

T.C
MARMARA UNIVERSITY
INSTITUTE OF GRADUATE STUDIES IN PURE AND APPLIED
SCIENCES

RECYCLING OF EXCAVATION SOIL FOR THE
PRODUCTION OF BUILDING MATERIAL

Glden ađın ULUBEYLİ

THESIS
FOR THE DEGREE OF MASTER OF SCIENCE
IN
METALLURGICAL AND MATERIALS SCIENCE
ENGINEERING

SUPERVISOR
Assoc. Prof. Dr. Recep ARTIR

İSTANBUL 2011

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June, 2011

Gülden Çağın ULUBEYLİ

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ÖZET

HAFRİYAT TOPRAĞININ BİR YAPI ELEMANI ÜRETMEK İÇİN GERİ KAZANILMASI

Yapı sektörü içinde karşılaşılan en büyük sorunlardan biri, yapının inşası sırasında büyük miktarlarda ortaya çıkan kazı toprağının değerlendirilemeyen kısmının depolanma ve depo yerine taşınma sorunudur. Özellikle büyük ölçekli inşaatlarda kazı toprağının büyük bir kısmı atıl hale geldiğinden, malzemenin tahliyesinde ve depolanmasında büyük sorunlara ile karşılaşılmaktadır.

Çalışmada özellikle hammaddesi bu şekilde atık ve değerlendirilmeye açık olan bir malzemenin farklı üretim yöntemleri ile tuğla benzeri bir yapı malzemesi olarak kullanılabilirliği araştırılmıştır. Bu amaçla literatür çalışmaları sonucunda kullanılmasına karar verilen 8 farklı bağlayıcı çeşidi ile hafriyat toprağı karıştırılmış ve klasik üretim yönteminde yüksek sıcaklıklarda fırınlanarak elde edilen tuğlanın, düşük sıcaklıkta bağlayıcıların birleştirici özellikleri kullanılarak üretimi yoluna gidilmiştir. Çünkü tuğlanın pişirme sıcaklığı oldukça yüksek olduğundan, üretiminde çok fazla enerji maliyeti bulunmaktadır.

Deneysel aşama sırasıyla hammadde karakterizasyonu, numune üretimi ve numune karakterizasyonu şeklinde üç ana bölümden oluşmaktadır. İlk bölümde bazı toprak deneyleri ve kimyasal analizler gerçekleştirilerek malzeme özellikleri belirlenmiştir. İkinci kısımda bağlayıcılar ve hammadde, 10 değişik karışım seti ile, 76 mm çapa ve 40 mm yüksekliğe sahip çelik kalıp içerisine 200 KN yük altında basılmıştır. Son bölümde ise üretilen numune üzerinde basınç mukavemeti, su emme, kuru birim hacim ağırlık, aktif çözünmüş tuz içeriği, boşluk oranı tayini deneyleri yapılmış ve tüm sonuçlar, mevcut kullanılan tuğla malzemeleri için oluşturulmuş iki TS Standardındaki değerler ile karşılaştırılmıştır. Sonuç olarak tüm test edilen özellikler bakımından standartlarda istenilen değerlere ulaşılmış veya çok yaklaşmıştır.

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ABSTRACT

RECYCLING OF EXCAVATION SOIL FOR THE PRODUCTION OF BUILDING MATERIAL

One of the greatest problems in the construction industry is how to store and transport the unused part of excavation soil which appears during construction with enormous amounts. Especially in large-scale construction projects, since huge part of excavation soil becomes waste material, a big problem arises while discharging and storing the material.

In this study, opportunities of using the brick produced with different production methods and called as a brick-like construction material of which raw material is waste and open to utilize were investigated. For this objective, the excavation soil was mixed with 8 different binders which were decided to use as a result of literature review. A brick-like material was produced by considering the bonding effect of binders at low temperatures, which in fact is produced by firing at high temperatures in the classical production technique. An excessive energy cost in producing this material is encountered because the firing temperature of brick is very high.

The experimental phase consisted of three sections such as characterization of raw material, and production and characterization of samples, respectively. Some soil experiments and chemical analyses were first carried out to determine the material characteristics. 10 different mixture sets including binders and raw materials were then pressed under a load of 200 kN in a steel mould of which diameter was 76 mm and height was 40 mm. Finally, some experiments such as compressive strength, water absorption, dry bulk density, active dissolved salt content, and void percentage were realized on the samples produced, and all of the results obtained were compared with the related brick values in TS Standards. Consequently, in terms of the tested characteristics, expected values in the Standards were either reached or approached very nearly.

June, 2011

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SYMBOLS

d_u	: Diameter
h	: Hour
h_u	: Height
M_{au}	: Mass in Air
M_d	: Dry Mass
M_g	: Magnesium Content
M_{mg}	: Molecular Weight of Magnesium
M_w	: Wet Mass
M_{wu}	: Mass in Water
V_{gu}	: Gross Volume
V_{nu}	: Net Volume
V_s	: Volume of the Mould
V_{vu}	: Volume of Voids
W_m	: Water Absorption
W_n	: Water Content of the Soil
x	: Volume of EDTA in the Second Titration
y	: Volume of EDTA in the First Titration
ρ_w	: Density of Water

ABBREVIATIONS

AAS	: Atomic Absorption Spectroscopy
ASTM	: American Society for Testing and Materials
CMC	: Carboxyl Methyl Cellulose
CSH	: Calcium Silicate Hydrate
EDTA	: Ethylene Diamine Tetra-Acetic Acid
EDX	: Energy Dispersive X-Ray
GGBS	: Grand Granulated Blast Furnace Slag
LOC	: Lower Oxford Clay
PC	: Portland Cement
SBD	: Solid-State Backscattered
SEM	: Scanning Electron Microscopy
SLS	: Sodium Ligno Sulfonate
TS	: Turkish Standards
XRD	: X-Ray Diffraction
XRF	: X-Ray Fluorescence

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CHAPTER I

INTRODUCTION AND AIM

I.1 INTRODUCTION

Soil excavation is defined as digging, grading, tunneling, trenching, and/or drilling below grade. It is difficult to carry out the process of excavation, because the process is mostly long and expensive. The excavated soil is dug by different machines, and can locate in various forms. These forms may include clay, soil, rock, etc. Every year, thousands tons of soil are excavated from the earth in construction industry. The excavated soil is not utilized efficiently. This valuable raw material can be recycled in several ways. For example, it could be used as a building material such as brick.

Bricks have been used over 5000 years as a construction and building material throughout the world. Today, the bricks are still being used for the same purpose. As the urbanization and civilization expand, demand for bricks gradually increases. Although clay-based brick has excellent durability as a construction material, the quality of bricks is still major concern in most places of the world. Data regarding the properties of masonry components such as bricks are abundant, but there is still much to learn about bricks. There are 70 factories producing brick and tile in Turkey. Ten of these factories work on tile only. The others (60 companies) produce both brick and tile. The capacity of brick production in Turkey is about 7.4 billion pieces or 22.1×10^9 kg per year. In terms of capacity, the utilization rate is 75% in Turkey (Report, 2001).

Considering previous research works about bricks, just a few of these studies was conducted on the low temperature-fired bricks and their engineering properties. For instance, one of them was about the behavior of brick masonry as a structural material under specific loading conditions. Also, changes of stress-strain characteristic and modulus of elasticity were reported (Mbumbia and Wilmars, 2001). Another research work was carried out in Cameroon region. It represented an effort to examine the

improvement of physical and mechanical properties of some Cameroonian lateritic soil bricks by stabilization through heat (at very low temperatures) with a view to finding their suitability for the construction of walls of single-family houses (Mbumbia et al, 2000). In another study, experimental results have been reported on the compressive strength and microstructure of unfired clay masonry bricks (Oti et al, 2009).

I.2 AIM

In this study, domestic excavated soil in Zonguldak region, which is located in north-west of Turkey, was collected and utilization possibilities were investigated in depth in laboratory conditions. A building material was produced by using excavated soil and binder, and experiments were made on the mixture materials to determine mechanical and physical properties. Then, these properties were compared to those of the commercial bricks.

As far as the conventional brick production is concerned, substantial amount of energy is required for the brick production. The aim of this thesis is to reduce and minimize the energy consumption in the brick-making process by lowering the firing or heat treatment temperature, and to recycle and convert excavation soil into a value-added product. Moreover, reduction in energy consumption and duration of firing process will be expected to provide lower production cost, and thus, economical savings will be obtained. Lower energy consumption, temperature, and lower thermal treatment means relatively reduced gas emissions release to the atmosphere, which could be resulted in less environmental pollution and less greenhouse gas effect. Therefore, environmentally friendly building material will also be intended to produce. The primary objective of this research study is to reduce firing temperature of brick by adding various organic and inorganic binders into the brick mixture. It is hoped that samples produced using excavation soils throughout this work could exhibit similar mechanical and physical properties of the commercial brick which is still in use in the construction materials market.

CHAPTER II

GENERAL BACKGROUND

II.1 LITERATURE REVIEW

In the literature, there are a number of research studies on this particular domain of civil engineering. Oti and Bai (2009) researched engineering properties of unfired clay masonry clay bricks. Mixes were formulated using a locally available industrial by-product (Ground Granulated Blast furnace Slag — GGBS) which is activated with an alkaline (lime or Portland cement) combined with clay soil. Portland cement was not used in the formulation of the unfired stabilized masonry bricks, except as a controlling device, which is a significant scientific breakthrough for the building industry. In this study, the laboratory results demonstrated that some properties such as compressive strength, moisture content, rate of water absorption, percentage of porosity, density, and durability assessment (repeated 24-hour freezing/thawing cycles) were all within the acceptable engineering standards for clay masonry units.

Oti et al. (2009a) reported an investigation concerning compressive strength and micro structural analysis of unfired clay masonry bricks. They blended binders comprising of lime-activated Ground Granulated Blast furnace Slag (GGBS) and Portland Cement (PC)-activated GGBS were used to stabilize Lower Oxford Clay (LOC) for unfired masonry brick production. The compressive strength of the stabilized bricks incorporating lime–GGBS–LOC was higher than that of PC–GGBS–LOC. Scanning Electron Microscopy (SEM) with a Solid-state Backscattered Detector (SBD) and Energy Dispersive X-ray (EDX) analysis was employed to obtain a view of the microstructure and to conduct an analysis on the morphology and composition of the dried unfired clay brick samples, after 28 days of moist curing. The quantification of the compound content of the unfired bricks showed the presence of Calcite (CaCO_3), Quartz

(SiO₂), Alumina (Al₂O₃) and Wollastonite (CaSiO₃) crystals. Traces of other crystals were also detected.

Oti et al. (2009b) found design thermal values for unfired clay bricks. They investigated a laboratory and theoretical method for determining the design values for thermal conductivity and thermal resistance of unfired clay masonry bricks from both experimental and theoretical design point of view. In order to determine the basic design thermal value and the design thermal resistance, a Laser-comp FOX 200 thermal conductivity meter equipped with WinTherm 32 a software package was employed for the laboratory data collection and analysis. Lime or Portland cement (PC)-activated Ground Granulated Blastfurnace Slag (GGBS) binder was used to stabilize Lower Oxford Clay (LOC) for unfired masonry brick specimen production.

Bideci et al. (2009) examined an investigation into the usability of different raw materials in brick production. They investigated the usability of different raw materials in brick production. To this end, brick clay was obtained from the brick factories in the Imrahor region in Ankara. Diatomite raw material from the diatomite plants of Ankara Sugar Factory, and expanded perlite from Eti Holding Inc. Perlite Enterprise in Cumaovası, Izmir were used. Raw materials were used with rates of 0%, 10%, 20% and 30%. Samples were fired at temperatures of 800°C, 900°C, and 1000°C. For proper brick production, the fired samples were subjected to water absorption, frost resistance, and compressive strength tests.

Chindaprasirt and Pimraksa (2007) reported about the properties of fly ash–lime granule unfired bricks. In this study, Granules were prepared from mixtures of fly ash and lime at fly ash to hydrated lime ratios of 100:0 (Ca/Si=0.2), 95:5 (Ca/Si=0.35) and 90:10 (Ca/Si=0.5) and after a period of moist curing, the microstructure and mineralogy of the granules were researched.

Diop and Grutzeck (2007) examined the low temperature process to create brick. They studied on making of durable alkali-activated aluminosilicate brick. As a distinctive feature, brick made with this technology can be obtained in a few hours using a pressurized vessel and elevated curing temperatures (100°C) or after three to four weeks, if the brick is cured under a tarp or in a solar heated enclosure in areas where energy is unavailable. The process is less expensive than conventional kiln firing of

brick, because the development of strength is a result of a chemical reaction that takes place at a relatively low temperature.

Millogo et al. (2007) investigated microstructure and physical properties of lime-clayey adobe bricks in a detailed manner. They presented micro structural changes of adobe bricks made of lime-clayey raw material. In that study, they carried out some analysis as X-ray diffraction, infrared spectrometry, differential thermal analyses, scanning electron microscopy, and energy dispersive spectrometry. The impact of these changes on the mechanical resistance and water absorption of adobe bricks were evaluated and finally found that lime additions resulted in the development of calcite and poorly crystallized calcium silicate hydrate (CSH).

Khattab et al. (2007) examined strength, durability, and hydraulic properties of clayey soil stabilized with lime and industrial waste lime. They reported the effect of utilization of industrial waste/lime (by-product of sugar factory) on some engineering properties of clayey soil selected from Mosul city in Iraq. These characteristics were unconfined compressive strength, permeability, soil-water characteristic curve, and durability. These tests were performed at different percentages of lime (2, 4 and 6%) and industrial waste/lime (2, 4, 6 and 8%) by dry weight of soil.

Abalı et al. (2005) examined about using of wastes produced in phosphoric acid plants and boron concentrators in producing structural brick. With this objective, they studied several parameters (firing temperature, heating rate) measured by several criteria such as natural drying shortening, water absorption and weight loss and so many experiments carried out. The results had shown that the industrial wastes produced by phosphoric acid plants can improve the performance of the bricks in terms of drying shortening, weight loss and water absorption.

Çelik et al. (2003) investigated usability of Isparta region pumice in the brick production. They used mixed specific rate of clays of Ankara zone and pumice of Isparta zone allowable in the brick product. To this aim, three different mixtures prepared: %100 clay on group (1), %75 clay + %25 1 mm pumice on group (2), %75 clay + %25 2 mm pumice on group (3), according to TS 705 compress pressure strength experiments made on these samples. The test results were compared with the standard values used in the building sector in Turkey.

Mbumbia and Wilmars (2002) investigated the behavior of low-temperature fired laterite bricks under uniaxial compressive loading. They presented the characteristics of the stress-strain relationship for predicting brick performance. When subjected to deformation, bricks behaved nonlinear plastic-elastic-plastic. These bricks were found to exhibit linear elasticity in a domain where strain varied between 2 and 5% according to the type. These values of strain were found to be higher than the maximum elastic strain (0.5%) observed for many ceramics.

Mbumbai et al. (2000) realized a case study in Cameroon about the performance characteristics of lateritic soil bricks fired at low temperatures. In their research, no admixture was used. According to the results of laboratory tests, the effect of homogenization obtained by crushing raw materials to reduce particle size, the cohesion of clay present in raw materials and the cementitious binder resulting from the transformation of some mineral phases at low temperatures were exploited.

Kumar (1999) examined Fly Ash-Lime-Phosphogypsum cementitious binders. In this work, he reported the results of an experimental investigation in which the compressive strength, water absorption, density, and durability of fly ash-lime-phosphogypsum bricks were investigated by using varying quantities of fly ash, lime, and calcined phosphogypsum.

Tasong et al. (1998) investigated mechanisms by which ground granulated blast-furnace slag prevents sulphate attack of lime-stabilised kaolinite. They researched the mechanisms by which ground granulated blast-furnace slag (GGBS) prevents sulphate attack of lime-stabilized kaolinite. Micro-structural and mineral phase analyses of compacted cylinders of lime-stabilized kaolinite with different percentages of lime replacement by GGBS have been carried out using scanning electron microscopy (SEM) and X-Ray Diffractometry (XRD). The cylinders were immersed in 4.2% Na_2SO_4 solution and also in demonized water for various time periods.

Özbayoğlu et al. (1997) gave shape to powder ulexite under pressure by using several binders. Compressive strengths of samples cured under low temperatures (20°C to 105°C) were investigated. Objective of the study was to examine powder usage in brick production.

II.2 WHAT IS BRICK?

II.2.1 Masonry

Masonry consistently ranks among consumers as the first choice in residential cladding materials. Brick and stone masonry have been favorites of builders and homeowners for hundreds of years. Masonry symbolizes strength, durability, and prestige, and at the same time adds warmth, color, and scale to a home. Masonry is most visible in building walls, but is also used in foundations, fireplaces, garden walls, retaining walls, floors, sidewalks, patios, and driveways. Natural stone as well as manufactured units of clay brick, concrete block, cast stone; structural clay tile, terra cotta, adobe, and glass block are all masonry materials. Brick, concrete block, and stone are the most popular and most widely used (Beall, 2004).

II.2.2 Bricks

Brick is obtained by moulding good clay into a block, which is dried and then burnt. This is the oldest building block to replace stone. Manufacture of brick started with hand moulding, sun drying, and firing in clamps. A considerable amount of technological development has taken place with better knowledge about the properties of raw materials, better machineries, and improved techniques of moulding, drying, and firing.

Brick can be made of several different materials, but the most common type of brick is made from ordinary clay soil. Clay brick is the oldest manufactured building material in the world, and it is still one of the most widely used. Sun-dried mud bricks are estimated to have been in use for about 10,000 years, and fired bricks since about 3,000 B.C. Sun-dried bricks are a traditional residential construction material in dry climates and are still used in many countries. More typically, modern clay brick is fired at over 1093°C in a large kiln to produce units that are very dense, hard, and durable. The color of the clay determines the color of the brick, and more clay can be combined to produce a variety of colors (Beall, 2004).

Brick textures vary depending on the moulding and forming process. Most bricks are shaped by extruding wet clay through a die and by slicing it to the appropriate size. Extruded brick may have holes cored through the middle which makes them lighter in

weight and allows mortar to physically interlock with the brick. Even though they may contain core holes, if the cores account for 15% or less of the cross sectional area of the brick, the units are still considered to be solid. By this definition, most bricks are considered solid masonry units (Beall, 2004).

II.2.3 Raw Materials of Brick

The raw materials for brick making are extracted from quarries and/or pits, and then, processed and mixed with water. The first stage is the extraction of raw materials. Generally, this is from unconsolidated deposits and blasting is not normally undertaken. The majority of manufacturers stockpile clay to minimize the quantity of extraction that is undertaken in wet weather when the movement of trucks on the clay material is difficult. Stockpiling also ensures better intermixing of the extracted material and minimizes segregation. Stockpiles may contain sufficient raw materials for a year's production. Stockpiling clay also weathers the raw material, and rainwater washes out some of the soluble salts, which might later cause efflorescence on the face of the brickwork. An alternative method of weathering the clay is to wash it thoroughly in a wash mill and store it in an open area traditionally called *clay back*. When the clay is removed from the stockpile, a full vertical cut is made from the stockpile. The objective of this stage is to produce a consistent material for the next stage of the brick-making process.

The processing of the clay involves grinding and working it to obtain plasticity and uniform workability. The fineness of the clay influences not only the external appearance of the finished brick but also physical characteristics such as compressive strength and water absorption. The mixing stage involves the addition of water to produce a homogenous material, and the quantity of water added to the clay depends upon the production method being used. There are three principal processes used to form or mould bricks, *soft mud moulding*, *extrusion/wire cutting*, or *semi dry pressing*.

In some works, other materials such as lime or crushed clinker may be added to act as fuel. Pigments may also be added to produce specific colors. In the next stage of the production process, which is shaping or forming the brick, it is necessary that the clay feed is consistent in content, grading, plasticity, and water content.

II.2.4 Classical Manufacture of Clay Bricks

The operations involved in the classical manufacture of clay bricks are represented diagrammatically in Figure II.1. After preparation of raw clay, the manufacture of fired brick is completed in four additional stages: *forming, drying, firing, and drawing and storage*. The basic process is always the same, and differences occur only in the moulding techniques. In ancient as well as more recent history, brick was exclusively hand-made since brick-making machines were invented in the late nineteenth century.

II.2.4.1 Forming

The first step in each forming method is tempering, where the clay is thoroughly mixed with a measured amount of water. The amount of water and the desired plasticity vary according to the forming method to be used.

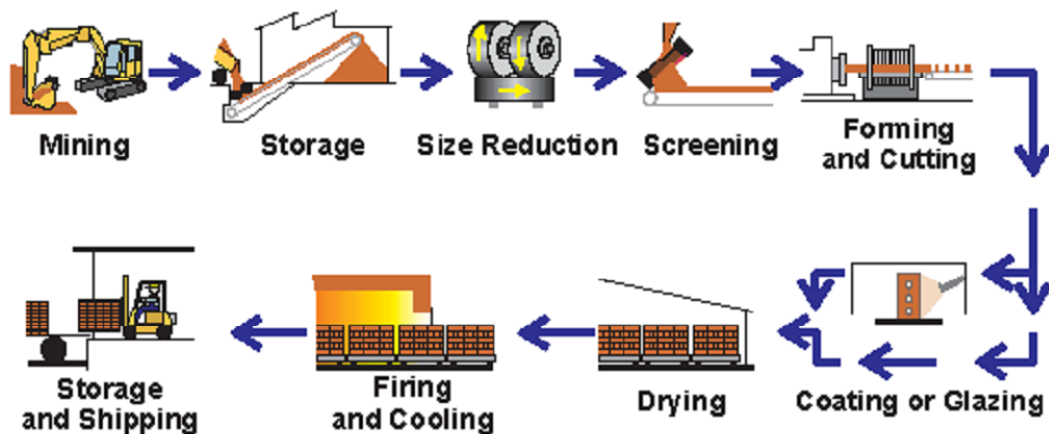


Figure II.1 Brick Manufacturing Process

A minimum amount of water, generally 12 to 15% moisture by weight, is mixed with the dry clay to make it plastic. After thorough mixing in a pug mill, the tempered clay goes through a de-airing process which increases the workability and plasticity of the clay and produces units with greater strength. The clay is then forced through a steel die in a continuous extrusion of the desired size and shape, and at the same time, is cored to reduce weight and to facilitate drying and firing. Automatic cutting machines using thin wires attached to a circular steel frame cut the extruded clay into pieces. Since the clay will shrink as it is dried and burned, die sizes and cutter wire spacing must be

carefully calculated to compensate. Texturing attachments may be affixed to roughen, score, scratch, or otherwise alter the smooth skin of the brick column as it emerges from the die. After cutting, clay slurry of contrasting color or texture may also be applied to the brick surface to produce different aesthetic effects.

II.2.4.2 Drying

Drying time takes only 24 to 48 hours, depending on the original moisture content. Drying temperatures range from 82 to 382°C, but must be carefully regulated, along with humidity, to prevent sudden changes which could crack or warp the units.

II.2.4.3 Glazing

Glazing is a highly specialized, carefully controlled procedure used in the production of decorative brick. *High-fired ceramic glazes* are the most widely used. The glaze is a blend of clays, ceramic frit, fluxes, and base metals sprayed on the units before firing, and then subjected to normal firing temperatures to fuse it to the clay body.

II.2.4.4 Firing

This is one of the most specialized and critical steps in the manufacture of clay products. Since many of the properties of brick and clay tile depend on the method and control of firing, the development over the years of more sophisticated kilns has been instrumental in improving the quality and durability of clay masonry. Firing consists essentially of subjecting brick units to gradually increasing temperatures until fusion chemically alters the structure of the clay. The firing process consists of six phases which are accomplished in the dryer kiln and in the preheating, firing, and cooling chambers of the firing kiln. The drying and evaporating of excess moisture are often called the water-smoking stage. This initial preheat may be done in separate dryers or, if high-fired glazes will not be added, in the forward section of the firing kiln. This exposure to relatively low temperatures of up to 380°C begins the gradual, controlled heating process. Dehydration, or removal of the remaining trapped moisture, requires anywhere from 280 to 1780°C, oxidation from 980 to 1780°C, and vitrification from 1580 to 2380°C. It is only within this final temperature range that the silicates in the clay

melt and fill the porosity between the more refractory materials binding and cementing them together to form a strong, dense, and hard-burned brick. The actual time and exact temperatures required throughout these phases vary according to the fusing characteristics and moisture content of the particular clay. Near the end of the vitrification phase, a reducing atmosphere may be created in which there is insufficient oxygen for complete combustion. This variation in the process is called flashing, and is intended to produce different hues and shadings from the natural clay colors. For example, if the clay has high iron oxide content, an oxygen-rich fire will produce a red brick. If the same clay is fired in a reducing atmosphere with low oxygen, the brick will be purpled. The final step in the firing of brick masonry is the cooling process. In a tunnel kiln, this normally requires up to 48 hours, as the temperatures must be reduced carefully and gradually to avoid cracking and checking of the brick.

II.2.4.5 Drawing and Storage

Removing brick from the kiln is called drawing. The loaded flatcars leave the cooling chamber and are placed in a holding area until the bricks reach room temperature.

II.2.5 Varieties of Forming Process

II.2.5.1 Soft Mud Process

The clay used in the soft mud process has water content in the range of 20-30%. It is so called because the processed clay has the appearance of a sloppy mud. The two variants of this process are hand moulding and machine moulding. Where the hand moulding technique is used, processed clay is thrown into a sand coated mould and the excess material is cut off with a wire. The characteristic folded appearance on the faces of the brick is caused by the dragging of the clay against the mould side as it is thrown. A skilled brick maker can produce up to 100 bricks per hour. Nowadays most production is by machine, which imitates the hand moulding process. The majority of bricks has a small frog (depression) formed by a raised central area on the bottom face of the mould. Due to the high water content of these bricks, they cannot be handled or stacked without deformation immediately after moulding. Also, because of the high drying shrinkage of

such wet mixes and the plasticity of the green brick, the size and shape of such units are fairly variable, and therefore, they are not suitable for use in thin mortar beds.

II.2.5.2 Extrusion Process

The stiff plastic process (sometimes called the extrusion process) involves grinding and mixing clay to produce material with a moisture content of 10-15%. This is extruded and cut into brick shaped pieces which are allowed to dry for a short period before being pressed into a die. The stiff clay retains its shape when ejected from the die. The relatively low moisture content indicates that shrinkage is low, and therefore, the size is easier to control. A further advantage of this process is that the drying time is relatively short. This type of unit will normally have at least one shallow frog. Engineering bricks, facing bricks, and other brick products with very accurate dimensions are normally produced by this process.

II.2.5.3 Wire Cut Process

The wire cut process involves using clay with moisture content in the range of 20-25%. A continuous ribbon of clay is extruded from the mixer through a rectangular die with a cross section the same as that of the required bricks. The ribbon of clay is then cut by a multiple wire-cutter into bricks. Cutting wires are set apart a distance depending on the height of the unit plus an allowance for shrinkage that takes place during drying and firing. The wire cut is made perpendicular to the face and ends of the brick. This type of production process produces a brick with no depressions in their bed faces. It also makes it relatively easy to include perforations along the length of the column by placing bars within the die head. Stains may be applied to the extruded clay to obtain a variety of colors.

II.2.5.4 Semi Dry Pressing Process

The semi dry pressing process involves placing clay with moisture content in the range of 17-20% in a press and applying hydraulic pressure to produce the required shape. The external appearance of the brick may be enhanced by techniques such as applying a surface layer of sand and firing the unit. Following the moulding stage, the

bricks are allowed to dry prior to being fired. The drying stage is important for two reasons: excessive shrinkage may cause cracking if not controlled, and preliminary drying reduces the overall energy requirement.

II.2.6 Size, Shape, and Color of Brick

Bricks are rectangular in shape but come in many different sizes. The easiest size to work with is called *modular* brick because its height and length are based on a 10 cm *module*. The measured dimensions of a masonry unit are called the *actual dimensions*, and the dimensions of a masonry unit plus one mortar joint are called the *nominal dimensions*.

Colors and textures vary depending on the clay and the methods used to form the brick. Reds, browns, tans, pinks, and buff colors are common. Brick manufacturers also sell color blends which combine light and dark shades, and more than one color of brick to create different effects. Brick comes in three types:

1-Architectural bricks are the most popular for residential and some small commercial construction because they often resemble old brick.

2-Standard bricks are more uniform. The dimensions do not vary as much from one brick to the next, the edges are sharper, and there are fewer and smaller chips and cracks.

3-Special bricks are more expensive precision bricks with tight limits on size variation, chips, and cracks. The edges are sharp and crisp, which gives them a very contemporary look. It is not very popular, even for commercial projects, and is not widely available (Beall, 2004).

II.2.7 Types of Bricks

Bricks may be broadly classified as:

1- Building bricks: These bricks are used for the construction of walls.

2-Paving bricks : These are vitrified bricks and are used as pavers.

3-Fire bricks : These bricks are specially made to withstand furnace temperature.

Silica bricks belong to this category.

4-Special bricks : These bricks are different from the commonly used building bricks with respect to their shape and the purpose for which they are made. Some of such bricks are listed below:

- Specially shaped bricks
- Facing bricks
- Perforated building bricks
- Burnt clay hollow bricks
- Sewer bricks
- Acid resistant bricks

II.2.8 Properties of Bricks

The following are the required properties of good bricks:

- Color: Color should be uniform and bright.
- Shape: Bricks should have plane faces. They should have sharp and true right angled corners.
- Size: Bricks should be of standard sizes as prescribed by codes.
- Texture: They should possess fine, dense, and uniform texture. They should not possess fissures, cavities, loose grit, and unfired lime.
- Soundness: When struck with hammer or with another brick, it should produce metallic sound.
- Hardness: Finger scratching should not produce any impression on the brick.
- Strength: Crushing strength of brick should not be less than 3.5 N/mm^2 . A field test for strength is that when dropped from a height of 0.9 m to 1.0 m on a hard ground, the brick should not break into pieces.
- Water Absorption: After immersing the brick in water for 24 hours, water absorption should not be more than 20% by weight. For some other types, this limit is 15 per cent.
- Efflorescence: Bricks should not show white patches when soaked in water for 24 hours and then allowed to dry in shade. White patches are due to the presence of sulphate of calcium, magnesium and potassium. They keep the masonry permanently in damp and wet conditions.

- **Thermal Conductivity:** Bricks should have low thermal conductivity, and thereby buildings built with them are cool in summer and warm in winter.
- **Sound Insulation:** Heavier bricks are poor insulators of sound while light weight and hollow bricks provide good sound insulation.
- **Fire Resistance:** Fire resistance of bricks is usually good. In fact, bricks are used to encase steel columns to protect them from fire.

II.2.9 Tests on Bricks

The following laboratory tests may be conducted on the bricks to find their suitability.

II.2.9.1 Compressive Strength

The compressive strength of the unit is the controlling factor. Bricks of various strengths are available to suit a wide range of architectural and engineering requirements.

II.2.9.2 Absorption Test

Brick specimens are weighed dry. Then, they are immersed in water for a period of 24 hours. The specimen is taken out and wiped with cloth. The weight of each specimen in wet condition is determined. The difference in weight indicates the water absorbed. The percentage absorption is the ratio of water absorbed to dry weight multiplied by 100. The average of specimens is taken. This value should not exceed 20 per cent.

II.2.9.3 Shape and Size

Bricks should be of standard sizes, and edges should be truly rectangular with sharp edges. To check it, some bricks are selected at random and they are stacked along the length, along the width, and then along the height. The following field tests help in ascertaining the good quality bricks:

- **Uniformity in Size:** A good brick should have rectangular plane surface and uniform in size. This check is made in the field by observation.

- **Uniformity in Color:** A good brick should have uniform color. This observation may be made before purchasing the brick.
- **Structure:** A few bricks may be broken in the field, and their cross-section may be observed. This section should be homogeneous, compact, and free from defects such as holes and lumps.
- **Hardness Test:** It is to scratch the brick with nail. If no impression is marked on the surface, the brick is sufficiently hard.
- **Sound Test:** If two bricks are struck with each other, they should produce clear ringing sound. The sound should not be dull.
- **Efflorescence:** The presence of alkaline in brick is not desirable because they form patches of grey powder by absorbing moisture. Hence, this test is performed in order to determine the presence of alkaline.

II.3 TYPES OF BINDERS

General characteristics of each binder used in the present study are introduced and explained in a detailed manner as follows.

II.3.1 Lime – Calcium Oxide (CaO)

Lime is an important binding material used in the construction sector. It has been used since ancient time. When it is mixed with sand, it provides lime mortar, and when mixed with sand and coarse aggregate, it forms lime concrete.

II.3.1.1 Types and Properties of Limes

The limes are classified into three types as follows:

1-Fat lime: It is composed of 95% of calcium oxide. When water is added, it slakes vigorously and its volume increases to 2 to 2.5 times. The followings are its properties:

- It hardens slowly
- It has high degree of plasticity
- It sets slowly in the presence of air
- It is white in color

- It slakes vigorously.

2-Hydraulic lime: It contains clay and ferrous oxide. Depending upon the percentage of clay present, the hydraulic lime is divided into the following three types:

- Free hydraulic lime (5 to 10% clay content)
- Moderately hydraulic lime (11 to 20% clay content)
- Eminently hydraulic lime (21 to 30% clay content)

Concerning its properties, the following features should be stressed:

- It sets under water.
- Its color is not perfectly white.
- It forms a thin paste with water and does not dissolve in water.
- Its binding property improves if its fine powder is mixed with sand and kept in the form of heap for a week, before using.

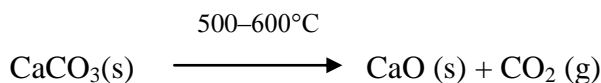
3-Poor lime: It contains more than 30% clay and has poor binding property. Its color is muddy. The mortar made with such lime is used for inferior works.

In general, limes are used in civil works for:

- white washing
- making mortar for masonry works and plastering
- producing lime sand bricks
- soil stabilization
- lining open hearth furnaces
- making cement.

II.3.1.2 Chemical Composition

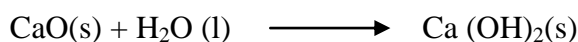
Calcium oxide is a white crystalline solid with a melting point of 2572°C. It is manufactured by heating limestone, coral, sea shells, or chalk, which are mainly CaCO₃, to drive off carbon dioxide.



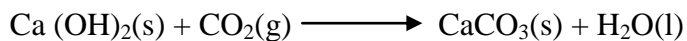
This reaction is reversible. Namely, calcium oxide will react with carbon dioxide to form calcium carbonate. The reaction is driven to the right by flushing carbon dioxide

from the mixture as it is released. Lime has many properties that make it quite valuable. It is so useful that it is today produced industrially on a vast scale.

When lime is mixed with water and sand, the result is mortar which is used in construction to secure bricks, blocks, and stones together. Mortar is initially a stiff paste that is laid between the bricks. It gradually hardens, cementing the bricks together. At room temperature, the reaction of lime with carbon dioxide is very slow. It is speeded by mixing lime with water. When lime is mixed with water, it forms calcium hydroxide, called slaked lime.



The reaction of calcium hydroxide with carbon dioxide is faster, producing a mortar that hardens more quickly.



Even with the increased reaction speed, mortar requires many years for complete reaction to occur. Perhaps the most commercially important property of lime is its ability to form solutions with silicates. When lime is heated with silica sand (SiO_2) and sodium carbonate (Na_2CO_3), a solution is formed, which does not crystallize when it is cooled. Instead, it hardens to an amorphous, clear, and nearly colorless solid, namely glass. Because it is a mixture and not a pure compound, glass does not have a distinct melting point. It gradually softens as it is heated. Therefore, it can be moulded and blown into many useful shapes.

II.3.2 Water Glass ($n\text{SiO}_2 \cdot \text{Na}_2\text{O}$)

Water glass is a common adhesive component used in the abrasive and casting industry and in other sectors. It is generally used as an alkali activator while composing materials, and recognized that alkali additions can activate pozzolanic materials to set and harden in their own right. Its main property, i.e. adhesive capacity, makes it possible to firmly bind ceramic components in a moulding mixture such as electro corundum

powder, powder components of ceramic binders for abrasive production, and organic additives like dextrin. The fluidity of soluble silicate plays an important role, as it provides uniform moistening and then gluing of moulding mixture components over the whole volume. Although the use of water glass possessing high fluidity and a decreased density provides rather uniform moistening of mixture components, the strength of moulded pre-forms in this case perceptibly decreases. The use of high density and lower fluidity water glass does not ensure a homogeneous distribution of the liquid silicate over the mixture volume, and does not achieve the required strength of moulded pre-forms (Polyakov and Polyokava, 2006).

II.3.3 Molasses

Molasses is a by- product of the sugar industry. It is the mother liquor remaining after removal of sucrose from the juice of sugar beet (Kavas et al, 2004). Molasses, a by-product from sugar industries, is not only cheap and eco-friendly material but can also be used as a binder for producing strong moulds. Limited work has been reported on molasses-bonded sand with calcium oxide and cement. For commercial exploitation of the molasses–cement system, it is essential to generate models for predicting the properties of molasses-bonded sand. There are a number of independent variables such as the percentage of binder and additive, setting time, temperature, etc., which influence the properties of the sand mix (Mandal and Roy, 2006).

II.3.4 CMC (Carboxymethyl Cellulose)

Sodium carboxymethyl cellulose (Na-CMC) is a type of cellulose which is ether synthesized by reacting alkali with sodium mono-chloroacetate under rigidly controlled conditions. Each unit contains three hydroxyl groups. The optimum water solubility as well as other desirable physical properties of CMC is achieved by a very low degree of substitution. When there is high degree of substitution, the CMC products improve compatibility with other soluble components. These cellulose ethers are long chain polymers. Their characteristics depend on average chain length and degree of substitution which in turn determines molecular weight of the polymer. The viscosity of

solution increases with the rise in molecular weight of the polymer. The viscosity is also affected by the degree of neutralization of carboxymethyl group (Vejani, 2009).

II.3.5 Borax-Boric Acid

Borates have a wide range of applications in various industries such as glass, ceramic, detergent, plastic, agriculture, and textile industries. Around 42% of the total world borate is used in glass sector, mainly in the production of borosilicate glasses, textile grade glass, and insulation grade glass fibers. Ceramics account for 11% of world borate consumption, where they are mainly used in glazes and enamel frits. One of the sources of boron in glass and ceramic industries is boric acid.

Boric acid (H_3BO_3) is produced industrially from borate minerals and brines. Alkali and alkaline earth metal borates such as borax, colemanite, ulexite, or kernite react with strong acids to form boric acid. Approximately 65% of the world borate reserves are located in Turkey where boric acid is obtained from colemanite ($2CaO.3B_2O_3.5H_2O$) (Mergen, 2002).

II.3.6 Humic Acid

Humic acid is a dark color material of which molecular weight changes between 50,000 and 100,000 g/mole. It can exist in decomposed organic matter, peat, coal deposits, and soil. Importantly, it contains high volume of carboxyl, hydroxyl, methoxyl, and oxygen made of carbonyl groups. Humic acid is not a fertilizer alone. Although the amount of nitrogen and sulphur content is very low, it is advertised as if being a fertilizer in markets. In reality, it provides only that micro nutrition substances are carried from soil to plant (Anonymous 1999). Therefore, both humic acid and fulvic acid are accepted as soil regulators (Özkan, 2008).

II.3.7 SLS (Sodium Lignosulfonate)

Lignosulfonate is a by-product in the pulping waste liquor from acid sulfite pulp mills. The unused industrial lignin is mostly burned in an energy recovery, but a large number of studies on lignin are focusing on finding alternative applications of higher value for lignin than fuel. Lignosulfonate contains both hydrophilic groups

(sulfonic, phenylic hydroxyl, and alcoholic hydroxyl) and hydrophobic groups (carbon chain).

Lignosulfonate has been used in various industrial processes. One of the largest applications for lignosulfonates is in the concrete admixture industry, where they are used as dispersing agents and set-retarding agents.

CHAPTER III

THE STUDY

III.1 BRICK PRODUCTION BY EXCAVATED SOIL

In this thesis, a brick-like construction material was produced with excavation soil and binders. The excavated soil used in this study was neither a specific type of soil nor obtained from a special area. The aim of this research was to reduce and minimize the energy consumption in the brick-making process by lowering the firing or heat treatment temperature and to recycle and convert excavation soil into a value added product. Moreover, reduction in firing energy consumption and duration was expected to provide lower cost of production. Lower energy consumption, temperature, and lower thermal treatment means relatively reduced gas emissions release to the atmosphere which could be resulted in less environmental pollution and less greenhouse gas effect. Therefore, environmentally friendly building material would also be intended to produce.

III.2.EXPERIMENTAL STUDIES

Experimental studies are composed of three main parts. These are: (i) characterization of raw material and selection of binders, (ii) preparation and curing of samples, and (iii) characterization of samples. Experimental parameters and all stages of experimental studies were shown in Fig. III.1.

III.3 CHARACTERIZATION OF RAW MATERIAL

III.3.1 Proposed Manufacturing Process of Clay Bricks

In the first step, as can be seen in Fig III.2, the excavated soil was obtained from a municipal area where the excavated soil of construction sites in Zonguldak region of Turkey is dumped.

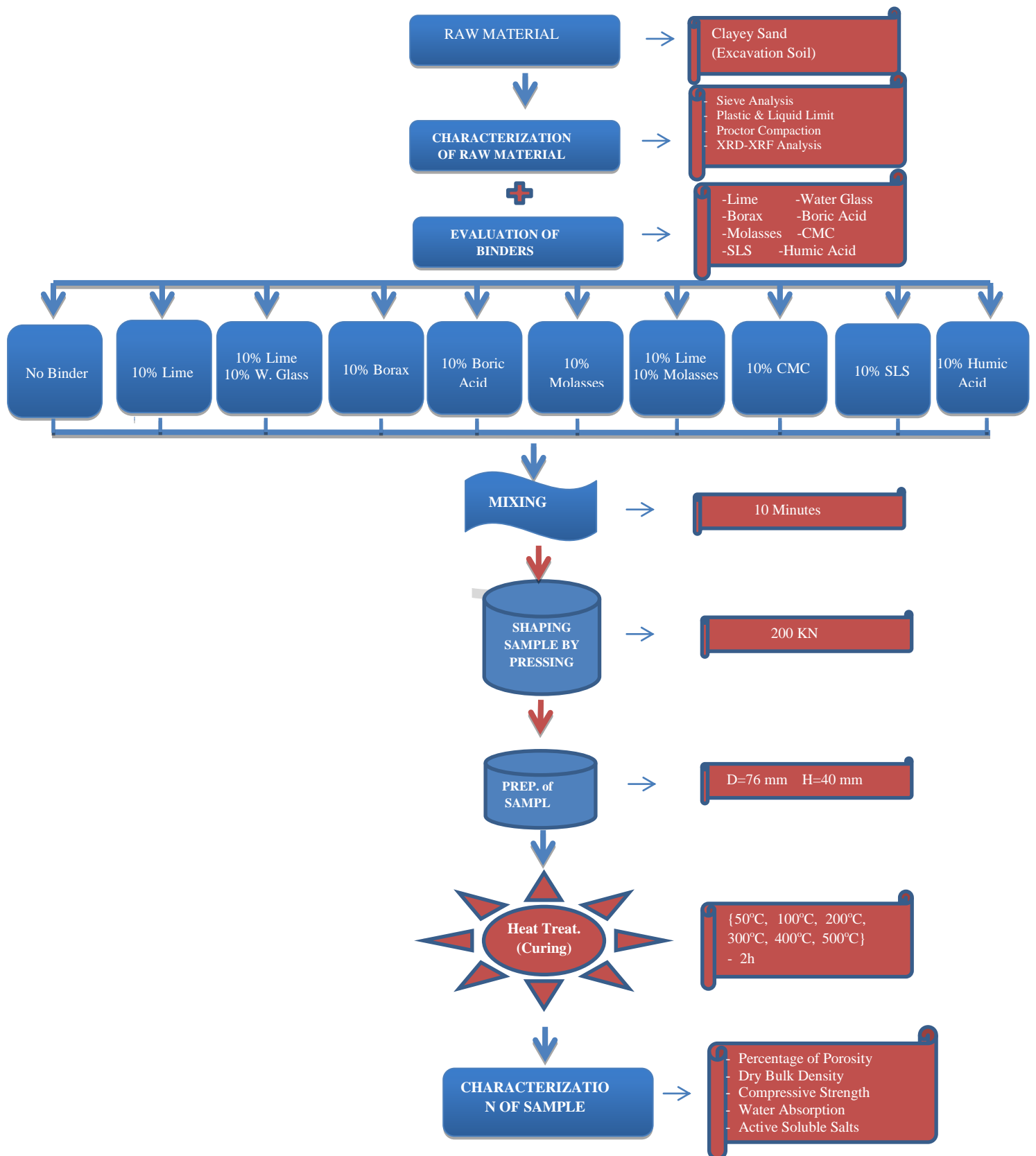


Figure III.1 Flow Chart of Experimental Studies

III.3.1.1. Sampling in the Bulk Soil

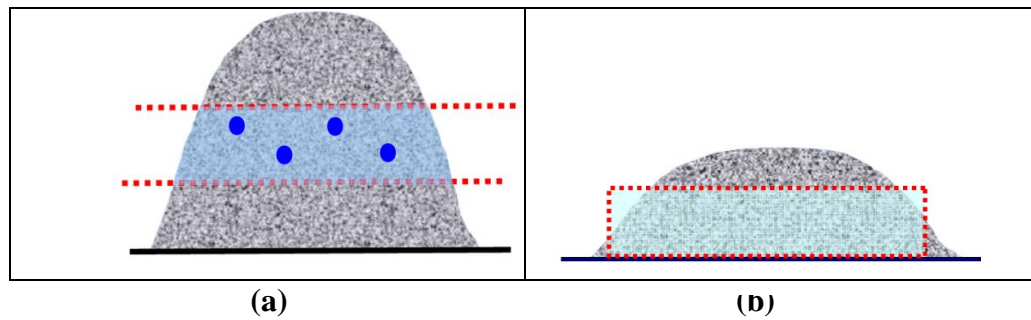


Figure III.2 Sampling in the Bulk Soil

Middle region of bulk soil was determined (Figure III.2/a). Then, 120 sampling attempts were made. Each one of them had 1 kg in weight. All of these samples were vacated on a smooth and clear surface, and possessed a circular shape. In other words, the total sampling material was mixed to make a homogenous composition by turning the entire sample over with a shovel three times. By the last turning, the entire sample was shoveled into a conical pile by depositing each shovelful on top of the preceding one. The conical pile had a uniform thickness and diameter. The pile diameter was approximately six times the thickness (Figure III.2/b)

III.3.1.2. Quartering Method

In order to reduce the total sampling size of excavation soil to testing size, quartering method was employed, which minimize variations in soil characteristics between the test sample and the bulk sample.



Figure III.3 Quartering Method

The flattened pile was divided into four equal quarters with the shovel (Figure III.3/a). Two diagonally opposite quarters were removed, including all fine material (Figure III.3/b). Spaces were brushed. The remaining material was successively mixed and quartered in the same manner as the testing sample. This process continued until the total sampling material was quartered.

III.3.2 Sieve Analysis (TS 130)

Before preparation of samples, some tests were experimented to characterize the excavation soil. The first one was the standard method of test for sieve analysis of fine and coarse aggregates.

In this experiment, a shaker and five test sieves of different sizes (4.75mm, 1.18mm, 450 μ m, 150 μ m, and tray) were used according to ASTM Standards (Figure III.4/a – III.4/b) with sieves by Han Kuk Jung of Korea and shaker by ÇMS Machine Industries.



Figure III.4 Sieves and Shaker

Before the soil was sieved, it was divided into a number of trays and dried at $105 \pm 5^{\circ}\text{C}$ for 24 h (Figure III.5/a). Then, big pieces of the dried soil were crushed by hammer to make the soil size smaller (Figure III.5/b).



Figure III.5 Material Before (A) and After (B) the Hammering Process

In total, soil material of 120 kg was sieved. In each sieving process, 1 kg of soil was vibrated 15 minutes at constant speed. After that, material passing through 4.75 mm sieve was used to prepare end samples since the dimension of soil should be harmonious with the gradation of binders and with the dimension of the mould. As a result, a total of 90 kg soil (76% of total material taken from the area) remained.

III.3.3 Determination of Liquid Limit (TS 1901)

For this experiment, mechanical liquid limit device (H-4230 by Humboldt) was used. Properties of this device and other apparatus were explained in Appendix 2.

In the first step, soil was mixed in a mixing dish. A representative portion was obtained from the total sample. It was sufficient to provide 200 g of material passing the 425- μm sieve. The portion of the prepared soil was placed in the cup of the liquid limit device at the point where the cup rest on the base was squeezed down and was spread by spatula into the cup up to the deepest point of about 10 mm.

The groove was formed by drawing the tool in the soil (beveled edge was forward) through the soil on a line joining the highest point to the lowest point on the rim of the cup (Figure III.6).

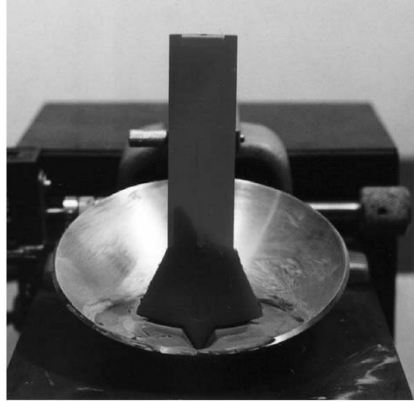
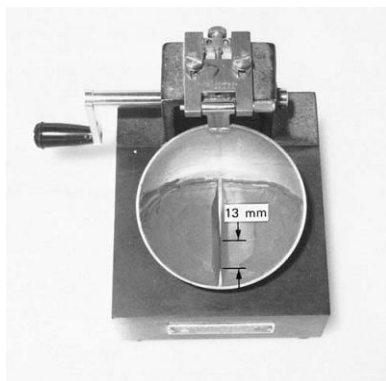


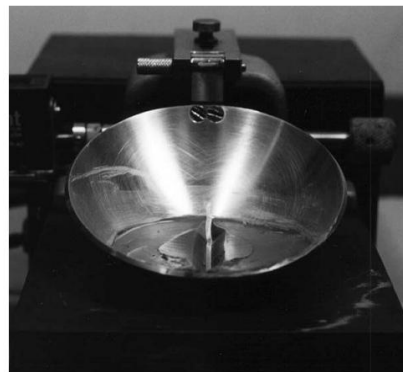
Figure III.6 Example of Grooving Tool Placed in a Properly Grooved Soil Pat

When cutting the groove, the grooving tool was held against the surface of the cup and was drawn in an arc, maintaining the tool perpendicular to the surface of the cup throughout its movement.

After formed the groove, the cup was lifted and dropped by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 13 mm.



(a)



(b)

Figure III.7 Soil Pat (a) Soil Pat after Groove Has Closed (b) Grooved Soil Pat in Liquid Limit Device

Consequently, number of drops was recorded, required to close the groove. A slice of soil was removed approximately the width of the spatula, extending from edge to edge of the soil cake at right angles to the groove and including that portion of the

groove in which the soil flowed together, was placed in a container of known mass, and was covered. The soil remaining in the cup was returned to the dish. The cup and grooving tool was washed and dried. The cup was reattached to the carriage in preparation for the next trial. The entire soil specimen in the dish was remixed adding distilled water to increase the water content of the soil and decrease the number of blows required to close the groove. (One of the trials shall be for a closure requiring 25 to 35 blows, one for closure between 20 and 30 blows, and one trial for a closure requiring 15 to 25 blows.) After, the water content of the soil specimen, W_n , was determined from each trial and initial masses of trial (container plus moist soil) were determined immediately after completion of the test.

III.3.4 Determination of Plastic Limit (TS 1901)

Portion (1.5 to 2.0 g) was selected from this plastic-limit specimen. The selected portion was formed into an ellipsoidal mass. The soil mass was rolled by one of the following methods (hand or rolling device). Hand method was used in this study.

Hand Method: The mass was rolled between the fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length. Its diameter was reached 3.2 mm by this method, taking no more than 2 min.

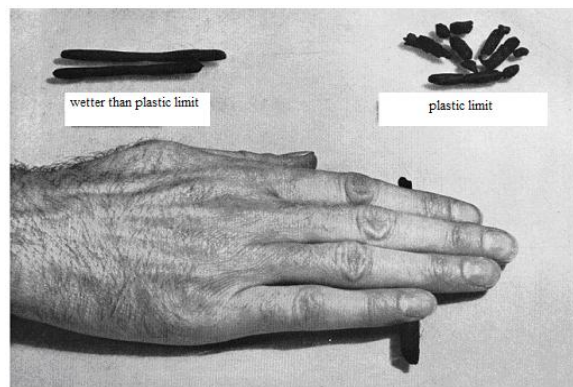


Figure III.8 Determination of Plastic Limit with Hand Method

When the diameter of the thread was become 3.2 mm, the thread was broken into several pieces. The pieces were squeezed together, were kneaded between the thumb and

first finger of each hand, were reformed into an ellipsoidal mass, and were re-rolled until the thread crumbles under the pressure required for rolling and the soil could no longer be rolled into a 3.2-mm diameter thread (Figure III.8). In this experiment, if crumbling occurs when the thread has a diameter greater than 3.2 mm, this shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread 3.2 mm in diameter. Lastly, clay soils was required much pressure to deform the thread, particularly as they approach the plastic limit (Figure III.9). With these soils, the thread broke into a series of barrel-shaped segments about 3.2 to 9.5 mm in length.

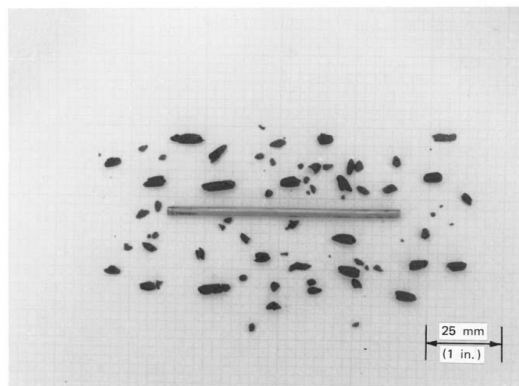


Figure III.9 Lean Clay Soil at the Plastic Limit (TS-1901, 2006)

The portions of the crumbled thread were gathered together and were placed in a container of known mass. Immediately the container was covered.

III.3.5 Proctor Test for Water Content of Soil

In this experiment, a 2.5 kg rammer falling 305 mm was used to compact the mixture in three layers into the Proctor mould. The Proctor mould with the base plate attached to 1 g was weighted and was recorded the mass as “ m_1 ”. The extension was attached to the mould and was placed the mould assembly on a solid base. The internal face of the extension was lubricated.

For one of the prepared samples, moist mixture was placed in the mould such that, when compacted, it occupied a little over one-third of the height of the mould body. And then, the 2.5 kg rammer that applying 25 blows was dropped from a height of 305 mm

above the mixture as controlled by the guide. The blows were distributed uniformly over the surface and were ensured that the rammer always falls freely.

The extension was removed, was struck off the excess mixture and was leveled off the surface of the compacted mixture carefully to the top of the mould using the straightedge. The materials and mould with the base plate to 1 g were weighted and were recorded the mass as “m₂”. The compacted mixture was removed from the mould and was placed it on the metal tray for determination of its water content.

Compaction test was carried out on each of the remaining prepared samples as described on above, to give a total of at least five determinations.

III.4 EVALUATION OF BINDERS

III.4.1 Binders Used in This Study

In this study, 8 different binders were used. While selecting binders, bonding materials that can be hardened in cold conditions were considered. Some of them were also waste products in particular. In the literature, just a few of these binders were employed in producing brick. At the same time, the selected binder was exposed to high temperatures and its effects on brick characteristics were investigated. Binder names and the related suppliers were given one by one in Table III.1.

Table III.1 Binder List

Binder No	Binder Name	Supplier
1	Water Glass	Filyos Refractory Brick Factory
2	Lime	Filyos Refractory Brick Factory
3	Sodium Ligno Sulfonate	As Chemistry Inc.
4	Borax	Eti Mine Inc. / Ankara
5	Boric Acid	Eti Mine Inc. / Ankara
6	Carboxy Methyl Cellulose	Motan Chemistry Inc.
7	Humic Acid	Kutsan Agriculture Inc. / Güpaş Fertilizer Production Inc.
8	Molasses	Erzurum Sugar Factory

III.4.2 Selection of Binder

The selection process of binder included 2 stages. In total, 10 mixture contents were created using 8 different binders to determine the most suitable mixture and to execute their characterization operations (see Table .III.2).

Table.III.2 Percentage of All Binders

Experiment No	Main Materials	Binder Materials	Percentage of Binder (%)	Percentage of Water (%)	Temperature of Cure 1 (°C)	Temperature of Cure 2 (°C)
1	Clayey Sand	-	-	5	50	105
2	Clayey Sand	Lime	10	5	50	105
3	Clayey Sand	Lime + W. Glass	Lime→10 W. G.→10	0	50	105
4	Clayey Sand	Borax	10	5	50	105
5	Clayey Sand	Boric Acid	10	5	50	105
6	Clayey Sand	Molasses (Syrup)	10	0	50	105
7	Clayey Sand	Lime + Molasses (Syrup)	Lime→10 Molasses (Syrup)→10	0	50	105
8	Clayey Sand	CMC	10	5	50	105
9	Clayey Sand	SLS	10	5	50	105
10	Clayey Sand	Humic Acid	10	5	50	105

For this objective, 3 trial samples were prepared for each of 10 mixtures. After they were cured at 2 different temperatures (50°C - 105°C), 5 mixtures that had the highest compressive strength values were chosen for continuation of the investigation (Table III.3).

Table III.3 Five Mixes and the Best Mix Selected

Experiment No	Main Materials	Binder Materials	Percentage of Binder (%)	Percentage of Water (%)	Temperature of Cure 1 (50 ° C)	Temperature of Cure 2 (105 ° C)	Temperature of Cure 3 (200 ° C)
1	Clayey Sand	-	-	5	50	105	200
2	Clayey Sand	Lime + Water Glass	Lime→10 Water Glass→10	0	50	105	200
3	Clayey Sand	Molasses (Syrup)	10	0	50	105	200
4	Clayey Sand	Lime + Molasses (Syrup)	Lime→10 Molasses (Syrup)→ 10	0	50	105	200
5	Clayey Sand	Humic Acid	10	5	50	105	200

Then, 3 trial samples were prepared for 5 mixtures selected at the first step. However, they were exposed to 3 different curing temperatures. These were 50°C, 105°C, and 200°C. Considering these three temperature values, the best mixture (Clayey sand + Lime + Molasses) that had the highest compressive strength was selected. The other experiments were also carried out on these samples.

III.5 MIXING PROCESS OF THE MATERIAL

For all experiment sets, a laboratory-type mixer (Kitchen Aid 5KSM150 Artisan) was used (Fig. III.10). It had 4 speed options (slow-middle-high-very high).

Middle option was selected and the mixing duration was fixed to 10 minutes. During this mixing process, liquid components such as water and molasses were sprayed on the mixtures in a homogeneous manner.



Figure III.10 Mixer

III.6 PREPARATION OF SAMPLE

III.6.1 Properties of Mould

The mould had 4 pieces which were made by cast iron. Since the soil material was pressed to the mould, thickness of the pieces was designed sufficiently big.

This 4-pieces disc section of the mould was because the mixture pressed could be easily put out without breaking (Fig. III.11/a). The diameter of disc section was 76 mm and the height was 40 mm. The second section of the mould was protective ring cover which kept the disc section together (Figure III.11/b). The third section prevented the mixture from spilling out of the mould before the pressing process (Figure III.12/a). In order to directly press the material, the fourth section was employed (Figure III.12/b).



(a)



(b)

Figure III.11 Mould (a) Section 1 (b) Section 2



(a)

(b)

Figure III.12 Mould (a) Section 3 (b) Section 4

III.6.2 Pressing

Seidner 7940 hydraulic pressing machine with maximum load capacity of 2000 kN was employed for shaping samples. It also had 10 speed options. For experiments in this study, mixtures were pressed under the load of 200 KN with a constant speed (5th option).



Figure III.13 Hydraulic Press

III.6.3 Dimensions of Samples

The diameter of the disc samples was 76 mm and the height was 40 mm. Therefore, compressive strength values were found using a correction coefficient in TS 771/1 standards.

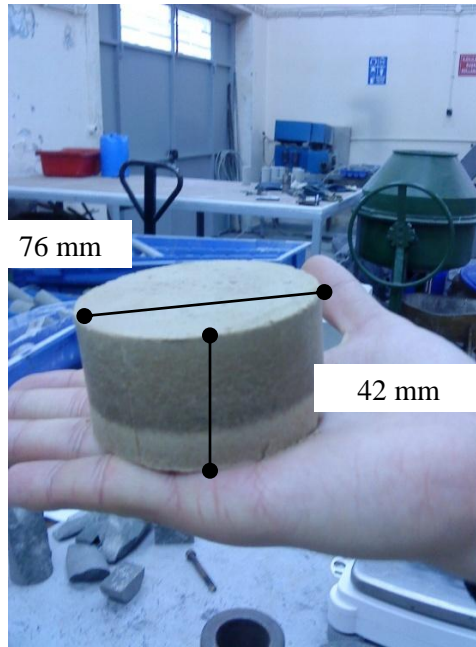


Figure III.14 Dimensions of the Samples

III.7 CHARACTERIZATION OF SAMPLES

To characterize the samples, some experiments were conducted. Their names and standards were given in Table III.4.

III.7.1 Net Volume and Percentage of Porosity (TS EN 772/3)

III.7.1.1 Apparatus

1. Tank, with adequate capacity to submerge a whole masonry unit.
2. Balances, readable to 0.1% of the compacted sample mass.

III.7.1.2 Procedure

The diameter (d_u) and height (h_u) of the specimen was measured by calipers. The specimen was immersed in water for at least 1 hour. The apparent masses (M_{wu}), was measured by two successive weighting's at 30 min intervals (If two weighing of specimens are not differ by less than 0.2%, the specimen can remove from the water). The result of the second weighing as the apparent mass (M_{wu}) was recorded. The surface water was removed with a damp towel and was determined the weight (M_{au}) immediately.

Table III.4 List and Standards of Experiments

NO	NAME OF EXPERIMENT	NUMBER OF SAMPLE	NAME OF SPECIFICATION	NUMBER OF STANDARTS
1	Methods of test for mortar for masonry units Part 3: Determination of net volume and percentage of porosity of clay masonry units by hydrostatic weighing	10	Determination of net volume and percentage of porosity of clay masonry units by hydrostatic weighing	TS EN 772-3
2	Methods of test for mortar for masonry Part10:Determination of dry bulk density of hardened mortar	10	Determination of dry bulk density of hardened mortar	TS EN 1015/10
3	Methods of test for mortar for masonry Part11:Determination of flexural and compressive strength of hardened mortar	10	Determination of flexural and compressive strength of hardened mortar	TS EN 772/1
4	Methods of test for mortar for masonry Part18: Determination of water absorption coefficient due to capillary action of hardened mortar	10	Determination of water absorption coefficient due to capillary action of hardened mortar	TS EN 771/1 App. C
5	Methods of test for masonry units Part5: Determination of the active soluble salts content of clay masonry units	10	Determination of the active soluble salts content of clay masonry units	TS EN 772/5

III.7.1.3 Calculations

The net volume of the specimen (V_{nu}) was calculated. Thus the mass of the specimen obtained by weighing it under water was subtracted from that obtained when weighing it in air ($M_{au} - M_{wu}$). The result was divided by the density of water (ρ_w). The net volume of the specimen was expressed to the nearest 10^4 mm^3 as

$$V_{nu} = (M_{au} - M_{wu}) / \rho_w \quad (\text{III.1})$$

The gross volume of the specimen (V_{gu}) was calculated by multiplying the diameter (d_u) and height (h_u) of the specimen measured to the nearest 10^4 mm^3 .

$$V_{gu} = d_u \times h_u \quad (\text{III.2})$$

The volume of porosity (V_{vu}) was calculated by

$$V_{vu} = V_{gu} - V_{nu} \quad (\text{III.3})$$

Calculate the percentage of porosity to the nearest 1 % by

$$(V_{vu} / V_{gu}) \times 100\% \quad (\text{III.4})$$

III.7.2 Dry Bulk Density (TS EN 1015/10)

III.7.2.1 Apparatus

1. Balances, readable to 0.1% of the compacted sample mass.
2. Drying Oven, thermostatically controlled, preferably of the forced-draft type, capable of continuously maintaining a temperature of $110 \pm 5^\circ\text{C}$ throughout the drying chamber.

III.7.2.2 Procedure

The specimen was dried in the oven at a temperature of $105 \pm 5^\circ\text{C}$ until the constant mass was reached. The specimen was considered to have reached constant mass

if two consecutive weightings 2 h. apart during the drying did not differ by more than 0.2% of the mass of the dry specimen. The mass was recorded of the specimen, $m_{s,dry}$ in kg to the nearest 0.1%.

III.7.2.3 Calculations

V_s is taken as the volume of the mould. The dry bulk density of each mortar specimen was calculated as the ratio of the recorded mass, $m_{s,dry}$ to the volume V_s . The mean value from the individual values of each mortar specimen was calculated, all values were rounded to the nearest 10 kg/m^3 .

III.7.3 Flexural and Compressive Strength (TS EN 772/1)

III.7.3.1 Apparatus

Hydraulic Press should provide the desired loading speed. The top loading head of the press should be adjustable according to the shape of sample. However, this head should not bend to any side during the loading process. It has ten different increasing loading speeds.

III.7.3.2 Procedure

Any slack material adhered to the surface of the sample touching the mould was moved. The press and loading heads were wiped with a clear cloth. Material was put into the mould without causing any overflow of the mixture and imbalance of the sample shape. The mould is then centrally put on the bottom tray of the press. The load is applied to the sample with a slowly increasing linear speed at second speed until a fracture was observed. The maximum load in the experiment was recorded, finally.

III.7.3.3 Calculations

Compressive strength was calculated by dividing the maximum load value to the loading area of the sample. For each sample, this value was rounded to the near 0.05 N/mm^2 . Average strength was found approaching the result by 0.1 N/mm^2 . Time for samples both during the experiment and at the time of putting out of the mould was recorded.

III.7.4 Water Absorption (TS EN 771/1 Annex-C)

III.7.4.1 Apparatus

1. Tank of water, of plan area depth enough to contain the specimens to be immersed and fitted with a means of maintaining a constant water level.
2. Stopwatch, graduated in seconds.
3. Weighing instrument, with an accuracy of 0.1% of the total, dry mass of the test specimen.
4. Ventilated oven, capable of maintaining a temperature of $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

III.7.4.2 Procedure

Ten specimens were tested for each cure temperatures. These were weighed and their dry mass, m_d , were recorded at their cure temperature. Constant mass shall be considered to have been reached if, during the drying process in subsequent weighting's with not less than a 24 h. interval, the loss in mass between the two determinations is less than 0.2% of the total mass. The specimens were allowed to cool to ambient temperature before they were weighed.

Each unit was placed in the tank of water at room temperature. That water is to be in contact with all faces of the unit and this may be achieved by resting the units on small pads which act as spacers. The units were left submerged for 24 h. The units were taken from the tank and were removed any surplus water from their surfaces using a damp cloth or sponge. The specimens were weighed and their wet mass, m_w , were recorded.

III.7.4.3 Calculation

The water absorption W_m of each of the units was calculated to the nearest 1%.

$$W_m = (m_w - m_d) / m_d \quad (\text{III.5})$$

III.7.5 Active Soluble Salts Content (TS EN 772/5)

III.7.5.1 Apparatus

1. Test sieves (150 μm test sieve), complying with the requirements of ASTM.

2. Polyethylene bottle or conical flask, typically with a capacity of 500 ml
3. Weighing instrument, readable to the nearest 0.1 mg
4. Burette, typically 10 ml
5. Pipette, typically 10 ml, 50 ml
6. Graduated flask, typically 1 l.
7. Volumetric flask, typically 100 ml
8. One-dimensional horizontal shaking equipment, capable of oscillating at $(120 \pm 5) \text{ min}^{-1}$, having a horizontal movement of 20 mm or a rotary shaker, capable of revolving at $30 \text{ min}^{-1} \pm 3 \text{ min}^{-1}$
9. Polyethylene bottle, typically with a capacity of between 1 l and 1.5 l.

III.7.5.2 Procedure

- Extraction procedure

20 g \pm 0.05 g of the sample was weighed and it was transferred to a 500 ml conical flask to using horizontal shaking equipment. 200 ml of distilled water at room temperature was added. The bottle was closed with a screw-on polyethylene top and it was shaken for 60 min. The suspended sample was filtered using an ash less blue ribbon filter paper.

- Determination of Na^+ , K^+ Ions with Atomic Absorption Spectroscopy Method (AAS)

50 ml aliquot of the soluble salts extract was put into a 100 ml volumetric flask and 4 ml of cesium chloride was added. It was filled to the mark with water and it was mixed.

Reference solutions of which Na^+ and K^+ concentrations are known was prepared. Of these solutions, spectroscopic lines were determined by using AAS. Finally, spectroscopic lines in the related sample were determined and thus the amount of concentration was found.

- Determination of Ca^{++} , Mg^{++} Ions with Alternative Method

- Preparation of EDTA test solution (0.5 %)

5 g of ethylene diamine tetra-acetic acid (EDTA) was dissolved in warm distilled water and it was stored in a polyethylene bottle.

- First titration (calcium + magnesium)

10 ml aliquot of the soluble salts extract was put into a 500 ml conical flask. 20 drops of hydrochloric acid was added, and then 10 ml of ammonia solution was added. It was diluted with distilled water to about 200 ml. 0,04 g of methyl blue complex one indicator, prepared by grinding together 0,2 g of methyl thymol blue complex one, with 20 g of potassium nitrate was added. Test solution was titrated with EDTA. When the color change was turned from blue into colorless, the volume titrated was measured, where the volume of EDTA use in the titration is denoted by y .

- Second titration (calcium)

10 ml aliquot of the soluble salts extract was put into a 500 ml conical flask. 20 drops of hydrochloric acid was added, and then 10 ml of potassium hydroxide solution was added. It was diluted with distilled water to about 200 ml. 0,015 g of calcium indicator, prepared by grinding together 0.1 g of calcium with 10 g of potassium chloride was added. Test solution was titrated with EDTA. When the color change was turned from fluorescent green to pink, the volume titrated was measured, where the volume of EDTA use in the titration is denoted by x .

III.7.5.3 Calculations

- Magnesium content

The mass of water soluble magnesium was calculated as a percentage of the mass of the original sample from the equation:

$$Mg = \frac{(y - x)M_{Mg}}{10} \text{ in percentage (\%)} \quad \text{(III.6)}$$

where y is the volume of the EDTA used in the first titration and x is the volume of EDTA used in the second titration.

- Calcium content
- It is calculated by using the equation “ $y - x$ ”.

CHAPTER IV

RESULTS AND DISCUSSIONS

IV.1 CHARACTERIZATION OF RAW MATERIAL (EXCAVATED SOIL)

IV.1.1 Sieve Analysis (TS 130)

Considering mould dimensions and previous studies, it was decided that the maximum sieve size was 1.18 mm. Sieve analysis was made to determine the material gradation. As can be seen from Table IV.1, the maximum diameter of aggregate (D_{max}) was 4.75 mm.

Table IV.1 Result of Sieve Analysis

SIEVE SIZE (mm)	INDIVIDUAL MASS RETAINED (g)	INDIVIDUAL PERCENT RETAINED (%)	CUMULATIVE PERCENT RETAINED (%)	CALCULATED PERCENT PASSING (%)
4.75 mm	0.00	0.00	0.00	100.00
1.18 mm	205.50	20.52	20.52	79.48
0.425 mm	220.90	22.06	42.58	57.42
0.150 mm	463.70	46.30	88.88	11.12
Tray	111.40	11.12	100.00	0.00

In terms of sieve analysis that decomposes fine and coarse aggregates, TS 130 suggests sieve space of 4.00 mm while ASTM suggests 4.75 mm. As can be seen from Fig.IV.1, the whole material was accepted as fine aggregate since it passed through sieve openings of 4.75 mm.

Fine material was an advantage for mould size and binder gradation. It was about 79.48% of the total material in weight, or approximately 95 kg.

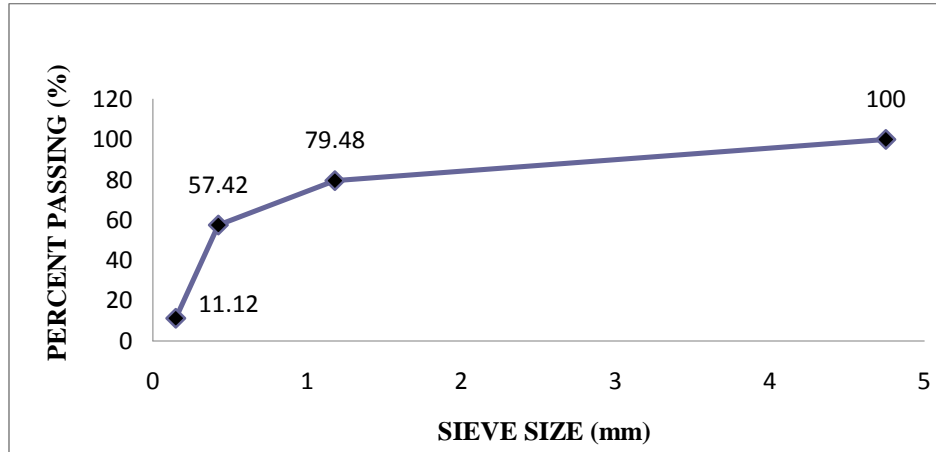


Figure IV.1 Curve of Sieve Analysis

IV.1.2 Determination of Liquid Limit (TS1901)

Liquid limit is the water content in which the soil changes its viscous fluid feature into plastic consistency. In Table IV.2, it was seen that the liquid limit value of the material was found out as 26.4%. In order to find the material structure, the plasticity index and plastic limit value were also needed.

Table IV.2 Result of Liquid Limit Analysis

	FIVE POINT				
	1	2	3	4	5
CONTAINER NO	K 1	K 24	K 6	K 8	K 10
NUMBER OF BLOWS	8	17	21	22	37
MASS MOIST SOIL + CONTAINER (g)	35.41	37.32	44.97	45.63	44.91
MASS DRY SOIL + CONTAINER (g)	32.33	34.25	40.30	40.98	40.71
MASS WATER (g)	3.08	3.07	4.67	4.65	4.20
CONTAINER (g)	22.01	22.80	22.47	22.35	22.58
MASS DRY SOIL (g)	10.32	11.45	17.83	18.63	18.13
WATER CONTENT (%)	29.9	26.8	26.2	25.0	23.2
LIQUID LIMIT (%)	26.4				

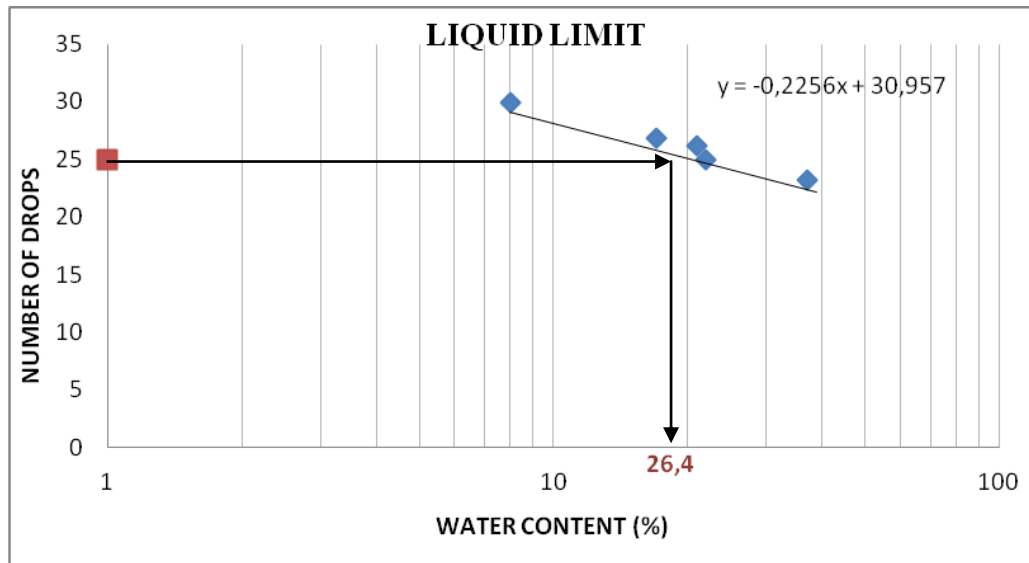


Figure IV.2 Determination of Liquid Limit Analysis

IV.1.3 Determination of Plastic Limit (TS 1901)

Plastic limit is the water content in which the soil changes its plastic feature into half plastic. Experimentally, it was determined as 15.5%.

Table IV.3 Result of Plastic Limit Analysis

PLASTIC LIMIT	CONTAINER NO	23	25	26
	MASS CONTAINER (g)	38.33	40.24	34.31
	MASS MOIST SOIL + CONTAINER (g)	69.77	72.64	64.87
	MASS DRY SOIL + CONTAINER (g)	65.44	68.27	60.88
	MASS WATER (g)	4.32	4.37	3.98
	MASS DRY SOIL (g)	27.11	28.03	26.57
	WATER CONTENT (%)	0.16	0.15	0.15
	PLASTIC LIMIT (%)	15.5		

The experiments also gave some ideas concerning optimum water content that should be used in main experiments. As can be seen from Table IV.3, it is evident that the soil loses its plastic consistency when the water content is more than 15.5%.

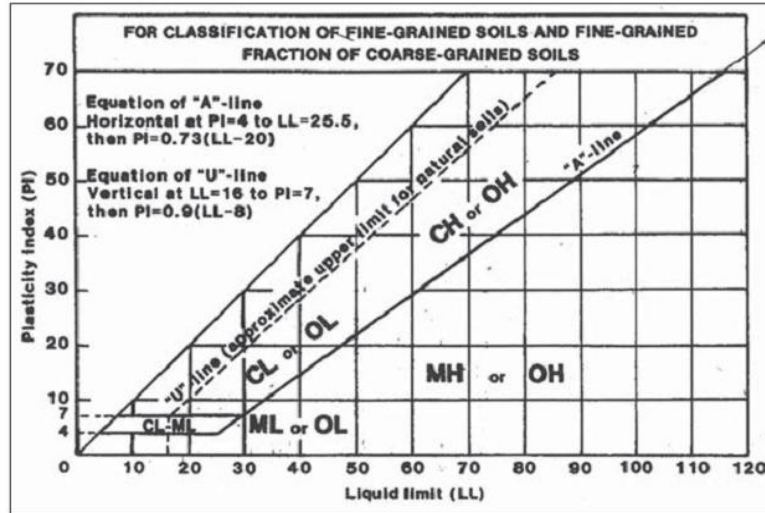


Figure IV.3 Casagrande Plasticity Card

In the Casagrande Plasticity Card,

CL: Low-plasticity inorganic clays or silted clays

ML: Low-plasticity inorganic silts or clayed silts

Plasticity index of the soil: $I_p = W_L - W_P$ $\Rightarrow I_p = 26.4 - 15.5 = 10.9$

As a result of the experiments, material was defined as low plasticity clay (CL) in Casagrande plasticity card (Figure IV.3).

IV.1.4 Proctor Test for Water Content of Soil

This experiment was realized by quantities in Table IV.4 using standard compaction device.

Table IV.4 Quantities of Compaction Test

COMPACTION TEST		1	2	3	4	5
WEIGHT OF MOLD + MOIST SOIL	(g)	3522.5	3627.4	3719.7	3772.2	3712.3
WEIGHT OF MOLD	(g)	1748.5	1748.5	1748.5	1748.5	1748.5
WEIGHT OF MOIST SOIL	(g)	1774	1878.9	1971.2	2023.7	1963.8
MOIST WEIGHT UNIT	(t/m ³)	1.88	1.99	2.09	2.15	2.08

At the end of the experiment, a graphic including dry unit volume weight and moisture content was drawn and optimum water content value was read.

Table IV.5 Result of Moisture Content

MOISTURE CONTENT W_N , %						
CONTAINER NUMBER	UNIT	14	16	23	12	9
MASS OF MOIST SOIL + CONTAINER	(g)	112.3	91.4	95.4	101.9	145.4
MASS OF DRY SOIL + CONTAINER	(g)	110.2	89.1	90.2	95.9	130.8
CONTAINER	(g)	48.5	56.4	38.4	48.9	42.9
MOISTURE CONTENT	(%)	3.4	7.0	10.0	12.8	16.6
DRY UNIT WEIGHT OF COMPACTION	(t/m³)	1.82	1.86	1.90	1.90	1.79
OPTIMUM MOISTURE CONTENT	(%)	11.57				
MAXIMUM DRY UNIT WEIGHT	(t/m³)	1.91				
SPECIFIC GRAVITY OF SOIL SOLIDS		2.68				

As a result of standard proctor compaction test, it was determined that optimum water content of the soil was about 12% (Fig.IV.4).

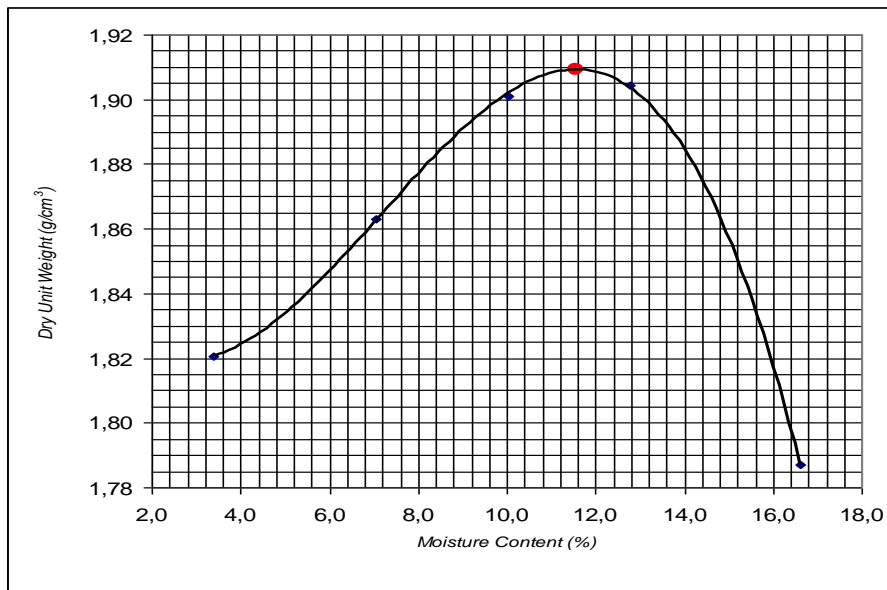


Figure IV.4 Curve of Moisture Content

The water content of plastic limit experiment was carried out before, which was found as 15%. Considering the results of these two experiments and past articles, it was decided that water content value in main experiments was 5% of the total material weight. As known, the lower the water content, the lower the porosity of the material. Thus, a sample that has much lower porosity and much higher strength can be obtained while pressed in the mould.

IV.1.5 XRD Analysis

According to the mineralogical analysis carried out by XRD, the content of excavation soil material was shown in Fig.IV.5.

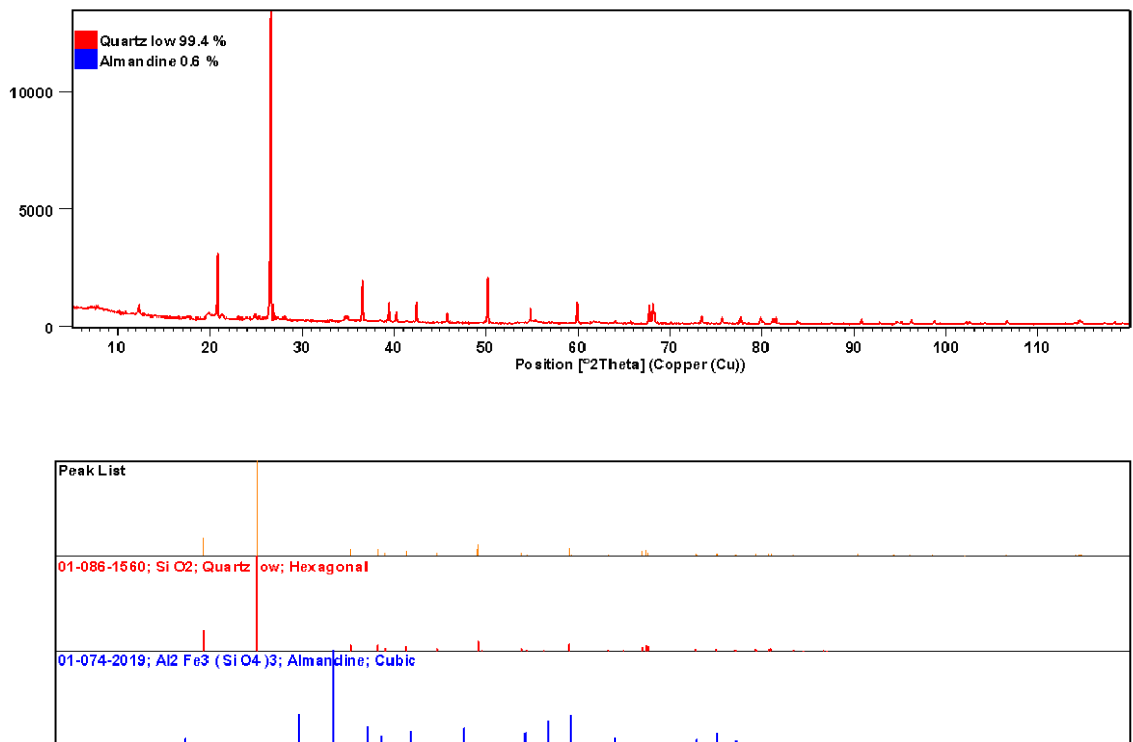


Figure IV.5 Result of XRD Analysis

In Fig.IV.5, especially two materials had maximum values. These were “quartz” [SiO₂] and “almandine” [Al₂Fe₃(SiO₄)₃], in a descending order.

IV.1.6 XRF Analysis

Chemical analysis of sample was made by investigating characteristic x-rays obtained from samples which were subjected to the concentrated x-rays. Milled sample was analyzed by Rigako RIX 2000 XRF equipment. All results of this experiment are following in Table IV.6. In some applications, the sample can be subjected to intensive protons or high-powered electrons instead of x-rays.

As a result of XRF analysis of the samples in this study, when they were compared to the oxide amount in a standard brick soil, it was understood that the chosen excavation soil possess an appropriate chemical analysis for brick production (Table IV.7).

Table IV.6 Result of XRF Analysis (%)

Cr₂O₃	Fe₂O₃	MnO	NiO	CaO	MgO
0.0366	8.5	0.186	0.02	1.22	0.589
Al₂O₃	Na₂O	P₂O₅	SiO₂	K₂O	TiO₂
18.3	0.0937	0.123	65.9	3.54	1.26

Table IV.7 Comparison between chemical structure of standard brick soil and the soil used in this study

OXIDE	MAX.(%)	MIN.(%)	SOIL USED (%)
SiO₂	64.5	42.7	65.9
TiO	1.2	1.0	1.3
Al₂O₃	20.6	16.3	18.3
Fe₂O₃	2.8	7.0	8.5
CaO	0.7	9.5	1.2
MgO	0.8	6.2	0.6
K₂O	1.7	3.6	3.5
Na₂O	0.1	0.8	0.1
SO₃	0.3	-	-
CaO₂	1.0	5.9	-
MnO +NiO+P₂O₅	6.3	11.5	0.3

IV.2 SELECTION OF BINDER

In this study, experiment sets were prepared by 8 binders and 10 different mixture contents. 3 samples were used for each set and each curing temperature. To determine the most suitable binder, two-stage pre-experiments were put into practice, considering compressive strength criterion. In the first stage of these experiments, compressive strength values were tested on samples produced with different binders and with two curing temperatures in laboratory conditions. Then, the second stage of pre-experiments was carried out in different curing temperatures by using five experiment sets that had the highest strengths, and thereby the concluding binder and experiment set were decided. These sets and their results were shown in Table (IV.8).

Table IV.8 Pre-experiment 1 for the binder selection

EXPERIMENT NO	MAIN MATERIALS	BINDER MATERIALS	BINDER ADDITION (%)	WATER ADDITION (%)	TEMPERATURE OF CURE 1 (°C)	TEMPERATURE OF CURE 2 (°C)	COMPRESSIVE STRENGTH OF CURE 1 (KN)	COMPRESSIVE STRENGTH OF CURE 2 (KN)
1	CLAYEY SAND	-	-	5	50	105	61	52
2	CLAYEY SAND	LIME	10	5	50	105	71	58
3	CLAYEY SAND	LIME + WATER GLASS	LIME→10 WATER GLASS→10	0	50	105	50	55
4	CLAYEY SAND	BORAX	10	5	50	105	53	45
5	CLAYEY SAND	BORIC ACID	10	5	50	105	48	35
6	CLAYEY SAND	MOLASSES (SYRUP)	10	0	50	105	25	40
7	CLAYEY SAND	LIME + MOLASSES (SYRUP)	LIME→10 MOLASSES (SYRUP)→10	0	50	105	25	65
8	CLAYEY SAND	CMC	10	5	50	105	22	28
9	CLAYEY SAND	SLS	10	5	50	105	5	15
10	CLAYEY SAND	HUMIC ACID	10	5	50	105	35	40

In this pre-experiments 1; percentage of binders was 10 % of main materials weight and percentage of water was 5 % of total materials (main materials + binder) weight. All the samples were cured for two hours. During the preparation of samples, all

timing values are the same each other. After the prepared samples, waiting time for breaking was the same each for a sample. In five and sixth experiments, 100gr syrup was solved by 100gr water. Solution addition to in the sample was 10 % of main materials weight. For the five sets chosen at the end of the first stage, experiments were repeated at the temperatures 50°C, 105°C, and 200°C (Table IV.9).

Table IV.9 Pre-Experiment 2 for the Binder Selection

EXPERIMENT NO	MAIN MATERIALS	BINDER MATERIALS	BINDER (%)	WATER (%)	TEMPERATURE OF CURE 1 (°C)	TEMPERATURE OF CURE 2 (°C)	TEMPERATURE OF CURE 3 (°C)	COMPRESSIVE STRENGTH OF CURE 1 (KN)	COMPRESSIVE STRENGTH OF CURE 2 (KN)	COMPRESSIVE STRENGTH OF CURE 3 (KN)
1	CLAYEY SAND	-	-	5	50	105	200	61	52	100
2	CLAYEY SAND	LIME + WATER GLASS	LIME→10 WATER GLASS→10	0	50	105	200	50	55	95
3	CLAYEY SAND	MOLASSES (SYRUP)	10	0	550	105	200	25	40	120
4	CLAYEY SAND	LIME + MOLASSES (SYRUP)	LIME→10 MOLASSES (SYRUP)→10	0	50	105	200	25	65	130
5	CLAYEY SAND	HUMIC ACID	10	5	50	105	200	35	40	80

In this pre-experiments 2; percentage of binders was 10 % of main materials weight and percentage of water was 5 % of total materials (main materials + binder) weight. All the samples were heat treated for two hours each for curing of binder or binder mixture. During the preparation of samples, all timing values are the same each other. After the prepared samples, waiting time to breaking is the same each for a sample. In five and sixth experiments, 100gr syrup is solved by 100gr water. Solution addition to the sample was 10 % of main materials weight. As a result of pre-experiments, the most suitable set would include “**clayey sand + lime (10wt %) + molasses (10wt %) (syrup)**”.

IV.3 CHARACTERIZATION OF SAMPLES

On the experiment set selected (clayey sand + lime + molasses), some tests were performed to find six different characteristics. Their names, required sample numbers, and related standards were given in Table III.4 in Chapter 3.

IV.3.1 Net Volume and Porosity Percentage (TS EN 772/3)

In this experiment, 4 curing temperatures were employed. These were 200°C, 300°C, 400°C, and 500°C. For each temperature, 10 samples were produced. Samples that were cured in temperatures less than 200°C dissolved in the water. Therefore, these relatively low temperatures were not taken into account. In the following tables (Table IV.10a, b, c, d), some values concerning average net volume, and volume and percentage of porosities were found for each one of curing temperatures.

Table IV.10 Net Volume and Porosities at Different Curing Temperatures

TEMPERATURE	200 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
NET VOLUME (cm ³)	170.0	165.1	161.8	166.1	164.0	169.6	167.4	166.2	164.5	163.1	165.8
POROSITY VOLUME (cm ³)	11.5	16.4	19.7	15.4	17.5	11.9	14.1	15.3	17.0	18.4	15.7
POROSITY (%)	6.3	9.0	10.9	8.5	9.6	6.6	7.8	8.4	9.4	10.1	8.7

(a)

TEMPERATURE	300 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
NET VOLUME (cm ³)	162.0	164.9	163.3	163.8	164.1	162.8	163.5	164.3	163.9	163.2	163.6
POROSITY VOLUME (cm ³)	9.5	6.6	8.2	7.7	7.4	8.7	8.0	7.2	7.6	8.3	17.9
POROSITY (%)	10.7	9.1	10.0	9.8	9.6	10.3	9.9	9.5	9.7	10.1	9.9

(b)

TEMPERATURE	400 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
NET VOLUME (cm ³)	163.3	156.6	156.8	157.4	156.8	158.2	157.6	156.9	159.3	158.7	158.2
POROSITY VOLUME (cm ³)	18.2	24.9	24.7	24.1	24.7	22.3	23.9	24.6	22.2	22.8	23.3
POROSITY (%)	10.0	13.7	13.6	13.3	13.6	12.8	13.2	13.6	12.2	12.6	12.9

(c)

TEMPERATURE	500 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
NET VOLUME (cm ³)	137.9	138.5	138.5	138.1	137.9	138.6	138.3	137.8	137.1	138.4	138.1
POROSITY VOLUME (cm ³)	43.6	43.0	43.0	43.4	43.6	42.9	43.2	43.7	44.4	43.1	43.4
POROSITY (%)	24.0	23.7	23.7	23.9	24.0	23.6	23.8	24.1	24.5	23.7	23.9

(d)

Considering temperature-porosity percentage and temperature-net volume interdependencies, it was seen that net volume and porosity percentage become in inverse proportion at higher temperatures. In Fig. IV.6, the higher the temperature is, the larger the porosity percentage exists. However, as shown in Fig. IV.7, the higher the temperature is, the smaller the net volume becomes.

Since the maximum temperature at which the curing conducted was 500 °C and dehydration continued, the crystal water in the samples went on being released. As the water was released, porosity volume grew and thus net total volume decreased. As can be seen from Figs.IV.6 and IV.7, the slope of curves dramatically increased between 400-500°C in particular.

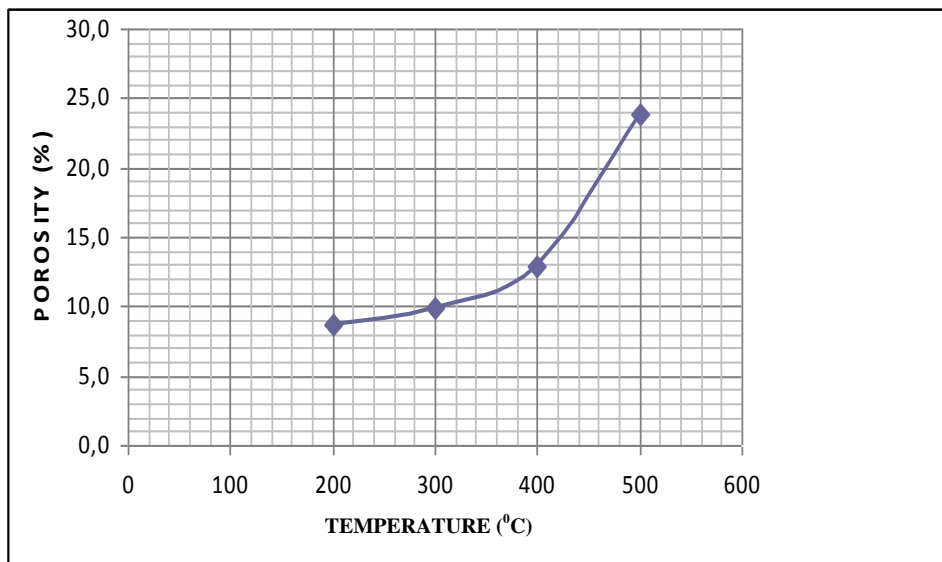


Figure IV.6 Porosity Percentage-Temperature Change

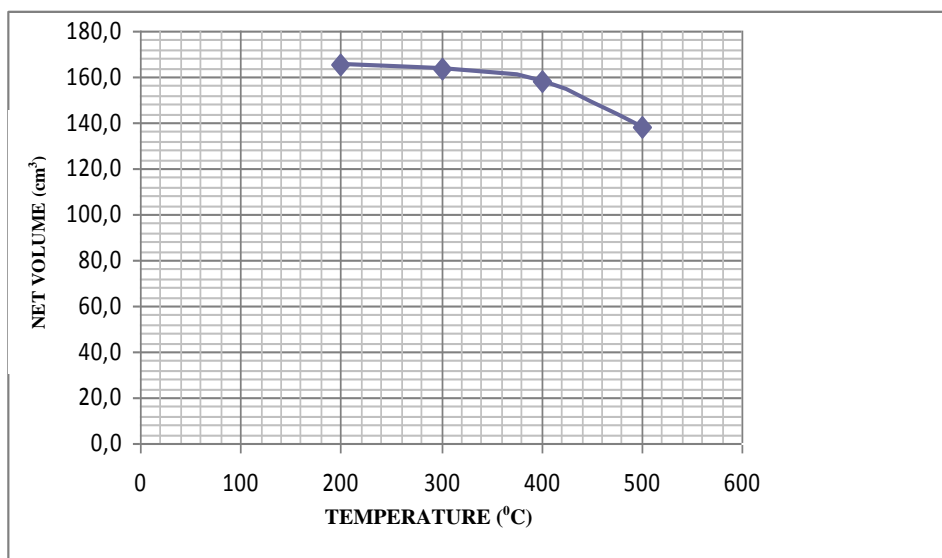


Figure IV.7 Net Volume-Temperature Change

IV.3.2 Dry Unit Volume Weights (TS EN 1015/10)

When the results obtained were compared with the TS EN 771/1 standard, it was found that brick samples produced in this study could be classified as high-density bricks. Their dry unit volume weights in the Standard are given in Table IV.11.

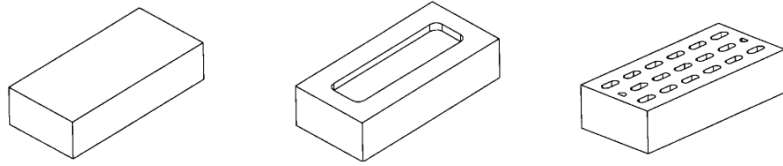


Figure IV.8 Varieties of Solid Brick in TS EN 771/1

Table IV.11 Brick Types and Dry Unit Volume Weights in TS EN 771/1

BRICK TYPE	UNIT VOLUME MASS (Kg/m ³)	
	MAX.	MIN.
2000 kg/m ³	2000	1801
1800 kg/m ³	1800	1601
1600 kg/m ³	1600	1401
1400 kg/m ³	1400	1201
1200 kg/m ³	1200	1001

Table IV.12 Average Dry Unit Volume Weight Values at Different Temperatures

TEMPERATURE	200 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
DRY UNIT VOLUME WEIGHTS (g /cm ³)	2.053	2.078	2.118	2.096	2.126	2.077	2.090	2.081	2.110	2.129	2.096

TEMPERATURE	300 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
DRY UNIT VOLUME WEIGHTS (g /cm ³)	2.113	2.069	2.066	2.070	2.082	2.103	2.101	2.068	2.068	2.060	2.080

TEMPERATURE	400 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
DRY UNIT VOLUME WEIGHTS (g /cm ³)	2.077	2.049	2.030	2.028	2.028	2.048	2.045	2.029	2.042	2.023	2.040

TEMPERATURE	500 °C										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
DRY UNIT VOLUME WEIGHTS (g/cm ³)	2.014	1.997	1.995	1.991	1.997	2.003	2.007	2.023	2.019	2.026	2.007

Up to 500 °C, dry unit volume weights of the samples were different from each other in the second digit of the percentage only. When considering temperature-dry bulk density relationship, it was seen that dry bulk density changes with temperature to little extent (Fig. IV.9).

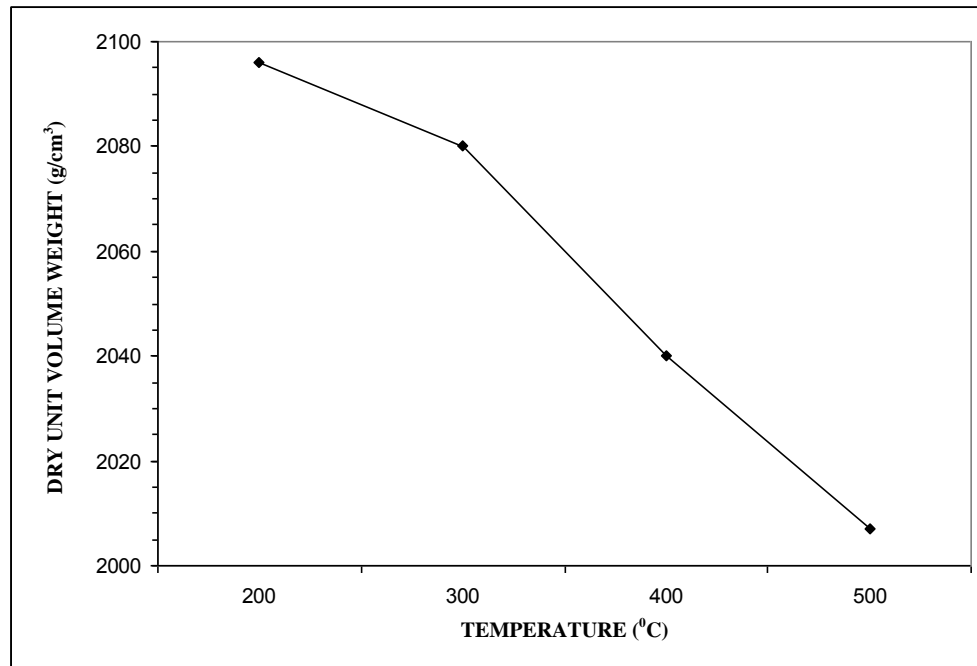


Figure IV.9 Dry Unit Volume Weight -Temperature Change

In terms of TS EN 772/1, the samples produced could be classified as the solid brick of 2000 kg/m³. As shown in Table IV.12, between the temperatures 200 and 500 °C, dry unit volume weight changed between the values 2096 and 2007 kg/m³.

IV.3.3 Compressive Strength (TS EN 772/1)

Compressive strength experiment was performed by 10 samples for each curing temperature. Here, the relationship between temperature and compressive strength of

samples was observed. For this objective, 6 curing temperatures were used. In Table IV.13, average values of maximum fracture loads were denoted for each temperature as a whole.

Table IV.13 Results of Compressive Strength at Difference Temperatures

TEMPERATURE	50°C		TEMPERATURE	100°C		TEMPERATURE	200°C	
SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)	SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)	SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)
1	4534	59.0	1	4534	76.3	1	4534	116.7
2	4534	46.5	2	4534	73.2	2	4534	107.0
3	4534	48.2	3	4534	71.1	3	4534	116.2
4	4534	57.8	4	4534	70.9	4	4534	112.4
5	4534	55.7	5	4534	73.6	5	4534	118.6
6	4534	51.2	6	4534	78.1	6	4534	116.8
7	4534	54.8	7	4534	75.4	7	4534	115.8
8	4534	53.9	8	4534	76.2	8	4534	117.5
9	4534	52.8	9	4534	78.2	9	4534	118.2
10	4534	51.9	10	4534	73.5	10	4534	116.8
Average	392.4	53.2	Average	382.5	74.6	Average	370.3	115.6

TEMPERATURE	300°C		TEMPERATURE	400°C		TEMPERATURE	500°C	
SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)	SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)	SAMPLE NUMBER	AREA (mm ²)	BREAKING LOAD (KN)
1	4534	112.0	1	4534	102.6	1	4534	88.7
2	4534	115.1	2	4534	106.7	2	4534	80.4
3	4534	110.2	3	4534	105.4	3	4534	104.6
4	4534	96.1	4	4534	113.4	4	4534	110.7
5	4534	114.4	5	4534	108.7	5	4534	110.8
6	4534	98.1	6	4534	105.4	6	4534	133.2
7	4534	100.5	7	4534	106.2	7	4534	110.2
8	4534	111.2	8	4534	104.3	8	4534	105.6
9	4534	113.5	9	4534	102.6	9	4534	104.2
10	4534	115.8	10	4534	101.8	10	4534	108.1
Average	365.8	108.7	Average	364.0	105.7	Average	360.9	105.7

Compressive strength value was calculated via fracture load divided by the loading area. For instance, at 50⁰C, fracture load was 53.2 KN and the loading area was 4534 mm² with the diameter of 76 mm. As a result, compressive strength was obtained in MPa as follows: $53.2 / 4534 = 11.73$ MPa.

These outcomes were multiplied by two coefficients in order to convert them to standard compressive strength values given in TS EN 772/1. The first one of these

coefficients was used for changing samples dried in furnace to those in air, and given as 0.8 in the Standard. The second was for transforming shape of samples in present experiments into a standardized shape. It was read as 0.75 (TS-772/1, 2002). For all temperature values, the results were calculated by applying the same standardization process. Compressive strength values were presented in Table IV.14.

Table IV.14 Results of Compressive Strength Test

TEMPERATURE (°C)	COMPRESSIVE STRENGTH (MPa)
50	7.02
100	9.9
200	15.3
300	14.4
400	13.9
500	13.9

Taking into consideration the change of compressive strength with temperature, maximum strength value was obtained at 200°C (Fig. IV.10).

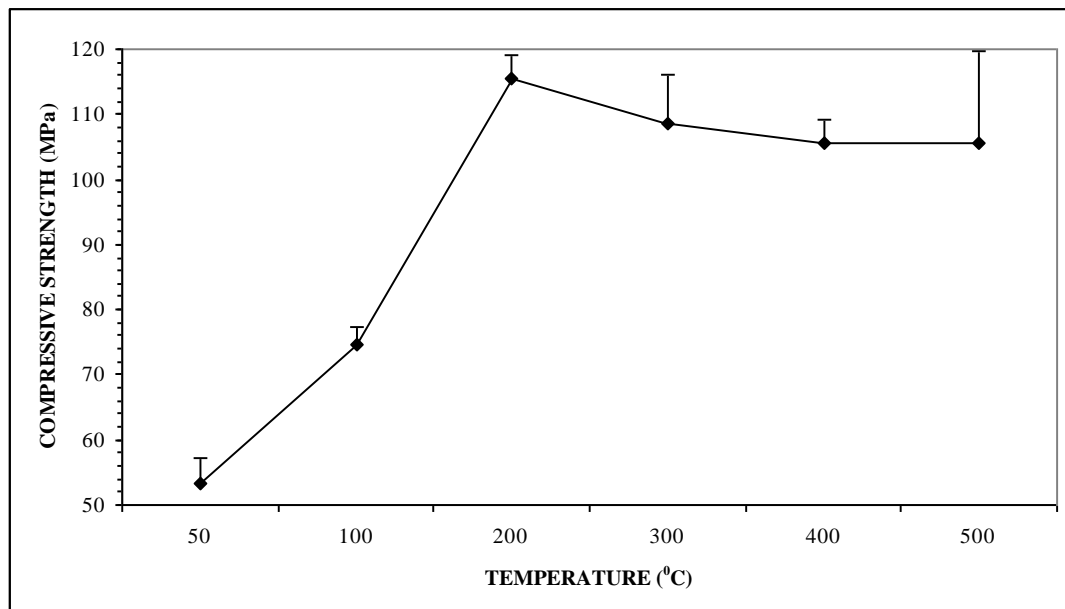


Fig.IV.10 Compressive Strength –Temperature Change for Clayey Sand + Lime (10wt %) + Molasses (10wt %) Containing Samples

IV.3.4 Water Absorption (TS EN 771/1 Annex-C)

In the experiment, 10 samples were employed for each temperature and their corresponding average water absorption values were obtained. All of the data were given in Table IV.15.

Table IV.15 Results of Water Absorption at Different Temperatures

TEMPERATURE	200 (°C)										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
WATER ABSORPTION (%)	4.5	5.4	4.6	4.3	4.3	3.9	4.1	4.6	4.4	4.4	4.4

TEMPERATURE	300 (°C)										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
WATER ABSORPTION (%)	5.9	4.9	4.9	5.0	5.4	4.7	4.7	4.6	5.1	4.6	5.0

TEMPERATURE	400 (°C)										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
WATER ABSORPTION (%)	5.0	6.8	6.6	6.7	7.2	6.0	7.1	8.2	8.4	7.9	7.0

TEMPERATURE	500 (°C)										
SAMPLE NUMBER	1	2	3	4	5	6	7	8	9	10	Average
WATER ABSORPTION (%)	10.1	10.3	10.2	10.5	10.5	10.4	10.5	9.8	9.8	9.8	10.2

What color samples took as a result of heat treatment for each temperature was illustrated in Fig. IV.11.



Figure IV.11 Appearances of Samples at Different Temperatures

Drawing graphics of water absorption-temperature change using values in Table IV.15, it is evident that the higher the temperature, the greater the water absorption (Fig. IV.12)

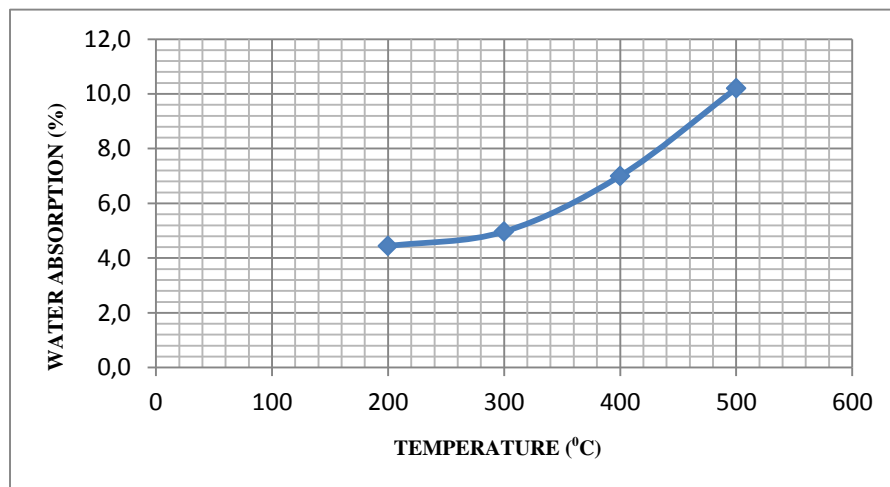


Fig.IV.12 Water Absorption –Temperature Change

IV.3.5 Active Soluble Salts Content (TS EN 772/5)

- Determination of Potassium

The experiment was made via a flame photometer. Since the sample was rarefied by 10 times, the result was to be multiplied by 10. The composition of samples that were cured at 200°C was clay soil + lime + molasses. The number of samples used in the

experiment was 6. They were crushed and then separated with 150 micron sieve. A sample of 100 g. was taken, representing all the samples.

For potassium concentration, reference solutions and sample's emission intensities can be seen in Table IV.16.

Table IV.16 Potassium Concentration-Emission Intensity

Potassium Concentration (ppm)	Emission Intensity
1	83
5	377
10	688
20	1115
35	1655
SAMPLE	532

Observing the values in Table IV.16, potassium concentration value corresponding to emission intensity of 532 for samples was found out as 8.61 mg/l or 0.086 ppm (Fig.IV.13).

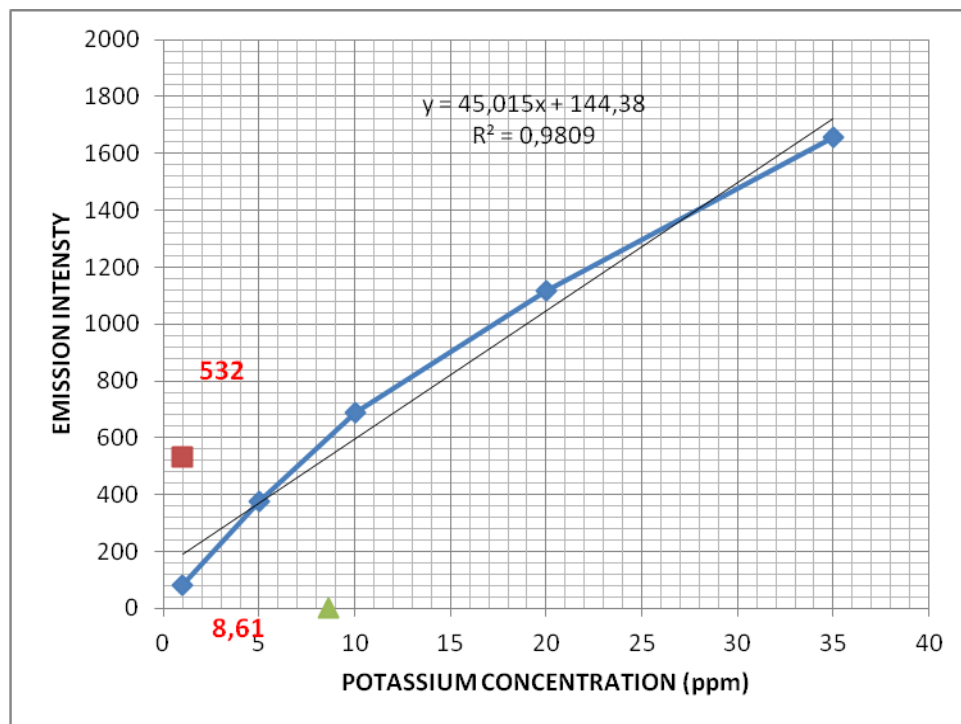


Figure IV.13 Potassium Concentration -Emission Intensity Change

Total material amount (g/ml): 20 g /200 ml

Potassium amount (mg/l): 86.1 mg /l

Potassium percentage (%): 0.086 (1 mg/l = 1 ppm)

- Determination of Sodium

The experiment was carried out by a flame photometer. Since the sample was rarefied 10 times, the result was to be multiplied by 10. The composition of samples that were cured at 200°C was clay soil + lime + molasses. The number of samples used in the experiment was 6. They were crushed and then separated with 150 micron sieve. A sample of 100 g was taken, representing all the samples. For potassium concentration, reference solutions and sample's emission intensities can be seen in Table IV.17.

Table IV.17 Sodium Concentration-Emission Intensity

Sodium Concentration (ppm)	Emission Intensity
1	21
5	103
10	178
50	518
100	783
SAMPLE	55

Total material amount (g/ml): 20 g /200 ml

Potassium amount (mg/l): 48.51mg /l

Potassium percentage (%): 0.048 (1 mg/l = 1 ppm)

- Determination of Calcium and Magnesium

The experiment was performed according to the alternative method cited in TS EN 772/3. The composition of samples that were cured at 200°C was clay soil + lime + molasses. Extracted solution was 20 g / 200 ml and the pattern taken was 25 ml. The experiment was repeated 2 times. Each repetition included 2 patterns. An evaluation was made by finding average of pattern results. Of these 2 patterns, rarefaction factors and EDTA values spent during titration in terms of a total of Ca and Mg values take part in Table IV.18. Resultant values were then multiplied by rarefaction factors to find the final values.

Table IV.18 EDTA Spent and Rarefaction Factor

	EDTA SPENT (ml)	RAREFACTION FACTOR
SAMPLE I	2.5	1
SAMPLE II	2.6	1

The formula that was used to have total concentration of Ca and Mg ions is given below. Data obtained using this formula can be seen in Table IV.19.

$$C_{Ca+Mg} = \frac{c_1 \times V_3}{V_0} \quad (IV.1)$$

where,

C₁: Concentration of EDTA Solution

V₃: Volume of EDTA used in Standardization (ml)

V₀: Volume of Sample Solution (ml)

C₁: 10 mmol/l

V₃: (2.5 +2.6)/2=2.55 ml

V₀: 25 ml

As a result;

C_{Ca+Mg} = 1mmol/l =100 mg/l

C_{Ca} =0.7 mmol/l =70 mg/l

C_{Mg} =1-0.7=0.3 mmol/l=30 mg/l

Table IV.19 Result of Concentration of Magnesium and Sodium

	% Values
Ca	0.07
Mg	0.03

According to TS EN 771/1, maximum ion amounts of 3 brick types can be seen in Table IV.20.

Table IV.20 Active Soluble Salts Content Categories According to TS EN 771/1

Category	Total % by mass not greater than	
	Na ⁺ + K ⁺	Mg ²⁺
S 0	No requirement	No requirement
S 1	0,17	0,08
S 2	0,06	0,03

Hence, the amount of sodium + potassium was determined as 0.134% which was harmonious with the Standard. At the same time, that of Mg ions was found as 0.03% which was also harmonious with the Standard.

CHAPTER V

CONCLUDING REMARKS AND RECOMMANDATIONS

In practice, excavation soil can be called as a waste material of construction industry. Although some part of the soil is used for different purposes in the construction job-site, a large part of it is usually removed. Therefore, transporting and storing this material becomes a costly process in reality. In this study, this waste material was produced as a brick-like structural material, and thereby it could reduce the energy consumption at low temperatures.

For this objective, experiments were made to determine the characteristics of raw material. By sieve analysis, it was first decided to use material of which grain diameter was smaller than 4 mm, as in classical brick production. In total, these fine aggregates constituted about 79.5% of the material of which weight was 120 kg. Water content values that indicate material characteristics in terms of liquidity and plasticity were then found out via experiments such as liquid limit and plastic limit. Compaction experiment was also carried out to find optimum water content. As a result, the material was determined as low-plasticity clay in the Casagrande plasticity card. Physical and mechanical characteristics are important rather than chemical structure in selecting clay. Plasticity is one of them and is denoted by plasticity index (I_p). For brick manufacturing, this index value should be between 8 and 25 (Özışık, 2000). That of excavation soil in this study was calculated as 10.9, which showed that the material was suitable for producing brick. During the production process, optimum amount of mixture water was found as 12% by the proctor compaction experiment. However, it was used as 5% in experiments to reduce the porosity of the material after drying process.

To explore the chemical structure of raw material, XRD and XRF techniques were employed and some comparative studies with the related standards were carried out (Table IV.7). It was seen that chemical contents of materials in practice and in theory

were almost same. This had vital aspect for obtaining positive results throughout the study.

While selecting binders, a procedure was applied to reveal how these binders that were chosen by literature review affected compressive strength values of samples (Table IV.8 - Table IV.9). Thus, the mixture that had the highest compressive strength was “clayey sand + lime (10wt %) + molasses (10wt %)”.

For sample characterization, some features of samples such as water absorption, compressive strength, porosity percentage, dry bulk density, and active soluble salts content were investigated. All of the related results of these experiments were compared with those of TS Standards.

In classical brick production, firing temperatures are very high (900-1100°C). However, in this study, maximum temperature required was 500°C. In literature, similar studies examining brick production at low temperatures are available but they did not use excavation soil as raw material. They worked on brick soil, and used cement and furnace slag as additive material in general.

In TS EN 772/3 standards, a specific value does not exist for water absorption. Porosity percentage should be declared by producer, and at the end of the experiment, the obtained value should not be higher than the declared one. Both in the literature and in the industrial examples, water absorption value of a standard solid brick should not be higher than 20%, as an average. In the present study, as the temperature rose, porosity percentage of the material increased from 8.7% to 23.9%. On the other hand, note that classical brick is baked at higher temperatures than 500°C and its porosity percentage normally decreases. Therefore, a porosity percentage of 8.7% at 200 °C in which maximum compressive strength was obtained showed that the material was quite compact and porosity-free.

As temperature rose, dry bulk densities of the samples decreased slightly (Fig.IV.9). This density of 2096 kg/m³ at 200°C declined down to 2007 kg/m³ at 500°C. According to TS EN 772/1, dry bulk densities of the samples produced could represent solid bricks of which density is 2000 kg/m³ in terms of maximum and minimum limit values.

As a result of experiments, maximum compressive strength was found as 15.3 N/mm² at 200°C. This outcome pointed out the type-3 brick in TS EN 771/1, which has density value of 2000 kg/m³. Although the values given in the Standards are obtained at 900-1000°C while producing classical brick, the samples in this study were manufactured at 200°C.

The value concerning water absorption rose with the same trend in temperature (Table IV.15) because water and other evaporative matters in material vaporize by the rise in temperature and thus the amount of porosity increases. Water absorption value was 4.2% at 200°C went up to 10.2% at 500 °C.

In TS EN 771/1, water absorption is not expressed with any number. However, in BS 3921 (English Standards), it is 7% for Class B and 4.5% for Class A. In the literature and in the practical examples, that of solid brick is not desired to be higher than 18% in general. As a conclusion, water absorption values in Standards and in this study were very close to each other.

In the experiment related with the active soluble salts content, 4 ions in the brick material which had maximum compressive strength at 200 °C were examined. The results obtained did not exceed the limits of ion percentages available in TS EN 771/1'e (Table IV.20).

Consequently, it is obvious that the experimental results of the samples of which components, i.e. excavation soil and some binders, were pressed together at low temperatures such as at 200 °C are almost the same with the Standards including values for classical bricks produced at high temperatures.

Following recommendations could be given which are as follows

- a) In this study, various curing temperature ranging from 50 up to 500 °C used. Higher firing temperatures could be studied to evaluate the effect of elevated temperature.
- b) Varieties of binder used up to 10 wt % in this study. Addition level could be increased and different binder could be used to see the effect of type of binder.

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BIOGRAPHY

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ACCEPTANCE AND APPROVAL DOCUMENT

The jury established by the Executive Board of the *INSTITUTE FOR GRADUATE STUDIES IN PURE AND APPLIED SCIENCES* on 4th of July 2011 (Resolution no: B.30.2.MAR.0.C1.00.00.sek./1702) has accepted Mrs Gülden Çağın ULUBEYLİ 's thesis titled "Recycling of Excavation Soil for the Production of Building Material" as Master of Science thesis in Metallurgical and Materials Engineering

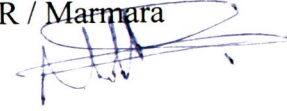
Advisor :Assoc.Prof. Dr. Recep ARTIR / Marmara
University, Technology Faculty



1. Member of the jury : Prof.Dr. Ayhan MERGEN / Marmara
University, Engineering Faculty



2.Member of the jury : Assistant Prof. Dr Mustafa ÇAKIR / Marmara
University, Technology Faculty



Date : 11.07.2011

APPROVAL

Mrs. has satisfactorily completed the requirements for the degree of Master of Science in at Marmara University. The Executive Committee approves that she was granted the degree of Master of Science on... 20.07.2011 (Resolution no.: 2011./15.02.)

DIRECTOR OF THE INSTITUTE

Prof. Dr. Meral ÜNAL

