

California Bearing Ratio Tests of Enzyme-treated Sedimentary Residual Soil Show No Improvement

(Ujian Nisbah Bearing California ke atas Sedimen Sisa Tanah Terawat Enzim Menunjukkan Tiada Sebarang Penambahbaikan)

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ABSTRACT

Environmental concerns have significantly influenced the construction industry regarding the identification and use of environmentally sustainable construction materials. In this context, enzymes (organic materials) have been introduced recently for ground improvement projects such as pavements and embankments. The present experimental study was carried out in order to evaluate the compressive strength of a sedimentary residual soil treated with three different types of enzymes, as assessed through a California bearing ratio (CBR) test. Controlled untreated and treated soil samples containing four dosages (the recommended dose and two, five and 10 times the recommended dose) were prepared, sealed and cured for four months. Following the curing period, samples were soaked in water for four days before the CBR tests were administered. These tests showed no improvement in the soil's compressive strength; in other words, samples prepared even at higher dosages did not exhibit any improvement. Nuclear magnetic resonance (NMR) spectroscopy tests were carried out on three enzymes in order to study the functional groups present in them. Furthermore, X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) tests were executed for untreated and treated soil samples to determine if any chemical reaction took place between the soil and the enzymes. Neither of the tests (XRD nor FESEM) revealed any change. In fact, the XRD patterns and FESEM images for untreated and treated soil samples were indistinguishable.

Keywords: California bearing ratio test; enzymes; improvement; soil

ABSTRAK

Kebimbangan terhadap alam sekitar secara signifikan mempengaruhi industri pembinaan mengenai pengenalan dan penggunaan bahan-bahan pembinaan alam sekitar yang mampan. Dalam konteks ini, enzim (bahan organik) telah diperkenalkan bagi penambahbaikan projek permukaan tanah seperti hamparan dan benteng. Penyelidikan ini telah dijalankan untuk menilai kekuatan mampatan sisa sedimen tanah yang dirawat dengan tiga jenis enzim, yang diperolehi melalui ujian nisbah bearing California (CBR). Sampel kawalan tanah yang tidak dirawat dan dirawat dengan empat dos (dos yang disyorkan serta dua, lima dan 10 kali dos yang disyorkan) telah disediakan, ditutup dan diolah selama empat bulan. Berikutan tempoh pengolahan, sampel telah direndam dalam air selama empat hari sebelum ujian CBR dijalankan. Ujian ini menunjukkan tidak ada peningkatan dalam kekuatan mampatan tanah; dalam erti kata lain, sampel yang disediakan walaupun pada dos yang lebih tinggi juga tidak menunjukkan apa-apa peningkatan. Ujian spektroskopi resonans nukleus magnet (NMR) telah dijalankan ke atas ketiga-tiga enzim dalam usaha untuk mengkaji kumpulan fungsian yang wujud di dalamnya. Selain itu, ujian pembelauan sinar-x (XRD) dan mikroskopi pancaran medan imbasan elektron (FESEM) telah dijalankan bagi sampel tanah tidak dirawat dan dirawat untuk menentukan jika terdapat tindak balas kimia yang berlaku antara tanah dan enzim tersebut. Kedua-dua ujian (XRD atau FESEM) tidak menunjukkan sebarang perubahan. Malah, pola XRD dan imej FESEM untuk sampel tanah tidak dirawat dan dirawat tidak dapat dibezakan.

Kata kunci: Enzim; penambahbaikan; tanah; ujian nisbah bearing California

INTRODUCTION

Environmental concerns have prompted the introduction of many types of regulations, the fulfillment of which has become a challenge for design engineers, building materials manufacturers and contractors in the construction industry. For example, the demand for new construction materials that are less harmful to the environment and can easily be reused without impact is significantly increasing in the 21st century (Cabalar & Canakci 2011).

In this regard, geotechnical engineering projects are directly linked to environmental issues, thus the improved sustainability of materials used in these projects may lead to sustainable development (Jefferis 2008). With roughly 40,000 projects worldwide that require soil improvement each year adding up to AU\$60 billion (DeJong et al. 2010), geotechnical engineers are being challenged to provide workable ground for the structures. Unfortunately, all phases of the geotechnical process, i.e. planning, design

and execution-are generally governed by financial interests rather than environmental issues (Abreu et al. 2008). For instance, the process used to manufacture construction materials such as cement and lime has a deteriorating effect on the environment, as the production of one ton of cement alone emits around one ton of CO₂ (Khedari et al. 2005). Additionally, up to 7% of the world's CO₂ emissions is caused by the production of ordinary Portland cement (Dessy et al. 2011).

Enzymes, which are organic materials, have recently been introduced to improve the properties of various pavement layers (sub-base and sub-grade), as well as other earthen works, such as embankments and levees. They take a concentrated liquid form and thus are easily soluble in water, which means they can be added to the water used for the compaction of soil layers. Enzymes are degradable materials that are broken down and dissolved with the passage of time. Due to lack of independent and unbiased testing, all available information and literature about these enzymes are generally provided by the suppliers. Enzymes are typically reformulations of other products, thus specific testing for a given enzyme is necessary (Kestler 2009).

Rural roads treated with TerraZyme showed better results which faced severe damage due to monsoon rains (Ahmad et al. 1999). A significant increase in strength was observed in soils treated with enzymes (Shankar et al. 2009; Shukla et al. 2003; Venkatasubramanian & Dhinakaran 2011). For example, Agarwal and Kaur (2014) conducted unconfined compressive strength (UCS) tests on a black cotton soil treated with TerraZyme and recorded enhancement in UCS results up to 200% after 7 days of curing. However, the study did not present any chemical tests or evidence to support or confirm the results. Atterberg's limits and strength tests were conducted on six single source and three blended soils treated with Permazyme 11-X and a decrease in plasticity index with some gain in strength was observed (Brandon et al. 2010).

In a similar study, Khan and Taha (2015) conducted UCS tests on a residual soil with three enzymes; however, they did not observe any improvement. Lacuoture and Gonzalez (1995) also used TerraZyme to improve different sub-base and sub-grade soils, but did not find any appreciable gain in strength. In another case, soil treated with Permazyme 11-X showed no enhancement in stiffness or improvement to resistance against freeze-thaw and wet-dry cycles (Milburn & Parsons 2004). Similarly, Mgangira (2009) treated soils with both Permazyme 11-X and EarthZyme, but no improvement was observed 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 an enzyme, but no notable improvement was detected (Rauch et al. 2003). Two other researchers, Tingle and Santoni (2003), selected two soils of low and high plasticity and treated them with different nontraditional additives, including enzymes. Saturated and unsaturated UCS tests were conducted on untreated and treated soil samples and

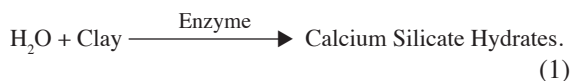
it was found that the enzymes did not increase the strength in either soil type.

As seen before, only a few peer-reviewed studies have been published on enzymes to enhance soil (Milburn & Parsons 2004) and the studies carried out so far to evaluate the suitability of enzymes have shown contradictory results. Therefore, the purpose of this study was to evaluate the effect of three enzymes on a soil considered to be suitable (presumably) by the enzyme suppliers. For example, Khan and Taha (2015) used small samples (extracted from Shelby tubes) for UCS tests, where minute errors in sample preparation and testing can significantly alter the results. Thus, a very basic but important test related to pavements, the California bearing ratio (CBR) test, was chosen for this study. Due to its larger size, CBR test minimizes the errors in sample preparation and testing that tests such as the UCS are prone to. Additionally, an extended time period of four months was selected for curing, in order to offer enough time for slow progressive enzyme activity.

Regarding the testing of enzymes themselves, this study adopted several means of assessment. Specifically, nuclear magnetic resonance (NMR) spectroscopy is an industry standard technique for the watchful examination of enzyme structures (Monasterio 2014). As such, NMR spectroscopy tests were conducted on the three enzymes used in this study, in order to obtain information about the functional groups present in the enzymes. Unlike previous studies, the current one was not limited to physical geotechnical tests, but X-ray diffraction (XRD) and field emission scanning electron microscopy (FESEM) tests were also carried out on untreated and treated soil samples to identify any chemical change that may have occurred.

STABILIZATION MECHANISM OF ENZYMES

Enzymes are assumed to work as catalysts and speed up the rate of chemical reactions without becoming a part of any final product. They attach themselves to the larger organic molecules to generate a reactant mediator. In soils, this mediator exchanges ions with the clay structure and breaks down the clay lattice, producing an effect that halts the absorption of water. The enzymes are then absorbed by the clay lattice and after the exchange, metal cations are freed, as illustrated in Figure 1 (Rauch et al. 2003; Scholen 1992). The enzymes promote the wetting action of water to produce a higher unit weight and this formulation facilitates cohesion among the soil particles (Parsons & Milburn 2003). In this case, enzymes catalyze the reactions between the clay and cations and boost the cationic exchange rate without becoming part of the final product. They do this by swapping adsorbed water with organic cations and neutralizing the negative charge on the clay particles. The organic cations also reduce the thickness of the electrical double layer. This allows the treated soils to be more densely compacted. Specifically, enzymes help to produce cementitious compounds through the following, general reaction (Agarwal & Kaur 2014):



TEST MATERIALS

ENZYMES

Three different types of enzymes from three different countries were selected for this study: DZ-1X (DZ) (Boron Innovations Pvt. Ltd., India), EarthZyme (EAR) (Cypher Environmental Ltd., Canada) and TerraZyme (TER) (Nature Plus, Inc., USA). All three enzyme suppliers were asked to provide material safety data sheets (MSDS) for

their products, but Boron Innovations Pvt. Ltd. did not provide the MSDS, although they were contacted repeatedly. However, some of the properties of the DZ-1X enzyme were determined in the laboratory and important information contained in the MSDS for EarthZyme and TerraZyme is provided in Table 1 (Khan & Taha 2015). Four dosages of each enzyme were selected for soil treatment, i.e. D1 (single dosage recommended by the supplier), D2 (two times the recommended dosage), D5 (five times the recommended dosage) and D10 (10 times the recommended dosage).

Suppliers for different enzymes state their recommended application rates in different terms and units. Therefore, it would be helpful to describe the two terms:

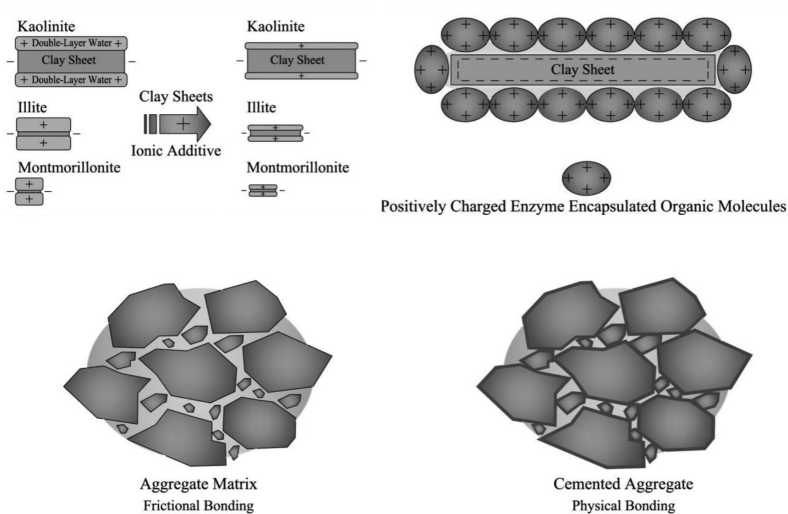


FIGURE 1. Proposed stabilization mechanism (Tingle et al. 2007)

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 sulphate	---	0.04%	---
Magnesium as the sulphate	---	1.2%	---
Total	---	100%	---
Specific gravity	1.0	1.0 to 1.1	1.0 to 1.1
pH (neat) ¹	4.5	3 to 6	2.8 to 3.5
Boiling point	>100°C	>100°C	>100°C
Ultimate biodegradability	---	DOC ² reduction >90% after 28 days	---
Composition	---	A blend of fermented carbohydrates, inorganic salts and surfactants	---

¹Concentrated enzyme, ²Dissolved organic content

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 (Rauch et al. 2002) and application mass ratio (AMR) is the mass ratio of concentrated chemical product to oven-dry material in the treated soil (Rauch et al. 2002).

The dosages suggested by the suppliers were very low and therefore the enzymes were diluted in water before mixing, with the optimum moisture content required to attain maximum dry density (MDD). Suppliers' recommended dosages, DMRs, AMRs and diluted application ratios are provided in Table 2.

TEST SOIL

The chosen soil was collected within the main campus of Universiti Kebangsaan Malaysia (UKM) in Bangi, Selangor, Malaysia. It is a sedimentary residual soil classified as low plasticity clay (CL) using a plasticity chart. The soil for all the tests was taken at one time, in order to reduce the chances of heterogeneity in soil when preparing soil samples.

SAMPLE PREPARATION

Rauch et al. (2002) devised a protocol for liquid stabilizers to prepare soil samples. They developed this protocol after consultation with a number of industry representatives and the Texas Department of Transportation. Some changes were suggested to this system after the completion of their studies. A summary of this revised protocol, 'Revised Protocol for Preparing Soil Test Specimens', is presented as follows:

After calculating the AMR from the rate of application provided by the suppliers, the next step in sample preparation was to dilute the concentrated enzyme to the suggested DMR. Initial water content to be mixed with the soil was calculated as;

$$w_o = \text{OMC} - \frac{\text{AMR}}{\text{DMR}} + 1\%, \quad (2)$$

where OMC is the optimum moisture water content. The soil mixed with this initial water content was allowed to mellow (standing time suggested by ASTM D698-7 for thorough absorption of water by the soil particles) for a minimum of 16 h in a properly sealed bag. Then, the diluted stabilizer used to achieve the recommended AMR was combined until a homogeneous mixture was formed. This mixture was again left in a sealed bag for 1 h before it was compacted in specified method. The compacted soil sample was then extruded from the mold and sealed immediately before curing. The sample was unsealed after the curing period and the required tests were carried out. The water content of this sample was assessed again after unsealing, to determine loss of water, if any.

RESULTS AND DISCUSSION

UKM SOIL PROPERTIES

The indices and properties of soil taken from UKM are provided in Table 3 (Khan & Taha 2015).

Some studies have suggested that enzymes may work well for soils containing 12 to 24% clay with a plasticity

TABLE 2. Recommended 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)			
Equivalent application mass ratio (AMR)*	27 mL per kg of soil	17 mL per kg of soil	22 mL per kg of soil
Diluted application ratios*			

*Maximum dry density of soil was taken for these calculations i.e. 1785 kg/m³

TABLE 3. Characteristics of UKM (Universiti Kebangsaan Malaysia) soil

Characteristics	Value/description
Plasticity Index ¹ (PI)	19.5%
Liquid Limit ¹ (LL)	42.3%
Clay fraction ²	29.6%
Soil classification ³	CL
Optimum moisture content (OMC) ⁴	16%
Maximum dry density (MDD) ⁴	1.785 gm/cm ³
pH	4.05

¹ "Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils", ASTM D 4318

² "Standard Test Method for Particle-Size Analysis of Soils", ASTM D 422

³ "Plasticity chart", ASTM D 2487

⁴ "Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort", ASTM D 698

index between 8 and 35 (Kestler 2009). The UKM soil almost falls into this category and thus it was considered appropriate for gauging the enzymes performance.

COMPACTION TEST

Standard compaction assessments (ASTM D 698) were performed to determine the compaction characteristics (maximum dry density and optimum moisture content) of the UKM soil. Generally, the three major factors used to determine the compaction or compaction curve are moisture content, soil type and compaction effort. Namely, as the compaction effort is increased, the MDD increases and OMC decreases for a given soil. A bell shaped curve (Figure 2) with a single peak represents the results of a typical clay-like soil with a liquid limit between 30 and 70 (Lee & Suedkamp 1972). The same compaction procedure was adopted to determine the compaction characteristics of UKM soil treated with the three enzymes at four dosages. However, no improvement was observed in the MDD of soil samples treated with any of the three enzymes.

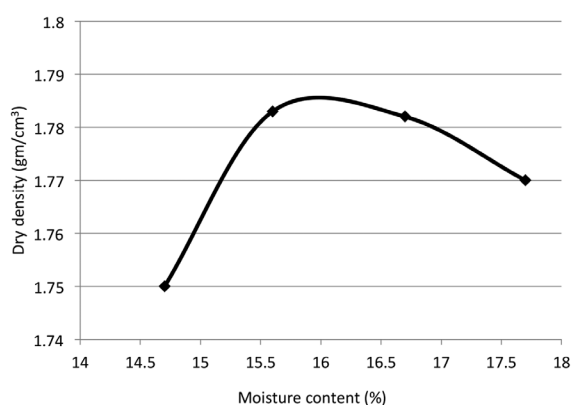


FIGURE 2. Compaction curve for UT (untreated) soil

Similar to the work conducted in this study, Milburn and Parsons (2004) carried out a number of tests on soil samples, including compaction tests on various soils (classified as CH, CL, ML, SM and SP) stabilized with lime, cement, Class C fly ash and Permazyme11-X. Two silty soils (ML and SM, having fines 88 and 30%, LL 30 and 20% and PI 7 and 3%, respectively) were treated with Permazyme11-X at the supplier's recommended dosage. Compaction tests were conducted to determine any increase in dry density, but only a minimal improvement of up to 4% in dry density was observed. In another study, Brandon et al. (2010) conducted Atterberg's limits, density, strength and R-value tests on six different soils treated with a commercially available enzyme called Permazyme. Compaction tests did not show a considerable increase in dry density; in fact, in some cases the dry density was observed to be reduced by a small amount. Rauch et al. (2003) evaluated the effect of three liquid stabilizers (an enzyme stabilizer, an ionic stabilizer and a polymer stabilizer) on five different soils. Two soils

were natural clays of high plasticity and three consisted of predominantly individual clay minerals: kaolinite, illite or sodium montmorillonite. Various tests, including compaction tests were carried out, but the enzyme stabilizer did not result in any improvement in dry density for any of the soils. Therefore, the findings of this study regarding density are quite consistent with those of previous studies. Specifically, improvement of the compaction characteristics (an increase in MDD and reduction in OMC) was not observed, contrary to the supplier's claims.

CBR TEST

The manual titled "Test Method for CBR of Laboratory-Compacted Soils1 (ASTM D 1883 – 99)" was followed for sample preparation and testing. All specimens (untreated and treated) were prepared at an OMC of 16%, with standard compaction effort. Also, the soil volume (six kg) taken for CBR tests was three times the soil used for standard Proctor mold. Therefore, the compaction effort was increased by three times, i.e. 75 strokes for each layer. The dry densities of CBR specimens were slightly lower than the MDD levels achieved through standard Proctor molds, but even the lowest value was only 2.5% less. Along with treated specimens, the untreated samples were also sealed and cured for four months to match the testing conditions applied to treated specimens. Following the curing period, all the specimens were tested after four days of soaking and the results are shown in Figure 3. It is clearly evident that none of the dosages of any of the three enzymes produced any improvement in CBR values. In Figure 4, the curves (deformation against load) for the untreated and three treated soil specimens are illustrated, which further verifies that the enzymes were unable to bring about any improvement.

Lateritic soil (LL=35, PI=10, F200=31%) from the Dakshina Kannada and Udupi districts of India was treated with TerraZyme and different tests, including a soaked CBR test, were conducted. A CBR value of 31% was

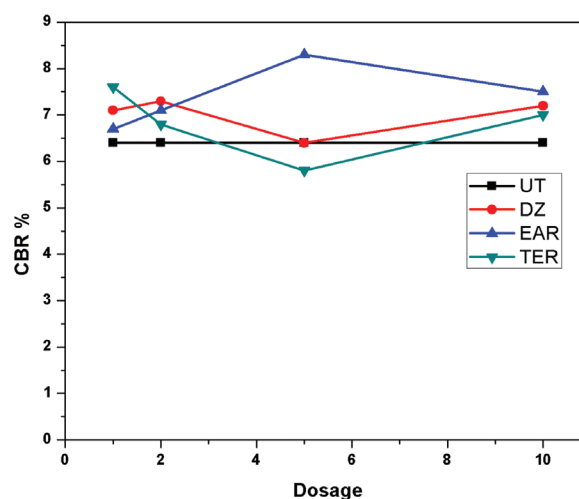


FIGURE 3. CBR values for different dosages of three enzymes

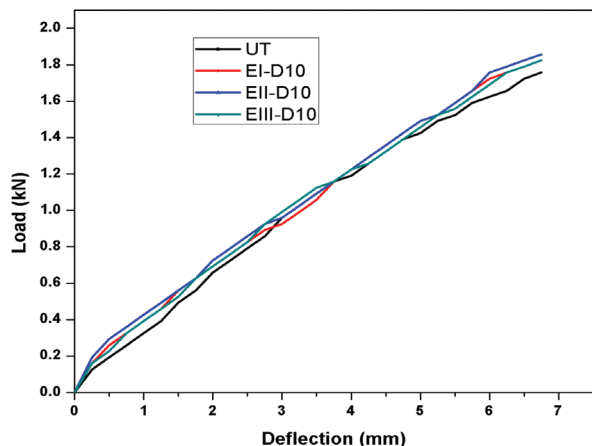


FIGURE 4. CBR test results (deflection against load)

reported after four weeks of curing the TerraZyme-treated soil, as compared to a CBR value of 8% for untreated soil, which represents an increase of 400% in CBR value (Shankar et al. 2009). The three soils (Soil 1: LL=46 and PI=6; Soil 2: LL=28 and PI=6; and Soil 3: LL=30 and PI=5) were each treated with an enzyme and two tests, CBR and UCS, were conducted. An increase of 157%, 613% and 673% after four weeks of curing was recorded for Soils 1, 2 and 3, respectively (Venkatasubramanian & Dhinakaran 2011). One of the enzymes used in the current study was TerraZyme and the UKM soil has properties quite similar to the soil used by Shankar et al. (2009), yet the results are contrasting and contradicting. To bring about a considerable change in strength, researchers agree that soil must go through some type of chemical change.

However, these results were not verified or supported by any chemical analysis or evidence in this case to justify such a change in strength. Whereas in the present study, geotechnical or mechanical tests were followed up by analytical tests (discussed below) to investigate chemical reactions, if any occurred.

NMR OF ENZYMES

The three enzymes (DZ, EAR and TER) used in this study were characterized using nuclear magnetic resonance (NMR) spectroscopy. To begin, all three samples were dissolved in deuterium oxide (D_2O). The results of the analyses of the three enzymes are provided in Figures 5 to 7. The peaks at 1 ppm (a triplet) and 2 ppm (a doublet) of the DZ enzyme figure represent alkyl groups. The peaks in the region from 3-6 ppm are likely due to water molecules present in the sample, while small peaks near 7 and 8 ppm represent some traces of aromatics in the DZ molecules. For the EAR enzyme, the presence of peaks near 1 ppm may be due to alkyl groups, while the peaks near 3.5 may be due to the presence of water molecules in the sample. Furthermore, H^1NMR results for the TER enzyme reflect a similar pattern as those of the EAR enzyme. The peaks suspected to be due to water molecules can also be seen here, as well as those due to alkyl functional group at about 1 ppm on the delta scale (Gottlieb et al. 1997). The H^1NMR results of these three enzymes (DZ, EAR and TER) compliment the previous FTIR findings regarding the functional groups. Two of the enzymes (EAR and TER) showed similar chemical shifts in H^1NMR , while DZ featured additional peaks, corresponding to aromatic molecules.

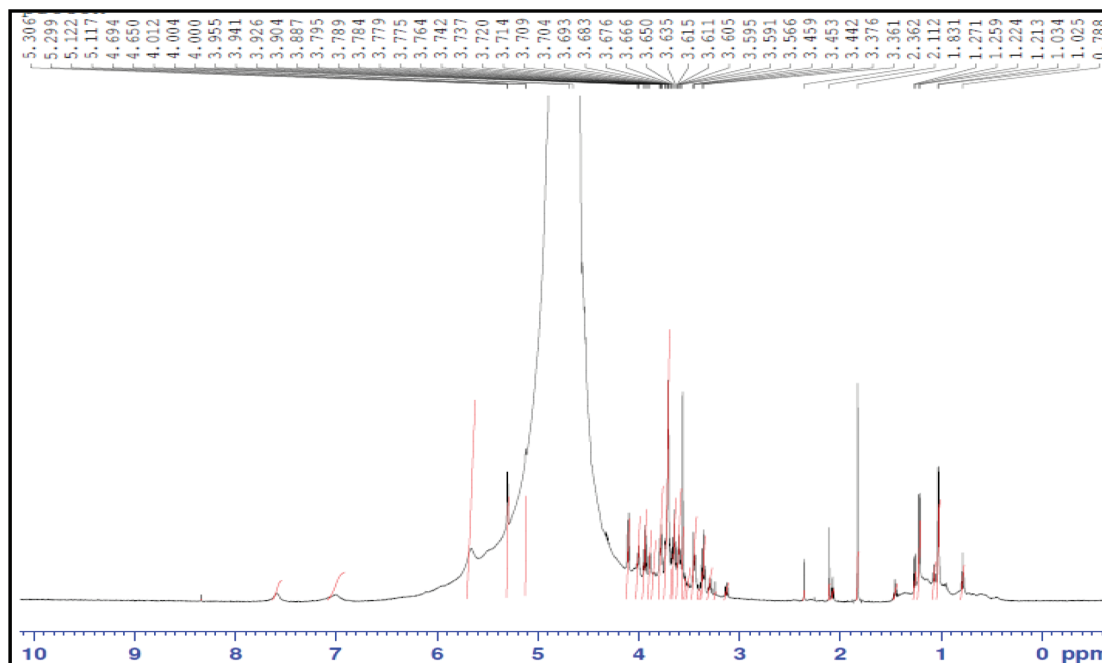


FIGURE 5. NMR spectroscopy results of DZ enzyme

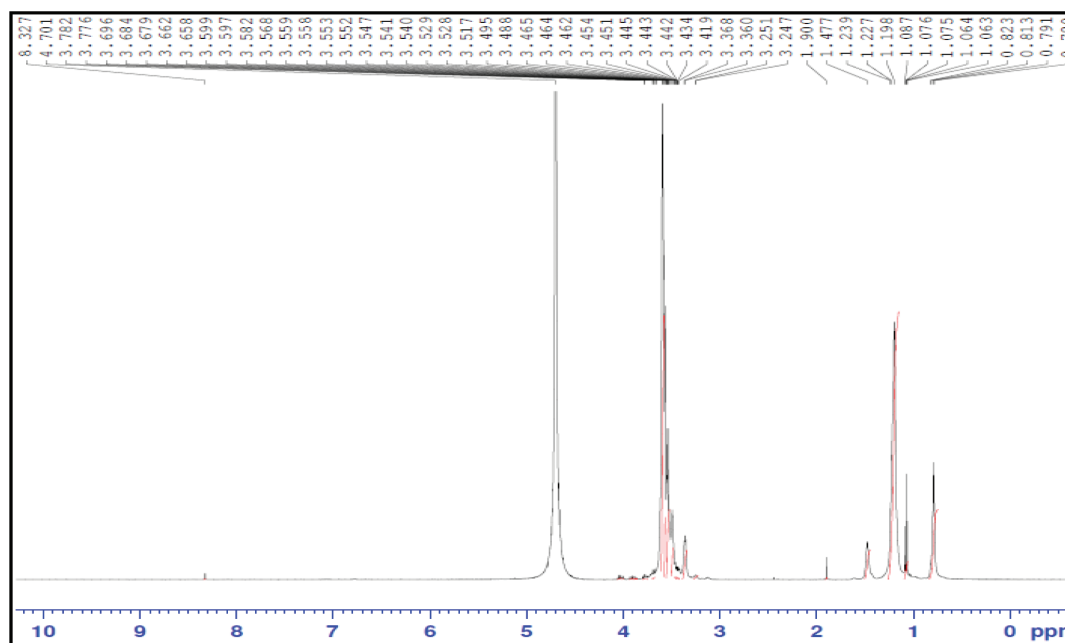


FIGURE 6. NMR spectroscopy results of EAR enzyme

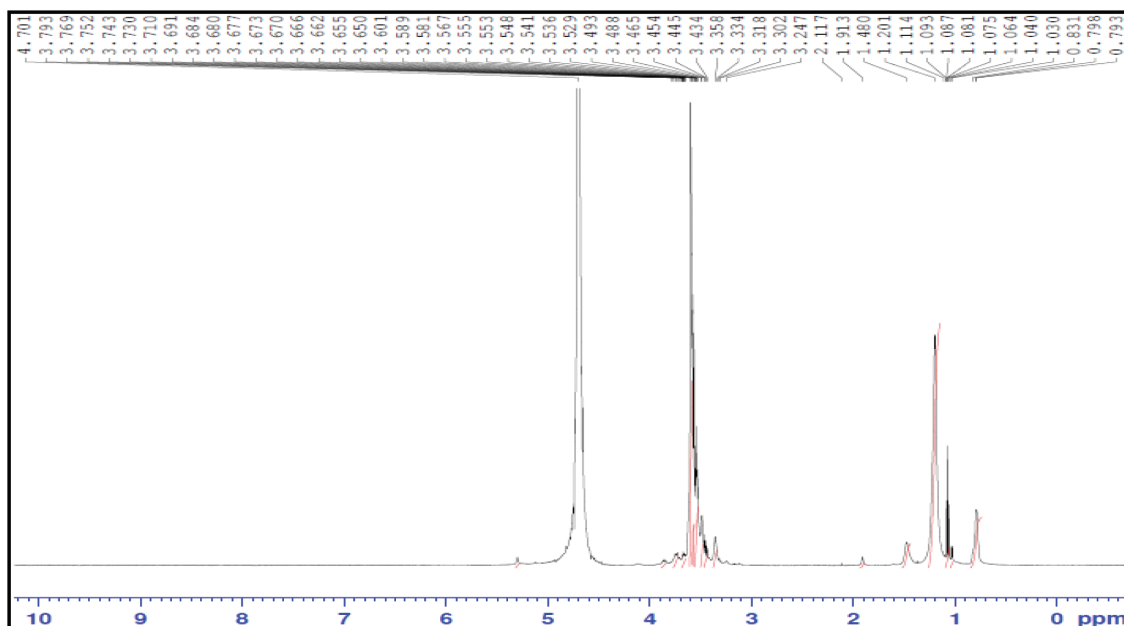


FIGURE 7. NMR spectroscopy results of TER enzyme

XRD AND FESEM

In addition to the tests outlined previously, XRD and FESEM assessments were performed on samples in this study. In the literature, Harrison and White (2008) recommended that XRD is a very reliable and standard technique for mineral identification in soils and rocks. As such, XRD and FESEM tests were conducted for untreated (UT) and treated (with all three enzymes) soil samples after four months of curing. The XRD results of the untreated and three treated soil samples were stacked and shown in Figure 8 for

comparison. It is evident that no chemical change took place to alter the chemical composition of the soil treated by any of the three enzymes. In fact, the peaks and the distance (2θ) for the untreated and treated specimens were identical, thus supporting the idea that no chemical change took place. In Figure 9, the FESEM images for untreated and treated soil specimens are shown. It can be seen that no gel has been formed in any of the images (treated specimens), suggesting that the enzymes' addition did not produce any binding material.

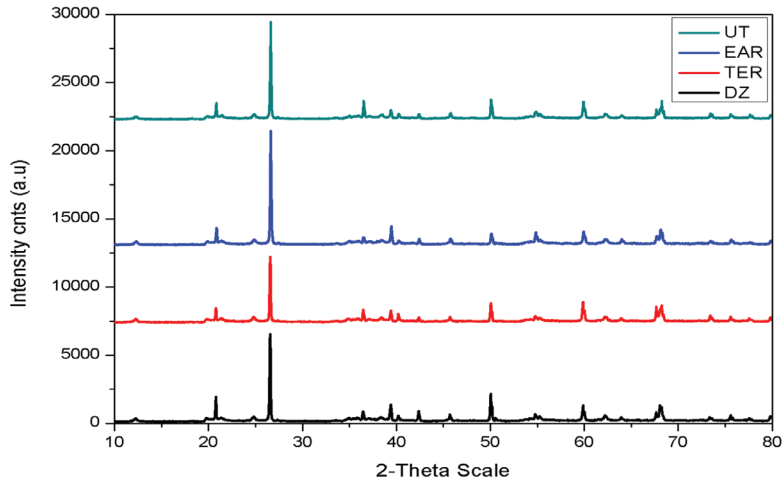


FIGURE 8. Comparison of XRD results for UT and soils treated with three enzymes

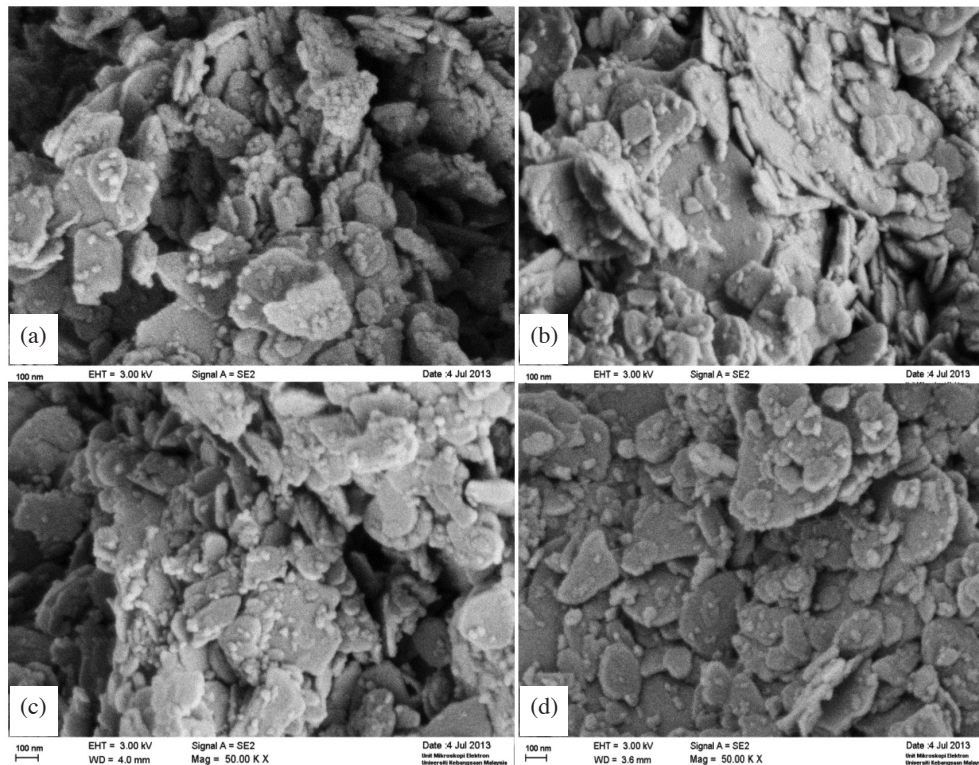


FIGURE 9. FESEM results for a) UT, b) DZ-D10, c) EAR-D10 and d) TER-D10

CONCLUSION

In this experimental study, the effects of three enzymes on CBR and compaction characteristics were evaluated. Standard Proctor tests were conducted to observe any change in optimum moisture content and maximum dry density among four dosages of the three enzymes. The same test was carried out on both control untreated and treated soil specimens and all samples were cured for four months. Following this, CBR tests were completed and XRD and FESEM tests were done to detect any chemical changes that may have developed.

It was observed that none of the two enzymes produced any explicit improvement in the two tests conducted, CBR and compaction characteristics. Minor improvement in individual cases may be attributed to general variations in the results associated with these geotechnical tests. Therefore, when using an invalidated stabilizer, it is essential to check its appropriateness before application on a larger scale.

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