



**Index testing and assessment of the suitability of  
bentonite from the Imerys Bentonite Mine, for use  
in Geosynthetic Clay Liners**

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the degree of Master of Science in Engineering Geology.

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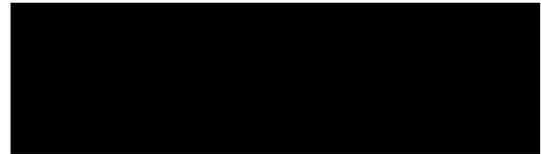
## Declaration

I, **Ahmad Patel**, hereby declare that this thesis is essentially my own work, except where otherwise indicated, and has not, to the best of my knowledge, been submitted for a degree at any other university.



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My sincerest gratitude,



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## Abstract

Geosynthetic Clay Liners (GCLs) have become a suitable substitute for compacted clay liners. Their use centres on liquid impoundments, such as to prevent leachates from landfills seeping into the groundwater system and as sealant in tailings dam. GCLs are thin sheets of bentonite encased between two geotextiles. The main component of GCLs is bentonite, which formed as an alteration product of volcanic ash, comprised mainly of montmorillonite. Bentonite exists as either sodium bentonite or calcium bentonite, depending on the type of montmorillonite present within it. To perform as an effective hydraulic barrier, sodium bentonite is the preferred type due to its high-water retention characteristics and swelling potential. Often times, the bentonite does not meet the desired swell index. As such, a process known as activation is undertaken, whereby soda ash ( $\text{Na}_2\text{CO}_3$ ) is mixed with borderline quality bentonite. This study investigated the suitability of bentonites from the Imerys mine in the Western Cape Province of South Africa for use in GCLs. In this research, both activated and non-activated bentonites were investigated. X-Ray diffraction analysis was conducted on the bentonites in order to determine their bulk mineralogical composition. Swell index test, fluid loss test, plate water absorption tests and Atterberg limits test were also conducted on samples of activated and non-activated bentonites. Furthermore, swell index tests were conducted to investigate the extent of beneficiation over time. The XRD results reveal that activated and non-activated bentonite have a smectite content of approximately 58 % and 67 % respectively with the major impurity being quartz. The swell index of non-activated bentonite was significantly lower than the activated bentonite. However, the activated bentonite did not swell to the required minimum of 24 ml/2g as it did not achieve full activation. Activated samples of bentonite tested at different times subsequent to activation reveal that the activation requires at least 4 weeks for the ideal ratio of 1:50, soda ash to bentonite, to fully activate. The fluid loss results also displayed results slightly above the required minimum, of 18 ml, as a result of the low swell index. Activated and non-activated bentonite has an absorption capacity of approximately 133 % and 121 %. The plasticity index is 101 % for activated bentonite, 15 % higher than non-activated bentonite. Moreover, a moderate correlation between plate water absorption and liquid limit was found for activated bentonite. The activated bentonite from Western Cape is suitable for use in GCLs provided the blend of bentonite is not of very low quality and sufficient time is given for activation to reach completion. Imerys bentonite is a medium quality bentonite with borderline index properties that requires beneficiation and time to achieve complete activation.

## **Research output**

During this research, the following paper was presented and published in the Proceedings of the African Regional Conference on Soil Mechanics and Geotechnical Engineering held in Cape Town in 2019. The full paper is attached in Appendix 1.

Patel, A., Hingston, E.D.C., James, G.M., Mukwevho, F.V. (2019). The effect of time on beneficiation of bentonites used in geosynthetic clay liners. *Proceedings of the 17<sup>th</sup> African Regional Conference on Soil Mechanics and Geotechnical Engineering*. pp 413-418.

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# Chapter 1

## Introduction

### 1.1 General background

Geosynthetic Clay Liners (GCLs) have gained much professional attention over the past decade as a substitute for compacted clay liners in cover systems, composite bottom liners, environmental protection barriers and as liners for canals, ponds or surface impoundments (Bouazza, 2002). GCLs are thinly manufactured hydraulic barriers comprising a layer of sodium bentonite encased between two geotextiles (Setz *et al.*, 2017). Sodium bentonite is the most critical component of a GCL due to its high water retention characteristics and swelling potential (Seiphoori *et al.*, 2016). Subsequent to installation, GCLs hydrate to a certain degree of saturation through the uptake of water from the subsoil. When the bentonite absorbs water under confinement, it swells, thereby reducing the void ratio. Thus, a lower void ratio can be obtained after the saturated condition, which results in a better performance of the GCL in terms of the limited hydraulic conductivity (Seiphoori *et al.*, 2016).

The primary function of GCLs is to act as hydraulic barriers for various liquids such as leachates and tailings in varied environments such as landfills and tailings dams (Bouazza, 2002). Therefore, investigations are aligned, but not restricted to properties that relate to hydraulic conductivity, such as fluid loss, free swell and absorption.

The focal point of this research was bentonite, which is the active ingredient of GCLs. There are two types of bentonites namely, sodium bentonite and calcium bentonite, each type denoting the predominant exchangeable ion. Sodium bentonite is preferred over calcium bentonite due to its better swelling properties. The primary constituent of sodium bentonite, which contributes to swelling, is montmorillonite (Norris, 1954). Montmorillonite comprises two silica tetrahedral sheets with a central octahedral sheet. Water molecules and cations occupy the space between the layers (Al-Ani and Sarapää, 2008). Generally, the typical sodium bentonite used in GCLs will contain 60–85 % montmorillonite (Von Maubeuge, 2002).

Sodium bentonite has a high swelling capacity whereas bentonite with lower amounts of sodium has a much lower swelling capacity. However, this can be improved by treatment with sodium carbonate or soda ash ( $\text{Na}_2\text{CO}_3$ ), to produce sodium exchanged bentonite, by increasing the concentration of exchangeable sodium ions within the bentonite (Al-Ani and Sarapää, 2008). Sodium ion has a large hydration radius, thus has the ability to retain high volumes of absorbed water. This is primarily responsible for the high swell index and sealing ability of a



### **1.3 Statement of the problem**

Hydration of bentonite without adequate confining pressure will result in free swell. This will continuously progress until saturation, as the bentonite absorbs water from the subgrade or precipitation.

In certain instances, the mined bentonite does not possess the desired swell potential. Thus, a process known as beneficiation is required. Beneficiation is a process whereby soda ash ( $\text{Na}_2\text{CO}_3$ ) is added to the bentonite to increase its sodium content (von Maubeuge, 2002). Customers require bentonite of a certain specification. Thus, all mined bentonite go through some processing prior to sale. Bentonite is sourced from Imerys to be used in the manufacture of GCLs. But would it meet the minimum fluid loss and swell index performance requirements? In this instance, how does non-beneficiated bentonite perform relative to beneficiated bentonite? What is the effect of time on the beneficiation process of bentonite used in GCLs? Problems such as inadequate swell defeat the main purpose of a GCL and destroy its primary function, which is to serve as a hydraulic barrier. However, it is not always feasible to conduct extensive tests prior to installation. Hence there is a need to determine complete suitability of the Imerys bentonite used in GCLs through extensive laboratory testing.

### **1.4 Research hypothesis**

Cape bentonite is a medium quality bentonite with borderline index properties that requires beneficiation and time to achieve complete activation.

### **1.5 Aim & objectives**

The aims of this research were to investigate the suitability of sodium bentonite from the Imerys bentonite mine for its use in GCLs and to examine the difference in the behaviour of beneficiated versus non-beneficiated bentonite.

The objectives of this research were:

- i. to determine the major mineralogical composition of acquired bentonite through X-Ray Diffraction (XRD) techniques;
- ii. to subject samples of bentonite to Fluid Loss (ASTM D 5891-02) tests on samples of bentonite in order to determine the amount of fluid loss under a specific air pressure;
- iii. to subject samples to Swell Index (ASTM D5890-1) tests in order to determine the amount of free swell a sample undergoes when submerged in water. Furthermore, the

- change in swell index over time was tested to determine the effect of time on activation of bentonite on samples of in-house activated bentonite;
- iv. to deduce water absorption of bentonite samples over a specified time period by conducting plate water absorption tests;
  - v. to determine the liquid and plastic limits of the bentonite by conducting Atterberg limits tests; and
  - vi. to deduce a relationship between the liquid limit and the plate water absorption.

## **1.6 Synopsis of chapters**

### **1.6.1 Chapter 1- Introduction**

Chapter 1 presents an introduction to the research. It starts by giving a brief background of GCLs and bentonite. It also touches on the definition of sodium activation. Thereafter, the location of the study is explained, followed by the problem statement. Lastly, the aims and objectives of the study are clearly stated.

### **1.6.2 Chapter 2 - Geological Setting and Mining Method**

Chapter 2 describes the regional geology of Heidelberg. Comprehensive explanations are given about the origin and formation of the bentonite beds within the stratigraphy. The typical lithology for each stratigraphic unit of the regional geology is defined. Further explanation is given to illustrate the method of mining and the processing procedures of the raw bentonite.

### **1.6.3 Chapter 3 - Literature Review**

Chapter 3 presents a detailed literature review pertaining to the research. To get an understanding of clay minerals and the composition of bentonite, the chapter starts by explaining the two elementary structures of individual clay minerals. Thereafter, it explains the combinations of these building blocks and their mineral classification. The chapter further elucidates on smectite minerals and their clay mineral classification. Bentonite is then introduced into the chapter and is discussed in detail. Subsequently, GCLs are described including its properties, problems and uses. Lastly, beneficiated bentonite is defined and explained.

### **1.6.4 Chapter 4 – Methodology**

Chapter 4 explicitly outlines the procedures involved in carrying out this research to fulfil the objectives set out. It details the test methods for each test conducted and explains why the

procedures were carried out in a particular manner. The test procedures include XRD, swell index, fluid loss, plate water absorption and Atterberg limits.

### **1.6.5 Chapter 5 – Results and Discussion**

Chapter 5 presents the results from the various tests and further discusses the results. These results are: XRD analysis, swell index, fluid loss, plate water absorption and Atterberg limits. This chapter presents the results of each test in individual sub-sections and immediately thereafter discusses the significance of the results. Results for swell index include investigations of the effect of time on the beneficiation of bentonite. The Atterberg limits section includes the correlation of liquid limit to plate water absorption.

### **1.6.6 Chapter 6 – Conclusion and Recommendations**

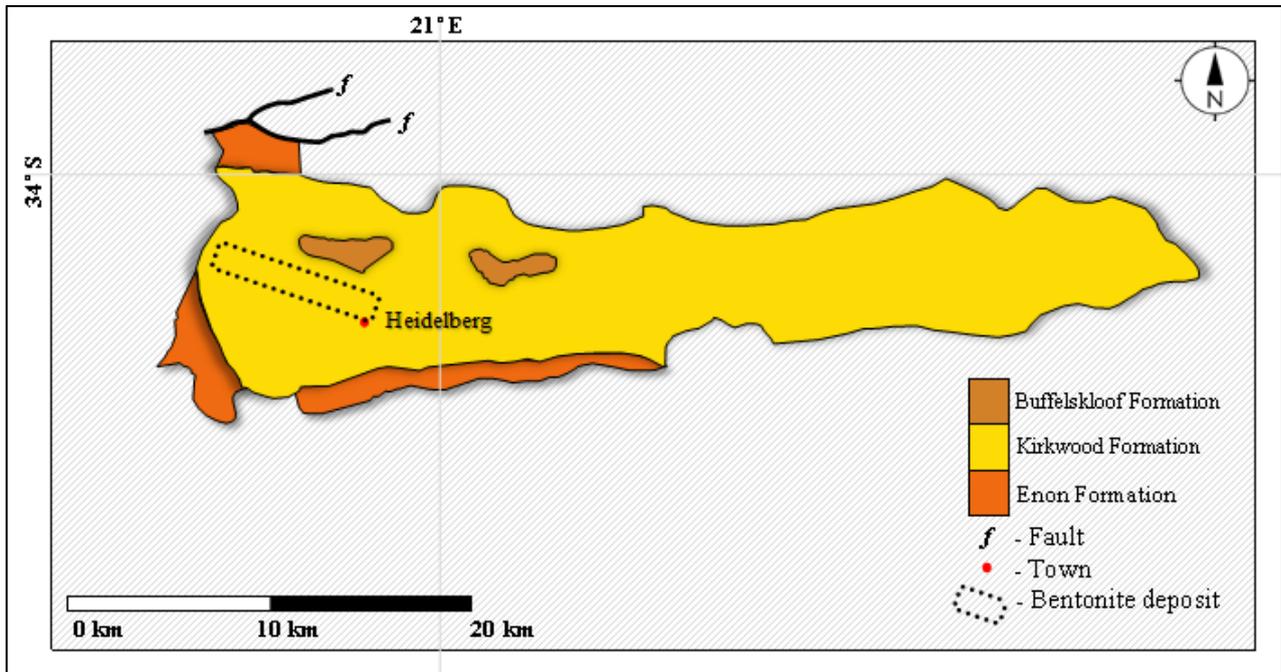
The concluding chapter highlights and summarises the main points of the research and reiterates the key aspects of the dissertation. This chapter also states the main findings of this research and the recommendations that should be implemented. It further explains the difficulties experienced, the constraints of this study and the necessity for future research.

# Chapter 2

## Geological Setting and Mining Method

### 2.1 Introduction

The Imerys mine lies within the Heidelberg Basin, which hosts the Uitenhage Group (Figure 2.1). During the Cretaceous, separation of east and west Gondwana and their eventual separation leading to South America and Africa, caused the development of these rift basins (Muir *et al.*, 2015). During the continental breakup, a series of normal faults developed in the Western Cape which provided accumulation space for what is now known as the Uitenhage Group, hosted in several rift basins such as the Heidelberg Basin (Muir, 2018). The Heidelberg Basin is situated in the vicinity of Heidelberg, Western Cape, South Africa. Deposited within this basin is the Enon Formation, the Kirkwood Formation and the Buffelskloof Formation of the Uitenhage Group (Muir *et al.*, 2017a). The bentonite deposits are found within the Kirkwood Formation.



**Figure 2.1:** Geology map of the Heidelberg area (after Council for Geoscience geological map, 2019).

### 2.2 The Uitenhage Group

During the Early Cretaceous separation of Africa and South America, a series of normal faults developed in the cape region of South Africa, which provided sedimentation space for the

Uitenhage Group to accumulate within these rift basins. The Uitenhage Group (Table 2.1) currently exists within several on-shore and off-shore basins (Renne *et al.*, 1996; Muir *et al.*, 2017a). The Uitenhage Group comprises 8 Formations, namely: the Hartenbos Formation, the Buffelskloof Formation, the Robberg Formation, the Sundays River Formation, the Brenton Formation, the Infanta Formation, the Kirkwood Formation and the Enon Formation (Muir, 2018) (Table 2.1). However, in the Heidelberg basin, only the Enon Formation, the Kirkwood Formation and the Buffelskloof Formation exist.

### **2.3 The Enon Formation**

The Enon Formation currently exists in several offshore basins and onshore basins within the Eastern Cape and Western Cape Provinces of South Africa (Muir, 2018). It is the oldest and most proximal unit of the Uitenhage Group and comprises predominantly conglomerate deposits.

The Enon Formation deposits are primarily thick beds of poorly sorted pebble/cobble conglomerates with subordinate sandstone and mudstone. Majority of the deposits were derived from the weathering of the Cape Fold Belt Mountains (Muir *et al.*, 2017a). The age of the Enon Formation is disputed. However, McLachlan and McMillan (1976) suggested that deposition of the Enon Formation occurred over a period of time, from the Jurassic until the Early Cretaceous.

### **2.4 The Kirkwood Formation**

The Kirkwood Formation has variable layers of fossil bearing sandstones, mudstones and shales (Muir *et al.*, 2017b). The age of these beds are broadly defined as Jurassic to Early Cretaceous. The Kirkwood Formation in general, is a sedimentary succession of intercalated sandstones and mudstones, with subordinate conglomerates, which are usually interbedded with sandstone and minor volcanoclastic deposits. The volcanoclastic deposits consist of tuff and bentonite (altered tuff), which is of significant economic importance (Muir *et al.*, 2017b). During the breakup of Gondwana, many volcanic activities occurred, any of which could have contributed to the deposits of volcanic ash within the rift basins. Ash-fall events are generally the mechanism of deposition of volcanic ash. However, it is unclear exactly which of the volcanic events caused the deposition of volcanic ash within the Western Cape rift basins (Muir *et al.*, 2017b).

**Table 2.1:** Summary of the Uitenhage Group and its formations (after Muir, 2018).

Ma	Era	Period	Group	Formation	Typical lithology
100 ma	Mesozoic	Early Cretaceous	Uitenhage	Hartenbos	Fine to coarse grained sandstone
				Buffelskloof	Conglomerates
				Robberg	Fine to medium grained sandstone
				Sundays River	Medium grained sandstone beds interbedded with mudstones
		Late Jurassic OR Early Cretaceous		Brenton	Massive claystones, siltstones, sandstone and conglomerates
		Late Jurassic – Early Cretaceous		Infanta	siltstones, claystones and shales
				Kirkwood	Mudstone, fine to coarse grained sandstone, conglomerates and bentonite
Enon	Conglomerates with interbedded fine to medium grained sandstone				
150 ma					

## **2.5 The Buffelskloof Formation**

Overlying the Kirkwood Formation in the Heidelberg Basin is the topmost Buffelskloof Formation, which is a conglomerate dominated unit of the Uitenhage Group. An angular unconformity separates it from the underlying units. The predominant constituents of the Buffelskloof Formation are conglomerate with very rare mudstone and siltstone (Viljoen and Cawthra, 2019).

## **2.6 Bentonite deposits at Heidelberg**

The bentonite deposit is located within the Heidelberg-Riversdale Basin. The basin was first formed during the Cape Fold Belt orogeny (i.e. Permo-Triassic) as a foreland basin. Thereafter, it continued to subside during the fragmentation of Gondwana due to the opening of the South-Atlantic (Viljoen & Cawthra, 2019). These basins formed on the southern downthrown side of normal mega faults (Horn and Strydom, 1998). Clastic sediments representing alluvial fans, braided rivers and lakes, filled the basins and together form the Uitenhage Group (Viljoen & Cawthra, 2019). Within this group, bentonite occurs in lacustrine mudstone of the Kirkwood Formation and formed as a result of the diagenesis of volcanic ash layers that were deposited in lakes receiving suspension-settled muds (Cole *et al.*, 2014).

During the formation of the Heidelberg-Riversdale basin, alkaline volcanic events occurred, leading to the deposition of volcanic ash in a salty lacustrine environment (Cole *et al.*, 2014). This saltwater-volcanic ash interaction led to the formation of sodium bentonite. A particularly active period of volcanism occurred between 162–72 ma in the Antarctic Peninsula that could have provided suitable sources for the bentonites hosted in lacustrine deposits of the Kirkwood Formation in the Heidelberg basin of southern Cape (Muir, 2018). The glass components of the ashes were chemically altered in this low energy environment and consolidated into distinct clay layers. These clay layers currently form the Heidelberg-Riversdale bentonite deposits (Christidis and Huff, 2009).

Alteration of volcanic glass may occur through vapour-phase crystallization, burial diagenesis, contact metamorphism, hydrothermal activity, and by hydrolysis either in alkaline or percolating groundwater. In most cases, bentonites form through alteration by a fluid such as shallow seas or lakes (Christidis and Huff, 2009). Leaching of alkali elements and high  $Mg^{2+}/H^{+}$  are required to form smectites during the alteration of volcanic glass. The conversion of volcanic glass to smectites involves movement of elements to and from the volcanic glass.

The loss of alkalis and a high magnesium activity promote the formation of smectite (Christidis, 1998).

There are multiple bentonite horizons mined at Heidelberg. Figure 2.2 shows one of the bentonite horizons mined at Imerys. In this illustration, the upper bentonite horizon is a grey-white layer within the lacustrine facies of the Kirkwood Formation, which is overlain predominantly by mudstones. This horizon extends laterally for approximately 30 m with a thickness of 1.5 m and a dip of 15° to the north. The high montmorillonite content and constant dimensions suggest an ash-fall origin of this bentonite. Other bentonite horizons range between 1 and 1.7 m in thickness and overlain mainly by siltstones (Muir *et al.*, 2017b).

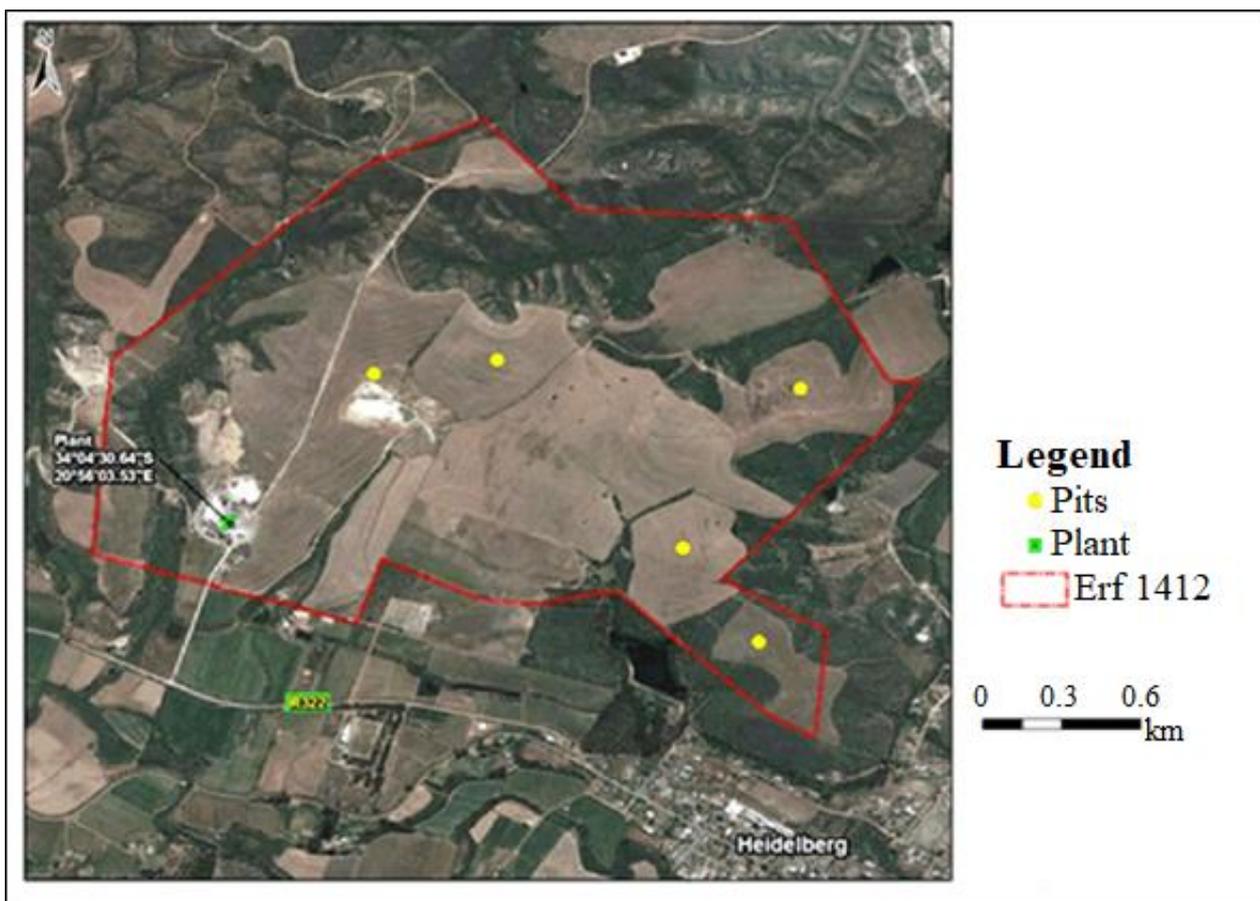


**Figure 2.2:** A light grey bentonite horizon (outlined in red), overlain and underlain by mudstone (after Cole *et al.*, 2014).

The bentonite reserves of South Africa are in excess of 8 million tons although only a portion is considered to be mined economically (Agnello, 2004). It is estimated that the bentonite reserve of the Heidelberg region is about one million tons (Gray *et al.*, 2013). The mining rights for this region is currently held by Imerys.

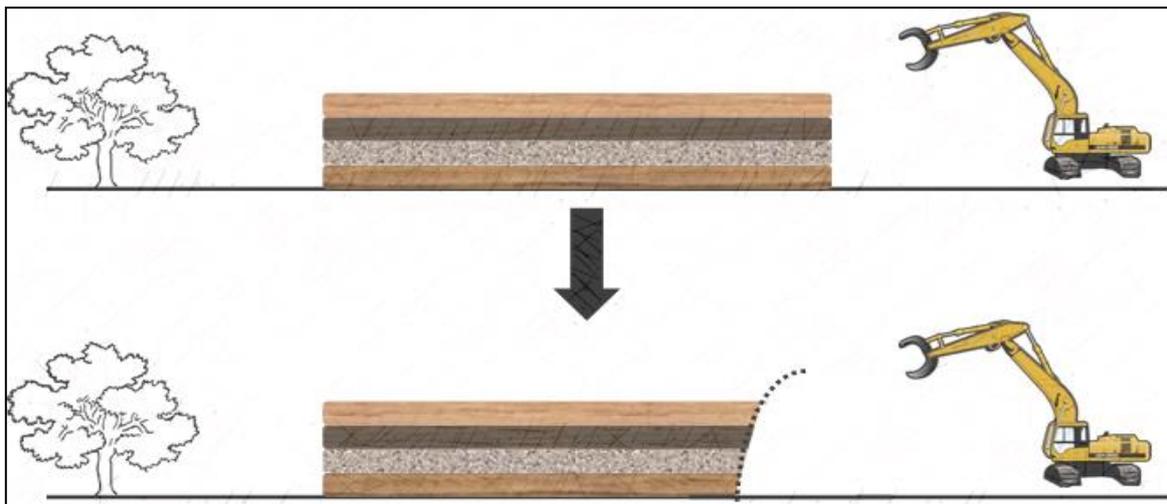
## 2.7 Mining of bentonite at Imerys Mine

The method of mining of the bentonite at the Imerys Mine is a shallow open pit method, which does not require extensive engineered benches or specific mining techniques. The bentonite was excavated, transported to a processing plant and the pit refilled and rehabilitated after completion. The pits were excavated to the bentonite layer, which was between 10 and 20 m below the surface. Bentonite was removed using an excavator. Since bentonite is soft, no drilling and blasting was required, as is the case with other conventional mines. The excavator simply cuts through the bentonite with the excavator bucket which is then loaded onto tipper trucks for transportation to the processing plant. An access ramp was constructed from the overburden to allow trucks and excavators into the pit. An observed pit was approximately 15 m deep and 50 m in diameter. The bentonite at Heidelberg is of varying quality excavated from several pits as indicated in Figure 2.3.



**Figure 2.3:** Satellite image showing the minable area (demarcated in red). The yellow dots show the locations of the mining pits. These pits may or may not be in operation (after Almond, 2014).

To ensure a uniform quality of bentonite, the bentonite from each pit is interlayered with each other as illustrated in Figure 2.4. Thereafter, slices are taken vertically to create a blend of all qualities and subsequently processed. The conversion process of natural sodium bentonite to activated bentonite follows a procedure by which approximately 1 % to 2 % of soda ash is added to the bentonite via a feed hopper containing soda ash. The activated bentonite then passes through a rotary drier where all excess moisture is removed. Finally, the dried bentonite is added to a roller crusher where it is milled to a fine powder. Bentonite with very low quality will be mixed with other bentonites of higher quality so that the quality of the end product has an average acceptable industry standard. This method also controls the amount of soda ash that is required. With a fairly consistent mix of bentonite, a fixed amount of soda ash can be added to every batch mined. This eliminates the need for constantly changing machinery settings and constant quality testing of bentonite.



**Figure 2.4:** Illustration of interlayering of bentonite to create a consistent mix after mining. Thereafter, taking vertical slices, mixing different qualities of bentonite.

# Chapter 3

## Literature Review

### 3.1 Introduction

This chapter starts by describing the definition and basic structure of clay. This leads onto the classification and categorisation of clays, based on structural configuration. The physicochemical properties of smectite are then described in relation to the classification of clays. The definition and constituents of bentonite as well as its uses are then explained. Furthermore, the chemistry and engineering properties of bentonite are elucidated at length. Thereafter, GCLs are introduced and their properties, such as hydraulic conductivity, are discussed. Moreover, some of the problems associated with GCLs are discussed. Finally, soda ash treated bentonite is discussed.

### 3.2 Clay minerals

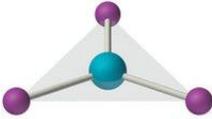
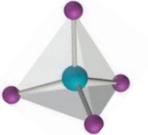
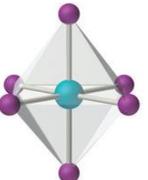
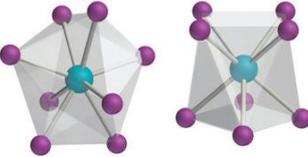
Clays were initially arbitrarily classified as earthly particles whose diameters were less than 2  $\mu\text{m}$  (Barton, 2002; Bergaya and Lagaly, 2006; Velde, 2013). However, the crystal structure and mineral family is what defines clay minerals. Clay minerals in general possess traits that are similar, such as their size, form, crystallographic structure and behaviour, whether physical or chemical (Velde, 2013). Their miniscule size and shape inherently produce a large surface area compared to the volume of the particle. The properties of clays are in fact dominated by their surfaces (Al-Ani and Sarapää, 2008; Velde, 2013). The reason for this is their phyllosilicate shape. The width and length in phyllosilicates are often about 20 times the thickness. The controlling factor of this specific crystallographic growth is the highly covalent ionic bonding present, comprising mostly oxygen and subordinate cations such as silicon and aluminium (Murray, 1999; Bergaya and Lagaly, 2006; Velde, 2013). The resulting atomic crystal structures are tetrahedral and octahedral sheets.

### 3.3 Structure of clay minerals

The elementary structural units of most clay minerals are a silicon–oxygen tetrahedron and an aluminium–hydroxyl octahedron. Although clay minerals comprise tetrahedral or octahedral sheets as their basic singular structure, the arrangements of these sheets account for the physical and chemical differences between the different types of clays (Murray, 1999; Barton, 2002; Al-Ani and Sarapää, 2008). Primarily, ionic bonds occur between oxygen and cations such as

aluminium, silicon, magnesium, potassium and sodium (Murray, 2006; Al-Ani and Sarapää, 2008; Velde, 2013). The ratio between anion to cation radii will determine the number of anion cations contacts.

**Table 3.1:** The limiting radii ratios, coordination number and geometric configuration of the oxygen ions (after Al-Ani and Sarapää, 2008).

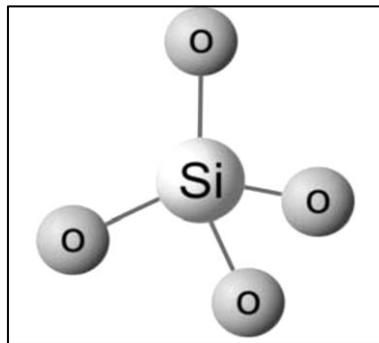
Cation:Anion ratio	Coordination number	Geometric configuration of anions (oxygen)
< 0.16	2	 (linear)
0.16 – 0.23	3	 (Triangular)
0.23 – 0.41	4	 (Tetrahedron)
0.41 – 0.73	6	 (Octahedron)
0.73 – 1.00	8	 (Cube)
> 1.00	12	Close packed sphere

The larger the ratio the more oxygen atoms can surround the cation. The oxygen can bond to the cation in varying geometries (Table 3.1) (Murray, 2006; Al-Ani and Sarapää, 2008). The number of anions in contact with a cation is known as the coordination number (Al-Ani and Sarapää, 2008). Ions forming clay minerals possess a coordination number of 4 or 6, hence produce tetrahedral or octahedral molecules.

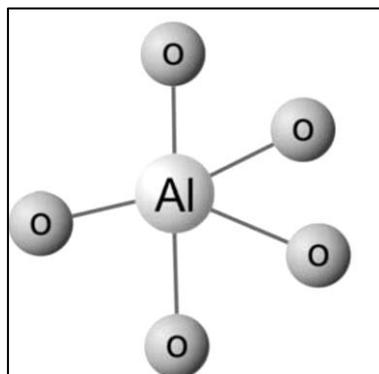
### 3.3.1 Tetrahedron

Clay structures are built of silicon-oxygen tetrahedron (i.e. four oxygen anion atoms bonded to the central silicon cation (Figure 3.1)) (Murray, 2006; Velde, 2013). Seldom, the aluminium cation replaces the silicon cation in the silicon-oxygen tetrahedron. The resulting molecular geometry is a tetrahedron. The net charge of a single tetrahedron is -4. Bonding of adjacent tetrahedron occurs between the three basal oxygens (Figure 3.1a) and hence only the apical oxygen remains with a charge of -1. The valency imbalances result in a net negative charge. Thus, singular units do not exist in isolation but combine to produce sheet structures (Mitchell and Soga, 2005; Craig, 2013).

(a) Tetrahedra



(b) Octahedra

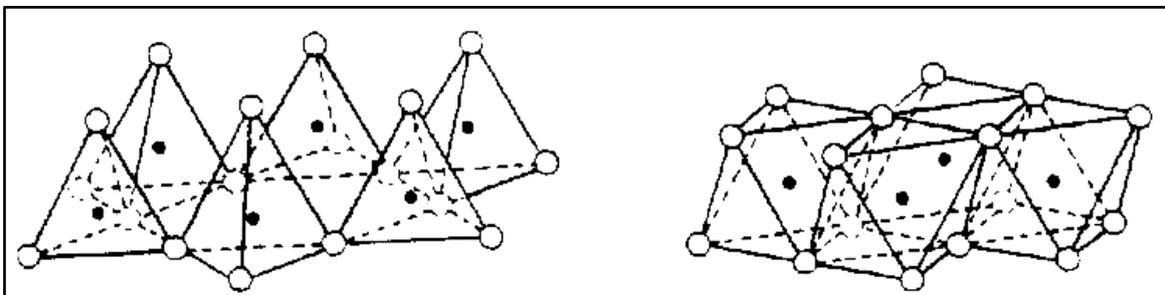


**Figure 3.1:** Tetrahedral and octahedral structure (O- Oxygen, Al- Aluminium, Si- Silicon)

(after Al-Ani and Sarapää, 2008).

### 3.3.2 Octahedron

Octahedral structures are formed with aluminium, magnesium and ferrous iron ions where six oxygen or hydroxyl group atoms lie at each octahedral apex (Figure 3.1b) (Velde, 2013). Unlike the tetrahedra, the number of cations can vary between two and three. However, a total positive charge of six is always maintained. When three ions are present, the structure is called tri-octahedral and when two ions are present it is called di-octahedral (Velde, 2013). For example, there could be three  $Mg^{2+}$  ions present or two  $Al^{3+}$  ions in the octahedral sites. These two types of octahedral are fundamental in classifying different clay minerals. Octahedral sheets are formed by sharing all hydroxyl groups with adjacent octahedral (Figure 3.2) (Mitchell and Soga, 2005).



**Figure 3.2:** Tetrahedral sheet (left) and Octahedral sheet (right) (after Murray, 1999).

## 3.4 Classification of clay minerals

Clay minerals occur naturally as a combination of tetrahedral and octahedral sheets. The arrangement of these sheets define the type of clay mineral. They are then further classified based on chemical considerations, such as net charge. Essentially, two combinations occur (Barton, 2002):

- (i) where the ratio of tetrahedral to octahedral sheets is 1:1, and
- (ii) where the ratio of tetrahedral to octahedral sheets is 2:1.

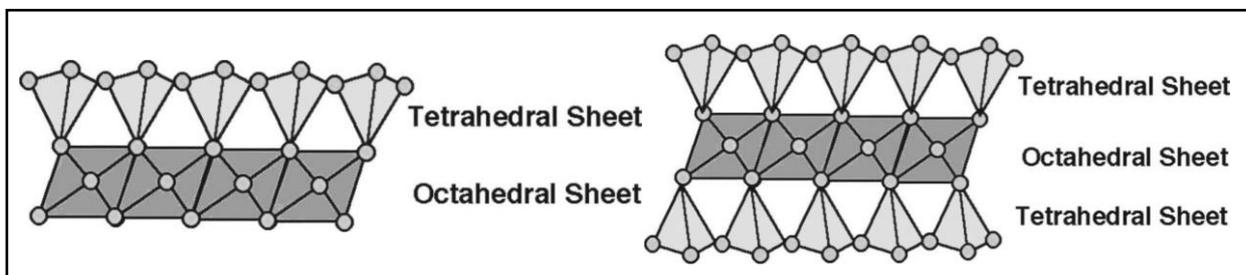
### 3.4.1 (1:1) Clay minerals

The 1:1 clay minerals contain one tetrahedral sheet and one octahedral sheet in their basic structural unit (Figure 3.3). These clay minerals exist in both di- and tri-octahedral variations. Van der Waals bonding occurs between the apical oxygen of the tetrahedral sheet and the octahedral sheet. The layers are held together tightly by hydrogen bonding, which restricts expansion and confines reactive action to the external surfaces only (Barton, 2002). The tetrahedral and octahedral units, when bonded to form a sheet, has a given and constant

thickness. The thickness of the tetrahedral layers is considered to be 3.4 Å (Ångström – a unit of measure equal to  $10^{-10}$  m) or 0.34 nm, whereas the octahedral layers are thinner (Velde, 2013). When one layer is interconnected to another through a shared oxygen atom, the combined thickness of the two will be less than the sum of the two individuals. A 1:1 clay mineral has a total thickness of 7 Å (Velde, 2013). Isomorphous substitution for  $\text{Si}^{4+}$  by  $\text{Al}^{3+}$  in this mineral is negligible. Hence, 1:1 minerals exhibit a low potential for adsorbing cations (Barton, 2002). The Kaolin Group mineral is representative of the 1:1 clay mineral (Bergaya and Lagaly, 2006).

### 3.4.2 (2:1) Clay minerals

In this type of clay mineral, an octahedral sheet is sandwiched between two tetrahedral sheets (Figure 3.3), wherein adjacent sheets are joined through Van der Waals bonds via the apical oxygens of the tetrahedral sheet (Kloprogge *et al.*, 1999; Barton, 2002; Mitchell and Soga, 2005). The clay mineral has a combined layer thickness of 10 Å (Velde, 2013). Ionic substitution is common and hence gives rise to varying minerals of diverse physicochemical properties. Expandable 2:1 clay minerals exhibit a similar layer structure to other mineral of the same category. However, 2:1 clay minerals vary widely in-layer charge and interlayer spacing due to the presence of weakly bonded cations, water, or polar organic molecules within their interlayers (Barton, 2002). Smectites generally refer to a group of expandable 2:1 minerals with a low charge. Montmorillonite, the most common member of this group, derives its charge from the octahedral substitution of  $\text{Mg}^{2+}$  for  $\text{Al}^{3+}$  (Barton, 2002). Bentonite is a combination of predominantly montmorillonite and other clay minerals like hectorite as well as impurities such as quartz, plagioclase and muscovite (Gray *et al.*, 2013).



**Figure 3.3:** Clay minerals of 1:1 sheet (left) and 2:1 sheet (right) (after Al-Ani and Sarapää, 2008).

### 3.5 Smectite minerals

Smectite is a 2:1 clay mineral, with two tetrahedral sheets and a central octahedral sheet with an average size of 0.5  $\mu\text{m}$  (Grim and Guven, 1978; Odom, 1984). The low charged structure permits hydrated ions or polar ions to be inserted between the layers (absorbed), increasing the interlayer distance leading to expansion or swelling (Murray, 2006; Velde, 2013). Moreover, a layer charge of 0.7 to 0.2 allows the layers to absorb hydrated cations and polar molecules between the 10 Å sheets (Mitchell and Soga, 2005; Velde, 2013). Bonding between consecutive layers is achieved by Van der Waals forces and by cations that balance charge deficiencies in the structure. These bonds are weak and easily separated through hydrous adsorption or other polar liquids (Mitchell and Soga, 2005).

The theoretical composition, of a smectitic mineral, in the absence of isomorphous substitutions is  $(\text{OH})_4\text{Si}_8\text{Al}_4\text{O}_{20} \cdot n \text{H}_2\text{O}$  (where  $n$  denotes the number of water molecules within the interlayer). However, in most smectites, there is substantial isomorphous substitution for silicon and aluminium by other cations. Aluminium in the octahedral sheet could be replaced by magnesium, iron, zinc, nickel, lithium, or other cations. Aluminium may replace about 15 % of the silicon ions in the tetrahedral sheet. Some of the silicon positions could be occupied by phosphorous (Grim, 1968; Lee and Shackelford, 2005; Mitchell and Soga, 2005). The cation exchange capacity (CEC) of relatively pure smectite can range between 70 and 130 meq/100 g. Majority of the CEC is due to charges resulting from structural substitution.  $\text{Na}^+$  is readily replaced by  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , hence leaching conditions would deplete  $\text{Na}^+$  (Odom, 1984; Lee and Shackelford, 2005).

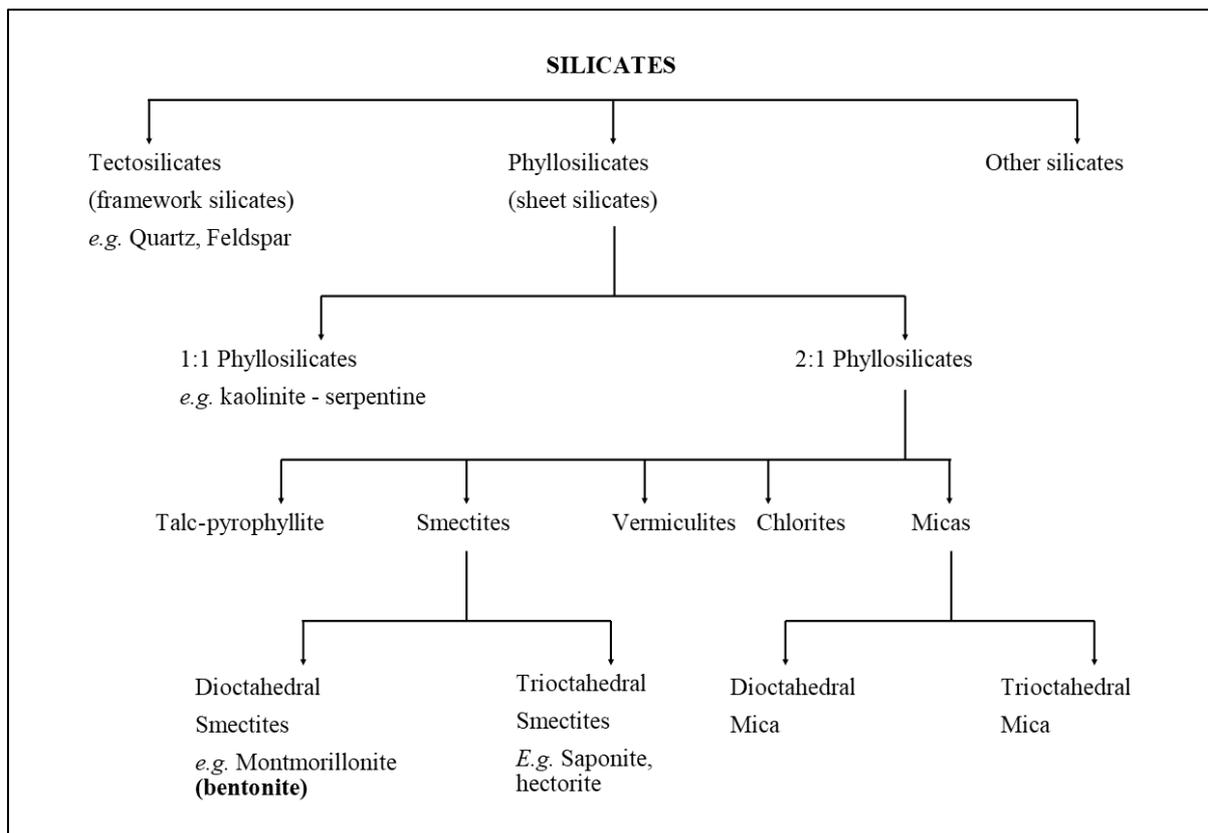
The most common smectite mineral is montmorillonite, which exists in two forms namely, Na-montmorillonite and Ca-montmorillonite. The layer charge deficiency of montmorillonite is balanced by the interlayer cation calcium or sodium (Al-Ani and Sarapää, 2008). Since bentonite is largely montmorillonite, bentonite also exists in two forms namely, sodium bentonite and calcium bentonite.

Bentonites predominantly comprise either Na-montmorillonite or Ca-montmorillonite and to a much lesser extent saponite and hectorite. These smectite (Figure 3.4) minerals that comprise bentonites have significantly differing physicochemical properties which govern their utility to a major degree (Murray, 1999).

### 3.6 Bentonites

The name ‘bentonite’ is often loosely used to denote any deposit of a smectite-rich mineral (Gates and Churchman, 2006). Bentonites constitute a rock type rather than a mineral. True bentonites are alteration products of volcanic ash deposited into shallow marine environments (Gates and Churchman, 2006). However, smectite is the major constituent in addition to other minerals such as quartz. The smectite in bentonites is primarily montmorillonite (Figure 3.4). Murray (1999) mentions two definitions of bentonite; defined by Ross and Shannon (1926) as a clay altered from glassy igneous material, usually a tuff or volcanic ash and redefined by Grim and Guven (1978) as any clay predominantly composed of a smectite mineral, regardless of its origin.

In this research, bentonites tested were volcanic ash deposited in a lacustrine environment and hence the definition by Ross and Shannon (1926) will apply.



**Figure 3.4:** Classification of silicates (after Al-Ani and Sarapää, 2008).

Bentonite was discovered in the late 1880’s near Fort Benton in Wyoming (Von Maubeuge, 2002). This magical soil termed “clay of a thousand uses” was first called ‘Taylorite’ after William Taylor, the first commercial producer of this clay. Later, the name was changed to bentonite after realising the initial name was taken (Von Maubeuge, 2002). Presently, bentonite

is often loosely used to denote any deposit of a smectite-rich mineral. The term bentonite is now well established for any clay that is composed predominantly of a smectite clay mineral, and whose physical properties are controlled by this clay mineral (Gates and Churchman, 2006). There are two types of bentonite: sodium bentonite and calcium bentonite, with the prefix denoting the predominant exchangeable ion. Sodium bentonite is used over calcium bentonite due to its better swelling properties. The primary constituent of sodium bentonite, which contributes to swelling, is montmorillonite (Norrish, 1954). Generally, the typical sodium bentonite used in GCLs will contain 60–85 % montmorillonite (Von Maubeuge, 2002).

### 3.6.1 Uses of bentonite

The uses of bentonite are numerous and thus, the demand is colossal. Table 3.2 shows a summary of some of the many uses of bentonite.

**Table 3.2:** Industrial uses of bentonites (after Grim and Guven, 1978).

drilling muds	medical formulations	crayons
foundry bondants	polishing and cleaning agents	cement
pelletising ores	detergents	desiccants
civil engineering/sealing	pharmaceutics	cosmetics
animal feed bondants	food additives	paint
decolourising edible oils and fats	adhesives	paper
industrial adsorbants (oils)	plastics	fillers
agricultural carriers	de-inking of paper	ceramics
absorbents (pet litter)	tape joint compounds	catalysts
beer, wine and liquor clarification	emulsion stabiliser	pencil lead

The primary uses of bentonites are in drilling muds, iron ore foundries, water impoundment and landfill liners (Figure 3.5), applied as Geosynthetic Clay Liners (GCLs) at the base of

landfills and as a capping. High swelling sodium bentonite will expand up to ten or more times their clay volume when subjected to hydration (Murray, 1999). This unique swelling property makes them necessary ingredients for GCLs. The swelling bentonite prevents flow of water through earthen structures such as dams, inhibits seepage of water from ponds and irrigation channels, and contains chemicals in landfills and in toxic waste impoundments (Murray, 1999). Furthermore, their thixotropic properties (the tendency to become less viscous when agitated) are suitable in bored pile construction applications. Calcium bentonites are used as animal feed bonds, act as absorbents for bacteria and certain enzymes. Granular bentonite is deodorised and treated for use as pet waste absorbent (Murray, 1999). The uses are extensive, hence the reason for its importance in industry.



**Figure 3.5:** Landfill with GCL lining (D&E Construction, 2011).

### **3.6.2 Particle size**

Clays are fine grained whilst those containing smectite clay minerals have extraordinary fineness (Pusch, 2015). Often, the particle size distribution is indicative of its smectite content. If the particle size is greater than 1  $\mu\text{m}$ , there is a good probability that other minerals are present as impurities (Pusch, 2015). Most smectites found in bentonites are classified as clay, which means their particle size is less than 2  $\mu\text{m}$  and can be as small as 0.2  $\mu\text{m}$  (Odom, 1984). The particle size is an important characteristic as it greatly influences the physicochemical properties of bentonites. The specific surface area, acidity and amphoteric character, all increase with decreasing mean particle size (Gates and Churchman, 2006). Many of the

physical characteristics for which bentonites are known, such as swellability and plasticity, are improved with decreasing particle size. In powder form, decreased particle size will enhance the ability of a clay to seal when hydrated and speed up the formation of a seal to water movement (Gates and Churchman, 2006). Naturally occurring sodium bentonite yields the smallest effective particle size and largest surface area making it most suitable for GCL applications (Odom, 1984).

### **3.6.3 Plasticity index**

Factors that affect clay fabric and rheology also define the plasticity of the bentonite. These are clay type, exchange cation identity and solution properties. Three moisture content limits are used to differentiate the plastic state of clay. The liquid limit, the upper limit, is the water content at which clays change from a plastic to a viscous semi-liquid. Liquid contents beyond the liquid limit will produce a non-malleable clay that cannot hold its form. The plastic limit is the water content that defines the lower limit at which a clay will display plastic behaviour. Water contents below this will yield a clay that is friable. The shrinkage limit is the water content at which no volume change is recorded with a continued loss in moisture. The plasticity index is the range between the plastic and liquid limits (Grim and Guven, 1978; Gates and Churchman, 2006; Al-Ani and Sarapää, 2008; Craig, 2013). It is reported that bentonite with a high plasticity index (~550 %) has a higher permeability to solutions of CaCl<sub>2</sub> than one composed of a bentonite with a lower plasticity index (~400 %) (Gates and Churchman, 2006).

### **3.6.4 Chemistry of bentonites**

Bentonites very rarely exist in its pure form. More often, impurities diminish the value for different purposes. The usefulness of bentonite is derived from the smectite content (Lee and Shackelford, 2005). The most dominant mineral impurities in bentonites are volcanic glass, opal, cristobalite, tridymite, quartz, mica, feldspar and interstratifications of illite-smectite and kaolinite-smectite (Gates and Churchman, 2006; Christidis and Huff, 2009). Gates *et al.* (2002) found impurities such as quartz, feldspar, cristobalite/opal, as well as minor amounts of clinoptilolite, mica/illite, gypsum and anatase in a bentonite from Australia. Majority of the bentonite deposits occur as Ca<sup>2+</sup> and Mg<sup>2+</sup> forms. It is rare to find Na<sup>+</sup> saturated form. Table 3.3 shows variation in composition of some well-known bentonite deposits around the world. The analyses are presented in terms of the percentage of major element oxides present.

**Table 3.3:** Composition of bentonite deposits around the world (after Gates and Churchman, 2006).

	% oxide							
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	Li <sub>2</sub> O
<b>Diocahedral smectites</b>								
Otay, <i>California</i>	67.72	19.36	1.28	7.321	4.087	0.062	0.01	-
Cheto, <i>Arizona</i>	67.59	19.85	1.71	6.508	3.919	0.118	0.074	-
JC Lane, <i>Wyoming</i>	67.78	22.70	4.70	2.490	2.860	0.060	0.030	-
Arumpo 130E, <i>New South Wales</i>	66.45	20.56	4.13	4.778	3.181	0.00	0.380	-
Arumpo, 'pure' <i>New South Wales</i>	66.65	19.69	4.56	4.798	2.724	0.00	0.758	-
Surrey, <i>United Kingdom</i>	65.05	18.40	8.45	4.119	3.143	0.029	0.104	-
Upton <i>Wyoming</i> (SWy)	65.56	23.72	4.93	2.523	2.937	0.027	0.047	-
Upton, <i>Wyoming</i>	64.90	24.05	4.95	2.920	3.098	0.090	-	-
Ossean, <i>South Africa</i>	65.92	22.55	3.03	4.856	3.699	0.036	-	-
Miles, <i>Queensland</i>	67.16	24.19	6.02	2.625	2.904	0.038	0.029	-
CE150, Miles, <i>Queensland</i>	64.26	22.26	5.67	3.797	3.569	0.033	0.026	-
Mt Binjour <i>Queensland</i>	63.77	26.25	1.71	3.868	4.052	0.074	0.016	-
DeLamar	62.11	32.09	0.50	1.330	3.298	0.009	0.711	-
Silver City, <i>Idaho</i>	60.77	31.99	2.02	0.859	3.012	0.015	0.660	-
<b>Triocahedral smectites</b>								
Hector, <i>California</i>	64.15	0.95	0.32	28.36	2.851	-		2.399
Hector, <i>California</i>	64.41	0.55	0.22	27.79	3.172			3.863
Burro Creek, <i>Arizona</i>	60.26	7.18	0.46*	27.82	3.429	0.075	0.017	-
Lake E, <i>West Australia</i>	58.73	6.17	2.47	29.28	2.730	0.084	0.121	-
Ballarat, <i>California</i>	59.05	5.31	0.98	31.68	2.980	0.04	0.004	-

\*The upper section of the table lists diocahedral smectites with chemical analyses ranging from the end members montmorillonite to beidellite. The lower section lists some triocahedral hectorites and saponites

Other elements, such as nickel, copper, titanium, zinc and cobalt, are present in trace amounts in most bentonites. These do not significantly impact on the chemistry. Most montmorillonitic bentonites will provide analyses within the ranges listed in Table 3.3 (Gates and Churchman, 2006).

In addition to particle size, relating to surface area, layer charge has a significant impact on chemical reactivity. These net charges originate from imbalanced compositions. Often, substituting cations have a lower charge resulting in a net negative charge (Lee and Shackelford, 2005).

Layer charge is directly responsible for several properties such as cation exchange capacity (CEC) and charge potential at layer surfaces. Layer charge moreover positively affects adsorption but negatively affects swelling and viscosity (Gates and Churchman, 2006).

#### **3.6.4.1 Cation exchange**

The net negative charge permits clay minerals to attract cations from solution provided normal pH conditions exist. The number of cations they attract per unit weight is directly related to their negative charge. Therefore, negative charge is commonly expressed as cation exchange capacity (CEC) (Gates, 2004).

Layer charge and CEC are identical for low charge smectites when the molecules carry no net electric charge (isoelectric point). The CEC can be lower than the layer charge, particularly in high charge smectites. The CEC for almost pure smectite ranges between 70 and 130 meq/100 g. About 80 % of the CEC is due to charges resulting from structural substitution and 20 % from broken bonds at crystal boundaries (Odom, 1984). The exchangeable ions are easily replaced,  $\text{Na}^+$  is readily replaced by  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , hence leached smectites are depleted of  $\text{Na}^+$  (Ross and Shannon, 1926; Odom, 1984).

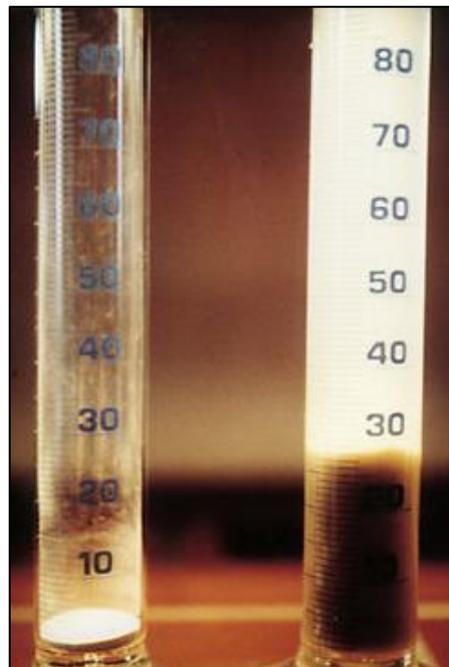
Exchangeable ions themselves play a significant role in determining the commercial use of smectitic bentonite. Sodium rich smectites tend to have a high swelling capacity whilst Na-leached smectites display a remarkably reduced degree of swelling. It is therefore incumbent to thoroughly study exchangeable ion content of commercial bentonite as serious consequences, such as a high hydraulic conductivity, could result. In general, a smectite with a high layer charge ( $> 0.85 e^-$  or  $> 110 \text{ meq } 100 \text{ g}^{-1}$ ) provides high contaminant adsorption and retention yet low crystalline and bulk swelling (Gates and Churchman, 2006).

### 3.6.5 Swelling of bentonite

Swelling occurs when water enters between clay particles resulting in an increase in volume. Smectites generally swell differently from each other based on their physicochemical characteristics of attraction and repulsion, between clay particles and between clay and the solution within the interlayer space. Two types of swelling are described; two-dimensional (crystalline) swelling and three-dimensional (bulk) swelling (Norrish and Quirk, 1954).

Two-dimensional swelling arises when a polar solvent enters interlayer space between clay particles thereby causing a separation. Bentonite predominantly comprises  $\text{Li}^+$ - or  $\text{Na}^+$ -smectite will exhibit unlimited swelling when taking up water provided the solution electrolyte concentration is very low. This occurs when repulsive forces dominate attractive forces.  $\text{Ca}^{2+}$ -smectite will display limited swelling. This means that while interlayer cations can collect water, forces of attraction outweigh the repulsive force necessary for the swelling to continue (Norrish, 1954).

The swell index (Figure 3.6) is a measure of volume change a unit mass of bentonite undergoes upon absorption of a liquid (Gates and Churchman, 2006), which measures bulk swelling in *ml* per 2 g of bentonite. Typical bentonite swelling index values range from 4 to 50 ml/2g and it depends on the exchange cation, the degree of dispersion and whether its initial fabric has been disrupted (Gates and Churchman, 2006).



**Figure 3.6:** Bentonite before swell (left) and after swell (right) (after Mudavath, 2018).

### 3.6.5.1 Attractive forces

Electrostatic forces of attraction between the exchange cations and the layer of smectites is the major force that inhibits clay swelling. Layer charge influences the degree of attraction. Higher layer charge results in greater electrostatic forces of attraction (Komine and Ogata, 1996; Gates and Churchman, 2006). This further results in a more densely packed layer of surface cations. Thus, bentonite comprising clay minerals of higher layer charge will certainly display limited swelling. Additionally, the solvent ionic strength influences clay swelling. Solvents of high ionic strength or containing divalent counter-ions (like  $\text{Ca}^{2+}$ ) inhibit clay swelling, whereas low ionic strength or monovalent counter-ions (like  $\text{Na}^+$ ) promote swelling (Komine, 2004; Gates and Churchman, 2006). Thus, as explained earlier, the reason Na-leached bentonite displays a remarkably reduced degree of swelling.

### 3.6.5.2 Repulsive forces

Repulsive forces promote clay swelling. Ions such as  $\text{Li}^+$  and  $\text{Na}^+$  possess high hydration potential. These small cations have a higher hydration energy and thus have a greater tendency to be hydrated. This hydration of the singular ion plus a small valency, significantly reduces the electrostatic forces of attraction (Komine and Ogata, 1996; Gates and Churchman, 2006). The volume of water adsorbed are governed correspondingly by the size and charge of the saturating cation, as well as by the value and localization or distribution of the charge of the adjacent silicate sheets that the exchangeable cations neutralize (Cases *et al.*, 1997).  $\text{Na}^+$ -bentonites possess the ability to rehydrate, whereas  $\text{K}^+$ -bentonite will not rehydrate (Gates and Churchman, 2006). Small divalent cations also possess high hydration energy and their attraction to the clay surface is greater than a monovalent cation, hence exhibit limited swelling (Gates and Churchman, 2006).

### 3.6.5.3 Factors affecting clay swelling

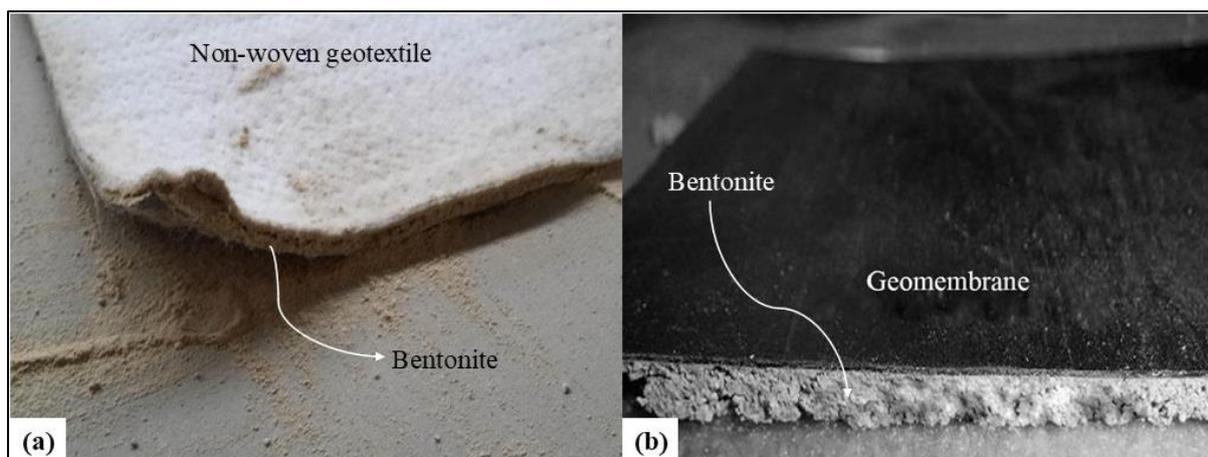
Gates and Churchman (2006) summarise 4 factors that affect clay swelling, which are discussed below in order of most to least importance. These factors are based on the works of several researchers (e.g. Mooney *et al.*, 1952; Norrish, 1954; Emerson, 1962; Posner and Quirk, 1964; Suquet *et al.*, 1977; Low, 1980; Low, 1987; Low, 1992 ):

- i. Electrostatic attraction vs. hydration-energy
  - If hydration energy of the exchange cation is large, and electrostatic attraction is low, then the repulsive force of cation hydration will overcome the electrostatic attractive force then crystalline swelling will occur.

- If the electrostatic attraction is large, such as having a high charge per unit cell, then crystalline swelling will be suppressed.
- ii. Layer charge
- High charged smectites with layer charge existing predominantly within the octahedral sheet tend to swell more than those with layer charge originating within the tetrahedral sheet.
  - Low charged smectites tend to experience greater crystalline swelling than high charge smectites. This is mostly independent on the location of layer charge.
- iii. Cation identity
- For monovalent cations, the ability to influence crystalline swelling decreases in the order: lithium ( $\text{Li}^+$ ) > sodium ( $\text{Na}^+$ ) >> potassium ( $\text{K}^+$ ) > rubidium ( $\text{Rb}^+$ ) > caesium ( $\text{Cs}^+$ ).
  - For divalent cations, the ability to induce crystalline swelling decreases in the order: magnesium ( $\text{Mg}^{2+}$ ) = calcium ( $\text{Ca}^{2+}$ ) > strontium ( $\text{Sr}^{2+}$ ) > barium ( $\text{Ba}^{2+}$ ).
- iv. Electrolyte concentration
- The ability of a smectite to experience crystalline swelling decreases with increasing electrolyte concentration, and vice versa.
  - Electrolyte solutions composed of multi- and divalent ions suppress swelling more than solutions composed of monovalent ions.

### 3.7 Geosynthetic Clay Liners (GCLs)

Geosynthetic Clay Liners (GCLs) comprise a thin layer of either sodium bentonite or calcium bentonite, bonded between a geomembrane or geotextile (Figure 3.7)(Bouazza, 2002). Those using geotextiles sandwich the bentonite by needle-punching, stitching or by using a non-polluting adhesive (Bouazza, 2002). Needle-punched GCLs comprise an encasing nonwoven filament geotextile where the needle punching process pierces fibres from the upper geotextile to the bottom geotextile (Kong *et al.*, 2017). This process entangles the fibres to the bottom geotextile bonding the sheathing layers together. Bonding may also be achieved through heating, causing the piercing geotextile to fuse to the bottom geotextile. The stitching method involves sewing the geotextiles together using stitching bonded yarns (Kong *et al.*, 2017). Geomembrane supported GCLs use non-polluting adhesives to bond the bentonite to a geomembrane.



**Figure 3.7:** Different types of GCLs. (a) Needle punched non-woven geotextile GCL. (b) Geomembrane supported GCL (after Kong *et al.*, 2017).

Though there are many types of GCLs, the fundamental difference and most importantly is the type of bentonite used (Bouazza, 2002). The bentonites used in GCLs can either be granular or powdered and sodium or calcium. The main advantages of GCLs are limited thickness, good endurance to differential settlements of underlying soil or waste, simple installation and low cost (Bouazza, 2002). The advantages and disadvantages are summarised in Table 3.4.

**Table 3.4:** Advantages and disadvantages of GCLs (after Bouazza, 2002).

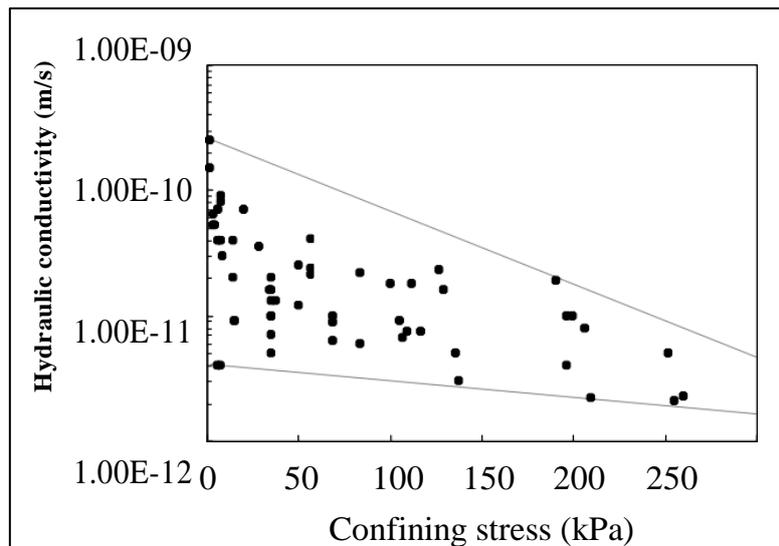
Advantages	Disadvantages
Quick installation. Low cost and low skilled labour. Low hydraulic conductivity. Excellent self-healing characteristics of small punctures. Resistance to the effects of freeze-thaw cycles. More airspace resulting from the smaller thickness. Field hydraulic conductivity testing not required. Hydrated GCL is an effective gas barrier. Reduce overburden stress.	Low shear strength of hydrated bentonite GCLs can be punctured during or after installation. Possible loss of bentonite during placement. Potential strength problems at interfaces with other materials. Possible increase of hydraulic conductivity due to compatibility problems with leachate if not pre-hydrated with water. Prone to ion exchange. Prone to drying if not properly covered.

### 3.7.1 Hydraulic conductivity of GCLs

The hydraulic conductivity is the most critical parameter that defines the effectiveness of GCLs (Kong *et al.*, 2017). Perceptibly, the hydraulic barrier is created by the bentonite within the

GCL. However, in geomembrane GCLs, the membrane also contributes to hydraulic retardation. Generally, hydraulic conductivities of geotextile GCLs measured in the laboratory of different water types range between  $2 \times 10^{-12}$  m/s and  $2 \times 10^{-10}$  m/s (Bouazza, 2002). Hydraulic conductivity also largely depends on the applied confining stress, as illustrated in Figure 3.8 (Bouazza, 2002).

GCLs are frequently used to contain liquids or leachates in addition to water. These leachates may have a very different effect on the hydraulic conductivity of GCLs. Compatibility testing, where the specimen is permeated with a sample of liquid to be contained, is vital prior to installation. Other features of GCLs that effect their hydraulic conductivity with liquids besides water are aggregate size, content of montmorillonite, thickness of adsorbed layer, pre-hydration and void ratio of the mineral component. Conversely, the main factors related to the liquid that influence the hydraulic conductivity are concentration of monovalent and divalent cations (Bouazza, 2002).



**Figure 3.8:** Variation of hydraulic conductivity versus confining stress (after Bouazza, 2002).

Hydraulic conductivity tests for various applications are generally tested in flexible wall parameters with regard to liquids deviating from the norm (Liu *et al.*, 2014). However, the flexible wall parameter test is time consuming hence the Fluid Loss test method evaluates the ability of bentonite in suspension to form a filter cake (i.e. a hydraulic barrier). Low fluid loss over a certain time will surely imply low hydraulic conductivity. It was found that with increasing fluid loss values the hydraulic conductivity increased (Liu *et al.*, 2014).

## **3.7.2 Problems associated with GCLs**

### **3.7.2.1 Puncturing**

GCLs may suffer lacerations during installation or from root encroachment or from wet-dry cycles leading to desiccation and cracking (Kong *et al.*, 2017). The obvious result is that it would compromise the hydraulic performance of the barrier by allowing a path for fluid flow. However, it has been observed that minor punctures can be effectively sealed by the swelling bentonite with inconsiderable compromise to the hydraulic conductivity as compared to an intact specimen (Bouazza, 2002). Lin and Benson (2000) found that ion exchange where calcium replaced sodium in the exchange complex, resulted in a decreased swelling capacity close to that of typical Ca-bentonite.

In general, the swelling capacity equates to the healing capacity and factors that affect swelling would affect healing as well. The only factor contributing to self-healing capacity is the amount of bentonite or smectite present to effectively heal a hole, provided it is less than 30 mm in diameter (Rowe and Li, 2016).

### **3.7.2.2 Thinning and piping**

The performance of a GCL depends to an extent on the thickness or mass of bentonite or the distribution of mass per area (Bouazza, 2002). The concern is notable as hydrated bentonite exhibits very low shear strength hence can expect lateral squeezing when loaded (Koerner and Narejo, 1995). A simple solution to this problem is prior to hydration, an adequate backfill of suitable thickness and particle size should be placed over a GCL (Koerner and Narejo, 1995; Bouazza, 2002). The subgrade on which the bentonite lies is as important as the cover soil. It should be suitable with respect to particle size and thickness (Bouazza, 2002).

Another issue encountered is internal erosion of the bentonite. The process of internal erosion involves the migration of fines due to the presence of a high hydraulic gradient typically common in fluid containment facilities (Bouazza, 2002). Rowe and Orsini (2003) unequivocally pointed out that *“The presence of large hydraulic gradients combined with clay soils that may be inadequately filtered creates the potential for internal erosion and possible hydraulic failure of the liner”*.

Rowe and Orsini (2003) further mentioned that GCLs possess the potential to display dispersive behaviour. High hydraulic gradients can cause internal erosion within the GCL. The problem is most severe when the gradients are capable of moving soil particles and is coupled with a situation where the repulsive forces between the clay particles exceed attractive forces. This causes dispersion of clay particles, which then enter into suspension, and is carried away

by flowing water. GCLs have the potential to exhibit this behaviour. Loss of bentonite from the GCL core by this mechanism may increase the hydraulic conductivity of the liner (Rowe and Orsini, 2003). This would increase the hydraulic conductivity, instantaneously defeating its purpose. Stark (1998) discussed migration of bentonite in GCLs due to the presence of wrinkles in an overlying geomembrane. The wrinkles create air pockets, which can cause hydrated bentonite to migrate into the airspace under the wrinkle. Fox *et al.* (1998a) conducted a field study wherein they assessed installation damage with varying GCL products, cover soil type, cover soil thickness, bulldozer type and number of times driven over by bulldozers, over the installed GCL after hydration. They concluded that significant damage resulting in bentonite migration can occur for soil cover thickness less than 305 mm and that significant migration can occur with increasing cover soil particle size and rate of loading. Giroud and Soderman (2000) proposed a limit of 10 g/m<sup>2</sup> for tolerable bentonite migration into a geonet drainage layer.

### **3.7.2.3 Gas migration**

Waste containment facilities tend to exude substantial amount of gasses. The migration of these gasses has gained a lot of professional interest. The migration of these gasses occurs by two major transport mechanisms: advective and diffusive flow (Vangpaisal and Bouazza, 2004). In advective flow, gasses migrate in response to a total pressure differential. To equalise pressure gradients, gasses migrate from high pressure to low pressure. In the context of landfill containment facilities, the primary cause for gas migration is pressure differentials caused by natural atmospheric pressure fluctuations (Bouazza, 2002). Low atmospheric pressure will cause gas to migrate out of the system increasing gaseous concentration near the surface whilst high atmospheric pressure will force air into the landfill thereby diluting near surface gasses (Vangpaisal and Bouazza, 2004). Other factors such as a change in water table or temperature can also cause pressure differences resulting in gas migration. Uncontrolled gas migration and monitoring can lead to catastrophic events. Williams and Aitkenhead (1991) reported that an explosion was caused by undetected methane gas migration from a landfill site in Loscoe, UK in March 1986. The explosion, although 70 m from the landfill, was caused by gas migration through geologic pathways.

Another concern related to gas migration is that the accumulation of gas could gradually increase positive pressures beneath a GCL (Vangpaisal and Bouazza, 2004). The positive pressures beneath the GCL may reduce the interface shear strength between the GCL and underlying soil layer due to insufficient normal forces acting on the GCL. This may lead to

slope failure. At present, gas migration has been identified as a design problem for landfill covers. Thus, Thiel (1999) put forward a methodology to design for gas migration. The methodology incorporates knowledge of the gas transmissivity of a chosen medium to design a spacing for highly permeable strip drains. The strip drains would discharge the gas either to vents or an active gas collection system. The gas relief layer typically consists of sand or a geonet composite. Less disastrous movement is gas movement by diffusion, where gasses diffuse into a lesser concentrated area. Thus, molecules migrate in response to a partial pressure gradient or concentration gradient of the gas (Vangpaisal and Bouazza, 2004).

#### **3.7.2.4 Slope stability**

Besides using GCLs as hydraulic barriers, their installation in conjunction with other materials is required to be stable. The GCL layer could be a plane of weakness and low shear strength. Slope failure can easily result along the GCL plane. GCLs are very weak compared to other materials and exhibit significant loss of shear strength with displacement. It is difficult to entirely quantify the shear strength of GCLs because they contain different component interacting with each other. The surrounding soil or material interacts with the GCL on either side and the internal component of the GCL interacting with each other (Bouazza, 2002).

Non-reinforced GCLs display a much lower shear strength than reinforced (needle-punched) GCLs. This is so because the interlocking threads increase the internal shear strength of the bentonite thereby increasing the shear resistance (Bouazza, 2002; Chiu and Fox, 2004). Studies conducted by Fox *et al.* (1998b) showed that failures generally occur at the geotextile bentonite interface. These failures will rupture the stitching or rip out the fibres of reinforced GCLs.

Bouazza and Bowders Jr (2009) mentioned four steps for successful design of slopes containing GCLs:

1. Define the geometry, loading conditions and consequences of a failure of the slope during construction, operation and after completion.
2. Select appropriate material properties for the GCL and all other materials in the slope. Consider rate of loading, deformations, normal stresses and fluid pressures.
3. Analyse and evaluate slope stability.
4. Take steps to mitigate the slightest of concerns about slope stability.

### 3.7.3 Soda ash treated bentonite

Bentonite used in GCL manufacturing often does not possess the desired properties. Borderline quality bentonites are mixed with soda ash ( $\text{Na}_2\text{CO}_3$ ) to increase the quantity of exchangeable sodium ions. Sodium ions, because of its large hydration radius, is primarily responsible for the sealing ability of a bentonite. This allows a larger volume of water retention thereby increasing swell. Bentonites with the highest swelling potential, highest liquid limit and a low water loss contain at least 60 % exchangeable sodium ions (Alther, 1987).

However, soda ash treated bentonite may degrade considerably over time as compared to natural sodium bentonite (Von Maubeuge, 2002). Investigations carried out by Von Maubeuge (2002) showed the difference in swell index of natural sodium bentonite (Wyoming Bentonite) and sodium activated calcium bentonite after certain time periods. The activated calcium bentonite may degrade considerably faster, through ionic exchanges, than natural sodium bentonite (Von Maubeuge, 2002). The results are shown in Table 3.5.

Although the use of activated calcium bentonite as an alternative can be accepted, it is by far not as effective as natural sodium bentonite. The soda ash activation process of calcium bentonites (and even natural sodium bentonites of lower quality) takes place in the field where a specified amount of soda ash is added to the un-beneficiated bentonite (Von Maubeuge, 2002).

**Table 3.5** Swell index of natural vs. sodium activated bentonite after certain time periods (after Von Maubeuge, 2002).

Time	Swell Index (ml/2 g)	
	Wyoming bentonite	Na <sup>+</sup> activated calcium bentonite
17 months	35	8
19 months	34	7
24 months	32	2
28 months	30	Not tested

Inhomogeneities in exchange can result in regions of higher permeability (Von Maubeuge, 2002). Several researchers (e.g. Alther, 1987; Szabó and Balázs, 2000; Mansour, 2001), have shown that naturally occurring sodium bentonite is considerably more effective than sodium activated calcium bentonite and is more resilient than sodium activated bentonite.

### 3.8 Summary

Clay minerals are defined by their crystal structure and mineral family. They generally possess traits that are similar, such as their size, form, crystallographic structure and behaviour, whether physical or chemical. Their phyllosilicate shape inherently produce a large surface area compared to the volume. The elementary units of most clays are silicon–oxygen tetrahedron and an aluminium–hydroxyl octahedron. The arrangements of these sheets account for the physical and chemical differences. Clay minerals occur as a combination of tetrahedral and octahedral sheets. The arrangement of these sheets define the type of clay mineral. The arrangements are either 1 tetrahedral sheet to 1 octahedral sheet or 2 tetrahedral sheets to 1 octahedral sheet. In the latter combination, the octahedral sheet is sandwiched between two tetrahedral sheets. Smectite is a 2:1 clay mineral with extraordinary properties. They possess a finer particle size compared to other clay minerals. The inherently low charge of smectite permits hydration and swelling.

Bentonite is often loosely used to denote any deposit of a smectite-rich mineral. The smectite in bentonites is primarily montmorillonite. The usefulness of a bentonite is often derived from its smectite content. Impurities in the form of other minerals diminish the quality and swelling ability of a bentonite. Certain inherent properties of smectite, such as CEC and electrostatic forces, affect the swelling potential of a bentonite. Not all bentonites possess exact physicochemical properties, hence will have different swelling abilities. Two types of bentonites exist; sodium bentonite and calcium bentonite. The preferred type for the use in GCLs, is sodium bentonite as it is proven to have a superior swelling ability. The wide ranging uses of bentonite make them a product that is highly in demand. Bentonites are used in GCLs to serve as hydraulic barriers that are commonly applied in landfills and dams. GCLs comprise a thin layer of either sodium bentonite or calcium bentonite, bonded between a geomembrane or geotextile. The most critical parameter that defines the effectiveness of GCLs is its hydraulic conductivity. There are problems associated with GCLs. They may suffer puncturing during installation or from root encroachment. This resultantly leads to a decrease in hydraulic conductivity. Other concerns related to GCLs are; uneven bentonite distribution or piping, gas migration underneath a GCL (particularly in landfill applications) and slope instability. Bentonite often does not possess the desired properties. Borderline quality bentonites are mixed with soda ash ( $\text{Na}_2\text{CO}_3$ ) to increase the quantity of exchangeable sodium ions. This allows a larger volume of water retention thereby increasing swell.

The intention of this research was to exhibit a thorough understanding of bentonite and its suitability. Lack of literature regarding activation processes and how different variables, such as time, affect the quality of activated bentonite indicates a knowledge gap. This research critically examines bentonite and bring to light the requirement for further research. It bridges the gap by introducing the effect of time on the activation of bentonite and opens the door to future research.

# Chapter 4

## Methodology

### 4.1 Introduction

This chapter describes the different tests carried out, in order to partly fulfil the objectives of the research. Prior to the testing of any sample, an extensive literature review was conducted to ensure a thorough understanding of GCLs and its key component, bentonite. Samples of beneficiated and non-beneficiated bentonite were obtained from the Imerys bentonite mine in Heidelberg, Western Cape, South Africa.

The laboratory tests conducted were XRD, swell index, fluid loss, plate water absorption and Atterberg limits. These tests were conducted at different locations. The XRD test was conducted in Pretoria by XRD Analytical & Consulting. The fluid lost test was conducted at the Kaytech Laboratory. The fluid loss test was done by Golder Associates in USA. The plate water absorption test was done at the University of KwaZulu-Natal (UKZN) Engineering Geology laboratory. The Atterberg limits test was initially conducted at UKZN but was also conducted by the eThekweni Soil Laboratory for validation. The swell index, plate water absorption and Atterberg limits test were conducted by the author.

### 4.2 Sample collection and storage

Samples were collected from stockpiles of activated and non-activated bentonite from the Imerys bentonite mine (Figure 1.1 of Chapter 1). Approximately 5 kg of each were packed in thick polyethylene bags and then stored in a controlled laboratory environment at 24°C and 65 % relative humidity. Samples were taken randomly from the storage bags for various tests which include Swell Index (ASTM D 5890), Fluid Loss (ASTM D 5891), Plate Water Absorption (ASTM E 946-92), X-Ray Diffraction and Atterberg Limits.

### 4.3 X-Ray diffraction

Samples of activated and non-activated bentonite powder, 100 % passing through the 75 µm sieve, were used. The samples were prepared by drying 10 g of activated and non-activated bentonite in a laboratory oven at 100°C. The dried bentonite was then crushed with a mortar and pestle and sieved using a 75 µm sieve. Powdered activated bentonite and non-activated bentonite were packed and sealed in a polyethylene bag then shipped to XRD Analytical and Consulting for the analysis.

Less than 2 g of powdered bentonite was placed in the X-Ray Diffraction (XRD) machine where monochromatic X-Rays are radiated in a vacuum, onto the powdered sample. By measuring the angles and intensities of the diffracted X-ray beams, the crystalline phases of the powdered bentonite could be determined (Hanawalt *et al.*, 1986).

The material was prepared for XRD analysis using a back-loading preparation method. It was analysed with a PANalytical Empyrean diffractometer with PIXcel detector and fixed slits with Fe-filtered Co-K $\alpha$  radiation. The phases were identified using X'Pert Highscore plus software. The relative phase amounts (weight %) were estimated using the Rietveld method. The Rietveld analysis is a method that uses a non-linear least squares algorithm to produce a refined profile to the experimental data, in order to quantify the mineralogy (Rietveld, 1967). This is done using specified software.

#### 4.4 Swell index

The swell index test follows the procedure according to the American Society for Testing and Materials (ASTM) standard, (ASTM D 5890 - 2011). The swell index test measures the volume of free swell of a 2 g sample of powdered bentonite in 100 ml reagent, which in this case, was de-ionised water.

The method involved drying about 15 g of bentonite in a laboratory oven at 100°C, to a constant mass for a minimum of 16 hours. The dried sample was then crushed to a fine powder using a mortar and pestle (Figure 4.1). The powdered bentonite was sieved with the 75  $\mu$ m aperture sieve.

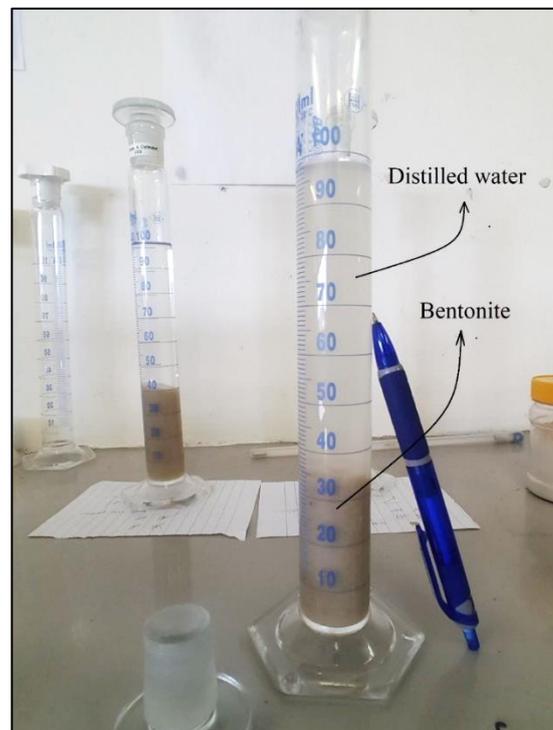


**Figure 4.1:** Mortar and pestle.

Samples passing the 75  $\mu\text{m}$  were used in the swell index tests. A total mass of 2 g of the sieved powdered bentonite was placed in a 100 ml graduated cylinder, filled with 90 ml of distilled water, in 0.1 g increments every 10 minutes. Once the entire 2 g sample was added to the cylinder, the water was then topped to 100 ml and left undisturbed for 16 hours (Figure 4.2). The temperature, pH and electric conductivity of the water were measured before and after each test.

Initial testing of Samples 1, 2 and 3 were conducted. Sample 1 comprised activated, processed (dried and milled) sodium bentonite; Sample 2 comprised unprocessed activated sodium bentonite and Sample 3 comprised unprocessed non activated sodium bentonite. Activation of Samples 1 and 2 occurred at Imerys mine whereby approximately 1 % soda ash was added to the bentonite.

To further investigate the effect of time on the beneficiation process, three samples of bentonite where activated in-house with different amounts of soda ash. Soda ash, obtained from Imerys bentonite mine, was added in specified amounts to non-activated bentonite. Samples A, B and C were mixed with 2 g, 4 g and 6 g of soda ash respectively, resulting in ratios of soda ash to bentonite of 1:50, 1:25 and 1:16 correspondingly. Swell Index tests were conducted at specific time intervals subsequent to activation. Each sample was tested within 24 hours and after 1 week, 2 weeks and 3 weeks, respectively.



**Figure 4.2:** Swell index test showing bentonite and distilled water in a 100 ml graduated cylinder.

## 4.5 Fluid loss

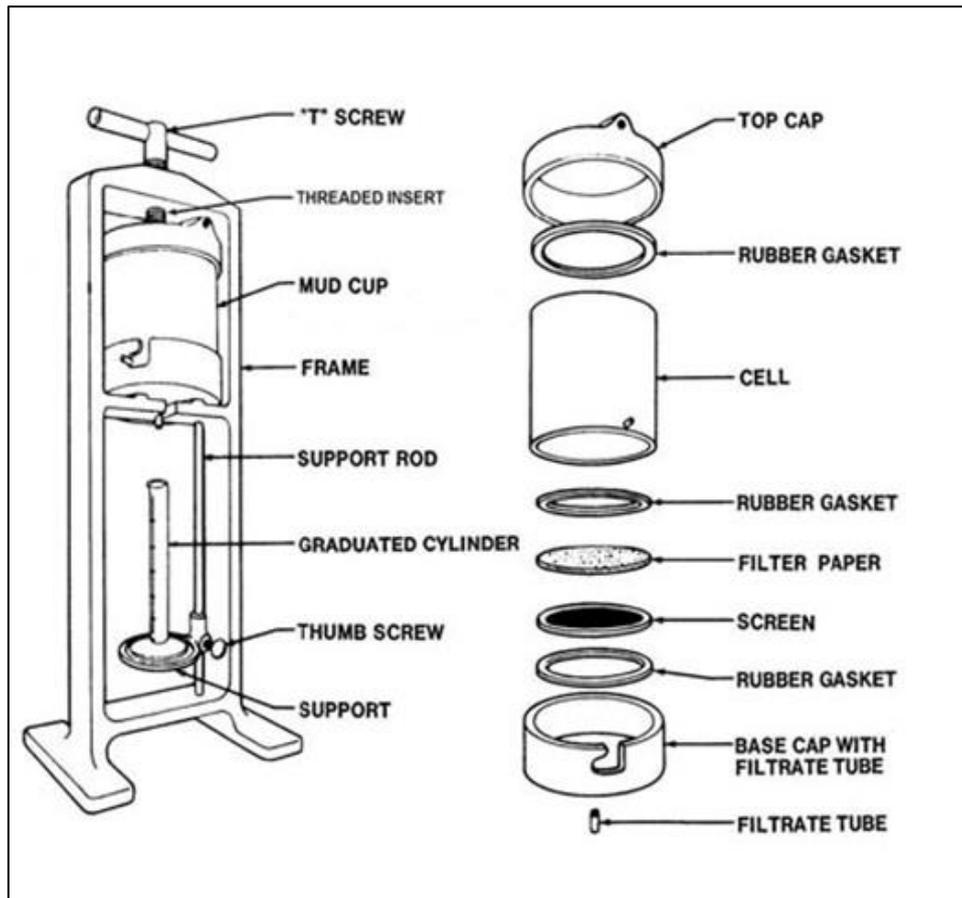
The tests were conducted by Golder Laboratories in the United States since there are no accredited laboratories in South Africa equipped to conduct the fluid loss test. For this reason, it was not feasible to conduct many tests. It was only feasible to conduct three test each, for activated and non-activated bentonites.

The fluid loss test was done in accordance with the ASTM (D 5891). It involves preparing 50 g of bentonite by drying in a laboratory oven, at 100°C for at least 16 hours, and milling to a powder, in this case, 100 % passing through the 75 µm sieve. A total mass of 22.5 g of this powdered bentonite was added to 350 ml of deionised water. It was then stirred on an automatic mechanical mixer. The mixer is designed such that it is capable of 11 000 revolutions per minute. The impeller must be sinuous shaped with an approximate diameter of 25 mm and a minimum weight of 5.1 g. The clay suspension was then mixed for a total of 20 minutes. Thereafter, the clay mixture was stored in a sealed container and left to rest for a minimum of 16 hours.

After aging (i.e. the process of leaving the bentonite overnight in a sealed container), the clay slurry is mixed again for an additional 5 minutes to break the gel strength. The fluid loss cell (Figure 4.3) assembly is prepared in the interim. Filter paper and rubber gaskets are added to the filter cell to prevent leakage. The bentonite slurry is added to the fluid loss cell and sealed. Air pressure of 700 kPa is applied to the sealed cell. Water draining from the filtrate tube is collected in a 10 ml graduated cylinder. After 7.5 minutes, any liquid in the 10 ml graduated cylinder is discarded. The liquid draining through the filtrate tube is collected for the next 22.5 minutes in a new graduated cylinder. The volume retained in the second graduated cylinder is recorded. The fluid loss is calculated as follows:

$$\text{Fluid loss} = 2 \times (\text{ml filtrate volume for the last 22.5 min}) \text{ ml} \quad (4.1)$$

Which simply means 2 times the amount of water collected in 22.5 minutes after the cell has been given 7.5 minutes to equilibrate.



**Figure 4.3:** Fluid loss cell set up (after Fadairo *et al.*, 2012).

## 4.6 Plate water absorption test

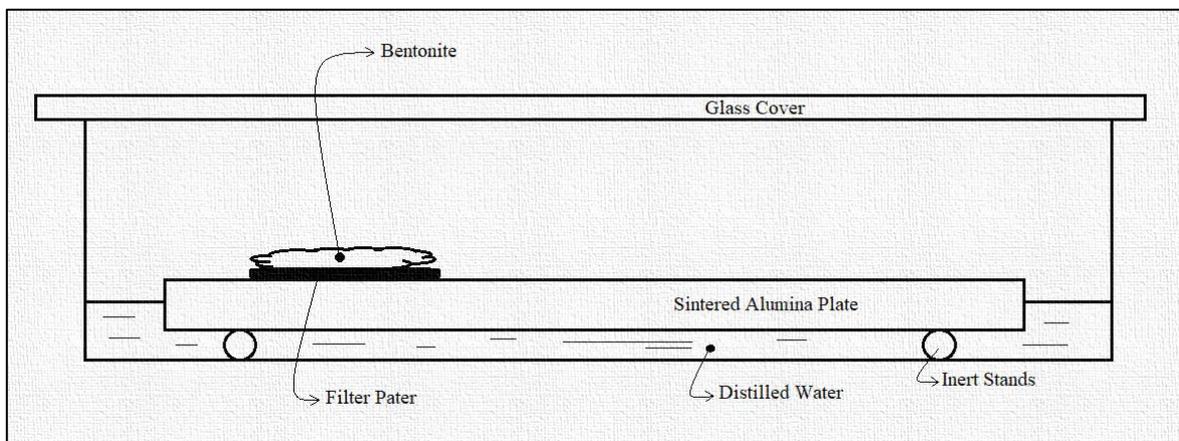
The plate water absorption test was designed to assess the binding ability of bentonite clay binders for iron ore palletisation (McDonald and Kawatra, 2017). The procedure originated from the ASTM E946-92 testing method, which was initially intended to test the absorption of dried bentonite over a specific period of time. Balling or pelletizing is a process where iron ore is processed into a pellet or ball. Bentonite acts as the binding agent in the production of iron ore pellets (Sastry and Fuerstenau, 1973).

In this case as well, the bentonite is required to have a specific absorption capacity. However, this method has been receded as McDonald and Kawatra (2017) pointed out that Plate Water Absorption (PWA) values did not correlate well with the overall pellet quality as the water used in the PWA test is distilled and in an operating pellet plant it is not. Due to this phenomenon, the published standard was withdrawn by ASTM in 1997. However, the PWA value is still used as a measurement metric when pelletizing facilities purchase bentonite.

Nevertheless, for this research, the PWA test was used to determine the absorption capacity of bentonite and to determine whether the PWA test has any correlation with fluid loss, swell index or Atterberg Limits.

The PWA test involves partially submerging a sintered alumina plate in distilled water in a bath as illustrated in Figure 4.4. Approximately 2 g of previously dried powdered bentonite is placed on a 9 cm diameter filter paper. The bentonite is spread over the filter paper within a 5 cm diameter template. The paper and bentonite are placed on the semi-submerged sintered plate, ensuring the bath is sealed, to absorb water for 18 hours (Figure 4.5). The height of the sample above the water surface was approximately 1.2 cm. The water temperature is recorded before and after the 18 hours. The bentonite and filter paper are then weighed. The average weight of the wet filter paper is subtracted from the weight of the hydrated filter paper plus the bentonite. This average weight is determined prior to testing by allowing 4 filter papers to absorb water over an 18-hour period without any bentonite.

To form a definitive correlation between water absorption and liquid limit, 5 samples of activated bentonite and 5 samples of non-activated bentonite were prepared and tested. The liquid limit for each sample was also tested and plotted linearly against the plate water absorption. Each absorption test followed the same aforementioned procedures.



**Figure 4.4:** Sketch of the plate water absorption test.

The water absorbed by the bentonite is calculated as a percentage of the dry mass as follows:

$$Absorption, \% = \frac{W_w - W_d}{W_d} \times 100 \quad (4.2)$$

Where:

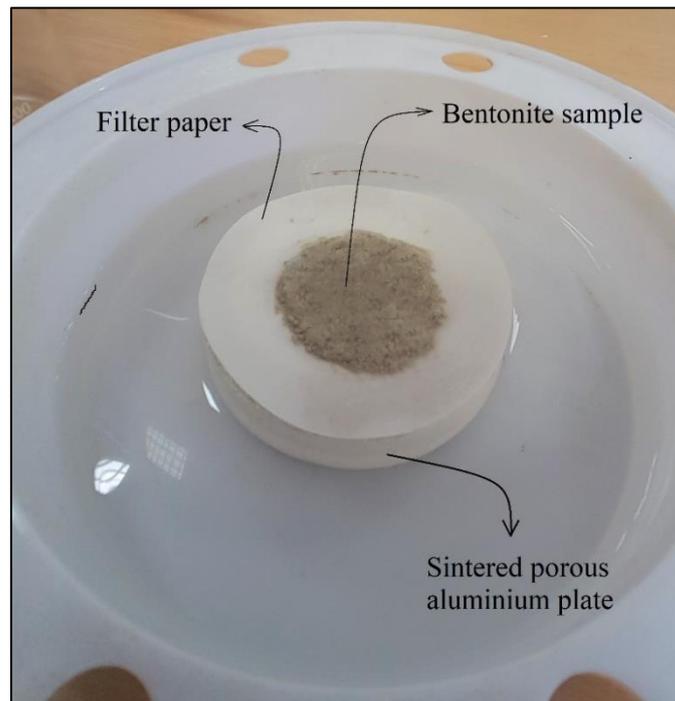
*Absorption* is the percentage of water absorbed

$W_w$  is the weight of the hydrated bentonite, g

$W_d$  is the weight of the dry bentonite, g

The purpose of conducting the PWA test in this research was to determine primarily, any comparisons between PWA and Atterberg limits.

Initial testing began with four tests being conducted to determine the absorption ability of the bentonite. In addition, five samples each, for activated and non-activated bentonite, were riffled and prepared for PWA and liquid limit test.



**Figure 4.5:** Laboratory plate water absorption test.

## 4.7 Atterberg limits

The procedures for the Atterberg limits tests follow the British Standard (BS); BS 1377-2:1990. The Atterberg limit is a measure of the critical moisture content, as a percentage of the dry mass, at which a clay soil changes consistency (White, 1949). Changing the liquid content of the clay will manifest in three distinct phases of consistency: the solid phase where the clay will display brittle behaviour, the plastic phase where the clay will exhibit a malleable consistency and the liquid phase where the clay will display flow like characteristics when slightly agitated. The moisture content above which a clay will act as a liquid is known as the Liquid Limit (LL). The moisture content below which a clay will display brittle behaviour is known as the Plastic Limit (PL). The moisture content range in which a clay will remain plastic is known as the Plasticity Index (PI) (White, 1949).

The PI is the difference between the LL and the PL:

$$PI = LL - PL \quad (4.3)$$

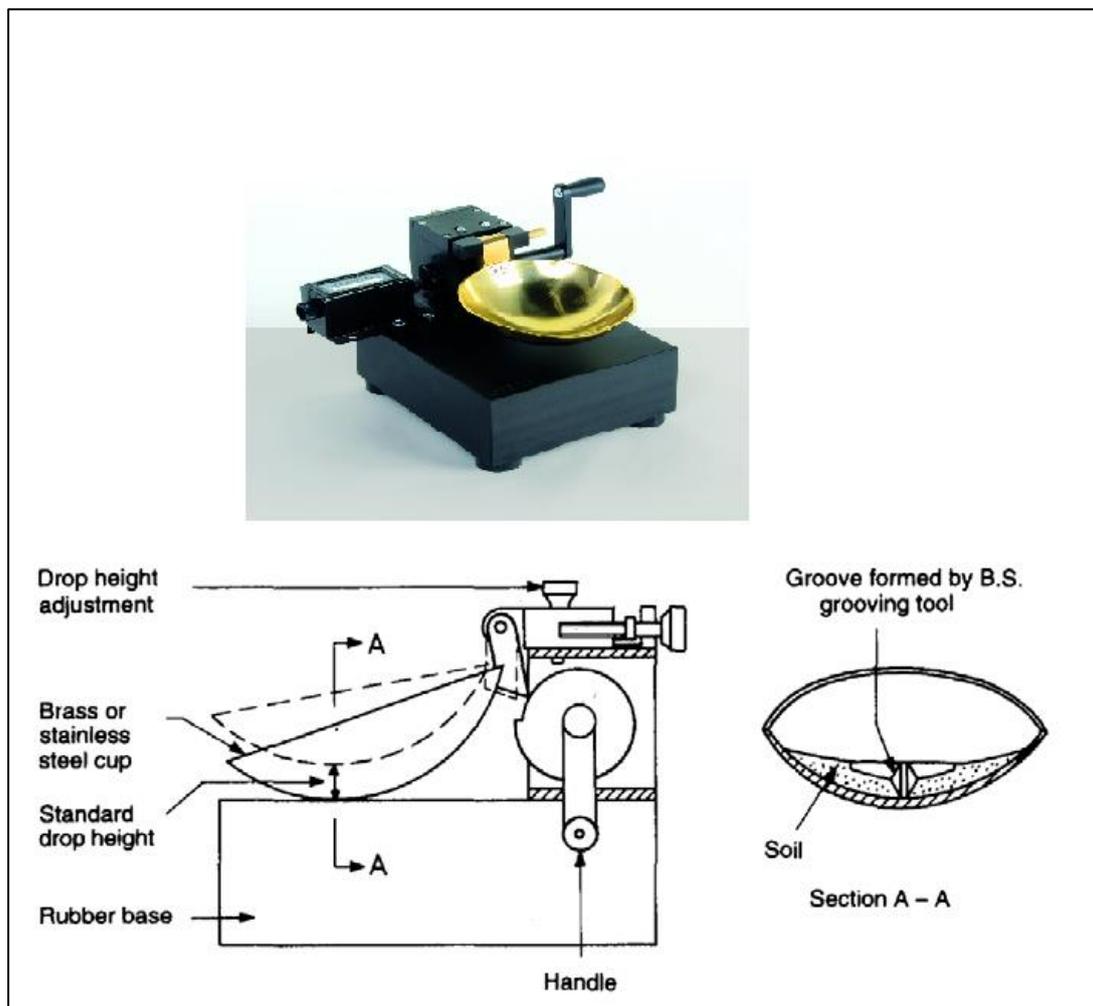
The test was conducted using powdered bentonite. The bentonite sample was prepared by mixing distilled water with the bentonite to make a paste. The bentonite paste was left to cure for at least 24 hours. The test then begins by placing a small amount of bentonite clay into the Casagrande device cup (Figure 4.6). The cup is movable in a vertical direction. When the handle is rotated the cup rises then taps down on the rubber base (Figure 4.7).



**Figure 4.6:** The Liquid Limit test using the Casagrande device.

A grooving tool was used to scrape a groove through the centre starting from the back and ending at the front. The handle was then rotated at a rate of 120 revolutions per minute (2 revolutions per second). The tapping motion of the falling cup causes the groove to close. The number of blows were counted until the two parts of the soil sample come into contact at the bottom of the groove along a distance of 10 mm. The soil closest to the closed groove was taken and tested for moisture content. The test was repeated at least four times, making sure each test results in a blow count between 10 and 50. The liquid limit corresponds to a blow count of 25 and is determined by plotting the results on a graph, where the number of blows is plotted as abscissa on a logarithmic scale and the corresponding water content as ordinate. The

straight-line curve corresponding to 25 blows gives the water content at the liquid limit boundary (Figure 4.9).

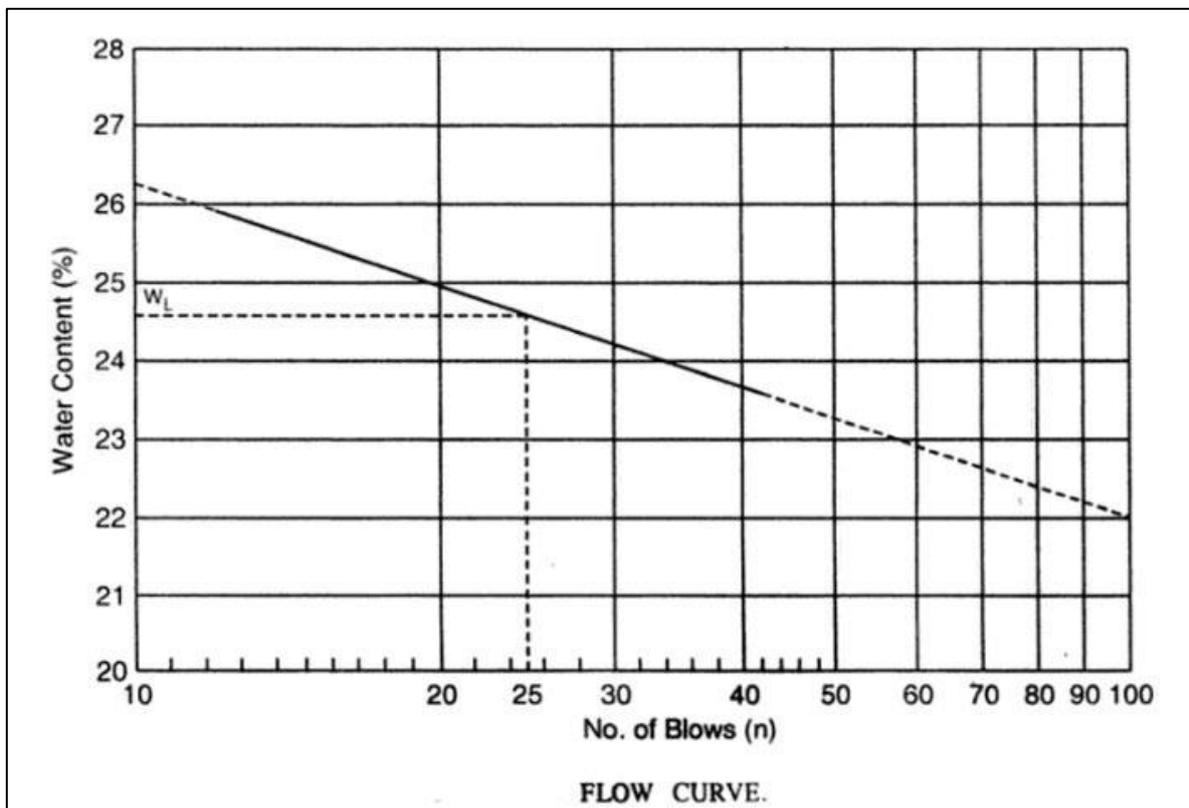


**Figure 4.7:** Casagrande device and sketch (after Clayton *et al.*, 1995).

The plastic limit was determined by mixing the clay with enough water so that it becomes malleable in the hand. About 8 g of clay was rolled into a ball. The ball was rolled between the fingers and glass plate to form a thread of an approximate uniform diameter of 3 mm. A rod of 3 mm diameter was used for reference. When a diameter of 3 mm was reached, the soil was moulded again into a ball. The process of rolling and moulding should continue until the thread forms cracks at 3 mm diameter (Figure 4.8). The clay has reached its plastic limit when the threads crack and crumble. The threads are then used for water content determination. The test was conducted three times and the average water content of the clay threads at their crumble point was taken as the plastic limit.



**Figure 4.8:** Plastic limit test.



**Figure 4.9:** Example of the liquid limit determination graph (after BS 1377-2, 1990).

The correlation between liquid limit and plate water absorption was investigated by conducting an additional five test each, for activated bentonite and non-activated bentonite.

These ten samples were prepared separately by drying and crushing. Each sample was then tested individually for liquid limits and plate water absorption. The resultant liquid limit data

was plotted as the abscissa ( $x$  values) and the plate water absorption data as the ordinate ( $y$  values).

The reasoning behind attempting to draw a correlation between the liquid limit and plate water absorption is to formulate a method to determine the performance of a bentonite absorption ability. Given that the liquid limit test is widely available this correlation aims to allow testing at any geotechnical soil laboratory. Another contributing factor was that previous studies (Sridharan and Nagaraj, 1999) have dealt with similar correlations and a comparison could be made with this study.

## **4.8 Post laboratory**

After all data was collected the results were analysed and all discussions are noted down in the relevant chapter. Where necessary, tests were compared to find links between them, especially between liquid limit and plate water absorption. Results for all tests were meticulously stored in a laboratory notebook to ensure correct record of all results. The crude data for all tests conducted can be found in Appendix 2.

# Chapter 5

## Results and Discussions

### 5.1 Introduction

This chapter presents the results and further discusses the results obtained from the various tests described in the previous chapter. The XRD results present the major mineral composition of the bentonite. The swell index shows unconfined swell. The fluid loss indicates the amount of water loss of a bentonite slurry, when subjected to air pressure. The plate water absorption shows the amount of water absorbed by the bentonite over an 18 hour period. The Atterberg limits reveal the liquid limit, plastic limit and linear shrinkage.

### 5.2 X-Ray diffraction

The results obtained from the XRD analysis for both the activated bentonite and non-activated bentonite are shown in Table 5.1. The results are presented in terms of the major minerals present.

**Table 5.1:** Mineral content of activated bentonite and non-activated bentonite.

	Percentage major minerals (%)	
	Activated bentonite	Non-activated bentonite
<b>Calcite</b>	1.0	1.8
<b>Cristobalite</b>	0.1	8.5
<b>Diopside</b>	1.7	2.4
<b>Mordenite</b>	2.5	4.0
<b>Muscovite</b>	2.6	3.7
<b>Plagioclase</b>	6.7	5.7
<b>Quartz</b>	18.6	15.7
<b>Smectite</b>	66.7	58.2

The results show a substantial amount of impurities in both the activated bentonite and non-activated bentonite. According to Gates and Churchman (2006), these impurities may adversely affect the swelling properties of the bentonite. Typically, bentonites contain around 60 % montmorillonite (von Maubeuge, 2002). The bentonites of this study can be regarded as typical

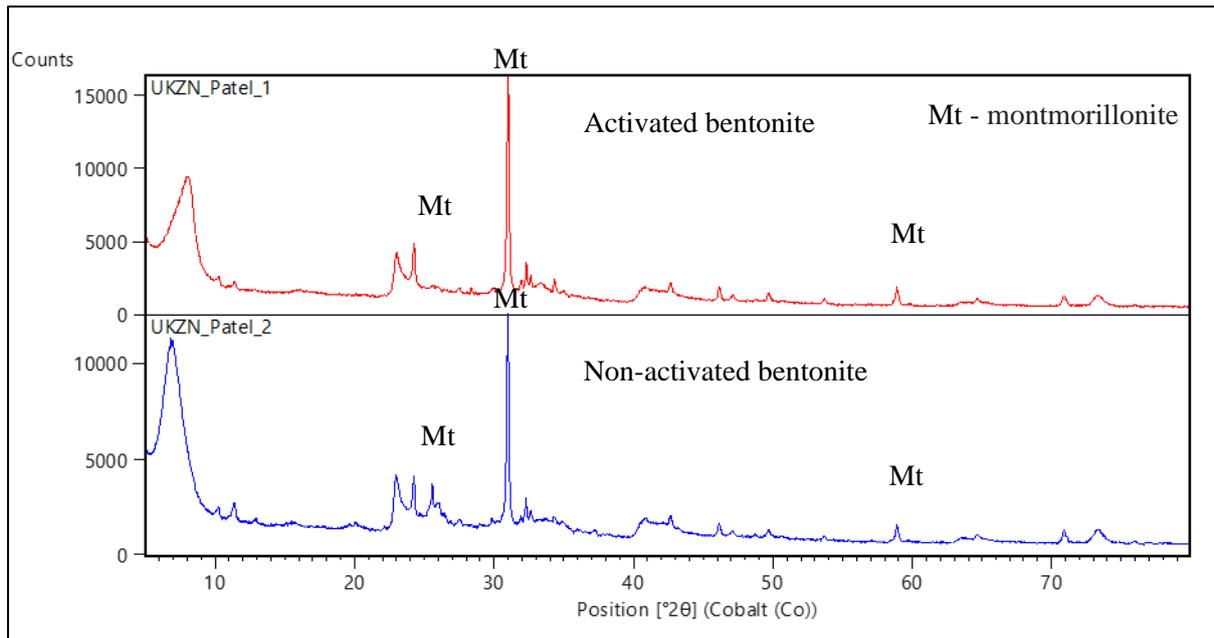
bentonites as their composition is dominated by smectite which is around 60 %. There is no standard compositional requirement for bentonites used in GCLs. Bentonites with impurities will not possess a desirable swell due to the impact that the non-smectite fraction has on the material's physical and chemical properties. The most detrimental types of impurities are non-swelling impurities which diminish the swelling ability of the bentonite (Gates and Churchman, 2006). This may be a contributing factor as to why the tested bentonites do not possess the desired swell index or fluid loss results as would be shown later in this chapter. According to Churchman *et al.* (2002), Wyoming bentonite has a smectite content of 90 % with far less accessory minerals (Table 5.2). The average quality of Imerys bentonite has a smectite content of less than 70 %. The quality and quantity of smectite and the chemical composition of bentonite critically influence the permeability and its performance as a sealing material (Dananaj *et al.*, 2005).

**Table 5.2:** Mineral composition of Wyoming bentonite (after Churchman *et al.*, 2002).

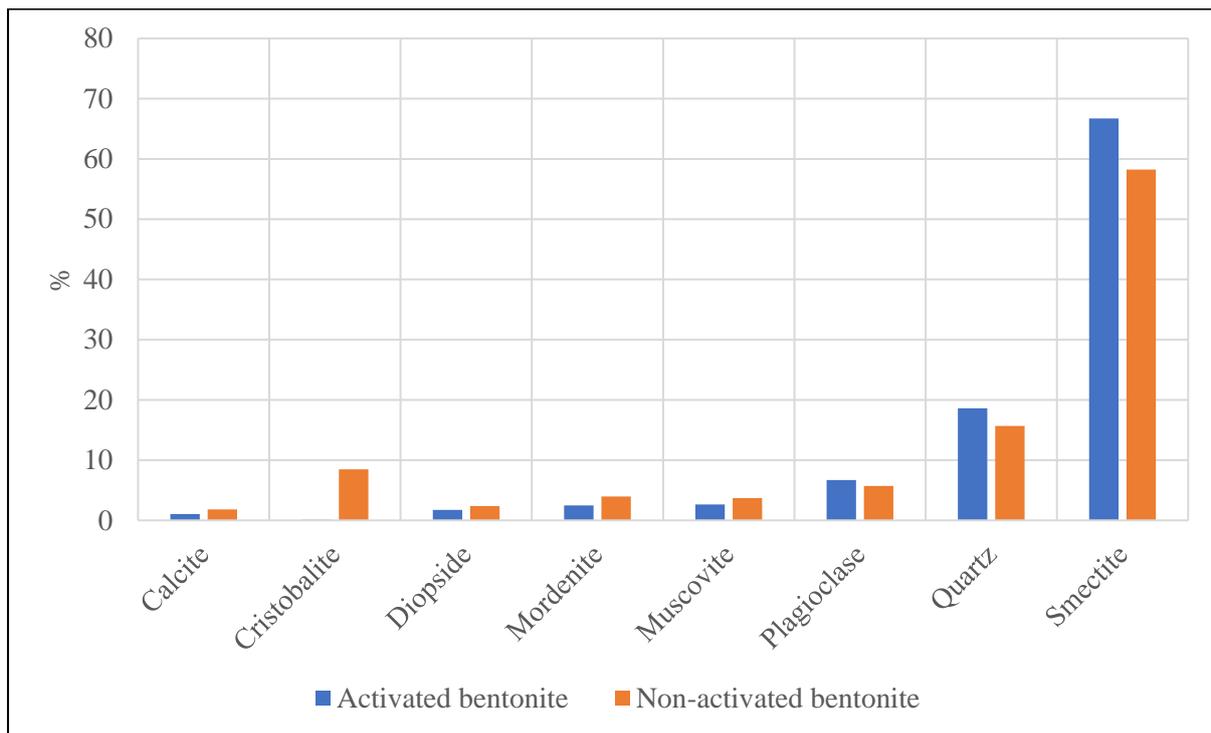
Mineral type	%
Montmorillonite	94
Gypsum	<1
Feldspar	<4
Quartz	<2

The XRD patterns of the activated bentonite and non-activated bentonite are shown in Figure 5.1 and is graphically represented in Figure 5.2. The peak positions of around 25, 30 and 60 are characteristics of montmorillonite. The other peaks correspond to impurities such as quartz, feldspars and calcite (Karnland *et al.*, 2006). The most significant impurity is quartz. The non-activated bentonite has a similar mineral composition to the activated bentonite, apart from cristobalite, which is notably higher.

It should be noted that von Maubeuge (2002) mentioned that the XRD method is somewhat unsuitable for fingerprinting bentonite and allowing a distinction between activated bentonite and non-activated bentonite. With XRD, the identification of mineral species and a simple quantitative estimation of their percentages within a sample is possible but an exact quantification requires several complementary analyses such as X-ray fluorescence (XRF) and scanning electron microscopy (SEM) (von Maubeuge *et al.*, 2007).



**Figure 5.1:** XRD pattern of activated bentonite and non-activated bentonite.



**Figure 5.2:** Mineral composition in percentages.

### 5.3 Swell index

#### 5.3.1. Activated bentonite and non-activated bentonite

The results obtained from the swell index tests for the 5 tests conducted on the activated bentonite and non-activated bentonite are shown in Table 5.3 and in Appendix 2.1. The average

swell index obtained for the activated bentonite is 15.6 ml/2 g in comparison to 9.8 ml/2 g for the non-activated bentonite.

**Table 5.3:** Results from the swell index tests for the activated bentonite and non-activated bentonite.

Swell index (ml/2 g)		
	Activated bentonite	Non-activated bentonite
Test 1	15	10
Test 2	16	9
Test 3	16	10
Test 4	16	10
Test 5	15	10
Average	15.6	9.8

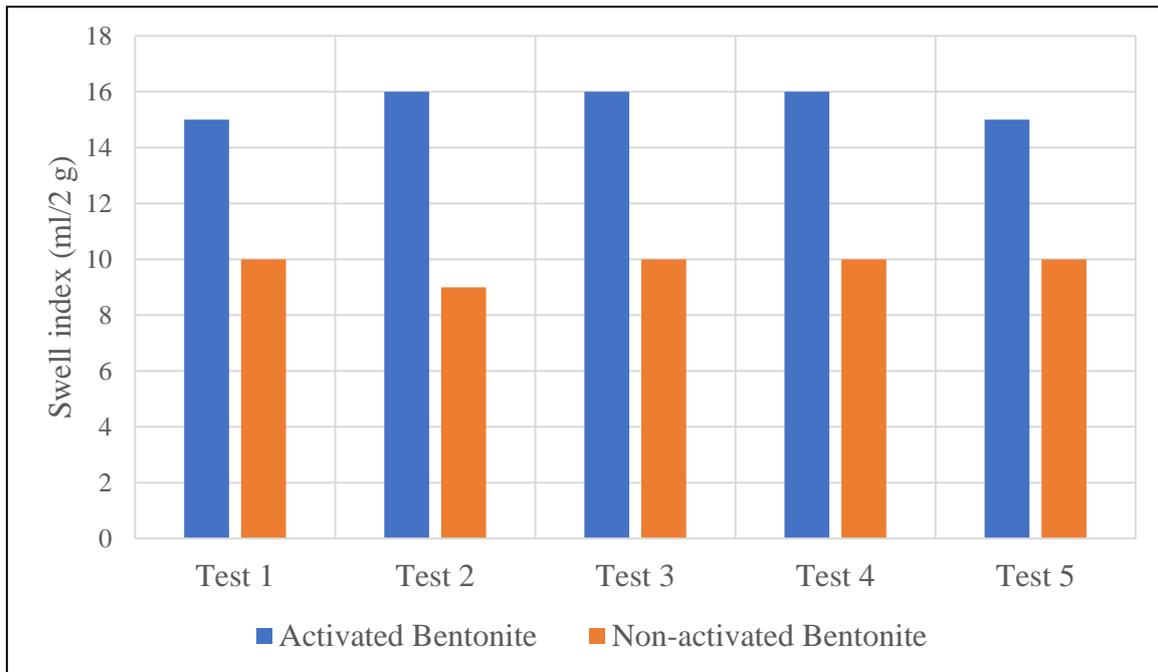
The results for the temperature, electrical conductivity (EC) and pH of the distilled water before and after each test, for the activated bentonite and non-activated bentonite, for the five tests carried out, are shown in Table 5.4.

**Table 5.4:** Temperature, EC and pH results of the distilled water before and after testing of the activated bentonite and non-activated bentonite.

Distilled water	Activated bentonite			Non-activated bentonite		
	Temperature (°C)	EC (µS/cm)	pH	Temperature (°C)	EC (µS/cm)	pH
Before testing	24	5	9.5	25	3	9.09
After Test 1	23	633	9.82	24.5	134	9.36
After Test 2	23	628	9.9	24.5	155	9.34
After Test 3	23	665	9.91	24.5	165	9.35
After Test 4	23	705	10.1	24.5	194	9.35
After Test 5	23	642	9.9	24	151	9.33

The results from the swell index show that the activated bentonite has a higher swell index than the non-activated bentonite. Activated bentonite has additional exchangeable sodium ions as a result of activation. Sodium ions, because of its large hydration radius, is primarily responsible

for the swelling bentonite (Alther, 1987). This explains why, the activated bentonite has a higher swell index than the non-activated bentonite. A magnitude of 5 to 6 ml/2 g increase in swell index is seen between the activated bentonite and the non-activated bentonite. This is about a 50 % to 70 % increase in swell. Figure 5.3 graphically shows the difference in swell index between the activated bentonite and non-activated bentonite.



**Figure 5.3:** Swell index of activated bentonite and non-activated bentonite.

The swell index for the activated bentonite does not reach the minimum requirement of 24 ml/2 g (ASTM D 5890). Further investigation was conducted as to why the swell index did not reach the required specification. This was experimented by testing the swell index of activated bentonite at specific time intervals after activation, as elaborated further in Section 5.3.2. The investigation concluded that at least 1 month is required after activation before the bentonite acquires the desired swell index. The activation process is an ionic exchange process whereby the sodium content of the bentonite is enhanced at a molecular level. The medium of this reaction being water. For complete activation, time is required for the reaction to reach completion (Patel *et al.*, 2019).

Wyoming bentonite, commonly referred to as MX80 bentonite, has a swell index of approximately 27 ml/2 g (Davies *et al.*, 2017). The swell index of Wyoming bentonite is much higher than the activated bentonite and non-activated bentonite. This may be attributed to the high montmorillonite content in the Wyoming bentonite.

The temperature, pH and EC of the distilled water before and the reagent after were measured. Since distilled water (de-ionised) was used, it can be assumed that no isomorphous ionic substitution occurred that could have affected the swelling potential of the bentonite. The temperature remains unchanged indicating no exothermic or endothermic chemical reaction between the deionised water and the activated bentonite and the non-activated bentonite.

The EC increases substantially. The initial EC for the distilled water used for the activated bentonite and non-activated is 5  $\mu\text{m}/\text{cm}$  and 3  $\mu\text{m}/\text{cm}$ , respectively. The EC increased to a maximum of 705  $\mu\text{m}/\text{cm}$  for the activated bentonite and 194  $\mu\text{m}/\text{cm}$  for the non-activated bentonite. Expectedly, ions enter into solution thereby increasing the electric conductivity. The EC of activated bentonite is higher than the non-activated due to the ionic exchange of sodium allowing the dissolution of accessory minerals into the surrounding solution. The most quickly dissolving components in natural bentonites are carbonates and sulphates (Muurinen and Lehtikoinen, 1999).

Using Equation 5.1, the total dissolved solids (TDS) of the reagent can be calculated using the EC values (Thirumalini and Joseph, 2009; Rusydi, 2018). Other equations do exist, such as Siosemarde *et al.* (2010). However, the equation by Thirumalini and Joseph (2009), and Rusydi (2018) is used due to its simplicity and ease of application. Both equations produced the same results (the first equation, the one used subsequently, is by Thirumalini and Joseph (2009) and Rusydi (2018) and the second equation is by Siosemarde *et al.* (2010)).

$$TDS=0.64 \times EC \quad (5.1)$$

The TDS of the distilled water equated to 3.2 ppm or 0.064 meq/L and 1.92 ppm or 0.03 meq/L for the activated bentonite and non-activated bentonite, respectively. This indicates virtually zero colloidal particles being present in the distilled water before testing. The water ensuing testing resulted in a maximum TDS value of 451.2 ppm or 9.02 meq/L for the activated bentonite and 124.2 ppm or 2.48 meq/L for the non-activated bentonite.

Kaufhold and Dohrmann (2008) did a study to identify the differences in the stability of bentonites in contact with deionized water. Stability, in this case, connotes detachment of colloidal particles and/or dissolution of the bentonite. They concluded that the bentonites release ultrafine colloidal particles and that the colloidal particles are mainly montmorillonite. Approximately 10 % of the elemental concentration measured in the water solution is from dissolution of the octahedral sheet. However, detachment of colloidal particles was found to be the dominating mechanism (Kaufhold and Dohrmann, 2008). Thus, in the case of this research,

it can be assumed that the contributor to an increased EC would be colloidal particles present in the reagent subsequent to testing.

The pH remains almost constant before and after the tests. However, the pH of the deionised water is around 9 to 9.5. This is not typical values for deionised water. The most plausible reason for this is uncalibrated lab equipment. However, this measurement does not influence the outcome of results but was merely used to detect a change in pH before and after each test.

### 5.3.2 In-house activated bentonite

In-house activation was conducted on three samples to investigate the effect of time on the beneficiation of bentonites.

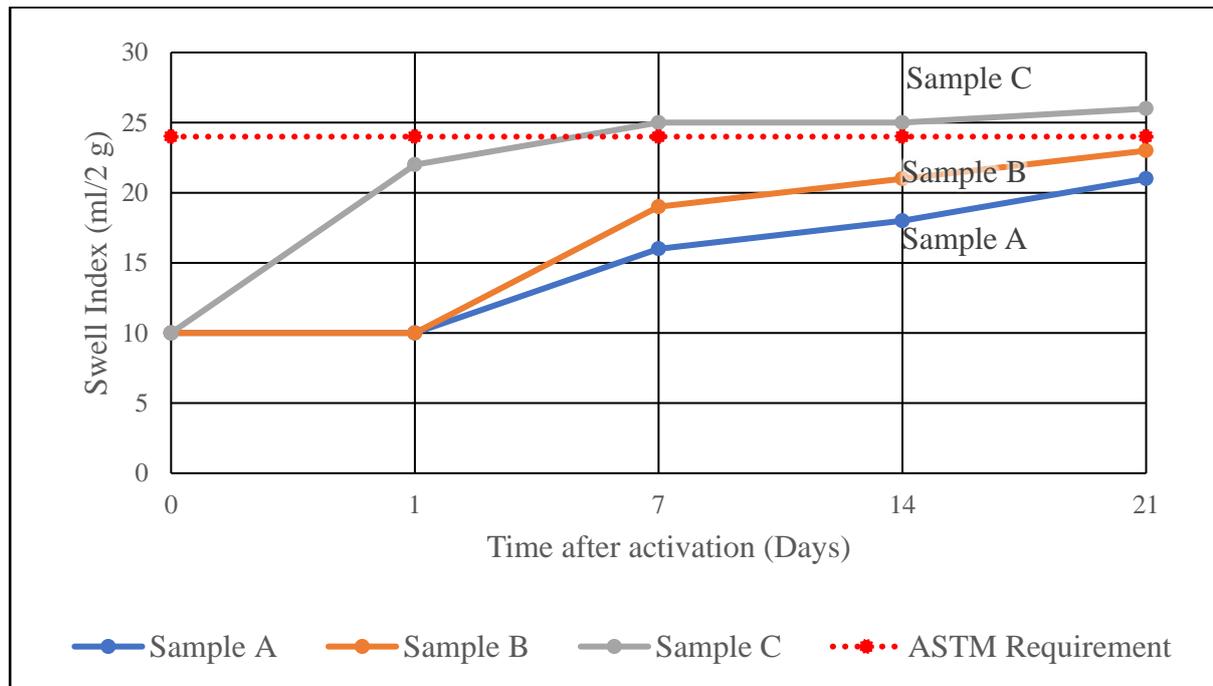
The results of swell index tests conducted on 3 Samples (A, B and C) at time intervals of 1 day, 7 days, 14 days and 21 days after activation are shown in Table 5.5 and in Appendix 2.2. Samples A, B and C contained 2 g, 4 g and 6 g of soda ash respectively, which equated to a ratio, by mass, of soda ash to bentonite of 1:50, 1:25 and approximately 1:16 respectively.

**Table 5.5:** Swell index over time after activation.

Days after activation	Swell index (ml/2 g)		
	Sample A	Sample B	Sample C
0	10	10	10
1	10	10	22
7	16	19	25
14	18	21	25
21	21	23	26

A plot of swell index versus time after activation is also shown in Figure 5.4 for Samples A, B and C. The plots show that Samples A and B show no increase in swell index within 24 hours. This shows that low amounts of sodium activation does not affect the bentonite over such a short period of time. However, Sample C shows an increase in swell index after 24 hours of activation, though lower than the required minimum. All three samples show a marked increase in swell index 7 days after the activation process. Although there is no change in the swell index for Samples A and B after 24 hours, Sample B however, swelled significantly more than Sample A beyond 24 hours. The most significant increase in swell for Samples A and B was after one week, with Samples A and B increasing by 6 and 9 ml/2 g of swell respectively, from

the initial swell index of 10 ml/2 g. However, two weeks of activation time is not sufficient for Samples A and B to reach the required minimum value of 24 ml/2 g. Sample C, however, attained the required minimum value after 1 week of activation. Thus, Sample C represents the best ratio required for activation over a short period of time, with a ratio of soda ash to bentonite of approximately 1:16. Although Sample C achieves majority of the required swell within the first week after activation, the swell index is enhanced significantly at the start and thereafter levels off. Samples A and B show a consistent increase in swell index over time with a decreasing rate of swell progressively. Swell indices for Samples A and B do not reach the benchmark even after 3 weeks of activation although Sample B is expected to reach a swell of 24 ml/2 g slightly over a 3-week period. Sample A will take slightly longer than Sample B. Further testing of these were halted as the laboratory in which these tests were conducted closed down.



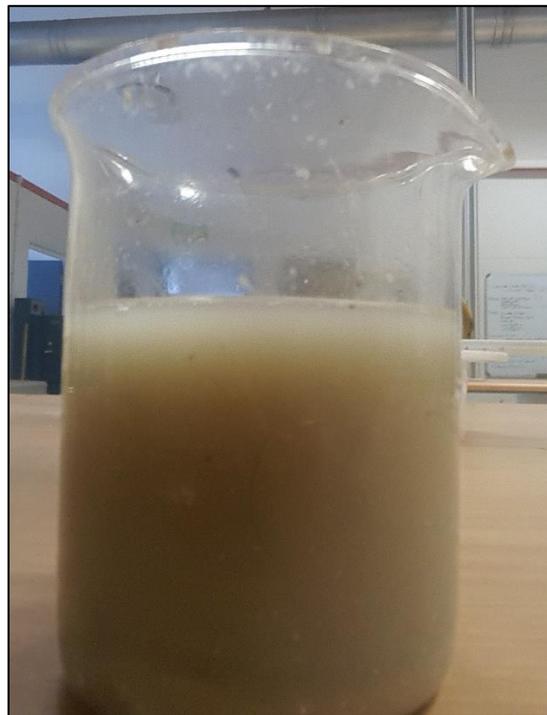
**Figure 5.4:** Swell index at time intervals after activation of Samples.

It was also observed that bentonite with higher levels of soda ash resulted in a dispersive sample. The bentonite dispersed throughout the distilled water making the water murky and difficult to read as there was no definite top surface. The sample containing 6 g of soda ash displayed a dispersive character when tested for swell index as compared to the 2 g and 4 g samples. Davies and Lacey (2009) explain that a sodic clay is dispersive, such that a substantial amount of sodium interferes with the structural stability of the soil. The reason being that when the ratio of sodium to other exchangeable ions (such as  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$  and  $\text{Na}^+$ ) is high, clay particles

are less tightly bound to each other and the soil easily disperse when the soil becomes wet. With this understanding, an addition of excess soda ash will add unnecessary sodium into the bentonite thereby increasing the ratio of sodium to other exchangeable ions. Figure 5.5 shows the different degrees of dispersion of a clay soil from least sodic to most sodic. A similar milky sample was observed for Sample C (Figure 5.6). This may be detrimental if the bentonite is used in a GCL.



**Figure 5.5:** Dispersion of soils. Least sodic to most sodic is from left to right (Davies and Lacey, 2009).



**Figure.5.6:** Dispersive Sample C, similar to that observed by Davies and Lacey (2009).

## 5.4 Fluid loss

The results from the fluid loss tests conducted on the activated bentonite and non-activated bentonite are shown in Table 5.6 and in Appendix 2.3.

**Table 5.6:** Fluid loss results of activated and non-activated bentonite.

Fluid loss (ml)			
	Average	Range	Number of tests conducted
Activated bentonite	23.2	23.0 – 23.4	3
Non-activated bentonite	26.8	25.7 – 27.9	3

Activated bentonite was expected to have a lower fluid loss due to a higher swelling and sealing ability (Liu *et al.*, 2014). The maximum ASTM requirement for fluid loss of bentonite used in GCLs is 18 ml. The tested bentonites, activated and non-activated, does not comply with the required maximum fluid loss.

Fluid loss is indicative of the hydraulic conductivity. Liu *et al.* (2014) conducted a study where they used fluid loss as a quick method to evaluate the hydraulic conductivity of bentonite within geosynthetic clay liners. Liu *et al.* (2014) further pointed out that the hydraulic performance of GCLs depends directly on the swelling capacity of the bentonite component when in contact with water. Studies have shown that an increase in hydraulic conductivity is mainly due to limited bentonite swelling (Shackelford *et al.*, 2010). The fluid loss test evaluates the capability of bentonite suspension to form a hydraulic barrier. A low fluid loss indicates low flow through the bentonite barrier within a given time and therefore implies a low hydraulic conductivity. In this study, failure to achieve the recommended fluid loss can be attributed to a lack of swell, as mentioned by Shackelford *et al.* (2010).

Lee and Shackelford (2005) investigated the effect of the quality of bentonite on the hydraulic conductivity. They found out that the hydraulic conductivity of high-quality bentonites is 3 times lower than the hydraulic conductivity of low-quality bentonites, when the bentonites were permeated with water. Furthermore, the high-quality bentonite is distinguished by a higher montmorillonite content as well as a higher plasticity index.

It is evident that, although the bentonites are being used in GCLs with the prerequisite of activation, the quality of the bentonite product alone, prior to activation, is of substandard quality. Activation, in a way, increases the quality of the bentonite to act as a hydraulic barrier. It is evident from this study that although the bentonite may have undergone some activation,

incomplete beneficiation will result in a deficient product. The beneficiation process may require additional time or additional treatment to achieve full activation.

## 5.5 Plate water absorption

The absorption ability of the bentonite was measured as the water content absorbed by the bentonite, after a fixed period of time. The results from the plate water absorption tests for both the activated bentonite and non-activated bentonite are presented in Table 5.7. Supporting data for the fluid loss tests are presented in Appendix 2.4. The results are presented as a percentage of the dry mass. The average absorption for the activated bentonite is 133.6 % whilst the average for the non-activated bentonite is 121.6 %.

**Table 5.7:** Summary of results from plate water absorption tests.

	Water absorption (% of the dry mass)				
	Test 1	Test 2	Test 3	Test 4	Average
<b>Activated bentonite</b>	119.1	129.1	144.1	142.1	133.6
<b>Non-activated bentonite</b>	120.7	117.7	124.1	123.7	121.6

The water absorbed is way above the dry mass of the sample. Bentonite, whether activated or non-activated, absorbs more water than the actual sample mass. The activated bentonite absorbed almost 10 % more water than the non-activated bentonite. This increase in absorption, which is intermolecular and intramolecular, is what gives rise to the increase in swell and resultantly decreases the fluid loss and thereby enhancing sealing behaviour. It is evident that the addition of soda ash increases the absorption capabilities of bentonite. Swelling occurs when water enters between clay particles resulting in an increase in volume (Norrish and Quirk, 1954), hence a greater absorption of water results in a higher swell.

Test 1 for activated produced results similar to that of non-activated bentonite. There are two possible reasons for this. This could be as a result of incomplete activation. It could also be as a result of randomly selecting a non-activated area. It is possible that some areas of the bentonite do not achieve activation during the beneficiation process as a consequence of indiscriminate processes. Since the sample size of the plate water absorption test is miniscule, it is likely a non-activated area of bentonite may have been randomly selected.

Bentonite with better absorption simply means that the bentonite has a higher ability to absorb water. Since absorption is directly related to swell, the plate water absorption would indicate the bentonite ability to swell.

## 5.6 Atterberg limits

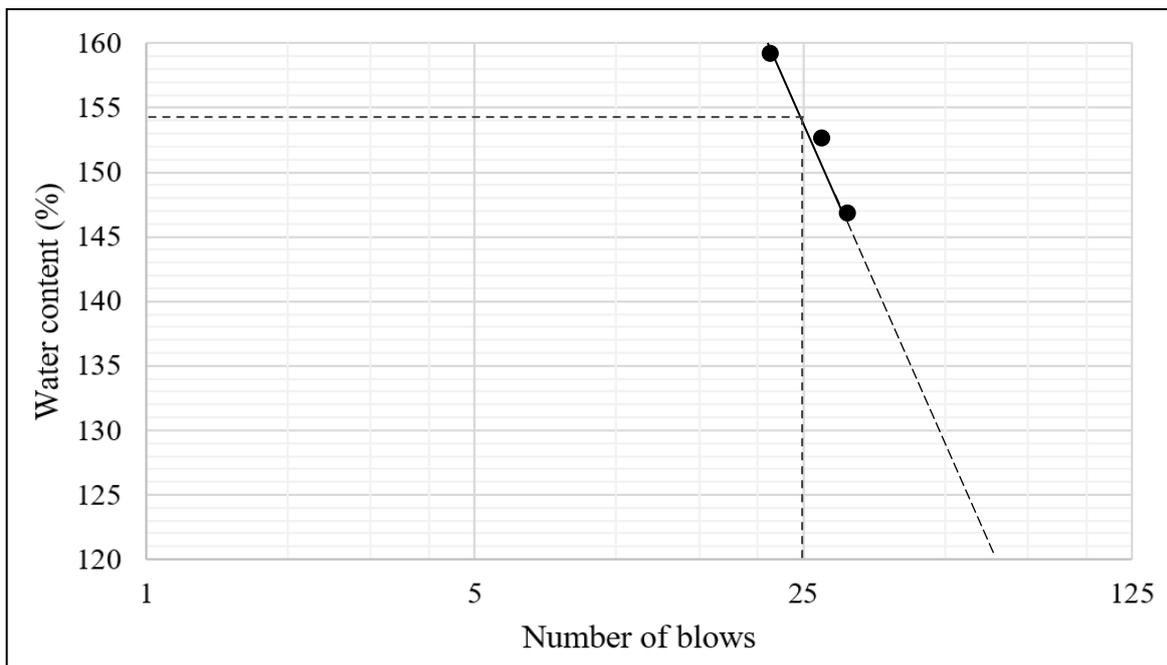
The results from the Atterberg limits tests are shown in Table 5.8 and in Appendix 2.6. Activated bentonite has 9 % higher liquid limit and 10 % higher plasticity index whereas the plastic limits are almost the same. The liquid limit is derived by corresponding the water content to a blow count of 25 on the liquid limit flow curve (Figure 5.7). The remaining flow curves for all the liquid limit tests are presented in Appendix 3. The plasticity index is derived by taking the difference between the liquid limit and plastic limit. Therefore, the plasticity index presented in Table 5.8 is the difference between the average liquid limit and the average plastic limit. The higher liquid limit for the activated bentonite suggests it has higher plasticity. The linear shrinkage results are identical. The linear shrinkage test was not of much significance to this study. It simply gives an indication to the degree of shrinkage when the bentonite is completely dry. The bentonite forms a powder when entirely dehydrated thus a straightforward measurement was impractical. Hence, the 25 % linear shrinkage is somewhat of an estimated figure.

**Table 5.8:** Atterberg limits results.

		<b>Atterberg limits (%)</b>	
		<b>Activated bentonite</b>	<b>Non-activated bentonite</b>
<b>Liquid limit</b>	<b>Average</b>	144	135
	<b>Range</b>	130 - 154	128 - 141
	<b>No. of tests</b>	8	8
<b>Plastic limit</b>	<b>Average</b>	50	51
	<b>Range</b>	48 - 51	49 - 53
	<b>No. of tests</b>	3	3
<b>Plasticity index</b>	<b>Average</b>	94	84
<b>Linear shrinkage</b>	<b>Average</b>	25	25
	<b>Range</b>	-	-
	<b>No. of tests</b>	1	1

Interparticle forces control the liquid limit of bentonite. The dominant interparticle force of bentonite is repulsion through osmotic activity (Warkentin, 1961). This repulsion keeps the

particles in a fixed configuration which prevents free movement (Warkentin, 1961). As the repulsion is decreased, particles move freely at lower liquid limits. Strength results from the force of repulsion resisting displacement of particles in the shear plane. Sodium increases the repulsion forces enabling a higher liquid limit. This is related to the higher repulsion found with monovalent ions, such as  $\text{Na}^+$  (Warkentin and Yong, 1960; Warkentin, 1961). Activation increases the concentration of  $\text{Na}^+$  within the bentonite thereby increasing the interparticle forces and hence increasing the swell index. Consequently, the activated bentonite has a higher liquid limit than the non-activated bentonite.



**Figure 5.7:** Liquid limit for activated bentonite, Sample A1.

There are many studies showing correlations of Atterberg limits to other soil properties. Farrar and Coleman (1967) went as far as correlating the cation exchange capacity (in meq %) with the liquid limit, with a high level of confidence (Equation 5.2).

$$C = -5 + 0.45W_L \quad (5.2)$$

Where  $C$  is the cation exchange capacity (CEC) and  $W_L$  is the liquid limit.

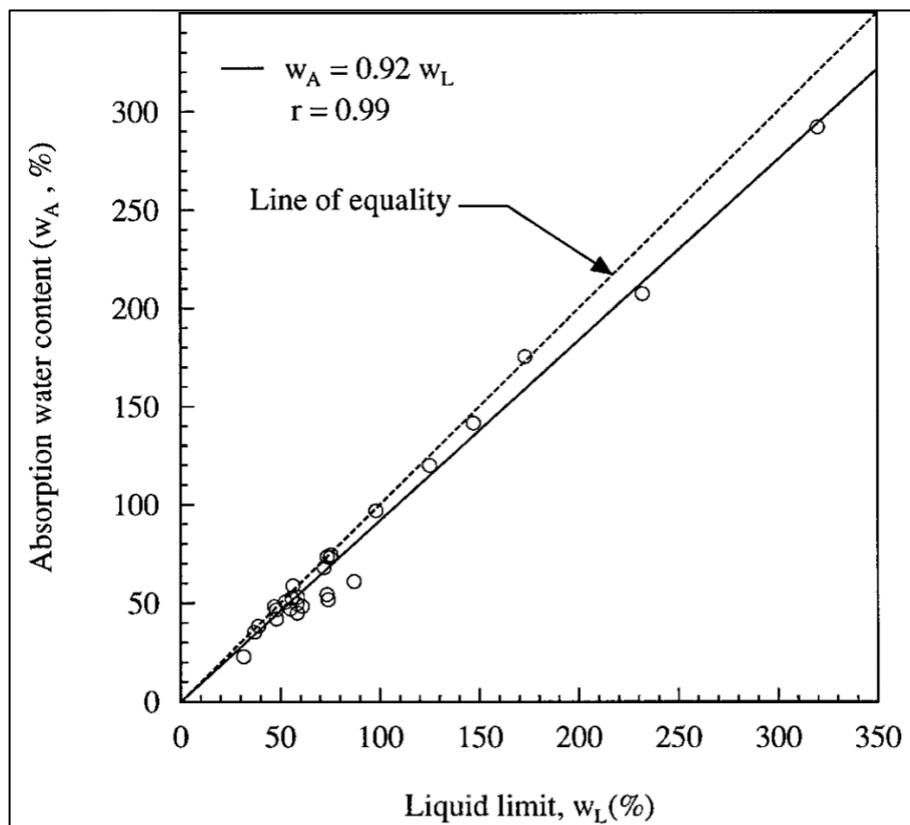
Using Equation 5.2 and average values for  $W_L$ , CEC values for the activated and non-activated bentonite are 59.8 meq/100 g and 55.75 meq/100 g, respectively. These are typical CEC values for bentonite as documented by Kayabali (1997), who recorded CEC values between 55 meq/100 g and 60 meq/100 g for sodium bentonite.

It is also possible to correlate the liquid limit to the absorbed water content. Sridharan and Nagaraj (1999) correlated liquid limit to water absorption. Their correlation between liquid limit and water absorption can be seen in Figure 5.8.

The absorbed water content is plotted against the liquid limit. Sridharan and Nagaraj (1999) shows that the absorbed water content is almost the same as the liquid limit of a clay soil. The correlation can be given by Equation 5.3

$$W_A = 0.92W_L \tag{5.3}$$

Where  $W_A$  is the absorbed water content and  $W_L$  is the liquid limit.



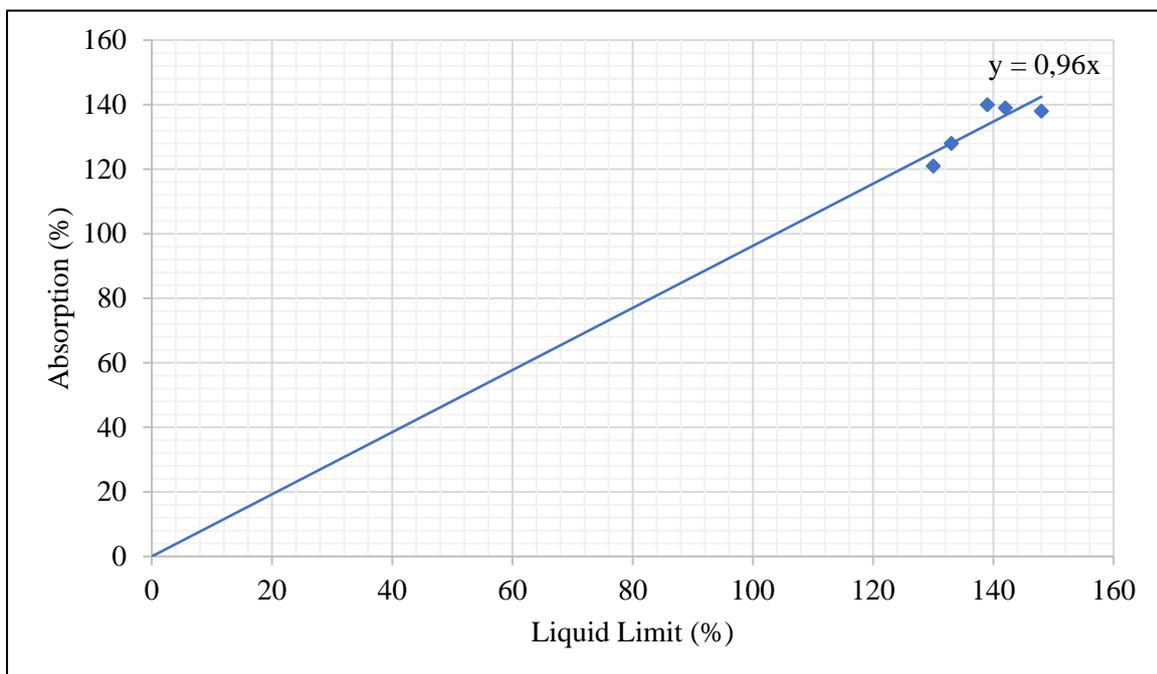
**Figure 5.8:** Correlation of liquid limit and absorption. The line of equality is where  $y = x$  (after Sridharan and Nagaraj, 1999).

To determine whether Sridharan and Nagaraj (1999) correlation of liquid limit to absorption applies to this study, a further 10 liquid limit and plate water absorption tests were conducted. The results are showed in Figure 5.9 for the activated bentonite and Figure 5.10 for the non-activated bentonite (data for liquid limit verses plate water absorption are presented in Appendix 2.7). The resultant linear regression curve for the activated bentonite, exhibits the formula given by Equation 5.4. Where the intercept is set to the origin.

$$W_a = 0.96W_L \quad (5.4)$$

Where  $W_a$  is the water absorption and  $W_L$  is the liquid limit.

The correlation equation derived from this research is somewhat comparable to Sridharan and Nagaraj (1999). This relationship is specifically important for bentonite studies considering liquid absorption is a key requirement in some bentonite applications. The correlation exists specifically when using distilled water and may be different if bentonite is subjected to test using a different liquid, such as acidic leachates.

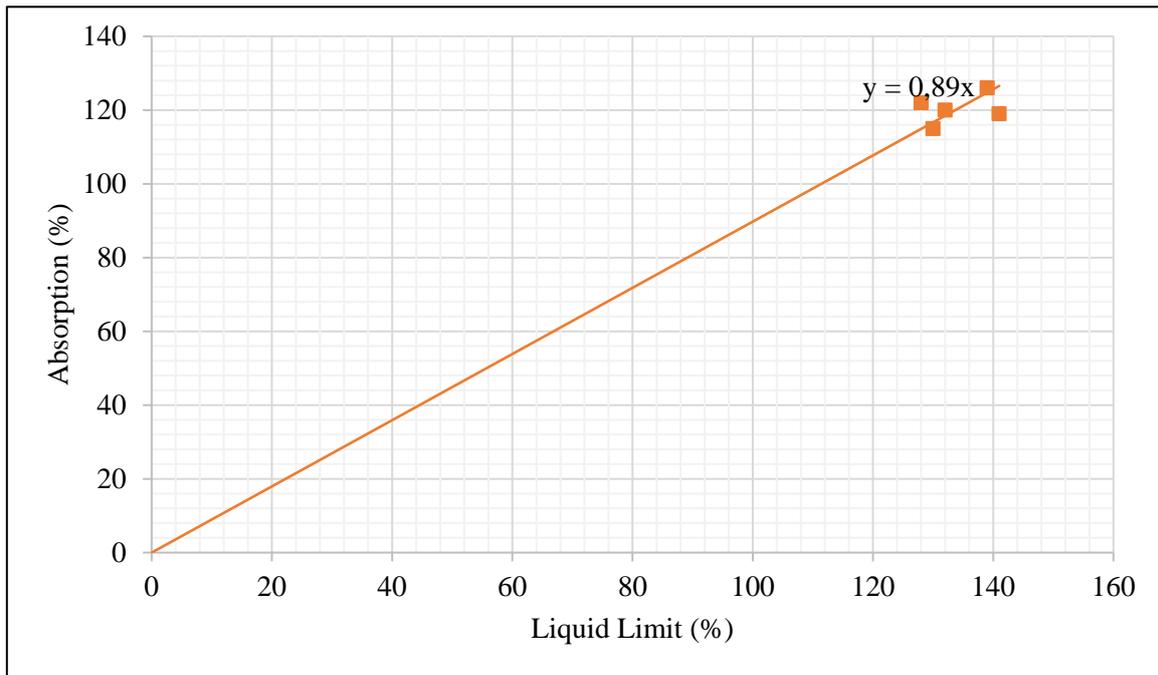


**Figure 5.9:** Absorption versus liquid limit for activated bentonite.

Non-activated bentonite displayed a more dispersed plot, lacking a convincing correlation. This linear regression curve produces the formula given in Equation 5.5.

$$W_a = 0.89W_L \quad (5.5)$$

A clearer trend is visible for the activated bentonite. However, a conclusive correlation could not be achieved for the non-activated bentonite. It is possible that this correlation applies only to sodium bentonite, studied in this research, and that different correlations could apply to soils of varying composition. It is conceivable that, in general, correlation is affected by the texture and fabric of the soil, and by the total surface area (Farrar and Coleman, 1967). Thus, soils different from those tested in this research may produce different results.



**Figure 5.10:** Absorption versus liquid limit for non-activated bentonite.

## 5.7 Summary of results

The XRD test revealed an almost identical mineralogical makeup except for cristobalite and smectite. Non-activated bentonite has approximately 8 % more cristobalite than activated bentonite, whereas activated bentonite has approximately 8 % more smectite. More than 30 % of these bentonites contain impurities which abates the swell index and hydraulic performance. Swell index tests produced results below the expected for activated bentonite. The average swell index is 15.6 ml/2 g and 9.8 ml/2 g for activated bentonite and non-activated bentonite, respectively. The expected swell index of activated bentonite was 24 ml/2 g, clearly falling short of this ASTM requirement. Further investigation revealed that inadequate activation may cause insufficient swelling. Two factors affect activation; the time required for the activation to reach completion and the amount of soda ash added to the bentonite. For a 2 % (i.e. a ratio 1:50) addition of soda ash, at least 4 weeks is required for the bentonite to reach complete activation. The electric conductivity of distilled water before and after the swell index test reveal substantial detachment of ultra-fine colloidal particles and/or dissolution of the bentonite. The pH and temperature of the distilled water before and after testing remained largely constant. The bentonite fluid loss displayed results of 23.2 ml for activated bentonite and 26.8 ml for non-activated bentonite, after 22.5 min. The activated bentonite has a lower fluid loss indicating a reduced hydraulic conductivity compared to non-activated bentonite. The quality and quantity of montmorillonite within the bentonite influences its hydraulic performance.

Activated bentonite has a higher montmorillonite content and is enhanced with sodium, through the addition of soda ash, hence the fluid loss is less than the non-activated bentonite.

The average absorption for the activated bentonite is 133.6 % whilst the average for the non-activated bentonite is 121.55 %. Absorption is directly related to swell. Therefore, a higher absorption results in a higher swell index, evidently shown as the activated bentonite swells more and has a higher absorption percentage than non-activated bentonite.

The plasticity index for activated bentonite is 10 % higher than non-activated bentonite and the liquid limit is 9 % higher for activated bentonite when compared to non-activated bentonite. Interparticle forces, responsible for the liquid limit, are enhanced by the addition of soda ash. This is related to the higher repulsion found with monovalent ions, such as  $\text{Na}^+$ . The liquid limit can be used to derive other soil properties, such as the CEC and absorption via correlation equations. Using these equations, the CEC for activated and non-activated bentonite is 63.4 meq/100 g and 56.65 meq/100 g, respectively. The plastic limit and linear shrinkage are virtually identical for activated and non-activated bentonite. The linear regression curve displays a moderate correlation between the liquid limit and plate water absorption for activated bentonite. A weaker correlation exists for the non-activated bentonite.

# Chapter 6

## Conclusion and Recommendations

### 6.1 Conclusion

Geosynthetic clay liners have become a common feature in many civil and environmental applications such as landfill hydraulic barriers, environmental protection barriers and as water impoundment liners. Their robust design and easy installation have favoured them over compacted clay liners. The abundance of bentonite deposits worldwide makes GCLs widely available. In South Africa, activated powdered sodium bentonite from the Imerys mine in Heidelberg Basin is used in the production of GCLs.

GCLs comprise powdered bentonite encased between two geotextile or geomembrane sheets. However, the fundamental component of GCLs is the bentonite used, which at times may not be of the desired quality. Consequently, a process known as beneficiation, whereby soda ash (sodium carbonate) is added to the bentonite, is carried out to enhance its quality. This results in the bentonite being referred to as activated bentonite. Soda ash principally increases the amount of exchangeable sodium ions within the bentonite, as the ionic exchange of sodium is the largest contributor of bentonite swell, when in contact with water.

The aims of this research were to investigate the suitability of sodium bentonite from the Imerys bentonite mine in the Western Cape Province of South Africa, for its use in GCLs and to examine the difference in the behaviour of activated bentonite versus non-activated bentonite. In addition, an attempt was made to draw correlations between liquid limit and plate water absorption. Through liquid limit data one may be able to approximate the bentonite performance of a GCL. This study subjected samples of activated and non-activated bentonite to laboratory test to determine the bulk mineralogical composition, swell index, fluid loss, plate water absorption and Atterberg limits.

XRD tests reveal that the Imerys bentonite, derived from the Heidelberg Basin, comprises approximately 60 % smectite. The balance comprises non-swelling impurities, which is mainly quartz. The major difference in mineralogy is that the activated bentonite has 8 % more smectite than the non-activated bentonite whereas the non-activated bentonite has 8 % additional cristobalite. Other impurities such as calcite, diopside, mordenite, muscovite, plagioclase and quartz are almost identical.

The swell index for the sodium activated bentonite and the non-activated bentonite does not reach the required minimum of 24 ml/2 g within the required 24 hours. In the case of activated

bentonite, this could possibly be due to incomplete activation. However, the swell index for activated bentonite is 5 ml/2 g more than non-activated bentonite. Subsequent to swell index testing, the EC of the water spiked considerably. Minimum EC values for deionized water were recorded at 5  $\mu\text{S}/\text{cm}$ . The maximum EC for the water after testing the activated bentonite was recorded at 705  $\mu\text{S}/\text{cm}$  and the maximum EC for the non-activated bentonite was recorded at 194  $\mu\text{S}/\text{cm}$ . The TDS can be calculated from the EC. The water ensuing after the swell index test resulted in a maximum TDS value of 451.2 ppm or 9.02 meq/L for the activated bentonite and 124.2 ppm or 2.48 meq/L for the non-activated bentonite. The increase in EC and TDS can be attributed to the ionic exchange of sodium allowing the dissolution of accessory minerals into the surrounding solution and the release of ultrafine montmorillonite colloidal particles. The fluid loss for activated and non-activated test produced results higher than the required minimum fluid loss of 18 ml. Average fluid loss results for activated and non-activated bentonite are 23.2 ml and 26.8 ml, respectively. This is attributed to low quality bentonite and incomplete activation.

The linear regression curve for liquid limit verses plate water absorption of activated bentonite show a modest linear relationship. The same cannot be said for non-activated bentonite. A clearer relationship may have been achieved with more laboratory tests, which time did not permit. The results show that the natural sodium bentonite is not suitable for GCLs. Activated bentonite, however, may sometimes fall short of the required specification due to insufficient activation or a low-quality blend

The key property that controls hydraulic performance, is the bentonite's swelling ability. It is unknown how the swelling is affected by different processing environments. The method of crushing and milling may eventually have an effect on the swelling. The temperature at which bentonite is dried and the time spent in the oven may change the physicochemical behaviour of the bentonite. This study, however, does reveal the effect of time on the activation of bentonite. Samples with ratios of soda ash to non-activated bentonite of 1:50, 1:25 and 1:16 were tested for swell index. The swell index over time was recorded to ascertain the best ratios of soda ash to non-activated bentonite and the time required for complete activation to occur. In practice, a very small amount of soda ash is added to non-activated bentonite. Consequently, 4 weeks is the minimum time required for non-activated bentonite to reach the desired degree of activation.

Considering all the laboratory results it can be concluded that Imerys bentonite is a medium quality bentonite with borderline index properties that requires beneficiation and time to

achieve complete activation. The appropriate ratio that suits beneficiation is 1:50 and the required time for beneficiation is at least 4 weeks.

## **6.2 Limitations**

This research was not without limitations. The geosynthetic industry is relatively new to the South African market and hence there has been very little academic interest. Apart from the Kaytech Geosynthetic Laboratory in Pine Town, which eventually shut down due to economic reasons, there are no accredited geosynthetic laboratories in South Africa. Research on geosynthetics is rather new in South Africa. It would have been ideal to continuously test samples throughout the course of this research. However, the Kaytech laboratory which is well equipped for all the necessary tests was shut down late 2018. Consequently, it was impossible to continue with extensive laboratory testing that was initially planned for this research. The outsourcing of laboratory testing has been time consuming and expensive as some samples had to be shipped to Gauteng and the United States of America for XRD and fluid loss testing, respectively.

## **6.3 Recommendations**

Particularly for this research, it is recommended that a change in the method of field activation is required. Quality control measures should be implemented to ensure complete activation of bentonite. The blending process of different qualities of bentonite should be thoroughly reconsidered. Bentonite in nature presents itself as a moist and very soft rock. Breaking down the size of larger pieces to at least gravel size would ensure complete activation. The method of field activation requires more attention to ensure complete activation of bentonite. A standard procedure of field activation is needed to guide operators and professionals.

A statutory body should be established to govern the method of activation and processing, particularly when used in GCLs. Mines and quarries processing bentonite for use in GCLs should be accredited to distribute the bentonite. On a national scale academic research interest is required to guide this industry, especially considering the fact that geosynthetics are becoming a common engineering and environmental geological feature.

## **6.4 Future research**

Future research is required to investigate optimum soda ash to bentonite ratios and the optimal moisture content at which activation should take place. The swell index, and to some extent, fluid loss tests are operator dependent qualitative tests wherein the results are dependent on a

variety of factors, such as the quality of water used, the duration and temperature at which the bentonites are dried and the operator's diligence to perform the arduous test correctly. Lengthy tests result in operator fatigue and deviant testing techniques. Research into modifying the current testing procedures are needed. Less operator dependent and more time efficient tests are required to assess swelling and hydraulic performance.

Geosynthetic clay liners are an absolutely robust technology that can be adapted to various environments. With the correct understanding of the intricate physicochemical behaviour and reactions to certain environments it can be used with phenomenal success.

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**Appendix 1**  
**Conference paper**

# The effect of time on beneficiation of bentonites used in geosynthetic clay liners

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Geosynthetic Clay Liners (GCLs) have gained much attention over the past twenty years as a substitute for compacted clay liners. GCLs are generally thin manufactured hydraulic barriers comprising a layer of sodium bentonite encased between two geotextiles. To perform as an effective hydraulic barrier, sodium bentonite is the preferred type and is the most important component of a GCL due to its high-water retention characteristics and swelling potential. The bentonite in most instances, does not meet the desired swell potential. As such, a process known as activation is undertaken, whereby soda ash ( $\text{Na}_2\text{CO}_3$ ) is mixed with borderline quality bentonites to increase the quantity of exchangeable sodium ions. This study investigated the effect of time on the different stages in the activation process of bentonite on samples taken from a bentonite mine in Heidelberg, Western Cape Province in South Africa. Samples at different stages of the activation process namely, non-activated bentonite, freshly activated bentonite and processed bentonite long after activation, were selected for swell index tests to assess their swelling ability with time. The results showed that the bentonite that had sufficient time to reach complete activation swells the most in comparison to the freshly activated and non-activated bentonite.

## 1 INTRODUCTION

Geosynthetic Clay Liners (GCLs) have become a common and reliable substitution for compacted clay liners (Von Maubeuge 2002). Their applications span from civil engineering to environmental protection such as liners in water impoundment facilities and as composite liners in landfills (Kong et al., 2017; Liu et al., 2013; Rowe and Orsini, 2003; Bouazza, 2002).

GCLs comprise powdered bentonite encased between two geotextile or geomembrane sheets. However, the fundamental component of GCLs is the bentonite used (Kong et al., 2017; Bouazza, 2002; Lin & Benson, 2000). Bentonite is mined in many different qualities and their post mining process varies between suppliers. Hence, beneficiation of bentonite is necessary to enhance its performance. Beneficiation is the process whereby soda ash ( $\text{Na}_2\text{CO}_3$ ) is added to bentonite to enhance its swelling properties, indirectly decreasing its hydraulic conductivity. The bentonite is then said to be activated. Activation and beneficiation are used synonymously (Von Maubeuge, 2002)

Many studies (e.g. Shirazi et al., 2010; Gates & Churchman, 2006; Komine, 2004) have investigated the properties of bentonite but few (e.g. Von Maubeuge, 2002; Yildiz et al., 1999; Erdoğan & Demirci, 1996; Bleifuss, 1973) have elaborated on the effect of

time on the beneficiation process of bentonite. In order to perform suitably, the bentonite in the GCL requires a minimum swell index of 24 ml/2 g (ASTM D 5890). Bentonite that has not been fully activated may not possess the desired swell index and the GCL produced will not be up to the required standard.

This study investigated, through a series of time related tests, the change in the swell the index of activated bentonite over time. The focus of this study was to verify whether activation requires time to reach completion and to investigate the changes of swell index subsequent to activation. Additionally, different ratios of soda ash to bentonite were tested to investigate the most effective ratio required for beneficiation.

## 2 GEOSYNTHETIC CLAY LINERS

Geosynthetic Clay Liners (GCLs) comprise a thin layer of either sodium bentonite or calcium bentonite, bonded between a geomembrane or geotextile (Kong et al., 2017; Sarabian & Rayhani, 2013; Sari & Chai, 2013; Bouazza, 2002). Those using geotextiles sandwich the bentonite by needle-punching, stitching or by using a non-polluting adhesive (Bouazza, 2002). Needle-punched GCLs comprise an encasing nonwoven filament geotextile where the needle punching process pierces fibres from the upper geotextile to the

bottom geotextile (Kong et al., 2017). This process entangles the fibres to the bottom geotextile bonding the sheathing layers together. Bonding may also be achieved through heating, causing the piercing geotextile to fuse to the bottom geotextile. The stitching method involves sewing the geotextiles together using stitching bonded yarns (Kong et al., 2017).

Bentonite in the geomembrane supported GCL is bonded to the geomembrane using a non-polluting adhesive. The adhesive is mixed with the bentonite and pasted onto a geomembrane (Bouazza, 2002; Kong et al., 2017).

### 2.1 Bentonite

Bentonite loosely refers to any smectite-rich mineral. Initially, bentonite referred to the alteration product of volcanic glass. There are two types of bentonite; sodium bentonite and calcium bentonite, each denoting the predominant exchangeable ion. Sodium bentonite is preferred over calcium bentonite due to its better swelling properties. The primary constituent of sodium bentonite, which contributes to swelling, is montmorillonite. Generally, the typical sodium bentonite used in a GCL will contain 60–85% montmorillonite (Gates & Churchman, 2006).

Montmorillonite is a 2:1 (denoting a ratio of two tetrahedral silicate sheets to an octahedral silicate sheet) clay mineral containing an octahedral silicate sheet sandwiched between two tetrahedral silicate sheets in which adjacent sheets are joined through Van der Waals bonds via the apical oxygens of the tetrahedral sheet, on either side. Smectites generally refer to a group of expandable 2:1 (denoting a ratio of two tetrahedral silicate sheets to an octahedral silicate sheet) minerals with a low charge. Montmorillonite, the most common member of this group, derives its charge from the octahedral substitution of  $Mg^{2+}$  for  $Al^{3+}$ . Bentonite is a combination of predominantly montmorillonite and other clay minerals such as beidellite (Barton 2002).

#### 2.1.1 Swelling of bentonite

Swelling occurs when water enters between clay particles resulting in an increase in volume. Smectites generally swell differently from each other based on their physiochemical characteristics of attraction and repulsion, between clay particles and between clay minerals and the solution within the interlayer space (Norrish, 1954). The swell index is a measure of volume change a unit mass of bentonite undergoes upon absorption of a liquid which measures bulk swelling in millilitres per 2 grams. Typical bentonite swelling index values range from 4 to 50 ml/2 g (Gates & Churchman, 2006).

#### 2.1.2 Beneficiation

Borderline quality bentonites are mixed with soda ash in order to increase the quantity of exchangeable so-

dium ions. However, soda ash treated bentonite degrades considerably over time when compared to natural sodium bentonite (Von Maubeuge, 2002). Although the use of activated calcium bentonite as an alternative can be accepted, it is by far not as effective as natural sodium bentonite. The reason why soda ash-treated sodium bentonite is normally used is because naturally occurring bentonite with the desired properties is hard to come by.

The soda ash activation process of calcium bentonites (and even natural sodium bentonites of lower quality) takes place in the field where soda ash is added to un-beneficiated bentonite. Non-exchange areas or not fully sodium activated bentonite can create high permeable areas (Von Maubeuge 2002). Many researchers (e.g. Mansour & Szabó, 2001; Balázs, 2000; Alther, 1987) have shown that naturally occurring sodium bentonite is considerably more effective and more resilient than sodium activated bentonite. However, the use of sodium activated bentonite cannot be avoided.

## 3 METHODOLOGY

Three samples of bentonite (Sample 1, Sample 2 and Sample 3) were obtained from Imerys bentonite mine in Heidelberg, in the Western Cape Province of South Africa (Fig. 1). Sample 1 comprised activated, processed (dried and milled) sodium bentonite whereby the activation process occurred at least three months prior to testing. Sample 2 comprised unprocessed activated sodium bentonite wherein the activation process occurred at least two weeks prior to testing. Sample 3 comprised unprocessed non-activated sodium bentonite. Activation at the mine is done by the addition of soda ash to the bentonite via a conveyor belt system at 1.1% per unit mass.

The bentonite is loaded onto the conveyor belt, which passes under a hopper loader that carries the soda ash. The soda ash is released automatically as the bentonite passes beneath.

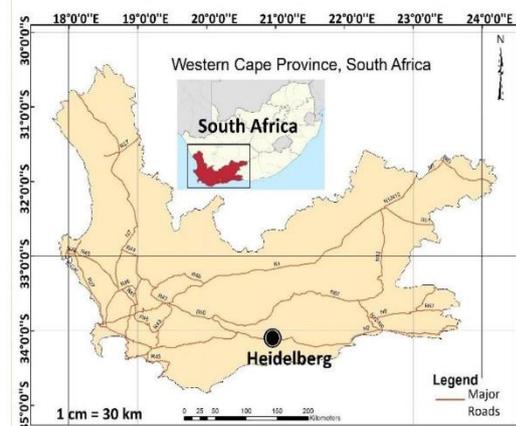


Figure 1. Location map of Heidelberg in the Western Cape Province, South Africa.

Furthermore, soda ash was obtained from Imerys mine and in-house activation was conducted at Kaytech Laboratories in Pinetown, South Africa on three Samples (A, B and C) wherein 2, 4 and 6 g of soda ash was added to 100 g of bentonite of Samples A, B and C, respectively. This equates to ratios of soda ash to bentonite of approximately 1:50, 1:25 and 1:16 for Samples A, B and C, respectively. Swell index tests were then conducted after 24 hours, 1 week, 2 weeks and 3 weeks of the activation of Samples A, B and C.

Swell index test followed the ASTM D 5890 test procedure. Soda ash was added to the bentonite prior to drying and milling.

The test procedure involved drying a desired amount of bentonite to a constant mass in a laboratory oven at 100°C for a minimum of 16 hours. Thereafter, the dried sample was crushed using a cast iron mortar and pestle (Fig. 2). The bentonite was then sieved on a mechanical sieve shaker. Crushed samples of 100% passing the 75 µm sieve were used in the swell index test.

The swell index test procedure (Fig. 3) involved filling a Class A graduated cylinder with 90 ml of distilled water.



Figure 2: Mortar and pestle used to crush the bentonite

Thereafter, 2 g of crushed bentonite was added to the cylinder in increments of 0.1 g with a 10 minutes interval between each increment. After the last increment of 0.1 g of bentonite, 10 ml distilled water was added to the cylinder, making sure to wash off any adhering bentonite on the sides, thus bringing the total volume of distilled water to 100 ml. The cylinder was left to stand for 2 hours and then gently shaken to release any air bubbles within the bentonite slurry. The cylinder was then left to stand for 16 hours. The results were read in ml/2 g at the top of the settled bentonite.

In addition, the pH, temperature and electrical conductivity (EC) of the distilled water, before and after



Figure 3. Swell index test set up

the test were recorded. The electrical conductivity was determined using a Hanna HI98312 water conductivity meter and the pH was determined using a portable Hanna HI98191 pH meter. The temperature was measured with a glass mercury thermometer. Since distilled water was used, the pH, temperature and EC were expected to be similar for all the tests prior to testing.

#### 4 RESULTS AND DISCUSSIONS

The average swell index for each sample is shown in Table 1.

Table 1. Average Swell Index for Sample 1, 2 and 3

	Swell Index
	ml/2 g
Sample 1	40
Sample 2	16
Sample 3	10

The differences are evident from the results obtained. Sample 1 displays a swell index way above the minimum requirement of 24 ml/2 g whereas Samples 2 and 3 fall short of the required minimum. This shows that non-activated sodium bentonite, from the Western Cape, does not possess the desired properties for use in GCLs without beneficiation. The soda ash treated bentonite (Sample 2) displays a low swell index. This could be attributed to insufficient time for activation. The activation process is essentially a chemical reaction that requires time to reach completion.

Since distilled water (de-ionised) was used, it can be assumed that no isomorphous ionic substitution occurred that could have diminished the swelling potential of the bentonite. Table 2 shows the pH, temperature and EC of the distilled water used in the tests. The electrical conductivity is directly related to the

total dissolved solids (TDS) of the reagent, which gives an indication of the dissolved ions.

Table 2. Average properties of distilled water used

	pH	Temp	EC
		°C	µS/cm
Sample 1	9.34	23	4
Sample 2	9.5	24	5
Sample 3	9.09	25	3

Using Equation 1, the TDS of the distilled water approximately equated to 3.2 ppm and 1.92 ppm for Samples 2 and 3 respectively.

$$TDS = 0.64 \times EC \quad (1)$$

These TDS values clearly indicate a lack of dissolved ions and thus shows that the distilled water did not affect the swelling potential of the bentonite tested. The bentonite swelling ability is purely related to its clay mineral composition and physiochemical properties.

The pH, temperature and EC of the water subsequent to testing, are shown in Table 3. It can be seen that the EC of the activated sodium bentonite (i.e. Samples 1 and 2) is higher than the non-activated bentonite (i.e. Sample 3). This could be due to more freely available sodium from the addition of soda ash. The pH of the reagent is almost constant and barely changes after testing. This would be expected as there are no dissolution or substitution of any  $H^+$  ions.

Table 3. Average properties of the solution after testing

	pH	Temp	EC
		°C	µS/cm
Sample 1	9.62	23	867
Sample 2	9.9	23	657
Sample 3	9.35	24	162

Figure 4 shows the plot of the swell index versus time, after activation, for Samples A, B and C. The plots show that Samples B and C show no increase in swell index after 24 hours. This shows that the sodium activation process does not affect the bentonite after 24 hrs. Sample C, however, shows an increase in swell index after 24 hours of activation though lower than the required minimum.

All three samples show marked increase in swell 7 days after activation. Although there is no change in the swell index between Samples A and B after 24 hours, Sample B however swelled significantly more than Sample A after 7 days.

The most significant increase in swell for Samples A and B was after two weeks, with Samples A and B increasing by 6 and 9 ml of swell respectively. However, two weeks of activation time is not sufficient for Samples A and B to reach the desired minimum value

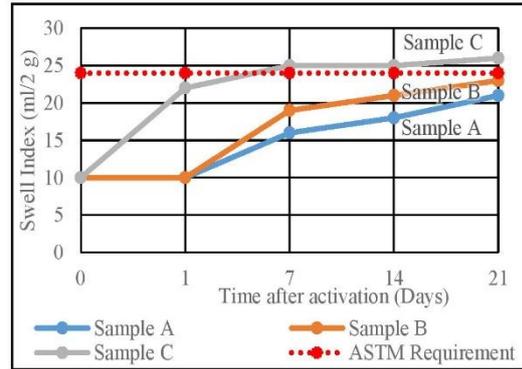


Figure 4. Swell index vs. time of Samples A, B and C, showing the ASTM requirement as a dotted line.

of 24 ml/2 g. Sample C, however, attained the required minimum value after 1 week of activation. Thus, Sample C represents the best ratio required for activation over a short period of time, with a ratio of soda ash to bentonite of 1:1.16.

Although, Sample C does not swell significantly more than the initial swell after activation. The swell index is enhanced drastically at the start and thereafter levels off. Samples A and B show a consistent increase in swell index over time with a decreasing rate of swell progressively. Swell indices for Samples A and B do not reach the benchmark even after 3 weeks of activation although Sample B is expected to reach a swell of 24 ml/2 g slightly over a 3-week period. Sample A will take slightly longer than Sample B. As this is an ongoing research, long term change in swell is still being monitored and the exact time frames required for the bentonite to reach the minimum swell is yet to be confirmed.

## 5 CONCLUSION

Bentonite presents itself naturally in various composition. Many a time, soda ash must be added to either sodium bentonite or calcium bentonite to enhance its properties, primarily being swell. The activation process is a chemical reaction whereby the sodium content of the bentonite is enhanced. The medium of this reaction being water. Hence, the activation process occurs at natural moisture content prior to drying and milling. For the completion of the activation process, time is required for the reaction.

As a preliminary study, this research shows that at least 4 weeks is required for the activation process to reach completion for a 2% addition of soda ash and 3 weeks for a 4% addition of soda ash. Over activation with soda ash may change the physiochemical properties of bentonite undesirably. Generally, activation in practice uses 1.1% of soda ash. In this case, 4 weeks will not be enough for the activated bentonite to reach the desired swell.

Bentonite in nature presents itself as a moist and very soft rock. Breaking down the size of larger pieces to at least gravel size would ensure complete activation. The method of field activation requires more attention to ensure complete activation of bentonite. A standard procedure of field activation is needed to guide operators and professionals.

## 6 ACKNOWLEDGEMENTS

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## **Appendix 2**

### **Laboratory results**

2.1 Swell index.

2.2 Swell index of in-house activated bentonite.

2.3 Fluid loss.

2.4 Plate water absorption.

2.5 XRD

2.6 Atterberg limits

2.7 Liquid limit vs absorption

## 2.1 Swell index

### Sample 1 – Activated bentonite

Test 1	Test 2	Test 3	Test 4	Test 5
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Swell Index (ml/ 2g)				
15	16	16	16	15

Temp after 16 hours (°C)				
23	23	23	23	23

Electric conductivity (µS/cm)				
633	628	665	705	642

pH				
9.82	9.9	9.91	10.1	9.9

#### Properties of the distilled water used

Temp (°C)	EC (µS/cm)	pH
24	5	9.5

### Sample 2 – Non activated bentonite

Test 1	Test 2	Test 3	Test 4	Test 5
--------	--------	--------	--------	--------

Swell index (ml/ 2g)				
10	9	10	10	10

Temp (°C)				
24.5	24.5	24.5	24.5	24

EC (µS/cm)				
134	155	165	194	151

pH				
9.36	9.34	9.35	9.35	9.33

#### Properties of the distilled water used

Temp (°C)	EC (µS/cm)	pH
25	3	9.09

## 2.2 Swell index of in-house activated bentonite

### After 1 day of activation

Swell index (ml/2 g)		
Sample A	Sample B	Sample C
10	10	22

### After 7 days

Swell index (ml/2 g)		
Sample A	Sample B	Sample C
16	19	25

### After 14 days

Swell index (ml/2 g)		
Sample A	Sample B	Sample C
18	21	25

### After 21 days

Swell index (ml/2 g)		
Sample A	Sample B	Sample C
21	23	26

## 2.3 Fluid loss

### Sample 1 – Activated bentonite

Fluid loss (ml)		
23.2	23.4	23.0
Average		23.2

### Sample 2 – Non activated bentonite

Fluid loss (ml)		
26.8	27.9	25.7
Average		26.8

## 2.4 Plate water absorption

Test 1	Test 2	Test 3	Test 4
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Mass of hydrated filter paper (g)			
1.31	1.29	1.34	1.33
Average			1.318

### Sample 1 – Activated Bentonite

Water absorption (%)			
119.1	129.1	144.1	142.1
Average			133.6

Temp of water (°C)			
22	23	22	22

### Sample 2 – Non activated bentonite

Water absorption (%)			
120.7	117.7	124.1	123.7
Average			121.6

Temp of water (°C)			
23	23	23	23

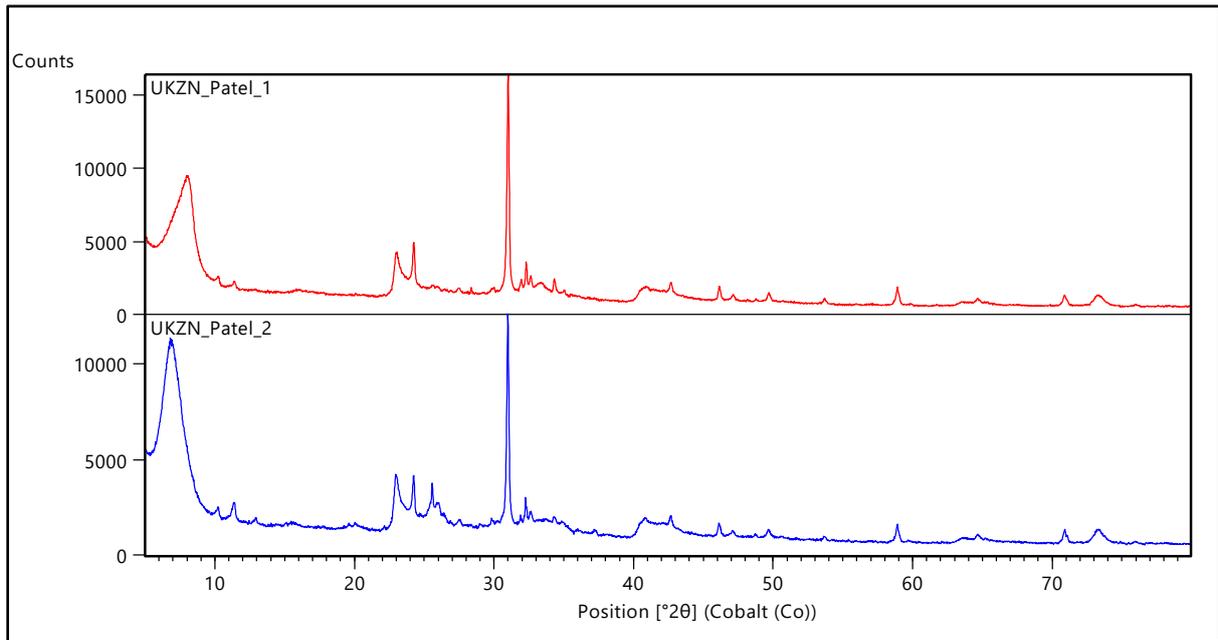
## 2.5 XRD

### Sample 1 – Activated Bentonite

% major minerals								
Calcite	Cristobalite	Diopside	Mordenite	Muscovite	Plagioclase	Quartz	Smectite12	Smectite14
1.05	0.1	1.73	2.47	2.64	6.7	18.6	45.32	21.39

### Sample 2 – Non activated bentonite

% major minerals								
Calcite	Cristobalite	Diopside	Mordenite	Muscovite	Plagioclase	Quartz	Smectite12	Smectite14
1.83	8.47	2.39	3.98	3.7	5.72	15.67	13.31	44.93



## 2.6 Atterberg Limits

### Liquid Limit

Activated Bentonite		Non-activated Bentonite	
Sample	Data (%)	Sample	Data (%)
A1	154	N1	137
A2	153	N2	139
A3	149	N3	134
CA1	130	CN1	130
CA2	133	CN2	132
CA3	142	CN3	128
CA4	139	CN4	141
CA5	148	CN5	139
Average	144	Average	135

### Plastic Limit

Activated Bentonite		Non-activated bentonite	
Sample	Data (%)	Sample	Data (%)
A1	50	N1	49
A2	48	N2	53
A3	51	N3	51
Average	50	Average	51

## Linear shrinkage

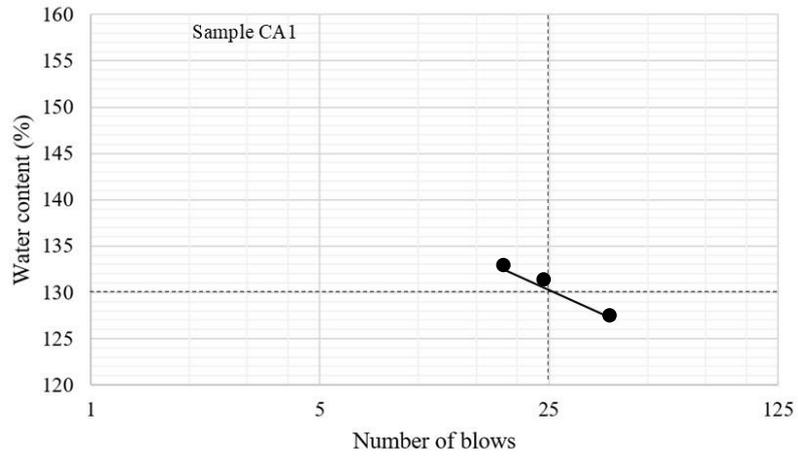
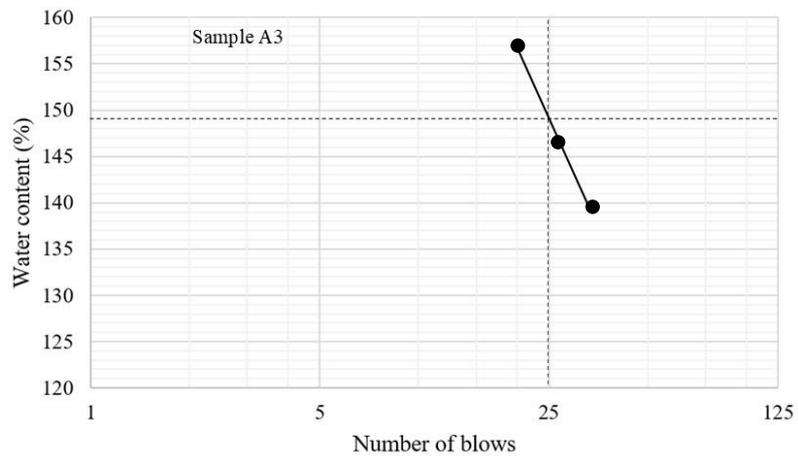
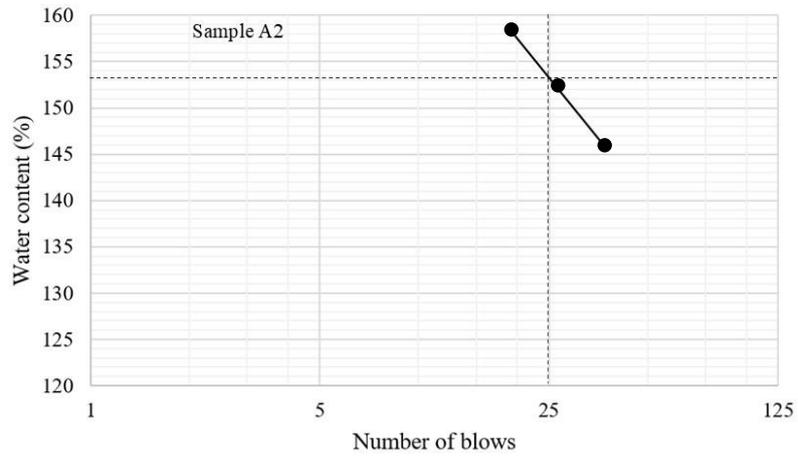
Activated Bentonite	Non-activated bentonite
25 %	25 %

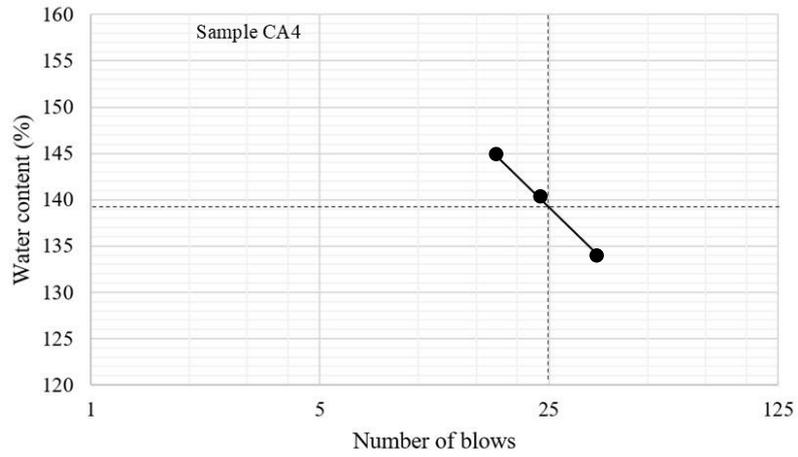
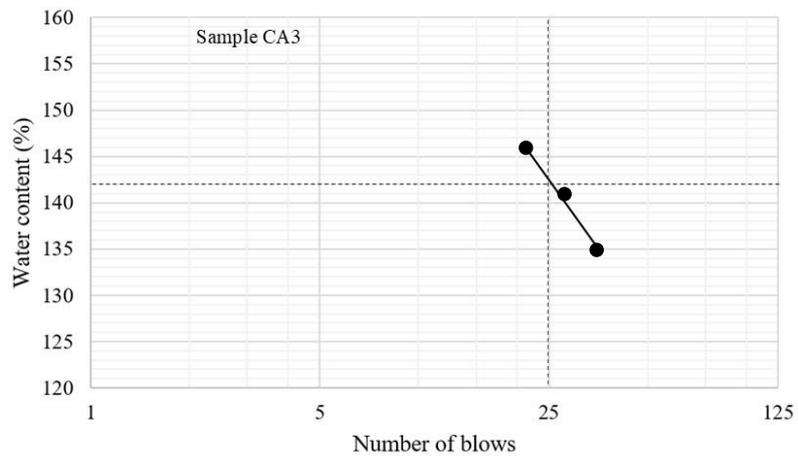
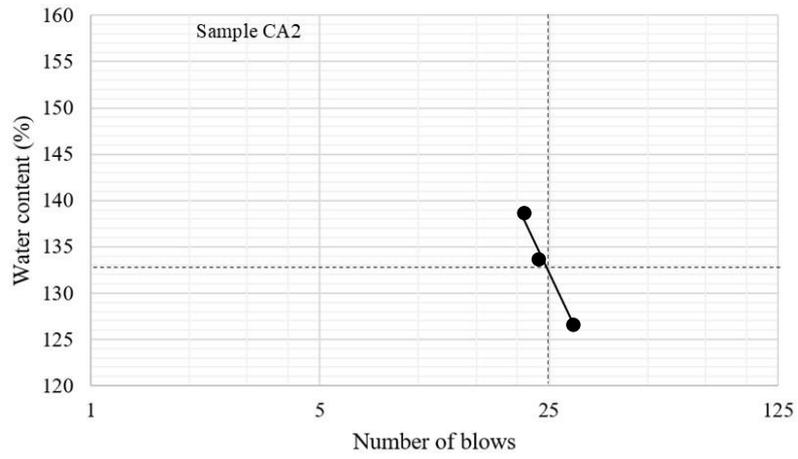
## 2.7 Liquid limit vs absorption

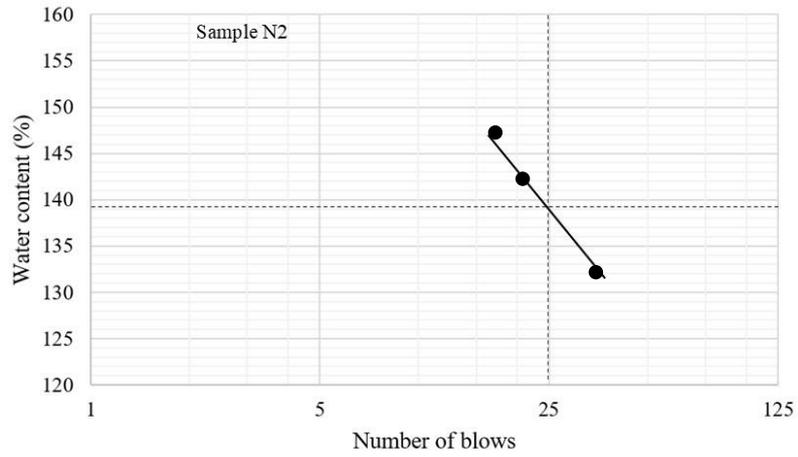
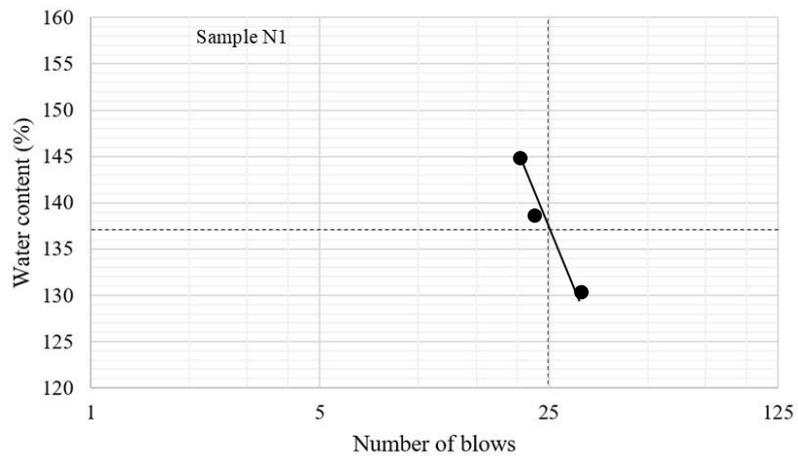
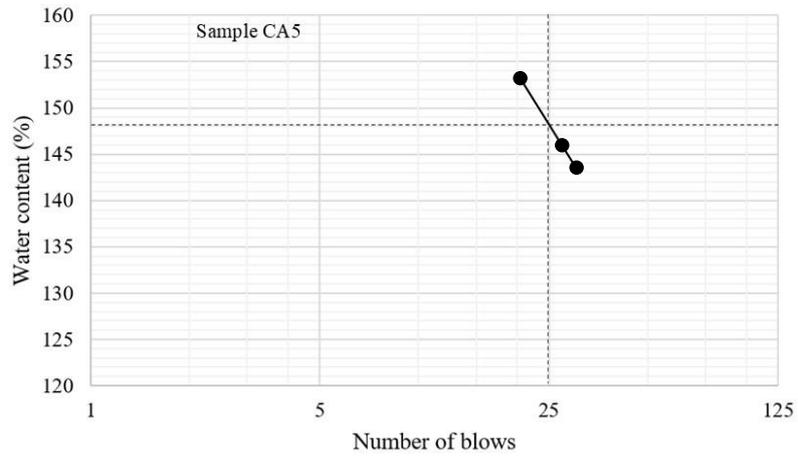
Activated bentonite	
Liquid Limit (%)	Absorption (%)
130	121
133	128
142	139
139	140
148	138
Non-Activated bentonite	
Liquid Limit (%)	Absorption (%)
130	115
132	120
128	122
141	119
139	126

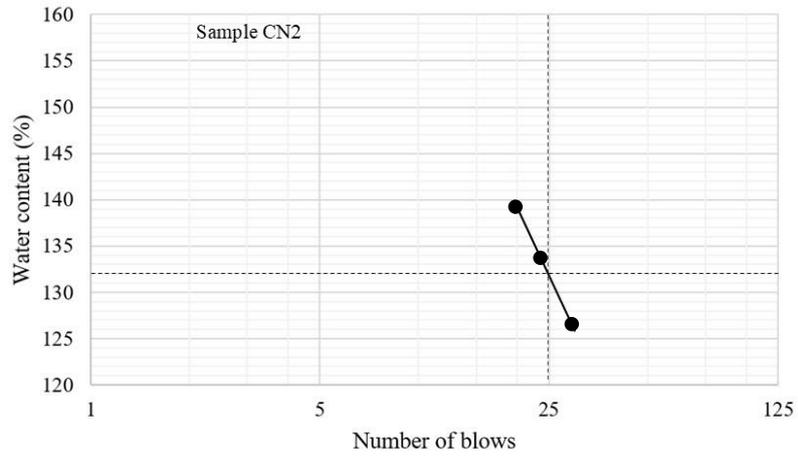
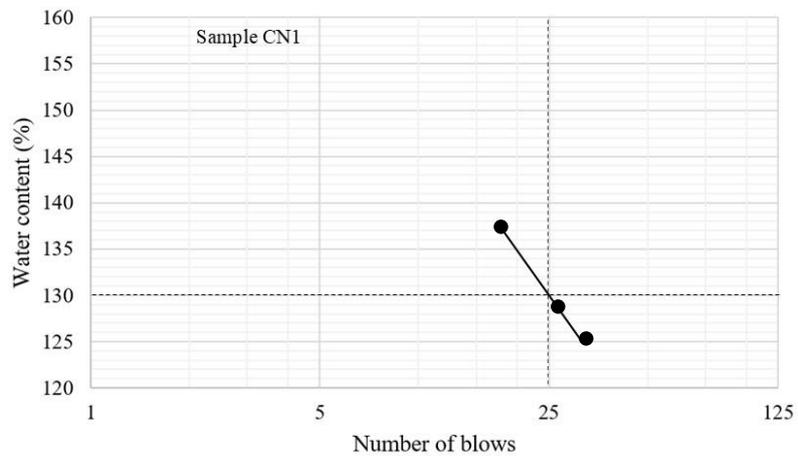
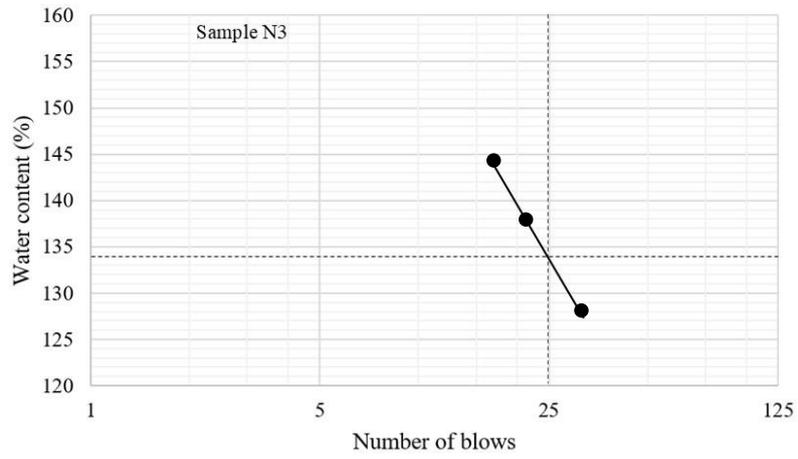
## **Appendix 3**

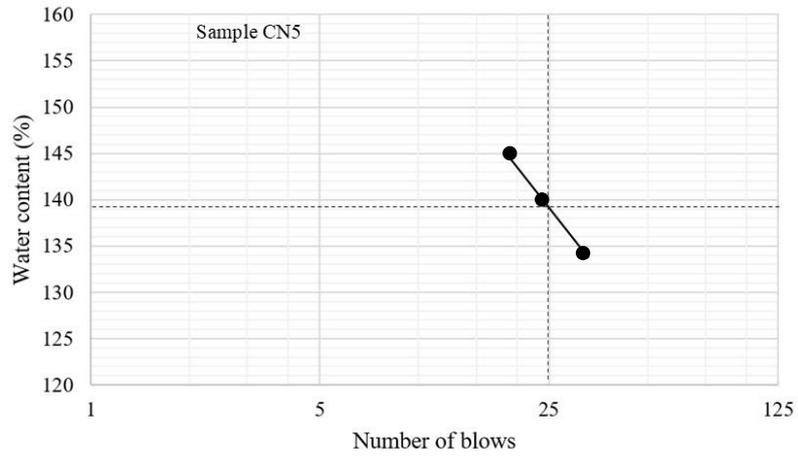
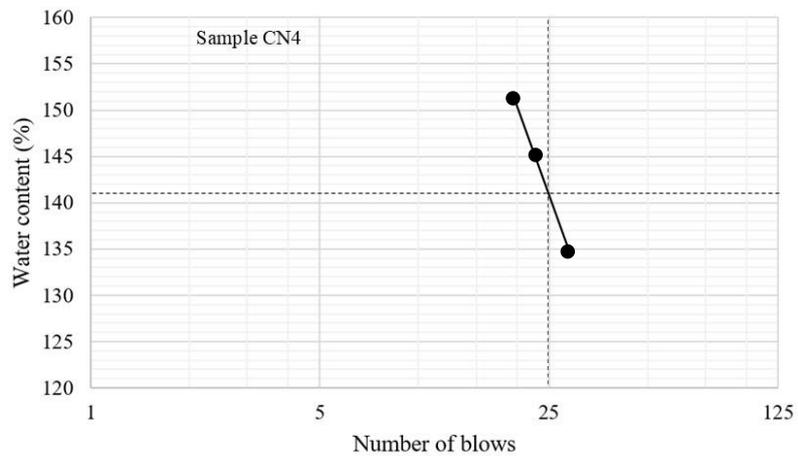
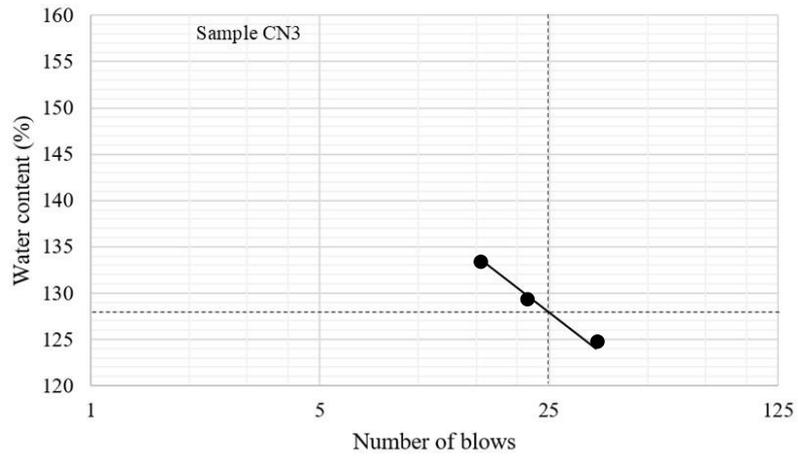
### **Liquid limit flow curves**











## **Appendix 4**

### **Test methods:**

4.1 Swell index

4.2 Fluid loss

4.3 Plate water absorption

# 4.1 Swell Index



Designation: D5890 – 11

## Standard Test Method for Swell Index of Clay Mineral Component of Geosynthetic Clay Liners<sup>1</sup>

This standard is issued under the fixed designation D5890; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers an index method that enables the evaluation of swelling properties of a clay mineral in reagent water for estimation of its usefulness for permeability or hydraulic conductivity reduction in geosynthetic clay liners (GCL).

1.2 It is adapted from United States Pharmacopeia (USP) test method for bentonite.

1.3 Powdered clay mineral is tested after drying to constant weight at  $105 \pm 5^\circ\text{C}$ ; granular clay mineral should be ground to a 100 % passing a 100 mesh U.S. Standard Sieve with a minimum of 65 % passing a 200 mesh U.S. Standard Sieve. The bentonite passing the 100 mesh U.S. Standard Sieve is used for testing after drying to constant weight at  $105 \pm 5^\circ\text{C}$ .

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *his standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards.<sup>2</sup>

D1193 Specification for Reagent Water

D4643 Test Method for Determination of Water (Moisture) Content of Soil by Microwave Oven Heating

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.04 on Geosynthetic Clay Liners.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E1 Specification for ASTM Liquid-in-Glass Thermometers  
E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E725 Test Method for Sampling Granular Carriers and Granular Pesticides

2.2 *United States Pharmacopeia Standard: USP-NF-XVII Bentonite*<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *geosynthetic, n*—a planar product manufactured from polymeric material used with soil, rock, earth, or other geotechnical engineering related material as an integral part of a man-made project, structure, or system.

3.1.2 *geosynthetic clay liner, n*—a factory manufactured hydraulic barrier consisting of clay supported by geotextiles, or geomembranes, or both, that are held together by needling, stitching, or chemical adhesives.

3.1.3 *moisture content, n*—that part of the mass of a geosynthetic clay liner that is absorbed water, compared to the mass of dry clay.

3.1.4 *oven-dried, adj*—the condition of a material that has been heated under prescribed conditions of temperature and humidity until there is no further significant change in its mass.

### 4. Significance and Use

4.1 Clay mineral is a major functional component of GCL systems that reduces the hydraulic conductivity of industrial, waste, or ground water through the liner.

4.2 Clay mineral quality can vary significantly and effect the hydraulic conductivity of the GCL composite. This test method evaluates a significant property of clay mineral that relates to performance.

### 5. Apparatus

5.1 *Mortar and Pestle or Laboratory Hammer Mill*, for grinding clay mineral to required particle sizing.

<sup>3</sup> Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852.

5.2 *U.S. Standard Sieve*, 100 mesh, 200 mesh, and automated sieve shaker.

5.3 *Drying Oven*, thermostatically controlled, preferably forced draft type, meeting requirements of Specification E145 and capable of maintaining a uniform temperature of  $105 \pm 5^\circ\text{C}$  throughout the drying chamber.

5.4 *Desiccator*, of suitable size containing indicator silica gel. It is preferable to use desiccant which changes color to indicate when it needs reconstitution.

5.5 *Laboratory Balance*, 100-g capacity,  $\pm 0.01$ -g accuracy and precision.

5.6 *Weighing Paper*, or small weighing dish.

5.7 *Glass Cylinder*, graduated TC (to contain), Class A volumetrically calibrated, with 1-mL subdivisions and ground glass stopper, high form with approximately 180-mm height from inside base to 100-mL mark.

5.8 *Wash Bottle*, for dispensing reagent water.

5.9 *Spatula*, flat-blade, to dispense clay mineral powder into cylinder; vibrating spatula should not be used since the delivery quantity may not be adequately controlled.

5.10 *Mechanical Ten-Minute Timer*.

5.11 *ASTM Calibration Immersion Thermometer*, (Specification E1).

5.12 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E145 and capable of maintaining a uniform temperature of  $105 \pm 5^\circ\text{C}$  throughout the drying chamber.

5.13 *Microwave Oven*—A microwave oven, preferably with a vented chamber, is suitable. The required size and power rating of the oven is dependent on its intended use. Ovens with variable power controls and input power ratings of about 700 W have been found to be adequate for this use. Variable power controls are important and reduce the potential for over heating the test specimen.

**NOTE 1**—Microwave ovens equipped with built-in scales and computer controls have been developed for use in drying soils. Their use is compatible with this test method.

5.14 *Balances*—All balances must meet the requirements of Specification D4753 and this section. A Class GP1 balance of 0.01 g readability is required for samples having a mass of up to 200 g (excluding mass of sample container).

5.15 *Sample Containers*, suitable containers made of material resistant to corrosion and change in mass upon repeated heating, cooling, exposure to materials of varying pH, and cleaning. Microwave sample containers should be microwave safe.

5.16 *Desiccator*, a desiccator cabinet or large desiccator jar of suitable size containing indicator silica gel. It is preferable to use a desiccant that changes color to indicate it needs reconstitution.

5.17 *Container Handling Apparatus*, gloves, tongs, or suitable holder for moving and handling hot containers after drying.

## 6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type I, II, or III (see Table X1.1). Such water is best prepared by distillation or the passage of tap water through an ion exchange resin.

6.2 Specification D1193 for reagent water, Type I, II, or III (see Table X1.1).

## 7. Hazards

7.1 Handle hot containers with a container holder.

7.2 Safety precautions supplied by the manufacturer of the microwave/oven should be observed.

7.3 Do not use metallic containers in a microwave oven (if used).

## 8. Sampling and Selection

8.1 Carry out sampling in accordance with Test Method E725.

## 9. Procedure

9.1 Grind the clay mineral sample to 100 % passing a 100 mesh U.S. Standard Sieve and a minimum of 65 % passing a 200 mesh U.S. Standard Sieve with a mortar and pestle or laboratory hammer mill as required.

9.2 The container to be used for drying should be oven dried thoroughly and subsequently placed into a desiccator until ready for use so that the tare weight of the container will be recorded.

9.3 Determine and record the tare of the specimen container.

9.4 Select representative test specimens.

9.5 Place the test specimen in the individual container. Determine the mass of the container and clay specimen as delivered using a balance selected on the basis of the sample mass. Record the value of the clay specimen.

**NOTE 2**—To prevent mixing of samples and yielding of incorrect results, all containers should be numbered and the container numbers shall be recorded on the laboratory data sheets.

9.6 Place the container with the clay specimen in the drying oven. Dry the clay specimen to a constant mass. Maintain the drying oven at  $105 \pm 5^\circ\text{C}$ . The time required to obtain constant mass will vary depending on the type of material, oven type and capacity, and other factors.

**NOTE 3**—In most cases, drying a test sample overnight (about 12 to 16 h) is sufficient for conventional ovens. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the change in mass after two successive periods (greater than 1 h) of drying is less than 0.1 %. In this case it should be verified that excessive drying does not influence the swelling performance of the clay. This can be done i.e. by comparing the swelling values after the first drying period (about 12 to 16 h) and the swelling values of bentonite being dried for a longer time period.

**NOTE 4**—If a microwave oven is used to dry the test specimen(s), the user of this test method should follow the drying procedures as stated in Test Method D4643. It is also recommended that the total mass of the test specimen(s) be a minimum of 100 g. It is further recommended to run a comparison test between the microwave oven and the drying oven to

demonstrate that the microwave oven gives similar values as the drying oven and that excessive drying does not change the swelling performance of the clay.

**NOTE 5**—Since some dry materials may absorb moisture from moist samples, dried samples should be removed before placing moist samples in the same oven. However, this would not be applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

9.7 After the material has dried to constant mass, remove the container from the oven (and replace the lid if used). Allow the material and container to cool to room temperature in a desiccation unit or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents or its being heated, or both. Determine the mass of the container and oven-dried material using the same balance as used previously. Subtract the tare of the container from the mass of the sample to determine the samples' constant dry mass. Record this value.

9.8 Weigh  $2.00 \pm 0.01$  g of dried and finely ground clay mineral onto a weighing paper.

9.9 Add 90 mL reagent water to the clean 100-mL graduated cylinder.

9.10 Remove not more than a 0.1-g increment of clay mineral with a volumetric spoon from weighing dish or paper and carefully dust it over the entire surface of water in the graduated cylinder over a period of approximately 30 s. Do not use a funnel that may concentrate the clay mineral in a poorly hydrated agglomerate. Allow the clay mineral to wet, hydrate, and settle to the bottom of the graduated cylinder for a minimum period of 10 min.

9.11 Add additional increments of the clay mineral powder in periods of 10 min, allowing the clay mineral to swell without air being trapped in between, following the details in 9.10, until the entire 2.00-g sample has been added.

9.12 After the final increment has settled, carefully rinse any adhering particles from the sides of the cylinder into the water column, raising the water volume to the 100 mL mark.

9.13 Place the glass stopper on the cylinder and allow it to stand undisturbed for a minimum of 16 h from the last incremental addition. After 2 h, inspect the hydrating clay mineral column for trapped air or water separation in the column. If present, gently tip the cylinder at a 45° angle and roll slowly to homogenize the settled clay mineral mass, allow the graduated cylinder with the hydrating clay mineral to remain undisturbed for a minimum of 16 h before recording the volume of the hydrated clay mass and its temperature.

9.14 After the minimum 16-h hydration period from the last increment addition, record the volume level in millilitres (mL) at the top of the settled clay mineral to the nearest 0.5 mL. Observe the distinct change in appearance at the upper surface of the settled clay mineral. Any low-density flocculated material (sometimes lighter in coloration to white) shall be ignored for this measurement. Record the observed volume of hydrated clay mineral.

**NOTE 6**—*Optional*—If a recognizable swelling still occurs after the minimum hydration period from the last increment addition (more than 10 % of the previous reading in a 4 hour period), continue recording the volume of the hydrated clay mass and its temperature to a maximum of 48 hours after the last increment addition.

9.15 Carefully immerse the thermometer and measure the temperature of the slurry. Record the temperature of the hydrated clay mineral to  $\pm 0.5^\circ\text{C}$ .

## 10. Report

10.1 Report the following information:

10.1.1 Source of clay mineral, including sample identification or lot number,

10.1.2 Method of sampling used,

10.1.3 ASTM standard test method number used to perform the test,

10.1.4 Any modifications to the test method or unusual observations which may effect the test results, and

10.1.5 Swell index as mL/2 g to the nearest 0.5 mL after the minimum 16-h hydration period from the last clay increment addition and if applicable to the maximum recorded hydration period from the last clay increment addition.

## 11. Precision and Bias

11.1 *Interlaboratory Test Program*—An interlaboratory study of the test method was run in 1999. The design of the experiment, similar to that of Practice E691. Seven different clay mineral samples were distributed to ten laboratories. Three sets of test results were generated for each sample by each of the laboratories.

11.2 *Test Results*—The precision information is given in Table 1. The average swell index values ranged from 20 to 36 for the seven clay mineral samples tested. However, since the statistics were not related to the magnitude of the test result, the precision values have been presented in terms of coefficients of variation, CV %.

11.3 *Bias*—The procedure in Test Method D5890 for measuring the swell index of clay mineral component of geosynthetic clay liners has no bias because the values of swell index can be defined only in terms of this test method.

## 12. Keywords

12.1 bentonite; clay; geosynthetic clay liner; microwave; oven drying; swell; swell index

**TABLE 1 Test Results**

Statistic	ILS Range
Within laboratory repeatability limit, CV % <sup>r</sup>	2 to 5 %
Between laboratory reproducibility limit, CV % <sup>r</sup>	7 to 22 %
95 % confidence limit	6 to 14 %
Within laboratory repeatability, 2.8 CV % <sup>r</sup>	
95 % confidence limit	20 to 61 %
Between laboratory reproducibility, 2.8 CV % <sup>r</sup>	

**APPENDICES**

**X1. Additional Reagent Information**

X1.1 See [Table X1.1](#).

**TABLE X1.1 Additional Reagent Information**

	Type I	Type II	Type III	Type IV
Electrical conductivity, max, $\mu\text{S}/\text{cm}$ at 298 K (25°C)	0.056	1.0	0.25	5.0
Electrical resistivity, min, $\text{M}\Omega\text{-cm}$ at 298 K (25°C)	18	1.0	4.0	0.2
pH at 298 K (25°C)	<sup>A</sup>	<sup>A</sup>	<sup>A</sup>	5.0 to 8.0
Total organic carbon (TOC), max, $\mu\text{g}/\text{L}$	100	50	200	no limit
Sodium, max, $\mu\text{g}/\text{L}$	1	5	10	50
Chlorides, max, $\mu\text{g}/\text{L}$	1	5	10	50
Total silica, max, $\mu\text{g}/\text{L}$	3	3	500	no limit
Microbiological contamination—When bacterial levels need to be controlled, reagent grade types should be further classified as follows:				
	Type A		Type B	
Maximum heterotrophic bacteria count	10/1000 mL		10/100 mL	
Endotoxin, EU/mL <sup>B</sup>	<0.03		0.25	
			100/10 mL	
			not applicable	

<sup>A</sup> The measurement of pH in Type I, II, and III reagent waters has been eliminated from this specification because these grades of water do not contain constituents in sufficient quantity to significantly alter the pH.

<sup>B</sup> EU = Endotoxin Units.

**X2. Check lists**



Added 90 ml of water to cylinder Yes  No

9.10 Added of no more than 0.1 g increments of clay (dusty addition) during approx. 30s onto the entire water surface (10 minute waiting period before next clay increment is added): Yes  No

9.11 Added remaining 0.1 g clay increments according to 9.10, without any air traps Yes  No

9.12 10 minutes after final clay increment, carefully rinsing with 10 ml of water Yes  No

9.13 Allow 16 hours to settle after final clay increment added. 2 hours hereafter inspect hydrating clay for e. g. trapped air. 45° cylinder tipping for homogenisation needed? Yes  No   
If tipped allow another 16 hours to settle.

9.14 Record volume to nearest 0.5 ml: \_\_\_\_\_ ml  
Recognizable swelling (more than 10% after 4 hours)? Yes  No   
If Yes record swell volume, water temperature and hours after last reading:  
Volume [ml] \_\_\_\_\_ water temperature °C or °F \_\_\_\_\_ time after 9.13 [h] \_\_\_\_\_

**10.1.4 Modifications / Other findings**

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Name and signature of person performing test Date: \_\_\_\_\_

Name, printed: \_\_\_\_\_ Signature: \_\_\_\_\_

Date of test performance \_\_\_\_\_

Name and address of lab performing test:

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## 4.2 Fluid loss



Designation: D5891 – 02 (Reapproved 2009)

### Standard Test Method for Fluid Loss of Clay Component of Geosynthetic Clay Liners<sup>1</sup>

This standard is issued under the fixed designation D5891; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 This test method covers an index method that enables the evaluation of fluid loss properties of a clay mineral film deposited on a filter paper from a 6 % solids slurry of clay mineral at 100-psi (-kPa) pressure as a measure of its usefulness for permeability or hydraulic conductivity reduction in geosynthetic clay liners (GCL).

1.2 This test method is adapted from American Petroleum Institute drilling fluid specifications for bentonite.

1.3 Powdered clay mineral is tested as produced; granular clay mineral should be ground to 100 % passing a 100 mesh U.S. Standard Sieve with a minimum of 65 % passing a 200 mesh U.S. Standard Sieve with the whole ground product used for testing.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*<sup>2</sup>

#### 2. Referenced Documents

- 2.1 *ASTM Standards*:<sup>3</sup>  
D1193 Specification for Reagent Water  
E1 Specification for ASTM Liquid-in-Glass Thermometers  
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method  
E725 Test Method for Sampling Granular Carriers and Granular Pesticides

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D35 on Geosynthetics and is the direct responsibility of Subcommittee D35.04 on Geosynthetic Clay Liners.

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<sup>2</sup> When bentonite is removed from a GCL product for testing, it may include adhesives that can influence test results.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

#### 2.2 *API Standards*:

API RP 131, Recommended Practice for Laboratory Testing of Drilling Fluids<sup>4</sup>

#### 3. Terminology

3.1 *Definitions*— For definitions of terms used in this test method, refer to API Standards and ASTM definitions for GCL products.

#### 4. Significance and Use

4.1 Clay mineral is the functional component of GCL that reduces the hydraulic conductivity of industrial waste or ground water through the liner.

4.2 Clay mineral quality can vary significantly and effect the hydraulic conductivity of the GCL composite. This test method evaluates a significant property of clay mineral that relates to performance.

#### 5. Apparatus

5.1 *Laboratory Balance*, 100 g capacity,  $\pm 0.01$ -g accuracy and precision.

5.2 *Weighing Paper*, or small weighing dish.

5.3 *Graduated Cylinder*, 500  $\pm$  5-mL graduated TD (to deliver) with 10-mL subdivisions, Class A volumetrically calibrated; 10  $\pm$  0.1-mL graduated cylinder, graduated TC (to contain) with 0.1-mL subdivisions.

5.4 *U.S. Standard Sieve*, 100 mesh, 200 mesh, and automated sieve shaker.

5.5 *Mortar and Pestle or Laboratory Hammer Mill*, for grinding clay mineral to required particle sizing.

5.6 *ASTM Calibration Immersion Thermometer*, 0 to 105  $\pm$  0.5°C (see Specification E1).

5.7 *Mixer*—11 000  $\pm$  300 rpm under load with single sine-wave impeller approximately 25 mm (1.0 in.) in diameter (mounted flash side up).<sup>5</sup> The impeller shall be replaced when it weighs a minimum of 5.1 g, from an original weight of about

<sup>4</sup> Available from American Petroleum Institute (API), 1220 L. St., NW, Washington, DC 20005-4070, <http://www.api.org>.

<sup>5</sup> For example, Sterling Multimixer Model 9B with 9B29X impeller blades available from Fann Instrument Co., P.O. Box 4350, Houston, TX 77210, has been found suitable for this purpose.

5.5 g. New blades will be weighed prior to installation in order to ensure conformance to manufacturing criteria. Mixer speed under sample loading shall be determined and documented once every 90 days unless the manufacturer has documented objective evidence to extend calibration time.

NOTE 1—Sterling Multimixer Model 9B with 9B29X impeller blades or equivalent may be obtained from the suppliers given in Footnote 9.

5.8 *Mixing Container*—Approximate dimensions are 180 mm (7 in.) deep, 97-mm (3<sup>13</sup>/<sub>16</sub>-in.) inner diameter at top, and 70-mm (2<sup>3</sup>/<sub>4</sub>-in.) inner diameter at bottom.<sup>6</sup>

NOTE 2—Mixing containers or equivalent may be obtained from the suppliers given in Footnote 8.

5.9 *Timers*, 30 min, two interval, mechanical or electrical, precision  $\pm 0.1$  min.

5.10 *Spatula*, flat blade, to dislodge clay mineral clumps adhering to the mixing container walls.

5.11 *Covered or Sealed Container*, of 400 to 600-mL capacity.

5.12 *Ambient Temperature/Low-Pressure Filter Press*, conforming to API RP 131, Section 3.2. This filter press consists mainly of a cylindrical cell having an inside diameter of 76.2 mm (3 in.) and a height of at least 64.0 mm (2.5 in.). This chamber is made of materials resistant to strongly alkaline solutions, and is so fitted that a pressure medium can be conveniently admitted into and bled from the top. Arrangement is also such that a sheet of 90-mm filter paper can be placed in the bottom of the chamber just above a suitable support. The filtration area is  $4580 \pm 60 \text{ mm}^2$  ( $7.1 \pm 0.1 \text{ in}^2$ ). Below the support is a drain tube for discharging the filtrate into a graduated cylinder. Sealing is accomplished with gaskets, and the entire assembly supported by a stand. A mini-press or half-area press does not directly correlate with the results obtained when using the above described standard-sized press. Pressure can be applied with any nonhazardous fluid medium, either gas or liquid. Presses are equipped with pressure regulators and can be obtained with portable pressure cylinders, midget pressure cartridges, or means of utilizing hydraulic pressure.

NOTE 3—Ambient temperature/low-pressure filter press conforming to API RP 131, Section 3.2, or equivalent, may be obtained from the suppliers given in Footnote 9.

5.13 *Filter Paper, 90-mm*, very dense, hardened with smooth lint-free surface, must be used.<sup>7</sup> These papers have high wet strength permitting application of high pressure during filtration. They also have good resistance to alkalis and acids.

## 6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, references to water shall be understood to mean reagent water

<sup>6</sup> For example, Hamilton Beach Mixer Cup No. M110-D, or equivalent, has been found suitable for this purpose. Mixing containers supplied by Fann Instrument Co., P.O. Box 4350, Houston, TX 77210.

<sup>7</sup> For example, Whatman No. 50, S & S No. 576, or equivalent, have been found suitable for this purpose.

conforming to Specification D1193, Type I, II, or III. Such water is best prepared by distillation or the passage of tap water through an ion-exchange resin.

6.2 Specification D1193 for reagent water, Type I, II, or III.

## 7. Hazards

7.1 *Safety Precautions*—Establish appropriate safety and health practices for high-pressure equipment prior to use.

## 8. Sampling and Selection

8.1 Conduct the sampling in accordance with Test Method E725.

## 9. Procedure

9.1 Grind the clay mineral sample to greater than 100 % passing a 100 mesh U.S. Standard Sieve, and a minimum of 65 % passing a 200-mesh U.S. Standard Sieve with a mortar and pestle or laboratory hammer mill as required.

9.2 Weigh  $22.50 \pm 0.01$  g of the whole composite of finely ground clay mineral with “as received” moisture, typically 5 to 10 %, onto a weighing paper. If bentonite is removed from a GCL product, the bentonite would be dried to less than 10 % moisture prior to weighing.

9.3 Measure  $350 \pm 5$  mL of reagent water with the 500- mL graduated cylinder and added to the mixing cup. Place the cup on the mixer, and add the clay mineral slowly over approximately 30 s.

9.4 After stirring for  $5 \pm 0.5$  min, remove the container from mixer, and scrape its sides with the spatula to dislodge any clay clinging to the container wall. Ensure that all of the dislodged clay mineral clinging to the spatula is incorporated into the suspension.

9.5 Replace the container on the mixer, and continue to stir for a cumulative total stirring time of  $20 \pm 0.1$  min. The container may need to be removed from the mixer and the sides scraped to dislodge any clay clinging to container walls after another 5 or 10 min of stirring.

9.6 Age the clay mineral suspension for a minimum of 16 h in a sealed or covered container at ambient temperature. Record the initial temperature, final temperature, and actual hydration aging time.

9.7 After aging the clay mineral suspension, shake vigorously to break its gel strength, and then pour the suspension into the mixer container. Stir the suspension on the mixer for  $5 \pm 0.5$  min to completely disperse the clay mineral slurry.

9.8 Assemble the dry filter cell with filter paper and gaskets, and immediately after remixing the clay mineral slurry, pour it into the filter cell and complete assembly of the filter cell. Place the filter cell in the filter frame and close the relief valve. Place a 10 mL graduated cylinder under the filter cell drain tube.

9.9 Set one timer for  $7.5 \pm 0.1$  min and the second timer for  $30 \pm 0.1$  min. Start both timers and adjust pressure on the fluid loss cell to  $100 \pm 2$  psi. Starting the timers and adding 100 psi pressure should be completed in less than 15 s. Supply pressure by compressed air, nitrogen, helium, or carbon dioxide.

9.10 At  $7.5 \pm 0.1$  min on the first timer, remove the graduated cylinder and any adhering liquid on the drain tube, and discard. Immediately place a clean dry 10-mL graduated cylinder under the drain tube, and collect the fluid for  $22.5 \pm 0.1$  min to the end of the second timer. This corrects the fluid loss value for any initial unpredictable spurt loss from the fluid loss cell. Remove the graduated cylinder after the second time interval and record the volume of fluid collected.

**10. Calculation**

10.1 Calculate the fluid loss in millilitres using Eq 1:

$$\text{Fluid loss volume} = 2(\text{mL filtrate volume for last 22.5min. interval}) \text{ mL} \quad (1)$$

**11. Report**

11.1 Report the following information:

- 11.1.1 Source of clay mineral, including sample identification or lot number,
- 11.1.2 Method of sampling used,
- 11.1.3 ASTM Test Method number used to perform the test,
- 11.1.4 Any modifications to the test method or unusual observations which may effect the test results,
- 11.1.5 Calculated fluid loss as millilitres to the nearest 0.1 mL, and
- 11.1.6 Temperature of the slurry at the start and completion of the test to the nearest 0.5°C.

**12. Precision and Bias**

12.1 *Interlaboratory Test Program*—An interlaboratory study of the test method was run in 1999. The design of the experiment, similar to that of Practice E691. Seven different clay mineral samples were distributed to seven laboratories. Three sets of test results were generated for each sample by each of the laboratories.

12.2 *Test Results*—The precision information is given in Table 1. The average fluid loss values ranged from 9 to 22 for the seven clay mineral samples tested. However, since the statistics were not related to the magnitude of the test result, the precision values have been presented in terms of coefficients of variation, CV %.

12.3 *Bias*—The procedure in Test Method D5891 for measuring the fluid loss value of the clay mineral component of geosynthetic clay liners has no bias because the values of swell index can be defined only in terms of this test method.

**TABLE 1 Test Results**

Statistic	ILS Range
Within laboratory repeatability limit, CV % <sup>r</sup>	1.8 to 4.7 %
Between laboratory reproducibility limit, CV % <sup>R</sup>	6 to 18 %
95 % confidence limit	5 to 13 %
Within laboratory repeatability, 2.8 CV % <sup>r</sup>	
95 % confidence limit	11.8 to 51 %
Between laboratory reproducibility, 2.8 CV % <sup>R</sup>	

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# 4.3 Plate absorption

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## Standard Test Method for Water Absorption of Bentonite by the Porous Plate Method<sup>1</sup>

This standard is issued under the fixed designation E 946; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of water absorbency of bentonite.

1.2 Bentonite is a montmorillonite clay material. It is added to moist iron-ore concentrates as a binder in forming balls prior to heat treatment in the agglomerating process for making iron-ore pellets.

1.3 The values as stated in SI units are to be regarded as the standard. The values in parentheses are given for information only.

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 1193 Specification for Reagent Water<sup>2</sup>
- E 1 Specification for ASTM Thermometers<sup>3</sup>
- E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens<sup>4</sup>
- E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal Bearing Ores and Related Materials<sup>5</sup>
- E 832 Specification for Laboratory Filter Papers<sup>4</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *absorption*—water absorbed by the bentonite expressed as a percentage of the dry weight of the bentonite sample.

3.1.2 *bentonite*—montmorillonite-form of absorptive colloidal clay used in processing iron-ore concentrates in preparation for heat-hardening.

3.1.3 *plate*—sintered alumina plate.

### 4. Summary of Test Method

4.1 The amount of water absorbed over an 18-h period by a 2-g sample of processed bentonite is determined gravi-

metrically by difference under specific fixed conditions and is calculated from the absorption equation in 11.2.

### 5. Significance and Use

5.1 The effectiveness of the bentonite is partially due to its water absorption capacity that relates to the quantity of the bentonite required to reach desired wet- and dry-ball strengths. The absorption test relates to the effectiveness.

5.2 The absorption test aids the supplier in determining his mining and processing requirements for meeting specifications. It aids the user in evaluating the effectiveness of the bentonite in the balling process.

5.3 This test method is not to be used to determine the effectiveness of the use of additives to bentonite to enhance the water absorbing properties.

### 6. Technical Hazards

6.1 Several conditions materially affect the test and these conditions are as follows:

- 6.1.1 Accumulation of soluble salts and fungal growth in the water affects the absorption properties of bentonite,
- 6.1.2 Absorption is temperature sensitive,
- 6.1.3 Absorption value is affected by time,
- 6.1.4 Loading density in grams per square centimetre has an important bearing on the sensitivity of the test, and
- 6.1.5 Water level has a significant effect on test results. It is, therefore, mandatory that the procedure for the test be adhered to carefully.

### 7. Apparatus (See Fig. 1)

7.1 *Sintered Medium Coarse Aluminum Oxide Plate*, 12 by 12 by 1-in.<sup>6</sup>, having 19 permeability units per plate, an approximate pore size of 164  $\mu\text{m}$  and a particle retention of 68  $\mu\text{m}$ .

7.2 *Inert Support Pad*, 1/4-in. thick by 1-in.<sup>2</sup> (4 required).

7.3 *Shallow Flat Base Glass Pan*, approximately 16 by 16 by 3 in.<sup>7</sup>

7.4 *Glass Cover*, 17 by 17 in. having a soft gasket strip to form a seal against the pan.

7.5 *Hardened Filter Paper*, 9-cm diameter. See Specification E 832.

7.6 *Drying Oven*, controllable to 105°C. See Specification E 145.

7.7 *Balance*, having a  $\pm 0.001$ -g sensitivity.

7.8 *Tared Covered Containers*, 10-cm diameter.<sup>8</sup>

7.9 *Thermometer*, accurate to  $\pm 1.0^\circ\text{C}$ . See Specification E 1.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>6</sup> Norton Industrial Ceramics Division, 50 N.W. Bond St., Worcester, MA 01606, Product #P-260 has been found satisfactory for this purpose.

<sup>7</sup> Glass pans are available from Scientific Supply Houses.

<sup>8</sup> Reference Fischer Scientific Catalogue 81, Reusable Petri Dish, Catalog No. 08-747-C, 100 by 15-mm diameter set has been found satisfactory for this purpose.

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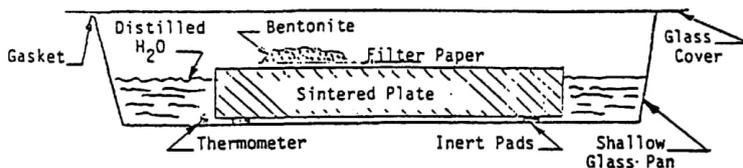


FIG. 1 Example of an Apparatus

7.10 *Washer-Type Template*, with a 5-cm diameter cut out for use in depositing the bentonite on the 9-cm filter paper.

**8. Materials**

8.1 *Bentonite Samples*, from the supplier or user inventory, ground to approximately 80 % of minus 200 mesh in accordance with Test Method E 276 and dried to a constant weight at 105°C (221 °F). The bentonite is described in 1.2.

8.2 *Distilled Water*—See Specification D 1193, Type II.

**9. Procedure**

9.1 Place a clean alumina plate that is supported by ¼-in. thick inert pads in the center of the pan and ensure the top of the plate is level. Before each test, add distilled water to within ¼ in. of the top of the plate, measuring to the water-plate contact, at least ⅜ in. away from the edge of the plate, then place the glass cover over the pan.

9.2 Allow the system sufficient time to reach ambient temperature.

9.3 Determine the average weight of a hydrated filter paper from the lot used in the absorbency test, by allowing four papers to absorb water on the alumina plate for 18 h and then weighing the papers.

9.4 Weigh 2 g (±1 mg) of processed bentonite, previously dried to constant weight at 105°C (220°F), onto the dry filter paper and spread it evenly within the 5-cm circle of the template on the 9-cm diameter paper. This can best be accomplished by using a vibrating spatula.<sup>9</sup> Remove the template and brush any remaining particles on top of the sample. Remove the template.

9.5 Place the paper and bentonite on the alumina plate. Up to four samples can be placed on the plate at one time.

9.6 Record the initial water temperature.

9.7 After 18 h, record the final water temperature. Carefully lift the filter paper and place it in a preweighed covered glass dish. Place a cover on the dish until ready to weigh.

9.8 Weigh the covered glass dish containing the hydrated paper and the hydrated bentonite.

<sup>9</sup> Vibrating spatula manufactured by Mettler has been found satisfactory for this purpose.

TABLE 1 Derivation of Temperature Correction Factor, K<sup>a</sup>

Sample Number	77-219	77-218	77-230	78-292	76-40
18-h Average Water Temperature, °F	63	73	82		
(°C)	(17.2)	(22.8)	(27.8)		
Water Absorption, Weight, %	664	650	671	676	907
	938	919	932	942	756
	768	795	795	781	745
	753	759	767	787	606
	610	620	624		
Regression Analysis Slope, weight, %, °C	3.14	4.14	3.13	2.83	3.26

<sup>a</sup> Average temperature correction factor (slope) = 3.30 % weight/°C and 1.83 % weight/°F.

9.9 Following each test, the water should be changed and the plate and pan cleaned.

**10. Calculation**

10.1 Determine the net weight of the hydrated bentonite by subtracting the glass dish weight and the average weight of the hydrated paper from the total weight.

10.2 Calculate and report the absorbed moisture, as to the percent weight increase, over the dry bentonite weight as follows:

$$\text{Absorption, \%} = \frac{(Ww - Wd)}{Wd} \times 100 - (K(Ta - Tr))$$

where:

- Absorption = water absorption (% weight) at 20°C/18 h,
- Ww = net weight of hydrated bentonite, g,
- Wd = net dry weight of dry bentonite, g,
- Ta = average of initial and final water temperature, °C,
- Tr = reference temperature = 20°C, and
- K = temperature coefficient (weight, %/°C) = 3.30.

**11. Precision and Bias**

11.1 No statement is made about either the precision or the bias of Test Method E 946 since the result merely states whether there is conformance to the criteria for success specified in the procedure.

**12. Keywords**

12.1 bentonite; gravimetric; water absorption

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