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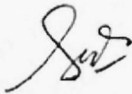
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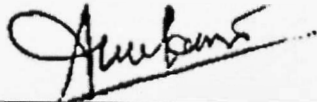
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IJAZAH: PhD (DOKTOR FALSAFAH)

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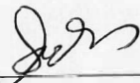
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HYDRAULIC CONDUCTIVITY AND HEAVY METAL RETENTION OF  
CONTAMINATED SOIL TREATED USING ENZYME INDUCED  
CALCITE PRECIPITATION METHOD

JODIN MAKINDA



UMMS

A thesis submitted in fulfilment of the  
requirements for the award of the degree of

Doctor of Philosophy

Faculty of Civil Engineering  
Universiti Teknologi Malaysia

MARCH 2023

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I declare that this thesis entitled "*Hydraulic Conductivity and Heavy Metal Retention of Contaminated Soil Treated Using Enzyme Induced Calcite Precipitation Method*" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

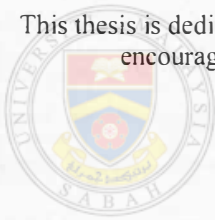
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## DEDICATION

This thesis is dedicated to my parents, for their love, support and encouragement along my PhD journey



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Contamination of heavy metals are known to affect the physical and mechanical properties of soil, particularly increasing the permeability due to the decrease of dielectric constant. This could increase the risk of transmission of heavy metal contaminants and threaten environmental sustainability. Hence, safer mitigation measures should be provided. Commonly, they are improved using conventional or alternative materials to meet the geotechnical requirement. However, most of these materials are costly, slow and long treatment cycles and environmentally unsustainable. Recently, soil improvement using biomediation or bio-inspired techniques have gained attention due to their cost-effectiveness, sustainability and environmental friendliness. Enzymatic induced calcium carbonate precipitation (EICP) is one such technique that utilizes free urease enzyme to produce calcium carbonate precipitation via urea hydrolysis process. This study investigated the properties of copper mine wastes from Mamut, Lohan and Bongkud, Sabah and used EICP to improve the engineering properties of heavy-metal contaminated soil. The study examined the effect of variation of cementation concentrations (0.5 M and 1.0 M), curing periods (1,3, and 7 days), degree of compactions (70 and 80 % of the maximum dry density (MDD)) and curing temperatures (5 °C, 15 °C and room) on the hydraulic conductivity, calcium carbonate content, as well as heavy metal retention. Results from the three locations indicate differences in grained particle sizes, acidity, specific gravity, natural moisture, organic content and heavy metal contaminant levels. While all three locations met the requirement of EICP biocementation, Lohan was selected due to the feasible coarse-fine grain composition and lowest MDD, in addition to its risky location and higher level of contamination. To mitigate the issue of soil heterogeneity and to produce reliable soil samples with same characteristics of heavy-metal contamination, a locally-sourced metal-spiked Stulang Laut was tested with Lohan. The effect of EICP in both Lohan and Stulang soils were found to be similar with higher reduction of hydraulic conductivity under higher concentration of cementation solution at 1.0 M and higher degree of compaction at 80%. The effects of curing duration can be seen as early as the first 24 hours. The precipitation rate then decreases with days, with the bigger drop observed on 1-day to 3-day curing for 0.5 M compared to 1.0 M. The highest permeability reduction is observed on sample cured under room temperature (25 °C) followed by 15 °C and 5 °C. Meanwhile, higher calcite carbonate content is observed at cementation concentration of 1.0 M. It was observed that the order of heavy metal-retention for EICP treated Lohan soil is  $Ni > Cu > Pb$  while for Stulang Laut, the order is  $Cu > Ni > Co$  with highest retention observed at room temperature. Regardless of the curing temperature, higher retention in both soils is observed at lower degree of compaction. The results from SEM images and X-ray diffraction were able to confirm the formation of calcite, their shape and distribution between the soil particles. In conclusion, optimum condition of 25 °C, 1.0 M EICP concentration, 80% MDD and 7-days curing and 25 °C, 1.0 M EICP concentration, 70% MDD and 3-days curing is the best treatment to reduce the hydraulic conductivity of heavy-metal contaminated soil and increase their retention-ability in immobilizing heavy metals, respectively.



Pencemaran logam berat diketahui menjejaskan sifat fizikal dan mekanikal tanah, terutamanya meningkatkan kebolehtelapan akibat penurunan pemalar dielektrik. Ini boleh meningkatkan risiko penghantaran bahan cemar logam berat dan mengancam kelestarian alam sekitar. Oleh itu, langkah mitigasi yang lebih selamat harus disediakan. Lazimnya, ia diperbaiki menggunakan bahan konvensional atau alternatif untuk memenuhi keperluan geoteknik. Walau bagaimanapun, kebanyakan bahan konvensional dan alternatif ini adalah kitaran rawatan yang mahal, perlahan dan panjang serta tidak mampan. Baru-baru ini, pembaikan tanah menggunakan teknik biomediasi atau bio-inspirasi telah mendapat perhatian kerana keberkesanan kos, kemampanan dan keramahannya terhadap alam sekitar. *Enzymatic induced calcium carbonate precipitation* (EICP) adalah satu teknik yang menggunakan enzim urease bebas untuk menghasilkan kerpasan kalsium karbonat melalui proses hidrolisis urea. Kajian ini menyiasat sifat sisa lombong tembaga dari Mamut, Lohan dan Bongkud di Sabah dan menggunakan EICP untuk menambah baik sifat kejuruteraan tanah tercemar logam berat. Kajian ini menguji kesan variasi kepekatan penyimenan (0.5 M dan 1.0 M), tempoh pengawetan (1,3, dan 7 hari), tahap pemadatan (70 dan 80 % daripada ketumpatan kering maksimum (MDD)) dan suhu pengawetan (5 °C, 15 °C dan suhu bilik) pada kekonduksian hidraulik, kandungan kalsium karbonat serta tahap mobiliti logam berat. Keputusan di ketiga-tiga lokasi menunjukkan perbezaan dari segi saiz butiran tanah, keasidan, graviti tentu, kandungan kelembapan, kandungan organik dan tahap pencemaran logam berat. Walaupun ketiga-tiganya memenuhi persyaratan, Lohan dipilih untuk rawatan biomediasi EICP memandangkan kesesuaian komposisi butiran tanah kasar dan halus serta ketumpatan kering maksimum yang terendah, tambahan pula tanah Lohan terletak di lokasi berisiko dan tahap pencemaran yang lebih tinggi. Untuk mengurangkan isu heterogeniti tanah dan untuk menghasilkan sampel tanah yang boleh dipercayai dengan ciri-ciri pencemaran logam berat yang sama, tanah Stulang Laut telah diuji dengan Lohan. Kesan rawatan EICP adalah sama ke atas Lohan dan Stulang Laut iaitu pengurangan kekonduksian hidraulik yang lebih tinggi di bawah kepekatan larutan penyimenan yang lebih tinggi pada 1.0 M dan tahap pemadatan yang lebih tinggi pada 80%. Kesan tempoh pengawetan boleh dilihat seawal 24 jam pertama. Kadar kerpasan kemudiannya berkurangan dengan hari, dengan penurunan yang lebih besar pada pengawetan 1 hingga 3 hari. Pengurangan kebolehtelapan tertinggi diperhatikan pada sampel yang diawet di bawah suhu bilik (25 °C). Sementara itu, kandungan kalsit karbonat yang lebih tinggi diperhatikan dalam kepekatan penyimenan 1.0 M. Telah diperhatikan bahawa susunan pengekalan logam berat untuk tanah Lohan yang dirawat EICP ialah Ni > Cu > Pb manakala bagi Stulang Laut, susunannya ialah Cu > Ni > Co. Tanpa mengira suhu pengawetan, pengekalan yang lebih tinggi diperhatikan dalam tahap pemadatan yang lebih rendah. Hasil daripada imej SEM dan pembelauan sinar-X dapat mengesahkan pembentukan kalsit, dan taburannya antara zarah tanah. Kesimpulannya, didapati bahawa biosimen EICP telah mengurangkan kekonduksian hidraulik tanah tercemar logam berat pada keadaan optimum 25 °C, 1.0 M EICP, 80 % MDD dan pengawetan 7 hari dan suhu 25 °C, 1.0 M EICP, 70 % MDD dan pengawetan 3 hari adalah rawatan EICP terbaik untuk mengurangkan kebolehtelapan tanah tercemar logam berat dan meningkatkan keupayaan pengekalannya dalam melumpuhkan logam berat, masing-masingnya.

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