



UNIVERSITI PUTRA MALAYSIA

***REINFORCEMENT EFFECTS OF NANO-MODIFIED COIR FIBRES ON
LIME-TREATED MARINE CLAY***

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LIME-TREATED MARINE CLAY**

By

VIVI ANGGRAINI

**Thesis Submitted to the School of Graduate Studies Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

November 2015

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DEDICATED

Mum and Dad, *Hj. Rasiha* and *Ir. H. Abdullah*

And

Beloved sons, *Athar* and *Tariq*



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Doctor of Philosophy

**REINFORCEMENT EFFECTS OF NANO-MODIFIED COIR FIBRES ON
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By

VIVI ANGGRAINI

November 2015

Chairman : Professor Bujang Bin Kim Huat, PhD
Faculty : Engineering

Marine clay soils under land-based structures develop shrinkage cracks due to the uneven distribution of moisture. Treatment of marine clay soils with lime is one of the widely used methods. However, the soils treated with lime will cause to brittle failure. Therefore, to improve the mechanical properties of treated soil, the lime treatment technique combined with inclusion of randomly distributed tensile reinforcement elements such as natural fibres (e.g., coir fibre) were used. However, the mechanical performance of the treated soil depends not only on the nature of the soil, moreover on the mechanical properties of the fibre as well the interaction between the fibre and the lime-treated soil.

This research was developed to further increase the performance of coir fibre in lime-treated soil as pile-supported earth platform. A nano impregnation method was applied through chemical treatment with different chemicals including CaCl_2 , MgCl_2 , AlCl_3 and FeCl_3 in order to impregnate fibres with nano-particles. To confirm the alteration of morphology in the fibres and understand the underlying mechanisms of chemically treated fibres, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) tests were performed. Furthermore, the mechanical properties of the composites were assessed. Unconfined compressive strength tests, indirect tensile strength tests, flexural strength tests, and triaxial compressive strength tests were carried out on original soil, lime-treated soil, and lime-treated soil reinforced with nano-treated and untreated fibre. Moreover, a durability test was conducted to scrutinize the change in the strength of the reinforced soil. Finally, the experimental results were used in a numerical analysis using commercially available software (ABAQUS CAE) to investigate the performance of the proposed treatment as pile supported earth platform. The physical model experiments were performed to validate numerical model.

The results revealed that the nano impregnation of fibres increased the tensile strength up to 200% compared with untreated fibres. The fibres modified with Ca(OH)_2 showed higher mechanical performance compared with the fibres modified by Mg(OH)_2 , Al(OH)_3 , and Fe(OH)_3 . The SEM/EDX results showed that cellulosic pores of the fibres

were filled with Ca nano-sized crystals ranging from 25 to 150 nm. The mechanical performance of the treated soil increased when chemically treated fibres were used. The compressive strength, indirect tensile strength, and flexural strength of the treated soil increased by 66, 122, and 60% when $\text{Ca}(\text{OH})_2$ -treated fibres were used compared with those of limed soil reinforced with untreated fibres. Moreover, the addition of nano impregnated fibres using $\text{Ca}(\text{OH})_2$ increased the shear strength parameters of marine clay soil with increases in the level of confining pressure and consequently led to a more ductile behaviour. The numerical analyses show the importance of the mechanical properties of the treated soils are effective in reducing the differential settlement up to 50% when the height of the earth platform used is 0.3 m. The research is important in that it confirms that the nano modification technique can not only increase the mechanical performance of the coir fibres but also improve the interfacial mechanical interactions between the fibre surface and soil particles, resulting in a higher performance of the composites used as a pile-supported earth platform.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai mementuhi keperluan untuk Ijazah Doktor Falsafah.

KESAN PENETULANGAN OLEH SABUT KELAPA TERUBAH SUAI NANO TERHADAP TANAH LIAT MARINE YANG TERAWAT KAPUR

Oleh

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Tanah liat marin di bawah struktur berasaskan tanah membangunkan retak pengecutan disebabkan oleh pengagihan kelembapan yang tidak sama rata. Rawatan tanah liat marin dengan kapur adalah salah satu kaedah yang digunakan secara meluas. Walaubagaimanapun, tanah yang dirawat dengan kapur akan menyebabkan kegagalan rapuh. Oleh itu, untuk menambahbaik prestasi mekanikal tanah dirawat, teknik rawatan kapur digabungkan dengan kemasukan secara rawak elemen tetulang tegangan seperti gentian semula jadi (contohnya, gentian sabut kelapa) di dalam tanah telah digunakan. Walaubagaimanapun, prestasi mekanikal tanah yang dirawat bukan sahaja bergantung kepada jenis tanah tetapi juga sifat-sifat mekanik gentian dan interaksi antara gentian dan tanah kapur yang dirawat itu.

Kajian ini telah dibangunkan untuk meningkatkan lagi prestasi sabut kelapa di dalam tanah yang telah dirawat dengan kapur sebagai sokongan cerucuk platform bumi. Satu kaedah pengisitepuan nano telah diaplikasikan melalui rawatan kimia dengan bahan kimia yang berbeza termasuk CaCl_2 , MgCl_2 , AlCl_3 dan FeCl_3 untuk mengisitepukan serat dengan zarah-zarah nano. Untuk mengesahkan perubahan morfologi dalam gentian dan memahami mekanisme asas sabut kelapa yang telah dirawat secara kimia, Ujian Mikroskop Imbasan Elektron (SEM) dan X-ray Serakan Tenaga Spektroskopi (EDX) telah dijalankan. Tambahan pula, sifat-sifat mekanikal komposit adalah dikaji. Ujian Mampatan Tak Terkurung, Ujian Kekuatan Tegangan Tak Langsung, Ujian Kekuatan Lenturan, Ujian Mampatan Tiga Paksi telah dijalankan ke atas tanah asli, tanah yang dirawat dengan kapur, dan tanah yang dirawat dengan kapur yang diperkukuhkan lagi dengan gentian yang dirawat dan tidak dirawat oleh nano. Selain itu, ujian ketahanan telah dijalankan untuk meneliti perubahan dalam kekuatan tanah yang bertetulang. Akhirnya, keputusan eksperimen telah digunakan di dalam analisis berangka dengan menggunakan perisian yang tersedia secara komersil (ABAQUS CAE) untuk menyiasat prestasi rawatan yang dicadangkan sebagai longgokan disokong platform bumi sebagai sokongan cerucuk platform bumi. Eksperimen model fizikal telah dijalankan untuk mengesahkan model berangka.

Keputusan menunjukkan bahawa pengisitepuan nano di dalam sabut kelapa telah meningkatkan kekuatan tegangan gentian sehingga 200% berbanding dengan gentian yang tidak dirawat. Gentian yang diubahsuai dengan Ca(OH)_2 menunjukkan prestasi mekanikal yang lebih tinggi berbanding dengan gentian diubahsuai oleh Mg(OH)_2 , Al(OH)_3 , dan Fe(OH)_3 . Keputusan SEM / EDX menunjukkan bahawa liang berselulos sabut kelapa dipenuhi dengan Ca bersaiz nano kristal antara 25-150 nm. Prestasi dari sifat –sifat mekanik tanah tanah yang dirawat adalah meningkat apabila gentian yang dirawat secara kimia telah digunakan. Kekuatan mampatan, kekuatan tegangan tidak langsung, dan kekuatan lenturan tanah yang dirawat didapati meningkat sebanyak 66, 122, dan 60% apabila gentian yang dirawat dengan Ca(OH)_2 digunakan berbanding dengan tanah yang dirawat oleh kapur diperkukuh dengan gentian yang tidak dirawat.

Selain itu, penambahan pengisitepuan nano di dalam gentian menggunakan Ca(OH)_2 meningkatkan parameter kekuatan ricih tanah liat marin dengan peningkatan dalam tahap tekanan mengurung dan seterusnya menjadikan kepada tingkah laku yang lebih anjal. Analisis berangka menunjukkan kepentingan sifat-sifat mekanik tanah dirawat bagi keberkesanan dalam pengurangan penyelesaian pengkamiran platform bumi. Nilai keberkesanan untuk tanah yang dirawat dengan kapur serta diperkukuhkan dengan semua jenis pengisitepuan nano gentian sabut kelapa adalah sehingga 50% di bawah pelbagai beban struktur apabila ketinggian efektif platform bumi adalah 0.3 m. Penyelesaian pengkamiran pada ketinggian kepala cerucuk semakin berkurangan sehingga 100%.

Kajian ini adalah penting kerana ia mengesahkan bahawa teknik pengubahsuaian nano bukan sahaja boleh meningkatkan prestasi mekanik gentian tetapi juga meningkatkan interaksi mekanikal antara muka di antara permukaan gentian dengan zarah tanah, menyebabkan prestasi yang lebih tinggi bagi komposit yang digunakan sebagai sokongan cerucuk platform bumi.

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I certify that a Thesis Examination Committee has met on 5 November 2015 to conduct the final examination of Vivi Anggraini on her thesis entitled "Reinforcement Effects of Nano-Modified Coir Fibres on Lime-Treated Marine Clay" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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TABLE OF CONTENTS

	Page
ABSTRACT	i
ABSTRAK	iii
ACKNOWLEDGEMENTS	v
APPROVAL	vi
DECLARATION	viii
LIST OF TABLES	xiii
LIST OF FIGURES	xiv
LIST OF ABBREVIATIONS	xviii
CHAPTER	
1 INTRODUCTION	1
1.1 Introduction	1
1.2 Problem of Statement	3
1.3 Objectives of Thesis	3
1.4 Scope of Study	4
1.5 Thesis Organisation	5
2 LITERATURE REVIEW	6
2.1 Introduction	6
2.2 Marine clay soil	7
2.2.1 Peninsular Malaysia marine clay soil	7
2.2.2 Characteristics of Klang marine clay soil	8
2.2.3 Marine clay soil problem	10
2.3 Soil stabilisation	11
2.4 Soil reinforcement	12
2.4.1 Coir fibres as soil reinforcement	16
2.4.2 Coir fibre structure	17
2.4.3 Natural fibre treatment	18
2.5 Potential application of lime and fibre reinforcement	21
2.6 Pile supported earth platform	22
2.6.1 Load transfer mechanism of pile supported load transfer platform	23
2.6.2 Numerical modelling	26
2.6.3 Physical modelling	29
2.6.4 Potential of lime and fibre as earth platform material	30
2.7 Summary	30
3 METHODOLOGY	32
3.1 Introduction	32
3.2 Sampling location	34
3.3 Material preparation	34
3.4 Original soil investigation	37
3.4.1 XRF analysis	37
3.4.2 XRD analysis	37
3.4.3 Salinity measurement	37
3.4.4 pH test	38

3.4.5	Organic content test	38
3.4.6	Plasticity index test	39
3.5	Fibre treatment	40
3.5.1	Quick precipitation method	40
3.5.2	Scanning electron microscope (SEM)/Energy dispersive X-Ray spectrometer	42
3.5.3	X-Ray diffractometry	43
3.5.4	Single fibre tensile strength test	43
3.6	Performance of coir fibre reinforced limed soil	43
3.6.1	Preparation of specimens	43
3.6.2	Unconfined compression strength test	45
3.6.3	Indirect tensile strength test	46
3.6.4	Flexural strength test	47
3.6.5	Triaxial compression strength test	48
3.6.6	Durability	49
3.6.7	Scanning electron microscopy test (SEM)	49
3.7	Use of lime and treated coir fibre reinforced soils as pile supported load transfer platform	50
3.7.1	Physical model	50
3.7.2	Numerical model	52
4	RESULTS AND DISCUSSION	55
4.1	Introduction	55
4.2	Results organization	55
4.3	Geotechnical properties of Klang marine clay soil	56
4.4	Morphological and mechanical characteristics of nano modified coir fibre through chemical treatment	58
4.4.1	Nanoparticles impregnate coir fibre mechanism	58
4.4.2	Morphological changes	59
4.4.3	Material characterization for untreated and treated fibres	63
4.4.4	Tensile strength of untreated and treated single coir fibre	65
4.5	Influence of lime and different amount of coir fibre on mechanical properties	66
4.5.1	Moisture density relations	66
4.5.2	Plasticity index	67
4.5.3	Unconfined compression strength	67
4.5.3.1	Effect of lime and various percentage of unmodified coir fibres	67
4.5.3.2	Effect of lime and nano modified coir fibres	68
4.5.4	Indirect tensile strength	71
4.5.4.1	Effect of lime and various percentage of unmodified coir fibres	71
4.5.4.2	Effect of lime and nano modified coir fibres	72
4.5.5	Flexural strength	74
4.5.5.1	Effect of lime and various percentage of unmodified coir fibres	74
4.5.5.2	Effect of lime and nano modified coir fibres	76
4.5.6	Triaxial compression test	79
4.5.7	Effect of repetitive wetting	84
4.5.8	Microstructural study	86

4.5.9	Pattern of failure	87
4.6	Physical and numerical model of earth platform	92
4.6.1	Comparison between experimental and finite element analysis (FEA)	92
4.6.2	Parametric studies	93
4.6.3	Settlement	93
4.6.4	Vertical stress	94
4.6.5	Efficacy	95
4.6.6	Bending performance of earth platform	97
5	CONCLUSIONS AND RECOMMENDATIONS	99
5.1	Summary	99
5.2	Conclusions	100
5.3	Recommendations	101
	REFERENCES	102
	APPENDICES	110
	BIODATA OF STUDENT	119
	LIST OF PUBLICATIONS	120

LIST OF TABLES

Table		Page
2.1	Materials investigated in some previous studies	15
3.1	The chemical and physical analysis of lime (Cao Industries Sdn Bhd)	36
3.2	The chemical and physical analysis of coir fibre	37
3.3	Concentration and composition of chemical used for treatment of coir fibre	40
3.4	Mixture of the tested materials of untreated coir fibres	44
3.5	Mixture of the tested materials of treated coir fibres	44
3.6	Model parameters of the unreinforced and reinforced soils	54
4.1	Basic Properties of Klang Marine soil used in this study	57
4.2	Chemical Composition of Klang Marine Clay	58
4.3	Atterberg limits of lime reinforced soil	67
4.4	Unconfined compressive strength after wetting and drying	85
4.5	Summary of the results at 90-day curing	91
4.6	Deflection of earth platform and vertical stress on soft soil ground midway	98

LIST OF FIGURES

Figure		Page
2.1	Quaternary Sediments in Peninsular Malaysia (after Stauffer, 1974)	8
2.2	Depth profile, specific gravity (Gs) and Atterberg's limit of the Klang clay deposit (Taha, 2000)	9
2.3	Fibre orientation in composites	13
2.4	Mechanism of fibre-reinforced soil (after Gray and Ohashi, 1983)	13
2.5	Structure of natural fibre (John and Anandjiwala, 2008)	17
2.6	Mechanism of alkali treatment of natural fibre (After Chowdhury et al.2013)	20
2.7	Rigid inclusion execution (Okyay and Dias, 2012)	22
2.8	Treated soil as pile supported load transfer platform	23
2.9	Analytical Model: Semi-cylindrical Earth Platform arches (after Low et al., 1994)	24
2.10	Geotextile overlying Cap Beams and Soft Ground (after Abusharar, 2012)	25
2.11	Numerical model grid (Jenk et al 2007)	26
2.12	Finite element model of geosynthetic reinforced piled embankment (a) and vertical stress (S22) on pile and soil layers (Bhasi & Rajagopal, 2007)	27
2.13	Finite element model that showing soil arching with 45° angle	28
2.14	The simulated zone and mesh distribution (Okyay & Dias, 2010)	28
2.15	Typical Scheebeli small-scale model test	29
3.1	Overall schematic presentation of the study	33
3.2	(a) The location of the sampling point of the marine clay at Port Klang, Malaysia	34
3.3	Grinding of marine clay soil	35
3.4	Homogenization of marine clay particles	35
3.5	Short coir fibre as reinforcement	36

3.6	Salinity test of Klang marine clay	38
3.7	Digital Calibrated pH Probes	38
3.8	Organic content test of Klang marine clay	39
3.9	Index plasticity test	39
3.10	Schematic diagram of the system used in the modification of coir fibre	41
3.11	SEM/EDX sample preparation	42
3.12	Sputter/coater samples	42
3.13	Chopped coir fibres	43
3.14	Compaction test of Klang marine clay	45
3.15	Unconfined compression strength test	46
3.16	Indirect tensile strength test	47
3.17	Three point bending test	48
3.18	Triaxial compression test	48
3.19	Soil submerged	49
3.20	Schematic diagram of earth platform model	50
3.21	Test setup and instrumentation detail of the physical model of an earth platform	51
3.22	Schema of the simulated zone and mesh distribution	53
4.1	Particle size distribution	57
4.2	Chemical Structure of (a) $\text{Ca}(\text{OH})_2$ modified coir fibre (b) $\text{Mg}(\text{OH})_2$ modified coir fibre (c) $\text{Fe}(\text{OH})_3$ modified coir fibre (d) $\text{Al}(\text{OH})_3$ modified coir fibre	59
4.3	Natural coir fibre (a) untreated (b) $\text{Mg}(\text{OH})_2$ treated fibres (c) $\text{Ca}(\text{OH})_2$ treated fibres (d) $\text{Fe}(\text{OH})_3$ treated fibres (e) $\text{Al}(\text{OH})_3$ treated fibres	60
4.4	Surface structure (a) unmodified (b-c) modified coir fibre	61
4.5	SEM images (a) cross-section of $\text{Mg}(\text{OH})_2$ impregnated fibre, nano average size 25 ± 4 nm (b) $\text{Ca}(\text{OH})_2$ nano impregnated fibre, nano average size 149 ± 19 nm (c) cross-section of $\text{Fe}(\text{OH})_3$ modified coir	62

	<p>fibre, nano average size 109 ± 15 nm (d) $\text{Al}(\text{OH})_3$ nano impregnated fibre, nano average size 119 ± 19 nm</p>	
4.6	EDX spectrum of (a) unmodified coir fibre (b) $\text{Mg}(\text{OH})_2$ modified coir fibre (c) $\text{Ca}(\text{OH})_2$ modified coir fibre (d) $\text{Fe}(\text{OH})_3$ modified coir fibre (e) $\text{Al}(\text{OH})_3$ modified coir fibre	63
4.7	X –ray diffractograms of the coir fibre particles (a) unmodified (b) $\text{Mg}(\text{OH})_2$ treated fibre (c) $\text{Fe}(\text{OH})_3$ treated fibre (d) $\text{Al}(\text{OH})_3$ treated fibre (e) $\text{Ca}(\text{OH})_2$ treated fibre	64
4.8	Tensile strength of the treated and untreated coir fibres	65
4.9	Dry unit weight vs water content	66
4.10	The values of compressive strength of specimen tested at 7, 28 and 90-day curing	68
4.11	Compressive stress-strain curves of reinforced soils after (a) 7 days (b) 28 days and (c) 90 days	69
4.12	Evolution of compressive strength	70
4.13	The values of indirect tensile strength of specimen tested at 7,28 and 90 days	71
4.14	Load-displacement curves of unreinforced soil and reinforced soils after (a) 7 (b) 28 and (c) 90 days	73
4.15	Evolution of tensile strength	74
4.16	The values of flexural strength of specimen tested at 7, 28 and 90-day curing	75
4.17	The values of Young’s modulus of specimen tested at 7, 28 and 90-day curing	76
4.18	Load-displacement curves of reinforced soils after 7, 28 and 90- day curing	77
4.19	Evolution of flexural strength	78
4.20	Evolution of Young’s modulus	79
4.21	Stress-strain relationship obtained from CU triaxial test at various confining stresses (a) 50 kPa; (b) 100 kPa; (c) 150kPa	80
4.22	Pore pressure in terms of effective strain in unreinforced and reinforced soil under triaxial compression shearing stage at different confining stresses (a) 50 kPa, (b) 100 kPa and (c) 150 kPa	81

4.23	Mohr circles in the case of total stress and effective stress for (a) S (b) SL (c) SLF (d) SLMF (e) SLFF (g) SLCF	84
4.24	Effect of wetting/ drying cycles on the compressive strength of the stabilized and fibre reinforced soil	86
4.25	SEM of fibres in soil	87
4.26	Failure characteristic of unreinforced and reinforced soil of compressive strength test	88
4.27	Failure characteristic of unreinforced and reinforced soil of indirect tensile strength test	89
4.28	Failure characteristic of unreinforced and reinforced soil of three point bending test	89
4.29	Failure characteristic of unreinforced and reinforced soil of triaxial compression test	90
4.30	Experimental observation of the settlements due to surcharge	92
4.31	Effect of height of earth platform on settlement from surcharge load	93
4.32	Effect of the earth platform's mechanical properties on settlement at various surcharge loads of the earth platform's height of 0.05 m	94
4.33	The vertical stress on soft soil ground midway between the pile heads for various earth platform materials	95
4.34	Performance of the material characteristics on the efficacy	96
4.35	The vertical stress on soft soil ground midway between the pile heads at 0.05 m height of earth platform	97

LIST OF ABBREVIATIONS

A	Area of cross-section of sample
Al	Aluminium
Al(OH) ₃	Aluminium hydroxide
AlCl ₃	Aluminium chloride
ASTM	American society for testing and material
BS	British Standard
C	Clay
c	Apparent cohesion
CEC	Cation exchange capacity
Ca	Calcium
CaCl ₂	Calcium chloride
Ca(OH) ₂	Calcium hydroxide
CU	Consolidated-undrained triaxial test
CD	Consolidated-drained triaxial test
D	Diameter of sample
D	Constrained modulus
D	Depth of soft ground
E	Young's modulus of elasticity
E	Efficacy
E'	Drained modulus
EDX	Energy dispersive X-ray spectrometer
F	Applied force
Fe	Iron
FeCl ₃	Ferric chloride
Fe(OH) ₃	Iron (III) hydroxide
G	Shear modulus
Gs	Specific gravity
H	Height of sample
H ₂ O	Water
L	Length of sample
LL	Liquid limit
Mg	Magnesium
MgCl ₂	Magnesium chloride
Mg(OH) ₂	Magnesium hydroxide
NaOH	Sodium hydroxide
NaCl	Sodium Chloride
OH	Hydroxide
OC	Overconsolidated

OCR	Overconsolidated ratio
OMC	Optimum moisture content
P	Applied force
PL	Plastic limit
PI	Plasticity Index
Rc	Compressive strength
Rt	Tensile strength
Rb	Bending strength
SEM	Scanning Electron Microscopy
UU	Unconsolidated-Undrained triaxial test
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
E_s	Elastic modulus of soft ground
E_s	Secant modulus
E_t	Tangent modulus
ESP	Effective stress path
e	Voids ratio
n	Porosity
r	Radius of soil sample
R	Radius of circular arc of geosynthetic
u	Pore pressure
ε	Strain
ν	Poisson's ratio
ν_u	Poisson's ratio (undrained)
ν'	Poisson's ratio (drained)
d	Dry density
σ	Normal stress
σ'	Normal effective stress
$\sigma_1, \sigma_2, \sigma_3$	Principal stresses
$\sigma'_1, \sigma'_2, \sigma'_3$	Principal effective stresses
σ_v, σ_h	Vertical and horizontal stresses
σ'_v, σ'_h	Vertical and horizontal effective stresses
σ_n	Stress normal to surface of failure
σ_l	Axial stress
σ_3, σ_c	Confining pressure
σ'_3, σ'_c	Effective confining pressure
$(\sigma_1 - \sigma_3)$	Deviator stress
$(\sigma_1 - \sigma_3)_f$	Deviator stress at failure
ϕ'	Angle of shear resistance based on effective stresses

σ_s	Vertical stress acting on top of soft ground midway between pile head
γ	Unit weight of earth platform
ϕ	Angle of shearing resistance of earth platform
s	Center to center spacing of piles
s'	Clear spacing between piles, $s' = s - b(m)$
K_p	Rankine passive earth pressure coefficient $K_p = (1 + \sin\phi_s)/(1 - \sin\phi_s)$;
θ	Half angle subtended by geosynthetic circular arc (degree)
t	maximum displacement of soft ground midway between pile heads
T	Axial tension force in geosynthetic

CHAPTER 1

INTRODUCTION

1.1 Introduction

Marine clay soils under land based structures develop shrinkage cracks due to uneven moistures distribution. Consequently, they exhibit considerable variation of shear strength, compressibility and tensile strength which cause differential movement, severe damage in foundations, buildings, roads, embankments, retaining structures, canal lining and etc. (Sivakumar Babu et al., 2008; Ramesh et al., 2010). The problems associated with marine clay soils can be controlled by different techniques such as isolating the soil using geo-membranes or providing an adequate thickness of cohesive non-swelling soil specially given in large-scale projects (Miller, 1997). However, they are expensive in small scale projects such as construction of bunds of smaller height.

One possible solution to this problem is addition of lime in order to immobilize water in marine clay by its chemical reactions and reduce plasticity index of the clay. A reduction in plasticity is usually accompanied by reduction of potential for swelling. Rajasekaran and Rao (1997) reported that lime is commonly used to change properties of soils due to its more stable performance, lower prices, and abundance. Lime is most effective for treating soils capable of holding large amounts of water (Locat et al., 1990; Bell, 1996; Rajasekaran et al., 1997; Rajasekaran and Rao, 2002; Dash and Hussain, 2011). However, soils treated with lime are subjected to a brittle failure (Ninov and Donchev, 2008). Therefore, it is better to amend it with a technique of reinforcement (Ranjan et al., 1994; Ziegler et al., 1998; Yetimoglu and Salbas, 2003; Ninov and Donchev, 2008). So, a possible solution involves inclusion of randomly distributed tensile reinforcement elements in the marine clay. Adding fibres can effectively reduce the number and width of shrinkage cracks and help to obstruct them (Ziegler et al., 1998; Estabragh et al., 2012).

The effectiveness of fibres depends upon the strength of fibre as well as how they interact with soil at normal stresses through adhesion. When a tensile force needs to mobilize in the fibres, as in drying shrinkage and desiccation cracks, adhesion restrains the fibres from pull out and thus allows its tensile resistance to develop. The mechanical properties of fibres reinforced lime treated soil have been investigated by various authors. A number of triaxial tests, unconfined compression tests, california bearing ratio (CBR) tests, direct shear tests, tensile strength tests and flexural strength tests have been conducted on the subject by several researchers in the last few decades (Prabakar and Sridhar, 2002; Yetimoglu and Salbas, 2003; Yetimoglu et al., 2005; Cai et al., 2006b; Tang et al., 2007; Tang et al., 2010; Estabragh et al., 2012; Hejazi et al., 2012; Divya et al., 2013; Estabragh et al., 2013; Hamidi and Hooresfand, 2013). All the previous studies have shown that the addition of fibre-reinforcement caused significant improvement in the strength and increased the stiffness of the soil.

At the same time, there has been a growing environmental consciousness and understanding of the need for sustainable development in recent years, which has raised interests in using natural fibres as reinforcements in soil. The reinforcement of soils with natural fibres such as roots, sisal, coir and palm has recently received a great deal of attention (Ghavami et al., 1999; Prabakar and Sridhar, 2002; Babu and Vasudevan, 2007; Sivakumar Babu et al., 2008; Subaida et al., 2008; Mwashu, 2009; Vinod et al., 2009; Ramesh et al., 2010; Bateni et al., 2011). Of all the natural fibres, coir fibres has the greatest tensile strength and it retains this property even in wet conditions (Eze-Uzomaka, 1991a; Ghavami et al., 1999; Sen and Reddy, 2011b). The reinforcing effectiveness of coir fibre is related to the nature of cellulose and its crystallinity. Cellulose is a natural polymer consisting of D-anhydro-glucose ($C_6H_{10}O_5$) repeating units joined by β -1,4-glycosidic linkages at C1 and C4 position (Nevell and Zeronian, 1985). Each repeating unit contains three hydroxyl groups. These hydroxyl groups and their ability to hydrogen bond play a major role in directing the crystalline packing and also govern the physical properties of cellulose.

Recently, few efforts have been made to enhance the interaction between soil and the coir fibres by modification of the fibres surface. One of the applied methods is the alkali treatment. In this method, a strong sodium hydroxide were used to remove lignin, hemicellulose and other alkali soluble compounds from the surface of the fibres in order to increase the number of reactive hydroxyl groups on the fibre surface to enhance chemical bonding. The removal of these substances also enhanced the surface roughness which increased the unconfined compressive strength of clay soil by 5 to 10% (Dutta et al., 2012). In another study by Ramesh et al. (2011) kerosene, bitumen and varnish were used to coat the coir fibres in order to modify the surface of fibres. It was observed that kerosene increased compression strength by 55% compared to uncoated coir fibre in soil. So far, however, no studies addressed the enhancement of tensile strength in coir fibre to be used in soil.

Nanotechnology has been a recent approach to modify natural fibres by impregnation of nano particles into fibres to improve their mechanical properties as well as introducing a new function onto the surface of fibres. (Chattopadhyay and Patel, 2009; Castellanos et al., 2012; Chowdhury et al., 2013; Khandanlou et al., 2013; Ridzuan et al., 2013).

In this study, the application of using randomly distributed coir fibre as tensile reinforcement elements and lime in the marine clay soil is investigated to be used as pile supported load transfer base layer. Finite element analyses of pile supported load transfer platform are performed using the program ABAQUS CAE 6.11 to investigate the load-transfer mechanism in the piled earth platforms by considering two major factors of influence: the mechanical properties of the earth platform and its height. The differential settlement is used to acquire experimental data for numerical model validation.

1.2 Problem of statement

Marine clays soils are present in many parts of the world and these deposits characterized by poor engineering properties such as high compressibility and very low shear strength. This sediment is mainly deposited along coastal areas of Peninsular Malaysia.

The rapid growth of industrialisation requires and extensive construction of infrastructure in Malaysia. Especially to new projects, the maintenance and upgrading of facilities also provided significant input to the overall developments include the coastal regions where ports and highways are located.

Even though some systematic studies are available on compressibility characteristic and shear strength of marine clay, not much work has been done its tensile strength aspects. Stabilization using lime was successfully done to increase strength and stiffness of marine clay soil. However, this method did not solve brittle problem of treated marine clay. For the utilisation of treated marine clay for the geotechnical structures, care should be taken to ensure that treated soil retains its ductile behaviour after failure.

The idea of reinforcing soil with tensile resisting elements such as synthetic and natural fibre has been commonly recognized in engineering practice. Using coir fibre as soil reinforcement has much advantages due to high tensile strength, good durability, environmentally friendly material and its ability to absorb water. However, the performance of the matrix (lime, fibre and soil) depends not only on fibres strengths but also how they interact with soil. Various techniques have been developed to modify natural fibres such as biological, physical, thermoplastic and nanotechnology. So far, no study addressed the enhancement of tensile strength of the fibres as well as its interaction with soils.

1.3 Objectives of the thesis

This study aims to investigate the mechanical properties of lime treated marine soil reinforced with modified coir fibre as pile supported load transfer platform. A practical approach was developed to impregnate fibres with nano particles of calcium hydroxide, magnesium hydroxide, ferric hydroxide and aluminum hydroxide. The overarching purpose of this study was to increase the tensile strength of fibres and to enhance their interaction with marine clay soil. The following objectives are identified for the successful completion of the aim of this research:

1. To investigate the morphological and mechanical characteristics of nano treated coir fibres as soil reinforcement.
2. To determine the effect of the nano modification of coir fibre and its interaction with lime reinforced marine clay soil.

3. To evaluate the performance of using nano modified coir fibre in lime treated soil as pile supported load transfer platform.

1.4 Scope and limitation of the study

The scope of the study can be presented in a form of three phases and these phases are:

- i. Phase one, in order to increase the strength as well as interactions of natural fibres and soil, green nano- impregnation method is applied. To improve the structures of the fibre, chemical treatment of fibre using different chemicals such as calcium chloride, magnesium chloride, aluminium chloride, and sodium hydroxide were carried out. To confirm the alteration of morphology in the fibres and understand the underlying mechanisms of chemically treated coir fibres, Scanning electron microscopy (SEM), X-Ray diffraction (XRD) and Energy-dispersive X-ray spectroscopy (EDX) tests were performed.
- ii. Phase two, to identify the applicability of the proposed nano-fibre treatment, the mechanical properties of the composites was assessed. Unconfined compressive strength tests, indirect tensile strength tests, flexural strength tests, and triaxial compressive strength tests were carried out on pure soil, lime-treated soil, and lime-treated soil reinforced with nano-treated and untreated fibres. Microstructure tests were performed to observe the interaction between fibres and soil. Moreover, a durability test was conducted to scrutinize the change in the strength of the reinforced soil due to the excessive moisture content in the soil.
- iii. Phase three, the experimental results from phase two were used in a numerical analysis using commercially available software (ABAQUS CAE). The physical model experiments were performed to validate numerical model of pile supported load transfer platform.

The followings are limitations of the present study:

- i. Marine clay soil samples are collected from Klang, Peninsular Malaysia.
- ii. 5% lime of dry weight of soil is used as additive.
- iii. The differential settlement is used to acquire experimental data for numerical model validation.
- iv. Two-dimensional numerical model is analysed to perform pile supported load transfer platform.
- v. Parametric study is developed based on numerical analysis.

1.5 Thesis organisation

This thesis presents different aspects of the potential of coir fibres as reinforcement in lime treated soft marine clay soil as pile supported earth platform. This thesis was organized into 5 chapters.

Chapter 1 gives a general introduction to the subject, problem statement, scope and limitation of the study and in addition of the objectives and outline of the thesis.

In providing a relevant background for the work described in this thesis, Chapter 2 contains a general literature review on characteristics and problem of Klang marine clay was used in the study. Special attention is given to various methods that have been used in marine clays stabilization for many years with various degrees are described. The gaps are identified and the importance of embarking on the current research work has been justified. In addition, the benefit of soft soil reinforcement for structural application such as pile supported earth platform including computational modelling, design method and lab-scale model are elaborated.

Chapter 3 describes the methodology used to fulfill the designated objectives for the research for reinforcing of marine clay soil. This chapter begins with a flow chart describing the general plan for the study, sampling location and continues with the required laboratory tests on some of the most significant physical, chemical and mechanical properties of natural marine clay soil, continue with coir fibre treatment, identification of the performance of lime and untreated coir fibre and treated coir fibre reinforced soil by performing mechanical tests, durability and microstructural tests. Finally, the numerical and physical models of the proposed composites as pile supported load transfer platform are analyzed.

Chapter 4 presents the results of the testing programs described in chapter three. The mechanical characterisation of untreated/treated single coir fibre as soil reinforcement has been investigated. Mechanisms of nano modified coir fibre and their interaction with soil matrix are also explained. The highest reinforcement potential of nano particles modified coir fibre and lime is exploited for marine clay reinforcement (i.e strength and durability). Attention is also paid to the mode of failure of fibre surface modification mixed with marine clay soil subjected to tensile and flexural loading. Furthermore, the interaction between fibres and limed soils are showed. Finally, numerical modelling is discussed and analysed and experimentally validated to reliably model the behaviour of nano modified coir fibre reinforced marine soil as pile supported load transfer platform over soft soil.

Chapter 5 is devoted to conclusions drawn from this study along with highlights topics for future work.

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