SCHOOL OF SCIENCE AND ENGINEERING

RHEOLOGY OF CLAY AND CLAY AS A BUILDING MATERIAL

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RHEOLOGY OF CLAY AND CLAY HOUSING IN BENSIM

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Capstone report

Student Statement:

“I, Houda Ghailane, declare that I have applied ethics to the design process and in the selection of the final proposed design. And that, I have held the safety of the public to be paramount and have addressed this in the presented design wherever may be applicable.”

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Abstract

The main objective of this project is to study the composite used for making bricks, and to look for some alternatives to enhance a process that has been used for decades in order to improve their energy efficiency. This study will mainly depend on a rheological testing, which will allow to first understand the behaviour of this material. The purpose of the rheological study is not only to understand the behaviour of this fluid, but also to compare the three types of clay from three different sites: the one used by Bab Mansour manufactory, the one used by “ExtraBric”, and finally the one in Bensmim. The effects of addition of wood ash and olive pomace ash to clays from the three sites at various concentrations 5%, 10%, 15%, 20%, 30%, and 40% were investigated. The purpose of this work is to study the properties of the bricks and to determine a new way of recycling of the wood ash and the olive pomace ash. The major property that will be covered is the compressive strength

Key words: Rheology, compressive strength, wood ash, olive pomace ash.
1. Introduction

1.1. Background

Minimizing the environmental impact and the energy consumption is one of the biggest concerns of Morocco. The construction field is a sector that knows a huge amount of carbon emission and consumes energy with high percentages. Importing energy costs considerable expenses to the government that is why it is nowadays focusing more on renewable energies as a source in order to avoid those costs. In order to make the construction field contribute in the savings of the energy, we must think about improving the materials used in this field as well as improving their making process. The green houses, for example has been an important first step in the development of the construction field.

Clay has been used for decades in construction, especially in poor regions. As previously mentioned it is a natural material, which makes it cheap, abundantly available, and easily accessible. Its high mechanical and thermal properties made of it one the indispensable constructing materials whether fired or non-fired.

Recently, many studies have been conducted in order to find a way to recycle waste materials as well as ash, making of them new raw materials to improve the compressive strength and the thermal conductivity of the existing building materials.

1.2. Project description

Bensmim region is known for its harsh weather, and people living there are unable to afford heating. Unfortunately they suffer a lot during winter periods. The living conditions in such cases are difficult and tough, that is why it is necessary to ensure a minimum of comfort. One of the most important interventions, that can be conducted, is improving the houses by using construction materials that are characterized with high
thermal insulation and compressive strength. Bensmim region is known for the abundant availability of red clay, and unfortunately most of the houses in the region are built in more modern ways that are expensive such as cement and concrete.

The purpose of this project is to improve the properties of fired clay ricks using Bensmim clay and some additives in order to obtain a fired brick of a good compressive strength as close as possible to the one of fired bricks manufactured in the two manufactories visited for the accomplishment of this project. In addition to that, a comparison will be established between the two processes followed by the two manufactories in order to improve the one of ExtraBric which has numerous issues.

Rheology in this project is fundamental since it would help understanding the behaviour of each type of clay and find the suitable composition to obtain bricks with better mechanical properties as close as possible to the manufactured bricks’ properties, without affecting the thermal ones.

1.3. Methodology

The first section of this report consists of a literature review explaining all the concepts that will be covered by this study. The second section discusses the manufacturing process followed by the two visited manufactories located in Meknes, and a comparison between the two. The following part will focus on the rheology experiments that were conducted in the Faculty of Science of Casablanca followed by a detailed analysis of the obtained results. The last section will deal with the effect of wood ash and olive pomace ash as additives and a discussion about the compressive strength experiments conducted in ENSAM University in Meknes.

2. Literature Review

2.1. Clay

Clays are natural minerals issued from the decomposition of rocks, and are
considered as heterogeneous materials at various scales. Macroscopically, clay are associated to others minerals such as quartz, feldspath, and others. At the microscopic scale, the structure of clays is represented as sheets. Clay minerals are simply mixtures of hydrated phyllosilicates. In others words, clays are hydrated phyllosilicates which structure is represented as sheets [9].

Clays are found in huge amounts in soils since they are of a huge importance for its stability. They are classified into two main subclasses: the ones that are said to be “active” called the swelling clays, and that are characterized by their ability to absorb large quantities of water in order to form gels that are stable, and the “passive” ones, called non-swelling clays that are characterized by causing landslides [4].

2.2. Mineralogy and crystallo-chemistry of clays

Clay minerals consist of particles of very small sizes with a diameter less than 2µm, and a structure composed of two main units: tetrahedral layers when the sheets are corner-linked and octahedral layers when it is about an edge sharing sheet.

![Figure 2.2.1: Tetrahedral sheet (a) and Octahedral sheet (b)](image)

The different types of clays are determined according to the arrangement of these layers. The structural organization of phyllosilicates is based on a frame of O²⁻ ions and
Those anions occupy the tops of the octahedral arrangement, and the cations ($\text{Si}^{4+}$, $\text{Al}^{3+}$, $\text{Fe}^{3+}$, $\text{Fe}^{2+}$, $\text{Mg}^{2+}$) in the cavities in tetrahedral or octahedral positions. This atomic arrangement determines the platy morphology of these minerals and depending on the number of the layers formed, the thickness of the sheet is determined, and the space between two parallel sheets is called the interlayers space [10].

When two cavities over three of the octahedral layer are occupied by $\text{Al}^{3+}$, or any other trivalent metallic ion, the structure is called dioctahedral. However, when all the octahedral cavities are occupied by divalent metallic ions, the structure is called trioctahedral. Though, in case of a tetrahedral isomorphic substitutions ($\text{Si}^{4+}\rightarrow\text{Al}^{3+}$, $\text{Fe}^{3+}$), and/or octahedral ($\text{Al}^{3+}\rightarrow\text{Mg}^{2+}$, $\text{Fe}^{2+}$, or $\text{Mg}^{2+}\rightarrow\text{Li}^{+}$), there is a charge deficiency that is compensated outside the layer by a compensator cation [10].

**a. Tetrahedral sheet**

This type of sheet consists of a tetrahedral arrangement of $\text{SiO}_4$, where three out of four divalent oxygen anions $\text{O}^{2-}$ are shared between the three neighbouring tetrahedrons. The shared anions are called basal oxygens and are located in the same plane, whereas the unshared ones are called apical oxygens. Only one $\text{O}^{2-}$ is shared between two adjacent tetrahedrons, however the fourth ion can bond to any polyhedron. Basal oxygens donate a (-1) charge to the central ion $\text{Si}^{4+}$, which leads to an addition of a hydron $\text{H}^+$ - the compensator cation- to the unshared oxygens in order to form hydroxyls which should lead to a neutral tetrahedral sheet [11].
b. Octahedral sheet

This type of sheets is an assembly of many octahedrons edges-sharing, in which the neighbouring ones share two oxygens with each other. In this case, the octahedral sites are filled by trivalent positively charged ions, usually Al$^{3+}$, and the hydron H$^+$ is linked to each O$^{2-}$ in order to ensure the charge balance. Unlike the tetrahedral sheets, OH$^-$ donates to each cation a charge of (-1/2) since two octahedrons share one same OH. Thus, each central ion Al$^{3+}$ is enclosed by three negative charges, which lead to electrical neutrality. When there are trivalent cations in the octahedral sites, there is a possibility to fill in two out of three octahedral sites. This dioctahedral structure is said to be the most common one in clay minerals. However, the trioctahedral structure occurs when the sites are occupied by divalent cations such as Mg$^{2+}$, and each remaining site is occupied in order to create a neutrality of the sheet [11].

**Figure 2.2.2:** sphere-packing model of a tetrahedral sheet (a) and its polyhedral model

(a) Octahedral sheet
(b) Tetrahedral sheet
Figure 2.2.3: sphere-packing model of an octahedral sheet (a) and its polyhedral model

2.3. Classification of clays

Various phyllosilicates classifications have been established depending on the norms of classification followed. A first type is based on the sheet charge and the number of metallic ions in the octahedral layer, and another one based on the location of the substitutes, their distribution, and the type of the compensator cations. The basic classification however is based on the thickness as well as the structure of the sheet [10].

a. 1:1 layer sheet

Also called T:O structure, with a thickness of 7Å [10] this type consists of the superposition of one tetrahedral and one octahedral sheet, sharing the apical oxygen of the tetrahedral sheet with the octahedral one. In this case, there is a first plane that is composed of the basal oxygens of the tetrahedral sheet, a second plane composed of shared $O^2-$ anions between the two types of sheets and the $OH^-$ from the octahedral sheet, and a last plane composed of $OH^-$ anions of the octahedral sheet [11].
b. 2:1 layer sheet

The T:O:T sheet is composed of two tetrahedral sheets and one octahedral in between, and is characterized by a thickness of 10 Å [10]. Unlike the 1:1 layer sheets, this type consists of four planes of negatively charged ions. The external ones composed of the tetrahedral sheet’s basal oxygens, and two other planes composed of shared oxygens between the octahedral and the two tetrahedral sheets in addition to the octahedral sheet’s hydroxyls [11].

The 2:1 layer sheet consists of two types holding the same structure and different thickness as a result of interlayer space. The second 2:1 layer sheet has a thickness of 14 Å [10]. An extension of this type will be the 2:1:1 layer sheet which consists on an addition of another octahedral sheet either an (Al-OH) sheet or an (Mg-OH) sheet.
2.4. Types of clay

a. Kaolinite

Kaolinite is a type of clay characterized by a structure composed of 1:1 layers piled together, where aluminium cations (Al$^{3+}$) occupy the octahedral sites and silicons (Si$^{4+}$) in the tetrahedral ones. The sheet of kaolinite is neutral, and the neighbouring layers are linked together through hydrogen bonding, between hydroxyls belonging to the outer plane of the neighbouring octahedral sheet and the basal oxygens of the tetrahedral sheet. Kaolinite is said to be the most common soil mineral and the most common one belonging to the 1:1 layer sheet classification. It is very rare that an isomorphous substitution occurs in kaolinites, neither in tetrahedral nor in octahedral sheets, and the ideal formula of kaolinites is Al$_2$Si$_2$O$_5$(OH)$_4$ [11].

![Figure 2.4.1: Crystallographic structure of kaolinite](image)

b. Illites

The formula of this type of clays is (K, H$_3$O)(Al, Mg, Fe)$_2$(Si, Al)$_3$O$_{10}$[(OH)$_2$,(H$_2$O)] [10], and its structure consists of a 2:1 layer sheet, and is composed of one alumina sheet (octahedral) and two silica sheets (tetrahedral). However, substitutions may occur and Si might be replaced by Al [9]. The sheets are negatively charged, a charge higher than the one of smectites [10]. Thus, potassium
cations are adsorbed in the interlayer space in order to compensate the charge imbalance. This type is said to be the closest to the muscovite which consists of more water and less potassium cations [9], and the difference between illites and smectites exist in the fact that the compensator ions K$^+$ are weakly exchangeable; in other words, illites have a weak cationic exchange [10].

![Figure 2.4.2: Crystallographic structure of illites](image)

c. Smectites

This is another 2:1 layer sheet type where the sheets are stacked in a disordered way. Each sheet is rotated in its plane compared to the previous one. In this case, the atoms substitutions are important. Both the disorder in the arrangement and the weak charge of the sheets makes their spreading and the adsorption of various molecules such as organic molecules, cations, and water easier at the level of the interlayer space that is spreading as well[9]. Unlike illites, compensator cations occupy the interlayer space in order to solve the charge deficiency problem, which implies the high cationic exchange of this type of clays [10]. The most common smectite is called montmorillonite [9], and the ideal formula is (OH)$_4$Si$_8$(Al$_{10}$/3, Mg$_{2}$/3)O$_{20}$,nH$_2$O.
d. Chlorites

Chlorites are phyllosilicates abundantly found as crystals in the magmatic rocks and that have 2:1:1 layer structure [10] with an surplus of negative charge [11], and whose formula is \((\text{Mg, Al, Fe})_6 [(\text{Si, Al})_4 \text{O}_{10}] \text{(OH)}_8 [10]\). The interlayer space in this type is occupied by an \((\text{Mg-OH})\) layer, and the aluminium is locally replaced by Fe [9]. Instead of potassium cations, the negative charge excess is compensated by an interlayer sheet of hydroxides that has a positive charge. This interlayer sheet being can be either a dioctahedral or trioctahedral one [11].
e. Vermiculites

This type of phyllosilicates consists of a 2:1 layer structure and is mainly trioctahedrals. Its formula is \((\text{Mg, Ca})_{0.7}(\text{Mg, Fe, Al})_6(\text{Al, Si})_8\text{O}_{22}(\text{OH})4.8\text{H}_2\text{O}\) [10], and it is characterized by its hydrated exchangeable cations [11]. Vermiculites is close to smectites, however their sheets are characterized by a higher charge deficiency that is caused by tetrahedral substitutions, and the charge balance is ensured in the interlayer spaced by cations, mostly \(\text{Mg}^{2+}\), and water layers [10].

![Figure 2.4.5: Crystallographic structure of Vermiculites](image)

f. Montmorillonites

This type is also a 2:1 layer sheet phyllosillicate belonging to the smectites category, and main constituent of the bentonite. Its formula is \((\text{Na, Ca})_{0.3}(\text{Al, Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2\ n\text{H}_2\text{O}\), and it is most of the time used as binder, a filter, and as an absorbent or also as adsorbents. The particles of this type of clay are formed when the layers stake. This type is considered as swelling clay, permitting to the water to go between the sheets. When dispersed in water montmorillonites become good plasticizer and thickening agent [23].
2.5. Rheology

Rheology is determined as the study of the flow and the deformation of the complex fluids. This science has two main key words that are the force and the deformation, and consists of measuring the deformation after applying a certain force required for a certain deformation, in order to understand the way the materials deform after applying a certain force [1]. The purpose of this science is to find a relation between stress and deformation in case of materials whose mechanical behaviour cannot be explained by neither the law of Newton, nor the law of Hooke [2]. The rheological experiments can be divided into two main categories depending on the nature of the deformation applied, whether it was a large or a small deformation. In case of small deformations, the measurements are done before the material breaks. This type of measurement gives us the necessary information about the structure of the suspension, and about the interactions between the particles. However, in case of large deformations, the measurements are done when the suspension is deformed or totally destroyed, and a good example of this case can be the viscometry measurements [3].

Two types of rheological experiments have been decided in case of small deformation measurements. First of all the transitional measurements, where we apply a shear stress during a defined period of time, then stop this application in order to
follow the relaxation and the recovery of the material. In case of applying a shear stress to the system, we call this “creep recovery”. The second type, the oscillatory measurements, also said dynamic measurements, is when the shear stress is applied at a range of frequencies [3]. The purpose of this type of measurements is to study the strength of the structure of the suspension as a function of the composition [4].

a. Laminar shear movement

A laminar shear movement is defined as a movement during which the material’s structure is laminated, or has very thin adjacent layers. The deformation of the material in this case is done by a relative sliding of the different layers without any matter transfer from a layer to another. This movement happens without any change in volume [5].

b. Shear stress

During a laminar shear movement, the layers are animated by the relative of one layer to another. This relative movement is explained by two successive layers, relatively moving with respect to each other, and as a result, friction forces appear. These forces are tangentially exerted on the surface of the layer, and are called shear forces or shear stress, noted τ (or σ), and defined by the following formula [5].

$$\tau = \frac{dF}{dS}$$

c. Shear strain and shear rate

To define the shear strain, it is preferable to consider the case of a laminar shear movement that presents plane symmetry such as the case of a material sheared between two parallel plates, a moving and a static one. For a
displacement $\Delta X$ and a distance $h$ between the parallel plates, the shear strain $\gamma$, in case of symmetry, is defined as the ratio of the displacement over the distance, and the shear rate $\dot{\gamma}$ as the derivative of the shear strain with respect to the time $t$. [5]

$$\gamma = \frac{\Delta X}{h} \quad \text{and} \quad \dot{\gamma} = \frac{d\gamma}{dt}$$

2.6. Rheological equation of the state

Every mechanical system follows a fundamental equation which aims to represent the relationship between the dynamic quantities which are responsible of the movement, and the kinematic ones which describe it. Also called constitutive equation, the equation of the state in rheology is the one that relates the shear strain to the shear stress, and that depends on both the properties and the composition of the material. Thus, the purpose of rheology is determining experimentally this relationship (the following function) using a Rheometer [5].

$$\gamma = f(\tau)$$

2.7. Viscosity

Viscosity is a property that characterizes the resistance of a material to a continuous deformation [1], the ability to resist flow, and reveals the energy of deformation dissipated [2]. After applying a certain force, the material response to this deformation will be either by showing an elastic or a viscous behaviour, or more frequently, a combination of both, which is called viscoelasticity [2]. Unlike in the case of elasticity, for the viscometry the shear stress is not related to the shear strain, but to the shear rate. It is thus a property of flowing materials, not solids. The simplest behaviour in rheology is summarized in a linear function relating the shear stress and the shear rate [1]. Most of the time, determining the viscosity, noted $\eta$, is enough to enable us to determine the precise characterization of the rheological behaviour of the
material [5].

\[ \eta = \frac{\tau}{\dot{\gamma}} \]

2.8. Newtonian fluids

For some substances, the viscosity \( \eta \) is independent from shear stress, and it implies proportionality between the shear stress and the shear rate. In this case, we call these fluids Newton-Stokes liquids or Newtonian fluids; since they follow Newton’s hypotheses [6]. The experimental study of the rheological behaviour of the flows is done using rheometers, which enables the measurement of the stress and the shear rate. Depending on the type of the rheometer used, the measurements will consist of applying a shear rate to obtain the shear stress; or the opposite determining \( \dot{\gamma} \) after applying a stress. The rheograms obtained in each case are either \( \tau(\dot{\gamma}) \), or \( \dot{\gamma}(\tau) \). If the obtained curve is a straight line that starts at \( \dot{\gamma} = 0 \) (or \( \tau = 0 \)), its slope represents the viscosity called absolute viscosity, defined as previously [5].

\[ \eta = \frac{\tau}{\dot{\gamma}} \]

Figure 2.8.1: Flow curve of Newton Liquid
2.9. Non-Newtonian fluids

In case of a non-linear flow curve, and that does not pass by the origin, we are facing a viscosity that varies with time, and that depends on the conditions of the flow such as geometry, shear stress, and rate; we then talk about non-Newtonian liquids, and there are many types of them. For such fluids, we don’t talk about absolute viscosity, but about apparent viscosity defined as follows at $\tau = \tau_0$ [7]

$$\eta = \left[ \frac{\tau}{\dot{\gamma}} \right]$$

a. Bingham liquids

Bingham fluids are classified as viscoplastic fluids; which means that they are known by the presence of a yield stress which needs to be exceeded in order to make the deformation (or the flow) possible. A fluid characterized by a linear flow curve for $\tau > \tau_0$ is called Bingham plastic, and has a plastic viscosity [7]. The rheogram of such fluid is a straight line that does not pass through the origin. Once the yield exceeded Bingham liquid acts similarly as a Newtonian liquid, which means that with the rise of $\dot{\gamma}$, there is a proportional increase of $\tau$. Since the fluid is characterized by its plastic viscosity $U$, the rheological equation of the state in this case becomes [5]:

$$\tau = \tau_0 + U\dot{\gamma}$$
Figure 2.9.1: Flow curve of Bingham liquid

b. Casson fluid

Casson fluids, or Hershel-Bulkley model are also part of the viscoplastic liquids. Same as for Bingham model, Casson model is also characterized by the presence of a yield stress, however does not have a Newtonian behaviour once above the yield stress. For this reason, we say has it behaves as an non-ideal plastic, and is also characterized by its plastic viscosity $\beta$. Similar to Bingham liquids, the apparent viscosity is negatively related to the shear stress and rate, which means that an increase in those quantities leads to a decrease in the apparent viscosity [5]. The rheological equation of state for a Casson fluid is the following:

$$\sqrt{\tau} = \sqrt{\tau_0} + \sqrt{\beta \dot{\gamma}}$$

Figure 2.9.2: Flow curve of Casson liquid
c. Shear thinning

Also called shear softening and pseudoplasticity this type of fluids do not have any yield stress and the apparent viscosity depends on the shear stress or rate since it decreases when the shear increases [5]. The shear thinning is said to be the most common non-Newtonian fluid that is time-independent. However, at very high or very low shears we notice a Newtonian behaviour for shear thinning suspensions [7]. The shear thinning rheograms show that for very low shear stresses (or rates), the viscosity is constant, and same for very high shear stresses (or rates) but at lower levels [8]. Unfortunately this type of fluids does not obey to any equation of the state that represents exactly the thinning behaviour; this is why the power law was elaborated by Ostwalden, which explains the second name of power law: Ostwalden law [5].

\[ \tau = K\gamma^n \]

The coefficient is to be determined, as well as the power n, which is always smaller than 1, either directly by the rheology software used, or by evaluating the rheogram in log-log coordinates. Using the logarithmic rheogram, we can directly determine the K coefficient which is the origin of the curve, and the power n represented by the slope of the curve. In this case the power law becomes [5]:

\[ \log\tau = \log K + n \log \dot{\gamma} \]

Figure 2.9.3: Flow curve of a shear thinning fluid
d. Shear thickening

Similar to shear thinning behaviour, the shear thickening, also said dilatant, do not show any yield stress, however, unlike the pseudoplastic fluid, the apparent viscosity and the shear rate (or stress) are positively related. This behaviour can be explained as follow: when the material is at rest, there is a minimum of voidage and the liquid is enough to fill in the voids; however at low shear rate, the motion of the particles is lubricated, thus we obtain a small shear stress. But for large shear rates, there is a slight dilatation of the material which causes a need in liquid to fill in the increased voids, thus the friction increases and the resulting shear stress is higher. This process causes the rise of the apparent viscosity with the rise of the shear rate (or stress) [7]. This kind of liquids is generally known for their low viscosity and their ability to solidify by agitation, and the equation followed is also the power law, however with an exponent $n$ higher than 1, and which gets higher when the behaviour of the fluids gets distant from the Newtonian behaviour [5].
e. **Thixotropy and rheopexy**

This other non-Newtonian fluid is said to be a time-dependent one. Thixotropic behaviour happens if after shearing the material at a constant rate, its shear stress, or apparent viscosity falls with the shearing time [7]. When the shearing stops, the particles tend to respond to the inter-particle forces, which requires quite a long time since the purpose is to reform completely the equilibrium of the initial structure of the material; thus, a decrease in viscosity occurs. When a thixotropy is occurring, it means that the applied shear tends to break the initial structure of the unsheared material [1].

Rheopexy, also said antithixotropy or negative thixotropy, happens when an
increase in viscosity with shearing time occurs. Unlike thixotropic material, in rheopexy the shearing aims to build-up the structure which breaks when at rest. However, to make the build-up of the structure, the shearing motion should be gentle since a strongest one will lead to a breakdown. [7]

It is not exceptional that for the same material, both rheopexy and thixotropy behaviours are displayed depending on the material concentration and/or the shear rate [7].

Figure 2.9.7: Flow curve of a Thixotropic and a Rheoplectic fluid

2.10. Rheometry

Rheometry refers to the experimental procedures and techniques used in determining the rheological properties of a fluid. The purpose of Rheometry here is to determine the relationship between stresses and deformations. The instrument used in these experiments is called a rheometer. Its purpose is to apply a shear stress to the fluid in order to measure its viscoelastic properties; or in more simple words, it relates the property of the fluid from how hard it is being pushed to how fast it is moving. There are two main categories of rheometers, the rotational and the extensional ones depending on the type of experiment to be conducted. If the purpose is to control the stress (or the strain), the rotational rheometer is the most suitable choice; however, if the main goal is
to apply an elongational stress, also said extensional stress (or strain), the best choice will be the extensional rheometer. In this study, the principal tool will be the rotational rheometer.

In a rotational rheometer, there are two parallel plates, one imposing the deformation which is the upper one, and second one fix. Such rheometers allow the measurement of a good range of torque, has a quick response, and most importantly it controls the stress (or strain) that will be applied. In this category of rheometers, we distinguish three main types: the plate-plate, the cone plate, and the concentric cylinders [17].

a. Parallel plates rheometer

This type of rheometers consists of two parallel flat plates, one stationary and the other rotating, which implies a laminar flow of the layers. In case of coating process, the upper plate becomes fix. In this case, the coating material is exposed to small gaps laminar flow. This type of rheometers in case of low, medium, or high viscosity liquids, for soft solids or gels, in case of samples with large particles. Both the shear stress and rate depend on the gap between the plates and the plate’s diameter as well. The shea stress and the plate’s diameter are negatively related, which means that a bigger diameter decreases the shear stress. However, the shear rate is negatively related to the gap between the two parallel plates [18].

![Representation of a plate-plate](image)

**Figure 2.10.1:** Representation of a plate-plate
b. Cone-plate rheometer

Unlike the previous type, this type of rheometers is known for the use of a cone with a known instead of an upper flat plate. The rotation of the cone creates a certain velocity which is linear at a very small cone angle. A small cone angle implies that there will be a homogeneous stress. Usually the gap is set by the manufacturer at a specific value; a value corresponding to the truncation of the cone. This type is the most common one used nowadays in order to study non-Newtonian fluids since it performs measurements of high shear rate [17]. Same as for the plate-plate rheometer shear stress increases as the diameter decreases, as well as for the shear rate which increases when the angle of the cone decreases [19].

![Cone-plate rheometer diagram](image)

**Figure 2.10.2:** Representation of a cone-plate

c. Concentric cylinder rheometer

Also called rotational cylinder rheometer, this type is used in case of liquids with low or medium, and never used for solids. It is characterized by its large surface for the contact between the instrument and the sample to study, which increases the levels of the toque, and thus allows the measurement of very low viscosities. The way it works consists of two cylinders, an outer one called “rotor” fix, and an inner
cylinder called “the stator” rotating continuously [19]. This type of rheometers is the one used in this study.

![Figure 2.10.3: Representation of a concentric cylinder](image)

2.11. Fired clay bricks

Clay bricks are one of the oldest construction materials used for decades. Fired clay bricks are said to be the revolution of masonry. When burning at very high temperatures, clay particles begin to melt and agglomerate to form a mass characterized by its stony shape [12]. The fact that fired clay bricks are characterized by their high compressive strength compared to the unfired clay bricks as well as their durability to weathering, makes of them the most used building material for decades, and will serve as a basis for recent construction materials [24]. This section will mainly focus on the properties of fired clay brick.

**a. Durability**

This property depends on whether the brick reaches the vitrification and fusion during the firing process. The major characteristics of fired ricks are directly related to the temperatures of firing, from the compressive strength to the water absorption. In addition to these two properties, the saturation coefficient is also considered as an indicator to predict the durability of the brick. However, since the used raw material
and the manufacturing process differ from a manufactory to another, these factors cannot be reliable in the determination of the firing temperature [24].

b. Color

Once again, this property depends on the nature of the raw material. It is the chemical composition, as well as the raw material minerals that determine the color of the brick once fired. The temperature of firing also plays an important role in determining the color of the product, and the oxide that mainly affects the color is the iron. All clays that contain iron will exhibit a red color once it oxidizes to form ferrous oxide. However, clays that contain a larger amount of lime will exhibit a dark red, and a brown color. In case of firing at low temperature, the clay will adopt a dark, or even black shade. When an identical raw material is fired at high temperature, the product is characterized by its red color, its high compressive strength, and its low ability to absorb water [24].

c. Compressive strength and water absorption

The compressive strength property, as well as the absorption is directly related to the raw material properties as well as to the manufacturing process, and the firing temperature [24]. The mineral composition and the porosity level are two main indicators of the compressive strength. This property is also related to the firing and drying process. In case of a good and controlled drying process, cracks will be avoided during the firing stage. Moreover, the temperature of firing strongly contributes in increasing the compressive strength. When the temperature is below 650°C, the remaining water is removed. And from 650°C to 850°C the chemical reactions start, and finally the vitrification occurs when the temperature varies from 950°C to 1200°C or higher depending on raw material [25]. For any type of clay and
any manufacturing process, to obtain a product with a high compressive strength and a lower ability to absorb water, high temperatures during the firing are required [24].

3. STEEPLE Analysis

STEEPLE analysis is considered as an innovative tool compared to the SWOT analysis since it approaches the macro-environmental factors. This analysis is intended to evaluate the different aspects and influences at that level. STEEPLE is an acronym for Socio-cultural, Technological, Environmental, Economic, Political, Legal, and Ethical. This section of the report will cover the STEEPLE analysis of the project.

3.1. Socio-cultural Factors

The socio-cultural implications include demographic characteristics, and consist of the social factors that are directly related to the revenue distribution. Ifrane surrounding rural areas, such as Bensmim, are known for their severe weather conditions. People from those regions still use clay blocks in their building, which is not providing them with the comfort they are expecting in their houses. Those two reasons determine the social implications of this research project. The most important aspect, the heating problem, will remain from the past thanks to the high thermal properties of clay, thus the lifestyle and conditions of those needy people will improve. Using clay composites with a high energy efficiency in terms of mechanical and thermal properties will decrease from the possibility of being exposed to health problems, and will increase from the population comfort.

3.2. Technological Factors

The purpose of the technological factors is mainly to reduce from the existing barriers. In this study, using the Rheometer is essential to understand the behaviour of clay used by ExtraBric, as well as Bab Mansour manufactory in manufacturing the
bricks, and compare them with the clay of Bensmim region. Besides, the X-rays will be used in order to determine each clay composition. The technological implications consist essentially on new creations and development. This project will cover the development of the bricks making process, the bricks mechanical and thermal properties, and the clay housing.

3.3. Environmental Factors

The environmental aspects evaluate the nature of the impact, whether it was positive or a negative, that the project may have. Those factors are most of the time enforced rules that has to be followed in order not to damage the environment. The purpose behind the rheological study is to understand the behaviour of the clay and come up with adequate results that will reduce from the waste of water used by the manufactories. Moreover, encouraging the use of clay bricks in construction will empower the development of ecological houses through the use of natural compounds, which will lower the carbon emission due to the manufacturing and transportation, and thus decreasing from the energy consumption.

3.4. Economic Factors

The economic factors affect directly the economic growth, the interest rate, the inflation rate, the exchange rate, the savings, and the customer potential power. Clay is generously found in nature and is very cheap as well; which is considered as an important economic benefit. Thus, using a natural and local material will enable an increase in the economic growth since the manufactories can easily obtain the raw material just by being located to an extraction site that will last for long, which will cost less for both the manufacturer and the customer. Another economic advantage in this case will be the maintenance costs that are very low. As mentioned in the previous point, this project has important environmental implications especially the decrease of the
energy consumption, which will lead to a decrease in the heating cost, as well as to a cost and construction reserves.

3.5. Political Factors

The political aspects embrace government guidelines, legal concerns, and political stability. Nowadays, the Moroccan government is encouraging the population to save energy, as well as the sustainable development. For these reasons, the Moroccan government is empowering projects that are looking for innovative ways to reduce from the energy consumption in the houses. Once again, the objective of this project is to encourage the use of a low cost natural material that has very low carbon emission, which implies the improvement of the environmental aspects, and thus we can say that this project conforms to the Moroccan political task.

3.6. Legal Factors

Legal inferences of this research project are represented by the impacts it may have in terms of employment laws, taxes regulations, and safety strategies. The main goal is to guarantee that all the country laws and regulations will be respected. This project does not disturb any of the safety strategies nor the law. However, it strengthens the energy efficiency.

3.7. Ethical Factors

Ethical aspects are anything that is related to social morals and values governing the person’s behaviour. These are seen as a basis to know what are the right and the wrong conduct. In this case, the ethics factors are summarized in the engineer’s code of ethics. From this code, six major norms should be followed by the engineers while performing their duties. First they must maintain the safety. Second, the services must be performed only in the domain of competences. Moreover, the issue public statements should be just
in a precise and straight manner. Also, the engineer must act as a faithful and trustee agent. Besides, engineers must avoid deceiving and dishonest acts. Finally, an ethical engineer is the one who behaves ethically, lawfully, responsibly, and honourably in order to develop the usefulness of the work, the reputation, and the honour. This project do not oppose theses fundamentals, thus the ethical factors are covered.

4. Part I: Manufactured bricks

In order to well understand the bricks making process, I have visited two different manufactories in Meknes. The first one was “Bab Mansour”, a manufactory using two different ways of making, a traditional process, and a modern process with the new techniques and machines. The second manufactory “ExtraBric” is the one that has been covered by a previous capstone student, Yosra El Boulli Rguibi, who worked on the eco-friendly bricks, foam bricks to be more precise. The reason behind this choice is to compare between the two processes in order to define the problem to work on and try to find solutions for them. The problem of “ExtraBric” is remained the same. The machines they are working with are too old compared to the ones used in “Bab Mansour”, which means that they still rely on the employee expertise to know if they are in the right path, for example in determining the amount of water. This part of the study will mainly focus on finding solutions to “ExtraBric” manufactory in order to answer to the needs and the issues that the engineer in charge stated.

4.1. Description of the mixture

The clay used by each manufactory has been sent to the Faculty of Science of Rabat-Agdal for the X-rays characterization in order to determine its chemical composition, and thus be able to know what compounds will be formed during the firing process. Unfortunately, the results are still not available.
Based on what the technician in charged said, the clay they are using is characterized by a high plasticity, which leads to a difficulty of removing the water added to facilitate the shaping and the molding of the mixture and can cause a severe shrinkage leading to a cracking during the drying process, making the addition of sand necessary. After been dried and fired, this characteristic is lost and the product obtained is distinguished as a brittle and hard material.

The composition clay-sand differs for the two manufactories, as well as the method of to determine the amount of sand to be added. For “Bab Mansour” manufactory, the mixture is composed of 80% of clay and 20% of sand, and the technique used to determine this amount of sand is simple. This technique consists of modelling the mixture clay-sand using hands, starting with an arbitrary percentage of sand until obtaining a shape of a rope with a diameter varying between 5cm and 6cm, and a length of 20cm. If the obtained dimensions are higher than the suggested ones, this means that more sand needs to be added. However, for “ExtraBric”, the mixture is composed of 70% of clay and 30% of sand. The technique used by this manufactory consists of making small blocks of clay-sand mixed with water, with a horizontal arrow in each side. Once molded, the block is subjected to a controlled drying process, and the length of the arrows is then measured. The amount of sand to be added depends on the shrinking percentage of the arrow that should not be less than 5.5% and should not exceed 6%. In case of a shrinkage percentage lower than 5.5%, this means that the quantity of sand added need to be lowered. However, in case of a percentage higher than 6%, more sand needs to be added to the clay for a perfect composition.
Figure 4.1.1: Blocks obtained during to determine the composition of the mixture following the method used by “ExtraBric”

4.2. Bricks making process

Bricks have been considered as the major construction materials since decades, and their making process has not known any evolution. The process steps followed are the same for all the manufactories which implies that the difference between manufactories will depend on the raw material used as well as the machines. In order to improve the material sustainability, new technologies has been developed and new studies has been conducted.

The purpose of this part of the project is mainly to compare between two different manufactories in Meknes, “ExtraBric” and “Bab Mansour” that follow the same process, but have different machines and a different source extraction.
a. Extraction of raw material

Both “ExtraBric” and “Bab Mansour” were extruding the clay they are using from a field nearby the manufactories for years. However, because of the houses which were built in that same area, they were not allowed to extract from there anymore, and thus started extracting from a new field less closer than the first one. Unlike “Bab Mansour” manufactory, “ExtraBric” does not have a permanent source from where they are bringing their sand which sometimes can be disadvantageous.
As soon as the clay is extracted, it is left in an open air area in order to first acclimatize, second to dry if it contains any water, and finally to decrease from the soluble salts, which will lead to a more homogeneous raw material.

![Figure 4.2.2: Extraction of raw material “Bab Mansour” manufactory](image)

**Figure 4.2.2:** Extraction of raw material “Bab Mansour” manufactory

![Figure 4.2.3: Open air area in “ExtraBric” manufactory where the extracted clay is left](image)

**Figure 4.2.3:** Open air area in “ExtraBric” manufactory where the extracted clay is left

**b. Preparation of the mixture**

This step of the process consists of the passage of the clay through different stages. The extracted clay goes through the crusher since the raw material is initially in a form of very big stones. The purpose here is to reduce the size and obtain a large size grains which are then subjected to a new type of crushing. This last consists of two medium cylinders rotating in two opposite senses in order to reduce the grain size to a range of 2cm to 3cm. The last step is a rolling mill which consists of two big cylinders that are also rotating oppositely, leading to thin clay layers of 1mm to 2mm thickness. The passage from a step to another is done through inclined vibrating screens that serve as a sieve in order to reduce more the particles size before reaching the
following phase. Each step knows an addition of a very small amount of water in order to facilitate the process.

Figure 4.2.5: Cylinders of the crusher that reduces the grain size

The second stage consists of the passage through a first mixing chamber along with the addition of another small amount of water, as well as the determined amount of sand. This first mixing chamber is specific for the mixture preparation, and the clay stays there for a certain period of time before the following step. The longer it stays, the better the quality is.

Figure 4.2.6: First mixing chamber
c. **Brick formation process**

Once it leaves the first preparation mixing chamber, the tempering step occurs. This stage consists of adding the necessary amount of water to obtain a viscoelastic mixture easy to shape. This step occurs in the second mixing chamber, also called plug mill, using shafts and blades. This step is followed by the molding, and the type of the molding machines used by the two manufactories is called the plastic clay molding machines or also extrusion columns. These machines have a rectangular opening from where the tempered clay comes out rectangular strips that are later cut using wires called wire cut bricks in order to obtain the required size. Between the plug mill and the extrusion columns, more water may be added if necessary. Within these extrusion columns, there is a chamber in which the process of removing air occurs. It consists of removing all the bubbles and holes in order to obtain a material with a better plasticity, and also in order to avoid cracks during the drying and firing processes.

![Figure 4.2.7: Inclined vibrating screen that serves as a sieve](image)

**Figure 4.2.7:** Inclined vibrating screen that serves as a sieve

**Figure 4.2.8:** Extrusion columns corresponding to the molding
d. Drying process

Unlike the traditional drying process that needs about 20 days to be completed, the new process used in the manufactories needs only 24 hours for a complete drying. After being molded and cut, the bricks are placed in the drying chamber in order to remove all the amount of moisture left to avoid the cracking during the firing process. This drying chamber is divided into sections of different temperature. To avoid cracking during the drying process, the drying temperature should increase progressively, which explains the sections of the drying chamber.

e. Firing

This last step of the manufacturing process consists of using tunnel kilns. After being dried, the bricks are transported wagons that enter the oven one after the other with a 30 min difference between each one, and going out with the same time difference. The fact that there are 38 wagon inside the kiln in “ExtraBric”, this means that the firing process lasts for 38 hours, and 30 hours for “Bab Mansour” with 27 wagon. During this process, the bricks acquire strength and hardness since chemical reactions occur which lead to a vitrification. The firing temperature differs depending on the nature if the clay, but the universal range varies from 850°C and 1000°C. If the fixed temperature is under the limit, the product will not acquire enough strength, and if it goes beyond it, the result will be a brittle product easy to break. This last stage is divided into five steps in order to avoid any loss due to the possible breaking. The first stage consists of a final drying since the bricks are subjected to a very low temperature when they enter the oven. After that, the dehydration step happens and which consists of final drying. At temperatures between 120°C and 650°C, the remaining water is removed and it is until 850°C that the chemical reactions occur, leading to the following step called the high temperature firing. The temperature in
decreased gradually and the low temperature firing happens. Finally, the cooling step happens, and during this step, the temperature drops significantly to the initial temperature at which the final drying occurred.

Figure 4.2.9: The kilns oven programming

4.3. Analysis and Comparison between “Bab Mansour” and “ExtraBric”

The process followed by the two manufactories is similar when it comes to the steps followed, but differs when it comes to the machines used, which makes the process of “Bab Mansour” better than the one of “ExtraBric”. The difference in the amount of sand added is not of a big importance since this one depends on the nature of the clay used by each manufactory. Same for the difference in the firing time and temperature which can be explained by the type of clay, this cannot be a difference to discuss. The most significant and important difference is in the brick formation stage. For “ExtraBric” manufactory, the tempering step all depends on the expertise of the person in charge. This person needs only to touch the mixture to say if the amount of water added was enough or not, which may sometimes be disadvantageous. The expertise differs from a person to another, and two people in charge who touch the mixture may not agree and have the same opinion. Also, in case the amount of water added is said to be excessive, this means that the process will stop and a loss in the material and the time will occur.
However, “Bab Mansour” manufactory is not limited to the expertise of their employees. Their molding machine has a pressure detector which helps in determining the exact amount to be added. Until reaching a pressure of 22Pa, the water keeps being added. The other difference between the two manufactories is the source of firing. “Bab Mansour” manufactory uses ecological sources such as olive pomace and almond peels and recycles this energy by using as a heating for the drying process, while “ExtraBric” uses only petroleum which is badly affecting the environment.

5. Part II: Rheology

5.1. Experimental procedure

The rheology tests were conducted in the Faculty of Science of Casablanca, using the rheometer Haake Rheostress 1, and controlled with the RheoWin software. This rheometer has an automatic lift in order to guarantee a more precise placing of the computing geometries [22]. The software controlling this rheometer is called RheoWin.

The measuring geometry that has been used to perform the rheological tests is the concentric cylinders; and the clay suspensions are placed in the cell DIN 53018 Z41 which diameter is equal to 41mm. The corresponding volume for this cell is 11.9 mL and the shear rate to be applied 0.008-3900 (s⁻¹).
The rheological tests were performed for different suspension at different concentrations 20%, 30%, 50%, and 70%. The first step was fixing the rheometer by establishing the point-of-contact after fixing the Z41 geometry in the software. Four types of clay were used to prepare the first set of suspensions, one from Bensmim region, from “Bab Mansour” manufactory, and the two types used by “ExtraBric”. The second set of measurements was done on the mixture of the two types of clay used by “ExtraBric” once varying the concentration of the grey clay and another type varying the concentration of yellow clay. Once the suspension ready, 11.9 mL of it is placed in the cell and the test is run to obtain two different curves for each suspension: $\tau = f(\dot{\gamma})$ and $\eta = f(\dot{\gamma})$. The curves obtained are attached in the appendices.

5.2. **Results**

The results obtained are all coherent and show that our samples are shear thinning fluids following the law of Ostwald Waele, with a melt flow index varying from 0 to 1.
These results are coherent with the type of clay we are working on, the one of Bensmim which was determined as an Illite from the X-rays results. The results in term of observations are also coherent with the experimental results. Indeed, the more liquid mixture was the one of Bensmim, followed by the suspensions of the grey clay from “ExtraBric”, then yellow clay from the same manufactory, the mixture of the two types of clays, and finally the clay of “Bab Mansour” was the most viscous. The gel appearance was reached only at high concentrations, mainly 70%, and here again the gelliest was the one brought from “Bab Mansour”. The following graphs show the tendency of the consistence and the melt flow index in terms of the increase of the concentrations. The corresponding values are attached in the appendix.

![Graph showing the tendency of K and MFI for “ExtraBric” grey clay with respect to the concentration](Image)

**Figure 5.2.1**: Tendency of K and MFI for “ExtraBric” grey clay with respect to the concentration
Figure 5.2.2: Tendency of K and MFI for “ExtraBric” yellow clay with respect to the concentration.

Figure 5.2.3: Tendency of K and MFI for “Bab Mansour” clay with respect to the concentration.
Figure 5.2.4: Tendency of K and MFI for Bensmim clay with respect to the concentration

All the graphs show clearly that the highest the concentration is, the lowest is the MFI, which explains that the gelly aspect of the suspension corresponds to lowest values of the melt index flow.

Since “ExtraBric” manufactory mix both the yellow and the grey clay, suspensions of the mixture at different concentrations of the two types of clay were subjected to rheological tests in order to understand the effect of each type of clay on the mixture, and come up with the best percentage for a better gelly structure. The suspensions were prepared at a constant concentration of grey clay first, then constant concentration of yellow clay. Based on the observations while preparing the mixtures, the addition of more grey clay leaded to the gelliest aspect. The results attached in the appendix as well as the graphs confirm the observations since the lowest MFI corresponds to the mixture with the highest amount of grey clay.
**Figure 5.2.5:** Comparison of the coefficient of consistence for all the suspensions with respect to the concentration

**Figure 5.2.6:** Comparison of the melt flow index for all the suspensions with respect to the concentration
5.3. Analysis and discussion

In this analysis we cannot rely on the values of the consistence K since sedimentation can occur, that is why this discussion will be based on the melt flow index. The obtained results enable us to determine the type of clays used, as well as their behaviour. All the fluids are following a power law with an MFI varying in a range of 0 to 1, which means that these clays have a shear thinning behaviour. The shear thinning behaviour can be explained by the fact that in case of low deformations, the molecules are disposed randomly and hang on each other. However when the shear rate is increased, the molecular chains line up and slide on each other instead on hanging on. The shear thinning behaviour is negatively related with the MFI, meaning that with the decrease of the MFI leads to a more gelly aspect. This implies that the clay particles will absorb more water.

The observation during the preparation of the clay suspensions also explain the obtained results and will contribute in the conclusions that will be made later on. These observations are detailed in the results section.

Illites are known for their covalent bond and are swelling clays. Actually, between the sheets there are potassium ions, which make the positioning of the water molecules between the sheets impossible, which explains the incapability of this type of clays in absorbing water, leading to a liquid aspect. This explains the values of the MFI for this type of clay that are very high compared to the others.

The clay brought from “ExtraBric” and “Bab Mansour” is both extracted from the same region, located in the road between Meknes and Sidi Kacem: road to Moulay Driss, which means that these two clays will belong to the same category, but with a possible different behaviour. Indeed, the decrease in MFI leads to the conclusion that these clays absorb water, which explains there gelly aspect. By analysing the link between this
property and the properties of the different types of clays, the conclusion about the type of clays used by these two manufactories can easily be drawn.

The Smectites and more particularly the montmorillonites form a gel when dispersed in water thanks to their tridimensional structure, that is why they are used as thickening and plasticizers agents Montmorillonites are part of the Smectites, and are 2:1 layer sheets type. These clays are characterized by the Van der Waals bonds that are relating the sheets, making the positioning of the water molecules between the sheets possible, which explains the presence of pending groups ready to react, and thus the absorption of water for a decreasing MFI and a more gelly structure. This leads us to the conclusion that the clay used by “ExtraBric” and “Bab Mansour” is a smectite, a montmorillonite to be more precise. These results are coherent with the ones obtained in previous studies conducted by [27] and [28].

6. Part III: Additives to fired clay bricks

6.1. Introduction

One of the recent challenges in the construction domain consists of developing new alternatives in order to produce a more sustainable building material with high thermal and mechanical properties. The use of additives will contribute in decreasing the effect of clay bricks manufacturing, and at the same time will improve the initial material properties. Obtaining a higher compressive strength is the purpose of most of recent studies that have been made in this field. Many years ago, people started to add different materials such as straw and sheep wool to the unfired bricks, and recently other additives such as cement and lime [13]. There are many types of admixtures that have different properties depending on the category they belong to. Most of the organic additives such as rice husk ash and saw dust mainly aim to leave pores in the material while burning which contributes in obtaining lighter products and more porous ones which allows the
heat to reach the deepest part of the material and avoid unburnt cores. Another category of additives includes pozzolanic materials, also called cementitious, such as Fly ash and Wood Ash. This type of materials acts as fusing agents and consolidating substances. When added to clay, the bond between particles is increased which leads to an increase in compressive strength [14].

This project focuses about the experimental study of the effect of Wood Ash and olive pomace on the properties of fired clay bricks. The main purpose of this part will be to compare the compressive strength of many samples made of three different types of clay before and after adding the two types of ash previously mentioned at various percentages.

a. Wood ash

Wood ash is characterized by being rich in CaCO$_3$ (calcium carbonate), which lakes of it one of the greatest binding agents, when added helps maintaining the shape. Wood as is a pozzolanic material with interesting stabilizing properties. It is one of the ancient stabilizers known in the history, with good binding and waterproof properties, which provide the material strength and avoid cracking thanks to its chemical composition mostly the presence of potassium components, which contribute in the bonding properties. Many researchers, such as Fajobi and Ogunbanjo (1994) have used wood ash in order to increase the compressive strength of adobe bricks [20].

b. Olive pomace ash

Olive pomace ash is rich of potassium and organic matter. Olive pomace has been used for a long time as a fertilizer since it contains large amounts of potassium. It has also been used as a soil amendment, as a remover of cooper ions, and as an additive to cementitious products and any other building materials. Many researchers evaluated
the retrieval of biomass ashes as one of the raw materials for any kind of building material [21]. Classified as an organic matter, it mainly contributes in leaving pores in the material while burning as well as in obtaining lighter products [20].

6.2. Experiments conducted

a. Raw material

Three different types of clay have been used to prepare our samples. The first one, a mixture of grey and yellow clay brought from “ExtraBric” manufactory, the second one brought from “Bab Mansour” manufactory, and the last one from Bensmim region. Concerning the ash, the two types are the olive pomace ash used by Al Akhawayn University, and wood ash from the fireplace of a friend’s home in Ifrane.

Figure 6.2.1: Clay extracted from Bensmim region (a) and grinded (b)

Figure 6.2.2: Clay brought from “ExtraBric” (a) and grinded (b)
In order to determine the composition of the raw material used, a sample of each was sent for X-rays to the Faculty of Science of Agdal-Rabat, and the results are still not ready yet. The results of a previous X-rays diffraction test led by Dr. Khaledoun and her colleagues are reported in the Appendix [15], and show that Bensmim clay is an Illite [16].

b. Experimental procedure

The first step of the procedure consists of preparing the raw materials. First of all, both the clays and the ashes were placed in the oven at 120°C for a drying process. Once this step completed, the clay was grinded using a mortar and a pestle then a blender in order to reduce the grain size to micrometres, and then sifted. Seven samples of each type of clay and each type of ash additive were prepared at different percentages. The total weight of the mixture was the same for all the samples but with
different components’ proportions. All were mixed with water by hands, and then put in circular molds obtained for the chemistry laboratory.

<table>
<thead>
<tr>
<th></th>
<th>Clay</th>
<th>Wood Ash</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sample 1</strong></td>
<td>100%</td>
<td>0%</td>
<td>23.5%</td>
</tr>
<tr>
<td><strong>Sample 2</strong></td>
<td>95%</td>
<td>5%</td>
<td>25.2%</td>
</tr>
<tr>
<td><strong>Sample 3</strong></td>
<td>90%</td>
<td>10%</td>
<td>25.2%</td>
</tr>
<tr>
<td><strong>Sample 4</strong></td>
<td>85%</td>
<td>15%</td>
<td>25.2%</td>
</tr>
<tr>
<td><strong>Sample 5</strong></td>
<td>80%</td>
<td>20%</td>
<td>29.5%</td>
</tr>
<tr>
<td><strong>Sample 6</strong></td>
<td>70%</td>
<td>30%</td>
<td>31.3%</td>
</tr>
<tr>
<td><strong>Sample 7</strong></td>
<td>60%</td>
<td>40%</td>
<td>32.1%</td>
</tr>
</tbody>
</table>

Table 6.2.1: Composition of “ExtraBric” samples using WA

![Figure 6.2.5: Samples using “ExtraBric” clay and WA before drying](image)

The idea about the amount of water to be added was determined first by the amount suggested by the manufactories, and second from the rheology results which were showing the best composition clay-water for better results. This is what explains the difference in the amount added depending on the type of clay used.

<table>
<thead>
<tr>
<th></th>
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<th>Wood Ash</th>
<th>Water</th>
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</thead>
<tbody>
<tr>
<td><strong>Sample 8</strong></td>
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<td>0%</td>
<td>21.7%</td>
</tr>
</tbody>
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62
Table 6.2.2: Composition of Bensmim samples using WA

<table>
<thead>
<tr>
<th>Sample</th>
<th>Clay</th>
<th>Wood Ash</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 9</td>
<td>95%</td>
<td>5%</td>
<td>23.4%</td>
</tr>
<tr>
<td>Sample 10</td>
<td>90%</td>
<td>10%</td>
<td>23.4%</td>
</tr>
<tr>
<td>Sample 11</td>
<td>85%</td>
<td>15%</td>
<td>24.3%</td>
</tr>
<tr>
<td>Sample 12</td>
<td>80%</td>
<td>20%</td>
<td>26.08%</td>
</tr>
<tr>
<td>Sample 13</td>
<td>70%</td>
<td>30%</td>
<td>27.8%</td>
</tr>
<tr>
<td>Sample 14</td>
<td>60%</td>
<td>40%</td>
<td>30.4%</td>
</tr>
</tbody>
</table>

Figure 6.2.6: Samples using Bensmim clay and WA before drying

Table 6.2.3: Composition of “Bab Mansour” samples using WA

<table>
<thead>
<tr>
<th>Sample</th>
<th>Clay</th>
<th>Wood Ash</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 15</td>
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<tr>
<td>Sample 16</td>
<td>95%</td>
<td>5%</td>
<td>37.5%</td>
</tr>
<tr>
<td>Sample 17</td>
<td>90%</td>
<td>10%</td>
<td>37.5%</td>
</tr>
<tr>
<td>Sample 18</td>
<td>85%</td>
<td>15%</td>
<td>38.2%</td>
</tr>
<tr>
<td>Sample 19</td>
<td>80%</td>
<td>20%</td>
<td>39.1%</td>
</tr>
<tr>
<td>Sample 20</td>
<td>70%</td>
<td>30%</td>
<td>40%</td>
</tr>
<tr>
<td>Sample 21</td>
<td>60%</td>
<td>40%</td>
<td>40%</td>
</tr>
</tbody>
</table>
Once the samples ready, they were left for 2 hours to rest before stating the controlled drying process. Samples were put in the oven at a starting temperature of 25°C which was increased by 10°C after each hour until stabilizing at 110°C in order to avoid the cracking of the handmade bricks. The drying process lasted for 24 hours.

After drying, the samples were left for cooling before processing to the firing process. At this stage, the samples were put in the oven, at a starting temperature of 200°C in order to eliminate the remaining water, and increasing every 30 minutes by 100°C until stabilizing at 850°C. The purpose of the progressive increase once again is to avoid to the bricks to break. After 24 hours, the temperature is again increased to
950°C, and then to 1100°C after 12 hours for a better vitrification. The same process is repeated for the making the bricks using olive pomace additive.

<table>
<thead>
<tr>
<th></th>
<th>Clay</th>
<th>Olive Pomace Ash</th>
<th>Water</th>
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<tbody>
<tr>
<td>Sample 22</td>
<td>95%</td>
<td>5%</td>
<td>21.7%</td>
</tr>
<tr>
<td>Sample 23</td>
<td>90%</td>
<td>10%</td>
<td>23.48%</td>
</tr>
<tr>
<td>Sample 24</td>
<td>85%</td>
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</tr>
<tr>
<td>Sample 25</td>
<td>80%</td>
<td>20%</td>
<td>26.1%</td>
</tr>
<tr>
<td>Sample 26</td>
<td>70%</td>
<td>30%</td>
<td>26.9%</td>
</tr>
<tr>
<td>Sample 27</td>
<td>60%</td>
<td>40%</td>
<td>27.8%</td>
</tr>
</tbody>
</table>

Table 6.2.4: Composition of Bensmim samples using OPA

<table>
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<th>Clay</th>
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</thead>
<tbody>
<tr>
<td>Sample 28</td>
<td>95%</td>
<td>5%</td>
<td>23.47%</td>
</tr>
<tr>
<td>Sample 29</td>
<td>90%</td>
<td>10%</td>
<td>24.34%</td>
</tr>
<tr>
<td>Sample 30</td>
<td>85%</td>
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</tr>
<tr>
<td>Sample 31</td>
<td>80%</td>
<td>20%</td>
<td>27.82%</td>
</tr>
<tr>
<td>Sample 32</td>
<td>70%</td>
<td>30%</td>
<td>29.56%</td>
</tr>
<tr>
<td>Sample 33</td>
<td>60%</td>
<td>40%</td>
<td>30.43%</td>
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</tbody>
</table>

Table 6.2.5: Composition of “ExtraBric” samples using OPA

<table>
<thead>
<tr>
<th></th>
<th>Clay</th>
<th>Olive Pomace Ash</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 34</td>
<td>95%</td>
<td>5%</td>
<td>23.8%</td>
</tr>
<tr>
<td>Sample 35</td>
<td>90%</td>
<td>10%</td>
<td>24.76%</td>
</tr>
<tr>
<td>Sample 36</td>
<td>85%</td>
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<td>25.7%</td>
</tr>
<tr>
<td>Sample 37</td>
<td>80%</td>
<td>20%</td>
<td>28.1%</td>
</tr>
<tr>
<td>-----------</td>
<td>-----</td>
<td>-----</td>
<td>-------</td>
</tr>
<tr>
<td>Sample 38</td>
<td>70%</td>
<td>30%</td>
<td>30.5%</td>
</tr>
<tr>
<td>Sample 39</td>
<td>60%</td>
<td>40%</td>
<td>31.9%</td>
</tr>
</tbody>
</table>

**Table 6.2.6:** Composition of “Bab Mansour” samples using OPA

**Figure 6.2.9:** Samples before drying using OPA and clay form Bensmim (a), clay from “ExtraBric (b), and clay from “Bab Mansour” (c)

**c. Observations**

During the preparation of the samples, many remarks were noted. First of all, the aspect of the mixture differed according to the type of clay and the type of ash added. Clay of Bensmim required the less amount of water in order to reach the viscoelastic state. Coming back to the rheology results, Bensmim clay is the less viscous one and the one that absorbs less water. This shows the coherence between the rheological test and the experiments conducted. However, when adding the olive pomace ash, more water is required, and even more when adding the wood ash. Also, this type of clay was the easier to mold compared to the clay for ExtraBric as well as Bab Mansour which was the most difficult to shape and required the highest amount of water.
During the drying process, it was noted that the samples that are mixed with the olive pomace ash were peeling, and the ones with the highest amounts of ash (30% and 40%) were totally damaged.

![Figure 6.2.10: Dried sample using clay from Bensmim and 40% OPA](image)

Once all the samples fired, it was noted that the stiffed ones were from Bensmim clay mixed with wood ash, followed by the ones from “Bab Mansour”, also mixed with wood ash, and finally the ones from “ExtraBric” clay. However, the samples mixed with olive pomace ash seemed to have lower compressive strength. Another remark related to the weight of the samples was noted. The samples mixed with olive pomace ash were lighter than the ones mixed with wood ash, lighter than the ones made of 100% clay. Also the lightest samples were the ones from “ExtraBric” clay, and the heaviest, the ones from Bensmim clay. Regarding the color, Bensmim
samples were the darkest, and the ones made of “ExtraBric’ and “Bab Mansour” clays were yellow.

![Image of samples after 48 hours of firing](image_url)

**Figure 6.2.12:** Samples after 48 hours of firing

### 6.3. Results

**a. Apparent porosity**

Porosity and compressive strength are negatively related, meaning that an increasing porosity corresponds to a decreasing compressive strength. The formula for the apparent porosity is the following with $W$ the saturated weight, $D$ the dried weight, and $V$ the volume [26].

$$\text{Apparent porosity} \% = \frac{W - D}{V} \times 100$$

The following graphs show the tendency of the apparent porosity which enables us to conclude that for the mixture clay-wood ash, the compressive strength will be higher for increasing percentages of ash. However, for the mixture olive pomace ash-clay, the compressive strength will decrease with the increase of the OPA percentage.
Figure 6.3.1: Apparent porosity for Bensmim clay with respect to the percentage of WA

Figure 6.3.2: Apparent porosity for ExtraBric clay with respect to the percentage of WA
Figure 6.3.3: Apparent porosity for Bab Mansour clay with respect to the percentage of WA

The graphs of the apparent porosity using OPA show that the decrease occurs at a specific percentage, which means that the improvement of compressive strength happens when adding 5% to Bensmim clay and ExtraBric, and do not happen in case of the clay brought from Bab Mansour.

Figure 6.3.4: Apparent porosity for Bensmim clay with respect to the percentage of OPA
Figure 6.3.5: Apparent porosity for ExtraBric clay with respect to the percentage of OPA

Figure 6.3.6: Apparent porosity for Bab Mansour clay with respect to the percentage of OPA

b. Bulk density

Bulk density and compressive strength are positively related, that is why it is important to calculate this property in order to confirm whether the wood ash and
olive pomace ash improve the mechanical property or not. The formula to calculate the bulk density is the following [26]:

\[ B \left( \frac{g}{cm^3} \right) = \frac{D}{V} \]

The graphs obtained are totally coherent with the previous results since the bulk density is increasing when adding more wood ash; however it is decreasing when adding more olive pomace ash except for 5% in case of Bensmim and ExtraBric.

**Figure 6.3.7:** Bulk density for Bensmim clay with respect to the percentage of WA

**Figure 6.3.8:** Bulk density for ExtraBric clay with respect to the percentage of WA
**Figure 6.3.9:** Bulk density for Bab Mansour clay with respect to the percentage of WA.

**Figure 6.3.10:** Bulk density for Bensmim clay with respect to the percentage of OPA.
**Figure 6.3.11:** Bulk density for ExtraBric clay with respect to the percentage of OPA

**Figure 6.3.12:** Bulk density for Bab Mansour clay with respect to the percentage of OPA
7. Conclusion

The purpose of this project was to find the perfect combination of ash additive in order to increase the compressive strength of the fired clay bricks, focusing more about the bricks that can be made from Bensmim clay. Unfortunately, the results of the compressive strength tests are not yet ready. However, the evaluation of the apparent porosity and the bulk density enabled us to conclude that the compressive strength when is improved when the additive is a pozzolanic admixture, and improved at 5% only when the additive is olive pomace ash which is considered as an organic material. These results are coherent with the bibliographical study that was conducted at the beginning of this work. The rheological tests were of a great importance since it enabled us to understand the behaviour of the three types of clays, enabling us to know which type of clay we worked on even if the X-rays results are not yet launched. In addition to that, the rheology contributed in the determination of the perfect composition clay-water in terms of water percentages to be added, and that are varying from 10% to 20% depending on the type of clay and the type of additive.

To sum up, this study encourages the use of the waste ash especially the wood ash which is a pozzolanic admixture working as a bonding agent. The clay houses will benefit from a good and cheap product, making the living conditions in the poor regions better.

As a future work, the next capstone student can work on new additives which will be complementary to the one investigated during this project, and which will improve the
thermal properties, without affecting the mechanical ones. Finding a new combination of
different types of clays and additives should be beneficial the main purpose of this work.
References


Appendices

Appendix 1: X-rays and fluorescence results

![Figure 1: X-rays of Bensmim clay](image)

<table>
<thead>
<tr>
<th>Chemical element</th>
<th>Percentage of chemical element (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>59.6</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>22.4</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>6.69</td>
</tr>
<tr>
<td>CaO</td>
<td>0.0777</td>
</tr>
<tr>
<td>MgO</td>
<td>0.97</td>
</tr>
<tr>
<td>K₂O</td>
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</tr>
<tr>
<td>TiO₂</td>
<td>0.832</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.458</td>
</tr>
<tr>
<td>P.a.f</td>
<td>5.34</td>
</tr>
</tbody>
</table>

Table 1: Chemical composition of Bensmim clay
Appendix 2: Rheograms obtained from the rheology tests on clay from “Bab Mansour”

**Figure 2:** Rheogram for “Bab Mansour” clay at C= 20%

**Figure 3:** Rheogram for “Bab Mansour” clay at C= 30%

**Figure 4:** Rheogram for “Bab Mansour” clay at C= 50%

**Figure 5:** Rheogram for “Bab Mansour” clay at C= 70%
Appendix 3: Rheograms obtained from the rheology tests on Grey clay from “ExtraBric”

Figure 6: Rheogram for “ExtraBric” grey clay at C= 20%

Figure 7: Rheogram for “ExtraBric” grey clay at C= 30%

Figure 8: Rheogram for “ExtraBric” grey clay at C= 50%

Figure 9: Rheogram for “ExtraBric” grey clay at C= 70%
Appendix 4: Rheograms obtained from the rheology tests on Yellow clay from “ExtraBric”

**Figure 10:** Rheogram for “ExtraBric” yellow clay at C= 20%

**Figure 11:** Rheogram for “ExtraBric” yellow clay at C= 30%

**Figure 12:** Rheogram for “ExtraBric” yellow clay at C= 50%

**Figure 13:** Rheogram for “ExtraBric” yellow clay at C= 70%
Appendix 5: Rheograms obtained from the rheology tests on Mixture of the two clays “ExtraBric” at constant concentration of Grey clay

Figure 14: Rheogram for the mixture of “ExtraBric” clays at C= 30% and $C_{grey}= 10%$

Figure 15: Rheogram for the mixture of “ExtraBric” clays at C= 50% and $C_{grey}= 10%$

Figure 17: Rheogram for the mixture of “ExtraBric” clays at C= 70% and $C_{grey}= 10%$
Appendix 6: Rheograms obtained from the rheology tests on Mixture of the two clays “ExtraBric” at constant concentration of Yellow clay

Figure 18: Rheogram for the mixture of “ExtraBric” clays at C= 30% and $C_{\text{yellow}}= 10\%$

Figure 19: Rheogram for the mixture of “ExtraBric” clays at C= 50% and $C_{\text{yellow}}= 10\%$

Figure 20: Rheogram for the mixture of “ExtraBric” clays at C= 70% and $C_{\text{yellow}}= 10\%$
Appendix 7: Values of MFI $n$ and consistence $K$

<table>
<thead>
<tr>
<th>Type of clay</th>
<th>Concentration</th>
<th>Rheological equation</th>
<th>Consistence $k$ (Pa.s$^n$)</th>
<th>Melt Flow Index $n$</th>
</tr>
</thead>
<tbody>
<tr>
<td>BAB MANSOUR</td>
<td>20%</td>
<td></td>
<td>0,08032</td>
<td>0,5464</td>
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<td></td>
<td>30%</td>
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<td></td>
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<td>0,744</td>
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<td>Ostwald Waele</td>
<td>0,02484</td>
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Table 2: Values of $K$ and $n$ for each type of clay

Table 3: Values of $K$ and $n$ for the mixture of the two clays from “ExtraBric”