الجمهورية الجزائرية الديمقراطيـة الشعبيــة
People's Democratic Republic of Algeria
وزارة التـعليـم العالـي والبحـث العلمــــي
Ministry of Higher Education and Scientific Research
جامعة عين تموشنت بلحاج بوشعيب
University of Ain Temouchent - Belhadj Bouchaib
Faculty of Science and Technology
Department of Civil Engineering and Public Works

Final Year Project
For the attainment of the Master's degree
Field : science and technology
Stream: Public works
Specialization: Roads and civil engineering structures.
Topic:

Physico-chemical and mechanical characterization of Maghnia bentonite

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Academic year 2023/2024

ملخص

تُستخدم البنتونيت على نطاق واسع في مختلف مجاالت الهندسة المدنية، ال سيما لخصائصها في العزل واللزوجة. تهدف هذه الرسالة إلى توصيف الخصائص الفيزيائية والكيميائية والميكانيكية للبنتونيت الخام والصناعي الهدف هو تحديد دور البنتونيت في عمليات الحفر SEMتم إجراء اختبارات تعريفية على كل من البنتونيت الخام والصناعي لتحديد خصائصها الرئيسية. وتم إجراء تحليالت حيود األشعة السينية(لفحص البنية المورفولوجية والمعدنية للتربة. وتم أيًضا)XRD المجهر اإللكتروني الماسح(و) .إجراء اختبار محدد لتقييم ظاهرة اللزوجة لكل من نوعي البنتونيت أظهرت االختبارات أن البنتونيت الصناعي أكثر فعالية في عمليات الحفر من البنتونيت الخام، مع مرشح ترشيح أكثر مقاومة للماء وتأثير لزوجة أسرع. وهذا يشير إلى أن البنتونيت الصناعي أكثر كفاءة في تطبيقات الحفر

الكلمات المفتاحية البنتونيت – توصيف – تكسية – حفر – طين – بروكتور

Abstract :

The Bentonite is a material widely used in various civil engineering fields, particularly for its sealing and thixotropic properties. This thesis aims to characterize the physical, chemical, and mechanical properties of both raw and industrial bentonite.
The Objectiveis To determine the role of bentonite in drilling operations.

Identification tests were conducted on both raw and industrial bentonite to determine theirmain characteristics. SEM (Scanning Electron Microscopy) and XRD (X-ray Diffraction)analyses were performed to examine the soil's morphology and mineralogical structure. Aspecific test was also conducted to evaluate the thixotropy phenomenon of both types of bentonite.
The tests revealed that industrial bentonite is more effective for drilling than raw bentonite,with a more impermeable filtration filter and a faster thixotropy effect. This indicates that industrial bentonite is more efficient in drilling applications.

Key words : bentonite, characterization, thixotropy, drilling, clay, proctor.

Résumé :

La Bentonite est un matériau très utilisé dans divers domaines du génie civil, notamment pour ses propriétés d'étanchéité et thixotropes. Cette thèse vise à caractériser les propriétés physiques, chimiques et mécaniques de la bentonite brute et industrielle.
L'objectif est de déterminer le rôle de la bentonite dans les opérations de forage.
Des tests d'identification ont été réalisés sur la bentonite brute et industrielle afin de déterminer leurs principales caractéristiques. Des analyses MEB (microscopie électronique à balayage) et XRD (diffraction des rayons X) ont été réalisées pour examiner la morphologie et la structure minéralogique du sol. Un essai spécifique a également été réalisé pour évaluer le phénomène de thixotropie des deux types de bentonite.
Les tests ont révélé que la bentonite industrielle est plus efficace pour le forage que la bentonite brute, avec un filtre de filtration plus imperméable et un effet thixotropique plus rapide. Cela indique que la bentonite industrielle est plus efficace dans les applications de forage.

Mots clés : bentonite, caractérisation, thixotropie, forage, argile, proctor**.**

Dedication

Dedication

First and foremost, I would like to express my gratitude to the Creator of the universe for endowing us with intelligence and keeping us in good health. We have successfully completed this year of study, and now it is time for me to share my joy and sincere dedications with all those who have supported and encouraged me.

This work represents the culmination of hard work and many sacrifices. I would like to extend my heartfelt thanks to the supervisor of this thesis, Professor Aïssa Mamoun Sidi Mohammed, for his constant support, insightful guidance, and encouragement throughout this project.

To my family, my parents, my brother, and especially my sisters, I am grateful for their help, courage, availability, knowledge, and patience. Their unwavering support and constant presence have been a source of inspiration and motivation, enabling me to find confidence in myself.

I also wish to thank all the professionals and friends who took the time to answer my various questions in the field, thereby facilitating the elaboration of this work.

Finally, a big thank you to the mechanical engineering administration for providing the quality tools necessary for my specialization through their program.

Acknowledgments

Acknowledgments

First and foremost, we thank God for helping us and giving us the strength and willpower necessary to complete this modest work.

I particularly want to express my gratitude to Mr. Aïssa Mamoun Sidi Mohammed for supervising this thesis, for the trust he placed in me, and for his invaluable advice.

Our deepest thanks also go to Dr. Guellil Mustapha for the honor he has done us by presiding over this jury, as well as to Mrs. Dounane Nawel for agreeing to be a member of it.

We also warmly thank La FARGE OUGAZ for allowing us to conduct the XRD test, and the University of Tlemcen for granting us permission to perform the SEM test.

Finally, we extend our gratitude to all the individuals who, directly or indirectly, contributed to the completion of this work.

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List of abbreviations

i. List of abbreviations

CEC: cation exchange capacity

AIPEA : the international association for stady of clays

PILC : pillared interlayered clays

CBR: the california bearing ratio

IBI : Immediate bearing index

ERD : extendend reach drilling

PDC : polycristalline diamond compact

BOP : blow out preventer

HPHT : high pressure high temperature

CP : poise or centipoise

KCI : potassium chloride

SEM : scaning electron microscopy

XRD : X-ray diffraction

EDX : energy dispersive x-ray

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General Introduction

v. General Introduction

The role of bentonite in drilling operations is crucial due to its unique properties and diverse applications. Bentonite, a clay characterized by its remarkable thixotropic properties, is essential for improving the efficiency and performance of drilling processes. This research aims to delve into the various aspects and benefits of using bentonite in drilling, focusing on its structural and compositional characteristics as well as its thixotropic behavior.

The first chapter provides an overview of clays, with particular attention to bentonite, including its definition, properties, and general uses. The second chapter is dedicated to the characterization of bentonite, encompassing its identification and various tests conducted according to French standards, such as Proctor tests, sedimentometry, gamma rays, methylene blue, and calcium carbonate (CaCO3) tests. These tests help understand the properties of bentonite and its behavior under different conditions.

The third chapter delves into the basics of drilling, including its definition, types, and various techniques used. It also examines the role of bentonite in drilling, its use, and importance. We then define the thixotropic phenomenon test of bentonite and measure this phenomenon theoretically.

Finally, in the fourth chapter, Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) tests are conducted to analyze the morphology and mineralogical composition of bentonite. We then compare the thixotropy of raw bentonite with that of industrial bentonite to assess their impact on drilling. For industrial bentonite, a heat treatment was applied, showing positive effects in terms of time savings and increased efficiency in drilling.

Chapter 1

Clays and Bentonites

1. **Introduction**:

Clays, essential members of the vast realm of natural materials, have long intrigued scientists, engineers, and researchers due to their remarkable complexity and diversity. Their ubiquitous presence in soils, rocks, and sediments makes them key players in a multitude of geological, environmental, and industrial processes. This chapter delves into one of the most fascinating and widely utilized types of clays: montmorillonite clays, with a particular focus on bentonite.

Firstly, we will explore the essence of clays, examining their geological origin and the complex processes that shape them over time. We will delve into the depths of their formation, whether sedimentary, metamorphic, or residual, to understand the factors that determine their composition, structure, and unique properties.

Next, we will concentrate on montmorillonite clays, with a specific emphasis on bentonite. We will analyze in detail the complex crystalline structure of these clays, highlighting their characteristic atomic arrangements that confer special properties such as expansion, plasticity, and adsorption. Understanding this subtle architecture is essential for comprehending the various applications of bentonite in diverse fields such as geotechnics, pharmacy, cosmetics, and more.

Subsequently, we will explore the physical and chemical properties of bentonite, examining how they influence its behavior and performance in different environments and applications. We will also discuss the main types of bentonite, highlighting their variations in terms of mineralogical composition, geological origin, and intrinsic properties.

Finally, we will address a particularly intriguing aspect of montmorillonite clays: intercalated clays. These complex structures, where water molecules or other substances insert between the clay layers, offer unique opportunities for modifying and functionalizing the properties of bentonite, paving the way for new applications and innovations in various fields.

In summary, this chapter provides an in-depth dive into the fascinating world of montmorillonite clays, highlighting the crucial role of bentonite and exploring the multiple facets of its origin, structure, properties, and applications. By better understanding these versatile and complex materials, we can fully exploit their potential to address contemporary technological challenges and shape a sustainable and innovative future. [15]

2. **Overview of Clays :**

2.1. Definition of Clays :

Clays are fine mineral substances. Their name derives from the Greek word 'argilos', meaning 'white'. According to G. Millot, ceramists classify materials based on their reaction to heat. These minerals are aluminum silicates, composed of a stacking of layers, earning them the name of phyllite or fibrous aggregates, with an average size of about 2 micrometers. The presence of electric charges on the surface as well as the exchange of cations between the layers are the main factors contributing to the hydration and swelling of clays. [2]

Figure 1.1 Clay Samples [14]

2.2. Charges on Clay Surfaces :

Clay minerals exhibit a non-neutral electrical surface. Two types of charges are observed: [2]

A permanent or structural charge resulting from ionic substitutions (such as Al3+ replacing Si4+ in the tetrahedral layer, or Mg2+ or Fe2+ replacing Al3+ in the octahedral layer), typically negative.

A variable surface charge depending on the pH of the environment, arising from chemical reactions on the mineral surface or the adsorption of cationic or anionic surfactants.

2.3. Classification of Clay Minerals:

The classification of clays according to the International Association for the Study of Clays (AIPEA) and other international authors. Phyllosilicates, which are clay minerals, are classified based on several criteria : [4]

2.3.1. Types of Layers :

Phyllosilicates can have layers arranged in different configurations, notably 2:1 (two layers of silica tetrahedra surrounding one layer of hydroxide octahedra) or 1:1 (a single layer of silica tetrahedra). This difference in layer structure affects the properties of the minerals.

2.3.2. Overall Charge of the Layer :

The layers of phyllosilicates can have an overall negative, positive, or neutral charge, depending on the chemical composition and ion substitution in the crystalline structure. This charge influences the interaction of clays with other chemical compounds and their behavior in different environments.

2.3.3. Nature of Interlayer Cations :

Between the layers of phyllosilicates, there may be interlayer spaces where ions can be present. The nature of these interlayer ions can vary and have an impact on the physical and chemical properties of clays.

The most common classification of clays is often based on the thickness and structure of the layers. According to this classification, there are generally four main groups of clays, which can be distinguished based on their structural characteristics and chemical composition.

1) Minerals with 7 Å :

They consist of alternating layers, with a tetrahedral layer containing silicon occupying certain sites, juxtaposed with an octahedral layer where aluminum occupies the sites. The distance between the layers is approximately 7 angstroms. In the tetrahedral layer, each SiO4-4 tetrahedron is connected to its neighbors by three of its vertices, while the fourth vertex binds to the cation of the octahedral layer. [5]

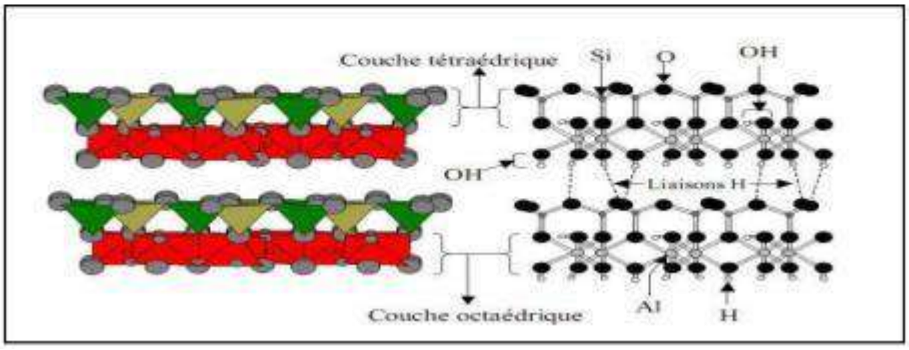
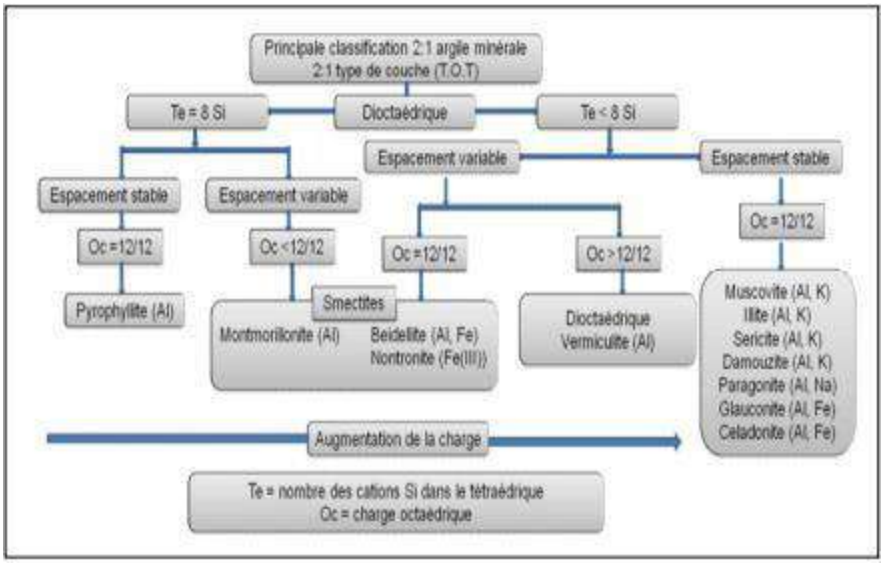


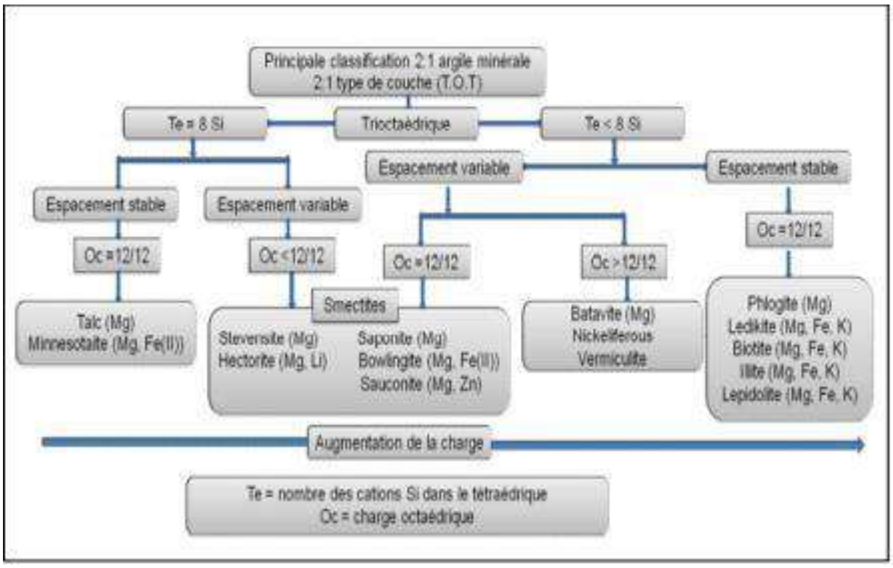
Figure 1.2 Representation of Silica Tetrahedral and Alumina Octahedral Stackings (case of kaolinite) [3]

2) Minerals with 10 Å :

The layer consists of two tetrahedral layers and one octahedral layer, thus characterizing a T:O:T or 2:1 type structure. Its dimension is approximately 10 angstroms in thickness. The various clays, classified according to the T-O-T elementary structure, are presented in Figures 2a and 2b. [3]



(a) Dioctahedral T-O-T clay minerals



(b) Trioctahedral T-O-T clay minerals

Figure 1.3: Classification of T-O-T clay minerals [6]

3) Minerals with 14 Å :

The layer consists of alternating T:O:T layers with interlayer octahedral layers. Its thickness is approximately 14 angstroms. [3]

4) Interstratified Minerals :

The thickness of the layer varies. These minerals are the product of the mixture, whether regular or irregular, of clays. [3]

3. **Montmorillonite Clays (Bentonites)** :

Basic chemical formula: 4SiO2 Al2O3 2H2O. Compared to kaolinite, it is richer in silica, giving it a more pronounced water adsorption and cation fixation capacity. It belongs to the smectite family, characterized by three-layer sheets: tetrahedral-octahedral-tetrahedral (2:1), with a typical thickness of 10 angstroms. These sheets exhibit substitutions within the octahedral layer, where aluminum ions (Al3+) can be replaced by magnesium ions (Mg2+), for example.

Montmorillonite is also known for its high cation exchange capacity (CEC), which typically ranges from 100 to 130 milliequivalents per 100 grams of clay calcined at 900°C. One of the most famous clays in this family is fuller's earth. It should be noted that this clay is never pure in its natural state but is often accompanied by mineral impurities such as quartz, muscovite, calcite, etc.

When composed of at least 75% montmorillonite, this clay is often marketed under the name bentonite, which is commonly used in various industrial, pharmaceutical, and environmental applications. [2]

3.1. Definition of Bentonite :

Bentonite, a clayey rock derived from volcanic processes, is distinguished by its malleability and excellent absorption capacity. It is widely employed across various industrial sectors for its remarkable properties. As a binding agent, it solidifies materials and holds them together in applications such as foundry and refractory brick manufacturing. Its ability to form tight seals makes it a preferred choice for sealing boreholes, retention ponds, and reservoirs. Additionally, its capacity to reduce friction makes it an effective lubricant in the construction, mining, and petroleum industries. [12]

Figure 1.4 Raw Bentonite

3.2. Origin of Bentonite :

The alteration and hydrothermal transformation of ash from volcanic tuffs rich in glass result in the formation of new clay minerals, mainly smectites. The clayey rocks thus created are called bentonites, named after the deposit near Fort Benton (Wyoming, United States). They contain over 75% montmorillonite, with the initial discovery dating back to 1847 near Montmorillon, in the department of Vienne (France). [7-8]

In Algeria, the most significant economically important bentonite deposits are located in Oran, the western region of Algeria. Particularly, the Maghnia quarry (Hammam Boughrara) has estimated reserves of one million tons, while the Mostaganem quarry (M'zila) has approximately two million tons of reserves. [9-10]

Figure 1.5 the Maghnia quarry (Hammam Boughrara)

3.3. Structure and Composition of Bentonite :

Despite their common elementary structure, different types of bentonite exhibit marked differences in terms of chemical composition and physical state of their constituents. These variations determine the specific properties of bentonite and influence its numerous technological applications.

Montmorillonite consists of an accumulation of lamellar plates, tightly packed together by electrochemical forces and containing interstitial water. Each plate comprises three layers arranged in a sandwich structure: a central layer of octahedral alumina (Al2O3) and two layers of tetrahedral silica (SiO2). Silicon and aluminum ions frequently undergo isomorphic substitutions by lower-valence metals, such as magnesium and iron. These substitutions result in a charge imbalance, compensated by exchangeable cations, including calcium ions (Ca2+), magnesium ions (Mg2+), and sodium ions (Na+), as well as water molecules bound by ion-dipole forces. These ions, no longer fitting into the lattice structure, migrate towards the external silica layers and are the main cause of hydration within the crystalline network.

Therefore, it can be assumed that each plate has the following general formulation:

$$\mathbf{[\ (Si^{Ai})_4\,(Al^{Fe\cdot Mg})_2\ O_{10}\ (OH)_2\ ]\ 2\cdot Mn\cdot mH2O}$$

where the first element in brackets refers to isomorphic constituents in the tetrahedral layers, the second element refers to isomorphic constituents in the octahedral layer; and the symbols M and mH2O refer to exchangeable cations and interstitial water, respectively. [11]

3.4. Formation of Bentonite:

Bentonite, a clay mineral of immense industrial importance, traces its origins to the deposition and subsequent alteration of volcanic ash. The genesis of bentonite begins with explosive volcanic eruptions, which disperse fine particles of ash into the atmosphere. Over time, these airborne particles settle onto the Earth's surface, forming layers of volcanic ash.

The transformation of volcanic ash into bentonite is a multifaceted process influenced by various environmental factors and geological conditions. Initially, the deposited ash undergoes chemical alteration facilitated by interactions with water, both from precipitation and groundwater sources. This alteration leads to the breakdown of primary minerals within the ash, particularly the amorphous volcanic glass.

As weathering processes act upon the volcanic glass shards, they release soluble ions such as silica ($SiO2$), aluminum (Al), magnesium (Mg), and iron (Fe). These ions serve as building blocks for the formation of secondary minerals, including clay minerals like montmorillonite. The alteration pathway involves hydrolysis reactions, ion exchange processes, and the crystallization of new mineral phases.

Hydrothermal fluids associated with volcanic activity can further influence the alteration of volcanic ash, introducing additional elements and compounds into the system. Environmental conditions such as temperature, pH, redox potential, and the availability of water play pivotal roles in determining the kinetics and pathways of mineral transformation.

The depositional environment of the volcanic ash also impacts the alteration process. Bentonite can form in a variety of settings, including marine, lacustrine, and terrestrial environments. Each environment presents unique geochemical conditions that influence the mineralogy and properties of the resulting bentonite deposit.

Over geological time scales, diagenetic processes exert further modifications on the bentonite deposit. Compaction, cementation, and recrystallization processes alter the texture and mineralogy of the clay-rich material, leading to the development of distinct lithological features characteristic of bentonite deposits. [15]

3.5. Mineralogy of Bentonite:

At the heart of bentonite's mineralogical composition lies montmorillonite, a member of the smectite group of clay minerals. Montmorillonite exhibits a characteristic 2:1 layered structure, consisting of tetrahedral and octahedral sheets stacked in a repeating sequence. The tetrahedral sheets comprise silicon (Si) and oxygen (O) atoms, while the octahedral sheets consist of aluminum (Al) and oxygen (O) atoms.

The isomorphic substitution of aluminum ions by magnesium (Mg) and silicon ions by aluminum (Al) within the crystal lattice imparts a net negative charge to the montmorillonite structure. This charge imbalance is balanced by the presence of exchangeable cations, such as sodium ($Na+$), calcium ($Ca2+$), and magnesium ($Mg2+$), in the interlayer spaces of the mineral.

Montmorillonite's high cation exchange capacity (CEC) allows it to adsorb and exchange cations with surrounding solutions, influencing its behavior in various environmental and industrial applications. The mineral's platy morphology and high surface area contribute to its exceptional swelling properties when hydrated, a characteristic feature exploited in applications ranging from drilling muds to cat litter.

In addition to montmorillonite, bentonite deposits may contain accessory minerals and impurities derived from the parent volcanic ash or introduced during the alteration process. These include minerals such as quartz, feldspar, mica, and organic matter, which can influence the physical and chemical properties of the bentonite deposit.[15]

3.6. Occurrence of Bentonite:

Bentonite deposits are found worldwide, occurring in a variety of geological settings and environments. The distribution and characteristics of bentonite deposits are influenced by factors such as volcanic activity, depositional history, tectonic processes, and weathering conditions. Understanding the occurrence of bentonite is essential for exploration, mining, and utilization of this valuable resource.

3.6.1. Volcanic Settings: Bentonite deposits commonly originate from the alteration of volcanic ash and tuff deposits. These deposits form during explosive volcanic eruptions, where fine particles of volcanic ash are ejected into the atmosphere and subsequently settle on the Earth's surface. The alteration of volcanic ash into bentonite occurs through chemical weathering processes driven by interactions with water and hydrothermal fluids associated with volcanic activity.

3.6.2. Marine Environments: Bentonite deposits can occur in marine sedimentary environments, particularly in areas with volcanic island arcs and active tectonic settings. Volcanic ash dispersed into marine waters settles onto the seafloor, where it undergoes alteration processes similar to those in terrestrial environments. Marine bentonite deposits are often associated with sedimentary basins and submarine volcanic activity.

3.6.3. Lacustrine and Fluvial Environments: Bentonite deposits may also form in lacustrine (lake) and fluvial (river) environments where volcanic ash is deposited in freshwater bodies. Volcanic eruptions near lakes or rivers can result in the rapid deposition of ash-rich sediments, which subsequently undergo alteration to form bentonite. Lacustrine and fluvial bentonite deposits are common in regions with active or dormant volcanoes.

3.6.4. Terrestrial Environments: In terrestrial settings, bentonite deposits can occur in various depositional environments, including floodplains, alluvial fans, and volcanic craters. Volcanic ash deposited on land surfaces weathers and alters over time, forming bentonite deposits within sedimentary sequences. Terrestrial bentonite deposits may exhibit characteristics such as stratification, grading, and cross-bedding, reflecting the dynamics of volcanic eruptions and sediment transport processes.

3.6.5. Structural Controls: The occurrence of bentonite deposits may be influenced by structural features such as faults, fractures, and folds. Tectonic activity can create pathways for hydrothermal fluids to circulate, enhancing the alteration of volcanic ash and promoting the formation of bentonite deposits. Structural controls also play a role in the localization and geometry of bentonite-bearing formations within the Earth's crust.

3.6.6. Secondary Deposition: Bentonite deposits can undergo secondary deposition and redistribution processes, leading to the formation of secondary or reworked bentonite beds. These secondary deposits may result from sedimentary reworking, erosion, transportation, and re-deposition of primary bentonite material by fluvial, aeolian (wind), or mass wasting processes.

3.6.7. Exploration and Resource Assessment: The exploration and assessment of bentonite resources involve geological mapping, stratigraphic analysis, geophysical surveys, and drilling programs. Geological models are developed to characterize the geometry, thickness, quality, and continuity of bentonite-bearing formations.

Understanding the occurrence and distribution of bentonite deposits is crucial for evaluating their economic potential and sustainable utilization in various industrial, agricultural, and environmental applications.

By considering the diverse occurrence of bentonite deposits in different geological settings and environments, geologists and resource professionals can effectively identify, evaluate, and exploit these valuable clay resources for various applications. [15]

3.7. The properties of bentonite :

3.7.1. Physical Properties:

Colloidal Suspension Formation: Bentonite can disperse in water to form stable colloidal suspensions. In this context, the term "colloidal" refers to bentonite's ability to form microscopic particles dispersed in a liquid medium without quickly settling.

Swelling upon Contact with Water: When bentonite comes into contact with water, it has the ability to swell significantly. This phenomenon is due to the unique crystalline structure of bentonite, which allows water absorption between its layers.

High Absorption Capacity: Bentonite has a high absorption capacity, meaning it can absorb and retain large quantities of water or other liquids. This characteristic makes it valuable in many applications, including controlling liquid spills and manufacturing animal litter.

High Viscosity and Plasticity: Bentonite exhibits high viscosity and plasticity, giving it some cohesion and allowing it to take on a malleable form when mixed with water. This property is particularly useful in applications such as drilling muds, where bentonite is used as a drilling fluid to stabilize wellbore walls.

Thixotropy: Thixotropy is the property where the viscosity of a material decreases when subjected to shear stress and then returns to its original viscosity when left at rest. Bentonite exhibits thixotropy, meaning its viscosity decreases when agitated or stirred but returns to its original viscosity when left at rest. This property is utilized in applications such as paint manufacturing and drilling products, where variable viscosity is desired based on application needs. [12]

3.7.2. Mechanical Properties:

Formation of Viscous and Malleable Suspensions: When bentonite is mixed with water, it forms viscous suspensions that can be molded and shaped as needed. This characteristic is essential in many industrial applications where bentonite is used as a binding agent or mold-forming agent.[12]

Ability to Act as a Viscous Fluid: When bentonite is agitated or stirred, it behaves like a viscous fluid, making it ideal for applications such as well drilling where it can be used as a drilling fluid to stabilize wellbore walls.

Maintaining Shape at Rest: Despite its high viscosity when agitated, bentonite retains its shape when left at rest. This feature is important in applications such as constructing watertight barriers where bentonite is used to seal joints and prevent water leaks.

Swelling Property: When bentonite comes into contact with water, it has the ability to swell significantly, increasing its initial volume. This swelling is due to the crystalline structure of bentonite, which allows water absorption between its layers.

Use in Geotechnical Applications: Due to its ability to form viscous suspensions and its swelling power, bentonite is widely used in geotechnical applications such as soil sealing, stabilization of wellbore walls, and dam construction.

Compressibility: Under certain conditions, bentonite can be compressible, meaning it can be compressed to form tablets or blocks. This characteristic is useful in applications such as remediating contaminated soils, where bentonite can be used to encapsulate contaminantsand reduce their dispersion in the environment.

3.8. Areas of Application of Bentonite:

Bentonite is utilized in numerous fields due to its unique properties. Here are some of the primary areas of application for bentonite:

a- Oil and Gas Drilling**:** Bentonite is widely used as a drilling fluid in the oil and gas industry to stabilize wellbore walls, control pressure, and remove drill cuttings.

b- Civil Engineering and Construction: In civil engineering, bentonite is used for foundation waterproofing, tunnel construction, waterproof barriers, and soil stabilization. It is also used in dam construction to prevent water leakage.

c- Environmental Engineering: Bentonite is used in environmental applications such as remediation of contaminated sites, waste management, wastewater treatment, and construction of retention ponds.

d- Mining Industry: In the mining industry, bentonite is used as a flocculation agent to separate fine particles from drainage water and as a binder in ore processing.

e- Foundry: Bentonite is used in the foundry industry as a binding agent to agglomerate sand particles and form molds for metal casting.

f- Pharmaceutical and Cosmetic Industry: Bentonite is used in the manufacture of drugs and cosmetic products due to its absorbent and adsorbent properties. It is used as an excipient in tablets and powders and as an ingredient in facial masks and skincare products.

g- Agriculture: In agriculture, bentonite is used as a soil amendment to improve its structure, water retention, and nutrient retention capacity.

h- Food Industry: Bentonite is used as a clarifying agent in the clarification process of fruit juices, wines, and beers. It is also used as a thickening and suspending agent in food products such as sauces, soups, and desserts.

These areas of application illustrate the versatility of bentonite and its contribution to many industrial sectors and diverse applications. [13]

3.9. Types of Bentonites :

The types of bentonite are generally classified based on their chemical composition, physical properties, and applications.

a- Sodium Bentonite: Also known as sodium bentonite, this variety contains a higher proportion of exchangeable sodium cations compared to other cations like calcium and magnesium. It is typically used in applications where viscosity reduction and filtration control are priorities, such as in oil and gas drilling.

b- Calcium Bentonite: Also called calcium bentonite, this bentonite contains a higher proportion of exchangeable calcium cations. It is often used in applications where stability and water retention are important, such as in the construction of waterproof barriers or as a flocculation agent in wastewater treatment.

c- Mixed Bentonite**:** This variety contains both sodium and calcium exchangeable cations, giving it intermediate properties between sodium and calcium bentonites. It is used in a range of diverse applications, including in geotechnical engineering, civil construction, and the mining industry.

d- Activated Bentonite: Activated bentonite is a type of bentonite that has been treated to enhance its absorption and adsorption properties. This treatment may involve thermal, chemical, or physical activation to increase its specific surface area and ion exchange capacity. Activated bentonite is widely used in applications such as wastewater treatment, oil and solvent purification, and liquid clarification.

e- Organophilic Bentonite: This is a bentonite that has been treated with organic agents to modify its structure and impart hydrophobic properties. Organophilic bentonite is often used as a swelling agent in oil-based drilling fluids and as a stabilizing agent in polymers and composites.

These types of bentonite exhibit different characteristics and performances that make them suitable for a variety of industrial, geotechnical, and environmental applications.

4. **Intercalated clays** :

4.1 Intercalation :

Intercalation in clays involves the insertion of chemical species between the layers of clay minerals, typically resulting in an increase in interlayer spacing. This expansion of interlayer spacing can be observed using techniques such as X-ray diffraction (XRD), where it manifests as a shift in the d(001) spacing.

This phenomenon is of significant importance in many applications as it allows for the modification of clay properties to suit various uses. For instance, intercalation can be employed to enhance the adsorption capacity of clays, improve their thermal or chemical stability, or facilitate controlled release of intercalated molecules in pharmaceutical or environmental applications.

4.2. Pillared clays :

Pillared Interlayered Clays (P.I.L.C), can be obtained through a process called "pillaring" (or pontage in French). This process involves two main steps:

1- Exchange of interlayer cations of the clay with metal polycations obtained by prior hydrolysis of the metal element.

2- Calcination at high temperature (300–500°C) to convert the polycations into pillars of metal oxides.

This transformation leads to an increase in the interlamellar basal distance d001 of the intercalated smectite compared to that of the starting clay mineral. This distance then undergoes a slight decrease in the final material of clay with metal oxide pillars due to dehydroxylation and dehydration during calcination.

The resulting pillared clays exhibit increased thermal stability and opening of interlayer spaces, resulting in an increase in their specific surface area and microporous volume. These properties offer new functionalities to clay minerals, paving the way for innovative and interesting applications.

We can classify pillared clays into three main categories:

a) Family of organo-clay complexes: These are modified (intercalated) bentonites by organic ions, such as tetraalkylammonium, alkylimidazolium, and tetraalkylphosphonium ions from ionic liquids. These organo-clay complexes are hydrophobic and organophilic, and can be used in the remediation of water contaminated with certain organic micropollutants such as pesticides, dyes, and phenols.

b) Family of inorgano-clay complexes: These are pillared clays with metal oxide pillars that possess exceptional adsorptive and catalytic properties.

c) Family of inorgano-organo-clay complexes: These are mixed pillared clays, such as montmorillonite intercalated with aluminum polycations and modified by the co-adsorption of a metal alkylammonium or another cationic surfactant.

5. **Conclusion** :

In conclusion, this chapter has explored montmorillonite clays, with a focus on bentonite, from their geological origin to their structure, properties, and various types. We have discovered that bentonite, with its unique characteristics, plays a crucial role in many industrial fields. However, challenges persist in terms of sustainability and environmental impact. By continuing our research and fully harnessing the potential of these materials, we can envision a future where clays contribute to technological innovation and sustainable development.

In the next chapter, we will begin the various practical identification tests for bentonite.

Chapter 2

Characterization of bentonite

1. **Introdction :**

In this chapter, we explore the detailed characterization of bentonite, comparing raw and industrially processed samples to understand their physicochemical and mechanical properties

To achieve this, we conducted a series of standardized tests. The Proctor test was used to evaluate the compaction properties of both raw and industrial bentonite, providing insights into optimal moisture content and maximum dry density, which are essential for construction and geotechnical applications. The sedimentation test helped determine the particle size distribution, crucial for understanding the rheological properties and settling behavior of the clay.

We assessed the plasticity and workability of bentonite through the Atterberg limits, measuring the liquid limit, plastic limit, and plasticity index. These properties are fundamental for predicting the soil's behavior under various moisture conditions. The methylene blue test was employed to measure the cation exchange capacity (CEC), a vital parameter for applications in environmental remediation and chemical industries.

Additionally, we measured the bulk density of the bentonite to provide information on the mass per unit volume, which is important for transportation and storage considerations. The carbonate content was determined using the calcimeter method, offering insights into the mineralogical composition, particularly the presence of carbonate minerals that can influence the clay's chemical reactivity and stability.

Each of these tests was conducted with rigorous procedures to ensure accuracy and reproducibility of results. This chapter systematically presents the methodologies, specific procedures, and results of these characterization tests, comparing raw and industrial bentonite samples to highlight their respective properties. The findings from these tests are critical in developing a comprehensive profile of bentonite, enabling its effective application in various fields.

2. Standard Proctor Trial:

NF P 94-093

2.1. Définitions et symboles :

w: moisture content, expressed as a percentage;

ρs: density of solid particles of the soil, expressed in tonnes per cubic meter;

ρd: density of dry soil, expressed in tonnes per cubic meter;

ρw: density of water, expressed in tonnes per cubic meter, conventionally equal to one ton per cubic meter

Sr: degree of saturation, expressed as a percentage:

$$Sr(\%) = \frac{W}{\rho w \left(\frac{1}{\rho d} - \frac{1}{\rho s}\right)}$$

I.CBR : is the California Bearing Ratio (CBR) index after immersion, expressed as a percentage;

IPI : is the Immediate Bearing Index (IBI), expressed as a percentage;

Dmax : is the maximum dimension of the largest particles contained in the soil. The presence of one or a few large scattered particles should not lead to considering their dimensions as the Dmax of the soil.

"Compaction energy" (expressed in kNm/m3) = $\frac{N*H*m*g}{V}$

N : is the total number of blows;

H : is the height of fall of the rammer;

m : is the mass of the rammer;

g : is the acceleration due to gravity;

V : is the volume of the compacted material in the mold.

2.2. Principle of the Trial :

The characteristics of the compaction Proctor test of a material are determined from tests called Normal Proctor Test or Modified Proctor Test. The two tests are identical in principle, only differing in the values of the parameters that define the applied compaction energy.

The principle of these trials consists of moistening a material at various water contents and compacting it, for each water content, according to a conventional process and energy. For each value of water content considered, the dry density of the material is determined, and the curve of the variations of this dry density as a function of water content is plotted.

In general, this curve, called the Proctor curve, presents a maximum value of the dry density of the material obtained for a particular value of the water content. These two values are called the optimal characteristics of Normal or Modified Proctor compaction, depending on the test performed.

NOTE 1: This document particularly considers the determination of the Proctor curve in five points. This is a minimum number, and any test resulting in the determination of this curve with more points is obviously compliant with the document.

NOTE 2: It is often advantageous to perform tests to determine the immediate CBR indices and/or after immersion and/or the Immediate Bearing Index simultaneously with the Proctor tests, since these tests are performed on molded specimens compacted in accordance with the procedure described in this document (see standard NF P 94-078).

2.3. Apparatus :

2.3.1. Specific Apparatus :

A- compaction pedestal : consisting of a concrete block with a flat horizontal surface of at least 30 cm × 30 cm and a thickness of at least 30 cm

B- Proctor mold : consists of a mold body, a base, and an extension. The mold body can be either single-piece or split.

C- Normal Proctor hammer : consists of a cylindrical ram of 51 mm ± 1 mm diameter. This ram slides within a sleeve that allows it a drop height of 305 mm ± 2 mm. The mass of the moving parts is 2,490 g ± 2.5 g

D- A straightedge : composed of a steel blade.

E- Mechanized compaction machines : can be advantageously used if they meet the following conditions:

- The mass, diameter, drop height of the ram, thickness of the sleeve when the machine has one.
- The distribution mode of the blows, specified in paragraph 7.2, is respected.
- The kinematics of the mechanism allow the mold holder to be in direct contact with the machine frame at the moment of impact of the hammer; if this is not the case, it should be checked for each material that the differences in measurement of the maximum density between the machine and manual mode do not exceed 1%.
- The machine is securely attached to a concrete base with a minimum thickness of 30 cm.

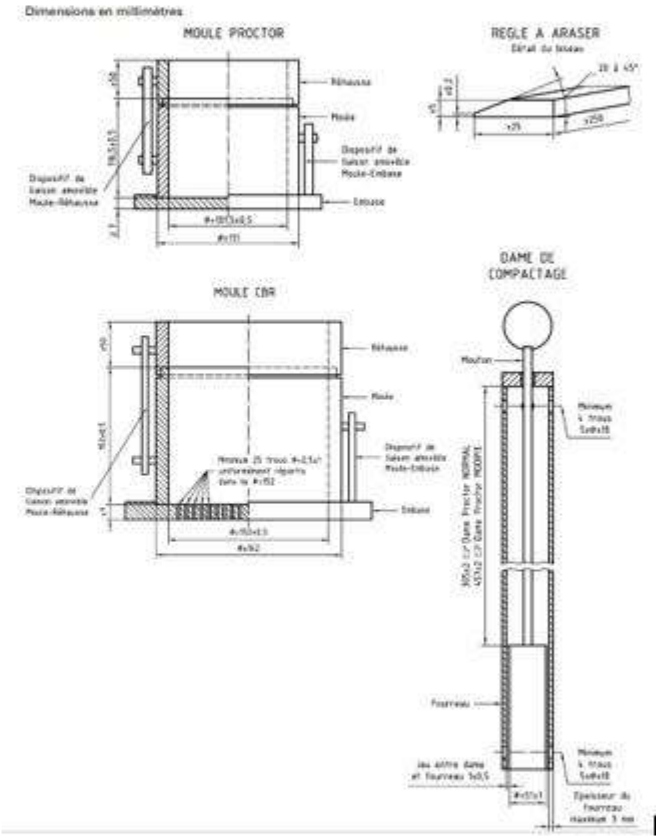


Figure 2.1 Proctor apparatus

2.3.1 Common Equipment :

A- Sieves with square mesh openings of nominal dimensions 5 mm and 20 mm;

B- Balances with maximum and minimum capacities compatible with the masses to be weighed, ensuring measurements are taken with an uncertainty of 1/1000 of the measured value;
C- A thermal chamber or drying apparatus to determine the moisture content of materials according to a standardized method (NF P 94-049-1, NF P 94-049-2, NF P 94-050);
D- Hermetically sealed containers (or bags) to preserve the moisture content of samples;
E- A water spray apparatus;
F- Optionally, a mechanical mixer-disintegrator with a tank volume of at least 10 dm3 (see example in Appendix B);
G- A device for extracting the specimen from the mold body (in the case of a single-piece mold body);
H- Various materials and tools.

2.3.3. Expression of results :

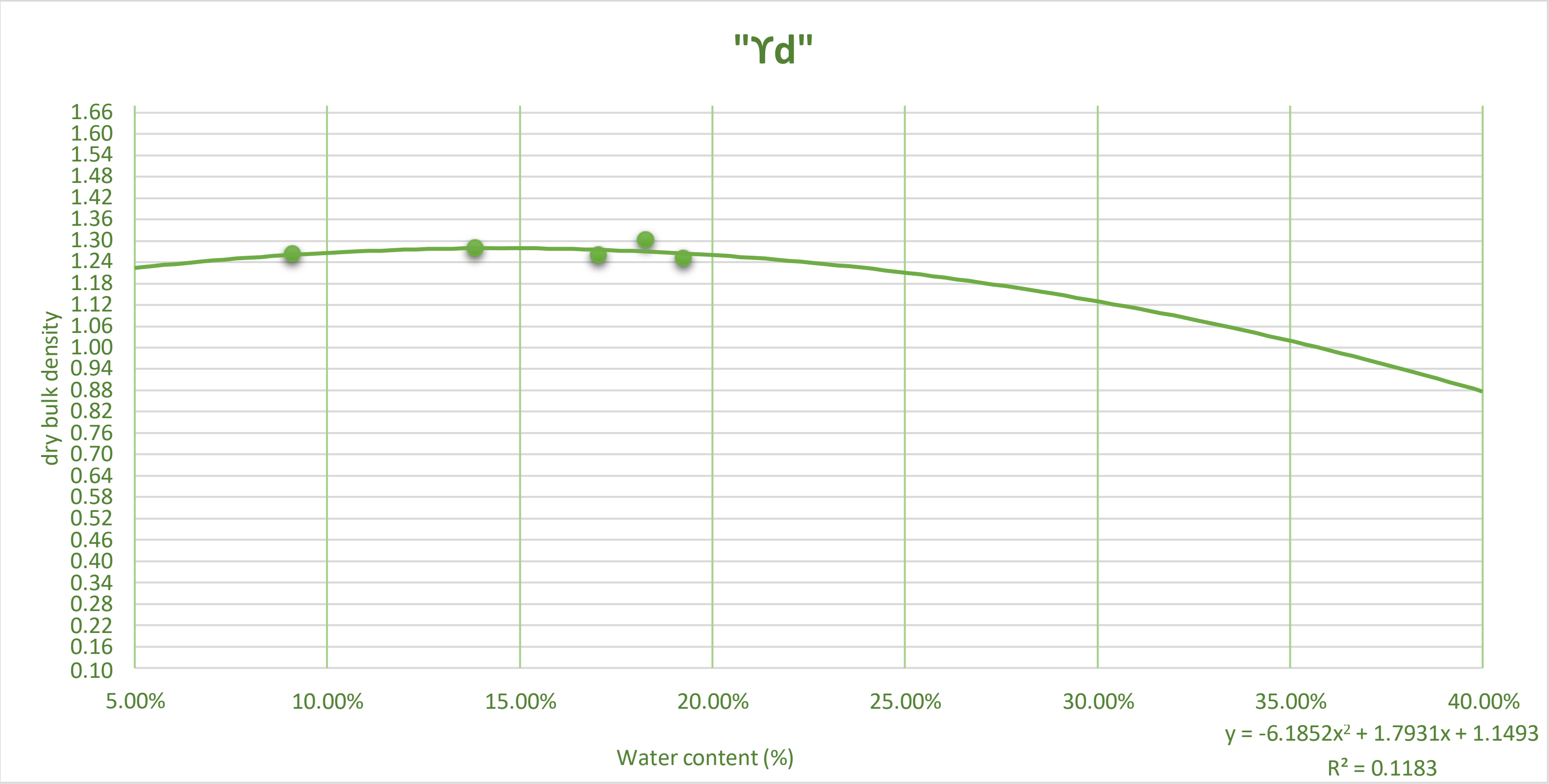
2.3.3.1. Case of Raw bentonite :

Number of trial	Trial 01	Trial 02	Trial 03	Trial 04	Trial 05	Trial 06	Trial 07	Trial 08
Percentage of water added (%)	5%	10%	15%	17%	19%	21%	23%	25%
Mold mass (g)	5735,00	5735,00	5735,00	5735,00	5735,00	5735,00	5735,00	5735,00
Mass of wet soil + mold (g)	7047	7123	7139	7155	7203	7237	7168	7160
Mold volume (cm^3)	953	953	953	953	953	953	953	953
Mass of wet soil (g)	1312	1388	1404	1420	1468	1502	1433	1425
Wet bulk density (t/m3)	1,376705142	1,456453305	1,473242392	1,49003148	1,540398741	1,576075551	1,503672613	1,495278069

Tab 2.1 Proctor test (raw 1)

Number of trial	Trial 01		Trial 02		Trial 03		Trial 04		Trial 05		Trial 06		Trial 07		Trial 08	
Tare number	f12	53	2k	8%	b14	b22	52	15	s1	t1	s2	t2	s3	t3	s4	t4
Mass of the tare (g)	9,50	9,52	9,81	9,56	9,4	9,8	10	9,6	22,7	22	22,2	22,7	22,4	22,7	25,6	22,3
Mass of wet soil + tare (g)	13,69	15,22	15,83	19,10	20	21,2	21,10	20,2	35	36,5	35	37,1	34	36,8	35	35,2
Mass of dry soil + tare (g)	13,3	14,8	15	18,1	18,4	19,6	19,4	18,4	33,4	34,3	33	34,6	32,3	34,5	33	32,8
Mass of water (g)	0,39	0,42	0,83	1	1,6	1,6	1,7	1,8	2	2,2	2,2	2,5	2,1	2,3	1,8	2,4
Mass of dry soil (g)	3,80	5,28	5,19	8,54	9,00	9,80	9,40	8,80	10,70	12,30	10,80	11,90	9,90	11,80	7,40	10,50
Water content (%)	10,26 %	7,95%	15,99 %	11,71 %	17,78 %	16,33 %	18,09 %	20,45 %	18,69 %	17,89 %	20,37 %	21,01 %	21,21 %	19,49 %	24,32 %	22,86%
Average water content (%)	9,11%		13,85%		17,05%		19,27%		18,29%		20,69%		20,35%		23,59%	
Dry bulk density	1,262		1,279		1,259		1,249		1,302		1,306		1,249		1,210	

Tab2.2 Proctor test (raw 2)



OPN

ϒd (Max)	ω (OPN)
1,279	14,495%

A -6,1852 B 1,7931 C 1,1493

Diagram 2.1 The Proctor optimum (raw bentonite)

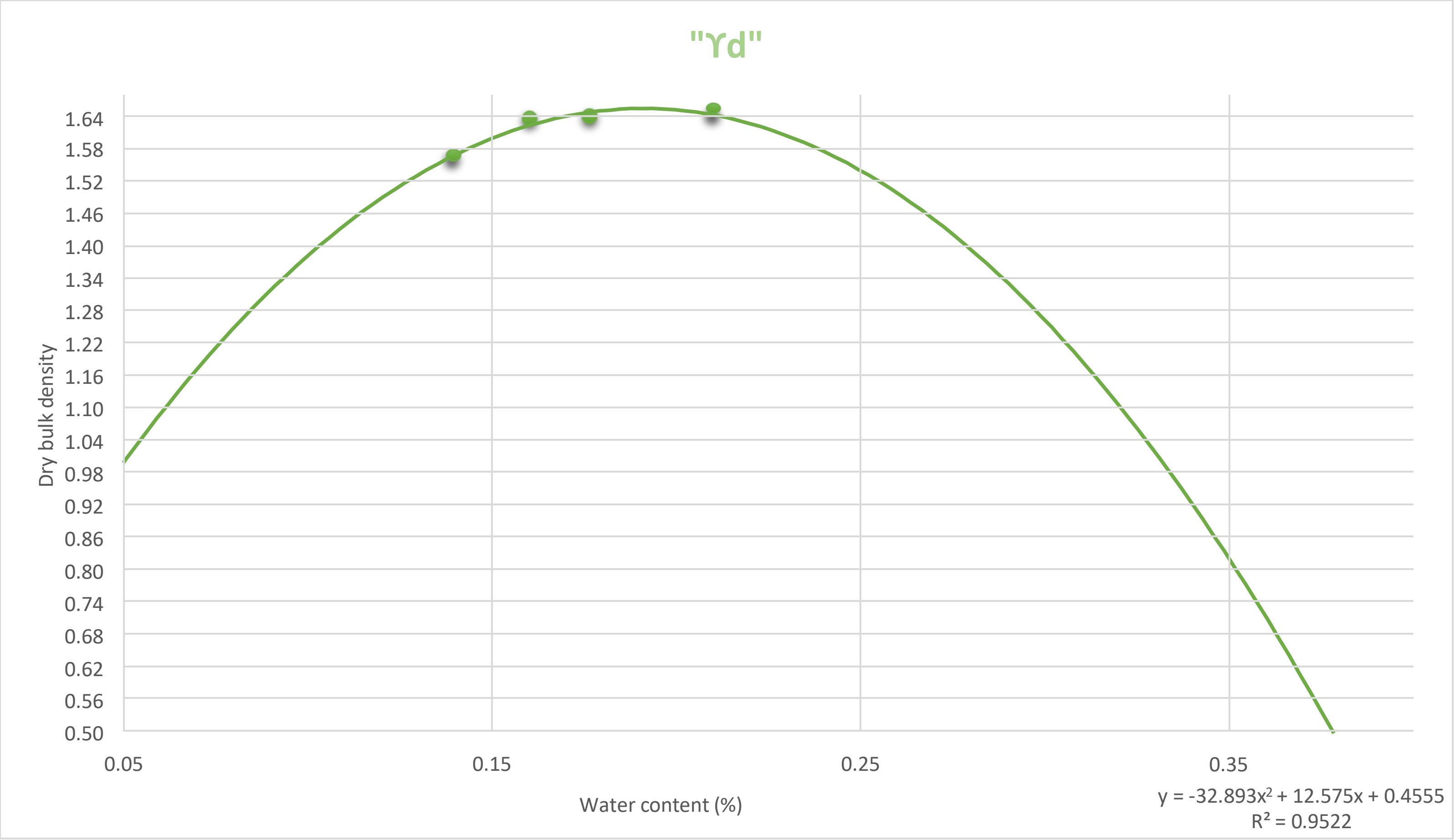
2.3.3.2. Case of Industrial bentonite :

Number of Trial	Trial 01	Trial 02	Trial 03	Trial 04
Percentage of water added (%)	15%	20%	25%	29%
Mold mass (g)	5733,00	5733,00	5733,00	5733,00
Mass of wet soil + mold (g)	7090	7135	7168	7013
Mold volume (cm^3)	953	953	953	953
Mass of wet soil (g)	1357	1402	1435	1280
Wet bulk density (t/m3)	1,423924449	1,471143757	1,505771249	1,343126967

Tab 2.3 Proctor test (indu 1)

Number of trial	Trial 01		Trial 02		Trial 03		Trial 04	
Tare number	f12	53	2k	8%	b14	b22	b30	b38
Mass of the tare (g)	9,50	9,50	9,5	9,9	9,5	9,6	9,70	9,50
Mass of wet soil + tare (g)	15,7	18,9	18,60	22,80	22,2	19,4	23,30	22,20
Mass of dry soil + tare (g)	15,1	17,8	17,2	20,8	19,6	17,5	20,50	19,70
Mass of water (g)	0,6	1,1	1,4	2	2,6	1,9	2,80	2,50
Mass of dry soil (g)	5,60	8,30	7,70	10,90	10,10	7,90	10,80	10,20
Water content (%)	10,71%	13,25%	18,18%	18,35%	25,74%	24,05%	25,93%	24,51%
Average water content (%)	11,98%		18,27%		24,90%		25,22%	
Dry bulk density	1,272		1,244		1,206		1,073	

Tab2.4 Proctor test (indu 2)



ϒd (Max)	ω (OPN)
1,273	13,602%

Diagram 2.2 The Proctor optimum (industrial bentonite)

3. **The determination of Atterberg limits**:

NF P 94-051

3.1. Definitions:

Liquid Limit (W_L): This is the highest water content at which a soil remains in a semi-liquid state, where it begins to flow under its own weight. It is determined using the Casagrande device.

Plastic Limit (W_P): This is the lowest water content at which a soil can be deformed without cracking. It is determined using the plasticity roll or the Atterberg's device.

Plasticity Index (I_P): This is the difference between the liquid limit and the plastic limit. It measures the range of water content over which the soil can transition from a solid to a plastic state without changing its liquid phase. This index is useful for characterizing the plasticity of a soil.

$$I_P = W_L - W_P$$

When water content is expressed in percentage, the plasticity index is a dimensionless number.

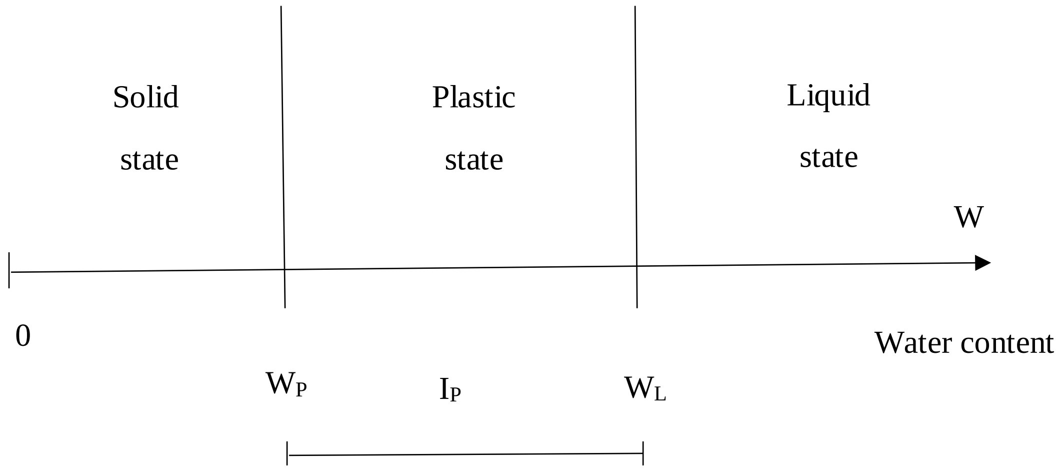


Diagram 2.3 Representation of Atterberg limits

3.2. The principle of determining Atterberg limits:

The trial is conducted in two phases:

- Search for the water content at which a groove made in a soil placed in a dish with imposed characteristics closes when the dish and its contents are subjected to repeated impacts.
- Search for the water content at which a manually prepared soil roll, of fixed dimensions, cracks

3.2.1. Apparatus:

a) Equipment for determining the liquid limit:

- Casagrande apparatus
- A drying oven
- A balance
- Capsules
- Spatula
- Trowels

b) Equipment for determining the plastic limit:

- A smooth marble plate or equivalent material for mixing and preparing soil samples for rolling.
- Capsules
- Spatula
- A balance
- A drying oven
- A glass plate, 5 cm wide and 10.5 cm long, with markings every 1 cm and a 0.5 cm shim, or any other device enabling the measurement of soil roll diameters of 3 mm

3.2.2. Operating procedure:

The operations described below are to be performed successively.

3.2.2.1. Soil preparation:

After soil sampling and homogenization by mixing, a mass of material m is soaked in a container of water at room temperature for at least 24 hours.

The mass m, expressed in grams, must be greater than 200 times the size of the largest soil particles appreciated visually and expressed in millimeters. Similarly, it must be such that sieving through the 400-micrometer sieve yields at least 200 grams of solid particles.

Once soaked, the material is wet-sieved through a 400-micrometer sieve. The wash water and the material retained on the sieve are collected in a tray.

After a settling duration of at least 12 hours, without any additive intended to accelerate sedimentation or using any centrifugation process, the clear water from the tray is siphoned off without entraining solid particles. The excess water is evaporated at a temperature not exceeding 50°C.

Figure 2.2 Soil preparation

3.2.2.2. Conducting the test:

a) The liquid limit:

Spread with the spatula, in the clean and dry dish, a mass of approximately 70g of paste, this paste spread in several layers to avoid trapping air bubbles, presenting at the end of the operation a symmetrical appearance as shown in the figure

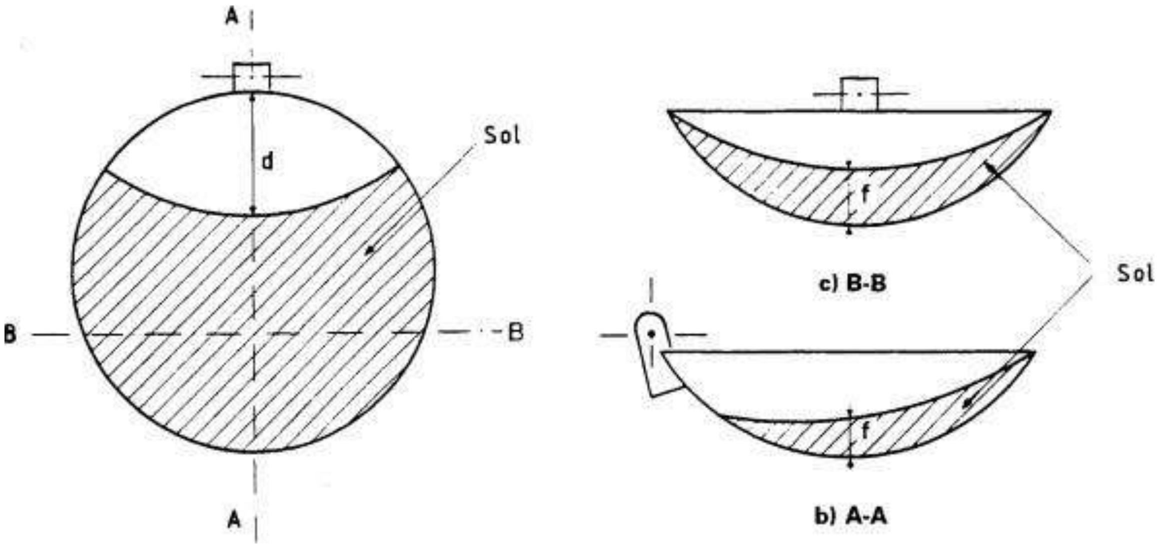


Figure 2.3 Filling the Casagrande cup

The paste covers the bottom of the cup except for a part d of about 3cm

Divide the paste in two as shown in the figure, using the grooving tool, holding it perpendicular to the surface of the cup and presenting its beveled part facing the direction of movement.

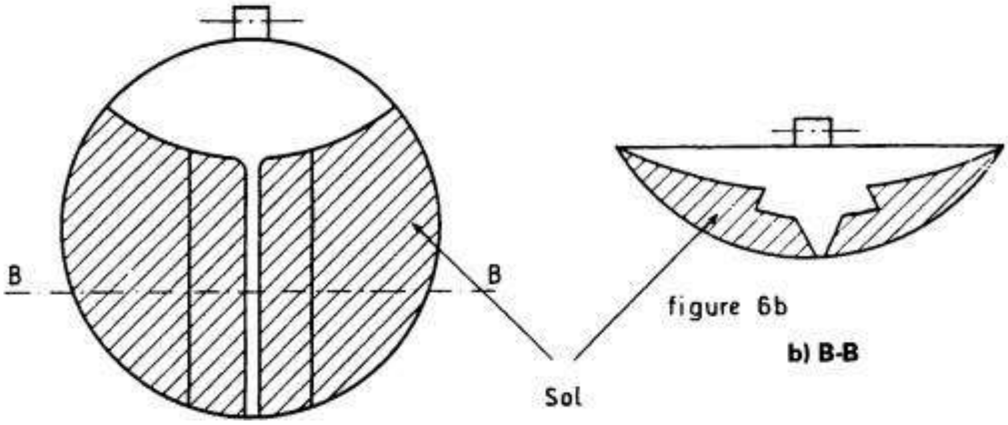


Figure 2.4 creating the groove

Gently fix the cup onto the metal support of the Casagrande apparatus

Operate the cam to subject the cup to a series of shocks at a rate of 2 impacts per second

Note the number N of impacts required for the lips of the groove to meet over a length of approximately 1cm as shown in the figure

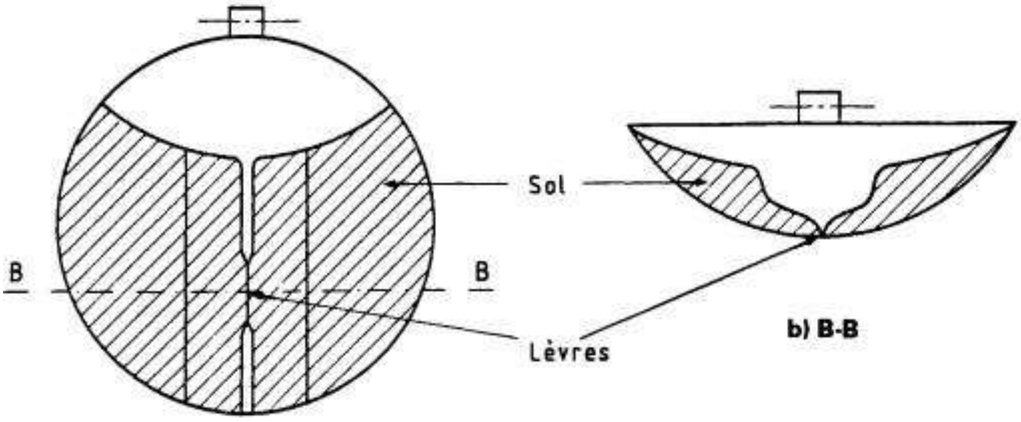


Figure 2.5 closing the groove

If N is less than 15, the process is repeated with a drier material, and homogenization is performed again

If N is greater than 35, the operation is repeated on a sample of paste to which a small amount of distilled or demineralized water has been added

The trial is continued only when N is between 15 and 35

Take from the cup, and using a spatula, approximately 5 g of paste, from each side of the lips of the groove and near the place where they closed, in order to determine its water content.

The sample is placed in a capsule or Petri dish of known mass and immediately weighed before being introduced into the oven for desiccation in accordance with standard NF P 94-050.

The whole operation is carried out at least four times on the same dough, but with a different water content each time.

The numbers of shocks in the test series must bracket 25 and the difference between two consecutive values must be less than or equal to 10

b) The plastic limit:

Form a pellet from the prepared paste

Roll the pellet on a smooth surface, by hand or possibly with the help of a press, until it forms a roll that gradually thins out until it reaches a diameter of 3 mm.

The hand movement should be regular.

The thinning of the roll is done continuously and without cutting in the lengthwise direction.

The roll, when it reaches a diameter of 3.0 mm + 0.5 mm, should be approximately 10 cm in length and should not be hollow.

The plastic limit is reached when, simultaneously, the roll cracks and its diameter reaches 3 mm + 0.5 mm.

If no cracks appear, the roll is reintegrated into the pellet. The dough is kneaded while being slightly dried, possibly under a flow of hot air at a temperature below 50°C. Form a new roll.

Once the cracks appear, take the central part of the roll and place it in a capsule or a Petri dish of known mass, weigh it immediately, and introduce it into the oven to determine its water content (standard NF P 94-050).

Perform a second test on a new pellet.

3.3. Expression of results:

3.3.1. The liquid limit:

The liquid limit W_L is the water content of the material conventionally corresponding to a closure of 1 cm of the groove lips after 25 impacts. It is calculated from the equation of the average line adjusted to the pairs of experimental values. This average line cannot be determined without a minimum of four points. The relationship is acceptable only if the difference in water content between the measured value and the calculated value for the same number of impacts does not exceed 3%. If this is not the case, repeat the measurement. The liquid limit wu is obtained for a value of N equal to 25. It is expressed as a percentage and rounded to the nearest whole number: the rounding interval is 1

3.3.2. Case of Raw bentonite:

M1 = Mass of empty container

M2 = Mass of container with wet sample

M3 = Mass of container with dry sample

- 1st trial:
  23 impacts

M1 = 9.4 g

M2 = 13.9 g

M3 = 12.3 g

W = (M2-M3) / (M3-M1)

W = 0.55 ==> 55%

- 2nd trial :
  33 impacts

M1 = 9.4 g

M2 = 14.3 g

M3 = 12.0 g

W = 0.88 ==> 88%

- 3rd trial:
  27 impacts

M1 = 9.8 g

M2 = 16.1 g

M3 = 13.2 g

W = 0.85 ==> 85%

Liquid limit (W_L) = 69% at 25 impacts

3.3.3. Case of Industrial bentonite :

- 1st trial:
  22 impacts

M1 = 9.5 g

M2 = 14.8 g

M3 = 12.2 g

W = (M2-M3) / (M3-M1)

W = 0.96 => 96%

- 2nd trial:
  33 impacts

M1 = 9.6 g

M2 = 13.3 g

M3 = 11.2 g

W = 1.31 => 131%

- 3rd trial:
  30 impacts

M1 = 9.7 g

M2 = 17.9 g

M3 = 13.6 g

W = 1.1 => 110%

Liquid limit (W_L) = 108% at 25 impacts

3.3.4. The plastic limit :

The plastic limit W_P is the conventional water content of a soil roll that cracks when its diameter reaches 3.0 mm ± 0.5 mm. Wp is the arithmetic average of the water contents obtained from two tests. The plastic limit value is expressed as a percentage, and the rounding interval is 1. If the values deviate by more than 2% from the average value, a new test is required.

3.3.4.1. Case of Raw bentonite :

- 1st trial:

M1 = 22.8 g

M2 = 27.1 g

M3 = 25.4 g

WP = 0.65 => 65%

- 2nd trial:

M1 = 22.6 g

M2 = 27.7 g

M3 = 25.7 g

WP = 0.39 => 39%

Wp=(0.65+0.39)/2=0.52

Therefore, the plastic limit Wp is 52%.

3.3.4.2. Case of Industrial bentonite :

- 1st trial:

M1= 22.6

M2= 23.9

M3= 23.4

W= 0.625=> 62.5%

- 2nd trial:

M1= 22.7

M2= 24.8

M3= 23.9

W= 0.75=> 75%

Wp=(0.625+0.75)/2=69

Therefore, the plastic limit Wp is 69%.

a) Plasticity Index :

I_P is The difference between the values of the liquid limit and the plastic limit

$$I_P = W_L – W_P$$

a.1) Case of Raw bentonite :

I_P= 17%

a.2) Case of Industrial bentonite :

I_P= 39%

4. **Granulometric Analysis of Soils, Sedimentation Method** :

NF P 94-057

4.1. Objective:

The purpose of this standard is to determine the weight distribution of the particle sizes of fine soil. It defines the terms used and the parameters measured, specifies the characteristics of the equipment, establishes the procedure to be followed, and details the results to be presented.

4.2. Scope of Application:

This standard applies to the elements of natural soil passing through the square mesh sieve with an 80 µm opening. However, particles smaller than 1 µm cannot be differentiated by this test.

The sedimentation granulometric analysis is a geotechnical test that complements the granulometric analysis by sieving of soil (standard NF P 94-056) and may be necessary for its description and classification.

4.3. General Information :

4.3.1. Principle :

The test, the subject of this standard, utilizes the fact that in a liquid medium at rest, the settling velocity of fine to very fine grains is a function of their size. Stokes' law provides, in the case of spherical grains of the same density, the relationship between the diameter of the grains and their sedimentation velocity. By convention, this law is applied to soil elements to determine equivalent particle diameters

4.3.2. Method:

The particles smaller than 80 µm, separated from the rest of the soil by sieving, are suspended in water supplemented with a deflocculant. The particles settle at different speeds depending on their size. Using a densimeter, the evolution over time of the density of the solution and the immersion depth of the device are measured. The weight distribution of the particle size is calculated from this data.

4.3.3. Terminology and Definitions :

- Tamisat : Material that passes through the meshes of a sieve.
- Sedimentation : Progression of a deposit of solid particles in a liquid at rest under the influence of gravity.
- Granularity : Dimensional distribution of grains in an aggregate.
- Density of a liquid : Ratio of the mass of a certain volume of the liquid to the mass of an equal volume of water under the same conditions of temperature and pressure.

4.4. Apparatus :

4.4.1. Soil preparation :

The following means are necessary for the preparation of the soil sample.

- Balance : A balance with maximum and minimum capacities compatible with the masses to be weighed, and such that the weighings are carried out with an uncertainty of 1/1,000 of the measured value.
- Sieve : A sieve with square mesh of 80 µm opening and a diameter of at least 250 mm
- Tray : An imperishable tray with minimum dimensions of 60 cm x 40 cm x 12 cm to collect the 80 µm sieved material
- Drying oven : A thermal chamber or a drying oven, with adjustable temperatures of 105°C and 50°C, of accuracy class C as defined in Appendix A of standard NF X 15-016.
- Mortar : A mortar with a minimum diameter of 20 cm along with its pestle made of flexible material to separate soil particles that have passed through an 80 µm sieve
- Mechanical stirrer : A mechanical stirrer with its container. The stirring speed must be adjustable up to 10,000 rpm. It must be of the "immersion" type, meaning that the shaft supporting the rotating paddle is inserted into the container containing the solution. This container must have a minimum capacity of 600 cm³
- Distilled or demineralized water and deflocculant : A mixture of distilled or demineralized water combined with a deflocculant to impregnate and disperse the sample. This mixture is made up of 440 cm³ of distilled or demineralized water and 60 cm³ of a 5% solution of sodium hexametaphosphate ($Na6(PO4)6, 10H2O$) prepared and stored away from light for less than one month.

4.5. Execution of the test :

The following equipment is necessary for carrying out the test:

Test tubes: Two cylindrical glass test tubes, transparent, graduated, with a minimum capacity of 2,500 cm³. They must have an inner diameter of 85 mm ± 5 mm, graduations at least at 500, 1,000, 1,500, and 2,000 cm³, and a variation in the cross-section of the test tube equal to or less than 1% of the average section.

- Distilled or demineralized water : A reserve of 4,000 cm³ of distilled or demineralized water
- Manual stirrer : A manual stirrer for homogenizing the suspension before testing.
- Densitometer : The densitometer must be capable of measuring with an accuracy of 0.0005. The operating range of the instrument is from 0.9950 to 1.0300. It must be torpedo-shaped with a center of thrust near the middle of the bulb.
- Thermometer : The two temperature measuring instruments must be capable of measuring with an accuracy of 0.5°C. The operating range of the thermometers is from 10 to 35°C.
- Stopwatch : The device must allow for time measurement to the nearest second.
- Test room thermometer : A control recording thermometer must be permanently placed in the testing room.

4.6. Operating procedure :

The operations described below are to be carried out successively in a test room where the temperature must be controlled.

4.7. Preparation of core sample :

a) The soil sample processing :

The sieved soil sample, sieved through a 80 µm sieve according to standard NF P 94-056, is collected with its washing water in a tray. The mixture is then left to settle. Once clear again, the water from the tray is siphoned off without entraining fine particles.

The tray and its contents are then placed in an oven. Conventionally, the evaporation of water is considered complete if the mass of the material in the tray does not vary by more than 2/1,000 between two successive weighings performed immediately after removal from the oven.

The minimum time interval between two weighings is:

- hours for drying at 105 °C,
- 8 hours for drying at 50 °C.

From the dried sieved material, disintegrated with a pestle in a mortar and then homogenized, take a test sample of 80 g + 10 g.

b) Sample soaking :

Introduce the test sample into the container used with the mechanical stirrer, add 500 cm3 of the mixture described in, and keep at room temperature.

Let soak for at least 15 hours at room temperature.

c) Mechanical stirring dispersion :

The trial sample is dispersed in the water and deflocculant solution using the mechanical stirrer, which must operate for a minimum of 3 minutes at 10,000 rpm.

4.7.1. Trial Execution :

a) Test initiation :

Immediately after the end of mechanical agitation, pour the dispersed suspension into a test cylinder.

Rinse the container as well as the shaft and blades of the mechanical stirrer with distilled or demineralized water. Collect the rinsing liquid in the test cylinder to avoid losing a portion of the soil sample during transfer.

Top up with distilled or demineralized water at room temperature until reaching 2,000 cm3.

Pour 2,000 cm3 of the same distilled or demineralized water into a second control cylinder and immerse a clean thermometer and densimeter into it.

Vigorously agitate the suspension vertically using the manual stirrer to achieve a uniform concentration throughout the height of the test cylinder.

Remove the manual stirrer and simultaneously start the timer; conventionally, this marks the beginning of the test.

Carefully immerse the densimeter into the suspension immediately after starting the timer.

b) Measurement execution :

Take readings from the start of the test for as long as necessary, at the following time intervals in minutes: 0.5 - 1 - 2 - 5 - 10 - 20 - 40 - 80 - 240 - 1440.

At each reading, record the density of the solution to the nearest 0.0001 and the temperature of the water in the control cylinder to the nearest 0.1°C.

Take densimeter readings at the top of the meniscus.

Conduct the first three readings at 0.5, 1, and 2 minutes without removing the densimeter from the solution.

From the third reading onwards, remove the densimeter from the solution after each measurement, clean it, and then immerse it in the distilled or demineralized water cylinder.

Starting from the fourth reading (at time t = 5 min), carefully immerse the densimeter in the solution at least 30 seconds before taking the measurement.

4.8. Expression of results :

4.8.1 Case of Raw bentonite :

		time (sec)	temperature	lecture Rc Rc= R+Ct+Cm+Cd		Is equal to 0,03368	Is equal to 0,00022	η=0,00179/(1+αθ+Bθ2					density of solid particles	density of distilled water			density of the suspension		
		time (sec)	**θ(c˚)**	***liquide (A)***	***temoin (B)***	**α**	**β**	***η***	***Hc (cm)***	***H(cm)***	***H1 (cm)***	***Ht(cm)***	***ρs (g/cm3)***	***ρw (g/cm3)***	***g(m/s2)***	***Di (mm)***	***ρt (g/cm3)***	***P(%)***	***% grains <à D***
30MIN		**30**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**0**	**18,4**	**2,83**	13,45	**2,79**	**0,999**	**9,81**	**0,0637**	1,0165	0,68085	100,00
1MIN		**60**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**0**	**18,4**	**2,83**	13,45	**2,79**	**0,999**	**9,81**	**0,0451**	1,0165	0,68085	100,00
2MIN		**120**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,05	**2,79**	**0,999**	**9,81**	**0,0302**	1,0165	0,68085	100,00
5 MIN		**300**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,05	**2,79**	**0,999**	**9,81**	**0,0191**	1,0165	0,68085	100,00
10MIN		**600**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,05	**2,79**	**0,999**	**9,81**	**0,0135**	1,0165	0,68085	100,00
30MIN		**1800**	26	1,017	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,19	**2,79**	**0,999**	**9,81**	**0,0078**	1,0160	0,66140	97,14

60 MIN		**3600**	26	1,017	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,19	**2,79**	**0,999**	**9,81**	**0,0055**	1,0160	0,66140	97,14
120 MIN		**7200**	26	1,017	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,19	**2,79**	**0,999**	**9,81**	**0,0039**	1,0160	0,66140	97,14
240 MIN		**14400**	26	1,0155	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,61	**2,79**	**0,999**	**9,81**	**0,0028**	1,0145	0,60304	88,57
1440 MIN		**86400**	26	1,014	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	13,04	**2,79**	**0,999**	**9,81**	**0,0012**	1,0130	0,54468	80,00

Tab2.5 sedimentometry (raw bentonite)

Di (mm)	*sieves (%)*
0,0637	100,00
0,0451	100,00
0,0302	100,00
0,0191	100,00
0,0135	100,00
0,0078	97,14
0,0055	97,14
0,0055	97,14
0,0028	88,57
0,0012	80,00

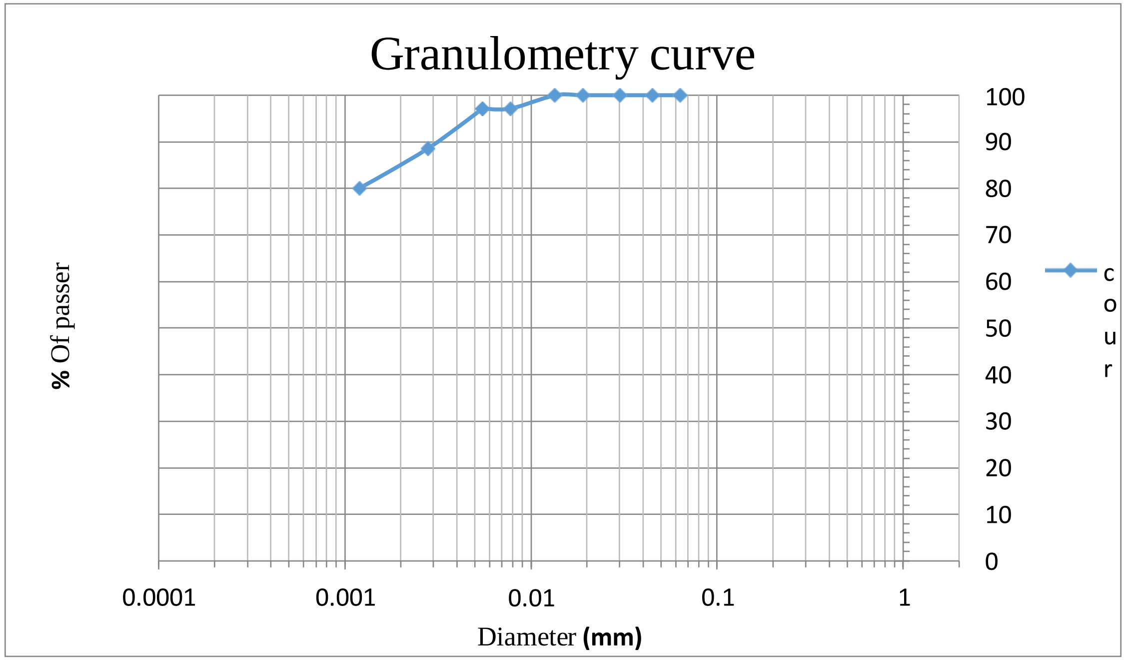


Diagram 2.4 Granulometry curve (raw bentonite)

4.8.2. Case of Industrial bentonite :

	time (sec)	temperature	lecture Rc Rc= R+Ct+Cm+Cd		Is equal to 0,03368	Is equal to 0,00022	η=0,00179/(1+αθ+Bθ2					density of solid particles	density of distilled water			density of the suspension		
	time (sec)	***θ(c°)***	***liquide (A)***	***temoin (B)***	***α***	***β***	***η***	***Hc (cm)***	***H(cm)***	***H1 (cm)***	***Ht(cm)***	***ρs (g/cm3)***	***ρw (g/cm3)***	***g(m/s2)***	***Di (mm)***	***ρt (g/cm3)***	***P(%)***	***% grains <à D***
30MIN	**30**	26	1,025	0,997	**0,03368**	**0,00022**	0,00088421	**0**	**18,4**	**2,83**	11,33	**2,55**	**0,999**	**9,81**	**0,0628**	1,0240	1,02654	100,00
1MIN	**60**	26	1,0225	0,997	**0,03368**	**0,00022**	0,00088421	**0**	**18,4**	**2,83**	12,03	**2,55**	**0,999**	**9,81**	**0,0458**	1,0215	0,92388	90,00
2MIN	**120**	26	1,022	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	10,77	**2,55**	**0,999**	**9,81**	**0,0306**	1,0210	0,90335	88,00
5 MIN	**300**	26	1,022	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	10,77	**2,55**	**0,999**	**9,81**	**0,0194**	1,0210	0,90335	88,00
10MIN	**600**	26	1,0205	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	11,20	**2,55**	**0,999**	**9,81**	**0,0140**	1,0195	0,84176	82,00
30MIN	**1800**	26	1,0195	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	11,48	**2,55**	**0,999**	**9,81**	**0,0082**	1,0185	0,80070	78,00

60 MIN	**3600**	26	1,0185	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	11,76	**2,55**	**0,999**	**9,81**	**0,0058**	1,0175	0,75964	74,00
120 MIN	**7200**	26	1,0175	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,05	**2,55**	**0,999**	**9,81**	**0,0042**	1,0165	0,71857	70,00
240 MIN	**14400**	26	1,015	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	12,76	**2,55**	**0,999**	**9,81**	**0,0030**	1,0140	0,61592	60,00
1440 MIN	**86400**	26	1,01	0,997	**0,03368**	**0,00022**	0,00088421	**1,4**	**18,4**	**2,83**	14,17	**2,55**	**0,999**	**9,81**	**0,0013**	1,0090	0,41061	40,00

Tab2.6 sedimentometry (industrial bentonite)

Di (mm)	***tamisas (%)***
0,0628	100,00
0,0458	90,00
0,0306	88,00
0,0140	82,00
0,0082	78,00
0,0058	74,00
0,0042	70,00
0,0030	60,00
0,0013	40,00

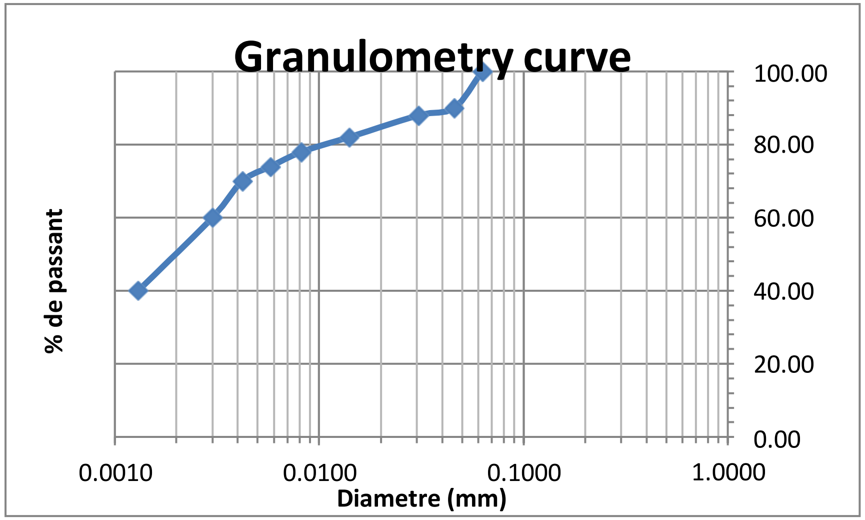


Diagram 2.5. Granulometry curve (industrial bentonite)

Determination of carbonate content, Calcimeter method :
NF P 94-048

4.4. Principle:

The experiment involves determining the volume of carbon dioxide (CO2) released under the action of excess hydrochloric acid (HCl) (under known temperature and atmospheric pressure conditions) by a sample prepared for the test.

4.5. Equipment :

- Calcimeter :

The calcimeter consists of a frame as well as (see the principle diagram in Figure 1):

- A reaction flask (5) of approximately 200 cm³ with its stopper mounted on the tube (6) connected to the condensing system and a test tube of at least 10 cm³ (or alternatively a reaction flask with a side arm of at least 10 cm).
- A cooling system (8).
- A burette of at least 150 cm³ graduated in at least one cubic centimeter increments (2) with a stopcock (3) at its upper end.
- A flask with a leveling bulb (1) of at least 100 cm³.
- A flexible tube (9) connecting the burette (2) to the leveling bulb (1).
- A cooling water bath (4) at room temperature.
- A stopwatch displaying seconds.

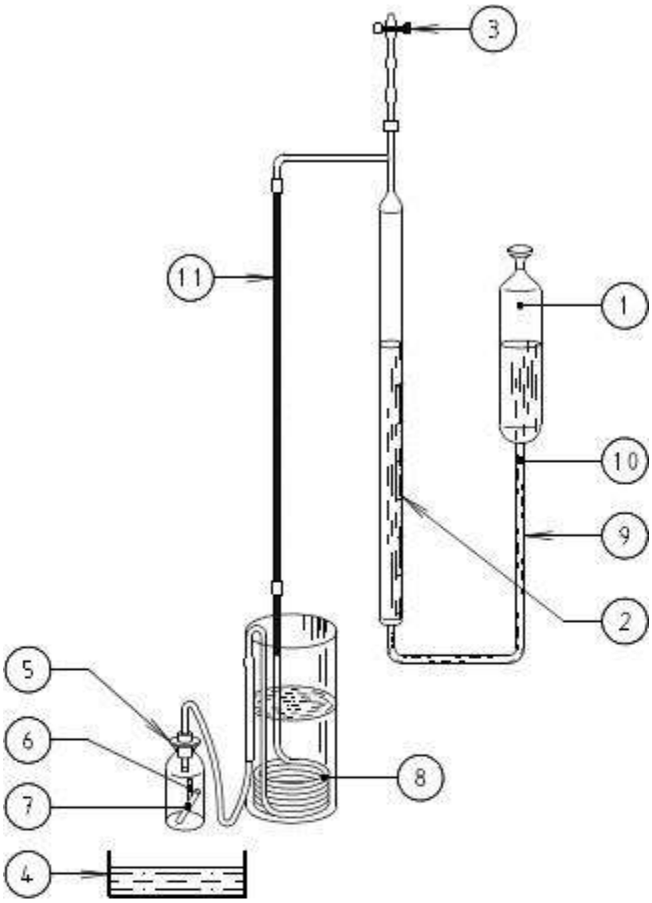


Figure 2.6 Calcimeter

(1)	Leveling bulb flask
(2)	Graduated burette
(3)	Stopcock
(4)	Cooling water bath at room temperature
(5)	Reaction flask
(6)	Test tube
(7)	Material under test
(8)	Cooling system
(9)	Flexible tubing connecting to
(10)	Liquid (with optional dye)
(11)	Gas released by the reaction

4.6. Materials and products :

- A mortar approximately 15 cm in diameter with its pestle for separating solid particles;
- A barometer (if necessary) with a measurement uncertainty of 1/1000 of the measured value. The usage range corresponds to the variation of atmospheric pressure at the test location;
- A balance with minimum and maximum capacities compatible with the masses to be weighed, ensuring weighings are performed with an uncertainty of 1/1000 of the measured value;
- A thermometer with a measurement accuracy of 1°C. The usage range of the thermometer is from 10°C to 35°C;
- A square mesh sieve with mesh openings of 400 µm or smaller;
- A pipette for hydrochloric acid;
- Standard laboratory equipment;
- A thermal chamber or drying oven with adjustable temperature at 105°C, accuracy class C, as defined in Appendix A of standard NF X 15-016, regarding temperatureonly;
- Distilled or demineralized water at room temperature;
- Concentrated hydrochloric acid with a density of 1.19 (recognized analytical quality);
- Pure calcium carbonate powder for analysis (if necessary).

4.7. Procedure:

The test is conducted on two test samples.

- Sample preparation

After preliminary drying, the material is ground in the mortar to obtain a powder that entirely passes through a sieve with mesh openings of 400 µm or smaller. The drying of the material continues at a temperature of 105°C until its mass does not vary by more than 2/1000 betweentwo successive weighings, spaced 1 hour apart.

After homogenization, take two test samples of identical mass according to Table 1.

In the absence of information on the presumed carbonate content, choose test samples with a mass of approximately 1 g.

Presumed percentage of bicarbonate content (%)	<4	4 à 9	10 à 19	20 à 39	40 à 79	≥80
mass of the dry sample aliquot in grams (g)	10	5	2,5	1	0,5	0,25

Tab2.7 Mass of a test sample depending on the presumed carbonate content

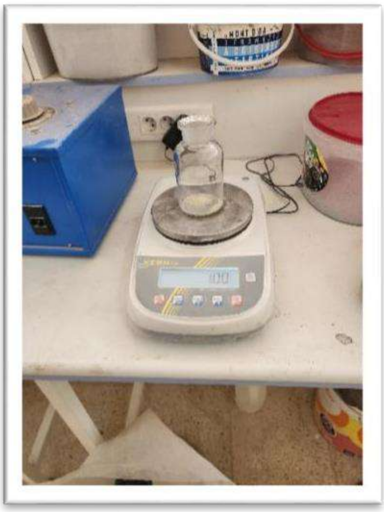


Figure 2.7 Sample preparation

4.8. Conduct of the test:

a) Introduce the test sample, once weighed (with mass m), into the clean and dry flask.

b) Set up a cooling system.

c) Fill the flask and burette with distilled or demineralized water. Align the low level of the flask with the zero mark at the top of the burette.

d) Introduce approximately 10 cm3 of concentrated hydrochloric acid into a test tube placed inthe flask (or, depending on the equipment used, into the side tubing of the flask using a curved-tip pipette).

e) Open the valve. Seal the reaction flask with its cap. The tubing passing through it is connectedto the top part of the burette.

f) Adjust the level of the flask to match the zero mark on the burette.

g) Close the valve. Bring the hydrochloric acid in the flask reservoir or test tube into contact with the material. As the gas evolution exerts pressure on the water in the burette, align the levels in the flask and burette by moving the flask. Agitate the flask until the water level stabilizes. Wait for approximately 5 minutes and place the flask in the cooling water bath at room temperature. After temperature equilibrium (about 5 minutes), bring the levels in the flaskand burette to the same horizontal plane and read the corresponding volume Vb on the burette. Note the temperature Tb of the test room and, if applicable, the atmospheric pressure.

h) Ensure that the gas collected in the burette does not contain hydrogen sulfide (H_2S) with itsvery characteristic odor. This would indicate that the test sample contained sulfides. If so, repeatthe test by adding a few cubic centimeters of 5% calcium acetate to the hydrochloric acid to neutralize any parasitic gas emissions in addition to carbon dioxide.

i) A continuous and slow gas evolution may be due to the presence of double calcium and magnesium carbonate $MgCa(CO_3)_2$ (dolomite). In this case, the test should be continued forat least 15 minutes.

j) Repeat the test with the second test sample.

Figure 2.8 Conduct of the test

4.9. Measures:

Simultaneously with the reading of the volume of gas evolved:

- Note the temperature of the test room.
- Record the atmospheric pressure.

In cases where one wishes to avoid measuring atmospheric pressure, the testing process described in paragraph 5.2 is applied to a reference sample of pure calcium carbonate. For this:

- Take, after oven-drying, a mass mt of 0.25 g of pure calcium carbonate for analysis.
- Proceed as described in paragraph 5.2.
- Read on the burette (2) the volume Vt of gas evolved.
- Note the temperature tt of the test room.

5.7 Expression of results :

CaCO3 content (%)	Geotechnical designation	
0 – 10	Clay	Soils
10 – 30	Marly clay	
30 – 70	Marl	
70 – 90	Marly limestone	Rocks
90 - 100	Limestone	

Tab 2.8 Classification of soils according to calcium carbonate content

5.7.1. Case of raw bentonite:

M= 1g

Hcl= 10 mlV=

44ml

Mass of CaCO3	Volume of CO2
100g	22400 ml
X g	44 ml

X = (44 * 100) / 22400X

= 0.20 g

Mass of CaCO3	Mass of the soil
0.20 g	1 g
Y g	100 g

Y = (0.20 * 100) / 1Y

= 20 g

So 100g of soil contain 20g of CaCO3: this sample contains 20% limestone.

That means it’s a « Marly clay »

5.7.2. Case of industrial bentonite:

M=1g

Hcl= 10mlV=

36ml

Mass of CaCO3	Volume of CO2
100g	22400 ml
X g	36 ml

X = (36 * 100) / 22400X

= 0.16 g

Mass of CaCO3	Mass of the soil
0.16 g	1 g
Y g	100 g

Y = (0.16 * 100) / 1Y

= 16 g

So 100g of soil contain 16g of $CaCO_3$: this sample contains 16% limestone.

That means it’s a « Marly clay »

5. Measurement of the methylene blue adsorption capacity of soil, Methylene Blue Value Test:

NF P 94-068

5.1. Definitions and symbols:

For the purposes of this document, the following definitions apply:

VBS: (Methylene Blue Value) represents the amount of methylene blue in a soil sample. It is expressed in grams of blue per 100 grams of the 0/50 mm fraction of the soil under study.

D_{max}: (Maximum Particle Size) is the maximum dimension of the largest elements present inthe soil (see NF P 11-301).

m_{h1} is the wet mass of the sample constituting the first test specimen (expressed in grams).

m_{h2} is the wet mass of the sample intended for drying, constituting the second test specimen (expressed in grams).

m_{h3} is the wet mass of the sample constituting the third test specimen (expressed in grams).

m_{s2} is the mass of the sample after drying, corresponding to the second test specimen (expressed in grams).

B is the mass of blue introduced into the solution (solution at 10 g/l).

V is the volume of the blue solution used (expressed in cubic centimeters).

C is the proportion of the 0/5 mm fraction in the 0/50 mm fraction of the dry material.w is

the moisture content, expressed as a decimal value.

5.2. Principle of the test :

The test consists of measuring, through titration, the quantity of methylene blue that can be adsorbed by the material suspended in water. This quantity is directly proportional to the 0/50mm fraction of the soil. The soil's methylene blue value is directly related to the specific surface area of the particles constituting the soil or the rock material.

The titration is performed by successively adding different quantities of methylene blue and controlling the adsorption after each addition. To do this, a drop of the suspension is taken and placed on a filter paper (see article 7), which creates a stain.

Maximum adsorption is reached when a persistent light blue halo appears at the periphery ofthe stain.

5.3. Testing apparatus and equipment :

- Specific equipment :

The specific equipment includes:

- A dosing device capable of injecting volumes of blue solution in increments of10 cm3, 5 cm3, and 2 cm3, and determining the total quantity injected with an accuracy of ± 1 cm3.
- A mechanical stirrer with blades, operating at speeds ranging from 400 rpm to 700 rpm. The blade diameter should be between 70 mm and 80 mm. The shape and dimensions of the blades should facilitate the movement of all soil particles.
- A cylindrical container (made of glass, plastic, or stainless steel) with a minimum capacity of 3,000 cm3 and an inner diameter of (155 ± 10) mm.
- A glass rod with a diameter of (8 ± 1) mm.
- Sieves with mesh sizes of 80 µm, 5 mm, and 50 mm.
- White filter paper with a basis weight of (95 ± 5) g/m2, thickness of (0.2 ± 0.02) mm, filtration speed of (75 ± 10) s for 100 ml (according to ASTM method), and retention diameter of (8 ± 5) µm.

- Current equipment :

The current equipment includes:

- Balances with scales compatible with the masses to be weighed and capable of measurements with a relative uncertainty of 0.2% of the measured value.
- Thermometer graduated in degrees (from 0 °C to 100 °C).
- Stopwatch or equivalent indicating seconds.
- Thermal chamber adjustable to 90 °C.
- Drying apparatus allowing determination of soil moisture content according to a standardized method.
- Hermetic bags or containers for preserving the moisture content of samples.

Figure 2.9 Methylene Blue Value test

5.4. Products :

The products to be used are as follows:

Methylene blue solution at (10 ± 0.1) g/l, manufactured less than one month ago and stored ina sealed container protected from light (the preparation method is described in Appendix A ofthis document).

Demineralized or distilled water.

Sample preparation :

- If the Dmax of the material is greater than 50 mm :

Take 10 kg of its 0/50 mm fraction, then proceed as indicated in 6.2.

- If the Dmax of the material is between 5 mm and 50 mm :
- Extracting a mass, m, of the wet material such that m > 200 Dmax (m in grams, Dmax in millimeters).
- Separating, through sieving and if necessary washing, the 0/5 mm fraction contained in this sample.
- Determining the weight proportion C of the 0/5 mm fraction (dry) contained in the material (or in its 0/50 mm fraction when Dmax > 50 mm).
- Proceeding as indicated in 6.3.

- If the Dmax of the material is less than or equal to 5 mm :

Extract a mass, m, of the wet material such that m > 200 Dmax (m in grams, Dmax in millimeters).

Quarter and homogenize the 0/5 mm fraction in order to prepare three test portions of approximately equal masses, approximately:

a) 30 g to 60 g in the case of highly clayey to clayey soils;

b) > 60 g in the case of moderately to slightly clayey soils.

The first test portion with mass mh1 is introduced into the 3,000 cm3 container, suspended in (500 ± 10) cm3 of demineralized water, and dispersed using the paddle agitator (speed (700 ± 100) rpm and positioned approximately 5 mm from the bottom of the container), for a minimum of 5 minutes and in all cases until the visual disappearance of any soil particle agglomeration in the suspension or any accumulationon the edges of the container.

The second test portion with mass mh2, intentionally reduced compared to the specifications of the water content standards, is used to determine the water content of each test portion.

The third test portion with mass mh3 is retained in case the test needs to be repeated.

5.5. Expression of results :

6.5.1. Case of raw bentonite :

V=380ML

M=30g

VBS = V / M = 12.66

SST = 20.93 * 12.66 = 265.11

VBS > 8 => Very clayey soil 6.5.2.

Case of industrial bentonite :

V = 350ml M =

30g VBS =

11.66

SST= 244.18

VBS > 8 => Very clayey soil

6. Determination of the density of soil solid particles using the water pycnometer method:

NF P 94-054

6.1. Object :

This standard aims to determine, using the water pycnometer, the density of solid soil particles.

It defines the terms used and the parameters measured, specifies the characteristics of the equipment, establishes the operating procedure, and outlines the results to be presented

6.2. Method :

The soil sample is dried in an oven and then weighed. The volume of the particles is determinedby weighing using a pycnometer, substituting water of known density for the solid particles.

6.3. Apparatus :

The following equipment is required:

- A thermal chamber or a drying oven with adjustable temperature settings at 50°C and 105°C, accuracy class C as defined in Annex A of the NF X 15-016 standard,
- A balance with minimum and maximum capacities compatible with the masses to be weighed, ensuring that weighings are performed with an uncertainty of ± 1/1,000of the measured value,
- A sieve with 2 mm square mesh openings,
- Drying dishes for the soil,
- A mortar with a pestle made of a soft material,
- Pycnometers with a minimum volume of 50 cm³, equipped with ground-glass stoppers with capillary tubes,
- A supply of at least 10 liters of distilled or demineralized water,

- A heat source capable of bringing the water in the pycnometer flask to a boil,
- A connection to a vacuum system capable of reducing the pressure to a value below 2.5 kPa and a device for measuring the pressure used,
- A control thermometer permanently placed in the test room.

6.4. Operating Procedure :

During the duration of the tests, the room temperature must be maintained within 3°C. The various steps to determine the density of the solid particles are described below.

6.5. Sample and Equipment Preparation :

First, ensure the provenance and geological nature of the soil, and perform a preliminary visual identification to determine if the soils are gypsiferous, lateritic, or if they contain organic matter. These soils are sensitive to heat. In case of doubt, treat the soil as if it were heat-sensitive.

A test sample of approximately 25 g is taken from the soil sample passing through the 2 mm sieve, and placed in a dish of known mass (m). The entire assembly is then placed in an oven at:

- 105°C if the soils are not heat-sensitive.
- 50°C if the soils are heat-sensitive. The drying duration varies between 1 and 8 days.

Drying is considered complete when the mass (md) of the sample does not vary by more than 2/1,000 between two weighings conducted immediately after removal from the oven, at least 4 hours apart.

The agglomerates of particles in the test sample are then separated using a pestle in a mortar.The

pycnometer and its stopper are weighed (m1) after ensuring they are clean and dry.

6.6. Pycnometer Test Procedure :

The trial sample is introduced into the pycnometer. The pycnometer is cleared of any particlesthat may have adhered to its external surface and then weighed with its cap (m1). The pycnometer's bulb is then filled with distilled or demineralized water maintained at room temperature. To ensure that no air bubbles remain attached to the particles, two procedures canbe performed depending on the sensitivity of the soils to heat:

a) by depression :

For all materials, and mandatory for those sensitive to heat such as gypsum, organic soils, laterites, etc., the liquid contained in the pycnometer is placed under vacuum. For this, the pycnometer is placed under vacuum, for at least 30 minutes and until complete disappearance of any gas release. After returning the bulb and its contents to atmospheric pressure, visually check that no gas bubbles are released after agitation. If this is not the case, repeat the process.

b) <u>by boiling :</u>

For materials insensitive to heat, the liquid contained in the pycnometer is brought to a boil for at least one hour. During this operation, no solid particles should escape from the bulb. Once the pycnometer and its contents have returned to room temperature, visually check thatno gas bubbles are released after agitation. If this is not the case, repeat the process.

When deaeration is completed, the cap with its marked tube is placed on the pycnometer bulb. The filling is done with distilled or demineralized water up to the mark and topped up, if necessary, after 30 minutes. The pycnometer is dried and weighed (m2). Then the pycnometeris emptied, cleaned, and refilled with distilled or demineralized water up to the mark on the cap. After 30 minutes, the level is again brought into coincidence with the mark. The assembly, once wiped, is weighed (ma).

Figure 2.10 Soil preparation by boiling

6.7. <u>Expression of results</u> :

The density of solid particles is determined from the following formula: ρs =

ρw $(M_2 - M_1) / (M_4 + M_2 - M_1 - M_3)$

where:

M_1 is the mass of the empty pycnometer,

M_2 is the mass of the pycnometer containing the test sample, M_3

is the mass of the pycnometer, soil, and water,

M_4 is the mass of the pycnometer filled with water,

ρw is the density of water conventionally taken as 1,000 kg/m^3.

The value of the density of solid particles is the arithmetic mean of two measurements performed on two test samples from the same soil sample. The rounding interval is 10 kg/m^3

7.7.1. Case of raw bentonite:

M_1= 177.17g

M_2= 203g M_3=

455g M_4=

438.4g

$\rho s = 2.79\ g/cm^3$

7.7.2. Case of industrial bentonite :

M1= 177.17g

M2= 201.98g

M3= 453.5g

M4= 438.4g

$\rho s = 2.55\ g/cm^3$

7. **Conclusion** :

The characterization of raw and industrial bentonite reveals distinct differences across several physical, chemical, and mechanical properties. The Standard Proctor Test shows comparable compaction properties, with raw bentonite requiring slightly more moisture for optimal compaction. The Atterberg limits indicate higher plasticity for industrial bentonite, making it more suitable for applications involving high water retention and swelling.

Further, industrial bentonite exhibits a more controlled particle size distribution, higher cation exchange capacity, greater swelling capacity, and a larger specific surface area. These enhanced properties of industrial bentonite result from extensive processing and purification, which makeit more effective and versatile for various industrial and engineering applications compared to raw bentonite. Understanding these differences is crucial for selecting the appropriate type of bentonite for specific use cases, ensuring optimal performance and efficiency.

Chapter 3

Drilling and the use of bentonite in it

1. Introduction :

Drilling, an essential technique in the oil, gas, and geothermal industries, requires specific materials to optimize performance and ensure operational safety. Bentonite, a clay naturallyrich in montmorillonite, plays a crucial role in this field. Primarily used for its suspension and lubrication properties, bentonite significantly enhances drilling efficiency by stabilizing well walls, controlling fluid pressure, and facilitating the removal of cuttings. This chapterdelves deeply into the physicochemical and mechanical characteristics of bentonite and examines how these properties can be leveraged to optimize drilling processes. Through detailed analyses and case studies, we will highlight the importance of bentonite in improving modern drilling techniques.

2. Generalities of Drilling in Oil and Gas Industry:

3. 2.1.Overview of Drilling :

Drilling in the oil and gas industry is the process of creating boreholes or wells to extract petroleum and natural gas from underground reservoirs. This process is highly complex and requires careful planning, precise execution, and advanced technology. [16, 17, 18]

2.2. Objectives of Drilling :

Exploration: To discover new oil and gas reserves.

Development: To develop known reserves by drilling production wells.

Production: To maximize the extraction of hydrocarbons from a reservoir.

Enhanced Recovery: To improve the extraction efficiency of existing wells.

2.3. Drilling Process :

The drilling process can be divided into several stages:

- Site Preparation: Before drilling starts, the site must be prepared. This includes building access roads, setting up the drilling rig, and ensuring that all necessary equipment and materials are available.
- Spudding: The initial phase where the well is started by drilling a large diameter hole for the surface casing.
- Drilling: Using the rotary drilling method, the drill bit is rotated to cut through the earth's layers. Drilling continues until the desired depth is reached. This includes the use of drilling muds to facilitate the process.
- Casing and Cementing: After reaching a specific depth, steel pipes (casings) are placedin the hole and cemented in place to ensure the well's integrity and prevent collapse.
- Completion: Once the target depth is reached, the well is completed, which involves placing the production casing and preparing the well for production.
- Production: Extracting oil or gas from the well.
- Abandonment: When a well no longer produces economically viable amounts of oil orgas, it is plugged and abandoned in accordance with regulatory requirements.

4. Types of Drilling :

Drilling techniques have evolved over the years to address various geological challenges and improve efficiency. The primary types of drilling used in the oil and gas industry include: [19,20,21]

- Rotary Drilling :

Principle:

Rotary drilling involves rotating a drill bit attached to a drill string to cut through rock formations.

Components:

- Drill Bit: The cutting tool at the end of the drill string.
- Drill String: A series of connected pipes that transmit rotational force from the rig to the drill bit.

Rotary Table or Top Drive: Equipment that provides the rotational force.

- Directional Drilling :

Principle:

Directional drilling allows the wellbore to be drilled at multiple angles, not just vertically. This technique is essential for accessing reserves that are not directly beneath the drilling rig.

Applications:

- Horizontal Drilling: A type of directional drilling where the well is turned horizontally within the reservoir to maximize exposure to the oil or gas.
- Extended Reach Drilling (ERD): Used to reach targets that are far from the drilling site, extending the horizontal reach of the well.
- Multilateral Drilling: Involves drilling multiple lateral wells from a single vertical wellbore, enhancing reservoir exposure.

- Horizontal Drilling :

Principle:

Horizontal drilling is a specific type of directional drilling where the wellbore is curved from a vertical to a horizontal direction within the reservoir.

Benefits:

- Increased Production: Greater exposure to the reservoir.

Reduced Environmental Impact: Fewer surface disruptions since multiple horizontal wells canbe drilled from a single pad.

- Enhanced Recovery: More effective drainage of the reservoir.

- Vertical Drilling :

Principle:

Vertical drilling involves drilling straight down from the surface to the target reservoir.

Application :

Common in simpler geological settings where the reservoir is directly beneath the drilling site.

- Multilateral Drilling :

Principle:

Multilateral drilling involves creating multiple branching wells from a single main borehole.

Benefits:

Enhanced Reservoir Contact: Maximizes the contact area with the reservoir.

Cost Efficiency: Reduces the number of surface facilities needed.

- Extended Reach Drilling (ERD) :

Principle:

ERD is a form of directional drilling that extends the horizontal reach of the well to access distant reserves.

Applications :

- Offshore Drilling : Allows access to offshore reserves from onshore drilling sites.
- Environmental Protection : Minimizes the footprint by reducing the number of drilling sites.

5. **Key Components of a Drilling Operation**: [22,23,24]

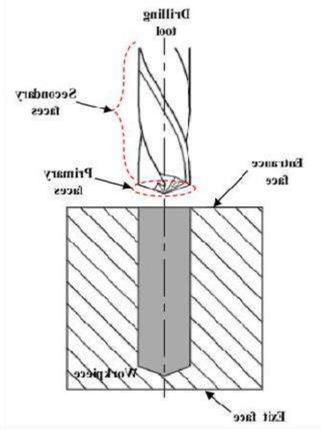


Diagram 3.1 Schematic diagram of drilling tool operation

A drilling operation involves various components and systems working together to efficiently and safely drill a well.

A. Drilling Rig :

A.1. Types:

- Land Rigs: Used for onshore drilling operations.
- Offshore Rigs : Includes jack-up rigs, semi-submersible rigs, and drillships for offshore drilling.

A.2. Components:

- Derrick or Mast: The tall structure that supports the drilling equipment.
- Substructure : The base that supports the derrick and other equipment.
- Drilling Floor: The platform where drilling operations are conducted.

B. Drill String :

B.1. Components:

- Drill Pipe: Steel pipes that transmit rotational force and drilling fluids.
- Drill Collars : Heavy, thick-walled tubes placed near the bit to apply weight on the drillbit.
- Kelly or Top Drive: Equipment that rotates the drill string.

C. Drill Bit :

C.1. Types:

- Roller Cone Bits: Have rotating cones with teeth that crush the rock.
- Fixed Cutter Bits: Includes polycrystalline diamond compact (PDC) bits and natural diamond bits that grind through rock.

C.2. Selection:

Depends on the rock formation and drilling conditions.

D. Mud Pumps :

D.1. Function:

Circulate drilling fluid (mud) through the drill string and back to the surface.

D.2. Types:

Triplex or duplex pumps, depending on the required pressure and flow rate.

E. Blowout Preventer (BOP) :

E.1. Function: A safety device used to prevent blowouts by sealing the wellbore in case of uncontrolled pressure surges.

E.2. Components: Includes rams and annular preventers that close around the drill pipe.

F. Drilling Fluid System :

F.1. Components:

- Mud Tanks : Store drilling fluids.
- Shale Shakers : Remove cuttings from the drilling fluid.
- Degassers : Remove gas from the drilling fluid.
- Centrifuges : Separate solids from the drilling fluid.

G. Control Systems :

G.1. Functions: Monitor and control drilling parameters such as weight on bit, torque, and mud flow.

G.2. Components: Include sensors, computer systems, and control panels.

4.1 Detailed Application in Drilling Operations:

1. Case Studies and Examples :

- High-Pressure High-Temperature (HPHT) Wells :

Bentonite-based muds are often used in HPHT wells due to their stability and ability to control filtration loss.

- Unconsolidated Formations :

In formations with loose or unconsolidated materials, bentonite helps prevent sloughing and caving by stabilizing the borehole walls.

2. Comparative Analysis :

- Water-Based vs. Oil-Based Muds :

Bentonite is primarily used in water-based muds. Compared to oil-based muds, bentonite muds are less expensive and environmentally friendly but may have limitations in highly reactive shales where oil-based muds perform better.

- Polymer-Based Muds :

In some cases, synthetic polymers are used instead of or in addition to bentonite to enhancemud properties. Polymers can provide better control over rheology and filtration but are usually more expensive.

3. Environmental Considerations :

- Non-Toxic and Biodegradable :

Bentonite is a natural clay, making it an environmentally friendly option compared to synthetic additives.

- Disposal and Recycling :

Used bentonite mud can be processed and recycled, reducing waste and environmental impact.

5. Use of Bentonite in Oil Drilling:

6. 5.1. Bentonite in Drilling Muds:

Bentonite is a type of clay that is primarily composed of the mineral montmorillonite. It is widely used in oil drilling due to its unique physicochemical properties, which make it an excellent component for drilling fluids. [25, 26, 27]

Bentonite serves several key functions:

5.2. Thixotropy:

Thixotropy is the property of certain fluids to become less viscous over time when subjected toshear stress and to regain viscosity when the stress is removed. This reversible transformation is crucial for drilling muds.

Its thixotropic nature means it becomes fluid when agitated and gels when static, aiding in borehole stability.

- Thixotropic Behavior of Bentonite Muds:
- Gel Formation :

When at rest, bentonite particles in the drilling mud form a network-like structure, causingthe mud to gel. This gel strength helps in suspending cuttings and preventing them from settling when circulation stops.

- Shear Thinning :

Under shear stress, such as during circulation, the structure breaks down, and the fluid becomes less viscous (shear thinning), allowing for easier flow.

- Regelation :

When the shear stress is removed, the bentonite particles reorganize, and the viscosity increases again, allowing the fluid to regain its gel strength.

- Importance in Drilling Operations :
- Cuttings Suspension :

Thixotropy ensures that cuttings remain suspended when the fluid is static, preventing themfrom settling and clogging the wellbore.

- Easier Pumping :

The shear-thinning property makes it easier to pump the drilling fluid through the drill stringand the annulus, reducing the energy required for circulation.

- Wellbore Stability :

The ability to form a gel when static helps maintain wellbore stability and supports the borehole walls, particularly important during pauses in drilling.

- Measuring Thixotropy :
- Gel Strength Measurements :

Gel strength is typically measured using a viscometer by determining the force required to break the gel structure after the fluid has been static for a certain period (usually measuredin lb/100 ft²).

- Rheological Tests :

Advanced rheological tests can measure the change in viscosity under varying shear rates and provide a detailed profile of the thixotropic behavior.

- Control and Optimization :
- Additives and Formulation :
  Polymers : Adding polymers to bentonite muds can enhance thixotropic properties, providing better control over viscosity and gel strength.
  pH Adjustment : The pH of the drilling fluid can affect the swelling and dispersion of bentonite particles. Maintaining a slightly alkaline pH (8.5-9.5) is optimal for bentonite performance.
  Weighting Agents : Materials like barite can be added to adjust the density of the drillingfluid without significantly impacting its thixotropic properties.
- Monitoring and Maintenance :
  Regular Testing : Continuous monitoring of viscosity and gel strength is essential to ensure the drilling fluid performs optimally. Adjustments can be made based on test results.
  Fluid Circulation: Proper circulation practices help maintain the desired properties of the drilling fluid, ensuring efficient drilling and wellbore stability.

5.3. Viscosity:

Viscosity is a measure of a fluid's resistance to flow. In the context of drilling fluids, it indicateshow easily the fluid can circulate through the wellbore, suspend cuttings, and transport them tothe surface.

- Types of Viscosity :
- Dynamic Viscosity :

Also known as absolute viscosity, it is the measure of a fluid's internal resistance to flow. Itis typically measured in poise or centipoise (cP).

- Kinematic Viscosity :

It is the dynamic viscosity divided by the fluid's density, measured in stokes or centistokes.

- Importance in Drilling Muds :
- Suspending Cuttings :

Adequate viscosity ensures that the drilling fluid can suspend and transport cuttings to the surface. Low viscosity fluids may fail to carry cuttings, leading to their accumulation at the bottom of the wellbore.

- Preventing Settling :

High viscosity helps prevent the settling of cuttings and weighting materials when circulation is stopped.

- Maintaining Circulation :

Optimal viscosity ensures smooth circulation of the drilling fluid, reducing the risk of blockages and ensuring efficient cooling and lubrication of the drill bit.

- Measuring Viscosity :
- Marsh Funnel :

A simple device used to measure the time it takes for a set volume of drilling fluid to flow through a funnel. The time is correlated with viscosity.

- Rheometers and Viscometers :

More precise instruments, such as rotational viscometers, measure the shear stress and shear rate of the fluid to determine its viscosity.

5.4. Stabilization:

Stabilization in the context of drilling fluids primarily refers to the maintenance of borehole integrity and prevention of wellbore collapse. Bentonite plays a crucial role in this process through several mechanisms.

A. Mechanisms of Borehole Stabilization :

- Formation of a Filter Cake :
- Filter Cake Formation:

Bentonite particles create a thin, low-permeability layer on the wellbore walls, known as a filter cake. This layer is formed by the deposition of bentonite particles as the fluid phase of the mud filters into the formation.

The filter cake reduces fluid loss into the formation and stabilizes the borehole by providing mechanical support to the wellbore walls. This is crucial in preventing the collapse of the borehole and maintaining wellbore stability.

A.1. Pressure Control :

- Hydrostatic Pressure :

The bentonite-based drilling fluid exerts hydrostatic pressure on the wellbore walls, counterbalancing the formation pressures. This prevents the inflow of formation fluids intothe wellbore and avoids blowouts.

- Pressure Balancing :

Maintaining the correct mud weight is essential for ensuring that the drilling fluid pressureis greater than the formation pressure but not so high as to fracture the formation.

A.2. Sealing Micro-Fractures :

- Micro-Fracture Sealing :

Bentonite particles are small enough to penetrate and seal micro-fractures in the formation.This helps in reducing fluid loss and enhancing borehole stability.

- Preventing Fluid Invasion :

By sealing these micro-fractures, bentonite helps prevent the invasion of drilling fluid intothe formation, which can cause wellbore instability and formation damage.

7. Advantages of Bentonite for Stabilization : _

6.1.Swelling and Gel Formation :

- Swelling Capacity :

Bentonite's ability to swell in the presence of water is a key factor in its stabilizing properties. The swelling increases the volume of the bentonite, which helps in forming a robust filter cake.

- Gel Formation :

When at rest, bentonite forms a gel-like structure that provides additional support to the wellbore. This gel structure helps in suspending cuttings and preventing their settling when circulation is stopped.

6.2.Rheological Properties :

- Thixotropy :

As mentioned earlier, bentonite exhibits thixotropic behavior. This means the mud becomes less viscous under shear (during circulation) and more viscous when static. This property helps maintain the stability of the wellbore by providing support during pauses indrilling.

- Viscosity Control :

The ability to control viscosity through the concentration of bentonite and the addition of other additives allows for the optimization of mud properties for specific drilling conditions.

8. **Challenges and Mitigation** : _

7.1.Mud Weight Management :

- Optimal Mud Weight :

It is crucial to maintain the correct mud weight to ensure sufficient hydrostatic pressure while avoiding formation fracturing. Overly high mud weight can lead to lost circulation, while too low a mud weight can cause wellbore collapse.

- Additives :

Weighting agents like barite can be added to increase mud weight without significantly affecting the rheological properties of bentonite-based mud.

7.2. Filter Cake Quality :

- Thin and Impermeable :

The ideal filter cake is thin yet impermeable. A thick filter cake can lead to stuck pipe issues, while an impermeable cake prevents excessive fluid loss.

- Monitoring and Adjustment :

Continuous monitoring of the filter cake properties is essential. Adjustments in the concentration of bentonite and the use of filtration control additives help maintain an optimal filter cake.

7.3. Compatibility with Formation :

- Formation Interaction :

Bentonite muds can interact with certain formations, such as reactive shales, leading to wellbore instability. This requires careful formulation and the possible inclusion of inhibitive additives.

- Chemical Additives :

Additives like potassium chloride (KCl) and other shale inhibitors can be used to stabilize reactive shales and prevent swelling and dispersion.

9. Best Practices for Stabilization with Bentonite : _

8.1.Pre-Drilling Planning : _

- Mud Program Design :

Designing a comprehensive mud program that considers the geological conditions of the wellbore. This includes selecting the appropriate type and concentration of bentonite and other additives.

- Geological Surveys :

Conducting thorough geological surveys to understand the formation characteristics and potential challenges.

8.2. Real-Time Monitoring :

- Monitoring Systems :

Utilizing real-time monitoring systems to continuously measure mud properties, including viscosity, density, pH, and filtration rates.

- Adjustments on the Fly :

Making on-the-fly adjustments based on real-time data to maintain optimal mud propertiesand ensure wellbore stability.

8.3. Maintenance and Recycling :

- Mud Recycling :

Implementing mud recycling systems to reclaim and reuse bentonite mud. This not only reduces costs but also minimizes environmental impact.

- Treatment Systems :

Using treatment systems to remove contaminants and restore the mud properties, ensuring consistent performance throughout the drilling operation.

10. Physicochemical Properties of Bentonite:

- Swelling Capacity:

Bentonite swells when hydrated, increasing its volume several times, which is crucial for forming a robust filter cake.

- Cation Exchange Capacity (CEC):

Bentonite can exchange cations, which affects its interaction with the formation and other components of the drilling fluid.

- PH:

The pH of bentonite affects its dispersion and interaction with other drilling fluid components.

11. Mechanical Properties:

Yield Point:

Bentonite's yield point helps determine the carrying capacity of the drilling mud.

Plastic Viscosity:

The plastic viscosity of bentonite-based muds affects the ease of circulation and the efficiency of cuttings transport.

12. Advantages of Using Bentonite in Drilling Muds

- Non-toxic and Environmentally Friendly:

Bentonite is a natural material, making it less harmful to the environment compared to synthetic additives. :[28,29,30]

- Cost-effective :

Bentonite is relatively inexpensive and readily available.

- Effective in Various Formations :

Its properties make it suitable for use in different types of geological formations.

13. Preparation and Use in Drilling Operations:

- Mixing:

Bentonite is mixed with water and other additives to create the drilling mud. The concentration and formulation depend on the specific drilling requirements.

- Monitoring and Maintenance:

The properties of the bentonite mud are continuously monitored and adjusted during drilling to ensure optimal performance.

14. The Thixotropy test:

13.1. Objective of Thixotropy Test on Bentonite:

The primary objective of conducting a thixotropy trial on bentonite is to understand and quantify its thixotropic properties.

The specific objective of our thixotropy trial, which involves testing both raw and industrial bentonite, is to compare the thixotropic properties of the two types. This comparison will help determine if the industrial processing of bentonite enhances and accelerate its thixotropic effect, making it more suitable and to save more time for specific applications like the drilling oil.

13.2. Apparatus:

1. Bentonite Samples : Prepare two samples of both raw and industrial bentonite accordingto the required specifications.

2. Beakers: Two beakers for each type of bentonite to hold the suspensions.

3. Stopwatch: To keep track of agitation and rest periods.

4. Laboratory balance: To accurately weigh the bentonite and water for the suspension.

5. Steel Tube: A 15 cm steel tube for containing the bentonite suspensions. Replacing the drill. 1.92 cm Interior Diameter (diameter inside) and 2.2 cm Exterior Diameter (diameter outside).

6. Distilled water: for preparing a homogeneous suspension

7. Mechanical stirrer: for homogenizing the suspension.

8. Reference soil: to replace the soil during drilling.

9. A container: For emptying the beaker of the bentonite once, the resting time is over.

10. Spatula: to mix the bentonite sample thoroughly just before the test.

11. Vernier caliper: to measure the diameter of the hole after the test.

Figure 3.1 Mechanical stirrer

Figure 3.2 Steel Tube

Figure 3.3 Laboratory balance

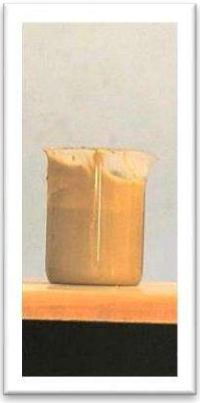


Figure 3.4 Liquid Bentonite

Figure 3.5 a container

Figure 3.6 Distilled water

Figure 3.7 Spatula

Figure 3.8 Vernier caliper

Figure 3.9 Beaker

13.3. Procedure:

1. Measure 200 g of bentonite using a laboratory balance and place it into a beaker.
2. Weigh an amount of water equivalent to the previously determined liquid limit of the bentonite.
3. Mix the bentonite with the water in a mechanical stirrer until achieving a perfectly liquid mixture, adding the water gradually.
4. Position a steel tube of defined diameter at the center of another beaker.
5. Introduce the reference soil into the beaker around the steel tube.
6. Apply the prepared bentonite onto the reference soil and inside the tube until it is completely filled.
7. Start the timer and let it rest for one minute.
8. Gently remove the steel tube and place it into an appropriate container.
9. Empty the beaker of any excess liquid bentonite to ensure complete hole emptying, usethe same container as used for removing the steel tube.
10. Precisely measure the diameter of the hole formed.
11. Repeat the process, varying the duration of the procedure at each iteration, meticulously recording the data obtained at each step.

Figure 3.10 The Thixotropy test

15. Conclusion:

This chapter has highlighted the indispensable role of bentonite in drilling operations. Thanks to its unique properties, such as swelling capacity, suspension power, and thermal stability, bentonite has proven to be a material of choice for optimizing drilling performanceand ensuring the stability of deep wells. Detailed analyses have demonstrated that incorporating bentonite into drilling fluids not only stabilizes well walls but also minimizesthe risks of cavity formation and structural failures. These properties contribute to better pressure management and reduced operational costs. In conclusion, bentonite remains an essential component in the arsenal of drilling technologies, and its future applications promise to be even more diverse and effective thanks to continuous advancements in material modification and characterization techniques.

Chapter 4

Measurement and Characterization of Thixotropy

1. **Introduction**:

Thixotropy, characterized by the fluidization of materials under agitation and their solidification at rest, is essential in various industrial sectors. This chapter focuses on methodsof characterizing thixotropy, specifically studying bentonite, a clay with remarkable rheological properties. We compare two types of bentonite: raw bentonite and industrial bentonite. ThroughScanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) analyses, we examine themorphological and mineralogical differences between these two forms of bentonite. Additionally, we evaluate the thixotropic phenomenon by measuring the time required for eachtype of bentonite to exhibit this effect. Our results show that industrial bentonite develops a thixotropic effect more quickly due to industrial treatments, leading to a more impermeable andefficient filter cake.

2. **Scanning Electron Microscopy (SEM)**: _

Scanning Electron Microscopy (SEM) is an advanced imaging technique that uses an electron beam to obtain high-resolution images of a sample's surface. Widely employed in various fieldssuch as materials science, biology, and metallurgy, this method is based on several fundamentalprinciples. An electron beam is generated and focused on the sample, where it interacts with the surface, producing various signals, including secondary and backscattered electrons. Thesesignals are then collected by detectors to form a detailed grayscale image, revealing the sample'stopography.

The SEM process involves several essential steps: sample preparation, which may include a conductive coating for non-conductive samples; placement of the sample in the microscope chamber; evacuation of the chamber to create a vacuum; alignment and focusing of the electron beam; adjustment of the microscope's parameters based on the specific characteristics of the sample; and finally, acquisition and analysis of the obtained images.

The advantages of SEM include extremely high resolution, greater depth of field compared to optical microscopes, and the ability to perform compositional analysis through additional detectors such as Energy Dispersive X-ray Spectroscopy (EDX) for elemental analysis. However, this technique also has limitations, such as the need for specific sample preparation,the requirement for a vacuum environment, and the high costs and complexity of using the equipment. Despite these challenges, SEM remains an essential tool for detailed examination of material microstructure in numerous scientific and industrial applications.

Figure 4.1 TM1000 Tabletop Microscope

Figure 4.2 the microscope chamber

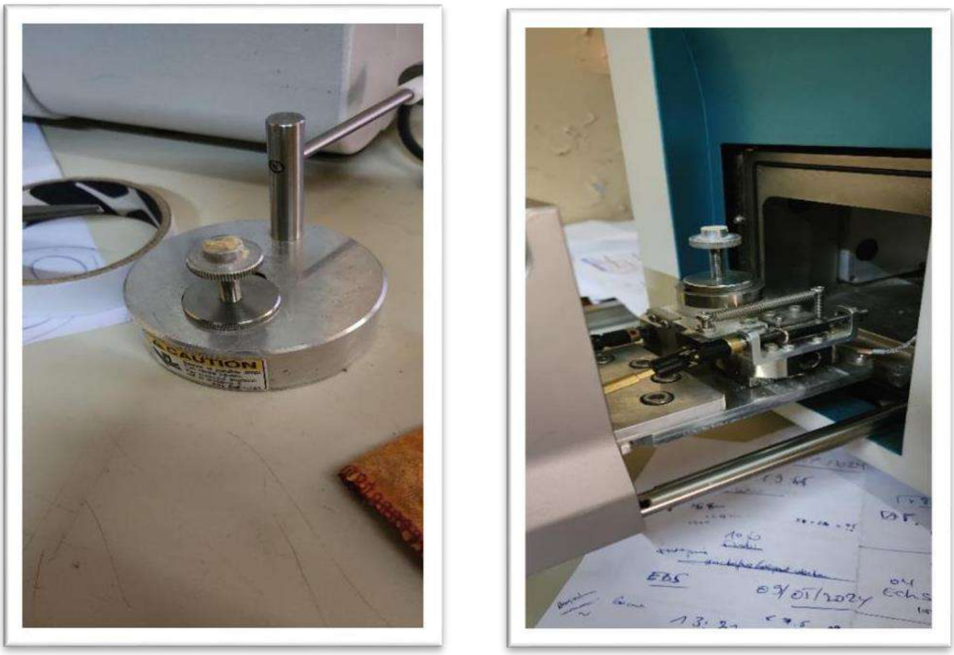


Figure 4.3 Placement of the sample in the microscope chamber

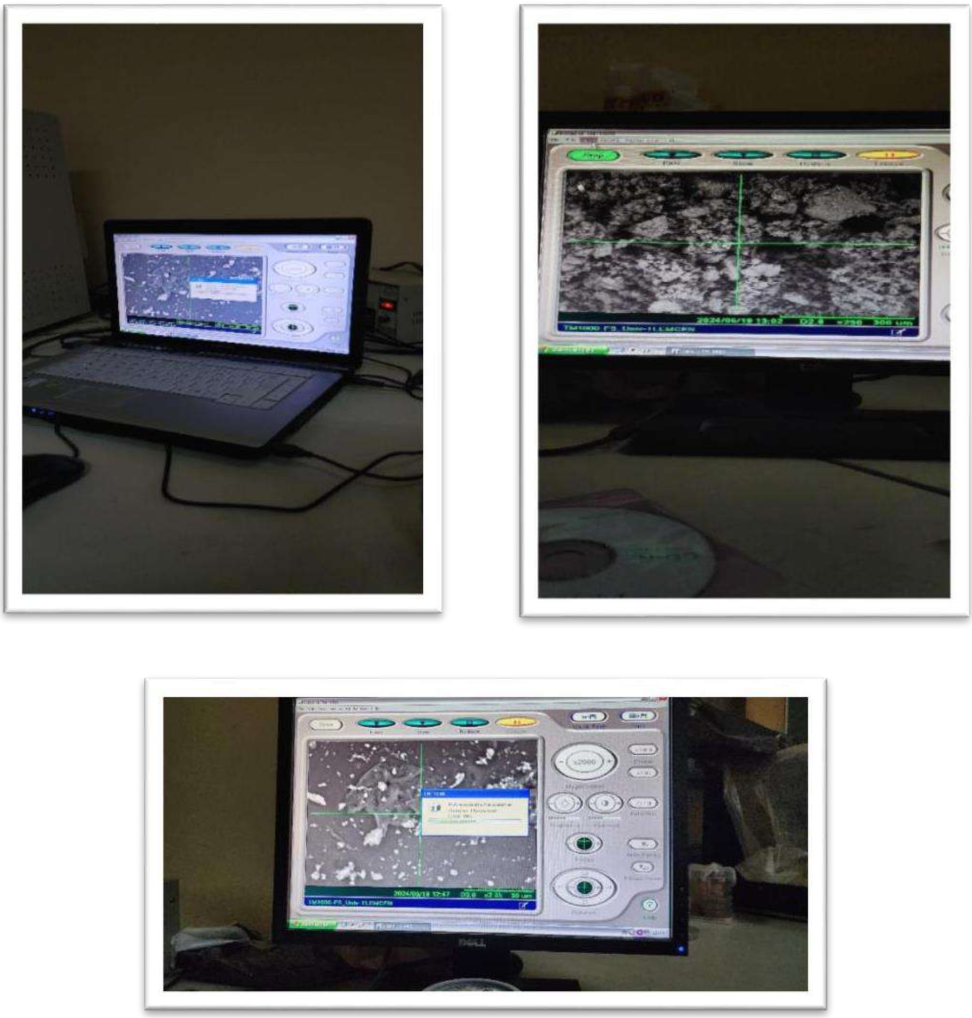


Figure 4.4 Acquisition and analysis of the obtained images

2.1. Discussion of Results:

Samples of raw and industrial bentonite were observed at magnifications of 250 and 1000 times respectively, using the TM1000-FS scanning electron microscope at the University of Tlemcen.The captured images provide a basis for detailed comparison of surface structures.

2.2. Results:

2.2.1. Industrial Bentonite:

Observation at 1.0k magnification reveals well-defined and densely compacted particle aggregates in industrial bentonite. Its structure is notably homogeneous, characterized by fine and regularly arranged particles. Particle compaction suggests increased organization and uniformity resulting from industrial treatments.

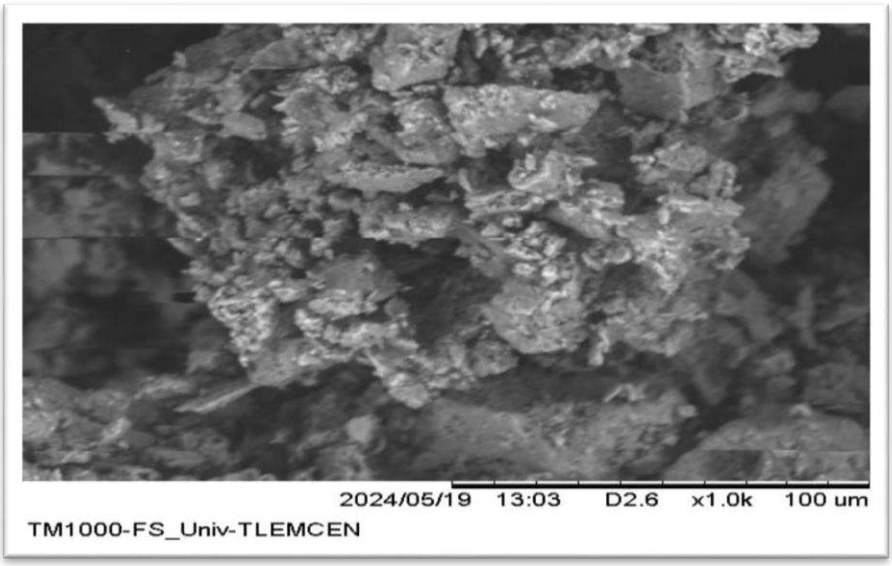


Figure 4.5 Observation at 1.0k magnification (industrial)

At a lower magnification of 250x, the aggregates appear coarser and less regular, but the distribution of particle sizes remains relatively structured.

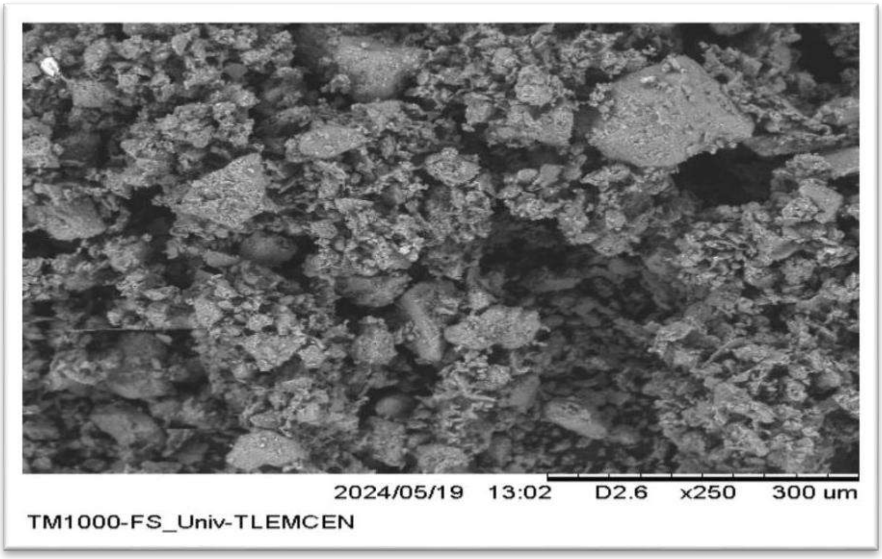


Figure 4.6 Observation at 250 magnification (industrial)

2.2.2. Raw Bentonite:

At 1.0k magnification, raw bentonite exhibits a more heterogeneous structure, with irregularly distributed particle aggregates. Particles show less compaction and a wider range of sizes, lending the texture a more disordered appearance typical of the absence of industrial treatment.

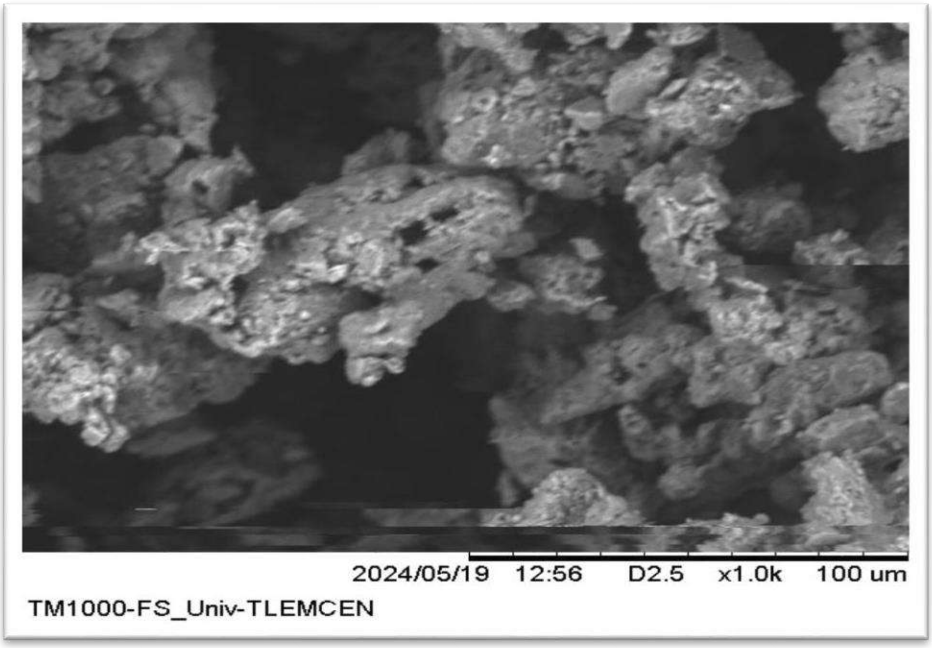


Figure 4.7 Observation at 1.0k magnification (raw)

At a magnification of 250x, the raw bentonite exhibits an irregular and porous structure characterized by a diverse range of particle sizes. The particles are loosely arranged, leaving visible voids and interparticle spaces throughout. The surface texture appears rough, featuring uneven edges and loosely bound aggregates. A broad distribution of particle sizes is observed,with larger aggregates surrounded by finer particles.

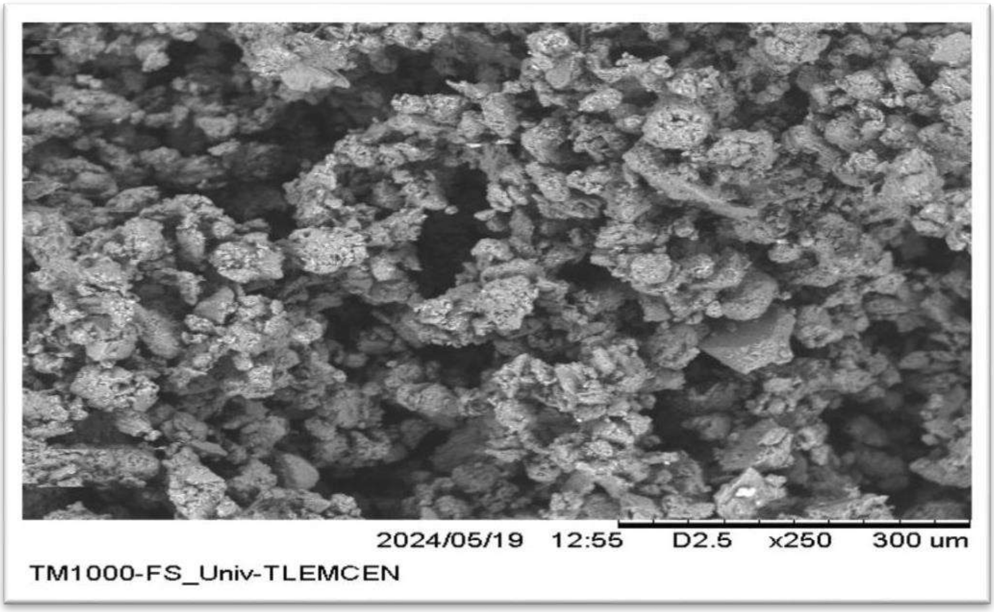


Figure 4.8 Observation at 250 magnification (raw)

2.3. Discussion:

The comparison of SEM images between raw and industrial bentonite elucidates significant disparities in their microstructural characteristics, which in turn have profound implications fortheir applications across various industries.

2.3.1. Homogeneity and Compaction:

Industrial bentonite demonstrates superior homogeneity and compaction compared to its raw counterpart. The industrial treatment processes, including grinding and extrusion, contribute tothe refinement of particle morphology, resulting in well-defined particles. These processes optimize the clay's physical properties, such as adsorption capacity and chemical reactivity, rendering it more suitable for specialized applications.

Conversely, raw bentonite exhibits a heterogeneous and less compacted structure, reflecting minimal processing. While this maintains the material closer to its natural state, it may result in variable performance in applications requiring consistency and predictability.

2.3.2. Particle Distribution:

Raw bentonite displays a varied distribution of particle sizes and a disordered structure, indicative of minimal mechanical treatment. This characteristic may influence its adsorption properties and efficiency in applications requiring high surface area and reactivity.

On the other hand, industrial bentonite demonstrates a more uniform particle distribution and a denser packing of particles, attributed to extensive processing steps such as grinding, sieving, and chemical treatments. These processes enhance the material's functional properties, including porosity and specific surface area, making it well-suited for applications such as liquid purification and drilling mud manufacturing.

2.3.3. Surface Characteristics:

The surface texture of raw bentonite is rough and uneven, contributing to its higher natural adsorption capacity. This property makes raw bentonite suitable for applications where high surface area and reactivity are advantageous, such as soil conditioning and drilling mud formulations.

In contrast, industrial bentonite exhibits a smoother and more uniform surface, indicating reduced porosity and possibly lower surface reactivity. However, this surface characteristic maybe tailored to meet the requirements of specific industrial applications, such as in pharmaceuticals, cosmetics, or as a binder in foundry sands, where consistency and predictability are paramount.

2.3.4. Implications for Applications:

The structural disparities between raw and industrial bentonite have significant implications fortheir respective applications. Raw bentonite's high porosity and irregular structure render it versatile for general use, particularly in applications where the preservation of its native characteristics is desirable.

On the other hand, industrial bentonite's controlled particle size distribution and compact structure make it ideal for specialized industrial applications that demand consistency and predictability. These include areas such as pharmaceuticals, cosmetics, and precision manufacturing processes where stringent quality standards are imperative.

In conclusion, the SEM analysis of raw and industrial bentonite underscores the profound influence of processing on their microstructural characteristics and functional properties. Whileraw bentonite maintains its natural heterogeneity, industrial treatment imparts uniformity and refinement, catering to specific industrial requirements. This comparative analysis highlights the importance of SEM in characterizing material properties and guiding their appropriate application across diverse industrial sectors.

3. **X-ray diffraction (XRD)**:

Mineralogical analysis through X-ray diffraction emerges as the predominant method in the field of materials science, offering precise identification of crystallized mineral phases within a sample. This technique involves meticulously irradiating a solid sample reduced to an extremely fine powder using an X-ray beam characterized by a specific wavelength (λ) and a determined angle of incidence (θ). When the material is irradiated, the X-ray beam undergoes diffraction at a specific angle θ based on the interplanar spacing d (the shortest distance between two parallel lattice planes) and the wavelength λ of the incident beam. This X-ray diffraction phenomenon is interpreted through Bragg's law, which identifies the directions where the interference of scattered rays is constructive, manifested as diffraction peaks.

Figure 4.9 The X'Pert Pro PANalytical diffractometer

3.1. Results:

SampleName	Date	Time	R_wp	Calcite	Dolimite	Quarts	Illite	Kaoliite	Chlorite	Pyrophylite	Albite	Microcline	Diaspore	Topaz
bentouite brute	21-mai-24	11:13:54	5,48	7,32	5,49	12,6	30,42	13,71	0,07	16,34	3,44	7,31	0,2	3,1
bentouite industrel	21-mai-24	11:20:28	5,957	5,74	1,38	19,22	27,18	13,28	0	15,33	6,13	8,39	0,1	3,24

Tab 4.1 XRD results

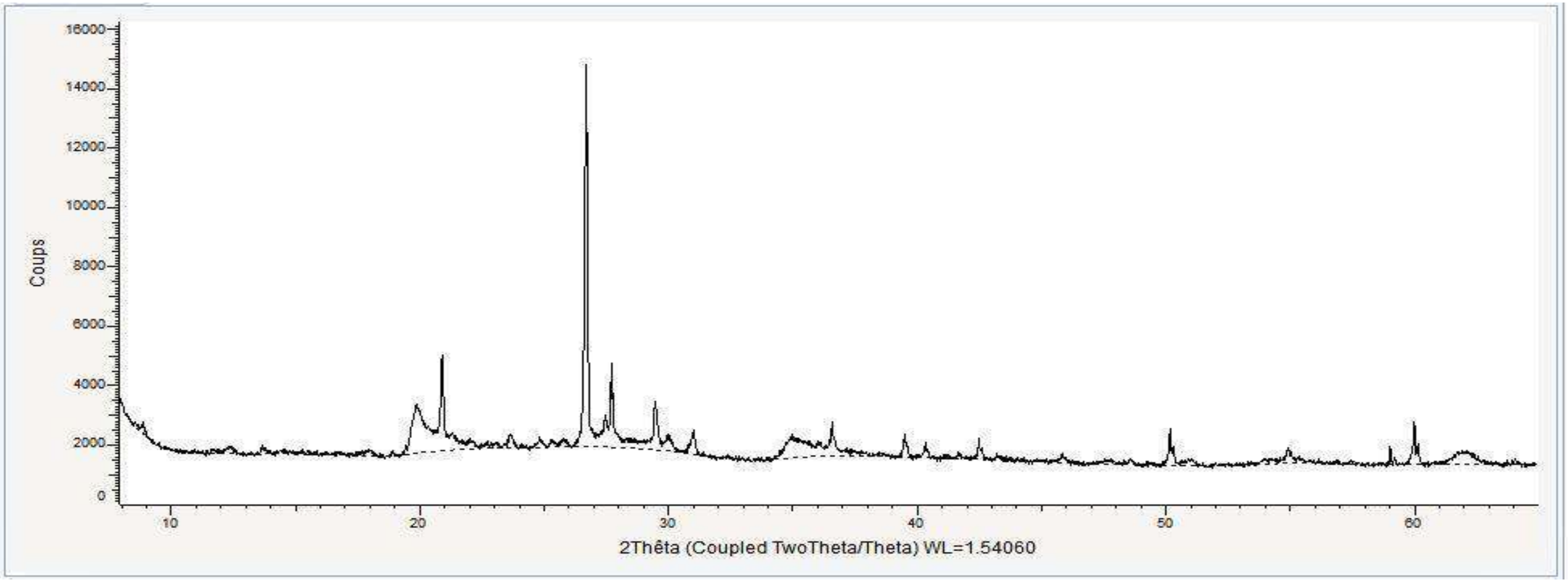


Diagram 4.1 Industrial bentonite

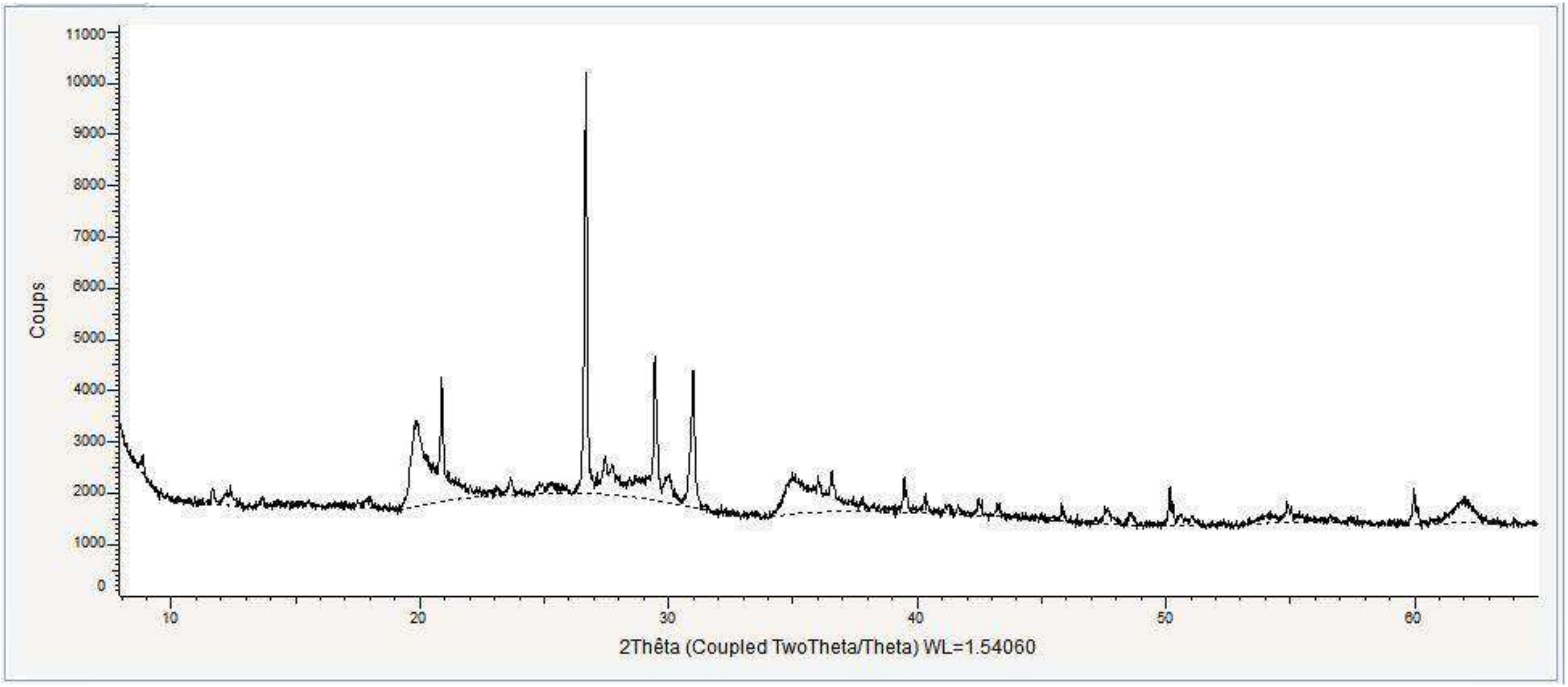


Diagram 4.2 Raw bentonite

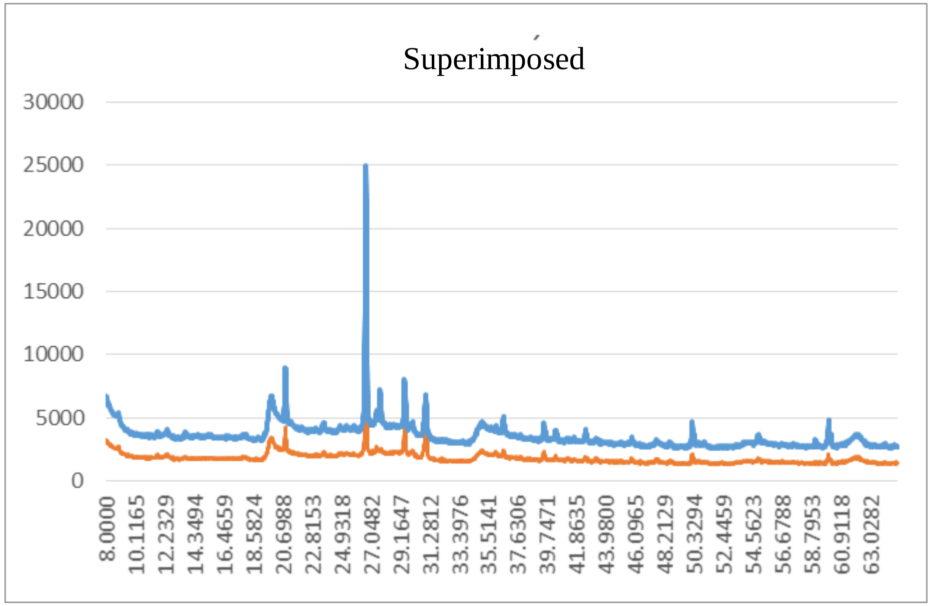


Diagram 4.3
Superimposed graph

3.2. Discussion:

DRX Bent (Raw):

The spectrum reveals the presence of mineral species characteristic of bentonite (see Diagram 4.2 Raw bentonite).

DRX Bent (Industrial):

The presence of the same species is noted, and in particular, we observe:

a) An increase in the intensity of the quartz peak (d 3.35). This increase generally indicates thermal activation of the bentonite and can be explained by the appearance ofamorphous silica.

b) A decrease in the peak corresponding to dolomite (impurity).

4. **The Thixotropy test**:

The objective of the thixotropy test, along with the procedure and materials used, has alreadybeen outlined in the previous chapter (Chapter 3). We will now proceed directly to the results.

4.1. The soils used in the test:

Soils characteristics	Raw bentonite	Industrial bentonite	Reference soil
Type of soil	Very Clayey soil	Very Clayey soil	sandy soil
Liquid limit	69 %	108 %	20 %
Example			

Tab 4.2 Soils used in the test

4.1.1. Raw bentonite:

- First test (1 minute):

✓ Hole diameter: 1.2 cm (62.5%)

Figure 4.10 Results of Thixotropy on raw bentonite (1minute)

- Second test (2 minute):

✓ Hole diameter: 1.32 cm (68.75%)

Figure 4.11 Results of Thixotropy on raw bentonite (2minute)

4.1.2. <u>Industrial bentonite :</u>

- First test (1 minute):

✓ Hole diameter: 1.49 cm (77.6%)

Figure 4.12 Results of Thixotropy on industrial bentonite (1minute)

- Second test (2 minute):

✓ Hole diameter: 1.62 cm (84.38%)

Figure 4.13 Results of Thixotropy on industrial bentonite (2minute)

4.2. Discussion of the results:

4.2.1. Raw bentonite:

The results show that the thixotropic effect of the bentonite suspension becomes more apparent with time. In the initial one-minute test, soil grains could penetrate the bentonite,(1) indicating a weak filter cake. However, in the longer second test, no soil grains were observed, suggesting a more impermeable filter cake (2) . The increase in hole diameter during the second test indicates that thixotropy requires more time to become effective. Therefore, prolonged testing durations lead to a more significant improvement in the filter cake's impermeability, highlighting the time-dependent nature of thixotropy.

4.2.2. Industrial bentonite:

In the case of industrial bentonite, it's evident from the first test that the filter cake is impermeable, as soil grains are unable to penetrate through the bentonite. The primary difference lies in the hole diameter, which enlarges in the second test compared to the first. Despite observing effective filter cake formation in the first test, the second test demonstrates an even more impermeable barrier.

4.2.3. Comparison:

A comparison between raw and industrial bentonite reveals notable differences in their performance. While both variants exhibit thixotropic behavior, industrial bentonite typically demonstrates superior qualities in barrier applications. In contrast to raw bentonite, industrial-grade bentonite often forms a more impermeable filter cake even in the initial stages of testing. This enhanced performance is attributed to the refinement and processing methods applied during industrial production, which optimize particle size distribution and increase the consistency of thixotropic properties.

Due to its refined characteristics and consistent performance, industrial bentonite is preferred for critical engineering applications such as drilling fluids, where time constraints are often crucial for project schedules and efficiency.

5. **Conclusion**:

This chapter highlighted the differences between raw bentonite and industrial bentonite in terms of morphology, mineralogical composition, and thixotropic behavior. The SEM and XRD analyses revealed that industrial modifications to bentonite enhance its structural and mineralogical properties. The study of the thixotropic phenomenon demonstrated that industrial bentonite develops its thixotropic effect more rapidly than raw bentonite. This acceleration is due to industrial treatments that optimize the characteristics of the bentonite, making the filter cake more efficient and impermeable. These findings are significant for industrial applications where thixotropy is crucial, underscoring the importance of industrial treatments in improving material performance.

General Conclusion

vi. General Conclusion

This in-depth study on bentonite has shed light on its crucial role in drilling operations, thanks to its thixotropic properties and unique characteristics. Through a series of dedicated chapters, we have explored various dimensions of bentonite, ranging from its definition and fundamental properties to its detailed characterization and specific applications in drilling.

The results of this study clearly demonstrate that industrial bentonite surpasses raw bentonite in the context of drilling applications. Tests have shown that industrial bentonite exhibits more efficient filtration properties, with a more impermeable filter, and demonstrates an accelerated thixotropic effect. These improved characteristics make industrial bentonite a more performant and reliable choice for drilling. In conclusion, the use of industrial bentonite in drilling leads to better operational efficiency and overall performance compared to raw bentonite. This study underscores the importance of selecting quality materials to optimize results in drilling applications.

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ملخص

تُستخدم البنتونيت على نطاق واسع في مختلف مجاالت الهندسة المدنية، ال سيما لخصائصها في العزل واللزوجة. تهدف هذه الرسالة إلى توصيف الخصائص الفيزيائية والكيميائية والميكانيكية للبنتونيت الخام والصناعي الهدف هو تحديد دور البنتونيت في عمليات الحفر SEMتم إجراء اختبارات تعريفية على كل من البنتونيت الخام والصناعي لتحديد خصائصها الرئيسية. وتم إجراء تحليالت حيود األشعة السينية(لفحص البنية المورفولوجية والمعدنية للتربة. وتم أيًضا)XRD المجهر اإللكتروني الماسح(و) .إجراء اختبار محدد لتقييم ظاهرة اللزوجة لكل من نوعي البنتونيت أظهرت االختبارات أن البنتونيت الصناعي أكثر فعالية في عمليات الحفر من البنتونيت الخام، مع مرشح ترشيح أكثر مقاومة للماء وتأثير لزوجة أسرع. وهذا يشير إلى أن البنتونيت الصناعي أكثر كفاءة في تطبيقات الحفر

الكلمات المفتاحية البنتونيت – توصيف – تكسية – حفر – طين – بروكتور

Abstract :

The Bentonite is a material widely used in various civil engineering fields, particularly for its sealing and thixotropic properties. This thesis aims to characterize the physical, chemical, and mechanical properties of both raw and industrial bentonite.
The Objectiveis To determine the role of bentonite in drilling operations.

Identification tests were conducted on both raw and industrial bentonite to determine their main characteristics. SEM (Scanning Electron Microscopy) and XRD (X-ray Diffraction) analyses were performed to examine the soil's morphology and mineralogical structure. A specific test was also conducted to evaluate the thixotropy phenomenon of both types of bentonite.
The tests revealed that industrial bentonite is more effective for drilling than raw bentonite,with a more impermeable filtration filter and a faster thixotropy effect. This indicates that industrial bentonite is more efficient in drilling applications.
Key words : bentonite, characterization, thixotropy, drilling, clay, proctor.

Résumé :

Les résultats de cette étude montrent clairement que la bentonite industrielle surpasse la bentonite brute dans le contexte des applications de forage. Les essais ont démontré que la bentonite industrielle possède des propriétés de filtration plus efficaces, avec un filtre plus imperméable, et présente un effet de thixotropie accéléré. Ces caractéristiques améliorées font de la bentonite industrielle un choix plus performant et fiable pour les forages. En conclusion, l'utilisation de la bentonite industrielle dans les forages se traduit par une meilleure efficacité opérationnelle et une performance globale accrue par rapport à la bentonite brute. Cette étude souligne l'importance de choisir des matériaux de qualité pour optimiser les résultats dans les applications de forage.
Mots clés : bentonite, characterization, thixotropy, drilling, clay, proctor.