

Mineralogical development in a lime treated clayey sand soil

BABA MUSTA¹, KHAIRUL ANUAR KASSIM¹ AND MOHD. RAZMAN SALIM²

¹Department of Geotechnic, Faculty of Civil Engineering
UTM 81310 Skudai, Johor, Malaysia

²Department of Environment, Faculty of Civil Engineering
UTM 81310 Skudai, Johor, Malaysia

Abstract: Three samples of lime treated clayey sand soils and a control sample were cured for one month at room temperature before being analysed using X-ray fluorescence (XRF), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The XRF data shows high abundances of SiO₂ (73.57%-80.45%), Al₂O₃ (10.77%-11.09%), L.O.I (4.38%-7.38%), Fe₂O₃ (2.32%-2.88%), K₂O (0.88%-1.03%) and CaO (0.03%-4.20%), whereas the other major elements are lower than 1.00%. The treated samples with 2% and 6% of lime show an increasing concentration of CaO about 1.41% and 4.20% respectively. The X-ray diffractograms and scanning electron micrographs detected the appearance of quartz and kaolinite in the control soil as well as in the treated soil samples. The development of new cementitious minerals in treated soil appear in low intensities in the X-ray diffractograms, due to their low crystallization. Scanning electron micrographs also show the development of new cementitious minerals, and modification of the surface micromorphology of the treated soil due to increasing concentration of lime.

Abstrak: Tiga sampel tanah pasir berlempung yang dirawat dengan kapur dan satu sampel kawalan telah diawet selama sebulan dalam suhu bilik sebelum dianalisis dengan menggunakan kaedah pendarflour sinar-X (XRF), pembelauan sinar-X (XRD) dan mikroskopi elektron imbasan (SEM). Data XRF yang diperolehi menunjukkan unsur-unsur yang melimpah ialah SiO₂ (73.57%-80.45%), Al₂O₃ (10.77%-11.09%), L.O.I (4.38%-7.38%), Fe₂O₃ (2.32%-2.88%), K₂O (0.88%-1.03%), manakala unsur-unsur major yang lain adalah kurang daripada 1.00%. Sampel-sampel yang telah dirawat dengan 2% dan 6% kapur pula menunjukkan kelimpahan CaO masing-masing meningkat lebih kurang 1.41% dan 4.20%. Difraktogram sinar-X dan mikrograf elektron imbasan mengesan kehadiran kuarza dan kaolinit dalam sampel kawalan dan juga dalam tanah yang telah dirawat. Pembentukan mineral bersimen yang baru dalam tanah yang telah dirawat ditunjukkan oleh keamatan rendah dalam difraktogram sinar-X, ini disebabkan oleh penghabluran yang sedikit. Mikrograf elektron imbasan juga menunjukkan pembentukan mineral bersimen yang baru, dan telah mengubah morfologi mikro tanah yang telah dirawat hasil daripada peningkatan kepekatan kapur.

INTRODUCTION

Lime is made from high-calcium or high-magnesium limestone. Three main processes of lime production are stone preparation, calcination and hydration (Boynton, 1966; O'Driscoll, 1988). In geoenvironmental engineering, quick lime (CaO) or hydrated lime (Ca(OH)₂) is used as a cementing agent to capsule contaminants via stabilisation processes. These processes thus reduce the risk posed by waste by converting the contaminants into a less soluble, mobile or toxic form. Clay minerals in soil will react with Ca²⁺ to produce cementitious minerals. The rate of cementitious mineral formation in different systems may vary greatly depending on the phase serving as the source of Al³⁺ (Odler and Colan-Subaurte, 1999). The crystal structure of the minerals can be identified using SEM and X-ray diffraction (Thaulow *et al.*, 1996). A study by Beaudoin *et al.* (1998) shows that the minerals are dependent on porosity, CaO/SiO₂ ratio, and ratio of the CaO-SiO₂-H₂O. Ford *et al.* (1999) in their study showed that the weathering of clay minerals and the time-dependent release of Al and Si ions controlled precipitate nucleation and

transformation of heavy metals such as nickel. The aim of this paper is to describe the distribution patterns of major elements and the mineralogical development of cementitious minerals on clayey sand soil treated with different concentration of lime.

METHODS OF STUDY

A clayey sand soil from sedimentary rocks of the Crocker Formation was collected from Kg. Kayu Madang, Telipok, Sabah (Figure 1). Deep weathering of the bedrock had produced a thick soil profile. The hand augered sample was brown yellowish in color. Physical properties of the soil sample are given in Table 1. The moisture content and pH were measured immediately upon arrived in the laboratory. The content of clay, silt and sand is 32.4%, 21.6% and 46.0% respectively, whereas the plasticity index is 26%. The soil is best classified as clayey sand.

The hydrated lime (Ca(OH)₂) sample was taken from Pasir Gudang, Johor. Different percentages of hydrated lime of 2%, 6% and 8% by weight respectively were added to the soil samples in plastic bags before curing at room

temperature for one month. The plastic bags were closed tightly to avoid any water loss and to maintain a constant moisture during the curing process.

At the end of the month, the control and treated soil samples were dried in an oven at 104°C. The samples were then ground into powder before being analysed. X-ray fluorescence (XRF) with fused discs was used to determine the major elements (Norrish and Hutton, 1969). A "Philips PW 1480 X-ray Digital" instrument controlled by Digital Software X 44 microcomputer software was used for this purpose. The calibration graph followed that of the 'Alphas on line' program (De Jongh, 1973, 1979). For mineralogical studies of the samples, X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed. The XRD analysis was carried out using a Siemens X-ray diffractometer with a scan speed of 1° per minute. Dried-powdered samples were used for XRD analysis of both the control sample and the treated samples. SEM was carried out using a Philips XL40 model with a pressure of 60 psi and a voltage of 10-20 kV. The fresh surfaces of control and treated sample with 2%, 6% and 8% of lime were spattered with a thin film of gold to eliminate any excess charge from the electron beam. Energy dispersive X-ray spectra (EDX) were obtained when necessary to confirm the identification of cementitious minerals.

RESULTS AND DISCUSSION

Geochemical Patterns

The results of XRF analyses for major elements in the soil sample, the soil treated with 2% of lime, the soil treated with 6% of lime and the lime sample are presented in Table 2. The XRF data shows that the major elements in the lime treated soils and the control sample are similar. The abundance of elements in order from high to low are SiO₂ (73.57%-80.45%), Al₂O₃ (10.77%-11.09%), L.O.I (4.38%-7.38%), Fe₂O₃ (2.32%-2.88%), K₂O (0.88%-1.03%) and CaO (0.03%-4.20%), whereas the other major elements are less than 1.00%. Otherwise, the treated samples with 2% and 6% of lime show an increased concentration of CaO of about 1.41% and 4.20% respectively. From the table it can be seen that the hydrated lime is high in CaO concentration (68.46%), followed by MgO (2.09%). The loss on ignition (L.O.I) increases with increasing of lime content. The increase in L.O.I is contributed by the water from the hydrated lime (Ca(OH)₂).

The geochemical data indicates that the high concentrations of SiO₂ in all samples is due to the contribution of quartz (SiO₂), kaolinite (Al₂Si₂O₅(OH)₄) and montmorillonite (Al₂O₃·4SiO₂·xH₂O). Kaolinite and montmorillonite contributed for the high concentration of Al₂O₃. Both SiO₂ and Al₂O₃ from the minerals mentioned above are believed to be the main source of Al³⁺ and Si⁴⁺ respectively for the development of cementitious minerals.

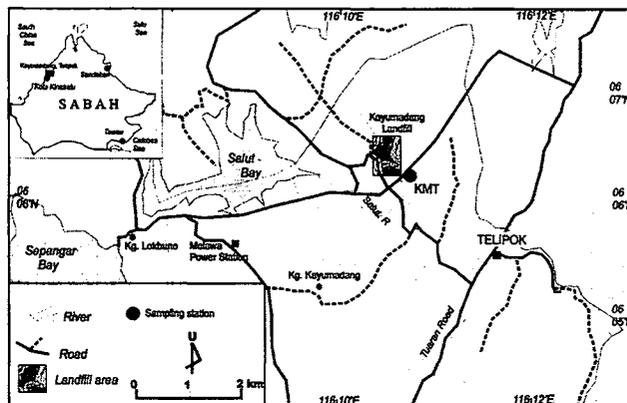


Figure 1. Map of the area of study showing the sampling station near the Kayumadang landfill area.

Table 1: The physico-chemical of soil from Kayumadang Landfill, Telipok Sabah.

| Physico-chemical properties | Value | Physico-chemical properties | Value |
|-----------------------------|-------|----------------------------------|-----------------------|
| Moisture Content (%) | 13.7 | Specific Gravity | 2.50 |
| Liquid Limit | 51 | Permeability (m/s) | 2.27x10 ⁻⁸ |
| Plastic Limit | 25 | Dry Density (Mg/m ³) | 1.73 |
| Plasticity Index (%) | 26 | W _{opt} (%) | 14 |
| Clay (%) | 32.4 | pH | 4.66 |
| Silt (%) | 46.0 | | |
| Sand (%) | 21.6 | | |

Mineralogy

X-ray diffractograms show high abundances of quartz and kaolinite, whereas montmorillonite is present as a minor mineral (Figure 2). The average of two theta (2θ) and crystal lattice spacing of minerals obtained by XRD in order from the high intensity to low intensity is given in Table 3. Quartz is originated from the parent rock and is relatively resistant to weathering (Ollier, 1969). The kaolinite is a secondary mineral produced from weathering of sedimentary rocks.

The X-ray diffractograms of the treated clayey sand soil with 2% of lime and 6% of lime are shown in Figure 2 (B and C). In Figure 2 (D; zooming peaks of soil treated with 2% of lime), new mineral development is indicated by new peaks with low intensity due to low crystallisation. When compared with the study by Rajasekaran and Rao (1998) on a lime treated clay soil, it is considered that CASH (calcium aluminate silicate hydrate), CSH (calcium silicate hydrate) and CAH (calcium aluminate hydrate) minerals were developed in this study. If this is true, then the reactions between lime with silica, lime with alumina and lime with clay minerals to give rise to cementitious minerals in this study would be similar to the reactions of the Transportation Research Board (1976) as shown below:

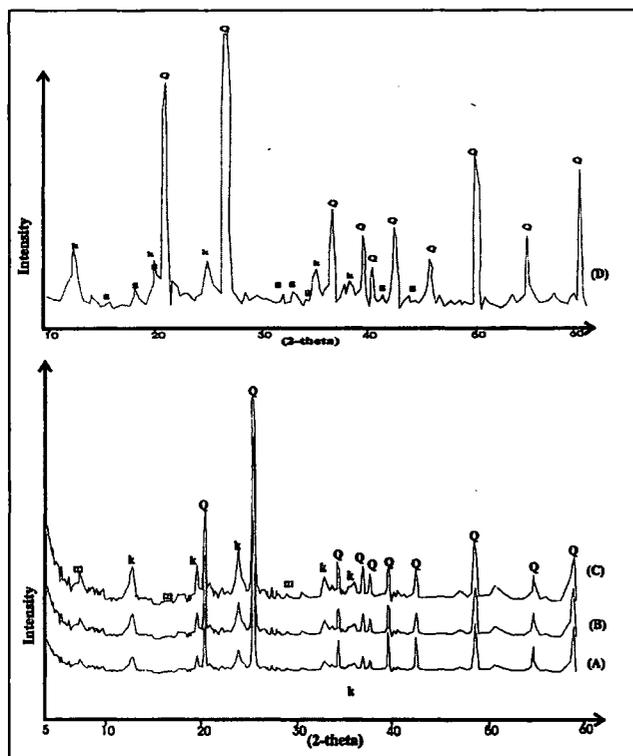
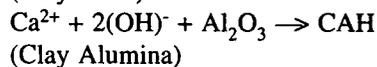
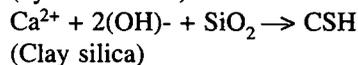
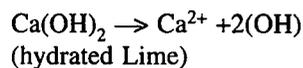


Figure 2. X-ray diffractograms of (A) untreated clayey sand soil; (B) treated with 2% of lime; (C) treated with 6% of lime and (D) soil treated with 2% of lime with low intensity of cementitious mineral enhanced by the zooming of peaks. Q: quartz; k:kaolinite; m:montmorillonite and g: cementitious mineral.



Morphological Patterns

The scanning electron micrographs in Figure 3a shows the morphology of kaolinite and quartz in the original soil. The photomicrograph also shows the appearance of pore spaces, as indicated by the dark background mostly at the edge of the minerals. Cementitious minerals are scattered on the surface and at the edges of the original minerals on the 2% lime treated clayey sand soil (Figure 3b). This results in modification of the micromorphology of the mineral surface (Figure 3b). The micrograph also shows the development of cementitious minerals in kaolinite. Figure 3c shows cementitious mineral produced at the edge and at the surface of the original mineral (6% of lime). This phase of cementitious minerals has marked similarities with the calcium silicate hydrated phase reported by Stephen *et al.* (1999). Figure 3d shows the similarity of the cementitious minerals with the flocculated and agglomerated sample treated with 8% lime. The flocculated phenomena at the surface of minerals at the same time could change the volume of micropore space, which could

Table 2: The abundance of major elements in clayey soil (KMT0%), soil treated with 2% of lime (KMT2%), soil treated with 6% of lime (KMT6%) and lime. Note: *bdl= below detection limit

| Major Elements | KMT 0% | KMT 2% | KMTC 6% | LIME |
|------------------------------------|--------|--------|---------|--------|
| SiO ₂ | 80.45 | 77.10 | 73.57 | bdl |
| TiO ₂ | 0.47 | 0.51 | 0.49 | 0.01 |
| Al ₂ O ₃ | 10.77 | 11.09 | 10.56 | 0.14 |
| Fe ₂ O ₃ (T) | 2.32 | 2.88 | 2.76 | 0.11 |
| MnO | 0.00 | 0.00 | 0.00 | 0.02 |
| MgO | 0.37 | 0.16 | 0.19 | 2.09 |
| CaO | 0.03 | 1.41 | 4.20 | 68.46 |
| Na ₂ O | 0.15 | bdl | bdl | bdl |
| K ₂ O | 1.03 | 0.93 | 0.88 | 0.02 |
| P ₂ O ₅ | 0.02 | 0.01 | 0.03 | 0.02 |
| L.O.I | 4.38 | 5.96 | 7.38 | 29.14 |
| TOTAL | 99.99 | 100.05 | 100.06 | 100.01 |

increase the permeability of the treated soil.

CONCLUSION

The XRF data shows that the major elements in lime treated soil and the control sample are similar. The abundance of elements in order from the high abundance to low abundance are SiO₂ (73.57%-80.45%), Al₂O₃ (10.77%-11.09%), L.O.I (4.38%-7.38%), Fe₂O₃ (2.32%-2.88%), K₂O (0.88%-1.03%) and CaO (0.03%-4.20%), whereas the other major elements are lower than 1.00%. The treated samples with 2% and 6% of lime shows an increasing concentration of CaO about 1.41% and 4.20% respectively. Loss on ignition (L.O.I) increases with increasing lime content due to the contribution of water from the hydrated lime.

The X-ray diffractograms show the presence of quartz and kaolinite in the clayey sand soil. These minerals contribute to the high contents of SiO₂ and Al₂O₃ as detected by XRF. The development of new cementitious minerals is also detected by the XRD but has low intensity due the low crystallisation.

The scanning electron micrographs showed the formation of new cementitious mineral from soil treated with lime. The micromorphology of the original soil was modified due to the flocculation of cementitious minerals, which could increase its permeability.

ACKNOWLEDGEMENT

This research was funded by the IRPA Grant Coded 72277. The authors would like to thank Mr. Abd. Aziz Ngah from Program Geology, UKM for providing XRF analyses. Mr. Azhari Ibrahim from Program Geology, UKM and Mr. Zainal Abidin from Faculty of Mechanical, UTM for helping in the XRD analyses. The authors are grateful to Mr Nazri from Faculty of Mechanical Engineering, UTM for the SEM analyses.

Table 3. The minerals content in soil samples showing the value of 2 theta and d-spacing using X-Ray Diffraction.

| Minerals | 2-theta (radian) | d-spacing (\AA°) |
|-----------------|-----------------------------------|----------------------------------|
| Quartz | 26.70, 20.93, 42.52, 36.62, 39.52 | 3.34, 4.24, 2.12, 2.45, 2.28 |
| Kaolinite | 12.40, 24.88, 19.96, 34.84 | 7.13, 3.58, 4.45, 2.57 |
| Montmorillonite | 6.18, 28.73 | 14.28, 3.11 |
| CASH | 18.03, 32.68, 34.11, 41.31, 15.58 | 4.91, 2.74, 2.63, 2.18, 5.68 |

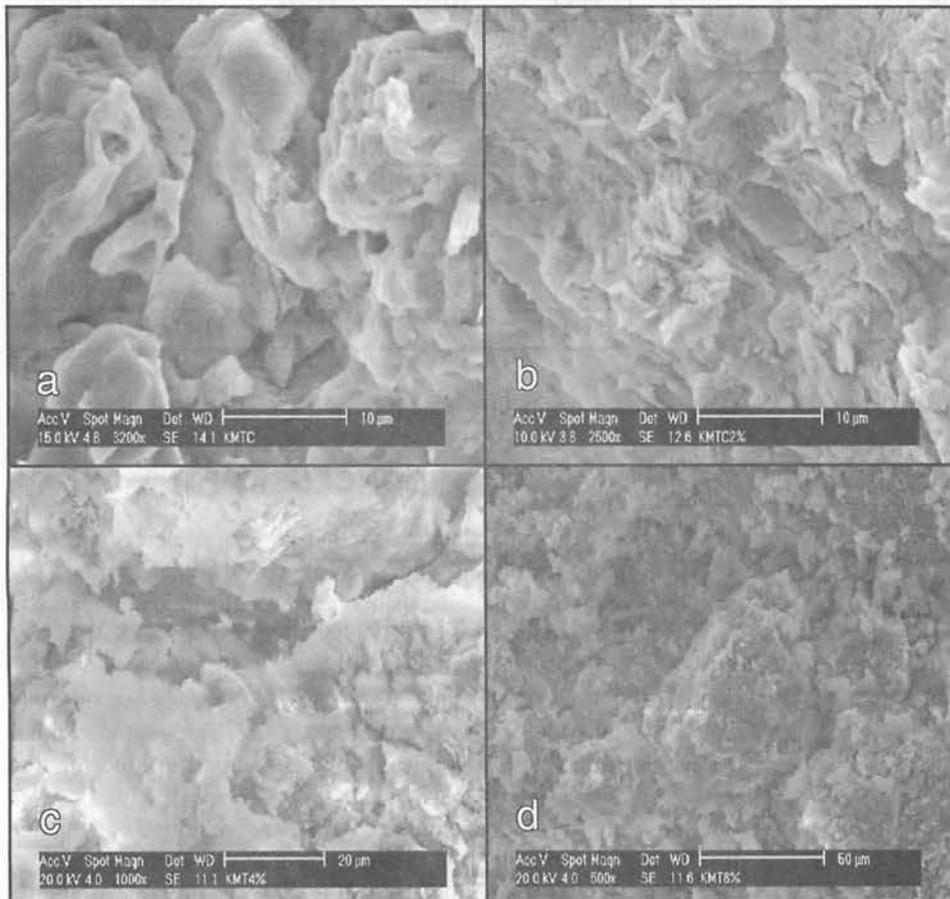


Figure 3: The scanning electron micrographs of (a) original clayey soil (b) treated with 2% of lime (c) treated with 4% of lime and (d) treated with 8% of lime.

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