PHYSICOCHEMICAL CHARACTERIZATION OF LIME AND CEMENT STABILIZED CLAYEY SOILS CONTAMINATED BY HEAVY METAL

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To the spirit of my mother and father

To my husband and my kids (Zina, Mohammed, Ruqia)

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ABSTRACT

The presence of contaminated soils due to industrials and mining activities is a major concern in today's heavily industrialized world. The contaminants lead to poor engineering properties for these soils. However, the effect of these contaminants on the geotechnical properties of clayey soils can be altered through chemical stabilization using traditional stabilizers, which result in achieving suitable material for construction purposes. On the other hand, the studies on the evaluation of microstructural, molecular, and leaching characteristics of treated contaminated clay soils are so limited. In this research, two contaminated tropical soils (mixed with different percentages of heavy metals) comprised mainly of kaolinite mineral i.e., Brown Kaolin and Laterite Clay. Similar contents of lime and cement (5% and 10% of dry weight) have been used for the stabilization process of contaminated clay soils. Two selected heavy metals Cu and Zn were chosen as nitrates to investigate their effects with time on the characteristics of the treated soils. The changes in the mineralogy, morphology and soil molecular structure due to the effect of stabilizers and heavy metals were explained based on X-ray Diffractometry (XRD), Energy Dispersive X-ray Spectrometry (EDAX), Field Emission Scanning Electron Microscopy (FESEM), Fourier Transform Infrared Spectroscopy (FTIR), Cation Exchange Capacity (CEC) and Brunauer Emmett and Teller (BET) surface area analysis. Leaching tests were also performed on cured contaminated samples, in order to investigate the potential of lime and cement stabilization technique to immobilize the heavy metals. The strength and compressibility of the stabilised soils with the effect of heavy metals were tested using unconfined compression and one dimensional consolidation tests. Based on the micro-structural characterization, strength and compressibility characteristics of the stabilized soils, cement was the most effective stabilizer than lime in terms of improving the strength and compressibility of contaminated treated soils. However, the heavy metals have more retarded effect on the cement treated samples than lime. This was due to precipitation of the metals onto the surface of calcium and aluminium silicates as insoluble hydroxides or sulphates. Thus, these compounds form an impermeable coating that acts as a barrier to inhibit cement hydration by impending transport of water into cement grain. Nevertheless, in the case of lime treatment, the metals ions adsorb and held as an amorphous mass in the pore spaces between the flocculated particles of treated samples. The pH values of leachate from all stabilized contaminated samples were found to be alkaline, indicating that the heavy metals were adsorbed on the surface of the cementations minerals. Finally, it can be concluded that the mechanical and the physico-chemical behaviours of the compacted specimens, as determined during testing, formed the basis for evaluating both the degree of immobilization of the heavy metal in the soil matrix, and the potential for rehabilitation of contaminated sites.

ABSTRAK

Kehadiran bahan-bahan tercemar dalam tanah disebabkan oleh industri dan aktiviti-akitiviti perlombongan kini telah menjadi kebimbangan utama dalam sektor perindustrian. Bahan-bahan tercemar ini telah melemahkan sifat-sifat kejuruteraan tanah tersebut. Walau bagaimanapun, kesan bahan-bahan tercemar ini ke atas sifat-sifat geoteknik tanah liat boleh diubah melalui penstabilan kimia dengan menggunakan penstabil tradisional, dimana ia sesuai untuk tujuan pembinaan. Selain itu, kajian mengenai penilaian mikrostruktur, molekul, dan ciri-ciri resapan tanah liat tercemar yang dirawat adalah begitu terhad. Dalam kajian ini, kandungan utama yang terdapat di dalam dua tanah tropika tercemar buatan (dicampur dengan peratusan logam berat yang berbeza) adalah kaolin (Kaolin Perang dan Tanah Liat Laterit). Kandungan kapur dan simen yang sama (5% dan 10% daripada berat kering) juga telah digunakan untuk proses penstabilan tanah liat yang tercemar. Dua logam berat iaitu Cu dan Zn telah dipilih sebagai nitrat untuk menyiasat kesan terhadap sifat tanah terawat. Perubahan dari segi mineralogi, morfologi dan struktur molekul tanah disebabkan oleh kesan penstabil dan logam berat telah diterangkan berdasarkan kaedah Pembelauan Sinar-X (XRD), Sebaran Tenaga X-ray spektrometri (EDAX), Pelepasan Imbasan Mikroskop Elektron (FESEM), Spektroskopi inframerah transformasi Fourier (FTIR), kation Bursa Kapasiti (CEC) dan Brunauer Emmett dan Teller (BET) analisis kawasan permukaan. Ujian resapan juga dilakukan ke atas sampel tercemar yang dirawat untuk mengenalpasti teknik penstabilan kapur dan simen dalam menyahkan logam berat. Kekuatan dan kebolehmampatan tanah yang terstabil melalui kesan daripada logam berat diuji menggunakan ujian mampatan tak terkurung dan ujian pengukuhan satu dimensi. Berdasarkan pencirian mikro-struktur daripada tanah yang dirawat dan keputusan yang diperolehi daripada kekuatan dan kebolehmampatan bagi kedua-dua jenis tanah, didapati bahawa simen adalah penstabil yang paling berkesan berbanding kapur untuk meningkatkan kekuatan dan kebolehmampatan tanah tercemar yang dirawat. Namun begitu, logam berat memberi kesan yang lebih dalam merencatkan proses rawatan tanah menggunakan simen berbanding kapur. Hal ini disebabkan oleh pemendakan logam ke atas permukaan kalsium dan aluminium silika yang merupakan hidroksida tidak larut atau sulfat. Dengan itu, sebatian ini membentuk lapisan yang bertindak sebagai penghalang penghidratan simen dengan menyekat pengangkutan air ke dalam zarah simen. Namun, di dalam kes rawatan kapur, ion-ion logam menyerap dan berkumpul sebagai jisim amorfus di dalam ruang liang di antara zarah terapung sampel yang dirawat. Nilai pH resapan dari semua sampel tercemar dirawat didapati alkali, menunjukkan bahawa logam berat terserap ke permukaan pensimenan mineral. Akhirnya, boleh disimpulkan bahawa tingkah laku mekanikal dan fiziko-kimia bagi spesimen dipadatkan seperti yang ditentukan semasa ujian, membentuk asas untuk menilai tahap kepegunan kedua-dua logam berat dalam matriks tanah, dan potensi penggunaan semula tanah tercemar setelah dirawat.

TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	TITLE	i
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENTS	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENTS	vii
	LIST OF TABLES	xiii
	LIST OF FIGURES	xv
	LIST OF ABBREVIATIONS AND SYMBOLS	xxxiii
	LIST OF APPENDICES	xxxix
1	**INTRODUCTION**	**1**
	1.1 Background	1
	1.2 Problem Statement	3
	1.3 Motivation and Significance of the Study	4
	1.4 Objectives of Study	5
	1.5 Scope of Study	6
	1.6 Thesis Structure	8

2 LITERATURE REVIEW 10

2.1 Introduction 10
2.2 The Contamination Issue 11
2.2.1 Inorganic Contaminants 12
2.2.2 Radioactive Contaminants 12
2.3 The Heavy Metals in Soil 13
2.3.1 Mineralization and Weathering 14
2.3.2 Landfill Site 15
2.3.3 Mining Activity 16
2.4 Level Toxicity of Heavy Metals 16
2.4.1 Sources of Heavy Metals in Malaysia 17
2.5 The Concentration of Heavy Metals 18
2.6 The Behaviour of Heavy Metals in Soil 20
2.6.1 Adsorption 21
2.6.2 Precipitation 22
2.6.3 Cation Exchange Reactions 23
2.6.4 Complexation 24
2.6.5 pH Values Effects 25
2.7 Clay Minerals 27
2.7.1 Kaolinite (1:1 Minerals) 28
2.7.2 Illite (2:1 Smectite Minerals) 29
2.7.3 Montmorillonite (2:1 Smectite Minerals) 31
2.8 Clay-Water Interaction 33
2.8.1 Ion Concentration in Clay-Water Interaction 34
2.9 The Mechanism of Stabilization/Solidification Technique 36
2.9.1 Immobilization Mechanisms 36
2.9.2 Leaching Mechanisms 38
2.10 Mechanism of Lime Stabilization 39
2.10.1 Chemical Clay-Lime Interactions 41
2.10.2 Lime Quantity 43
2.11 Mechanism of Cement Stabilization 45
2.11.1 Cement-Clay Interactions 46

2.11.2 Soil Improvement and Cement-Clay Interactions 48
2.12 Hydration of Cement Compounds 51
2.12.1 Hydration of silicates compounds 51
2.12.2 Hydration of aluminate and ferrite 54
2.13 Mechanisms of Retardation 55
2.13.1 Heavy Metals Interactions 58
2.14 Chemical and Mineralogical Properties of Stabilized Soils 60
2.14.1 Micro-Structural Characterization 60
2.14.1.1 X-ray Diffraction Studies 60
2.14.1.2 Microscopic Studies 64
2.14.2 Molecular classification 68
2.14.2.1 FTIR Studies 68
2.14.3 Leaching Characterization 71
2.14.4 Cation Exchange Capacity Analysis 78
2.15 Physical Analysis of the Stabilized Soil 79
2.15.1 Compaction Characteristics 79
2.15.2 Compressive Strength 82
2.15.3 Compressibility of Stabilized Soils 85
2.16 Acceptance criteria of solidified waste 87
2.17 Summary 88

3 EXPERIMENTAL DESIGN AND METHODOLOGY 89

3.1 Introduction 89
3.2 Material Specification 91
3.2.1 The Soils 91
3.2.2 The Stabilizer 92
3.2.2.1 Hydrated Lime 92
3.2.2.2 Portland Cement 93
3.2.3 Contaminating metals added during the Investigations 94
3.3 Water Quality 95
3.4 Laboratory Full-Scale Mix Design Preparation 96

3.4.1 Lime and Cement Content 96
3.4.2 Compaction Characteristics 97
3.4.3 Sample Preparation 98
3.4.3.1 Preparation of UCS Samples 98
3.4.3.2 Preparation of Oedometer Test Samples 103
3.5 Sample Characterization 104
3.5.1 Micro-Structural Characterization 105
3.5.1.1 X-ray Diffraction 105
3.5.1.2 Scanning Electron Microscopy & X-ray Spectrometry 109
3.5.2 Molecular Characterization 112
3.5.2.1 Fourier Transform Infrared Spectroscopy 112
3.5.3 Leaching Characterization 114
3.6 Chemical Properties Analysis 117
3.6.1 Cation Exchange Capacity 117
3.6.2 pH Measurement 120
3.7 Engineering Properties 122
3.7.1 Surface Area Value (BET test) 122
3.7.2 Unconfined Compressive Strength 124
3.7.3 One Dimensional Consolidation Test 126
3.7.3.1 Soil Compressibility 126
3.8 Summary 128

4 RESULTS AND DISCUSSION 129

4.1 Introduction 129
4.2 Material Properties 130
4.2.1 Particle Size Analysis (PSA) 131
4.2.2 Atterberg Limits 132
4.2.3 Initial Consumption of Lime Test 133
4.2.4 Compaction Characteristics 134
4.3 Characterization of Stabilized Soils 138
4.3.1 Micro-Structural Characteristics 138

4.3.1.1 XRD 138

4.3.1.1.1XRD for Treated Un-Contaminated Clay Soils 138

4.3.1.1.2 XRD for Treated Contaminated Clay Soils 147

4.3.1.2 Field Emission Scanning Electronic Microscopy 154

4.3.1.2.1 FESEM for Treated Un-Contaminated Clay Soils 154

4.3.1.2.2 FESEM for Treated Contaminated Clay Soils 162

4.3.1.3 EDAX 169

4.3.2 Molecular Characteristics 177

4.3.2.1 FTIR 177

4.3.3 Leaching Characteristics 187

4.4 Chemical Analysis of Cured Samples 191

4.4.1 pH 191

4.4.2 Cation Exchange Capacity (CEC) 195

4.5 Physical Analysis of Cured Samples 199

4.5.1 BET Surface Area 199

4.5.2 Unconfined Compressive Strength (UCS) 203

4.5.2.1 The effect of heavy metals content 203

4.5.2.2 The effect of heavy metal type 205

4.5.2.3 The effect of stabilizer content 207

4.5.3 One-dimensional consolidation test 219

4.5.3.1 The Compressibility of Contaminated Stabilized Clay Soil 221

4.6 Summary 229

5 CONCLUSIONS AND RECOMMENDATIONS 234

5.1 Introduction 234

5.2 Conclusions 234
5.2.1 Stabilization Process of Uncontaminated Soils 235
5.2.2 Stabilization Process for Contaminated Soils 239
5.2.3 Leaching Characteristics 243
5.3 Recommendations 245

REFERENCES 247

Appendices A-G 270-340

LIST OF TABLES

TABLE NO.	TITLE	PAGE
2.1	The composition of particular industrial waste streams and sludges (after Landreth, 1982)	12
2.2	The Concentrations of Heavy Metals from the Previous Studies	19
2.3	Summary of previous studies on the mechanism of lime stabilization	43
2.4	Typical composition of cement clinker (after Janz and Johansson, 2002)	46
2.5	IR bands of kaolinite (KGa) and montmorillonite (SWy) clay along with their assignments (Madejova and Komadel, 2001)	69
2.6	Aqueous concentrations of various metals for different mix designs(after Goswami and Mahanta, 2007)	75
2.7	Leachate pHs and metal concentrations in TCLP leachates (Asavapisit et al.,2005)	77
2.8	Solidified waste acceptance criteria	88

3.1	Chemical composition of the hydrated lime	93
3.2	Chemical composition of Portland cement	94
3.3	The chemical and physical properties for selected regents	95
3.4	Mix proportions of specimens	102
4.1	The values of atterberg limit for the natural soils	133
4.2	Variation of Al:Si and Ca:Si ratios for various cement Laterite clay soil mix design with curing time	171
4.3	Variation of Al:Si and Ca:Si ratios for various lime Laterite clay soil mix design with curing time	173
4.4	Variation of Al:Si and Ca:Si ratios for various cement Kaolin clay soil mix design with curing time	175
4.5	Variation of Al:Si and Ca:Si ratios for various lime Kaolin clay soil mix design with curing time Studies	177
4.6	The functional peaks in the FTIR spectra of Laterite clay Soil	178
4.7	Summary of the Obtained Results	229

LIST OF FIGURES

FIGURE NO.	TITLE	PAGE
2.1	Global productions and consumption of selected toxic metals,1850-1999 (after Järup, 2003).	18
2.2	Schematic diagram showing specific and non-specifically adsorbed counter ions in diffuse double layer (DDL) model (after Yong, 2000).	21
2.3	Schematic diagram showing the inner Helmholtzplane (IHP) and outer Helmholtz plane (OHP) on the clay particle (after Yong, 2000).	22
2.4	General distribution of cations held as exchangeable ions at different pH values (after Brady and Weil, 1996)	26
2.5	Solubility of some soil mineral species in relation to pH (afte Loughnan, 1969)	26
2.6	Basic arrangements of silica tetrahedral (Top) and aluminum octahedral(Bottom) (after Mitchell and Soga, 2005)	27

2.7	Synthesis pattern for the clay minerals (after Mitchell and Soga, 2005)	28
2.8	Diagrammatic sketch of the kaolinite structure (after Mitchel and Soga, 2005) and (After Das, 1997)	29
2.9	Photomicrograph of well-crystallized kaolinite (after Tovey, 1971)	29
2.10	Diagrammatic sketch of the structure of Illite (after Mitchel and Soga, 2005) and (after Das, 1997)	30
2.11	Electron photomicrograph of Illite (after Tovey, 1971)	31
2.12	Schematic diagrams of the structures of Montmorillonite (after Mitchell and Soga, 2005, and Das, 1997)	32
2.13	Electron photomicrograph of Montmorillonite (after Tovey, 1971)	32
2.14	Some possible mechanisms of water adsorption by clay surfaces: (a) hydrogen bonding, (b) ion hydration, (c) attraction by osmosis, and (d) dipole attraction (after Low, 1961)	34
2.15	Distribution of ions adjacent to a clay surface according to the concept of the diffuse double layer (after Mitchell and Soga, 2005, and Das,1997)	35
2.16	The mechanisms of metal fixations (after USEPA, 200	38

2.17	Leaching mechanisms (after Cote et al., 1985)	39
2.18	Steps of Cement Hydration (after Kurtis, 2002)	47
2.19	Reaction mechanisms of soil and hardening agents (after Saitoh et al. 1985)	49
2.20	Cement reaction mechanisms in clay soil with time (after Saitoh et al. 1985)	50
2.21	Reaction mechanism involved in the development of C_3S-Pozzolana grains (after Ogawa and Roy, 1982)	50
2.22	Solidification and stabilization of heavy metals by hydrates of Portland cement (after Conner, 1990)	59
2.23	XRD Patterns of Cement- and Lime-Stabilized Fly ash (after Hilmi and Aysen, 2000)	62
2.24	Influence of the heavy metals on the hydration product of C3S (1: Ca(OH)2; 2: C3S; 3: aO(Zn(OH)2)•2H2O) (after Qiao et al.,2007)	62
2.25	Influence of the heavy metals on the hydration product of C_3A (after Qiao et al., 2007)	63
2.26	XRD pattern of Type I Portland cement before and after hydration (after Wang and Vipulanandan, 1999)	63

2.27	SEM image of (a) untreated Singapore marine clay,(b) 10% cement-treated marine clay,(c)30% cement – treated marine clay, and (d)50% cement-treated marine clay, all the samples treated with constant water content for 28 days curing time by(after Chew et al.,2004)	65
2.28	SEM micrographs of 7- and 28- day cured main (solidified soil) and control specimens at magnitude of 2000: (a) 7-day cured control; (b) 7-day cured solidified soil; (c) 28- day cured control; and (d) 28-day cured solidified Soil (after Ganjidoust et al., 2009)	66
2.29	Scanning electron micrographs of lime treated Buckingham soil (after Choquette et al., 1987)	67
2.30	IR spectra of kaolinite (KGa-2) using KBr pellet (after Madejova and Komadel, 2001)	69
2.31	Mid-infrared spectra of three source clays (after Du et al., 2009)	70
2.32	FTIR spectra of a) Zn-OPC, cured in air b) Zn-OPC, cured in carbon dioxide (after Yousuf et al., 1993)	71
2.33	Mixing methods for extraction tests (after Boardman, 1999)	73
2.34	Leaching Test Procedure (after Yin et al., 2007)	74
2.35	Levels of TCLP Pb release with or without quicklime	76

	treatment for different solid mixes, following 28 days of specimen curing (after Dermatas and Meng, 2003)	
2.36	Levels of TCLP Cr (III) release with or without quicklime treatment for different solid mixes, following 28 days of specimen curing (after Dermatas and Meng, 2003)	76
2.37	Changes in compaction curve by addition of lime to a low plasticity clay soil (after Mallela et al. 2004)	81
2.38	Plot of dry unit weight versus water content of the uncemented and the cemented samples compacted under standard and modified Proctor energies (after Horpibulsuk et al., 2010)	82
2.39	Variation of UCC strength with lime content (after Sen and Mukesh, 2011)	83
2.40	Strength developments as a function of cement content (after Horpibulsuk et al, 2010)	84
2.41	Compressive strength of two specimens without and with heavy metals as a function of curing time day (after Ganjidoust et al., 2009)	85
2.42	Compression curve for different content of cement (after Ho et al., 2010)	86

3.1	Flow chart of research methodology	90
3.2	The image of natural Laterite Clay soil from its excavated site	92
3.3	Compaction standardize equipment	98
3.4	Preparation of Laterite Clay Soil	99
3.5	Samples preparation	100
3.6	Typical samples (labeling and keeping)	101
3.7	Preparation of One-consolidation Oedometer test samples	104
3.8	X-ray Diffraction Techniques	105
3.9	Diffraction from crystal planes according to Bragg's Law (after Mitchell and Soga, 2005)	106
3.10	X-ray powder diffractometer scheme	107
3.11	Preparation of samples for XRD-Diffraction analysis	108
3.12	Bruker D8 advance diffractometer-UTHM- FKM-Batu Pahat –Johor-Malaysia	108
3.13	Schemes for the technical process of SEM test	110

3.14	Gold sputter coater	111
3.15	A JSM-6701F JEOL (FESEM)-UTM-FKM-Skudi-Johor-Malaysia	112
3.16	A simplified optical layout of a typical FTIR spectrometer	113
3.17	Perkin Elmer Spectrum 2000 instrument(FTIR)-UTM-FS-Skudi-Johor-Malaysia	114
3.18	Batch Mechanical Stirrers	115
3.19	Preparation the samples for centrifuging	116
3.20	Collected the filtered samples for ICP analysis	116
3.21	Inductively Coupled Plasma spectrometer	117
3.22	Titration Samples Equipment	119
3.23	Samples Preparation	119
3.24	Hannah model pH Meter	121
3.25	Orion model pH Meter	121
3.26	Quantachrome Autosorb-1 surface area analyzer (BET), FS, UTM, Johor, Malaysia	123

3.27	Triaxial loading frame, ADU, the processing unit, and some types of sample failures	125
3.28	One Dimensional Oedometer frame, sample cell frame, and ADU control system	127
3.29	A fully automated oedometer samples test	128
4.1	Particle size distribution curve of Laterite clay	131
4.2	Particle size distribution curve of Kaolin clay	132
4.3	Plasticity Chart	133
4.4	Results of the ICL test on Laterite clay and Kaolin clay soils	134
4.5	Variation of dry density with water content for lime-Laterite clay soil samples	136
4.6	Variation of dry density with water content for cement-Laterite Clay soil samples	136
4.7	Variation of dry density with water content for lime Kaolin clay soil samples	137
4.8	Variation of dry density with water content for cement treated Kaolin clay soil samples	137
4.9	X-ray diffraction for untreated laterite clay soil LCUT	139

4.10	X-ray diffraction patterns for cement (a) and lime (b) treated laterite clay soil after 7, 100, and 200 days curing	142
4.11	X-ray diffraction for untreated kaolin clay soil KUT	143
4.12	X-ray diffraction patterns for cement (a) and lime (b) treated kaolin clay soil after 7, 100, and 200 days curing	146
4.13	X-ray diffraction patterns of cement treated laterite clay contaminated by copper (a) and zinc (b) at 7, 100, and 200 curing	148
4.14	X-ray diffraction patterns of lime treated Laterite clay contaminated by copper (a) and zinc (b) at 7, 100, and 200 curing	150
4.15	X-ray diffraction patterns of cement treated Kaolin clay contaminated by copper (a) and zinc (b at 7, 100, and 200 curing	152
4.16	X-ray diffraction patterns of lime treated Kaolin clay contaminated by copper (a) and zinc (b) at 7, 100, and 200 curing	153
4.17	FESEM image of untreated Laterite clay	154

4.18	FESEM images of 10% cement treated Laterite Clay after7, 100, and 200 days	156
4.19	FESEM images of 10% lime treated Laterite Clay after7, 100, and 200 days	157
4.20	FESEM image of untreated Kaolin clay soil	158
4.21	FESEM images of 10% cement treated Kaolin Clay after7, 100, and 200 days	159
4.22	FESEM images of 10% Lime treated Kaolin Clay after7, 100,and 200 days	161
4.23	FESEM micrographs of cement treated Laterite soil contaminated by copper (left side) and zinc (right side) at 7,100, and 200 days curing	163
4.24	FESEM micrographs of lime treated Laterite soil contaminated by copper (left side) and zinc (right side) at 7, 100, and 200 days curing	165
4.25	FESEM micrographs of cement treated Kaolin clay soil contaminated by copper (left side) and zinc (right side) at 7, 100, and 200 days curing	167

4.26	FESEM micrographs of lime treated Kaolin clay soil contaminated by copper (left side) and zinc (right side) at 7, 100, and 200 days curing	168
4.27	EDAX spectrums of untreated, cement, and cement treated contaminated Laterite clay soil after 200 days curing time	170
4.28	EDAX spectrums of untreated, lime treated, and lime treated contaminated Laterite clay soil after 200 days curing time	172
4.29	EDAX spectrums of untreated, cement treated, and cement treated contaminated Kaolin clay soil after 200 days curing time	174
4.30	EDAX spectrums of untreated, lime treated, and lime treated contaminated Kaolin clay soil after 200 days curing time	176
4.31	FTIR Spectra for natural Laterite clay soil	178
4.32	FTIR spectrums of natural and cement treated Laterite clay at different time intervals	179
4.33	FTIR spectrums of copper and zinc doped with cement treated Laterite clay at different time intervals	180
4.34	FTIR spectrums of natural and lime treated Laterite	

	clay at different time intervals	181
4.35	FTIR spectrums of copper and zinc doped with lime treated Laterite clay at different time intervals	182
4.36	FTIR Spectra for natural Brown Kaolin clay soil	183
4.37	FTIR spectrums of natural and cement treated Kaolin clay at different time in	184
4.38	FTIR spectrums of copper and zinc doped with cement treated Kaolin Clay at different time intervals	185
4.39	FTIR spectrums of natural and lime treated Kaolin Clay at different time intervals	186
4.40	FTIR spectrums of copper and zinc doped with lime treated Kaolin Clay at different time interval	186
4.41	Levels of concentration of Cu and Zn release into the aqueous solution with respect to 5% and 10% cement treatment for Laterite clay samples	188
4.42	Levels of concentration of Cu and Zn release into the aqueous solution with respect to 5% and 10% lime treatment for Laterite clay sample	189

4.43	Levels of concentration of Cu and Zn release into the aqueous solution with respect to 5% and 10% cement treatment for Kaolin clay samples	190
4.44	Levels of concentration of Cu and Zn release into the aqueous solution with respect to 5% and 10% lime treatment for Kaolin clay samples	191
4.45	Variation of pH for cement treated Laterite Clay mix designs before and after contamination	192
4.46	Variation of pH for lime treated Laterite Clay mix designs before and after contamination	193
4.47	Variation of pH for cement treated Brown Kaolin Clay mix designs before and after contamination	194
4.48	Variation of pH for lime treated Brown Kaolin Clay mix designs before and after contamination	194
4.49	Time-dependent changes in CECp of cement-Laterite samples mix design	196
4.50	Time-dependent changes in CECp of lime-Laterite samples mix design	197
4.51	Time-dependent changes in CECp of cement-Brown Kaolin samples mix design	198

4.52	Time-dependent changes in CECp of lime-Brown Kaolin samples mix design	198
4.53	BET results for cement treated Laterite Clay samples before and after contamination at different intervals	200
4.54	BET results for lime treated Laterite Clay samples before and after contamination at different time intervals	201
4.55	BET results for cement treated Brown Kaolin Clay samples before and after contamination at different time intervals	202
4.56	BET results for lime treated Brown Kaolin Clay samples before and after contamination at different time intervals	203
4.57	Normalized strength of 5%cement treated Laterite clay contaminated by different percentages of copper	205
4.58	Normalized strength of 5% lime treated Laterite clay contaminated by different percentages of copper	205
4.59	Normalized strength of 5% cement treated Laterite clay contaminated by zinc	206
4.60	Normalized strength of 5% lime treated Laterite clay	

	contaminated by zinc	207
4.61a	Normalized strength of 5%cement treated Laterite clay contaminated by different percentages of copper	208
4.61b	Normalized strength of 10%cement treated Laterite clay contaminated by different percentages of copper	209
4.62a	Normalized strength of 5%lime treated Laterite clay contaminated by different percentages of copper	210
4.62b	Normalized strength of 10% lime treated Laterite clay contaminated by different percentages of copper	210
4.63a	Normalized strength of 10%cement treated Laterite clay contaminated by different percentages of copper	211
4.63b	Normalized strength of 10%cement treated Laterite clay contaminated by different percentages of zinc	212
4.64a	Normalized strength of 10%lime treated Laterite clay contaminated by different percentages of copper	212
4.64b	Normalized strength of 10%lime treated Laterite clay contaminated by different percentages of zinc	213
4.65a	Normalized strength of 5%cement treated Kaolin clay contaminated by different percentages of copper	214

4.65b	Normalized strength of 5%lime treated Kaolin clay contaminated by different percentages of copper	215
4.66a	Normalized strength of 5%cement treated Kaolin clay contaminated by different percentages of zinc	215
4.66b	Normalized strength of 5%lime treated Kaolin clay contaminated by different percentages of zinc	216
4.67a	Normalized strength of 10%cement treated Kaolin clay contaminated by different percentages of copper	217
4.67b	Normalized strength of 10%lime treated Kaolin clay contaminated by different percentages of copper	217
4.68a	Normalized strength of 10%cement treated Kaolin clay contaminated by different percentages of zinc	218
4.68b	Normalized strength of 10%lime treated Kaolin clay contaminated by different percentages of zinc	219
4.69a	void ratio-axial stress relationship for Laterite clay soils stabilized with cement	220
4.69b	void ratio-axial stress relationship for Laterite clay soils stabilized with lime	221
4.70	Relationship of void ratio with axial pressure for uncontaminated and copper-zinc contaminated cement stabilized Laterite clay at different curing times (a) 7days, (b) 28 days, (c) 100 days, and (d) 200 days	223

4.71	The variations of compression index for uncontaminated and copper-zinc contaminated cement stabilized Laterite clay at different curing time	224
4.72	Relationship of the void ratio with axial pressure for uncontaminated and copper–zinc contaminated lime stabilized Laterite clay at different curing times (a) 7days (b) 28 days, (c) 100 days, and (d) 200 days	225
4.73	The variations of compression index for uncontaminated and copper –zinc contaminated lime stabilized laterite clay at different curing time	227
4.74	The variations of compression index for uncontaminated and copper–zinc contaminated cement stabilized Brown Kaolin clay at different curing time	228
4.75	The variations of compression index for uncontaminated and copper–zinc contaminated lime stabilized Brown Kaolin clay at different curing time	228

LIST OF ABBREVIATIONS AND SYMBOLS

AAS	-	Atomic Adsorption Spectrophotometer
ADU	-	Acquisition data unit
Ag	-	Silver
Al	-	Aluminum
Al^{3+}	-	Aluminum cation
Al_2O	-	Aluminium Oxide
$Al(OH)_3$	-	Aluminum hydroxide
ASTM	-	American Society of Testing Material
As	-	Arsenic
Au	-	Gold
B	-	Born
Ba	-	Barium
BET	-	Brunauer Emmett and Teller
BS	-	British standard
C	-	Carbon
Cc	-	Compression Index
Ca	-	Calcium
Ca^{2+}	-	Calcium Cation

$CaCO_3$	-	Calcium carbonate
CaO	-	Calcium oxide
$Ca(OH)_2$	-	Calcium hydroxide
$CaSO_4$	-	Calcium sulphate
CAH	-	Calcium Aluminate Hydrate
CASH	-	Calcium aluminate silicate hydrate
C_3A	-	tricalcium aluminate
C_4AF	-	tetracalcium aluminoferrite
CEC	-	Cation Exchange Capacity
Cl	-	Chloride
CSH	-	Calcium Silicate Hydrate
C_3S	-	tricalcium silicate
C_2S	-	dicalcium silicate
CT	-	Cement Treated
Cu	-	Copper
Cu^{2+}	-	Copper cation
$Cu(NO_3)_2$	-	Copper dinitrate trihydrate
Cd	-	Cadmium
Cr	-	Chromium
Co	-	Cobalt
CO_2	-	Carbon dioxide
d	-	distance of interplanar spacing as function of θ
D	-	Dielectric constant of medium
DDL	-	Diffuse Double Layer
DOE	-	Department of Energy

EDAX	-	Energy dispersive x-ray analysis
EE	-	Equilibrium extraction
Fe	-	Iron
Fe^{2+}	-	Iron (II) cation
Fe^{3+}	-	Iron (III) cation
Fe_2O_3	-	Ferric Oxide
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared
G	-	Gibbsite
H	-	Hydrogen
H+	-	Hydrogen cation
H2O	-	Water
ICL	-	Initial Consumption of Lime
ICP	-	Inductively Coupled Plasma
K	-	Potassium
K^{+}	-	Potassium cation
KBr	-	Potassium bromide
KC	-	Kaolin Clay
KCT	-	Kaolin Cement Treated
KLT	-	Kaolin Lime Treated
KUT	-	Kaolin Untreated
LC	-	Laterite Clay
LCCT	-	Laterite Clay Cement Treated
LCLT	-	Laterite Clay Lime Treated
LL	-	Liquid limit

LOI	-	Loss on ignition
LT	-	Lime treated
LCUT	-	Laterite Clay Untreated
LVDT	-	Linear Variable Displacement Transducer
MDD	-	Maximum dry density
mEq	-	milliequivalents
Mg	-	Magnesium
MgO	-	Magnesium oxide
Mn	-	Manganese
n	-	order of diffraction
n_0	-	electrolyte concentration
Na	-	Sodium
Na^+	-	Sodium cation
Na_2O	-	Sodium oxide
NH^{4+}	-	Ammonium ion
Ni	-	Nickel
NMHPPE	-	Netherlands Ministry of Housing, Physical Planning and Enviroment. Leidschendam
NO_3	-	Nitrate
NWC	-	Nuclear Weapons Complex
O	-	Oxygen
$(OH)^-$	-	Hydroxide ion
OMC	-	Optimum Moisture Content
OPC	-	Ordinary Portland Cement
P	-	Phosphorous

Pb	-	Lead
PI	-	Plasticity index
PL	-	Plastic Limit
ppm	-	parts per million
PP	-	Potassium propanoate
Q	-	Quartz
S	-	Sulfur
S/S	-	Stabilization/ Solidification
SEM	-	Scanning Electron Microscope
Si	-	Silicon
SiO_2	-	Silica
SO_4	-	Sulfate
SSA	-	Specific Surface Area
T	-	Temperature
UCS	-	Unconfined Compressive Strength
USEPA	-	Uinited Stated. Environmental Protection Agency
UT	-	Untreated
v	-	volume of gas adsorbed per unit weight of clay at a pressure
vm	-	volume of gas adsorbed for monolayer coverage
XRD	-	X-ray diffraction
Zn	-	Zinc
Zn^{2+}	-	Zinc Cation
$Zn(NO_3)_2$	-	Zinc nitrate tetra hydrated
1/k	-	the effective thickness of the diffuse layer
v	-	cation valence

ε_0	-	permittivity of vacuum
ε	-	Strain
μ	-	Micro
λ	-	Wave-length
θ	-	critical angle of incidence of the x-ray beam on the crystal plane

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	The data of the particle size distribution (PSA)	270
B	Table B.1 : XRD results of untreated, lime -cement treated, and cement-lime treated Laterite and Kaolin clay soils contaminated by copper and zinc	275
C	Samples of data obtained from EDAX analysis	297
D	Calibration of ICP for Cu and Zn analysis	320
E	Data obtained from leaching test	325
F	Datat obtained from CEC test	330
G	Samples of data obtained from BET analysis	334

CHAPTER ONE

INTRODUCTION

1.1 Background

Raising public awareness regarding the harmful effects of environmental contamination (land, water and air) has grown considerably over the last two decades partly because of the active participation of environmental groups. The United Nations held a Conference on the Environment and Development (UNCED, 1992) in Rio de Janeiro to initiate international collaboration on environmental problems. Afterwards, the conference acknowledged basic principles and established a plan of actions for the achievement of sustainable development, referred to as the 'Rio Declaration' (UNCED, 1992). Part of this declaration calls for researchers to focus on finding applications and optimal technical approaches for the management of toxic chemicals.

Due to the growth of industrial activities that produce materials and chemicals that comprise large amounts of heavy metal-bearing wastes. Thus, the disposal of chemical contamination has become one of the major problems currently faced by developing countries. This has consequently led to increasing pressures on governments to urgently enact new legislation for the control and disposal of such waste.

Large sources of contamination come from industrial processes. Furthermore, the composition of contaminants varies enormously from one industrial process to another. Typical sources of contamination are copper, zinc and lead smelting industries; the manufacture of duplication and photography equipment; as well as pharmaceuticals and sewage treatment, among many others. Through the study of waste materials-known as waste streams or sludge-produced by modern processes, well qualified indications for environmental contamination caused by industrial process can readily be obtained. These contaminants include sedimentation in rivers, canals, lakes and reservoirs as a result of air and/or water transport as well as direct contact with land (Lutz and Minehan,1986). Therefore, any readily applicable technology that easily handles large amounts of contaminated soil and sedimentation that does not require excessive amounts of expensive additives, is of great importance.

As regards contamination issues in Malaysia, it poses major problems as the country's environmental degradation shows similar trends to that of most of other nations (DOE, 1999). In terms of the heavy metal contamination of water, it has been noted that the two major sources of water pollution are the industrial manufacturing sector and wastewater treatment, accounting for 43.7% and 45.5% of contaminants, respectively (Musta, 2003).

Generally , soil contamination in Malaysia remains under control and at low levels. Nevertheless, some sectors such as mining waste containment areas and landfills are of growing interest because of the high risk to the environment from chemical degradation, particularly heavy metals that can pollute land and water systems via leaching.

In cases of landfill design, to avoid heavy metals such as copper, zinc, lead and nickel contained in hazardous wastes from passing into the adjacent environment, the initial step is landfill location and design for containments should be removed as far as possible from groundwater tables and withdrawal wells. The second step is to modify or stabilize the clay liners which increase the soil's capacity to adsorb and immobilize heavy metals. Furthermore, the waste itself may also be stabilized with suitable chemicals such as lime or cement in order to improve the physical properties of the soil that subsequently increase its durability and strength and also reduce its compressibility(Asavapisit et al, 2005). Therefore, understanding and analyzing physical characteristics, chemical properties, mineralogical identification, as well as microsturacture studies of both soil and waste, were the main interest of this thesis.

1.2 Problem Statement

Owing to rapid economic growth over the past decades there have been increased in road transport (interstate highways) and construction projects. Indirectly, an increased presence of weak clay deposits at various contaminated sites has also been noticed because of the growth of numerous industrial centers in the world. These are often contaminated by metal pollutants primarily due to inadequate waste disposal and chemical leakage, as well as fall-out from atmospheric emissions. In addition, the variability of contaminants in clay deposits and sediments makes it difficult to find unique treatment systems that satisfy all requirements for ‘safe’, long-term storage of such material. Hence, various techniques, in situ and ex situ, have been used which aimed to reduce the impact of these metals on the soil. Furthermore, the main objective of these processes was either to modify the path from the source or to inhibit negative effects in the future (Spence and Shi, 2004;USEPA, 1999, 2004). Disposal methods that are currently available as well as on-going research include spreading, dehydration and disposal in landfills; encapsulation and burial; in addition to stabilization and disposal on land or in landfills (Musta, 2003).

With regards to excavation, replacing contaminated soil with high quality soil imposed significant costs on construction projects. In addition, this may involve only the excavation and relocation the contaminants from one site to another but the trace of elements cannot be destroyed. Therefore, it is not always possible, in many cases, especially with the huge areas of contamination due to its high costs.

Hence, stabilization/solidification, as an alternative and convenient technique that is less environmentally hazardous, has been applied. It is one of the obvious advantages of choosing stabilization/solidification compared to other soil remediation techniques are its cost effectiveness means, relatively quickly and the ability to use this technology through in-situ and ex-situ. This method includes the use of chemicals and emulsions as auxiliary soil compactors as binders and water-repellents that modify soil behaviour and are more appropriate and efficient (Park, 1999 and Ganjidoust et al., 2009). Furthermore, regarding economic considerations, it is less costly when compared to other treatments such as vitrification, organic encapsulation (e.g. thermo-regulation of plastic), or electro-osmosis.

Generally any technique that is uncomplicated and capable of handling large amounts of contaminated soil and, furthermore, does not require large amounts of costly additives is of great importance in this research. . However, to realize the potential effectiveness for the modification of specific engineering properties of the site's tropical soil, it is essential to study time-dependent, soil-stabilizers- heavy metal interactions in terms of leaching and microstructural characteristics, in addition to molecular and mechanical characteristics.

1.3 Motivations and significance of this study

The process by which heavy metals interfere with the strength and compressibility of cement and lime stabilizers is not fully understood. Hence, there is a need for a comprehensive study, specifically in Malaysia. Indeed, to this writer's knowledge, there are no known research publications indicating the effects of heavy metals on the compressibility of treated contaminated clay

In addition, there are no known studies that precisely quantify the types and amounts or effectiveness of required binders on the chemical stabilization process of contaminated soils that prevents the leaching of contaminates to ground level. Moreover, as a specific Malaysian study, to date there is no evidence of any microstructural studies having been conducted that would help explain the engineering behaviours of cement-lime stabilized soils that are contaminated with heavy metals.

Therefore, by identifying the physicochemical behaviour of contaminated clay soils, this study would address the abovementioned gaps in knowledge and may be of some significance for geotechnical engineers and/or land development agencies in their planning of projects that involve landfill sites, contaminated soils, and mining waste containment. Furthermore, it should also be mentioned that this study takes a novel approach to geotechnical engineering studies by evaluating and associating the geotechnical, environmental, and chemical components of the micro-structural, molecular, and chemical characteristics of treated contaminated clay soils, specifically in Malaysia.

1.4 Objectives of Study

This thesis aims to identify and assess the various chemical reactions involved in the process of stabilizing contaminated tropical soils. To achieve this goal, the following objectives were established:

a) Evaluating the microstructural characteristics of heavy-metals-stabilizer matrices in contaminated soil by determining the changes induced in morphology, mineralogy, and elemental composition of treated samples.

b) Studying the molecular structure in terms of functional groups and local bonding in an attempt to assess the extent of the stabilizer's penetration and the crystalline structure of heavy metals in the clay and its intermediate layers.

c) Determination the effect of such stabilizers on heavy metal mobility within the clay structure and its pore water chemistry by performing leaching tests on cured samples. Furthemore, to verify and complement the data obtained in previous step by performing additional chemical analysis, to include the total exchange capacity (CEC) and pH as well as an analysis of the surface area of cured samples

d) Monitoring the trend developments of strength and compressibility in order to assess whether or not heavy metals enhance or inhibit 'stabilizer hydration' by applying unconfined compression strength and consolidation tests on samples before and after soil contamination by heavy metals.

1.5 Scope of Study

To accomplish the objectives of this study, the behaviour of artificially contaminated treated clay soils were investigated. To implement this, laboratory tests were carried out which included the following assessments: engineering properties (strength and compressibility), chemical, mineralogical, and micro-structural assays of treated soils before and after contamination. Full-scale testing samples were prepared and cured in a similar manner to those described by the British Standard (BS 1924, Part 2: 1990).

Two types of soils were selected for this study; both are typical of the structural properties and environmental conditions experienced in tropical regions. The first type is a brown kaolin powder supplied from Tapah located in Malaysia peninsular. The second is highly weathered lateritic clay that is commonly found in tropical regions, obtained from the campus of Universiti Teknologi Malaysia (UTM). Hence, both can serve as indices for comparison purposes with a view to predict interactions of heavy metal–stabilizers with pure industrial soil and natural tropical soil.

Several types of chemical stabilizers have been used in previous studies. In this study, traditional stabilizers such as hydrated lime $Ca(OH)_2$ and ordinary Portland cement (OPC) were used. These stabilizers were selected due to their low cost and available prevalence, especially in Malaysia.

The sources of contamination used for this study were copper nitrate trihydrateate ($Cu(NO_3)_2$ * 3 H_2O), which provided the copper (II) ion (Cu^{2+}), and zinc in the form of zinc nitrate tetrahydrate ($Zn(NO_3)_2$ * 4 H_2O), which provided the zinc (II) ion (Zn^{2+}). Both were of analytical reagent grade, supplied in solid forms by Merck KGaA-Germany. The contaminants, Cu^{2+} and Zn^{2+}, were selected due to their frequent presence at many contaminated sites as reprted by many previous studies in Malaysia (Ismail *et al*.,1993 ;. Baker *et al*., 1985; Yin *et al*.,2007; and Zain *et al*.,2004) The heavy metals were mixed with treated soil individually to check their effects on the stabilization process.

Contaminated clay soils were prepared in the laboratory by mixing each selected soil with different amounts of both copper and zinc. The respective percentages of pollutant were assumed as indicative values commonly found in contaminated soil (0.1%) from industrial sources (soil that typically surrounds copper or zinc manufacturing processes (at 5%), and more highly contaminated soil (at 10%).

For this research, the strength and compressibility behaviour of contaminated and uncontaminated clay soils treated with lime-cement were determined at 7, 14, 28, 100 and 200 days of curing. This curing sequence was chosen because it encompasses the period of the modification process which then provides reasonable approximations of the long term stabilization process.

Moreover, it was important to compare the effectiveness of both stabilizers and, furthermore, to evaluate the interactions of clay minerals with heavy metal stabilizers. Similar amounts of stabilizers and heavy metals were used for all samples.

1.6 Thesis structure

This thesis aims to investigate the physicochemical mechanisms of treated contaminated clay-lime/cement specimens.

Chapter 1 contains a brief background on the current issues of contamination of surrounding environment. In addition, it describes the role of chemical stabilization and the solidification of contaminated clay along with the need to understand mechanisms associated with the process.

Chapter 2 critically review in depthe the literature on the subject of sources of heavy metals in soil and their concentrations in contaminated soil, and provides examples of contaminated sites. It also goes beyond the basics of clay mineralogy to present a greater understanding of contaminant interactions. Furthermore, several chemical stabilization techniques and their recent revisions are described and then subsequently followed by hypothesized mechanisms for the formation of reaction products. We considered this an essential component that would allow engineers to better appreciate the complex chemical interactions that are discussed in later chapters in the thesis.

Chapter 3 describes our complete chemical analytical approach and the methods used to monitor the structure of the treated contaminated soils under observation. All laboratory assays used to determine the geotechnical characteristics of the soil studied followed procedures as described in the British Standard. Moreover, spectroscopic and microscopic techniques were described to analyze the characteristics of contaminated stabilized soils based on precedent standards as made available in published papers.

Chapter 4 presents the microstructural and molecular characteristics of soils that are stabilized by lime and cement, in addition to their physical and geotechnical properties. This was done in order to study the effect of stabilizers on the geotechnical properties and microstructural properties in treated soils. In addition, it describes our investigation of physicochemical behaviours of stabilized contaminated soils in the laboratory via chemical analysis, as well as micro-structural microscopic techniques, and geotechnical analysis.

Finally, **Chapter 5** summarizes the findings by presenting the results of this study and further highlights areas where future research might be undertaken.

4) The study of heavy metals effects on treated soil focused on mechanical properties such as strength and compressibility in addition to chemical and microstructural aspects. We therefore recommend further analyses of contaminated soils to evaluate the effect of heavy metals on compaction, atterberg limits, particles size distribution, and permeability.

5) This study also based its purview on the use of traditional stabilizers due to their suitability and low cost. The latter is extremely important in the treatment of contaminated soils, especially when contaminated areas are extensive. However, alternative binders of an acidic nature should be compared with the effects of alkaline binders.

6) There is a lack of knowledge regarding requirements for the design and construction of contaminated sites. Hence, with the support of government agencies, specific contaminated soil sites that pose problems can be made available to geotechnical, environmental engineers and researchers from different establishments throughout Malaysia. The data obtained using diverse remediation techniques can then be shared-with and reviewed-by the national research community and industrial players. Depending on results from laboratory scaled investigations and actual field conditions, standard guidelines regarding the design of any construction on contaminated area or landfill can then be established with better qualifications for engineers to predict degrees of improvement when using different stabilizers.

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