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Microanalytical Investigation on the Consolidation Characteristic of Lime Composites

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ABSTRACT
This paper presents a series of geotechnical and geochemical tests used for investigation into consolidation behaviour of lime composites as a part of a comprehensive study in geotechnical group in Curtin Univeristy. The consolidation test was analysed the engineering characteristics of one sand-clay mixtures (pure composite) and six lime-modified samples (lime composites). Both the experimental outcomes and analytical data were justified the efficiency of lime addition for modifying the soil compressibility bihaviour. The key role of pozzolanic effect on sand composites were recognized by micrograph images of SEM, elemental examination of EDS, and mineralogical phases of XRD. It was happened due to availability of Calcium (Ca), oxygen (O), silicon (Si), aluminium (Al), as the major chemical factors for pozzolanic reactions. Subsequently, the polymerisation phenomenon was generated as a result of creation of aluminosilicate compounds, then generation a more uniform area among the composite's particles.
Keywords - Consolidation, Lime Stabilisation, EDS, SEM, XRD.

I. Introduction

One of the environmental friendly and cost-effective techniques for constructing the geotechnical projects such is soil stabilisation, [1-8]. It is performed by different methods such as mechanical stabilisation, utilisation of binders, or application of geosynthetic clay liner (GCL) in soil [1-6], [9-13]. Chemical stabilisation is the process of adjustment used to improve the properties of local soil by adding additives like lime. Normally, lime modification can improve some basic soil properties like strength and compaction together with improving the compressibility characteristics of unstabilised soil [1-6, 14-16]. As the main element, consolidation properties are considered in the wide range of soil's projects. Consolidation is a technique through water particles and voids are extruded whereby load implantation that connected with the volume of air, the soil's permeability and pozzolanic activity [1-6, 17, 18]. For minimising the settlement in civil engineering structures, the rate of soil settlement can be limited by lime modification. Pozzolanic reactions and flocculation agglomerations are two key mechanisms of lime treatment, for improving the consolidation characteristics of soil [6, 19, 20]. The polymerization process happens in consequence of the lime's pozzolanic reaction can establish either (a) calcium silicate or (b), aluminium silicate hydrates that bind the atoms on a nanometre scale[6-8, 19, 20]. The pozzolanic chemical equations are illustrated[6, 19] as:

$Ca(OH)_2 + SiO_2 \rightarrow$ C–S–H gel (a)

$Ca(OH)_2 + Al_2O_3 \rightarrow$ C–A–H gel (b)

Moreover, flocculation agglomeration plays a pivotal role in the improvement of soil performance such as plasticity, workability, soil strength, and load deformation. It is caused by dissolution of the available calcium resulting from lime in water, thereby replacing monovalent hydrogen ions (OH_) with divalent calcium ions (Ca^{2+}). This process leads to a reduction in the soil's water absorption capacity, controls the separation between soil layers, and allows the establishment of larger soil units, [1-8, 19]. The stabilisation of diverse soils has been examined by many researchers; however, it seems that the obtained results are diverse and need to be considered further [21-23]. Studies on lime show that the main achievement of lime treatment is in the development of the soil's resistance. Nevertheless, after adding a specific amount of lime, this tendency towards resistance was actually reversed by adding greater and greater dosages of lime to the soil [1-6, 19, 21, 24]. Based on the Eades and Grim method,[19, 24] research was carried out by pH evaluation in order to assess the optimum dosage of lime needed to form a lime composite. Results showed that the range of lime dosage would be between 4% and 8% if the pH value was approximately 12.4[6, 19]. Other studies report that varying amounts of lime: 2.5% - 5% [19] and 1%-3%, [6, 21, 23, 25] are required for archiving the same amount of pH.

Therefore, with regard to the diverse results and limited studies on the geotechnical and chemical aspects of lime's efficacy on sand composites the necessity of more research in this area was recognized.

As a part of a comprehensive project in geotechnical group in Curtin Univeristy this study [1-6] was carried out to investigate on engineering and chemical mechanism of lime modification from a microanalytical viewpoint. In addition of a series of consolidation tests which were assessed the geotechnical efficiency of lime application, Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD) was used to analyse the chemical, mineralogical and elemental characteristics of lime composite. This paper is extracted from a master thesis of first author.

II. Material and processing method

2.1 Material characteristic

The soil samples used in this research were prepared by mixing the Baldivis sand and the kaolin clay. The hydrated lime was used as a stabiliser [6].

2.2 Sample preparation

The same proportion of sand and clay was used for preparing the sand-clay composite and six modified samples were then treated with different dosages of lime (i.e., 1%-3%). The reason of using mix of sand and clay has been due to lack of research on mixed soils. Samples were saved with a curing time of one hour at 22.5 ºc [6].

2.3 Characterisation techniques

A series of microscopic analyses was undertaken to study the physical, chemical, elemental and mineralogical reactions between lime and material particles in pure composite, and lime composite. Investigation into the microstructural characteristics of the one pure combination specimen and six lime mixtures (i.e., 0.5% lime-3% lime) was carried out by SEM, EDS, and XRD tests [1-6].

2.4 Consolidation Test

In accordance with ASTM D2435, an automatic consolidation device was carried out a series of tests on the saturated and partially saturated samples.Usual practice of consolidation tests followed and the results were analysed based on the “void ratio-effective stress curves” and “settlement- log time curves” [4, 6, 8]. Some parts of this data are achieved in previous researches in this area [4, 6].

III. Result and discussion

3.1 Laboratory results

Table I presents the obtained results of one-dimensional consolidation tests. In general, lime modification reduces the consolidation-swelling behaviour of pure compounds. The initial void ratio, compression index, swell index was considered for evaluating the lime efficiency.

Moreover, Table I illustrates that by adding 0.5% of lime to a pure sample, there was a remarkable decrease in the compression index of the soil, which dropped to $2.6{*}10e^{-2}$. Then, the modified sample with 3% lime had the lowest settlement by $9.9{*}10e^{-3}$ amount. furthermore, as this composite had the minimum amount of initial void ratio as well as the lowest coefficient of consolidation, whereas in the other samples, the unmodified sample had the maximum C_v and the highest e_0[6].

Table I
Consolidation test result (C_c: Compression Index, C_s: Swelling Index, E_0: Initial void ratio, C_v: Coefficient of consolidation) L: Lime [4, 6].

Specimens (%)	**C_C**	**C_S**	**e_0**	**C_V (cm^^{2}) / sec**
0 L	$4.6{*}10e^{-2}$	$1.6{*}10e^{-2}$	0.52	$8.20{*}10e^{-5}$
0.5 L	$2.6{*}10e^{-2}$	$1.4{*}10e^{-2}$	0.46	$8.14{*}10e^{-5}$
1 L	$2.1{*}10e^{-2}$	$1.3{*}10e^{-2}$	0.41	$8.02{*}10e^{-5}$
1.5 L	$1.7{*}10e^{-2}$	$1.1{*}10e^{-2}$	0.38	$7.56{*}10e^{-5}$
2 L	$1.5{*}10e^{-2}$	$9.1{*}10e^{-3}$	0.37	$7.455{*}10e^{-5}$
2.5 L	$1.2{*}10e^{-2}$	$7.4{*}10e^{-3}$	0.36	$7.450{*}10e^{-5}$
3 L	$9.9{*}10e^{-3}$	$5.8{*}10e^{-3}$	0.36	$7.35{*}10e^{-5}$

In the area of swell properties of pure combination, the same tendency can be observed with the compression index results of lime treated sand. Table I shows that the swelling index was reduced by incrementing the dosage of lime, with the minimum amount of C_s reaching $5.8{*}10e^{-3}$ in 3% lime treated samples [6].

3.2 SEM and EDS investigation

Comparisons of the microstructural scale of untreated composite and lime- modified mixtures were carried out with scanning electron microscopy (SEM) micrographs. The micrographs of selected materials (i.e., lime, untreated and treated samples) were accurately analysed for any changes in the microstructure of lime-treated composites for evaluating the chemical effects of lime [6]. The surface characteristics of lime are illustrated by SEM micrograph, Fig 1.

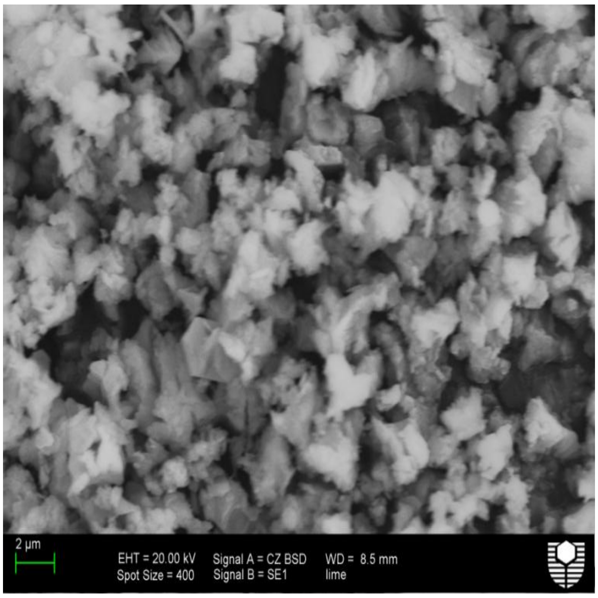

Fig. 1; SEM micrograph of lime particle with 8.5*mm* working distance and 20*kv* voltage energy at 2μm.

The micrograph of the modified compound is shown in Fig 2. The black areas on the micrographs represent voids in the samples. As is shown in Fig 2 the microstructure of the lime composite is completely dense and could establish a flat area between the microstructures of the atoms in the material. In the case of the lime treatment, the amount of calcium compound was very apparent on the lime-modified micrograph, and the sample's microstructure was comparatively dense. Fig 2 reveals that the calcium in the lime created a composite whit soil particle which can be seen in the micrograph images, shown as a bright polygon form around the soil's atoms [6].

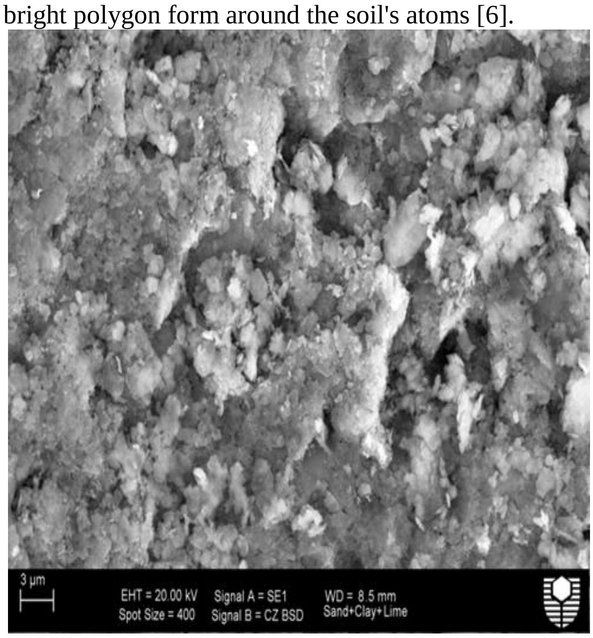

Fig. 2; Secondary and backscattered electron SEM micrograph of lime composite at 8.5*mm* working distance and 20*kv* voltage energy at 3μm.

For identifying the elemental and chemical properties of composites, EDS was presented the unique atomic structure of lime, untreated composite and lime-modified by line identification and X-ray mapping data. As shown in Fig 3 calcium (Ca) is known as the most dominant element of lime components compared to oxygen (O), silicon (Si), aluminium (Al), iron (Fe) and magnesium (Mg)[6].

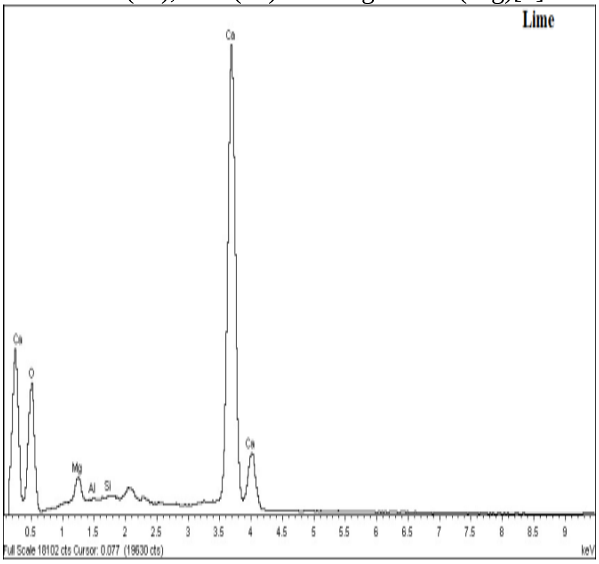

Fig. 3; Hydrated lime's line identification. X-ray-Ray spectroscopy indicates the domination of Calcium among other components such as oxygen (O), aluminium (Al), magnesium (Mg), and silicon (Si).

On the other hand, the EDS spectrum of pure combination in Fig 4, and lime-treated samples in Fig 5, presents the difference between chemical composition in modified and unmodified mixtures due to the effects of the addition of lime. It is clearly obvious that in pure composites the low dosage of calcium could play a pivotal role in reducing the possibility of a pozzolanic reaction due to the non-creation of calcium silicate or aluminium silicate hydrates [6].

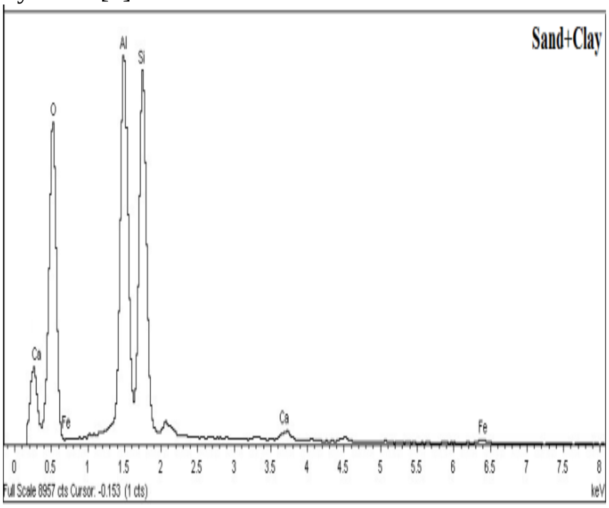

Fig. 4 clay-sand composite's line identification of X-ray-Ray spectroscopy indicates the small peak that corresponds to ca.

Both EDS patterns of untreated and lime-modified samples indicate peaks corresponding to Al, Si, Ca, O, and Fe. However, an increase in the number of peaks that correspond to Ca, the appearance of the Mg peak, and increments in the levels of other peaks is revealed as a result of composite-modification by lime. Directly associated with the increments is the chance of the establishment of the C–S–H gel and C–A–H gel as a part of the pozzolanic effect.

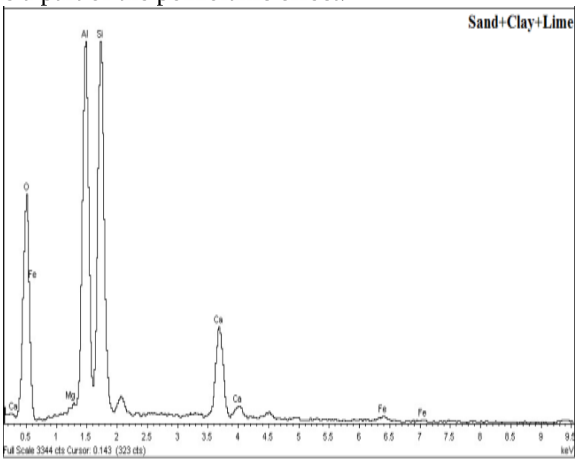

Fig. 5 Lime composite's line identification of X-ray-Ray spectroscopy shows a high level of chemical elements in the lime composite.

As is shown by Fig 5, Ca characteristics peak at 4 *keV*, Mg characteristics peak at 1.3 *keV*, and Fe characteristics peak at 7 *keV* and this peaking appears to be due to lime-modification. These changes directly increase the chance of a pozzolanic reaction by increasing the ratio of chemical elements in the composite [6].

3.3XRD analysis

The mineralogical analysis of the material plays a pivotal role in determining the changes in the mineralogical phases of pure composite due to pozzolanic reactions. The XRD graphs of the lime, clay, sand, sand-clay mixture, and the lime treated mixture of sand-clay are presented in Fig 6[6].

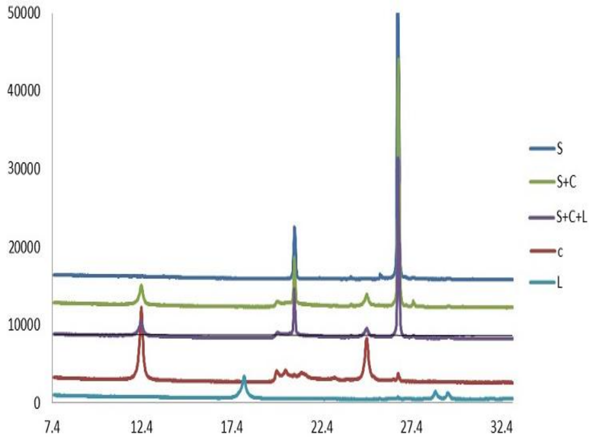

Fig.6 X-ray diffraction patterns for materials illustrate the mineralogical phases of the material. The XRD results show the position of the reflection peaks.

The remarkable diffraction lines were presents specific characteristics of each material. Some compounds like Aragonite and Calcite [$CaCO^3$], Periclase [MgO], Portlandite [Ca(OH)] and Quartz [SiO^2] were detected as main component of lime. "In addition, the XRD data was confirmed the reaction among lime atoms by detecting the Ettringite crystallized phase hydrates [$3CaO\text{-}Al^2O^3\text{-}3CaSO^4\text{-}32H^2O$] [6, 19, 26]. Furthermore, due to reaction between sand's quartz particles and aluminosilicate of clay the diffraction peaks of C–S–H hydrate became higher [6,19]. Then, the pozzolanic reaction could trigger on by hydrate of calcium silica and aluminium silica.

On the other hand, as illustrated, kaolin clay was identified by two main peaks at 12.54 and 25.05 2θ angles. Furthermore, with regard to the presence of quartz minerals in sand particles, two strong diffraction lines can be observed at 20.85 and 26.58 2θ angles [6].

IV. Conclusion

This study has been part of a big project in geotechnical group in Curtin Univeristy. Two aspects of consolidation (i.e. Odometer test and material characteristic tests) were investigated. Regarding the utilisation of the lime stabilisation in many of construction projects, as well as geotechnical aspects, the elemental, mineralogical and chemical mechanisms of this process were analysed. Investigation was conducted into the aspects of the specimens' microstructure by Scanning electron microscopy (SEM) micrographs, components were examined by Energy-dispersive X-ray spectroscopy (EDS), composition was examined by X-ray diffraction (XRD). The following conclusions can be drawn despite of some of the possible limitations of the laboratory tests.

From a geotechnical point of view, the results obtained for the consolidation and swelling properties of the pure composite indicate the remarkable improvement in the compressibility and swelling behaviour of the lime combination. In all treated specimens, the efficiency of the addition of lime was observed in reducing the compression index and swelling index of samples. The results were achieved in the range of $4.6{*}10e^{-2}$ _$9.9{*}10e^{-3}$ for C_c, $1.1{*}10e^{-2}$_$4.9{*}10e^{-3}$ for C_s, and $8.27{*}10e^{-5}$_ $7.35{*}10e^{-5}$(cm^2) / sec for C_v.

Based on the SEM/ EDS results, a more uniform area was created between the micro-particles of the atoms in the composite. By the inclusion of lime the calcium compound was produced. Increasing the ratio of calcium (Ca), oxygen (O), silicon (Si), aluminium (Al), iron (Fe), and magnesium (Mg) in the lime-modified mixture was directly associated with the creation of aluminosilicate components. Then, it was established the pozzolanic reaction [6].

The XRD data justified the establishment of pozzolanic reaction because of combination between

calcium and specimens particle after creation of calcium silicate and aluminium silicate hydrates.
Thus, the present research has demonstrated that lime plays a key role in improving the properties of the composite's materials from an elemental, chemical and mineralogical viewpoint.

V. Acknowledgment

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