Geological Society of Malaysia Annual Geological Conference 2002
May 26–27 2002, Kota Bharu, Kelantan, Malaysia

Effect of lime on permeability and microstructure of soil

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Abstract: Unstabilised and stabilised clayey sand soil with 6% of lime were cured in 190 mm and 100 mm diameter of cylindrical plexy-glass mould for 4 weeks to study the effect of lime on permeability and microstructure of the soil. The permeability of soils were measured for every 1 pore volume (PV) solution by falling the head method during the leaching test. The leaching test was conducted until 7 PV solutions. Scanning Electron Microscopic (SEM) was used to study the microstructures of both soils before and after leaching tests. The initial permeability of stabilized soil is typically lower compared to the unstabilised soil after curing for 4 weeks. The permeability of unstabilised soil samples was 7.02 x 10^{-9} m/s and the stabilised soil was 2.40 x 10^{-9} m/s. The unstabilised samples show the immediate decrease of permeability to 1.85 x 10^{-9} m/s with leaching 2 PV leaching solutions, whereas the stabilized samples show the immediate decrease of permeability to 1.86 x 10^{-10} m/s after 1 PV leaching solution. Further increase in PV values almost maintained the permeability of stabilized and unstabilised soils with average values of 1.42 x 10^{-10} m/s and 2.33 x 10^{-9} m/s respectively. The phenomenon of decrease of permeability is due to the clogging of fine particles in pore space and formation of cementitious minerals. The scanning electron micrographs showed the structure of layered kaolinite, angular shape of quartz and high pore space in the unstabilised soil. After leaching at 7 PV solutions, the unstabilised soil at the top layer indicated packed microstructure and good reorientation of clay particles. Whereas, the structures at the bottom layer showed a more packed structure, flocculated and with low pore space. The scanning electron micrographs showed the formation of cementitious mineral in stabilized soil. After leaching with 7 PV solutions, the dissolution of cementitious minerals occurred and formed new channel. However, the dense cementitious minerals at the bottom layer were flocculated, link with one another and clogged up the fine particles in pore spaces. The test result indicates that addition of lime could modify the microstructure and reduce the permeability of the soil.

Abstrak: Tanah pasir berlempung yang tak distabilkan dan tanah yang distabilkan dengan 6% kapur dalam bekas selinder pleksi-kaca berukuran 190 mm dan diameter 100 m telah diawet selama empat minggu, bertujuan menentukan kesan kapur ke atas ketelapan dan struktur mikro tanah tersebut. Ketelapan tanah yang ditentukan dengan kaedah turus menurun ditentukan pada setiap 1 isipadu pori (PV) larutan semasa ujian larut lesap. Ujian larut lesap dijalankan sehingga 7 PV larutan. Kajian mikroskopik pengimbas elektron (SEM) pula telah digunakan untuk mengkaji struktur mikro kedua-dua tanah sebelum dan selepas ujian larut lesap. Ketelapan awal tanah yang distabilkan adalah rendah berbanding tanah yang tak distabilkan selepas di awet selama 4 minggu. Ketelapan bagi tanah tak distabilkan ialah 7.02 x 10^{-9} m/s dan tanah yang distabilkan ialah 2.40 x 10^{-9} m/s. Sampel tidak distabilkan menunjukkan pengurangan dengan pantas nilai ketelapan menjadi 1.85 x 10^{-9} m/s setelah dilarutlesapkan dengan 2 PV larutan. Manakala sampel yang distabilkan pula menunjukkan pengurangan ketelapan dengan pantas kepada 1.86 x 10^{-10} m/s setelah larut lesap 1 PV larutan. Peningkatan nilai PV seterusnya menunjukkan nilai ketelapan dikekalkan pada 1.42 x 10^{-10} m/s dan 2.33 x 10^{-9} m/s masing-masing bagi tanah distabilkan dan tanah tak distabilkan. Fenomena pengurangan nilai ketelapan adalah disebabkan oleh halangan butiran halus dan pembentukan mineral bersimen. Mikrograf pengimbas elektron menunjukkan struktur berlapis kaolinit, butiran bersudut kuarza dan ruang pori yang tinggi dalam sampel tanah yang tak distabilkan. Sampel tanah tak distabilkan selepas 7 PV pada lapisan atas menunjukkan struktur mikro yang padat dan butiran lempung yang teratur dengan baik. Manakala struktur lapisan bawah pula menunjukkan struktur yang lebih padat, mengalami flokulasi dan dengan sedikit ruang pori. Mikrograf pengimbas elektron menunjukkan tanah yang distabilkan menghasilkan mineral bersimen. Setelah dilarutlesapkan dengan 7 PV mineral bersimen mengalami pelarutan dan membentuk alur baru. Walaubagaimanapun bahagian bawah lapisan tanah menunjukkan mineral bersimen yang lebih tumpat mengalami flokulasi, bersambungan antara satu sama lain dan halangan butiran halus dalam ruang-ruang pori. Keputusan kajian tersebut menunjukkan penambahan kapur boleh mengubah struktur mikro dan mengurangkan ketelapan tanah.

INTRODUCTION

Lime as a stabilisation agent could improve the physical and chemical properties of clays. In the landfill area adding sufficient lime to clay liner is effective to immobilise the contaminant such as heavy metals. The immobilisation of heavy metals depends on the formation of cementitious minerals, which formed due to the pozzolanic reaction between Ca^{2+} from lime and Al^{3+} or Si^{4+} from clay minerals. The microstructure and mineralogy of cementitious minerals can be identified using Scanning Eelectron Microscope and X-ray diffraction (Thowlow *et al.*, 1996; Rajasekaran

Table 1. The concentrations of major elements in unstabilised soil, stabilised soil and hydrated lime.

Major Elements (%)	Unstabilised	Stabilised	LIME
SiO_2	64.78	61.89	bdl
TiO_2	0.80	0.83	0.01
Al_2O_3	19.41	16.33	0.14
Fe_2O_3(T)	3.79	4.76	0.11
MnO	0.01	0.01	0.02
MgO	bdl	0.58	2.09
CaO	0.11	4.32	68.46
Na_2O	bdl	bdl	bdl
K_2O	2.24	2.09	0.02
P_2O_5	0.12	0.09	0.02
L.O.I	8.74	9.08	29.14
Total:	100.00	99.98	100.01

*bdl:below detection limit.
*L.O.I:Loss On Ignition.

Table 2. The physico-chemical of unstabilised and stabilised soil from Kg Bongkud, Ranau, Sabah.

Physico-chemical properties	Unstabilised	Stabilised
Moisture Content (%)	18.62	16.99
Organic Matter (%)	0.96	0.96
Specific Gravity	2.62	2.68
SSA (m^2/g)	13.8	14.20
Liquid Limit (%)	51	44
Plastic Limit (%)	23	26
Plasticity Index (%)	28	18
Shrinkage Limit (%)	45.39	46.59
Dry density (mg/m^3)	1.73	1.71
Optimum moisture content W_{opt}(%)	16.2	16.5
Bulk density (Mg.m3)	2.00	1.98
Void ratio (e)	0.57	0.52
pH	3.8	11.7
Porosity (%)	0.34	0.36
Pore volume (ml)	216	227
Permeability (m/s)	7.02×10^{-9}	2.40×10^{-9}

& Rao, 1998; Arabi & Wild, 1986; Hilmi & Aysen, 2000 and Baba Musta *et al.*, 2001). The cementitious minerals act as ion-sieving filters and separators to prevent contamination of the groundwater from the transmission of polluted liquids through the bottom of landfills. Tsai & Vesilind (1998) suggested that the decreased impermeability of lime stabilized clay liner was due to the formation of calcium silicate hydrate and calcium aluminate hydrate which blocked the flow channels. The permeability of soil also depends on particle size distribution, particle shape and texture, mineralogical composition, voids ratio, degree of saturation, soil fabric, nature of fluid, type of flow and temperature (Head, 1982). Gordon *et al.* (1989) revealed that the maximum laboratory hydraulic conductivity of clay soils used for clay liner is 1.0×10^{-9} m/s. In laboratory work the permeability and the reaction between soil and liquid can be measured by means of leaching test. The availability of cementitious minerals to immobilised heavy metals through the leaching test have been previously reported by Shively *et al.* (1986), Gosh & Subbarao (1995), Lombardi *et al.* (1998), Wang *et al.*, (2001) Mehmet & Bilge (2001) and Mckinley *et al.* (2001).

Although several studies reported the effectiveness of lime in improving permeability and modified microstructure of soils, limited information is available on lime treated clayey sand soil from tropical areas especially in the present studied area. Clayey sand soil from the weathered Crocker Formation shows extensive distribution in the study area. Study of the permeability of the soil is an important criterion to evaluate the suitability as a clay liner. Therefore, the objectives of this study are to investigate its permeability of lime treated clayey sand soil during leaching test and to determine the microstructure of the original minerals and cementitious minerals after leaching test by using SEM.

METHODOLOGY

A clayey sand soil sample from the mixture of sedimentary rocks of the Crocker Formation and an intrusive igneous rock was collected from Kg. Bongkud, Ranau, Sabah. The field observation showed soil of the sedimentary rocks underlying the igneous rock. The igneous rock can be recognised clearly by the saprolite formation at the bottom of the profile. Deep weathering of the rocks have produced a thick soil profile. The hand augered soil sample was dark brown in colour. The concentrations of elements in the soil are given in Table 1. Base on the data obtained, the high concentration of silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3) are important to supply Al ions and Si ions respectively for the pozzolanic reaction. Physico-chemical properties of the soil sample are presented in Table 2. The particle size distribution of unstabilised soil is 0% course sand (0.6–2.0 mm), 6.47% medium sand (2.0–0.6 mm), 37.04% fine sand (0.06–0.2 mm), 30.82% silt (0.002–0.06 mm), and 25.68% clay (< 0.002 mm). The Atterberg limit of the soil is as follows: liquid limit of 51% and plastic limit of 23%. The plasticity index is 28%. The hydrated lime [$Ca(OH)_2$] that is used as a stabilising agent was taken from the lime treatment company at Pasir Gudang, Johor. The abundance of CaO and MgO in lime is 68.46% and 2.09% respectively (Table 1). The X-ray diffractograms indicated the mineralogy of unstabilised soil consists of kaolinite, quartz and feldspar (Fig. 1).

The duplicate original samples were prepared by mixing the soil with water 3% higher than optimum moisture content. Whereas, about 6% by weight of lime were added to the soil before being placed in plexi-glass mold with 130 mm x 50 mm diameter. The optimum moisture content and different of density for preparing the unstabilised samples and stabilised samples were obtained from the compaction standard Proctor test summarized in Table 2. The soil was compacted using static compaction with ELE Digital Tritesting Instrument before curing at room temperature for 28 days under anaerobic conditions. The molds were closed tightly to avoid any water loss and to maintain the moisture constant during the curing process. At the end of the curing period the unstabilised sample (control sample) and stabilised samples were saturated with distilled water. Falling head was performed to study the permeability both in the unstabilised and stabilised soils. After leaching 7 PV solutions, the samples were oven dried for further chemical and microstructure analysis. Scanning electron microscopy (SEM) was employed to study the microstructure of the soil. SEM was carried out using a Philips XL40 model with a pressure of 60 psi and a voltage of 15–20 kV. The samples 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 identification of the cementitious minerals. The samples were then ground into powder form before being analysed. X-ray fluorescence (XRF) with fused discs was used to analyse the concentration of major elements (Norrish & Hutton, 1969). A "Philips PW 1480 X-ray Digital" instrument controlled by Digital Software x 44 microcomputer software was used for this purpose.

RESULT AND DISCUSSION

Effect of Lime on Physico-chemical Properties

The physico-chemical properties of stabilised soil are presented in Table 1. Base on the data obtained, by adding 6% lime the specific gravity (SG) and specific surface area (SSA) of stabilized soil increased slightly from 2.62 to 2.68 and from 13.8 m^2/g to 14.20 m^2/g respectively. The SG and SSA increment of stabilised soil was due to the high density and high surface area of the cementitious mineral. The low plasticity of cementitious minerals resulted in the decrease of the plasticity index of the stabilised soil from 28% to 18%. Whereas the shrinkage limit of the stabilised soil increased 1.2% i.e. from 45.4% to 46.6%. Generally the particle size of the stabilised soil has increased as shown in Figure 2. The particle size distribution of the stabilised soil is 9.28% course sand (0.6–2.0 mm), 44.75% medium sand (0.2–0.6 mm), 30.82% fine sand (0.2–0.006 mm), 12.61% silt (< 0.006 mm), and 2.52% clay (< 0.002 mm). The Atterberg limit of the stabilised soil is as follows: liquid limit of 53% and plastic limit of 23%. The compaction curves of unstabilised and stabilised soils are given in Figure 3. From the figure, it shows that the dry density of stabilised soil with 6% lime

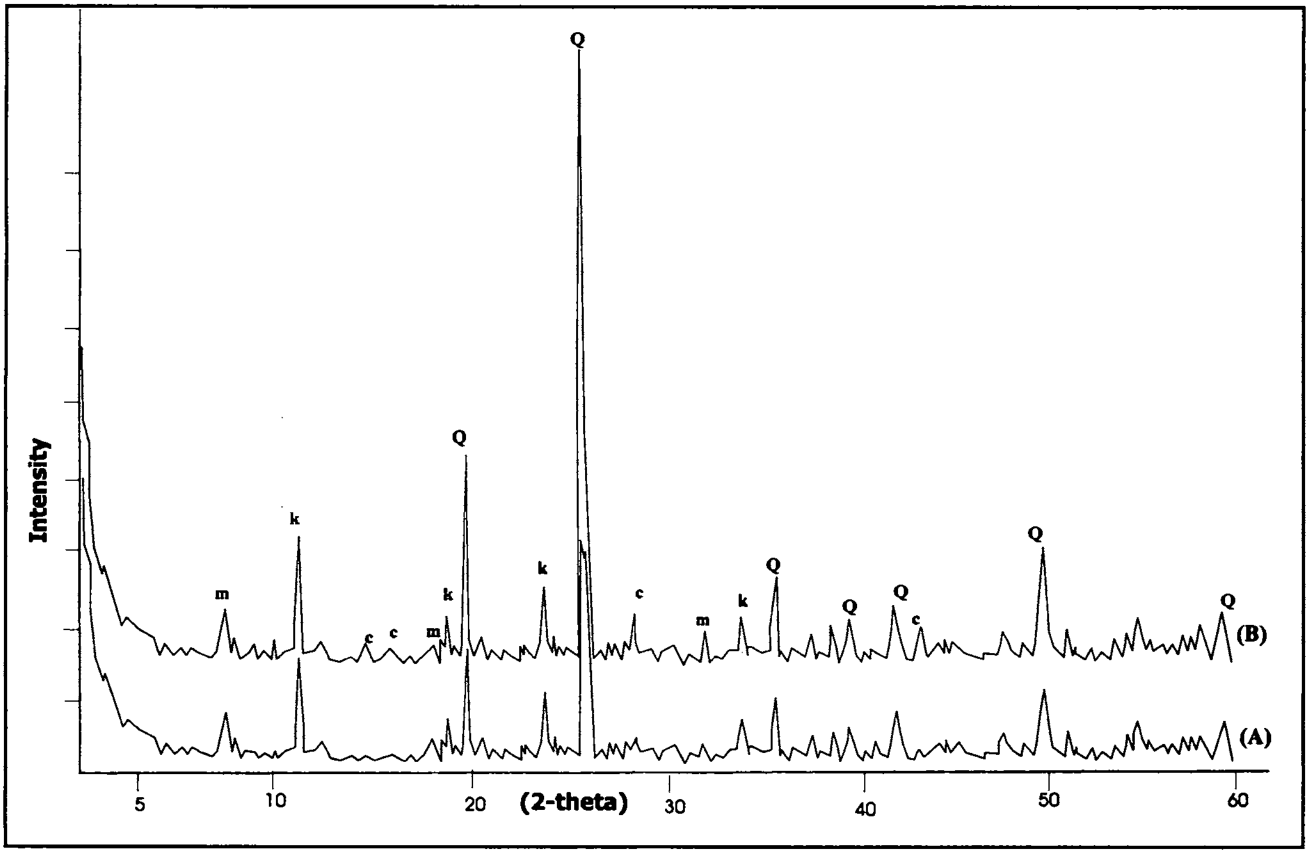

Figure 1. The XRD diffractograms of (A) unstabilised clayey sand soil, (B) stabilised with 6% of lime. Q: Quartz; k: kaolinite; m: muscovite; c: cementitious minerals.

has decreased from 1.73 mg/m^3 to 1.71 mg/m^3. However, the optimum moisture content has increased from 16.2% to 16.5%. This is due to the absorption of water by the soil for the formation of cementitious mineral. The X-ray diffractograms of stabilised soil shows the appearance of quartz, kaolinite and muscovite as the original minerals. The cementitious minerals were not detected clearly by the XRD due to the poor crystallization.

Effect of Lime on Microstructure

The scanning electron micrographs of the unstabilised soil, stabilised soil with 6% lime, unstabilised soils after leaching test and stabilised soils after leaching test are shown in Figure 4. Figure 4A shows the structure of random arrangement of clay particle and high pore spaces in unstabilised soil. The high percentage of pore indicated by the dark background area of the photomicrographs. This is the reason for the high permeability of the unstabilised soil. From the data obtained in Table 1, the volume of voids in the unstabilised soil is 0.57 and the pore volume is 216 ml. After leaching 7 pore volume of solution the top layer of soil became more packed and dense, clogging of fine particles and reorientation of clay particle resulted due to the pressure of solution during the leaching process (Fig. 4B). At the bottom layer the soil structure is well pack, flocculated and with low pore space as compared to the top layer (Fig. 4C). The microphotographs also showed the formation a small channel, which allowed the flow of solution through the bottom of the leaching cell. The stabilised soil with 6% lime shows the cementitious minerals, which is light in color, and are scattered at the edges and the surface of the original minerals (Fig. 4D). The formation of the cementitious minerals was due to the pozzolanic reaction. The cementitious minerals created a bridge at the edge and at the surface of minerals. The small dark background area indicates the reducing pore space compared to the unstabilised soil. Figure 4E shows the microstructure of the upper layer of stabilised soil after leaching test. The photomicrograph shows the flocculation reactions on the stabilised soil. The formation of channel at the middle of the micrograph was due to the dissolution of cementitious minerals during the leaching process. However, the bottom layer of stabilised soil still maintained the well packed assemblages of mineral. The dense cementitious minerals at the bottom layer were flocculated, linked with one another and clogged of the fine particles in pore spaces but the pore spaces slightly increased as compared with the stabilised soil before the leaching test (Fig. 4F).

Effect of Lime on Permeability

The initial permeability of stabilized soil is typically lower compared to the unstabilised soil after curing for 4 weeks. The permeability of unstabilised soil samples was 7.02 x 10^{-9} m/s and the stabilised soil was 2.40 x 10^{-9} m/s. The permeability patterns from 0 PV to 7 PV of both stabilised and unstabilised soil are presented in Figure 5.

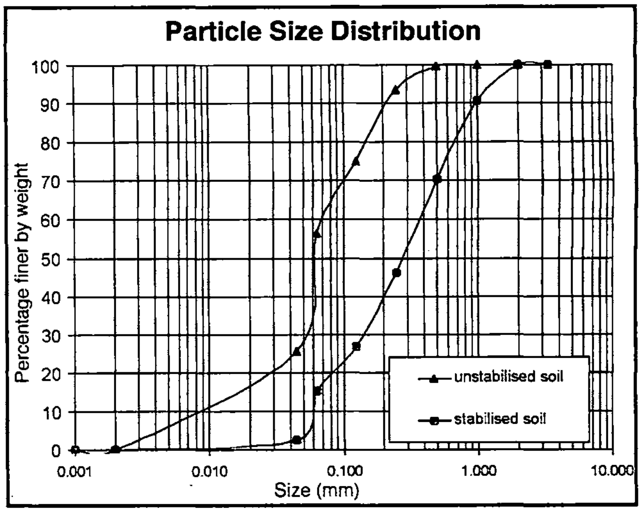

Figure 2. The particle distribution of unstabilised and stabilized soils.

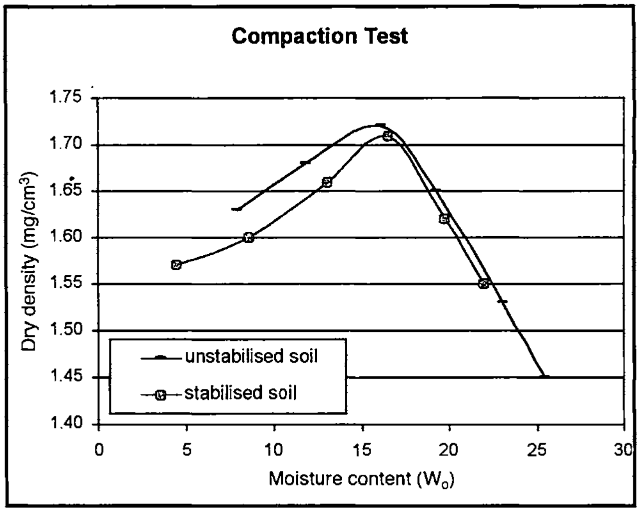

Figure 3. The compaction curve of unstabilised and stabilized soils.

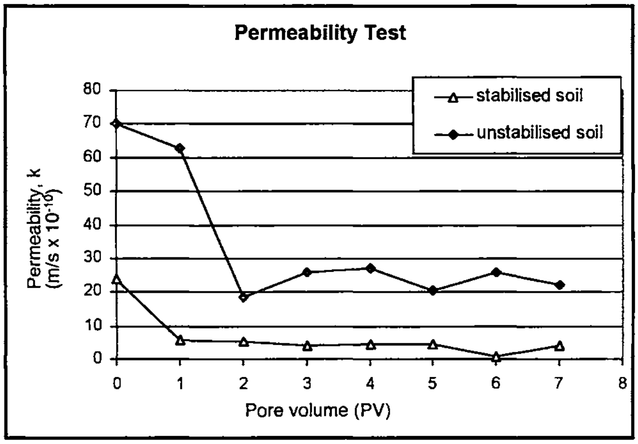

Figure 5. The permeability of unstabilised and stabilized soils from 0 pore volume to 7 pore volume.

Base on the figure, the unstabilised samples show the immediate decreased of permeability to 6.29 x 10^{-9} m/s with leaching 2 PV leaching solutions, whereas the stabilized samples show the immediate decrease of permeability to 1.86 x 10^{-10} m/s after 1 PV leaching solution. The increase of double layer thickness within the clay particle surface and clogging of fine particles might caused the immediate reducing of permeability. Followed by 2 PV to 7 PV leaching solution, the permeability of unstabilised soil was maintained at the range of 2.06 x 10^{-9} m/s to 2.70 x 10^{-9} m/s. Generally, the permeability of unstabilised soil with average values was 2.33 x 10^{-9} m/s. In this stage the permeability observed could be due to the optimum thickness of the double layer achieved and maximum clogging of fine particles into the pore spaces. This is due to the equilibrium achieved between the negative charge of the clay particle surface and positive charges from the cation in the liquid near the particle surface. Based on the figure, the range of permeability achieved for the stabilised soil at 1 PV to 7 PV was 1.01 x 10^{-10} m/s to 1.86 x 10^{-10} m/s, and the average was 1.42 x 10^{-10} m/s. The test result indicated that there is a decrease in permeability almost 20 times as compared to the untreated soil. This indicates that beside the increased of double layer thickness and clogged of fine particles, the formation of bridge and flocculation of cementitious minerals could also reduce the permeability of the stabilised soil. The result also shows that permeability could be decreased if the lime content is sufficient for the pozzolanic reaction to form cementitious minerals and flocculation of the clay particles.

CONCLUSION

1. The random structural arrangement of the clay particle and high pore spaces in the unstabilised soil is the reason for the high permeability of the unstabilised soil (k = 7.02 x 10^{-9} m/s) as compared to the stabilised soil (k = 2.40 x 10^{-9} m/s). After leaching 7 pore volume of solution the microstructure of the soil become packed, clogged with fine particles, occurrence

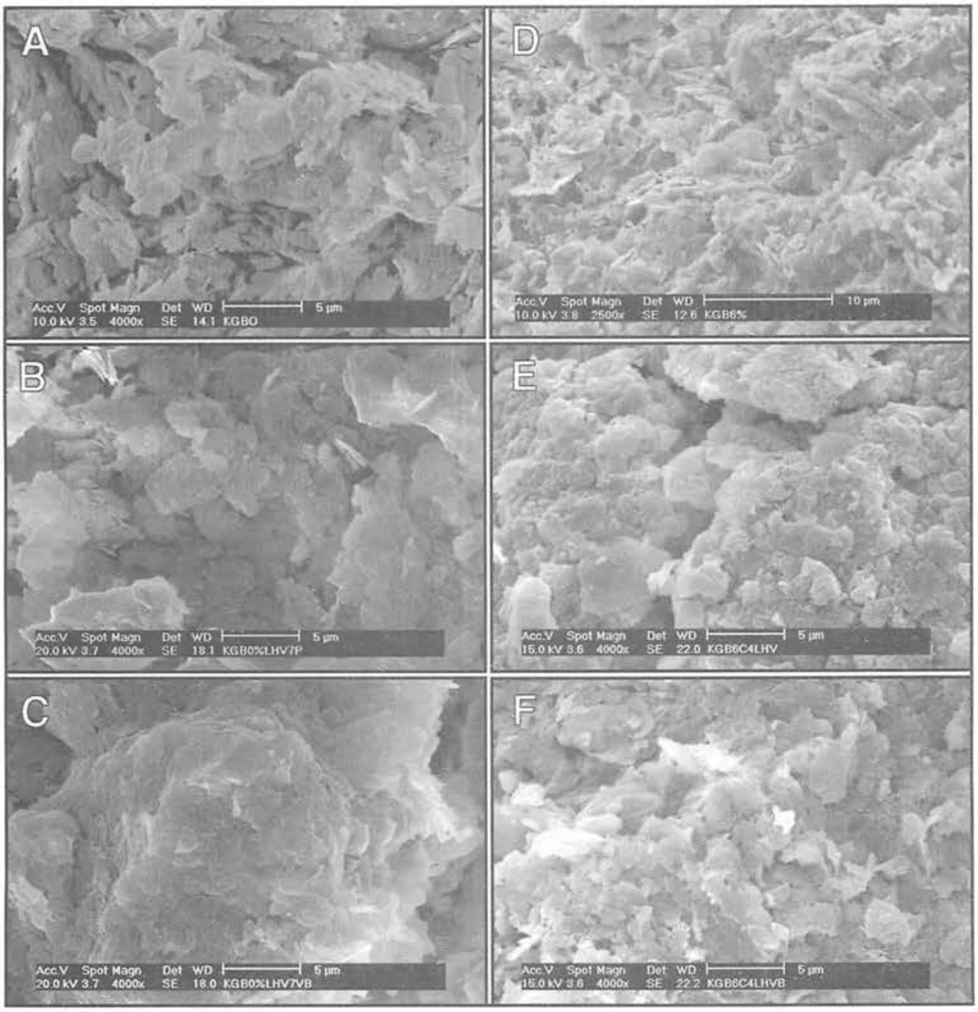

Figure 4. The scanning electron micrographs of (A) unstabilised soil (B) top layer of unstabilised soil after leaching test (C) bottom layer of unstabilised soil after leaching test (D) soil stabilised with 6% of lime (E) top layer of soil stabilised with 6% of lime after leaching test (F) bottom layer of soil stabilised with 6% of lime after leaching test.

of the clay particle reorientation and low pore space area. What resulted was observed to be more dense and the reduction the permeability of the unstabilised soil with in the range of 2.06 x 10^{-9} m/s to 2.70 x 10^{-9} m/s.

2. The microphotographs of the stabilised soil with 6% lime showed the formation of the cementitious minerals at the edge and surface of the original minerals, creating bridge, and flocculation within the minerals. After leaching with 7 pore volume solutions, the cementitious minerals at the upper layer of the soil become dissolved, which allowed the movement of the solution. However, at the bottom layer the stabilised soil still maintained the well packed assemblages of mineral, thus still low in permeability at the range of 1.01 x 10^{-10} m/s to 1.74 x 10^{-10} m/s.

ACKNOWLEDGEMENT

This research was funded by the Intensification Research of Priority Area (IRPA) Grant Coded 72277. The authors are grateful to Mr. Jerfi Samin from Faculty of Mechanical Engineering, Universiti Teknologi Malaysia for the SEM analyses. The authors would like to thank Mr. Abd. Aziz Ngah from Program Geology, Universiti Kebangsaan Malaysia for providing XRF analyses and Mr. Zainal Abidin from Faculty of Mechanical Engineering, Universiti Teknologi Malaysia for helping in the XRD analyses.

REFERENCES

ARABI, M. & WILD, S., 1986. Microstructural Development in Cured Soil-lime Composites. *Journal of Materials Science, 32*, 497–503.

BABA MUSTA, KHAIRUL ANUAR KASSIM & MOHD. RAZMAN SALIM, 2001. Mineralogical Development in a Lime Treated Clayey Sand Soil. *Proceedings Annual Geological Conference 2001*, Pangkor Island, Perak.

GORDON, M.E., HUEBNER, P.M. & MIAZGA, T.J., 1989. Hydraulic conductivity of three Landfill Clay liners. *Journal of Geotechnical Engineering, 115(8)*, 1148–1160.

GOSH, A. & SUBBARAO, C., 1995. Hydraulic conductivity and Leachate characteristics of Stabilized Fly ash. *Journal of Environmental Engineering, 124(9)*, 812–820.

HEAD, K.H., 1982. *Manual of Soil Laboratory Testing: Volume 2, Permeability, Shear Strength, and Compressibility Tests.* Pentech Press, London.

HILMI, L.A. & AYSEN, L.M., 2000. Microstructural development of Stabilized Fly ash as Pavement Base material. *Journal of materials in Civil Engineering, 12(2)*, 157–163.

LOMBARDI, F., MANGIALARDI, T., PIGA, L. & SRINI, P., 1998. Mechanical and leaching properties of cement solidified hospital solid waste incinerator fly ash. *Waste Management, 18*, 99–106.

MCKINLEY, J.D., THOMAS, H.R., WILLIAMS, K.P. & REID, J.M., 2001. Chemical Analysis of Contaminated Soil Strengthened by the addition of Lime. *Engineering Geology, 60*, 181–192.

MEHMET ALI YUKSELEN & BILGE ALPASLAN, 2001. Leaching of Metals from Soil Contaminated by Mining Activities. *Journal of Hazardous Materials, B87*, 289–300.

NORRISH, K. & HUTTON, J.T., 1969. An accurate X-ray Spectrographic Method for the Analysis of a wide range of Geological Samples. *Geochim. Et Cosmochima Acta, 33*, 431–453.

RARASEKARAN, G. & RAO, S.N., 1998. X-Ray Diffraction and Microstructural studies of Lime-Marine Clay Reaction Products. *Geotechnical Engineering, 29(1)*, 1–27.

SHIVELY, W., BISHOP, P., GRESS, D. & BROWN, T., 1986. Leaching tests of Heavy Metals Stabilized with Portland Cement. *Journal WPCF, 58*, 234–241.

THOWLOW, N., JAKOBSE, U.H. & CLARK, B., 1996. Composition of Alkali Silica Gel and Ettringite in Concrete Railroad Ties: SEM-EDX and X-ray Diffraction Analyses. *Cement and Concrete Research, 26(2)*, 309–318.

TSAI, T. & VESILIND, P.A., 1998. A new Landfill Liner and Reduce Ground-water Contamination from Heavy Metals. *Journal of Environmental Engineering, 124*, 1061–1095.

WANG, Y.M., CHEN, T.C., YEH, K.J. & SHUE, M.F., 2001. Stabilization of an Elevated Heavy Metal Contaminated Site. *Journal of Hazardous Materials, B88*, 63–74.