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GYPSEOUS SOIL STABILIZATION BY ALKALINE ACTIVATION METHOD

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By

SHAYMAA SADEQ KADHIM

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

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DEDICATION

This thesis is dedicated to Allah, my husband, my parents, and my children.

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

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Chairman : Professor Bujang Kim Huat, PhD
Faculty : Engineering

Gypseous soils cover a large area in Iraq and other parts of the world. In general, these soils are problematic and very sensitive to the moisture content or any wet conditions. In fact, water is able to dissolve the gypsum/salt in the soil and consequently the soluble particles are leached out, causing huge changes in the volume and geotechnical properties of the soil mass. Although cement and lime can improve some engineering properties of gypseous soils, they have several shortcomings, especially when viewed from an environmental perspective (e.g. carbon dioxide emissions). Moreover, when gypseous soil is treated with the calcium-based materials (i.e. cement/lime), the stabilized soil has low durability due to the formation of ettringite, especially when the soil has a high amount of gypsum. Therefore, it is significant to investigate a proper method to stabilize gypseous soil. Alkali-activated binders were used as a new method of soil stabilization in this study. Due to the energy efficiency, the environmental friendly nature of the process, and the excellent resulting engineering properties, alkali-activated binders are fast emerging as materials of choice for soil stabilization.

In this research, soil was collected from Babylon in Iraq. Different types of gypseous soils with different gypsum contents were prepared in order to identify the role of gypsum content in the soil. Fly ash class F was used as a precursor along with two types of activators at different molarities. Mechanical tests including compressive strength and mass loss and microstructural tests including XRD, BET, SEM, EDX, and TGA were performed on mortar before and after treatment and the effect of sulfate attack were investigated in the process. Gypseous soils were then treated with different alkaline activators for different curing times. Afterwards, UCS tests and collapsibility tests using the hydraulic Rowe cell system were performed to assess the effect of the treatment. Microstructural analyses were also performed to investigate the underlying mechanism. Finally, undrained triaxial tests were carried out to investigate the mechanical behaviour of the treated soils.

This research also includes an attempt to find an empirical correlation to predict the collapse index based on soil properties using the results of collapsibility tests.

The results showed that the alkaline activation method could stabilize the soils effectively. The collapse index decreased, and the mechanical performance of the treated soils improved. The microstructural analyses confirmed the durability of the stabilized mass. The study was important as it confirmed that the alkaline activation method played a dramatic role in the improvement of gypseous soil.

In addition, series of Rowe cell and shear strength tests are performed on these three models of collapsible soils under various conditions. The results indicate that the most important parameters affecting soil collapsibility are; fine percent, initial dry unit weight, prewetting pressure and water content. Collapse potential decreases with the increase in initial dry unit weight and water content. It is found that only a relatively small fine percentage is required to yield significant collapse, and collapse potential increases with pressure at wetting and fine particles increase. Rate of increase in the collapse potential decreases as fine particles percentage increases. In un-soaked samples with 13% gypsum (G13), while for 25% gypsum content (G25), the collapse potential was 7.95 (moderately severe), and finally for the high gypsum content of 45% (G45), the collapse potential increased to 10.75 and was rated as severe. As can be seen that a large reduction in collapse potential was recorded with a high concentration of activator. For instance, the collapse potential of gypseous soil with 45% gypsum activated with 30% fly ash activated with 8-M KOH was 8.69%, but when the molarity of the activator was increased to 12 M under the same condition, the collapse potential decreased to 3.7% after 7 days of curing.

On the other hand, the increase in the fly ash content from 10 to 30% reduced the collapse potential at different rates. As can be seen, the collapse potential for the soil with 45% gypsum content was 10.75 and decreased to 8.69, 5.88, and 3.7 in 12-M KOH samples and to 9.09, 6.64, and 4.48 in NaOH samples at 7 days when 10, 20, and 30% geopolymer fly ash was used to stabilize the gypseous soil.

The result of UCS for the gypseous soil with 45% gypsum content treated with fly ash activated with 12-M KOH. It can be observed that the compressive strength of the untreated gypseous soil was 0.531 Mpa and it was increased after treatment with fly ash geopolymerized with 12-M KOH; the addition of 30% activated fly ash led to a significant enhancement, giving a compressive strength of 2.216 MPa with a strain 4.668 after 7 days of curing.

Two collapse-predictive mathematical models are proposed by using the results of 165 Rowe cell tests. These models are of high and acceptable correlation factor of (r^2=0.875and 0.87) and verified by experimental data.

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PENSTABILAN TANAH BERGIPSUM OLEH KAEDAH PENGAKTIFAN ALKALI

Oleh

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Tanah bergipsum meliputi kawasan yang luas di Iraq serta kawasan-kawasan lain di dunia. Secara umum, tanah ini bermasalah dan sangat sensitif pada kandungan lembapan atau keadaan basah. Sebenarnya, air mampu melarutkan gipsum / garam di dalam tanah dan seterusnya zarah larut terlarut lesap, menyebabkan perubahan yang besar pada isipadu dan sifat geoteknikal jisim tanah. Walaupun simen dan kapur boleh meningkatkan beberapa ciri kejuruteraan tanah bergipsum, namun ia mempunyai beberapa kelemahan terutamanya apabila dilihat daripada perspektif alam sekitar (misalnya, pelepasan karbon dioksida). Tambahan pula, apabila tanah bergipsum dirawat dengan bahan-bahan berasaskan kalsium (iaitu simen / kapur), tanah yang distabilkan ini mempunyai ketahanan yang rendah kerana pembentukan *ettringite*, terutamanya jika tanah mempunyai jumlah gipsum yang tinggi. Oleh itu, kajian tentang kaedah yang betul bagi menstabilkan tanah bergipsum adalah penting. Dalam kajian ini pengikat yang diaktifkan oleh alkali digunakan sebagai satu kaedah baru dalam menstabilkan tanah. Kerana kecekapan tenaga, sifat proses yang mesra alam, serta dapat menghasilkan sifat kejuruteraan yang baik, pengikat yang diaktifkan oleh alkali semakin pesat digunakan sebagai bahan pilihan bagi menstabilkan tanah.

Dalam kajian ini, tanah telah diambil dari Babylon di Iraq. Tanah bergipsum yang berlainan jenis dengan kandungan gipsum yang berlainan telah disediakan untuk mengenal pasti peranan kandungan gipsum di dalam tanah. Abu terbang kelas F telah digunakan sebagai pelopor bersama-sama dengan dua jenis pengaktif pada kemolaran yang berbeza-beza. Ujian mekanikal termasuk kekuatan mampatan dan kehilangan jisim serta ujian mikrostruktur termasuk XRD, BET, SEM, EDX, dan TGA telah dilakukan pada mortar sebelum serta selepas rawatan dan seterusnya kesan serangan sulfat disiasat. Tanah bergipsum kemudiannya dirawat dengan pengaktif alkali yang berbeza-beza bagi masa pengawetan yang berbeza-beza. Kemudian, ujian UCS dan ujian boleh runtuh menggunakan sistem sel hidraulik Rowe telah dijalankan untuk menilai kesan rawatan. Analisis mikrostruktur juga dilakukan untuk menyiasat

mekanisme dalamannya. Akhir sekali, ujian tiga paksi tak bersalir telah dijalankan untuk menyiasat perlakuan mekanikal tanah yang dirawat. Kajian ini juga melibatkan usaha untuk mencari korelasi empirik bagi meramal indeks keruntuhan berdasarkan sifat-sifat tanah menggunakan keputusan ujian boleh runtuh. Hasil kajian menunjukkan kaedah pengaktifan alkali dapat menstabilkan tanah dengan berkesan. Indeks keruntuhan menurun dan prestasi mekanikal tanah yang dirawat meningkat. Analisis mikrostruktur mengesahkan ketahanan jisim yang distabilkan. Kajian ini penting kerana ia mengesahkan bahawa kaedah pengaktifan alkali memainkan peranan yang luar biasa dalam pembaikan tanah bergipsum.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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LIST OF ABBREVIATIONS

G	Gypseous soil
G13%	Gypseous soil with 13% gypsum
G25%	Gypseous soil with 25% gypsum
G45%	Gypseous soil with 45% gypsum
F.A	Fly ash
KOH	Potassium hydroxide
NaOH	Sodium hydroxide
C-S-H	Calcium silicate hydrate
A-S-H	Aluminium silicate hydrate
N-A-S-H	Sodium aluminium silicate hydrate
GGBS	Ground granulated blast furnace slag
UCS	Unconfined compression test
MDD	Maximum dry density
OMC	Optimum moisture content
LL	Liquid limit
PL	Plastic limit
PI	Plastic index
XRD	X-ray diffraction
XRF	X-ray fluorescence
SEM	Scanning electron microscopy
EDX	Energy-dispersive X-ray spectroscopy
TGA	Thermogravimetric analysis
BET	Brunauer–Emmett–Teller surface area analysis
C_p	Collapse potential

K	Coefficient of permeability
OPC	Ordinary Portland cement
S.R	Sulfate-resistant cement
MgSO4	Magnesium sulfate
Na_2SO_4	Sodium sulfate

CHAPTER ONE

INTRODUCTION

1.1 Background

In general, one of the most common means of stabilizing gypseous soil is by chemical treatment using cement or lime as an additive to the soil. Gypseous soils are considered as an issue and a riddle, which is the reason why these soils merit consideration. Gypseous soils (with light, moderate, or high gypsum contents) cover several parts of populated areas, especially in arid and semi-arid regions such as the Arabians peninsula, Russia, Armenia, the United States and Spain (Fattah, Al-ani, and Al-lamy 2013). The gypsum content is not only able to restrict water and dissolve in water but can also cause degradation impacts on concrete, metal, and building materials. In spite of that, understanding of the genesis and engineering properties of gypseous soils is essential and can be advanced by conducting research and tests on aspects including the formation process and the reaction of these soils from the construction side. The availability of gypsum in soil influences the soil improvement properties therefore several tests was done to determine the effects of treatment on chemical and engineering properties of gypseous soil and the effects of the soil environment.

The term “gypseous soil” is used for soils that contain gypsum. When gypseous soils are in the dry state, they tend to be stable and more cohesive than they are in wet conditions due to the behaviour of the cementation agent (gypsum) between the soil particles and the water. In other words, there is a great decrease in strength of the soil with an immediate increase in compressibility, especially when the soil is wet or leaching as a result of the dissolution of the gypsum (cementitious agent), so the bonds between the soil particles break down (Nashat, 1990). Gypseous soils are viewed as collapsible soils. In this manner, they are typically thought to be a hazard and they display collapsible behaviour corresponding to huge changes in the soil volume (Razouki et al. 1994).

In order to reduce the collapse index, improvement of the foundation behaviour would be a practical, more economical, and environmentally friendly method, a fundamental investigation that should be embraced to utilize a new technique in geotechnical design to be established in gypseous soil by using fly ash class F (10, 20, and 30%) and activator liquid (NaOH, KOH).

The geopolymer materials are unaffected by sulfate attack compared to cement. Sulfate attack can lead to loss of strength, expansion, sliding of surface layers, and ultimate disintegration when the sulfate ions intrude into the concrete and react with $Ca(OH)_2$ in cement to generate more gypsum. In addition, there is a rise in CO_2 emission as a result of using cement or lime. In this study, alkali activators are used instead of cement and lime because the former have the ability to resist sulfate attack whereas cement is attacked by solutions containing sulfates. Furthermore, the gel produced may not have $Ca(OH)_2$ or mono-sulfo-aluminates due to being formed from materials when calcium content is low. Therefore, when gypseous soil that is stabilized with alkali activation is exposed to sulfate solution, it may not allow gypsum to expand and may even lessen the formation of ettringite in the matrix, meaning that the gel binder produced by alkali activation may not be affected by sulfate attack. The majority of the material in an alkali-activated binder is generally derived from industrial by-products. Therefore it is considered as an environmental friendly product. These binders have been identified as offering the potential for notable greenhouse gas emissions reduction when compared with Portland cement. Consequently, replacing ordinary Portland cement (OPC) or lime with alkali activator can largely reduce energy consumption and CO_2 emission. Therefore, the hypothesis behind using geopolymer [fly ash class F + activator liquid (NaOH or KOH)] instead of cement as stabilizer materials.

1.2 Problem Statement

Gypseous soil demonstrates volumetric changes due to dissolution of gypsum by water flow and increased permeability in the soil matrix, which causes uneven settlement or collapse, especially in wet state. Consequently, engineering construction on gypseous soil is extremely risky as several problems such as cracks, tilting, or differential settlement and composition failure may occur. Gypseous soils are widely distributed in arid and semi-arid areas of the world such as the Arabian peninsula, Russia, Armenia, the United States, Iraq, Iran, and Spain (Boyadgiev & Verheye, 1996; Casby-Horton *et al*., 2015). So far, soil stabilization with cement is a the most common treatment technique and it has been widely used to reduce the collapsibility of gypseous soils under engineering constructions, such as foundations and pavements. Nevertheless, the utilization of cement to treat soils with a large amount of gypsum often triggers side effects (Khattab, 1986).

In general, collapsible soils have porous textures with a high void ratio and relatively low densities. In dry state, they possess high apparent strength, but they are susceptible to large reductions in void ratio upon wetting (Jotisankasa 2005). When the soil is subjected to the wet condition, firstly the metastable texture collapses because the gypsum dissolves and the bonds between grains break down (Seleam, 1988). Then the soil particles become rearranged into a denser state of packing as the dissolved gypsum leaches out, and finally collapse occurs (Bolzon, 2010).

One of the practical techniques used to immobilize gypsum, preventing it from leaching out, is to stabilize it with cement. However, the major problem with stabilization of soil using cement is internal sulfate attack. Degradation of cement paste as a result of chemical reactions between hydrated Portland cement and sulfate ions from an outside source is known from crack propagation and expansion in the cement matrix. Sulfate attack can cause a significant reduction in the strength and mass due to disintegration of C-S-H (Hartell et al. 2011). Production of gypsum from the reaction of Ca^{+2} from C-S-H and calcium hydroxide with SO_4^{-2} is one of the major deterioration mechanisms in sulfate attack (Fallis 2013). Furthermore, exposure of alumina containing hydrates to aggressive water leads to the formation of ettringite, which can also cause expansion and disintegration of the cement matrix. As in cement-stabilized gypseous soil, the sulfate attack is induced in the wet state of the soil due to the availability of a high content of sulfate ions in gypsum, which leads to disintegration of the stabilizing binder. The low integrity of the matrix, as a result, increases the permeability of the binder, facilitates infiltration of water, and increases leaching out of the gypsum. Increased porosity of the soil along with disintegration of stabilizers increases the collapse potential of the soil (Najah, Campus, and Pinang 2013).

Recently, geopolymer binders have been introduced as good replacements for cement due to their comparable mechanical properties along with greater resistance to chemical attacks (John L Provis 2014). The term "geopolymer" was initially coined by Davidovits in the 1970s and was later utilized to describe a class of solid materials synthesized through the reaction of an aluminosilicate powder with an alkaline solution. The geopolymer materials are generally industrial byproducts or other economical materials supplied in powder form and activated with an alkaline activator, which is usually a concentrated aqueous solution of alkali hydroxide, silicate, carbonate, or sulfate (Duxson et al. 2007). The strength gain in the geopolymer is related to the formation of geopolymer gel, which depends on rapid dissolution of the aluminosilicates and release of the tetrahedral units of $[SiO_4]^-$and $[AlO_4]^-$ in the solution. These tetrahedral units are joined by sharing oxygen atoms instead of a polymeric precursor to form aluminosilicate hydrate (A-S-H) (John L. Provis 2014). Due to the energy efficiency and environmental friendly nature of the process as well as the excellent engineering properties, geopolymer binders are fast emerging as materials of choice for highly demanding civil-engineering applications.

The overarching purpose of this study is to use the low-calcium fly ash in gypseous soil stabilizing, which may reduce its collapsibility potential. Fly ash geopolymer may cover the gypsum particles in soil to prevent any contact between the gypsum particles and water. Furthermore, the absence of calcium in the binder structure can provide a sulfate-resistant property by which the vulnerability of the soil to the collapse potential (C_p) is reduced with an increase in gel binder amount. The findings of this study will help to understand the underlying mechanism by which geopolymer can control the internal sulfate attacks caused by the high quantity of gypsum in the soil matrix and to assess the collapse behaviour of gypseous soil stabilized with fly ash geopolymer.

1.3 Aim of the Research

The major aim of this present research is to attempt to stabilize gypseous soil characteristics using alkali activation method. Types of soils having different gypsum contents are used to study the effect of the alkali activator on improving the collapsibility and compressive and shear strength characteristics for various percentages of gypsum in the soil.

1.4 Objectives of the Research

The objectives include:

1. Characterizing the chemical, microstructural composition, assessing the compressive strength and sulfate-attack resistance of fly ash geopolymer binder with different activator types and molarities;
2. Determining the effective dose of the geopolymer such as the amount of fly ash and the type and molarity of activator liquid that must be mixed with soils having different quantities of gypsum;
3. Evaluating the changes in the geotechnical characteristics of the alkali-activated fly-ash treatment of the gypseous soil and its effect on the collapsibility characteristics of the soil after treatment;
4. Investigating the influence of sulfate SO_4^{-2} attack and its underlying mechanism of influencing the collapsibility characteristics, unconfined compressive strength, and permeability of the treated soil
5. Investigating an empirical model to estimate the collapsible index based on the amount of fly ash, molarity of activator liquid and the quantities of gypsum .

1.5 Scope of the Research

The research will concentrate on investigating the phenomenon of geopolymerization method in gypseous soil stabilization, considering the effects of this method on the collapsibility, chemical, and shear properties of gypseous soil before and after the treatment along with the effect of sulfate.

In this study, the behaviours of the alkali activator, industrial waste powder, and the chemical components during the reaction process were investigated based on the type of gel binder and its chemical properties. Disturbed and remoulded gypseous soil samples were collected for laboratory investigations. Artificial gypseous soil was prepared by adding $CaSO_4$ to the soil to make three types of gypseous soils (slightly, moderately, and highly gypseous). In this research , the gel binder is prepare from the industrial waste and the chemical solutions (fly ash + NaOH or KOH) used to stabilize the gypseous soil in the presence of different amounts of gypsum. The reaction and the underlying mechanism of the binder gel are investigated under exposure to sulfate attack on the geopolymer paste through the compressive strength test, mass loss test, X-ray diffraction (XRD), thermogravimetric analysis (TGA), Brunauer-Emmett-Teller (BET) surface area, scanning electron microscopy (SEM), and Energy-dispersive X-ray spectroscopy (EDX) analyses before and after soaking in sulfate solution. The effect of the stabilization method on the geotechnical characteristics of gypseous soil was investigated. On the other hand, the effect of internal sulfate attack was studied by soaking the soil samples before and after stabilization in laboratory tests, namely collapsibility, permeability-leaching (Rowe cell hydraulic pressure), unconfined compressive strength (UCS), and triaxial tests with XRD, SEM, and EDX analyses of the gypseous soil.

1.6 Significance of the Research

The application of the chemical treatment method to the stabilization of gypseous soil is performed by using alkali activators with industrial waste powder (fly ash class F) instead of the traditional additives such as cement or lime to prevent the problem of sulfate attack and improve the soil compressive strength by adding alkali activators with industrial waste powder (fly ash class F), which will play an important role in the future.

Preliminary reports from case studies show the beneficial role of alkali activation-stabilization techniques. Indeed, this technology can be used in situ by adding the activator to the soil and mix it for different gypsum contents. Some researchers applied this method to some types of soils such as expansive, clayey, and soft soils, and they showed remarkable improvement when the method was used.

On the other hand, every method may have some side effects such as changes in chemical compounds because of the alkali reaction, but there has been no research about the method's disadvantages until now. Furthermore, this technique is still in progress and the number of studies in this field is limited, so the disadvantages or side effects are unknown so far; however, there are some researches on the use of these materials in concrete instead of cement.

In fact, to understand the whole process and to gain more information and knowledge, some steps have to be followed. Firstly, the laboratory tests have done to show how the stabilization with alkali activation can affect and improve the soil properties. Secondly, these tests need to be repeated several times to find the optimum percentages of powder and alkali activator that give an acceptable improvement. Finally, the simulated data or results that emerge from these tests should be analysed. Hence, that will help in recognizing the advantages or disadvantages of the work.

The method of stabilization by alkali activation may be a workable technique in Iraq compared with other chemical techniques since it does not need any special equipment for mixing; not only that, but it is also beneficial to use ground granulated blast furnace slag (GGBS), a type of industrial waste that is available in Iraq. Indeed, the geopolymerization technique still has some un-investigated parts that need further attention and attempts to find a full explanation.

1.7 Thesis Organization

The thesis is presented in five chapters. Chapter One illustrates the importance of this study, including the aim, scope, and methodology with general information regarding the improvement of gypseous soil. Chapter Two presents a literature review on gypseous soil, including the formation and characteristics, collapse behaviour, shear strength characteristics, and studies of the treatment of gypseous soil. The third chapter describes the strategy used to equivalent the designated objectives of the research for stabilizing gypseous soil. This chapter considers the materials used and classification tests as well as physical and chemical, collapse, shear, unconfined compression, soaking effect, permeability, compressive strength, and micro-structural tests. The fourth chapter reports the results of the testing programme together with the analysis and discussion of test results with drown curves. Chapter Five presents a brief summary of the research techniques and findings and then outlines simple specific conclusions of the study and recommendations for future studies.

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