$$

They treat adsorbed water with organic cations and neutralize the negative charge on the clay particles. The organic cations also trim down thickness of the electrical double layer. This allows the treated soils to be compacted more densely [36].

2. Materials and Methods

2.1. Materials

The detail of three enzymes and soils (three reference clays and one blended soil) used in this study is as follows:

2.1.1. Enzymes

Enzymes are usually reformulated from other products; therefore, to eradicate the chances of obtaining the same product the three enzymes selected for this study were procured from three different countries. These enzymes are DZ-1X® (DZ) (Boron Innovations Pvt. Ltd. Ahmedabad, India), EarthZyme® (EAR) (Cypher Environmental Ltd., Winnipeg, MB, Canada) and TerraZyme® (TER) (Nature Plus, Inc., Stratford, CT, USA) respectively. The properties determined in laboratory and the information enclosed in Material Safety Data Sheet (MSDS) are given in Table 1 [14]. The enzyme dosages are designated as D1 (proposed by the supplier), D2 (two times of the proposed dose), D5 (five times of the proposed dose) and D10 (ten times of the proposed dose).

Table 1. Physical and chemical properties of enzymes.

Item	DZ-1X	EarthZyme	TerraZyme
Water	—	21.06%	>50%
Alcohols, C12-C16, ethoxylated	—	—	<30%
Fermented vegetable extract	—	—	<20%
Non-ionic surfactants	—	55%	—
Polysaccharides	—	2%	—
Oligosaccharides	—	3%	—
Disaccharides	—	5%	—
Monosaccharide	—	8%	—
Lactic acid	—	3.5%	—
Potassium as the chloride	—	1.2%	—
Aluminum as the sulfate	—	0.04%	—
Magnesium as the sulfate	—	1.2%	—
Total	—	100%	—
Specific gravity	1.0	1.0 to 1.1	1.0 to 1.1
pH (neat)	4.5	3 to 6	2.8 to 3.5
Boiling point	>100 °C	>100 °C	>100 °C
Ultimate biodegradability	—	Dissolved organic content reduction >90% after 28 days	—
Composition	—	A blend of fermented carbohydrates, inorganic salts, and surfactants	—

"—" means data not available.

The recommended application rates of different enzymes by the suppliers are stated in different terms and units. For that very reason, it would be advantageous to describe the following two terms:

- "Dilution mass ratio (DMR) is the mass ratio of concentrated chemical product to water, used to express the product dilution in water prior to soil application" [37].
- "Application mass ratio (AMR) is the mass ratio of concentrated chemical product to oven-dry material in the treated soil" [37].

Since the proposed dosages by the suppliers were too low to accurately measure, it was therefore decided to dilute the concentrated enzymes with water before mixing with soil. Suppliers' proposed dosages, DMR, AMR and diluted application ratios (DAR) for S1 (maximum dry density taken for these calculations was 1675 kg/m^3) are given in Table 2. As different dosages (D1, D2, D5 and D10) of enzymes were used to prepare soil samples, the quantity of the DMR increased accordingly. For example, the "5/1000" DMR proposed for DZ by the supplier increased to "25/1000" to prepare DZ-D5 samples.

Table 2. Proposed dosages, dilution ratios, and diluted application ratios of enzymes.

Stabilizer	DZ	EAR	TER
Suppliers recommended dosage	1 L per 4.2 m^3	1 L per 33 m^3	1 L per 25 m^3
Equivalent Dilution Mass Ratio (DMR)	$\frac{5}{1000}$	$\frac{1}{1000}$	$\frac{1}{1000}$
Equivalent Application Mass Ratio (AMR) *	$\frac{1}{7035}$	$\frac{1}{55,275}$	$\frac{1}{41,875}$
Diluted application ratios (DAR) *	28.5 mL per kg of soil	18 mL per kg of soil	24 mL per kg of soil

* Maximum dry density (1675 kg/m^3) of S1 soil was used to calculate AMR and DAR.

2.1.2. Reference Clays

Three common reference clays namely kaolinite, illite, and bentonite were selected for the study. Kaolinite and illite clays were obtained from Kaolin (Malaysia) factory under the trade names of "S-300" and "KM800" respectively. Bentonite used in this study is a high swell sodium bentonite containing sodium montmorillonite, which was procured from Linachem Selangor Malaysia.

Different properties of three reference clays i.e., kaolinite, illite and bentonite as provided by the suppliers and additionally determined by conducting various geotechnical laboratory tests following the relevant American Society for Testing and Materials (ASTM) recommended procedures and equipment are listed in Table 3.

Table 3. Properties of three reference clays.

Characteristics	Bentonite	Illite	Kaolinite
SiO_2	60.85	29.43	85.76
Al_2O_3	14.82	52.37	9.11
Fe_2O_3	4.38	1.85	0.38
K_2O	—	8.21	1.34
CaO	3.67	—	—
MgO	—	1.76	—
Na_2O	3.13	—	—
TiO_2	—	1.36	—
MgO	3.09	—	—
Heat loss	8.22	5.02	3.41
Specific gravity	2.66	2.46	2.54
Liquid Limit (LL)	466	64.8	42.8
Plastic limit (PL)	69.5	22.7	17.1
Plasticity Index (PI)	396.5	42.1	25.7
Optimum moisture content (OMC)	30.9%	23.6%	17.1%
Maximum dry density (MDD)	1.16 g/cm^3	1.19 g/cm^3	1.55 g/cm^3
Soil classification *	CH	CH	CL

* Unified soil classification system (USCS). "—" means data not available.

2.1.3. UKM Soil Mixed with 10% Bentonite (S1)

Another soil chosen for this study was Universiti Kebangsaan Malaysia (UKM) soil. The UKM soil is a sedimentary residual soil classified as CL by plasticity chart. Gradation curve of UKM soil is given in Figure 2. UKM soil was mixed with 10% bentonite for different tests and it is designated as S1 in this study. This mixing percentage was chosen due to very low hydraulic conductivity of bentonite. Higher quantity of bentonite would increase the time required for volume change tests considerably. Kabir and Taha [38] and Taha and Taha [39] used the UKM soil in their experimental studies. Some of the indices and properties of UKM soil and S1 (UKM soil + 10% bentonite) and different properties of UKM soil determined by Kabir and Taha [38] and Taha and Taha [39] are given in Table 4 for comparison. When the values are compared with the results of previous studies, there are slight differences among the values which are inevitable in these tests and thus are acceptable.

Enzymes may work well for soils containing 12 to 24% clay fraction with plasticity index between 8 and 35 [13]. Thus, UKM soil is quite appropriate for enzymes performance.

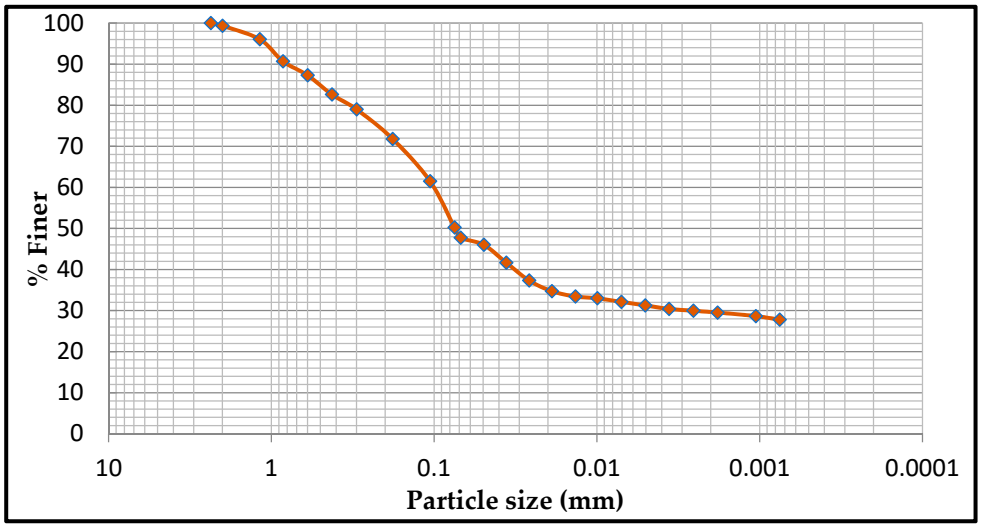


Figure 2. Particle size distribution of UKM soil.

Table 4. Characteristics of UKM and S1 soil.

Characteristics	Kabir and Taha [38]	Taha and Taha [39]	UKM Soil	S1
Plasticity Index (PI)	18.8%	16.96%	19.5%	38.3%
Liquid limit (LL)	36%	—	38.4%	70.3%
Silt fraction	18%	29.16%	20.64%	18.77%
Clay fraction	23%	18%	29.6%	36%
Optimum moisture content	15.8%	14.29%	16%	19.4%
Maximum dry density	1.78 g/cm^3	1.84 g/cm^3	1.79 g/cm^3	1.68 g/cm^3
Soil classification *	SC	SC	CL	CH

* Unified soil classification system (USCS). "—" means data not available.

2.2. Samples Preparation and Testing

Rauch et al. [33,37] conducted their experimental study on three liquid stabilizers with a protocol, which was devised after consulting several industry representatives and the Texas Department of Transportation. After completing their study, they noticed some deficiencies in the employed protocol, hence accordingly some changes were suggested for future research to prepare soil samples with liquid stabilizers. For the current study, the "Revised Protocol for Preparing Soil Test Specimens" was chosen and its application in this study is presented hereafter:

First, AMR and DMR were calculated from the application rate suggested by the supplier and then concentrated enzyme was diluted to the required DMR. Initial moisture content w_o was determined by [37]:

$$w_o = OMC - AMR/DMR + w_1 \quad (2)$$

OMC is the optimum moisture content and w_l is the loss of water during soil sample preparation which was estimated around 1%. The soil mixed with this initial moisture content was sealed in plastic bag and placed to mellow for minimum 16 h (standing time suggested by ASTM D698-7 for thorough absorption of water by the soil particles). After mellowing, the diluted stabilizer was added and mixed thoroughly to attain a homogeneous mixture. The soil was again sealed in plastic bag and was allowed to mellow for further 1 h. Soil samples were then compacted by standard Proctor test and properly sealed just after their extrusion from the molds. Once the curing time was completed the soils samples were unsealed and the required tests were performed.

The above procedure can be explained by taking the example of sample preparation of S1 for DZ-1X enzyme with single dosage (DZ-D1). The OMC for S1 was determined as 19.4%. The soil taken for sample preparation was 2 kg, thus the total water required for mixing was 20.4% (OMC + wl). Diluted solution was prepared by mixing 5 mL of DZ in 1 L of water (DMR = $\frac{5}{1000}$). DAR for this trial was calculated as 28.5 mL for 1 kg of soil therefore dilute solution required for this dosage was 57 mL (for 2 kg soil). Now the initial moisture content (w_o) for 2 kg of soil was calculated as:

$$w_o = 388 - 57 + 20 = 351 \text{ mL} \quad (3)$$

This quantity of 351 mL of water was added to the soil, mixed thoroughly and allowed to mellow for 16 h. On completion of mellowing period, 57 mL of dilute enzyme solution was added, and the mixture was mellowed for 1 h again before it was compacted in the mold.

Three mechanical tests i.e., shrinkage, swell, and UCS were carried out in this study. Standard Proctor (ASTM D 698) was selected to determine OMC and maximum dry density (MDD) of S1 and three reference clays. The same standard was followed to prepare samples for all the tests. Instead of preparing conventional samples of Shelby tube size (38 cm by 76 cm), the sample size for all the tests was chosen to have a diameter of 10 cm and height of 10 cm. The larger samples were prepared to minimize the error in sample preparation and testing. It is to be noted that the height to diameter ratio is one rather than the recommended ratio of two, as the emphasis is on the comparative behavior than the actual ultimate compressive strength [30,40]. Untreated samples for these tests were also prepared, sealed, and kept with treated samples. The basis was to replicate the conditions for any change in moisture content of treated samples and gain in strength due to aging.

For S1, the samples were prepared for shrinkage, swell, and UCS tests. Three sample for each dosage were prepared and average of the three readings was taken as final value. All the samples were cured for four months. For three-dimensional free swell test, soil samples were placed on porous stone plates which were immersed in water to calculate the percentage increase in volume. After the completion of this test, these samples were used for soaked UCS tests. The shrinkage tests were conducted by keeping the samples prepared at OMC in a constant temperature room to determine the decrease in volume due to loss of moisture content. UCS tests were carried out on cured samples to compare the results with soaked UCS test results. However, for the reference clays (bentonite, illite, and kaolinite) the samples were prepared and cured for four months and only UCS test was conducted. XRD, tests were carried out on untreated and treated soil samples to identify and detect any chemical change, if occurred.

Rauch et al. [29] suggested unconfined three-dimensional free swell test after conducting one-dimensional free swell test on soils treated with an enzyme. It was realized that conventional one-dimensional test where small soil specimen is tested in oedometer cannot give precise results. Therefore, three-dimensional free swell test, in which larger samples can be tested, was selected for this study to determine the increase in volume due to capillary soaking. UKM soil was classified as CL, thus with low swell potential due to moisture entrapment. For this purpose, high swell sodium bentonite was added in the UKM soil. A percentage of 10% bentonite was selected to mix with UKM soil as higher percentage would have taken very long time to saturate the sample due to its very low hydraulic conductivity. The liquid limit (LL = 70.3) and plasticity index (PI = 38.3) values of S1 soil also justified that the selected CH soil (fat clay) was suitable for this test.

Samples for swell test, shrinkage test and UCS were prepared by following the procedure laid down by National Lime Association [41]. Although the procedure is to prepare lime treated sample, but similar procedure was adopted by Tingle and Rosa [30] and Santoni et al. [40] to make larger sized samples with various stabilizers. Samples were prepared at OMC and MDD which was determined for untreated soils by following ASTM D698-7 procedure. After the completion of curing period the samples were unsealed, and the readings were taken to measure the initial volumes of the samples. Three vertical readings (120° apart) with Vernier caliper and three peripherals using measuring tapes were recorded to calculate the initial volume (Vi). After recording the measurements, the samples were

immediately wrapped with wet absorptive fabric. The samples were then put on the porous stone plates that were immersed in water (Figure 3). The water level was maintained at a level where it just reached the top of the plate and was in contact with fabric, but the samples did not come directly in contact with water. Water reached the sample base through these porous stone plates and made the sample saturated due to capillary action. The samples were kept saturating for 24 days. They could have been allowed to saturate even more, but the samples started to disintegrate from the base and could be noticed as the water started becoming gloomy. The samples were then taken out of the water container and absorptive fabric was removed to take the readings for final volume (Vf). The change in volume was; $\Delta V = Vf - Vi$. The expansiveness or swell potential (%S) was calculated as:

$$(\%)\ S = \Delta V/Vi \times 100 \quad (4)$$

For shrinkage tests, the samples were unsealed after the curing period of four months and measurements were taken to calculate the initial volume of samples. Samples were then placed at a constant temperature (25 °C) to dry smoothly. The readings were taken after one week as the rate of loss of moisture was very slow. It took 110 days to reach a point where the decrease in volume was almost negligible. The final measurements were taken and the percentage decrease in volume or % shrinkage (% Sh) was calculated as explained in swell test

$$(\%)\ Sh = \Delta V/Vi \times 100 \quad (5)$$



Figure 3. Samples placed on porous plates for swell test.

3. Results and Discussion

This section covers results of five tests conducted in the study including three-dimensional swell test, Shrinkage Test, Unconfined compressive strength test, Field Emission Scanning Electron Microscope (FESEM) and XRD to discuss the performance of soil before and after addition of enzymes.

3.1. Three-Dimensional Swell Test

The expansion in percentage or swell potential (% S) for untreated and the specimens treated with three enzymes for different dosages are shown in Figure 4. The average swell potential of the three untreated samples was 6.63%. Samples treated with enzymes showed swell potential with little variation. The maximum improvement was observed for EAR-D5 samples, where swell potential reduced to 6.48%. This reduction in swell potential is only 2.26% of the untreated samples. The maximum error was 0.21 for TER-D10 samples. It is apparent from the figure that the swell potential for untreated and treated samples is alike and the three enzymes were unable to substantially decrease the swell potential for any of their dosages. Rauch et al. [37] observed similar behavior

through one-dimensional swell tests on two native natural clays and three references clays (kaolinite, illite, and montmorillonite) treated with an enzyme.

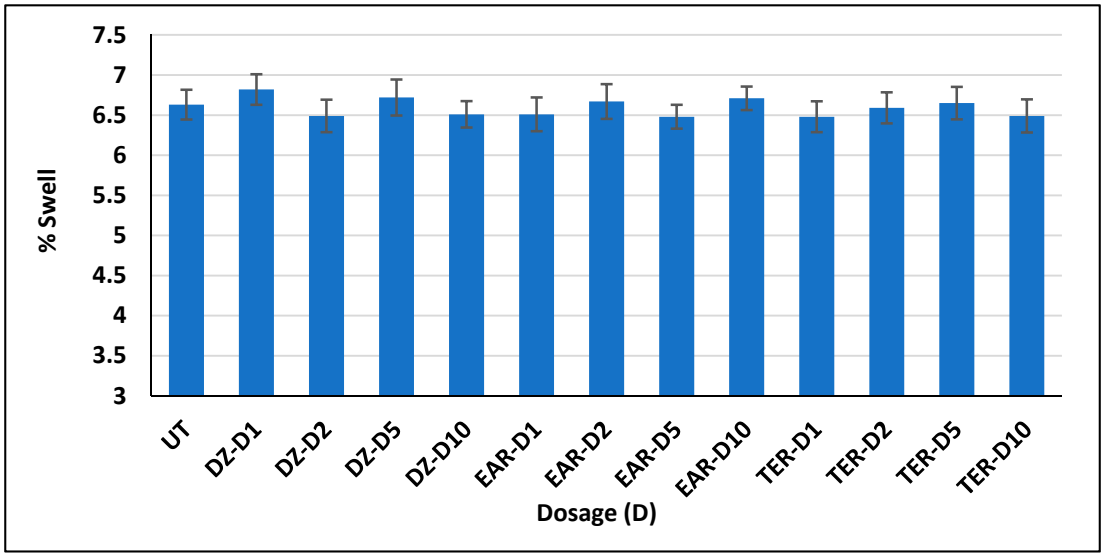


Figure 4. Swell percentage of untreated and treated S1 samples for different dosages of enzymes.

3.2. Shrinkage Test

The shrinkage of untreated and treated samples is shown in Figure 5. The swell tests were conducted first, and not much reduction in swell potential for treated samples was observed (Figure 6). Thus, it was expected that shrinkage potential will not change by great deal. Shrinkage potential of untreated samples was recorded 8.47%. Among the treated samples, the values are marginally higher but still lower than the shrinkage of untreated samples. A maximum reduction in shrinkage was shown by EAR-D1, which is 2.83%.

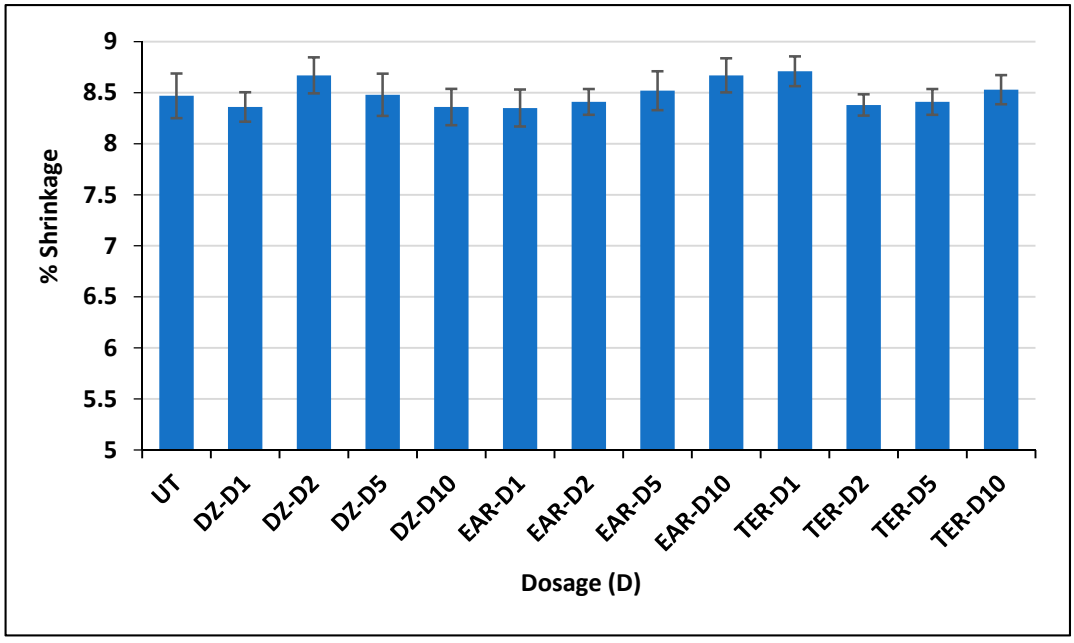


Figure 5. Shrinkage percent of untreated and treated S1 samples for different dosages of enzymes.



Figure 6. Samples after swell test.

3.3. Unconfined Compressive Strength Test

UCS tests were conducted for S1 (UKM soil mixed with bentonite) and three reference clays. After considering the results of previous investigations where generally small sized samples (3.8 cm to 7.6 cm) were used due to easy availability of Shelby tubes, it was decided to prepare larger samples (stabilization of clay soils with non-traditional additives, stabilization of silty sand with non-traditional additives). The samples were prepared in Proctor mold and cured for four months before testing. After completion of swell test, the soaked samples were tested for UCS as well by following ASTM D2166 procedure (Standard Test Method for Unconfined Compressive Strength of Cohesive Soil). The unsaturated (unsoaked) UCS results of S1 are given in Figure 7. The UCS of untreated samples was 143 kPa. The maximum strength of 150.18 kPa was recorded for EAR-D5 and TER-D5 samples, resulting in an increase of 5%. This increase cannot be considered to be notable improvement in strength and similarly soaked UCS tests did not reveal any improvement in strength too (Figure 8). Figures 9–11 show the UCS for three reference clays i.e., bentonite, illite, and kaolinite, respectively. The results are consistent and did not show any improvement in strength for different soils. Maximum increase in UCS was 3.94%, 7.78% and 8.68% for bentonite, illite, and kaolinite, respectively.

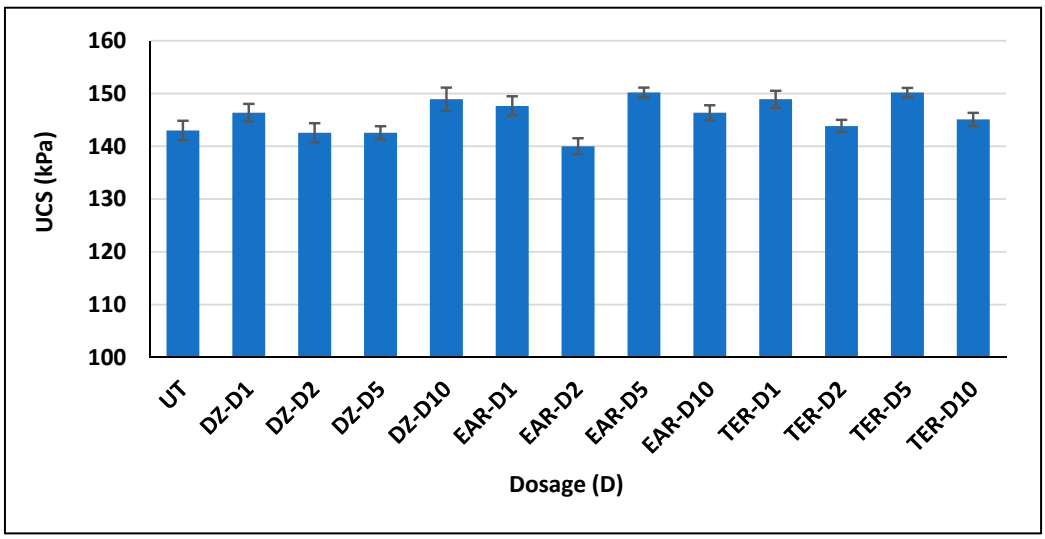


Figure 7. Unsoaked UCS of S1 for different dosages of enzymes.

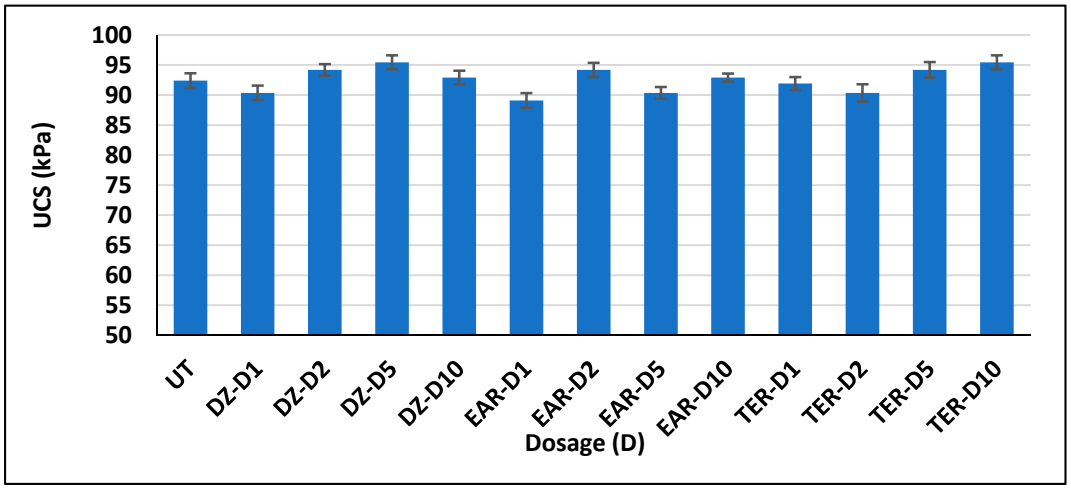


Figure 8. Soaked UCS of S1 for different dosages of enzymes.

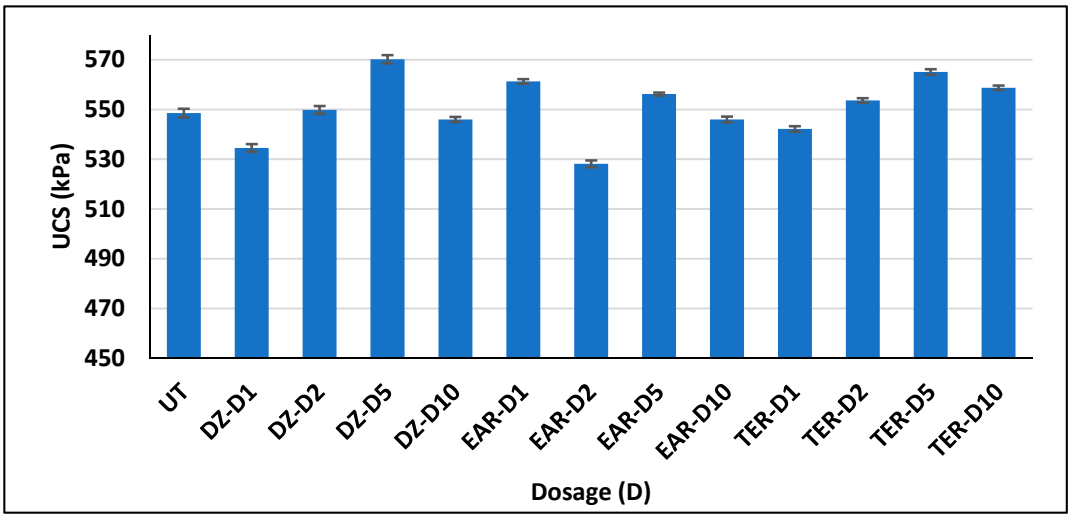


Figure 9. UCS of bentonite for different dosages of enzymes.

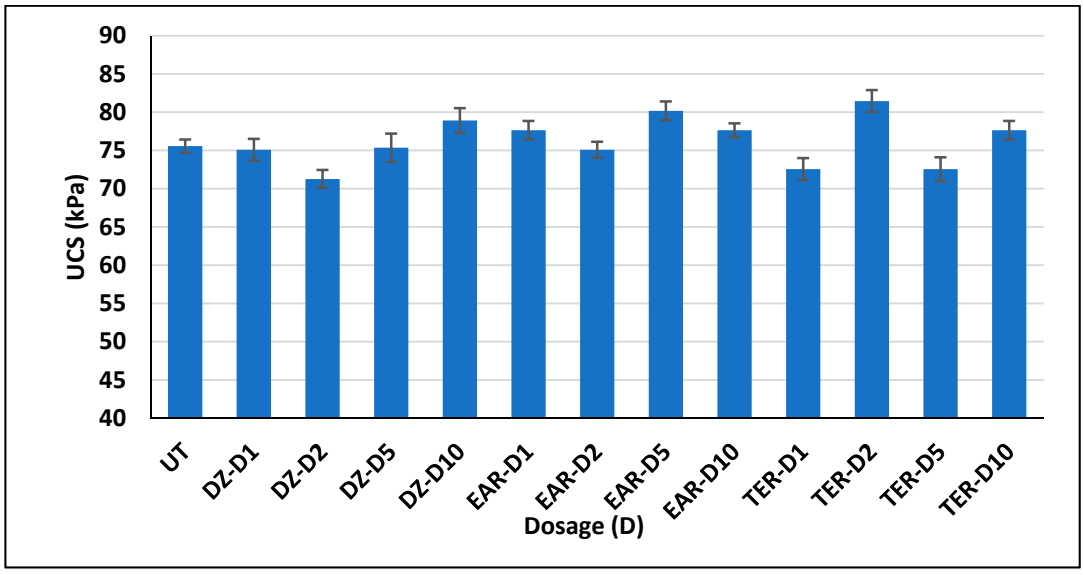


Figure 10. UCS of illite for different dosages of enzymes.

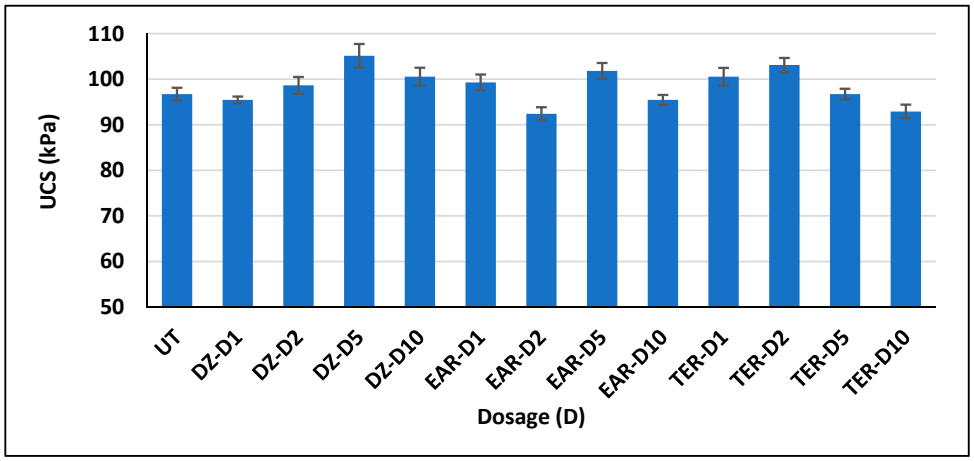


Figure 11. UCS of kaolinite for different dosages of enzymes.

Shankar, Rai [24] and Venkatasubramanian and Dhinakaran [23] found considerable increase in UCS for soils treated with enzymes. A maximum increase of 450% and 400% was observed by Shankar, Rai [24] and Venkatasubramanian and Dhinakaran [23], respectively. Shankar, Rai [24] used TerraZyme in their study which is one of the enzymes used in this study. In their studies many-fold increase in strength was reported yet results were not supported by identifying any chemical reaction between soil and enzyme.

3.4. X-Ray Diffraction (XRD) and Field Emission Scanning Electron Microscope (FESEM)

XRD is a very reliable and dominant technique for minerals identification in soils and rocks [29]. XRD tests for three reference clays (bentonite, illite, and kaolinite) were conducted for untreated (UT) and treated soil samples after four months of curing. The XRD results of untreated and treated soil samples for bentonite, illite, and kaolinite are stacked and shown in Figures 12–14, for comparison. The XRD patterns for the bentonite, illite, and kaolinite treated samples are consistent with the respective untreated diffractogram. Certainly, the d-spacings for the treated and untreated samples for bentonite, illite, and kaolinite are closely identical. It is evident that no chemical change took place to alter the chemical composition of the soils treated by any of the three enzymes.

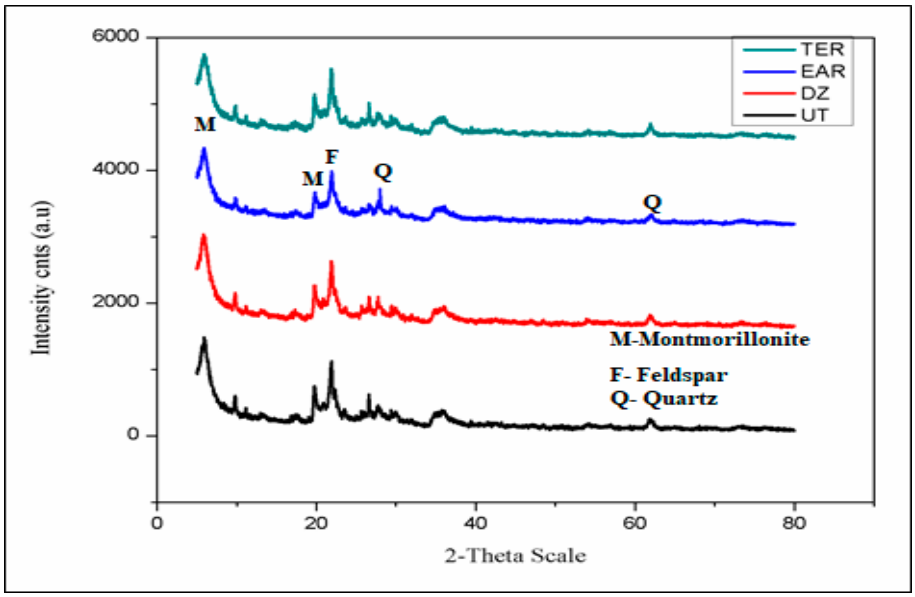


Figure 12. Comparison of XRD results for untreated (UT) bentonite and treated with enzymes.

The peaks and the distance (2θ) for the untreated and specimens treated with three enzymes are identical thus suggesting that no chemical change took place. Thus, it is expected that no chemical changes had occurred, as found in swell, shrinkage, and UCS tests. The test results are consistent with the XRD results of Rauch [29] of three reference clays (bentonite, illite, and kaolinite).

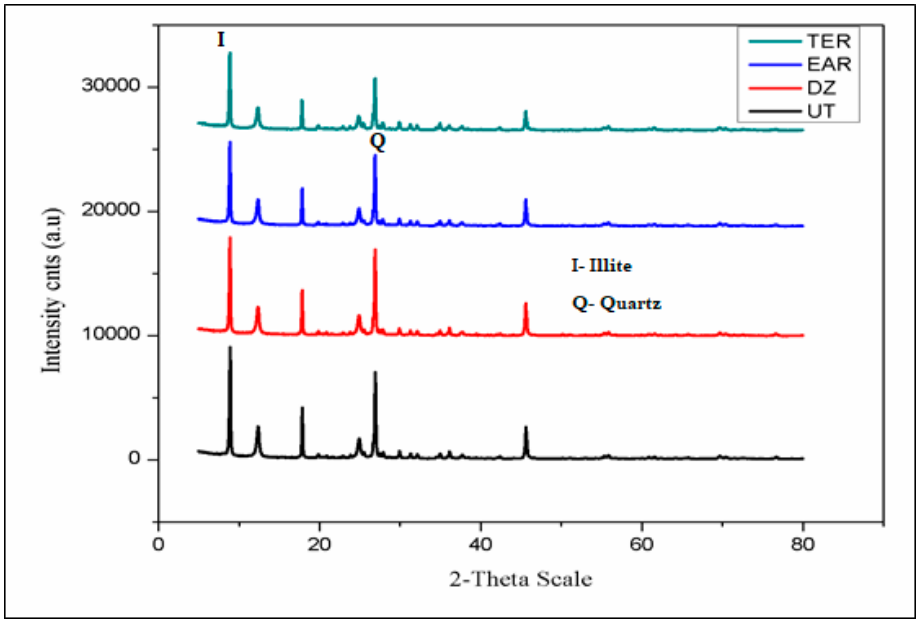


Figure 13. Comparison of XRD results for untreated (UT) illite and treated with enzymes.

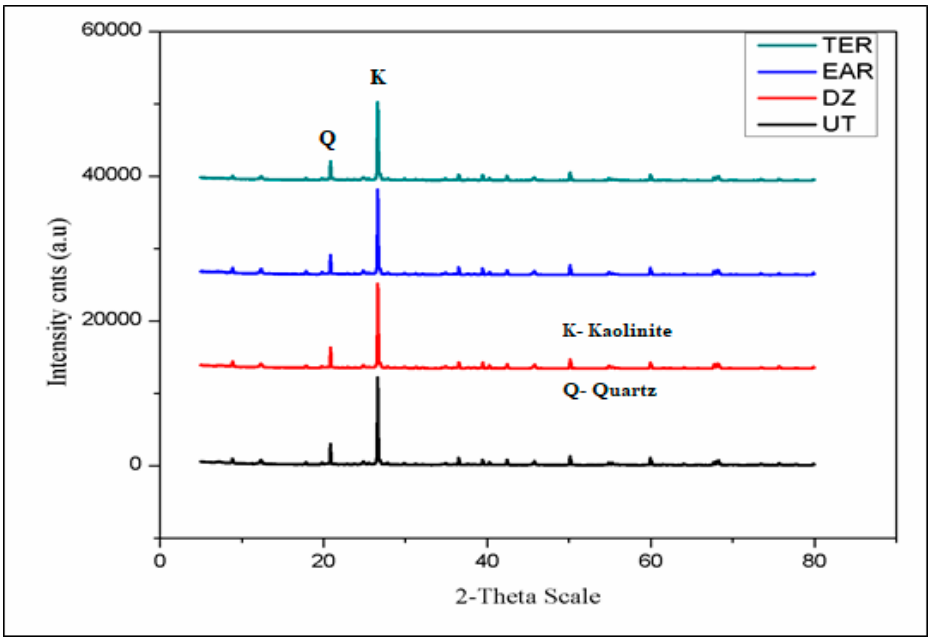


Figure 14. Comparison of XRD results for untreated (UT) kaolinite and treated with enzymes.

FESEM images of untreated and samples treated with D10 dosage of S1 soil are given in Figure 15. The treated samples seem to be more accumulated than the untreated sample, and the clay features are less evident. Though, there does not appear to be a change in the composition of the material. The voids or pores can be seen as shadows in Figure 15a due to looseness of particles in untreated samples. These results are steady with the hypothesis suggested by Scholen [32] and Rauch et al. [33],

which says that the enzymes combine with organic molecules, which then surround the clay minerals and abolishing the negative charge on the clay surface and reducing the clay's attraction for water.

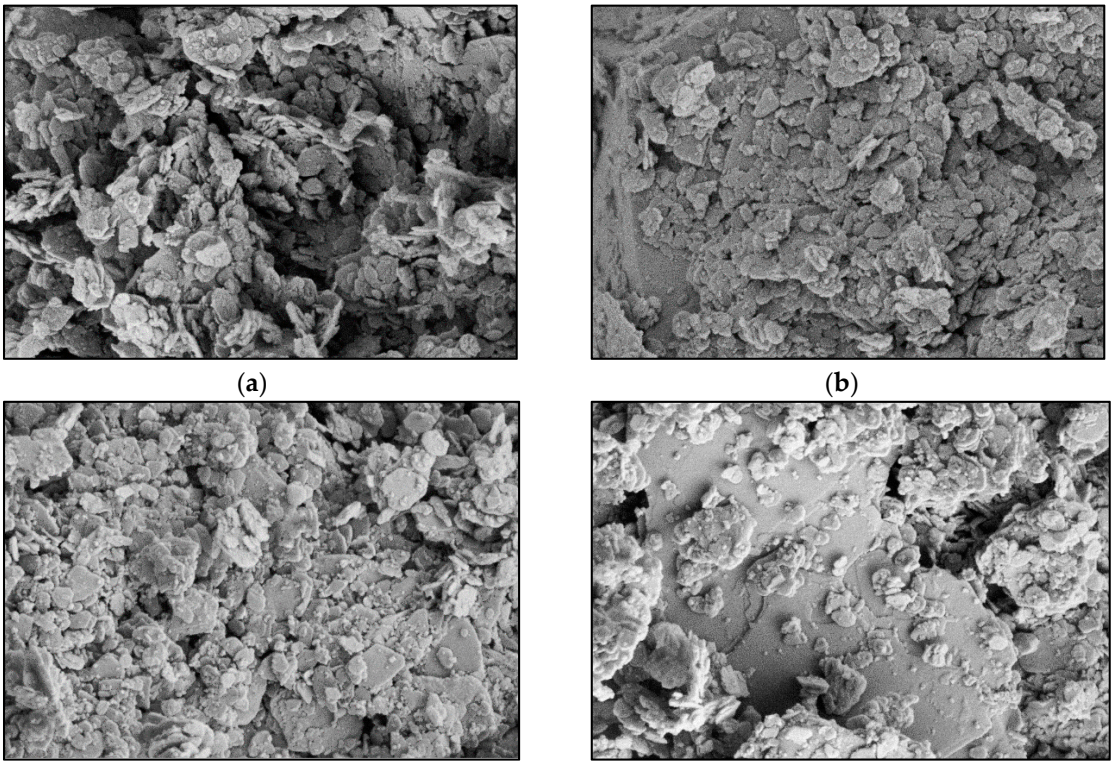


Figure 15. Comparison of FESEM results at a magnification of 20,000 for (**a**) untreated (UT), (**b**) DZ-10, (**c**) EAR-10 and (**d**) TER-10.

4. Conclusions

This study has been designed to analyze the performance of enzymes on strength and volume change characteristics of clayey soils. The effects of three enzymes on volume change (swell and shrinkage) and strength characteristics of clayey soils were evaluated. Standard Proctor test was conducted to determine optimum moisture content and maximum dry density of different soils. Then the same test was carried out to formulate controlled untreated and treated soil specimens (S1 and three reference clays) with four different dosages of three enzymes and cured for four months. Samples of UKM soil mixed with bentonite (S1) were tested for volume change (shrinkage and swell tests) and UCS on soaked and unsoaked samples, whereas for reference clays UCS test was carried out. XRD tests were conducted to identify any chemical change, if occurred. After completion of swell test, the soaked samples were tested for UCS as well by following ASTM D2166 procedure (Standard Test Method for Unconfined Compressive Strength of Cohesive Soil). The UCS of untreated samples was 143 kPa. The maximum strength of 150.18 kPa was recorded for EAR-D5 and TER-D5 samples, resulting in an increase of 5%. This increase cannot be considered to be notable improvement in strength and similarly soaked UCS tests did not reveal any improvement in strength too. Shrinkage potential of untreated samples was recorded 8.47%. Among the treated samples, the values are marginally higher but still lower than the shrinkage of untreated samples. It was observed that all three enzymes did not produce any significant improvement in different tests conducted for the study. Even, very high dosages of three enzymes did not bring any considerable improvement in three reference clays. Little improvement, in some cases, can be associated with the hypothesis that the enzymes do not produce any chemical change, though they only avert moisture absorption to carry the particles closer. Therefore, in case of using invalidated stabilizer it is essential to check its appropriateness before using it on larger scale.

Author Contributions: Conceptualization, T.A.K. and M.R.T.; literature review, T.A.K. and A.W.; methodology, T.A.K. and M.R.T.; software, T.A.K., M.M.K. and S.A.R.S.; formal analysis, T.A.K. and M.A.A.; investigation, T.A.K. and M.R.T.; writing—original draft preparation, T.A.K. and M.R.T.; writing—review and editing, A.R.K. and M.W.; supervision, M.R.T. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Ministry of Education Malaysia through Universiti Kebangsaan Malaysia.

Acknowledgments: The authors gratefully acknowledged the Ministry of Education Malaysia for their initial financial support for the research project through Universiti Kebangsaan Malaysia under the project code DPP-2014-047 and the Centre for Research & Instrumentation Management (CRIM) for XRD and FESEM tests.

Conflicts of Interest: The authors declare no conflict of interest.

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