

IDENTIFICATION OF MICROSCALE CHARACTERISTICS OF TREATED SUBGRADE MATERIALS AND HOW THEY RELATE TO MACROSCOPIC PROPERTIES

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ABSTRACT

Treatment of subgrade materials with non-traditional additives continues to be widely used as a solution for improving their engineering properties. The assessment for the suitability of these products is mostly based on a combination of both standard and non-standard test methods that determine properties at a macro-level. These tests do not reveal the microstructure details that may be responsible for the mechanism that contributes to the interaction between the product and the soil particles. The paper presents an example to demonstrate that characterization of materials at the micro-scale, using small specimens provides valuable information and offers the potential to develop new approaches to complement existing methods in the evaluation of treated materials and give an indication of expected performance at a macro-scale.

1 INTRODUCTION

Liquid chemicals are widely used for the improvement of soil stability in road construction. There are a number of these products on the market. Some of these products have been on the market in South Africa for over 30 years yet many road engineers are still not in favour of their use (Savage, 2006). A systematic and focused research programme on liquid chemical stabilisers in South Africa can be traced back to the 1990s, within the Built Environment Unit of the CSIR, see for example Paige-Green and Coetser (1996) and Paige-Green (1999). However, these studies were mainly on sulphonated petroleum products (SPPs).

Recently, enzyme-based liquid stabilizers, the focus of this paper, have been introduced to the road construction industry worldwide. In South Africa there are currently two enzyme-based liquid stabilisers that are more prominent than others. Studies on enzyme-based liquid stabilizers include work by Scholen (1992; 1995); Petry (1997); Petry and Das (2001); Rauch *et al* (2002); Santon *et al.* (2002); Tingle and Santoni (2003); Van Veelen (2006); Velasquez *et al* (2006); Visser (2007) and Tingle *et al.* (2007). Discussions in the above literature reveal that in general several issues remain unresolved when dealing with these products. Laboratory studies generally show the lack of significant consistent improvement in engineering properties of treated soils, for example in Tingle and Santoni (2003) and in Tingle *et al* (2007). Field tests have been reported in Velasquez *et al* (2006) where better performance from enzyme treated roads was found. Van Veelen (2006) and Visser (2007) have also reported significant improvement in CBR values of enzyme-treated material in field test panels. The assessment of the suitability of these products is mostly based on a combination of both standard tests for the determination of geotechnical engineering properties and non-standard test methods such as those in Jones and

Ventura (2003) and Jones (2007). These tests do not reveal the microstructural details that may be responsible for the mechanisms that contribute to the interaction between the product and the soil particles and the accompanying geotechnical engineering properties. Although this interaction may not necessarily lead to significant improvement in engineering properties of the treated soils, because of the premise that effectiveness of additives in soil stabilization is dependent on a good inter-particle bonding, it is considered essential to characterize the materials at the micro-level. An investigation was therefore conducted to determine whether some of the available advanced testing methods for material characterization at the micro-level can be used to determine the bonding mechanism associated with enzyme-based liquid chemical stabilisers.

The characterisation of the treated materials at a micro-scale can lead to an understanding of the expected performance of the materials at the macro-scale. It also offers the opportunity for modifying the products in order to provide significant improvements in the stability of treated soils. The objective of the paper is to present some aspects of ongoing work, which forms part of a long term study programme on the behaviour of materials treated with non-traditional chemical stabilisers being undertaken at the CSIR Built Environment Unit.

2 EXPERIMENTAL INVESTIGATION

2.1 Stabilizer products

For this investigation two enzyme-based products that are commercially available in South Africa were used. They will be referred to as Products A and B. Compositional characterization revealed four major inorganic anions in the enzyme-based products. These were fluoride, chloride, nitrate and sulphate. The concentration levels within the products were different. Relatively higher levels were found in product A than in product B. The order of concentration levels was also different. For product A the order was $\text{Cl}^- > \text{F}^- > \text{SO}_4^{2-} > \text{NO}_3^-$ and $\text{Cl}^- > \text{SO}_4^{2-} > \text{F}^- > \text{NO}_3^-$ for product B.

2.2 Sample preparation

Following the compositional characterization, small samples were prepared for the microstructure characterization. Two soil samples were selected for the test program, soil A, a black clay and soil B, reddish brown residual chert gravel. Soil A had a plasticity index of 35% and soil B had a plasticity index of 7% with linear shrinkage values of 11% and 5% respectively. Both soil samples were first sieved to produce material < 0.150 mm and < 0.075 mm. The use of this material size was dictated by the need to achieve the most effective bonding, enhance surface interactions directly with chemical groups on soil particles when mixed and the available equipment to produce the small size samples. Using a SpeedVac vacuum pump and a Fourier transform infrared spectroscopy (FTIR) tablet press, it was possible to produce 0.34 ± 0.1 mm thick tablet size samples from both materials. The optimum pressure at which samples could be compressed was 10 kPa. The sample diameters ranged between 9 and 11 mm. The samples were left to cure in a humidity controlled chamber at a relative humidity of 90% and at temperature of 25°C. The limiting factor in the preparation of the samples using this method is that the production of these tablets was time consuming. Figure 1 shows the typical samples produced.



Figure 1: Typical tablet size samples

2.2 Sample analysis

Microstructural characterization of the samples was done using a scanning electron microscope (SEM) model A LEO 1525 SEM. The SEM was operated at a voltage of 20 kV, with the Robinson back scattered detector (RBSD) with an aperture of 60 μm at a working distance of 12 mm. The magnification level was varied depending on the level of detail required and is indicated on the SEM images. SEM analysis to identify the microstructure characteristics required scanning over the samples to visually evaluate the features. The images that are presented represent the generally observed features. Figure 2 shows the SEM used in the analysis. The analysis was done at the National Metallurgical Institute of South Africa (NMISA)



Figure 2: NMISA scanning electron microscope (SEM)

3 RESULTS AND DISCUSSION

The interaction between the soil material and the enzyme-based liquid stabiliser solution were investigated by scanning electron microscopy (SEM) analysis. In an attempt to relate the micro structural characteristics to macro-scale behaviour, the interaction between the soil material and the enzyme-based liquid stabiliser solution was investigated using the unconfined compressive strength.

3.1 Microstructure analysis

The following section presents some of the results of the scanning electron microscopy (SEM) analysis in an attempt to quantify features at the micro-level that may be indicators of the expected performance of subgrade materials after treatment. The following figures show the observed surface micro structural features of both untreated and treated soil samples. Only results from samples made from <0.075 mm sieve material are presented as samples from < 0.150 mm sieve material of the chert easily crumbled.

Figure 3 shows the microstructure characteristics of the untreated soil sample A. From image (b) of Figure 3, which is at a magnification of 5000x, the micro-cracks ranging in width between 1 and 3 μm are noticeable. The maximum width of these cracks was 10 μm . It should be remembered that these were tablets formed by pressing the soil at a pressure of 10 kPa and hence the even surface. The images show a compressed structure.

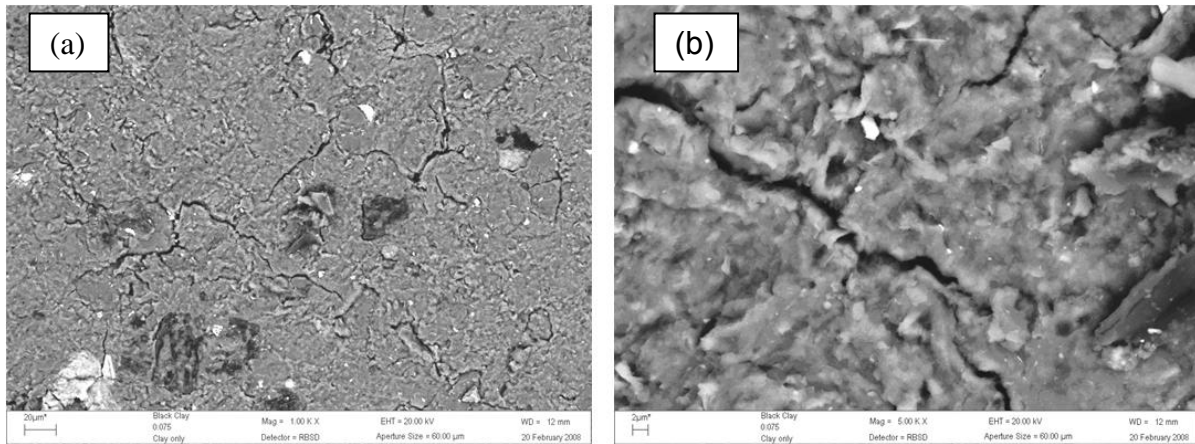


Figure 3: Overall microstructure images of (a) untreated pressed soil sample 1 (mag. 1000x), (b) untreated soil sample 1 (mag. 5000x)

Figures 4 and 5 show the microstructure image of soil 1 after treatment with the two different enzyme-based liquid stabilisers, at different magnification scales. The images reveal that treatment at this rate did not significantly change the microstructure of the sample. However, from Figure 5 at a magnification of 5000x, there is a noticeable difference in the surface structure of the samples treated with the different products. Figure 5(a) reveals a closely compact surface structure compared with that in Figure 5(b). This is interpreted as indicating that the resulting structure is dependent on the type of product.

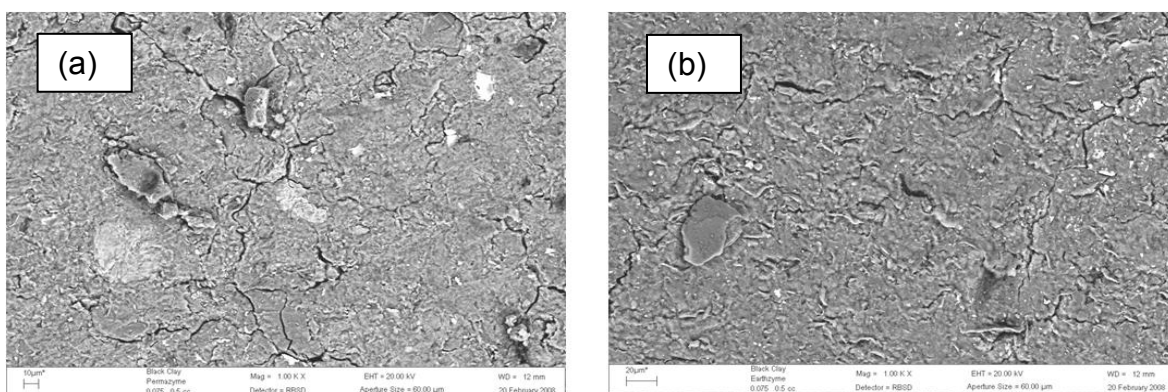


Figure 4: Microstructure images of (a) soil 1 treated with product A at 0.5cc/5l (mag. 1000x), (b) Soil 1 treated with product B at 0.5cc/5l (mag. 1000x)

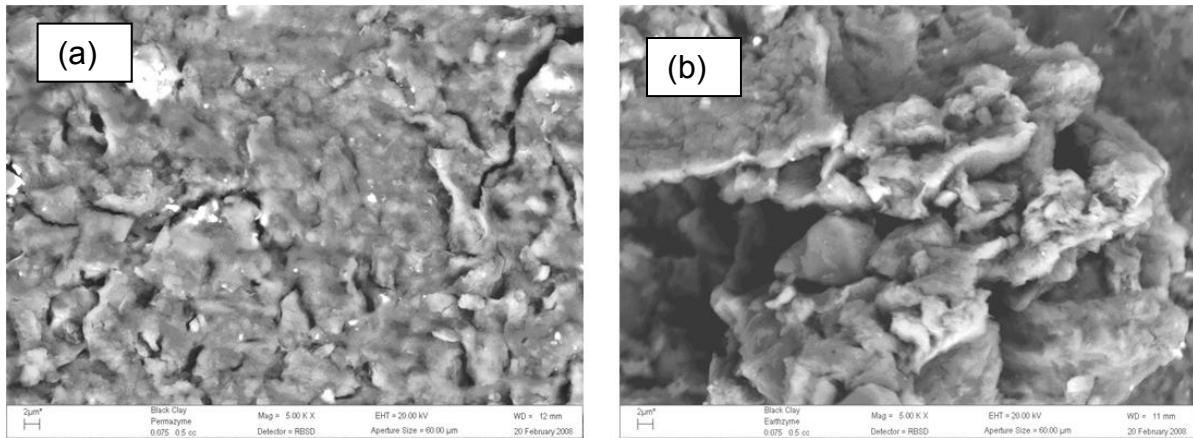


Figure 5 Microstructure images of (a) soil 1 treated with product A at 0.5cc/5l (mag. 5000x), (b) soil 1 treated with product B at 0.5cc/5l (mag. 5000x)

Figure 6 shows the surface microstructure of untreated chert, soil 2. Figure 7 shows the surface microstructure of the same material after treatment with product A in Figure 7(a) and product B in Figure 7(b). The SEM images of the untreated soil showed a generally fragmented structure compared with the structure of soil 1.

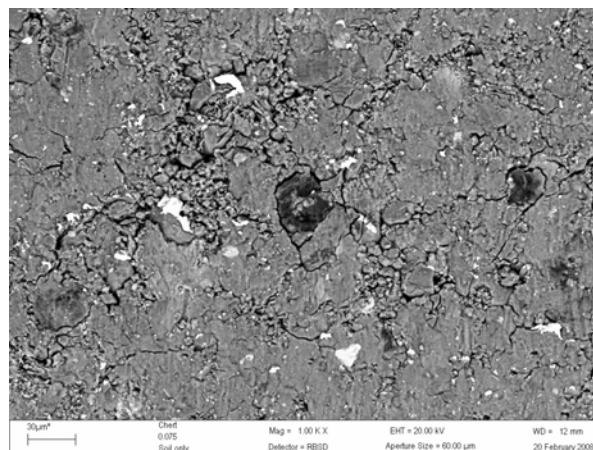


Figure 6: Overall microstructure image of untreated soil sample 2 (mag. 1000x)

The SEM micrographs in Figure 7 reveal a significant difference in the structure between the treated materials. Figure 7(a) shows the presence of tentacle-like/network structures on the surface but not for the sample treated with product B. However, the observed structures were not evenly spread. The results suggest that the bonding mechanism for product A with respect to this type of material is both chemical and mechanical.

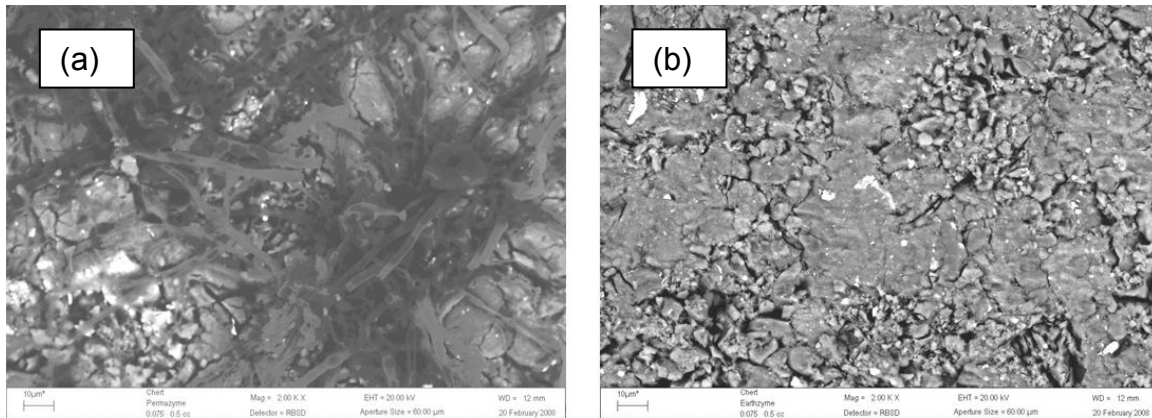


Figure 7: Microstructure images of (a) soil sample 2 treated with product A at 0.5cc/5ℓ (mag. 2000x), (b) soil sample 2 treated with product B at 0.5cc/5ℓ (mag. 2000x)

While the tablet samples were produced using three application rates, 0.5 cc/5ℓ, 1.0 cc/5ℓ and 1.5 cc/5ℓ, the effect of the rate of application is illustrated using the results of 1.5 cc/5ℓ concentration. The images in Figures 8 and 9 should be compared with the images in Figures 3(a), Figures 6 and 7. The images in Figures 8 show a marked difference from the microstructure characteristics observed at an application rate of 0.5 cc/5ℓ. It is interesting to note the presence of the tentacle-like structures on sample 1 which were absent at an application rate of 0.5 cc/5ℓ. Figure 8(a) revealed that the network structure was observed to spread evenly over the surface of soil sample 1. No such network structures were observed on the sample with treatment using product B even at this application rate. Wider micro cracks are observed in Figure 8(b).

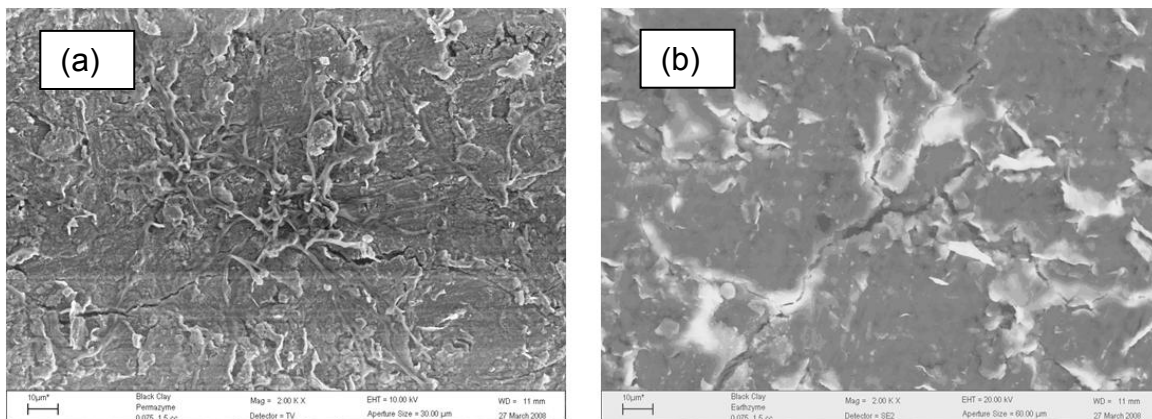


Figure 8: Microstructure images of (a) soil 1 treated with product A at 1.5cc/5ℓ (mag. 2000x), (b) soil 1 treated with product B at 1.5cc/5ℓ (mag. 2000x)

Figure 9(a) clearly shows the presence of the surface network structures on sample 2 treated with product A. Again no such network structures were revealed on sample B with treatment using product B. The images for sample 2 treated with product B generally revealed a fractured structure at the 1.5cc/5ℓ application rate.

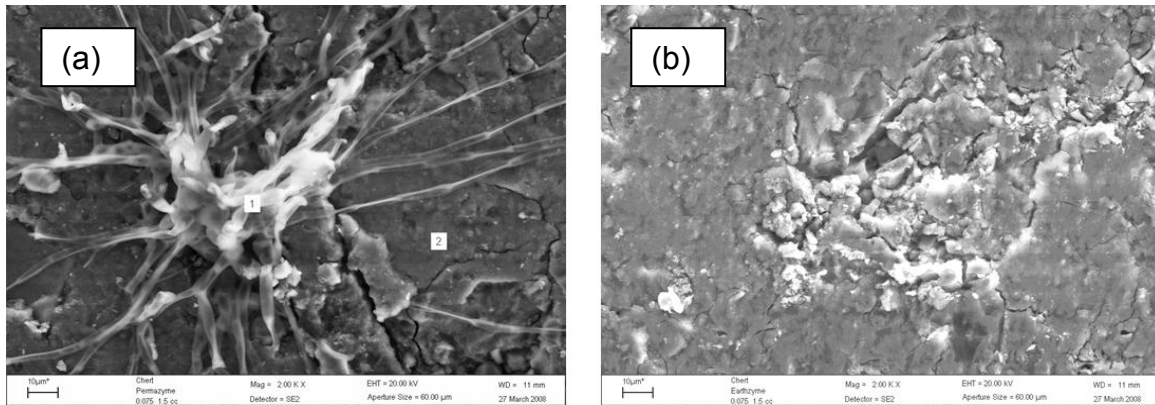


Figure 9: Microstructure image of (a) soil 2 treated with product A at 1.5cc/5l (mag. 2000x), (b) soil 2 treated with product B at 1.5cc/5l (mag. 2000x)

The results from the scanning electron microscopy analysis of the treated material show that the addition of the enzyme-based products modifies the microstructure of the material matrix, but whether this leads to significant improvement in engineering properties is another question. The images from the scanning electron microscopy analysis reveals that the type of microstructure changes that take place after treatment will be dependent on the product which in turn is a function of the compositional make up of the product itself. It was also observed that the development of the surface network structures was after a month from the time the tablet samples were manufactured. Images in Figure 8 and 9 were taken at two months. These two factors have an implication on the method of assessment of the suitability of these products. Effort should be made towards understanding the long term behaviour of these products.

3.2 Unconfined compressive strength

The unconfined compressive strength test was used to assess whether the observed characteristics at the micro-scale can be used as indicators to assist in the prediction of expected performance with respect to strength improvement following the treatment with the stabilizers. The effects of the treatment level and age on the unconfined compressive strength are shown in Figures 10 to 13

Figures 10 and 11 show the results for sample 1. It can be observed that the treatment leads to some increase in unconfined compressive strength especially for product A on soil 1. The results of the testing on soil A show some consistency in the effects of treatment based on product concentration. However, there is no significant change with time especially at the higher concentration levels. It can be observed that product A generally gives better results than product B at the same age and concentration level. The effect of B on the sample is that of reduced unconfined compressive strength after treatment. This phenomenon has been observed in the use of other liquid stabilisers and Savage (2006) has suggested an explanation for this. However, what is surprising is that the phenomenon is observed at all the application rates.

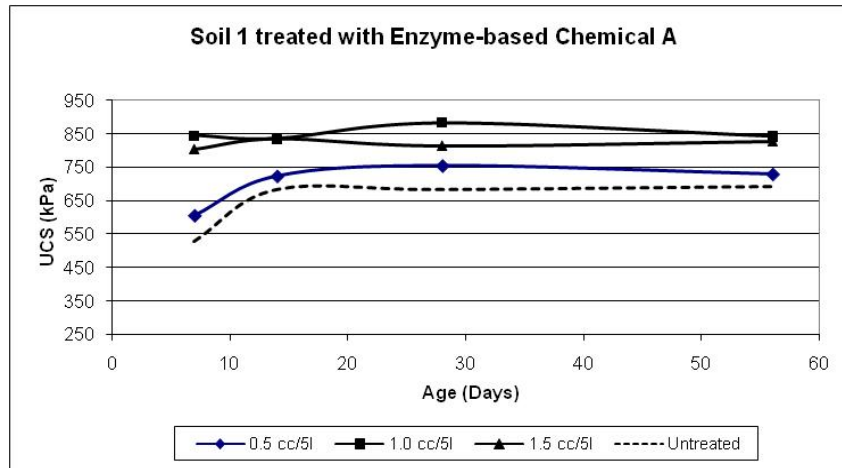


Figure 10: Effect of treatment on soil 1 with product A

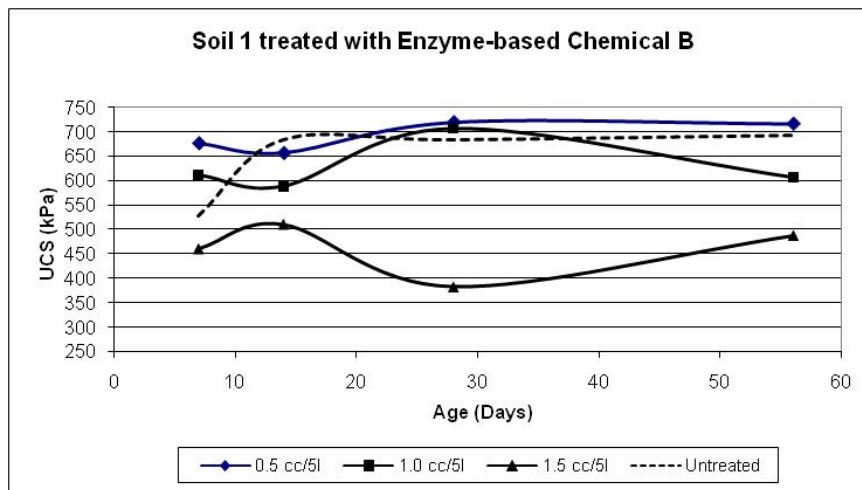


Figure 11: Effect of treatment on soil 1 with product B

Figures 12 and 13 show the results for soil sample 2. The results for samples treated with product A shown in Figure 12 are more consistent with the level of application as well as age compared with the results of samples treated with product B shown in Figure 13. Soil sample 2 was gravelly material with a lower plasticity than sample 1. The results indicate that the treatment with product A results in a more stable matrix structure than is the case with treatment using product B. It is interesting to note that the SEM images of the small samples treated with product A revealed the presence of surface network structures. It would therefore seem to indicate that the observed microstructure contributes to a more stable matrix. The treatment of sample 2 with product A clearly shows an increase in strength with time, consistent with observations made by Savage (2006), Velasquez *et al* (2006), and Visser (2007) about these products. The results show that although product B is an enzyme-based product like product A, it is not as effective as product A and this result is consistent with observations made at the micro-scale.

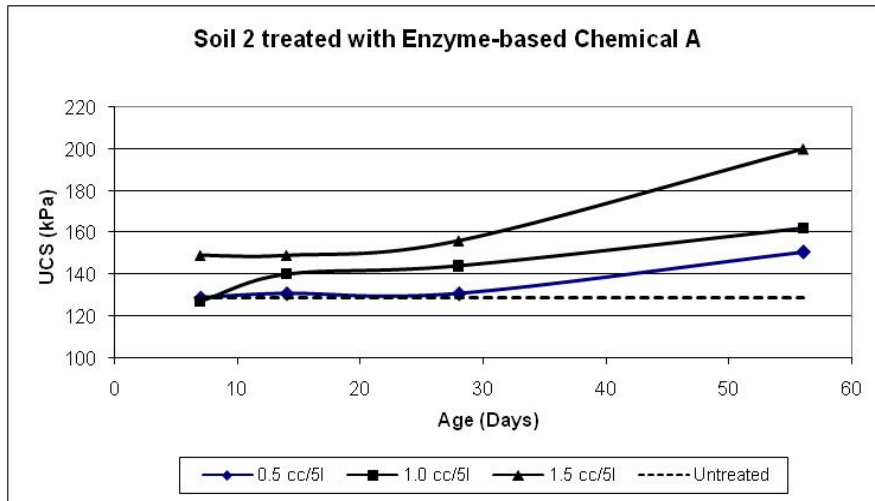


Figure 12: Effect of treatment on soil 2 with product A

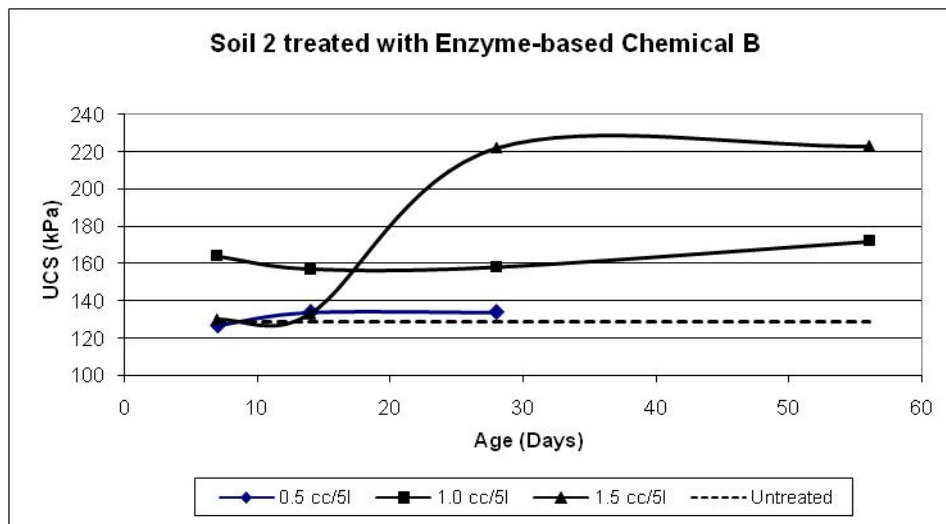


Figure 13: Effect of treatment on soil 2 with product B

Much of the commercial literature related to the enzyme stabilizers discusses their mechanism of action as being purely that of a catalyst. Scholen (1992) has suggested the stabilization mechanism of the enzymes as catalysts that speed up chemical reaction, help the soil bacteria to release hydrogen ions resulting in pH gradients on the surface of the clay particles, which assist in breaking up the structure of clay. The enzymes affect the clay lattice, initially causing them to expand and then tighten. The breaking down of the lattice causes the cover-up effect which prevents further absorption of water. The binding of clay particles is formed by aggregation. Based on the described mechanism it is therefore suggested that micro structural characteristics observed on the surfaces of the samples treated with product A is the evidence of the interaction between the soil and the product.

4 CONCLUSION

The difference in environmental conditions, between laboratory and field, due to factors such as temperature, humidity and moisture is acknowledged. But the objective of the study was to investigate the possibility of identifying material characteristics at the micro-scale that can assist in predicting the performance of treated subgrade using tests that determine properties at the macro-scale.

The results of the investigation reveal that the potential exists to achieve this, but this requires use of advanced methods of microstructure analysis. The study has demonstrated the capability of employing microstructure characterization using small samples to explain the difference in the performance of two enzyme-based products.

The study has also shown that characterizing the microstructure of small samples, prepared in a well controlled environment, and using advanced equipment provided valuable information towards understanding the behaviour at the macro-scale. The use of the scanning electron microscope has assisted in explaining the difference in the performance of two enzyme-based products. The observed connectivity of the microstructure and the presence of a surface network binding the particles, following treatment with product A differs from that seen on samples treated by product B and is considered to be the underlying explanation in the difference in performance of the two products. This is considered to be the result of the compositional characteristics of the products. The results of the study using the scanning electron microscopy image analysis revealed that the surface microstructure changes observed on the samples were time dependent, and thus could easily be overlooked when using conventional geotechnical testing methods.

The implication of this study is that the methods currently being used to assess the suitability of the enzyme-based liquid chemical stabilisers require further development to provide knowledge on binding interactions between soil and these products. Through the understanding of the mechanisms of the interaction between the soil particles and the product solutions at the micro-scale it is possible to modify these products in order to improve their effectiveness to provide significant improvement in engineering properties in a consistent manner.

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