EVALUATION OF FOUR SEMI-PERMEABLE MEMBRANES IN THE OSMOTIC TECHNIQUE FOR ESTABLISHING SOIL-WATER RETENTION CURVE (SWRC) OF MANSULI CLAY

NURUL SYAFIQAH BINTI MOHD AZMI

MASTER OF SCIENCE

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of degree of Master of Science.

_____

(Supervisor's Signature)

Full Name : DR. HAJI MOHD YUHYI BIN DR. HAJI MOHD TADZA

Position : SENIOR LECTURER

Date : 2/2/2018

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted to any other degree at Universiti Malaysia Pahang or any other institutions.

_____

(Student's Signature)

Full Name : NURUL SYAFIQAH BINTI MOHD AZMI

ID Number : MAC15003

Date : 2/2/2018

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LIST OF SYMBOLS

MPa	Mega Pascal
Rb	Rubidium
Cs	Caesium
Sr	Strontium
K^+	Potassium ion
Na^+	Sodium ion
Mg^{2+}	Magnesium ion
Fe^{2+}	Ferrous ion
Al^{+3}	Aluminum ion
O^{2-}	Oxygen ion
OH^-	Hydroxyl ion
Ca^{2+}	Calcium ion
Si^{4+}	Silicon ion
S_m	Matric suction
μ_a	Air pressure
μ_w	Water pressure
S_o	Osmotic suction
S_T	Total suction
kPa	Kilo Pascal
g	Gram
L	Liter
S	Suction
c	PEG concentration
$^\circ$C	Degree Celsius
n	Length of the chain
d_{PEG}	Diameter of PEG molecules
nm	Nanometer
%	Percentage
Da	Dalton
m/s	Meter per second
3-D	Three-Dimensional

Θ_s	Saturated water content
N	North
E	East
m	Meter
s	Second
hr	Hour
ml	Milliliter
mm	Millimeter
µm	Micrometer
m^2/g	Square meter per gram
m^2	Square meter
pH	Potential of Hydrogen
meq/100g	Milliequivalent per 100 grams
meq	Milliequivalent
Si	Silicon
Al	Aluminum
Fe	Iron
K	Potassium
Mg	Magnesium
Ti	Titanium
Na	Sodium
Ca	Calcium
Mn	Manganese
°	Degree
NaCl	Sodium chloride
Br	Brix index
J	Water flux
P_m	Permeability of semi-permeable membrane
mg	Milligram
PtCo	Platinum-Cobalt
$CaCl_2$	Calcium chloride
KNO_3	Potassium nitrate
KCl	Potassium chloride

K_2SO_4	Potassium sulphate
LiCl	Lithium chloride
K_2CO_3	Potassium carbonate
G_s	Specific gravity
w_i	Initial water content
w_h	Hygroscopic water content
LL	Liquid limit
PL	Plastic limit
SL	Shrinkage limit
SSA_T	Total specific surface area
Fe^{3+}	Ferric ion
Mn^{2+}	Manganese ion
<	Less than
w	Water content
S_r	Degree of saturation
e	Void ratio
P	Phosphorus
V	Vanadium
S	Sulfur
Ba	Barium
Zr	Zirconium
Zn	Zinc
Rb	Rubidium
Cr	Chromium
Ni	Nickel
Cu	Copper

LIST OF ABBREVIATIONS

AEV	Air Entry Value
AFM	Atomic Force Microscopy
ASTM	American Society for Testing and Materials
BET	Brunauer-Emmett-Teller
BS	British Standard
BSCS	British Soil Classification System
CA	Cellulose Acetate
CEC	Cation Exchange Capacity
CV	Clay with Very high plasticity
EGME	Ethylene Glycol Monoethyl Ether
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
LOI	Loss On Ignition
MW	Molecular Weight
MWCO	Molecular Weight Cut Off
NA	Nutrient Agar
NTU	Nephelometric Turbidity Units
PA	Polyamide
PDA	Potato Dextrose Agar
PEG	Polyethylene Glycol
PES	Polyethersulfone
Pf	Polysulfone
PIP	Piperazine
PPA	Polypiperazine-amide
SEM	Scanning Electron Microscopy
SSA	Specific Surface Area
SWRC	Soil-Water Retention Curve
TF	Thin Film
TMC	Trimesoylchloride
UMP	Universiti Malaysia Pahang
VET	Vapour Equilibrium Technique
WEV	Water Entry Value

XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

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ABSTRAK

Lempung mengembang banyak digunakan dalam pelbagai aplikasi geoteknikal dan geoalam sekitar di kebanyakan negara terutamanya sebagai bahan kambusan dan bahan hadangan di repositori pelupusan sisa radioaktif nuklear peringkat tinggi. Kefahaman terperinci mengenai sifat lempung mengembang tempatan adalah amat penting untuk memastikan prestasi jangka panjang kepada sistem repositori pelupusan sisa di Malaysia. Sedutan dan kandungan air adalah parameter paling penting yang mengawal sifat lempung mengembang. Perubahan dalam kandungan air yang disebabkan oleh perubahan dalam sedutan biasanya diramalkan dengan menubuhkan pengekalan tanah-air lengkung (SWRCs). Dalam makmal, teknik osmosis dan teknik keseimbangan wap telah biasa digunakan untuk mengawal sedutan dalam tanah dan untuk menubuhkan SWRCs. Beberapa penyelidik mendedahkan bahawa teknik osmosis telah digunakan secara meluas untuk mengenakan sedutan rendah kurang daripada 1 MPa tetapi kajian untuk sedutan tinggi adalah terbatas. Batasan teknik osmosis di sedutan tinggi dikaitkan dengan intrusi molekul polietilena glikol (PEG) ke dalam specimen lempung kerana kegagalan membran separa bolehterlap dalam menyekat peredaran molekul PEG ke dalam spesimen lempung dan menjejaskan penentuan SWRC yang tepat. Dalam kajian ini, ciri-ciri fizikal, kimia, mineralogi dan mikrobiologi lempung mengembang Mansuli tempatan telah ditentukan mengikut prosedur standard. Selain itu, pengeringan dan pembasahan SWRCs telah dibentuk dengan menggunakan teknik osmosis, keseimbangan wap dan cermin-penyaman takat-embun untuk meramalkan sifat lempung Mansuli pada sedutan gunaan daripada 0.06 sehingga 262.75 MPa. Pada sedutan gunaan tinggi, ujian osmosis telah dijalankan dengan menggunakan membran separa bolehterlap poliethersulfona (PES), filem nipis (TF) dan polipiperazina-amida (PPA) untuk menghalang intrusi molekul PEG ke dalam spesimen lempung. Prestasi membran separa bolehterlap sebelum dan selepas ujian osmosis telah dikaji menggunakan ujian fluks air, kebolehterlapan dan mikroskopi tenaga atom (AFM). Berdasarkan hasil kajian, lempung Mansuli dikelaskan sebagai lempung dengan keplastikan sangat tinggi (CV) kerana kawasan permukaan spesifik yang tinggi dan kapasiti pertukaran kation yang tinggi. Lempung Mansuli mengandungi vermikulit sebagai mineral utama (36.8%) dan lempung tempatan ini adalah jenis vermikulit yang kaya dengan magnesium dengan purata berpemberat valensi sebanyak dua. Secara keseluruhannya, lima spesies mikrob telah ditemui dalam lempung Mansuli. Intrusi PEG berlaku pada sedutan gunaan tinggi (iaitu di antara sedutan 3 dan 10 MPa) dalam ujian osmosis yang menggunakan membran selulosa asetat (CA) dan kandungan air yang diperolehi daripada ujian osmosis didapati lebih rendah daripada kandungan air yang diperolehi daripada ujian keseimbangan wap. Dua daripada kulat yang dikenalpasti iaitu *Penicillium funiculosum* dan *Hypocrea aureoviridis* dipercayai bertanggungjawab terhadap penguraian membran CA dalam ujian osmosis. Pada akhir kajian, didapati bahawa membran PES mampu untuk mengurangkan kesan intrusi PEG ke dalam spesimen lempung pada sedutan tinggi dan kandungan air yang diperolehi daripada ujian osmosis didapati serasi dengan kandungan air yang diperolehi daripada ujian keseimbangan wap. Pengeringan dan pembasahan SWRCs yang lancar dan tepat bagi lempung Mansuli telah dibentuk dengan menggunakan membrane PES. Oleh itu, kajian ini mengatasi batasan teknik osmosis di sedutan tinggi dan ramalan tepat mengenai sifat kejuruteraan lempung tempatan dapat disediakan. Kajian ini juga memberikan maklumat mengenai kemungkinan pengunaan lempung Mansuli sebagai bahan bendungan untuk pembangunan repositori sisa nuklear di Malaysia.

ABSTRACT

Expansive clays are extensively used for various geotechnical and geoenvironmental applications in many countries especially as the backfilling and barrier materials in high-level radioactive nuclear waste disposal repositories. Detailed understanding of the behaviour of local expansive clay is extremely crucial to ensure the long term performance of the waste disposal repositories system in Malaysia. Suction and water content are the most important parameters that control the behaviour of expansive clay. Changes in water content due to changes in suction are commonly predicted by establishing the soil-water retention curves (SWRCs). In the laboratory, osmotic technique and vapour equilibrium technique were commonly used for controlling suction in the soil and to establish SWRCs. Several researchers revealed that osmotic technique has been widely used for applying lower suction less than 1 MPa but studies for higher suction are limited. The limitation of osmotic technique at higher suction is associated with the intrusion of polyethylene glycol (PEG) molecules into clay specimen due to failure of the semi-permeable membrane in restricting the passage of PEG molecules into clay specimen and affecting the precise determination of SWRCs. In this study, physical, chemical, mineralogical and microbiological properties of local expansive Mansuli clay were determined following the standard procedures. Besides that, drying and wetting SWRCs were established using osmotic, vapour equilibrium and chilled-mirror dew-point techniques to predict the behaviour of Mansuli clay at applied suction of 0.06 to 262.75 MPa. At higher applied suction, the osmotic tests were carried out using polyethersulfone (PES), thin film (TF) and polypiperazine-amide (PPA) semi-permeable membranes in order to prevent the intrusion of PEG molecules into clay specimen. The performance of semi-permeable membranes before and after osmotic tests was investigated using water flux test, permeability test and Atomic Force Microscopy (AFM). Based on the findings, Mansuli clay is classified as clay with very high plasticity (CV) due to high specific surface area and cation exchange capacity. Mansuli clay contains vermiculite as the main mineral (36.8%) and this local clay is magnesium-rich type vermiculite with weighted average valency of about two. In total, five species of microbes were found within Mansuli clay. The PEG intrusion occurred at higher applied suction (i.e. suctions between 3 and 10 MPa) in osmotic test using cellulose acetate (CA) membrane and the water contents obtained from osmotic test were found to be lower than water contents obtained from vapour equilibrium test. Two of the fungus identified namely *Penicillium funiculosum* and *Hypocrea aureoviridis* were believed to be accountable for degradation of CA membrane in osmotic test. At the end of the research, it was found that PES membrane was able to minimise the effect of PEG intrusion into clay specimen at higher suction and the water contents obtained from osmotic test were found to be in good agreement with that obtained from vapour equilibrium test. Smooth and precise drying and wetting SWRCs of Mansuli clay were established using PES membrane. Thus, this study overcomes the limitation of osmotic technique at higher suction and a better prediction of engineering behaviour of local clay can be provided. This study also provides information for possible use of Mansuli clay as barrier material for the development of Malaysia nuclear waste repositories.

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