EFFECT OF SUPERABSORBENT POLYMER COMPOSITE ON THE COMPRESSIBILITY BEHAVIOUR OF LOW PLASTIC CLAYS

NOOR BALQISH BINTI MOHD DIN

B. ENG(HONS.) CIVIL ENGINEERING

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

DECLARATION OF THESIS AND COPYRIGHT			
Author's Full Name	: NOOR BALQISH BINTI MOHD DIN		
Date of Birth	: <u>17th NOVEMBER 1994</u>		
Title	: EFFECT OF SUPERABSORBENT POLYMER COMPOSITE ON THE COMPRESSIBILITY BEHAVIOUR OF LOW PLASTIC CLAYS		
Academic Session	: 2016/2017		
I declare that this thesis	is classified as:		
CONFIDENTIA	L (Contains confidential information under the Official		
□ RESTRICTED	(Contains restricted information as specified by the		
☑ OPEN ACCESS	I agree that my thesis to be published as online open access (Full Text)		
I acknowledge that Univ	versiti Malaysia Pahang reserves the following rights:		
 The Thesis is the Property of Universiti Malaysia Pahang The Library of Universiti Malaysia Pahang has the right to make copies of the thesis for the purpose of research only. The Library has the right to make copies of the thesis for academic exchange. 			
Certified by:			
(Student's Signa	ture) (Supervisor's Signature)		
<u>941117-06-5778</u>	DR. HJ MOHD YUHYI BIN HJ MOHD TADZA		
New IC/Passport N Date: 19 June 2017	umber Name of Supervisor Date:19 June 2017		

NOTE : * If the thesis is CONFIDENTIAL or RESTRICTED, please attach a thesis declaration letter.



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 the Bachelor Degree of Civil Engineering

(Supervisor's Signature)			
Full Name	:		
Position	:		
Date	:		



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 for any other degree at Universiti Malaysia Pahang or any other institutions.

(Student's Signature) Full Name : NOOR BALQISH BINTI MOHD DIN ID Number : AA13138 Date : 19 June 2017

EFFECT OF SUPER ABSORBENT POLYMER COMPOSITE ON COMPRESSIBILITY BEHAVIOUR OF LOW PLASTIC CLAYS

NOOR BALQISH BINTI MOHD DIN

Thesis submitted in fulfillment of the requirements for the award of the Bachelor Degree in Civil Engineering

Faculty of Civil Engineering and Earth Resources UNIVERSITI MALAYSIA PAHANG

JUNE 2017

ACKNOWLEDGEMENTS

I would like to express my gratitude to all those who gave me the possibility to complete this thesis. I am deeply indebted to my supervisor, Dr Hj Mohd Yuhyi Hj Mohd Tadza from Faculty of Civil Engineering & Earth Resources, Universiti Malaysia Pahang, who believing, stimulating suggestions and encouragement helped me in all the time of research for this project and writing of this thesis.

I am obliged to my parents; Mohd Din Mat Nor and Normawati Mat Hassan, my siblings; Tasha, Maisarah and Thurshina and also my brother in law, Aidi and my nephew, Umar and my grandmother and grandfather; Meriam and Maarof and my true supporter, Fikri. Special thanks for all of you made doa for smoothness of the research and writing.

This work would not have been possible without the support and encouragement of my coursemates and roomates .I would like to thank my close friends, Fatin and Haziqah , for always give supports when I need them.

I also want to thank all the staffs from Environmental Laboratory, Faculty of Civil Engineering & Earth Resources, Universiti Malaysia Pahang, for all their assistance and support on the laboratory experiments.

ABSTRAK

Kaolin diketahui mempunyai kadar plastik yang lebih rendah daripada mana-mana tanah liat yang lain, dan ia biasa ditemui di kawasan di Malaysia. Dalam kajian ini, penggunaan kaolin adalah sesuai kerana ia adalah salah satu daripada tanah liat yang biasa terdapat di Malaysia. Tidak seperti bentonit, kaolin mempunyai pengekalan rendah air menjadikannya tidak begitu komersial dalam pelbagai bidang industri, tetapi ia boleh penyelesaian dengan penambahan SAP, bahan dengan rangkaian hydrophilic yang boleh menyerap dan mengekalkan sejumlah besar air atau larutan akueus. Sifat-sifat fizikal dan kimia dengan atau tanpa SAP telah dikaji dan kandungan SWCC terbentuk. Teknik yang digunakan untuk mendapatkan data yang menggunakan "chilled-mirror dew-point technique". Ujian oedometer juga dijalankan untuk mengetahui lengkung tekanan nisbah ruang. Ia menunjukkan bahawa terdapat potensi dalam meningkatkan keupayaan mengekalkan keupayaan air. Kesimpulannya, SAP dapat membantu kaolin meningkatkan kadar air penyerapan, mampatan tanah dan meningkatkan kadar keplastikan tanah.

ABSTRACT

Kaolinite is known to have lower plasticity than any other clay, and it is commonly found in the area of Malaysia. In this study, the usage of kaolin is appropriate since it is one of the commonly found clay in Malaysia. Unlike bentonite, kaolin has low retention of water makes it less commercial in the various fields of the industry, but it can be solves with addition of Super Absorbent Polymer (SAP); a materials with hydrophilic networks that can absorb and retain huge amounts of water or aqueous solution. The physical and chemical properties with or without SAP were studied and suction-water content Soil-Water Characteristic Curve (SWCC) is formed. The techniques used for obtaining the data is using chilled-mirror dew points techniques. Oedometer test also carried out to know the pressure void ratio curve. It shows that there is a potential in increasing the ability of retaining water ability. In conclusion, SAP able to help kaolinite boost their water rate of absorption then enhance the plasticity of the soils.

TABLE OF CONTENT

DEC	CLARATION	
TIT	LE PAGE	
ACH	KNOWLEDGEMENTS	ii
ABS	STRACT	iiiii
ABS	STRAK	iv
TAB	BLE OF CONTENT	v
LIST	T OF TABLES	ix
LIST	T OF FIGURES	X
LIST	T OF ABBREVIATIONS	xi
CHA	APTER 1 INTRODUCTION	1
1.1	Background study	3
1.2	Problem statement	4
1.3	Research objectives	4
1.4	Scope of study	4
1.5	Chapter summary	4
CHA	APTER 2 LITERATURE REVIEW	6
2.1	Introduction	6
2.2	Application of clays	6
2.3	Diffuse double layer of clay	7
2.4	Oedometer test	9
2.5	Superabsorbent polymer (SAPs)	9

	2.5.1	Characteristics of SAP	10
	2.5.2	Application of SAP	11
CHAF	PTER 3	METHODOLOGY	12
3.1	Introd	uction	12
3.2	Selecti	on of materials	12
3.3	Sample	e preparation	12
3.4	Physics	al properties of FMC and S300	13
	3.4.1	Specific gravity	14
	3.4.2	Liquid Limit	14
	3.4.3	Plastic Limit	15
	3.4.4	Shrinkage Limit	15
	3.4.5	Free swell	15
	3.4.6	Organic content	16
	3.4.7	Cation exchange capacity	16
	3.4.8	Surface area	17
	3.4.9	Mineral composition	17
3.5	Soil-wa	ater retention curve	17
	3.5.1	Osmotic technique	17
	3.5.2	Chilled-mirror dew-point technique	17

3.6	Shear	strength	18
	3.6.1	Vane shear	19
	3.6.2	Shear strength	19
CHA	PTER 4	RESULTS AND DISCUSSION	20
4.1	Introd	uction	20
	4.1.1	Specific gravity	20
	4.1.2	Liquid limit	20
	4.1.3	Plastic limit	21
	4.1.4	Initial water content	21
	4.1.5	Water content	21
	4.1.6	Loss on ignition	21
	4.1.7	Free swell	22
	4.1.8	Soil physical characteristics	22
4.2	Oedon	neter test	23
	4.2.1	Water content-Pressure curve	23
4.3	Water	content pressure curve	25
CHA	PTER 5	5 CONCLUSION	29
5.1	Introd	uction	29
5.2	Recom	amendations	29

REFERENCES

APPENDICES

33

30

LIST OF TABLES

Table 3.1	Standard used for physical properties	13
Table 4.1	General table data for soil physical characteristics	22
Table 4.2	Oedometer result for FMC	23
Table 4.3	Oedometer result for FMC+SAP	23
Table 4.4	Oedometer result for S300	24
Table 4.5	Oedometer result for S300+SAP	24

LIST OF FIGURES

Figure 2.1	Diffuse double layer theory	8
Figure 2.2	Stern model	8
Figure 2.3	Swelling mechanisms of superabsorbent polymer	10
Figure 3.1	WP4C Potential dew meter for soil suction reading	18
Figure 4.1	The water content-pressure curve for FMC with and without SAP	25
Figure 4.2	The water content-pressure curve for S300 with and without SAP	26
Figure 4.3	Void ratio-pressure curve of FMC with and without SAP	27
Figure 4.4	Void ratio-pressure curve of S300 with and without SAP	28

LIST OF ABBREVIATIONS

SAPs	Superabsorbent polymer
SWCC	Soil-water retention curve
Mg	Magnesium
Al	Aluminium
DDL	Diffuse double layer
Ca	Calcium
Na	Sodium
К	Pottasium
СООН	Carboxylic acid groups
H ₂ O	Water

CHAPTER 1

INTRODUCTION

1.1 Background study

The global industrial issues is an imperative issue that require assertive attention from authorities. The industrial waste had already classified by Karl et al., (2009) become greater than over the last century. Bentonite commonly preferred used as engineered clay barriers for waste disposal in industries of oil and gas (Yang and Barbour, 1993).Rock formed of highly colloidal and plastic clays, and mainly its composition is montmorillonite which is a clay mineral of the smectite group. It is produced by in situ devitrification of volcanic ash (Adamis and William, 2005). The industries and factories dumping the waste without any safety control and caused the environment polluted. The rapid development in technology has been lead to the industrialization and increasing in life demands. The industries are crucial to the progress of civilization. To accomplish the high worldwide demand for goods, there is almost no alternative to industrialization. Thus, waste material by human activities quite large in number and need the big area of landfill for disposal. The layer is landfill usually are layered by certain layer of clay. The collection system of leachate must be effective and have to be inspected and that is why landfill construction liner crucial to minimize the effect of environmental (Bouazza et al., 2002). Highly plastic clays exist in various form, such as slurry clays or loose powder which compacted at low dry densities have been used for construction of liners of landfill (Bouazza and Bowders, 2010). Bentonite clay is well suit with the buffer barrier requirements or containment of landfills and all the facilities which need lining system because of its swelling capacity (Pusch and Yong, 2005). It has the high swelling capacity and able to form aqueous stable suspensions (Luckam, 1999). The bentonite are considered to surround the final disposal as an active component engineered barriers of radioactive wastes from nuclear reactor. Due to its low permeability and ion diffusivity and its ability to self-healing through swelling (Pusch, 1982).

The best known member of the smectite group is montmorillonite. The structure of montmorillonite is classified as dioctuhedrul which having two thirds of the octahedral sites which is occupied by trivalent cations. The structural charge of dioctahedral montmorillonite originating from the substitution of Mg^{2+} for Al^{3+} in the octahedral sheet. The mineral that exist in clay are silicate, oxide, feldspar, hydroxide, carbonate, liquid aluminium and organic matter. When exposed to wet condition, the clay particles is in pieces will form greasy and sticky properties while it will hard in dry condition. Clay have three types that are montmorillonite, illite and kaolinite. Kaolin or known also as china clay is a mixture of varies clay which is mainly composed of kaolinite and also contains quartz, mica, feldspar, illite and montmorillonite. Kaolinite is made up of tiny sheets of triclinic crystals with pseudohexagona morphology. It also has some cation exchange capacity and formed by rock weathering (Adamis and William, 2005). For illite, besides it is a component of ball clays, it also one of main components of common clay with chlorite. Illite has less substitution of aluminium for silicon even though it closely resembles micas and/or partial replacement of potassium ions between the unit layers by other cations, such as hydrogen, magnesium, and calcium (Adamis and William, 2005). Even so, the montmorillonite site is hard to obtain because it is located in heritage land and strictly prohibited to explore of. Thus, the high cost will be involved if montromorillonite need to be imported from foreign country. The uses of bentonite only limited to agriculture industry since its ability to increase the capacity of soil to store water fertilizer. Mixture of bentonite and chemical fertilizer also increase the fertilizer use efficiency and meanwhile lower the fixations of soil for potassium and phosphorus (Li et al., 2010). In this study, kaolinite is used since its abundance in soil (Schroeder and Erickson, 2014). The kaolinite has the strong hydrogen bonding to retain water (Luckham., 1999). According to Koch (2002), the kaolinite show the poor absorption capacity. For layers with low permeability, kaolinite which is the member of clay minerals can be used as a less sensitive components.

The major impact from human activities could lead to the degradation of organic soil matter. From previous research, super absorbent polymers (SAPs) can solve the issues by applied SAPs to the soil in order to bind the water and cations (Huettermann et al., 2009). SAPs acts as granules to hold soil moisture in arid areas. According to Zohurian-Mehr MJ et al. (2008), SAPs have the tendency to absorb and retain large amount of water or aqueous solution. SAPs also can promote in internal curing of cement

in order to increase the hydration of the material and self-desiccation and able to control self-desiccation shrinkage thus decrease the risk of early-age autogenous shrinkage cracking (Wyrzykowski et al., 2013; Lura, 2003). SAPs, special hydrogels are extensively commercialized in hygienic uses particularly disposable diapers and female napkin where they can capture secreted fluid, such as blood, urine, etc (Zohurian-Mehr MJ et al., 2008). Agricultural grade of such hydrogels are used as granules for holding soil moisture in arid areas.

In this study, FMC and S300 are selected which have different properties and background mineralogy. Both of FMC and S300 kaolinite will be mixed with superabsorbent polymer (SAP) to increase its water absorption capacity. The oedometer test is carried out to obtain the void ratio-pressure/suction curve and water content-pressure/suction curve. The various loads will be applied to a soil sample and the deformation response will be measured. Deformation of the soil in the field can be predicted to a change in effective stress. The soil water characterictic curve (SWCC) is usually used to estimate unsaturated soil property functions such as permeability functions, water storage functions, shear strength functions, and thermal property functions (Fredlund et al., 2011). SWCC of FMC and S300 will be determined in this study. Water content and void ratio of each soils at each applied suctions are required after establishing the suction-degree of saturation SWCCs (Mohd Tadza, 2011).

1.2 Problem statement

In landfill, there are certain specification before the waste disposal. The requirement should be complied with high specific surface by using minerals also capacity of high cation exchange which is optimized clay minerals such as Na-montmorillonite. S300 and FMC have low plasticity characteristics as compared to bentonite. With addition of SAP, the plasticity characteristics of FMC and S300 increased. The kaolinite-enriched layers and bentonite-enriched layers are built up which have a low permeability, good absorption properties and also low sensitivity against chemical attack. Furthermore, the kaolinite-SAP not yet developed to enhance the quality of kaolinite to show the properties of montmorillonite.

1.3 Research objectives

The aim of this research is as follows, i) to identify the effect of SAPs on plasticity of FMC and S300 kaolin, ii) to identify the effects of SAPs on compressibility behaviour of FMC and S300 kaolin and iii) to identify the effects of SAPs on water retention of FMC and S300 kaolin.

1.4 Scope of study

In this study, FMC and S300 from clay factory was considered. It is taken from the seal bag, then place in completely seal plastics to ensure there are no extra air affect the soil. Test performed in the laboratory to determine the physical properties of FMC and S300, using standards that allows the test is carried out smoothly. The soil water characteristic curve is established by using a chilled mirror dew point method. This method is to determine the difference in SWCC and plasticity characteristic of both soils with or without addition of Superabsorbent Polymer (SAP).All tests are carried out in ambient laboratory works.

1.5 Chapter summary

CHAPTER 2 presents literature review in detailed considered to the studies undertaken. The chapter briefing on introduction of clays, clays itself, diffuse double layer of clay, application of clays, oedometer test, superabsorbent polymer followed by characteristics and application of SAP.

CHAPTER 3 presents and discussed the introduction of methodology that will be adopted, selection of materials used, sample preparation, physical properties and soil-water retention curve.

CHAPTER 4 outlines the adopted experimental methodologies and apparatus used in the study, the result of oedometer test conducted, the water content-pressure curve and the void ratio-pressure curve.

CHAPTER 5 presents the conclusion and recommendation in this study for the next to explore the usage of SAP in the study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviews more information about history background, characteristics and applications of the related material which involved in this research project. Further view explained the essential of pressure-voids ratio and their significance.

2.2 Applications of clay

Clay has variety of uses in the construction industry nowadays. The mineral that exist in clay are silicate, oxide, feldspar, hydroxide, carbonate, liquid aluminium and organic matter. When exposed to wet condition, the clay particles is in pieces will form greasy and sticky properties while it will hard in dry condition. Thus, the properties of itself will absorb the water more. In landfill, there are certain specification before the waste disposal. The requirement should be complied with high specific surface by using minerals also capacity of high cation exchange which is optimized clay minerals such as Na-montmorillonite (Koch, 2001). One of the important application of clays in the industry is in development of landfill for disposal of waste. Barrier system is needed for separation of waste and leachate from leaking to groundwater system (Rowe, 2005). Besides, bentonites which has thermal stability is particular interest for containment barriers in facilities of nuclear waste storage (Meunier et al., 1998). In addition, clay is used in construction such as tile, rick, sewer pipe, cement, fibreglass etc. and manufacturing such as kiln furniture, dinnerware and refractories, clays are used in the calcined form as fillers, carriers, extenders and grogs (Burst, 1991). Treatment of liquid wastes in wetlands, ponds and impoundments and also isolation walls for storage are others application of clays in slurry form (Rowe et al., 1997; Reeves et al., 2007). It has least permeable of rocks and low porosity and yet it is ideal for containment for aquifers and hydrocarbon reservoirs (Reeves, 2006). Bentonite can provide confinement for hazardous wastes with its unique properties to prevent surrounding environment is contaminated (Mohd Tadza, 2011). Usually, clays with particle sizes from 2-5µm are best suitable fillers which is very fine-grained kaolinite is used for textiles and papers (Obaje et al., 2013). Kaolinites have been used for centuries in pharmaceutical preparations of intestinal adsorbent drugs and other therapeutically useful applications. In the preparation of pastes, ointments and body lotions, the clays such as montmorillonite, kaolinite and attapulgite are used. The best suitable for the manufacture of white Portland cement is kaolinite. (Bogue et al., 1995). Bentonites were used for lining of landfills, to minimise the hydraulic conductivity of mineral liners and geo synthetic clay liners were offered as a thin and flexible lining membrane (Koch, 2002).

2.3 Diffuse double layer of clay

In dry clay, negative charge are balanced by exchangeable cations like Ca, Mg, Na, and K surrounding the particles that being held by electrostatic attraction. As water added to the clay, the cations and anions float around the clay particles. This can be explained by using diffuse double layer theory (DDL). Moreover, because of the very small distances between the negatively charged edge surfaces, a significant fraction of the external water will be strongly influenced by the diffuse double layer (Wersin, 2004).

Due to its isomorphous substitution, clay particles are negatively charged, incomplete positions are available for metal ions and release of protons from the hydroxides. As the result of negatives charges at the surface of clay particles, electrostatic forces exists between negative charges and exchangeable cations. The negatively charges attracts a positive ion, and the region that attracted positive ion and negative charge surface of clay which makes it known as diffuse double layer. This occur at interface of clay surface and the soil solution. This layer actually made up from permanent negative charge available in the clay surface and the cations in the soil that balance the negative charge. The nature and natural properties are highly dependent on type of mineral and the chemistry of pore water.



Figure 2.1: Diffuse double layer theory

Figure 2.1 shows the diffuse double layer theory. The clay hold the innermost layer of double layer water strongly known as absorbed water. The orientation of this water gives clay soils their plastic properties and it has greater viscosity. Initially, attraction from the negative colloid causes some of the positive ions to form a firmly attached layer around the surface of the colloid; this layer of counter-ions is known as the Stern layer.



Figure 2.2: Stern model

The figure 2.2 shows that attached counter-ions in the Stern layer and what we refer as the double layer is the charged atmosphere in the diffuse layer.

The type and concentration of ions in solution will determine the thickness of this layer. As a net positive charge of ions extends away from the surface, the diffuse layer is called 'diffuse'. The net positive charge of the solution will be less as it further from the surface. Distance from the surface of the particles makes the force of attraction between water and clay decreases. All of the water held to clay particles by force of attraction is known as double layer water. For one cation to leave the double layer it must be substituted by another from the soil solution. The amount of adsorbed water in the double layer relative to the total volume of pores varies greatly with mineral type. (Van Olphen et al., 1963).

2.4 Oedometer test

Oedometer test is commonly used to determine the one-dimensional compressibility behaviour of soils either using saturated soil specimen (either undisturbed or remoulded). Several types of laboratory tests (i.e., falling head permeability test, Free Swell oedometer test, pressure plate test, shrinkage test and constant suction consolidation test) were performed to identify the appropriate soil properties and variables which control the swelling behavior of an unsaturated soil by carried out oedometer test (Shuai, 1996).

2.5 Superabsorbent polymer (SAPs)

There are materials that have the ability to absorb and retain large volumes of water and aqueous solution known as Superabsorbent polymers (SAPs)(Zohuriaan-Mehr MJ et al., 2008). They are currently used in many areas including hygienic and bio-related uses (particularly in disposable diapers), agricultural uses (e.g., water reserving in soil, soil conditioning, and controlled release of agrochemicals), pharmaceutical dosage forms, separation technology, fibres/textiles, water-swelling rubbers, soft

actuators/valves, electrical, construction, packaging, artificial snow, sludge/coal dewatering, fire-extinguishing gels, etc. (Kabiri et al., 2011).

2.5.1 Characteristics of SAPs

Superabsorbent polymer (SAP) materials are hydrophilic networks that can absorb and retain huge amounts of water or aqueous solutions. Many of the existing superabsorbents are formed from unsaturated carboxylic acid monomers including acrylic acid, methacrylic acid, alkylacrylates, and acrylamides which are rendered water insoluble by crosslinking (Smith et al., 1995). They can uptake water as high as 100,000%. Common SAPs are generally white sugar-like hygroscopic materials, which are mainly used in disposable diapers and other applications including agriculture (Zohuriaan-Mehr MJ et al., 2008). It is crucial to understand the reasons why SAPs can swell before discussing further the synthesis of superabsorbent polymers. There are several mechanisms to the process of swelling, all of which contribute to the final swelling capacity (or centrifuge retention capacity CRC – which is the amount of 0.9 wt% saline solution that a SAP can retain under free swelling conditions when surface water has been removed in a centrifuge).



Figure 2.3: Swelling mechanisms of Superabsorbent polymer

Figure 2.3 shows a diagrammatic representation of part of the polymer network. The polymer backbone in SAP is hydrophilic i.e. 'water loving' because it contains water loving carboxylic acid groups (–COOH). When water is added to SAP there is a polymer/solvent interaction; hydration1 and the formation of hydrogen bonds are two of these interactions. The effects of the H2O which contains hydrogen and oxygen atom decrease the energy and increase the entropy of the system. Due to the hydrophilic nature of SAP the polymer chains have a tendency to disperse in a given amount of water (i.e. they are trying to dissolve in the water), which leads to a higher number of configurations for the system and also increases entropy.

2.5.2 Application of SAPs

SAP caused a huge revolution in the personal health care industries in just over years. In late 1990, the world production of the SAP resins was more than one million tons (Zohuriaan-Mehr MJ et al., 2008). SAP amendment to soils reduces the evapotranspiration rate of the plants. They induce a significantly higher growth rate in plants growing on SAP amended soil. They bind heavy metals and mitigate their action on plants. They mitigate the effects of salinity. The benefits of SAP amendment to soils substantially outweigh their costs (Huttermann et al., 2009). The water retention of sand and soil was enhanced by using the SAPs (Chen et al., 2004). Utilization of SAPs as micro-reservoirs for chemical substances that would been released under specific conditions such as temperature, change of the chemical composition of pore solution, passage of time etc. (Mechtcherine et al., 2014). It have been used widely in sanitary goods, hygienic goods, wiping cloths, water retaining agents, dehydrating agents, sludge coagulants, disposable litter mats for pets, condensation preventing agents, and release control agents for various chemicals (Pascente, 1998). The presence of a polymer of SAPs in the soil diminished the kinetic drying of the soil, which will have as an application a reduction in water loss in the process of irrigation (Bakass et al., 2002).

CHAPTER 3

METHODOLOGY

3.1 Introduction

The characteristics of FMC Kaolin and S300 can be determined by experimental testing. The properties that are investigated that are physical and chemical. The different tests such as moisture content, particle size distribution, Atterberg limit, free swell test and Loss on Ignition test. Cation- Exchange Capacity is used to obtain the chemical properties. Other than that, void ratio pressure graph of FMC-SAP and S300-SAP can be acquired with several techniques including oedometer test.

3.2 Selection of materials

The soil samples of FMC and S300 are available in factory of clays and be kept in Soil and Geotechnical Laboratory of Universiti Malaysia Pahang. The sample is securely kept in seal plastic bags to prevent any air or water go through that may affects the soil moisture content.

3.3 Sample preparation

Since the appearance of the soil is in white powder alike, the sample does not need much preparation, which can be classified as a really fine particles. The sample is not sieve before the experimental tests were carried out. There are only certain test that require the condition of oven dry for 24 hours to calculate the moisture content of each sample. The sample then mixed with water and total of water and sample be measured as 250g. The sample is prepared in slurry form to increase its workability to be used. For sample without addition of SAPs, the sample just mixture of water and sample. For sample with addition of SAPs, the SAPs is added in certain amount. In this study, the percentage of SAPs used is five percent from total mass of mixture. The sample of FMC and S300 is calculated by using 1.2LL considering water content of FMC, FMC+SAP, S300 and S300+SAP. The sample are kept only a week in order to avoid the sample loses its hydration and after a week, new sample need to be prepared.

3.4 Physical properties of FMC and S300

The physical properties of soil signifies the soil texture, colour, horizontation and consistency. It is tested before able to proceed to the soil suction measurement. Laboratory testing included are specific gravity test, Atterberg Limit test, Shrinkage Limit Test, Free Swell test, Loss on Ignition test, and specific surface. All tests followed standards shown in Table 3.1.

Physical properties	Testing Method
Specific Gravity, Gs	Density Bottle (Small pycnometer) method (BS 1377: Part
	2: 1990: 8.3)
Liquid Limit. LL	BS 1377: Part 2: 1990:4.3
Plastic Limit, PL	BS 1377: Part 2: 1990: 5.3
Shrinkage Limit, SL	Standard Test Method for Shrinkage Factors of Soils by
	the Wax Method(ASTM D4943 – 08)
Water Content, w	Oven drying at 105 °C (BS1377: Part 2:1990)
Swell Index, Cs	Free swell test (Holtz and Gibbs, 1956)
Loss on Ignition	BS 1377: Part 3: 1990: 4.3
Chemical Properties	Testing Method
Cation Exchange	Ammonium acetate method
Capacity	

 Table 3.1: Standard used for physical properties

Table 3.1 outlines the standard used for physical properties of FMC and S300 kaolinite. For physical properties, the test carried out are specific gravity, Gs by using Density Bottle (Small pycnometer) method (BS 1377: Part 2: 1990: 8.3), followed by liquid limit, LL, plastic limit, PL and shrinkage limit, SL with the method of BS 1377: Part 2: 1990:4.3 BS 1377: Part 2: 1990: 5.3 and Standard Test Method for Shrinkage Factors of Soils by the Wax Method (ASTM D4943 – 08) respectively. For water content, w, the sample need to oven drying at 105 °C (BS1377: Part 2:1990). Swell Index, Cs, the test is free swell test (Holtz and Gibbs, 1956) and Loss on Ignition with BS 1377: Part 3: 1990: 4.3. To test for chemical properties which is cation exchange capacity, testing method used is ammonium acetate method.

3.4.1 Specific gravity

This test followed BS 1377-2 (2012). About 10 g oven dried soil samples were transferred into density bottle. The distilled water was added about half to three-fourth of the density bottle and placed in the vacuum desiccator. The soil samples were left in the desiccator for at least one hour until no further loss of air was apparent. The distilled water was added until the density bottle full and left for an hour in room temperature. Then, the soil and water were removed from the bottle. The density bottle was refilled with water until full and were left for an hour. The test repeated twice for the same soil sample. The specific gravity can be calculated using Eq. 3.1.

$$Gs = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 - W_2)}$$
(3.1)

where W_1 = weight of bottle + Stopper W_2 = weight of bottle + Stopper + Dry soil W_3 = weight of bottle + Stopper + Soil + Water W_4 = weight of bottle + Stopper + Water

3.4.2 Liquid limit

Liquid limit test were conducted using cone penetrometer follows BS 1377-2 (2012). About 250 g oven dried soil passing 425 μ m were left air dried for at least 30 minutes. Distilled water was added to the soil sample to form paste and then transferred to the cylindrical cup of cone penetrometer apparatus, ensuring that no air is trapped in the soil sample. The penetrometer was adjusted that the cone point touches the surface of the soil paste. The vertical clamp was released to penetrate into soil paste under its own weight for five seconds. The test was repeated for three times of values of penetration in the range of 13.5 to 27.5 mm. The graph of water content versus cone penetration was plotted. The moisture content corresponding to cone penetration of 20 mm was taken as liquid limit of the soil.

3.4.3 Plastic limit

Plastic limit test referred BS 1377-2 (2012). Soil paste was rolled out a thread on a flat surface. The plastic limit is defined as the moisture content where the soil paste begin to break apart at diameter 3.2 mm.

3.4.4 Shrinkage limit

Shrinkage limit test was referred to Standard Test Method for Shrinkage Factors of Soils by the Wax Method (ASTM D4943:2008). Soil sample was added with distilled water until it reaches 1.2 times liquid limit of the soil sample. Weight of empty container is recorded. Then, grease was applied on internal of the container and weight again. The soil sample was transferred to the cylindrical cup of cone penetrometer apparatus, ensuring that no air is trapped in the soil sample and the weight is recorded. The soil sample was left air dried until no changes in the soil weight. The weight of metal cup with soil sample after oven dried were recorded. The dry soil sample is tied with thread and coated with wax. The weight of dry soil with thread and weight of dry soil with thread and wax were recorded. The dry soil with thread and wax is weight in air and in water. The graph of void ratio versus water content was plotted. The moisture content corresponding to void ratio of the soil was taken as shrinkage limit of the soil.

3.4.5 Free swell

Free swell test followed ASTM D-4829. 10 ml soil sample was poured into a cylinder and distilled water was added until it reaches 50 ml. The soil sample was left air dried until there is no changes in its volume. The free swell test can be determined by using the Eq. (3.2).

$$FS = \frac{V_1 - V_0}{V_0} \times 100 \tag{3.2}$$

where FS = free swell

 V_1 = soil volume after swelling

 V_o = volume of dry soil

3.4.6 Organic content

Organic content was determined by loss on ignition test based on BS 1377-3 (2012). Soil sample dried for at least one hour, cooled in a desiccator for 30 minutes was added into the crucible until half full and weigh. The soil sample was oven dried for 24 hours before it was cooled and reweigh. Then, the soil sample was heated in a furnace with 440°C for three hours, cooled in a desiccator and weigh again. The heating repeated until no further changes in the soil weight. The loss in ignition can be calculated by using Eq. 3.3.

Organic Content
$$= \frac{M_{s} - M_{A}}{M_{s} - M_{c}} \times 100$$
(3.3)
where M_{s} = mass of crucible and oven dried soil sample
 M_{A} = mass of crucible and soil sample after ignition
 M_{c} = mass of crucible

3.4.7 Cation exchange capacity

5g of soil were mixed with ammonium acetate solution in a centrifuge tube. Ammonium hydroxide solution and acetic acid were used to adjust the pH value to 7. The sample were subjected to a rotation of 115 rpm for 15 minutes and were left overnight. The sample were then filtered using Whatman 42 filter paper and vacuum pump. 30 ml of 1 M ammonium acetate solution were poured four times. The liquid extracted from each test were collected and then analyzed using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES).

3.4.8 Surface area

The specific surface area of the soil were determined by using Bruker SB Tiger Accelerated Surface Analyzer ASAP 2020. The specific surface area of each soil samples were compared.

3.4.9 Mineral composition

An X-ray diffraction (XRD) device, Rigaku Miniflex II was employed to determine the mineral compositions of each soil sample. The changes in the mineralogical compositions of the soil samples were compared.

3.5 Soil –water retention curve

Soil powder specimens were mixed with deionized water at increments of about 2%. The soil-water mixtures were then allowed to cure for seven days before being tested. In this study, osmotic technique was used to control suction. Total suction is obtained using Decagon WP4C chilled-mirror dew-point hygrometer.

3.5.1 Osmotic pressure

Dialysis cellulotic membrane 14,000 was soaked in distilled water for about 30 minutes prior being used. Solution of polyethyleneglycol (PEG) 20,000 was prepared by adding 25, 75 and 125 g of PEG powder with 250 ml distilled water in a beakers and magnetic stirrer was put into the beakers. The PEG solutions have to be stirred continuously by using magnetic stirrer. After that, 4 g slurry sample was enclosed in the semi-permeable membrane and then immersed into the PEG solutions prepared earlier. The mass of the sample were obtained every day until no further changes occur. After the constant mass were reached, the sample were then oven dried for 24 hours and the resulted oven-dried mass of the sample were determined. The suction of PEG solutions at different concentration were measured using chilled-mirror dew-point technique.

3.5.2 Chilled-mirror dew-point technique

In Decagon's chilled mirror devices, a test specimen is inserted into a sealed chamber that contains a mirror together with means to chill and detect condensation on the mirror. All suction measurements in this study were carried out at 25°C using the "precise mode". During measurements, the specimen holder was filled with approximately 5 g of soil-water mixtures, less than half of the volume (6 cm3). This step was taken to minimize contamination to the testing chamber. Initially, all specimens were thermally pre-conditioned to meet the device inner block's temperature before being tested. The suction were taken from chilled mirror device. Soil sample was taken out from the device and weigh. After 24 hours, the dry weight of the soil sample was obtained and water content was calculated. The graph of gravimetric water content versus log total suction is plotted.



Figure 3.1: WP4C Potential dew meter for soil suction reading

The figure 3.1 shows the apparatus of WP4C Potential dew meter for soil suction reading. The reading can be taken when TS-Tb is in range of -0.11 until 0.19. The sample need to be cooled for a while to ensure WP4C Potential dew meter can read the sample.

3.6 Shear strength

The soil sample were added with deionized water with increment of 2%. The sample were kept in sealed prior being tested. In this study, vane shear was used to determine shear strength of every soil sample.

3.6.1 Vane shear

The soil sample transferred to the cylindrical cup, ensuring that no air is trapped in the soil sample. The vane shear were gently lowered into the specimen to their full length without disturbing the soil specimen. The top of the vanes were adjusted to 25 mm below the top of the specimen. The vanes were rotated using motor until the specimen fails. Final reading of the angle of deflection were recorded. The undrained shear strength can be calculated by using Eq. 3.4.

$$c_u = \frac{T}{\pi \left[\frac{d^2h}{2} + \beta \frac{d^3}{4}\right]}$$
(3.4)

Where c_u = undrained shear strength

T = torque

•

d = diameter of the shear vane

h = height of the shear vane

$$\beta = \frac{2}{3}$$
 for uniform mobilization of undrained shear strength

T is given by
$$\frac{\text{spring constant}}{180 \times \text{difference between initial and final reading}}$$

3.6.2 Shear strength

The soil sample were added with deionized water with increment of 2%. The sample were kept in sealed prior being tested. In this study, vane shear was used to determine shear strength of every soil sample.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Physical properties

This chapter will discuss the results obtained from all the tests that had be done for the research. The soil properties of FMC Kaolin and S300 such as initial water content, specific gravity, liquid limit, shrinkage limit, free swell, cation-exchange capacity and loss on ignition were discussed in this chapter.

4.1.1 Specific gravity

Based from the results, it is shown that specific gravity of soils with SAP is lesser than normal condition. This is probably because the water retain in SAP mixture is higher than normal soils which make it less value than normal.

4.1.2 Liquid limit

From the result, there is an obvious difference in FMC and FMC+SAP which are 80% and 120% respectively. The values indicates that is possible for clay to have a potential of retaining water because it is suggested there are inter-particle forces that have remarkable rule in determining the liquid limit value. Inter-particle attraction and repulsive determine the particle arrangement and regulates the liquid limit. According to Srihadran et al., (1988), it was anticipated that kaolinite soils with higher liquid limit and higher degree of particle flocculation should have less shrink and will occupy higher sediment volume than anticipated. For S300 and S300+SAP, the liquid limit were 41% and 83.85% respectively.

4.1.3 Plastic limit

The results shows that the value of FMC with SAP is the highest compared with others, the reason behind is that SAP helps the soils to have more expansion and enhance their ability. It can be said that the swelling properties of the soils is greatly assist by Super Absorbent Polymer and may compete with Bentonite, one of the most expansive clay in clay family (Seed et al., 1964).

4.1.4 Shrinkage limit

Last of the series in Atterberg Limit where value of normal soils and with SAP showed an increase. It is stated that it depends on the clay types and clay content, the changes in smecite/kaolinite are associated with micropore volumes and swelling capacity. Since FMC and Speswhite has really fine particles, it might be have lesser micropore volumes and hard for water to pass through. SAP can boost the ability and allow the micropore volumes and swelling capacity become bigger then affect the SL value (Boivin et al., 2004).

4.1.5 Water content

For water content, there is only a slight increase in value in either normal or SAP mix. The probability is that the SAP is not really affect the soils since the water in each kaolinite is lesser than 1.0 and it could not have more effect compared when large amount of water is present.

4.1.6 Loss on ignition

Other than any other properties, there is a decrease in LOI of mixture with SAP than the normal soils. The cause is probably because of the chemical nature of the clay itself, where it naturally it has more nutrients in the soil. In this case however, FMC and S300 ignored the bound water and it is tended that higher rate of water absorption usually will have a higher value of LOI (Howard et al., 1990).

4.1.7 Free swell

The last physical properties that were determined swelling potential from the soils with or without SAP. It looks that one with SAP can almost reach 180% from the original state, because of the diffuse double-layer repulsive forces (Prakash et al., 2004).

4.1.8 Soil physical characteristics

The table below shows the value for each physical characteristics of the soil and the difference on number in each of one without and with addition of SAP.

Physical	FMC	FMC+SAP	S300	S300+SAP
properties				
Specific gravity,	2.62	2.33	2.62	2.70
G_s				
Liquid limit, LL	80	120	41	83.85
Plastic Limit, PL	49.40	64.87	27.90	48.79
Shrinkage Limit,	67.41	-	38.5	40
SL				
Water content, w	1.011	1.678	1.223	1.000
Loss on Ignition	0.112	0.056	0.0007	0.00785
Swell index, C	10	80.0	1	70.0
CEC	1.45	20.90	0.81	10.10

Table 4.1: General table data for soil physical characters.

The table summarises the specific gravity of FMC, FMC+SAP, S300 and S300+SAP are 2.62, 2.33, 2.62 and 2.70 respectively. For liquid limit, FMC is 80, FMC+SAP is 120, S300 is 41 and S300+SAP is 83.75. Plastic limit for FMC, FMC+SAP, S300 and S300+SAP are 49.40, 64.87, 27.90 and 48.79 respectively. Shrinkage limit for FMC is 67.41, 38.5 for S300 and 40 for S300+SAP. Water content for FMC, FMC+SAP, S300 and S300+SAP are 1.011, 1.678, 1.223 and 1.000 respectively. The loss on ignition for FMC is 0.112, 0.056 for FMC+SAP, 0.0007 for S300 and 0.00785 for S300+SAP. Foe swell index, the results are 10, 80, 1 and 70 for FMC, FMC+SAP, S300 and S300+SAP respectively. CEC value for FMC is 1.45, 20.90 for FMC+SAP, 0.81 for S300 and 10.10 for S300+SAP.

4.2 Oedometer test

The results of oedometer test conducted of FMC and S300 kaolinites are in tabulation of data and shows in graph as below.

4.2.1 Water content-pressure results

Loading (g)	Pressure(kPa)	Water content (%)	Void ratio, e
125	63.7	0.619	1.64
250	127.4	0.579	1.53
500	254.77	0.534	1.41
2000	1019.1	0.536	1.42
16000	8152.9	0.478	1.25
32000	16305.7	0.479	1.25

Table 4.2 Oedometer results for FMC

The void ratio of the sample are slightly decrease from 1.64 to 1.25 when the pressure applied increased from 63.7 kPa tp 16305.7 kPa.

Table 4.3 Oedometer results for FMC+	SAP
--------------------------------------	-----

Loading (g)	Pressure(kPa)	Water content (%)	Void ratio, e
125	63.7	3.075	12.02
250	127.4	2.660	10.40
500	254.77	2.656	10.38
2000	1019.1	2.180	8.52
16000	8152.9	2.722	6.34
32000	16305.7	2.705	6.30

There is great decrease of void ratio from 12.02 to 6.30 when SAP is added to the FMC.

Loading (g)	Pressure(kPa)	Water content	Void ratio, e
		(%)	
125	63.7	0.251	0.80
250	127.4	0.244	0.78
500	254.77	0.241	0.77
2000	1019.1	0.230	0.74
16000	8152.9	0.425	0.73
32000	16305.7	0.428	0.72

Table 4.4	Oedometer	results	for	S300
	Occonnecter	results	101	0500

Table 4.4 shows oedometer results for S300 where void ratio slightly decrease when the pressure applied is increased.

Loading (g)	Pressure(kPa)	Water	content	Void ratio, e
		(%)		
125	63.7	4.568		12.33
250	127.4	4.136		11.17
500	254.77	3.634		9.81
2000	1019.1	3.606		9.74
16000	8152.9	4.172		9.68
32000	16305.7	4.539		9.61

Table 4.5 Oedometer results for S300+SAP

The addition of SAP to S300 shows decrease in void ratio in Table 4.5 when the pressure is increased.

4.3 Water content-pressure curve



Figure 4.1 Water content-pressure curve for FMC with and without SAP.

From figure 4.1, the FMC with addition of SAP is higher than FMC without addition of SAP.



Figure 4.2 Water content-pressure curve for S300 with and without SAP

The figure 4.2 shows the increase in S300 with addition of SAP as compared without addition of SAP.



Figure 4.3 Void ratio-pressure curve for FMC with and without SAP

In the figure, the FMC with addition SAP has slightly higher than FMC without SAP.



Figure 4.4 Void ratio-pressure curve for S300 with and without SAP

The figure shows the S300 with SAP have higher void ratio than S300 without SAP.

CHAPTER 5

CONCLUSION

5.1 Introduction

From the results, the study of the behaviour clay by using superabsorbent polymer (SAP) can be summarized where

- 1. The plasticity characteristics of FMC and S300 can be determined
- 2. The water retention of FMC and S300 can be determined.
- 3. The compressibility behaviour of FMC and S300 can be determined. Thus, the objectives of the study is achieved.

5.2 Recommendation

The study can let others to explore the application and properties with addition of superabsorbent polymer (SAP).

REFERENCES

Adamis, Z., & Williams, R. B. (2005). Bentonite, kaolin and selected clay minerals. -387.

- Bakass, M., Mokhlisse, A., & Lallemant, M. (2002). Absorption and desorption of liquid water by a superabsorbent polymer: Effect of polymer in the drying of the soil and the quality of certain plants. Journal of applied polymer science, 83(2), 234-243.
- Bouazza, A., Zornberg, J. G., & Adam, D. (2002). Geosynthetics in waste containment facilities: recent advances.
- Bouazza, A. and Bowders, J.J. 2010. Geosynthetic clay liners in waste containment facilities.CRC Press/Balkema.: 254.
- Bogue, R. H. (1955). The chemistry of Portland cement. Soil Science, 79(4), 322.
- Burst, J. F. (1991). The application of clay minerals in ceramics. Applied Clay Science, 5(5-6), 421-4
- Chen, P., Zhang, W. A., Luo, W., & Fang, Y. E. (2004). Synthesis of superabsorbent polymers by irradiation and their applications in agriculture. Journal of applied polymer science, 93(4), 1748-1755
- Fredlund, D. G., Sheng, D., & Zhao, J. (2011). Estimation of soil suction from the soilwater characteristic curve. Canadian geotechnical journal, 48(2), 186-198.
- Huettermann, A., Orikiriza, L. J., & Agaba, H. (2009). Application of superabsorbent polymers for improving the ecological chemistry of degraded or polluted lands. CLEAN–Soil, Air, Water, 37(7), 517-526.
- Kabiri, K., Omidian, H., Zohuriaan-Mehr, M. J., & Doroudiani, S. (2011). Superabsorbent hydrogel composites and nanocomposites: a review. Polymer composites, 32(2), 277-289.
- Koch, D. (2002). Bentonites as a basic material for technical base liners and site encapsulation cut-off walls. Applied Clay Science, 21(1), 1-11.

- LI, Z. X., LIANG, Q., & HAN, L. (2010). Application Progress of Bentonite in Agriculture and Animal Husbandry Environmental Protection. Environmental Science & Technology, S2.
- Luckham, P. F., & Rossi, S. (1999). The colloidal and rheological properties of bentonite suspensions. Advances in colloid and interface science, 82(1), 43-92.
- Mechtcherine, V., & Reinhardt, H. W. (Eds.). (2012). Application of super absorbent polymers (SAP) in concrete construction: state-of-the-art report prepared by Technical Committee 225-SAP (Vol. 2). Springer Science & Business Media.
- Meunier, A., Velde, B., & Griffault, L. (1998). The reactivity of bentonites: a review. An application to clay barrier stability for nuclear waste storage. Clay Minerals, 33(2), 187-196.
- Obaje, S. O., Omada, J. I., & Dambatta, U. A. (2013). Clays and their industrial applications: Synoptic Review. International Journal of Science and Technology, 3(5), 264-270
- P. Lura, O.M. Jensen, K. van Breugel. (2003).Autogenous shrinkage in high-performance cement paste: an evaluation of basic mechanisms Cem Concr Res, 33 (2). 223-232.
- Pascente, J. E., & Pascente, T. J. (1998). U.S. Patent No. 5,849,210. Washington, DC:U.S. Patent and Trademark Office
- Pusch, R. (1982). Mineral–water interactions and their influence on the physical behavior of highly compacted Na bentonite. Canadian Geotechnical Journal, 19(3), 381
- Pusch R. and Yong R. N. 2005. Microstructure of Smectite Clays and Engineering Performance (Spon Research) (1st Edition). Taylor and Francis, 1st Edition
- Reeves, G. M. Sims, I. and Cripps, J. C. 2007. Clay Materials Used in Construction.Geological Society Engineering Geology Special Publications No. 21. Cromwell Press.43.
- Reeves, G. M., Sims, I., & Cripps, J. C. (2006). Clay materials used in construction. Geological Society of London.

- Rowe, R. K. Booker, J.R. and Quigley, R.M. 1997. Clayey barrier systems for waste disposal facilities. Taylor and Francis. 1st Edition.
- Shuai, F. (1996). Simulation of swelling pressure measurements on expansive soils (Doctoral dissertation).
- Smith, S. J., & Lind, E. J. (1995). U.S. Patent No. 5,462,972. Washington, DC: U.S. Patent and Trademark Office.
- Tadza, M., & Yuhyi, M. (2011). Soil-water characteristic curves and shrinkage behaviour of highly plastic clays: an experimental investigation. Cardiff University.
- Van Olphen, H. (1964). An Introduction to Clay Colloid Chemistry. Soil Science, 97(4), 290.
- Wyrzykowski, M., & Lura, P. (2013). Controlling the coefficient of thermal expansion of cementitious materials–A new application for superabsorbent polymers. Cement and Concrete Composites, 35(1), 49-58.
- Yang, N. and Barbour, S.L. 1993. A review of the influence of clay–brine interactions on the geotechnical properties of Ca-montmorillonitic clayey soils from western Canada. Canadian Geotechnical Journal, Vol.30,: 920-934.

APPENDIX A SAMPLE APPENDIX 1

For Appendices Heading, use TITLE AT ROMAN PAGES style.

APPENDIX B SAMPLE APPENDIX 2

For Appendices Heading, use TITLE AT ROMAN PAGES style.