EFFECTS OF FIRING TEMPERATURE, AND RATIO OF STEEL/IRON SLAG ON THE MECHANICAL AND PHYSICAL PROPERTIES OF CLAY BRICKS, THE CASE OF JIMMA TOWN

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Declaration

I hereby declare that the thesis work entitled: “Effects of Firing Temperature, and the ratio of Steel/Iron Slag on the Mechanical and Physical Properties of Clay Bricks; The Case of “Jimma Tow”, submitted to the school of Graduate studies of Jimma University, Jimma Institute of Technology is a record of an original work done by me under the guidance of Dr. Olu Emanuel Femi and Mr. Zekarias Gebreyes Eticha. This project work is submitted in the partial fulfillment of the requirements for the award of the degree of Master of Science in Ceramics Engineering. The result embodied in this thesis has not been submitted to any other University or institute for the award of any degree or diploma.

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ABBREVIATIONS

BF…………………….Blast Furnace

BOF………………… Basic Oxygen Furnace

BFS………………….. Blast Furnace Slag

EAFS………………… Electric Arc Furnace Slag

XRD………………… X-Ray Diffraction

XRF…………………. X-Ray Fluorescence

SEM………………..… Scanning Electron Microscope

EDS…………………. Energy Dispersive spectroscopy

WAP………………… Water Absorption Property

LOI…………………...Loss on Ignition

CTM………………… Compressive Testing Machine

ASTM……………….. American Society for Testing and Materials,

MPa………………..… Mega Pascal

L……………………. Maximum load applied to bricks specimen

A……………………. Cross sectional area of bricks

RMM……………….. Research and Metallurgical Microscopy

CTM………………... Compressive Test Machine

RCA………………… Recycle Concrete Aggregate

Abstract

A construction brick made from clay soil and iron slag with variable temperature has been synthesized. In the process iron slag was collected from steely RMI Bishoftu Ethiopia and clay soil was taken from Jimma town Frustalle area. The samples was then grinded grounded properly and sieved to have a fine sample. The samples clay was mixed with a proportion containing; no slag, 10% slag, 20% slag and 30% slag and water to attain a plastic property and the brick was synthesized. The synthesized brick is then fired at a temperature of 900^0C. 1000^0C and 1100^0C and samples were arranged for characterization. The characterization was made in to three parts; the mineralogical and morphological property, the physical property and the compression strength. The mineralogical characterization result shows the clay composed of quartz as a main content while feldspar and other are the main content at the same time the content of quartz in the clay brick decreases with increasing in temperature. The mineralogical characterization result of iron slag also shows the main component of hematite while Ca_2SiO_4 and $CaAl_2O_4$ are the other component. The increase in firing temperature cause in the decomposition of Ca_2SiO_4 and $CaAl_2O_4$ and favor the formation of quartz, crystobollite and kaolinite, this indicated that the samples have good mechanical and physico-sintering properties. Finally the physical and mechanical characterization result indicates, the addition of iron slag improves the property of the brick. But the result outcome indicates that the weight loss on ignition violet the expected result and the percentage of firing shrinkage at 30% slag addition above 1000^0C did not change.

Key Words: Clay soil, Iron slag, Mineralogical and morphological property, Physical property.

Chapter One

Introduction

1.1 Historical Background

One of the oldest materials in the history of building and construction used by humankind is clay brick.Clay'ssoil as a raw material for clay bricks are mostly valued due to their ceramic characteristics. It was a fundamental building material in the Mesopotamian, Egyptian and Roman periods. Clay brick continued to be used during medieval and modern times. Despite several modifications of the clay brick intensive research is required to make the clay product economical, light weight and easy to produce [1].

In the last decades, the studies involving ancient building technologies and materials are of historical importance due to cultural and economic reasons. Clay brick, in the forms of sun dried and burnt, has been used since the beginning of civilization, ten thousand years ago. It was selected widely because it was easily produced, lighter than stone, easy to mold and formed a wall that was fire resistant and durable. The use of brick masonry, often in combination with stone masonry and timber roof, or floors, is well distributed all over the world [2].

The clays are burn in to bricks of various colors due to the presence of rock particles in it. The most important properties of clays that make them highly desirable as brick materials are the development of plasticity when mixed with water, and the hardening under the influence of fire, which drives off the water content [3].

Formerly, the characterization of old clay bricks was a hard task due to the difficulties in collecting samples, and the lack of standard procedures for testing. Currently it is believed that, characterization is relevant to understand damage, to assess safety, to define conservation measures and even to make a decision on reusing or replacing existing materials as modern materials and the like[4].

Recently different raw materials are replacing the clay bricks with small percentage to modify the mechanical properties of it [5]. At the same time, the evolution of technology in the countries forced us to depend on industry-led modernization and focus the researcher on waste disposal system and reusing it as other input. A high volume of production of raw materials like steel/iron has also generated at the same time a considerable amount of waste materials which can create adverse impact on the environment has been disposed [6].

Consequently, the use of waste has emerged as a viable technology in brick making and other activities. Using this iron/steel slagoffer two major advantages; recycling of the waste to minimize the disposal problem and preserve the precious fertile soil, which is essential for the cultivation of agricultural crops [7].Keeping this advantage in to consideration, this research design to synthesize a construction clay brick made from clay soil and iron/steel slag. In the process, the morphology and microstructure, the physical and mechanical properties are tested by varying the mix ratio and firing temperature

1.2 Statement of the Problem

Following the technological advancements, those productive industries expand in Ethiopia, Iron/steel rolling industries are among, the factories and those industries use iron scrap to processes and manufacture sheet metals, re-bar and the like. Since those scraps are collected from different areas, the waste product of those iron/steel industries become a threat for environment because it contains carcinogenic elements like chromium and cobalt. Therefore, removing or reusing them in a way that cannot harm the human being is the main task for the researcher. At the same time, the natural resource used for brick construction are depleted with time and the clay brick made from it are needed to withstand all external factors like mechanical, electrical, chemical and physical effects. In this regards finding a suitable material that enhance the mechanical property should be the task for materials and ceramic engineers. Additionally all of the clay bricks production process in Jimma town are historically a long term experience and are produced with limited types of clay bricks soil found in the town. Furthermore, the temperature of fired clay bricks production is remain undetermined and has been done traditionally. In this regard one of the main contributing factors for this problem is poor firing temperature controlling mechanism. So in addition to the study of the firing temperature controlling mechanism of these production process, determination of the effect has an effect on the optimized values of compressive strength, water absorption and structure of clay bricks. This are the facts which drawn the attention for this study.

1.3 Objectives of the study

Using industrial waste in the construction ceramic materials have a dual advantage; minimizing the environmental risk and getting alternative materials for the brick production. Hence, it requires intensive research to convert wastes released from one industry to raw material for this sector. Therefore, the general as well as specific objectives of the research set as follows;

1.3.1 General Objective

To examine the effect of firing temperature and Iron/steel slag addition on the mechanical and Physical property of construction clay bricks.

1.3.2 Specific Objective

- To investigate the chemical composition of Iron/steel slag useful for construction purposes.
- To identify and compare the property of raw clay brick and iron/steel slag added brick.
- To synthesize and identify the effect of iron/steel slag on the clay brick.
- To investigate the Mineralogical and Morphological property of clay brick.
- To Test the mechanical and water absorption property of the as-synthesized clay brick.

1.4 Research Design

In conducting this research some required steps has been accomplished, to mention some of them;

i. Collecting some secondary data (information) related to clay bricks and iron slag, reading and making a comprehensive literature review to get the gap on the area.
ii. Making a visit to the areas of clay soil and the iron/slag source to identify the problems practically and to collect sample raw materials.
iii. Designing the experimental procedure to study the effect of iron/steel slag addition and firing temperature on the clay brick.
iv. Analyzing the experimental data to reach to the conclusion
v. Forwarding the concluding remark and recommend for further study.

1.5 The thesis Layout

This thesis contains five main chapters. The first chapter is introduction part, includes background of the study, problem statement, objective of the study and significance of the research. The second chapter explains the finding from the review of different literatures including; thesis report, books, historical record and website, which are relevant to the study. Chapter three describes methodologies used in this thesis including data collection, laboratory test on the sampled clay and iron slag, selection of site, bricks production process, laboratory tests of unfired bricks ,laboratory tests of fired bricks and methods of data analysis. The fourth chapter presented the result and discussed of the laboratory test results. Chapter five includes conclusion, and recommendation for further studies.

CHAPTER TWO

2. Review of Related Literature

2.1 Iron Industrial Wastes, World Overview

The iron and steel industry is the most important element of a nation's industrial economic infrastructure, and the consumption of steel per capita of population is an indicative index of industrialization and progress. Iron and steel making is one of the most important industrial sectors which have a great impact on the global growth and economy. The steel production is sharply increased in the recent years to reach more than 1662.8 million tons in 2014-15, up by 1.2% compared to 2013. By 2050, steel usage is expected to increase to become 1.5 times higher than present levels in order to meet the needs of a growing population [8].

Chinese crude steel production reached 804 million tons in 2015, accounting for 73% of Asian and 50% of world crude steel production. India was the 3rd largest crude steel producer during 2015 with a production of 89 million tons [9]. In India, the crude steel production has shown a sustained rise since 2010–16 as shown in Table 1

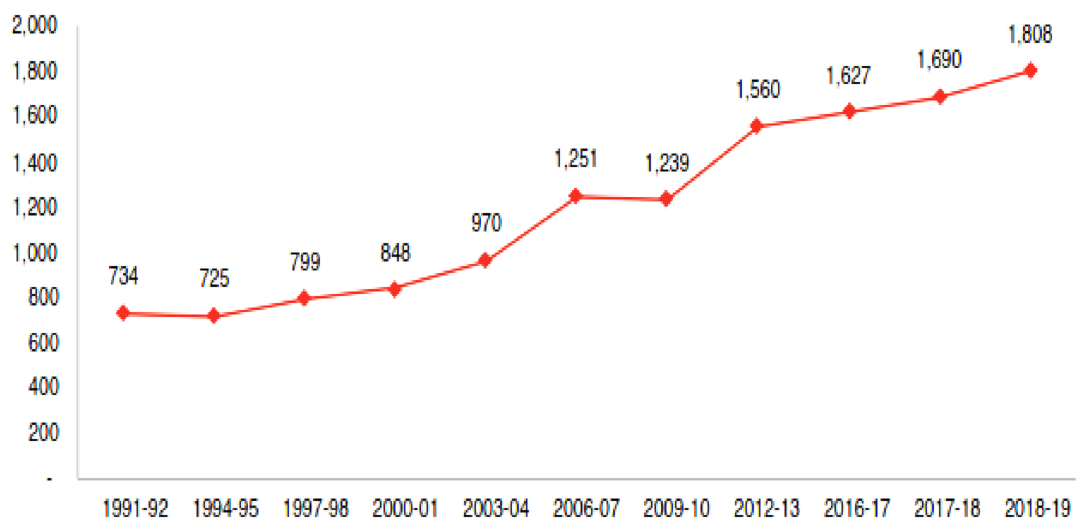

Figure 2.1: Year-wise Crude Steel Production in the world (Source: [10])

In total, 70% of the world steel is produced utilizing the Blast Furnace (BF), where iron ore is reduced to pig iron, which is afterwards converted into steel in the Basic Oxygen Furnace (BOF. On average, or one ton of steel 200 kg (in the scrap-based steelmaking) and 400 kg (in the iron ore-based steel making) of by-products are produced[11].

The two different process routes available to produce steel products, namely the blast furnace with oxygen steelmaking and the electric arc steelmaking route. In both the process the Blast furnace process plays an important role in production of any steel product, thus, causes production of waste too[12]. But, in the stainless steel making process, lots of wastes are generated from the factories. Approximately one ton of stainless steel wastes was originated when producing three tones of stainless steel [13]. On the other hand large volumes of slag are produced during the process of removing metals from ore and, thus, extensive slag dumps arepresent at both historical and modern smelting sites. This waste will fill the lad if it is damped put side which can affect the cultivable lands as well as it will pollute the ground waste.

2.2 Steel Slag

Steel slag, a by-product of steel making, is produced during the separation of the molten steel from impurities in steel making furnaces [14]. The slag can be produced as a molten liquid melt and is a complex solution of silicates and oxides that solidifies upon cooling. Slag is a waste product from the pyro metallurgical processing of various ores [15].

The slag generated from this process is referred to as Blast Furnace Slag (BFS).The hot metal is then converted to steel in a basic Oxygen furnace where the carbon percentage is controlled to obtain desired properties such as high strength. Other types of slag results from this process are known as the integrated production method. In min-mills, steel scrap is melted in an electrical arc furnace.

The use of such materials not only result in conservation of natural resources but also helps in maintaining good environmental condition by effective utilization of these wastes material. Slag can be act as an alternative construction material with superior environment friendly qualities and better product features.

Table 2.1: Chemical Composition of steel slag (Blast Furnace Slag (BFS), Basic Oxygen Furnace (BOFS) and Electric Arc Furnace Slag (EAFS)[16]

Chemical Analysis	**BFS**	**BOFS**	**EAFS**
Silicon Dioxide(SiO_2)	34.05	9.68	22.56
Aluminum Oxide(Al_2O_3)	13.35	0.41	3.52
Iron(Fe)	0.83	21.00	20.20
Ferric Oxide (Fe_2O_3)		7.52	
Calcium Oxide(CaO)	37.20	44.29	37.00
Magnesium Oxide(MgO)	4.54	4.45	3.08
Sulphur(S)	1.44	0.18	
Sulphur Trioxide(SO_3)			0.24
Manganese Oxide (MnO)		8.50	10.20
Phosphorous Oxide (P_2O)	2.92		
Phosphorous Pent oxide (P_2O_5)		3.47	0.68
Potassium Oxide(K_2O)			0.03
Chromium Oxide (Cr_2O_3)			2.04
Titanium Oxide (TiO_2)			0.45
Sodium Oxide (Nao_2)	1.45		
Barium Oxide (BaO)	3.95		
Potassium Oxide (K_2O)	0.27		
Basicity Index	1.09	4.57	1.64

The Mineralogical composition of the those furnace slag shown in the above table can be considered as an ideal staff for construction brick synthesis since it has some ceramic materials (Minerals) that can give a thermo-physical strength for the clay[17-19].

2.3 Clay Brick

The clay brick is one of oldest building materials yet known[20] and its widespread use is mainly due to the availability of clay minerals used for the production and durability in both load bearing and non-load bearing structures[21, 22]. It was a fundamental building material in ancient periods and the use of clay brick was increased and became specialized in order to maximize its benefits during the Roman period. The durability of the bricks is its ability to withstand an applied load without failures. The clay brick is used widely in the construction industry all over the world. The global brick production is estimated about 1.5 trillion annually and Asian countries only accounts about 89-90 % of the global production [23].

The report presented by (Altayework Tadesse, et.al, 2013) indicatesthe use of clay brick as a walling material is very low and is accounted as 0.1 percent. Some of the reasons are the cost of the brick due to the scarcity of the raw material and limited shape of production. So finding a viable substitute of input raw materials for clay production will alleviate the problems[24]

Table 2.2: Distribution of Households by Construction Material of Walls of a Dwelling Unit and Place of Residence in Ethiopia[19]

Mineral of Walling	**Place of Residence**		
	Country (%)	**Rural (%)**	**Urban (%)**
Wood and mud	76	74.8	82.4
Wood and Thatch	7.8	9.1	1.0
Reed/Bamboo	3.3	3.9	0.4
Stone and Mud	8.7	9.1	6.3
Stone and Cement	0.7	0.0	4.3
Hollow Blocks	0.5	0.1	3.1
Bricks	0.1	-	0.4
Others	2.8	2.9	2.1

For brick manufacturing, the main imput of the brick clay soil have to possess some specific properties and characteristics. Such clays must have plasticity, which allowed to be shaped or molded when mixed with water; they must have sufficient wet and air-dried strength to maintain their shape after forming at the same time it should withstand the bad condition.

The clay bricks depended on the mineralogy of the clay materials used to manufacture it, the manufacturing process and firing temperature. The fired clay bricks are extremely durable and hence, there have been numerous archaeological masonry buildings standing for centuries as a testimony of the survival of the clay-based fired bricks. In general, clay bricks are classified into various groups based on their mineralogy.

The mineralogical, physico-sintering and mechanical properties of clay bricks are varied depending on the degree of firing at high temperature and these properties are interrelated to each other. The physico-sintering and mechanical properties of different clay bricks were investigated at different firing temperatures and the brick sample fired at 1100 °C showed the best mechanical properties[23].

2.4 Process of fired clay bricks Manufacturing

The fundamentals of brick manufacturing have not changed over time. However, technological advancements have made contemporary brick plants substantially more efficient and have improved the overall quality of the products[25].

Brick is still produced with an ancient technology. The phases of the production of traditional clay brick is the same as manufacturing techniques elsewhere

There are four different operations are involved in the process of manufacturing of bricks: Namely, Preparation of clay, Molding, Drying, Burning. Once it passes this major steps the clay bricks property are characterized[26].

2.4.1. Preparation of clay for brick manufacturing:

Preparation of clay for bricks manufacturing is done in six steps:

Unsoiling of clay:- The need of clay for the preparation of bricks should be the pure one, since the top soil contains impurities, most of the top layer of soil about 200mm depth is thrown away. This process is termed as unsoiling.

Digging: After the removal of top layer, the clay is dug out from the ground and spread on the plain ground.

Cleaning: In this stage, the clay is cleaned of stones, vegetable matter etc. if large quantity of particulate matter is present, then the clay is washed and screened. The lumps of clay are converted into powder with earth crushing rollers.

Weathering: The cleaned clay is exposed to atmosphere for softening. The period of weathering may be 3 to 4 weeks or a full rainy season. Generally, the clay is dug out just before the rainy season for larger projects.

Blending: If we want to add any ingredient to the clay, it is to be added in this stage by making the clay loose and spread the ingredient over it. Then take small portion of clay into the hands and tuning it up and down in vertical direction. This process is called blending of clay.

Tempering: In this case, water is added to clay and pressed or mixed. The pressing will be done by feet of men or hands for small scale projects. So, the clay obtains the plastic nature and now it is suitable for molding.

2.4.2. Molding of clay for brick manufacturing

Molding is a process of giving a required shape to the brick from the prepared brick earth. The brick is taking its shape in this stage, in this process prepared clay is mold into brick shape (generally rectangular). This process can be done in two ways according to scale of project.

- Hand molding (for small scale)
- Machine molding (for large scale)

2.4.3. Drying of raw bricks

The drying process helps to control shrinkage, save fuel and time during burning. Some reseaches use sand added to the clay during mixing by some manufacturers. This reduces shrinkage since the drying shrinkage is dependent upon pore spaces within the clay and the mixing water. To protect the cracking of the brick during burning (Firing) the sample is allowed to dry by the natural process under sunlight. The bricks are laid in stacks, and this stacks should be arranged in such a way that circulation of air in between the bricks is free. Most of the time the drying of bricks may take 3 to 10 days depending on the weathering condition. In Some situations artificial drying is adopted under special dryers or hot gases [27].

2.4.4. Burning of bricks

The critical stage in the manufacture of burnt clay bricks is the firing stage; it determines the shape, color and the durability of the burnt bricks. The clay after drying is allowed to dehydrate, the water which has been retained in the pores is driven off and the clay loses its plasticity. In the process, the dried bricks are burned either in clamps (small scale) or kilns (large scale) up to certain degree temperature. In this stage, the bricks will gain hardness and strength so it is important stage in manufacturing of bricks.The temperature required for burning most of the time ranges up to 1100^{o}C. Hence burning should be done properly to meet the requirements of good brick.

At temperatures of approximately 450^{o}C the clay structures begins to break down due to the decomposition of the hydroxyl (OH) groups and organic matter starts to burn off. After the reaction, an amorphous or glassy structure is formed. At 863^{0}C calcium carbonate present in the raw material begins to decompose into calcium oxide and carbon dioxide.This reaction triggers molecular rearrangement causing shrinkage.

2.5 Ingredients of Clay Bricks and their function

Bricks are ceramic materials obtained by firing raw clay at temperatures ranging between 650 and 1000^{0}C. Color, composition and mechanical properties are dependent on the nature of the clay (kaolinite, illite, smectite), kiln environment (oxidative or reductive), kiln operator, temperature and firing process, nature and amount of the temper (quartz, carbonates, shards, grounded fired clays)[28].

The chemical composition of old bricks allows the identification of possible deficiencies that occurred during their production, like the presence of organic matter, lime nodules, harmful soluble salts and other impurities that might influence the durability of the brick. Soluble salts and other impurities are one of the most important factors of brick decay and are frequently found in old clay brick fabrics[29]. Chemical composition can also provide information about firing temperature and degree of vitrification[30] which is relevant for the manufacturing of new replacement bricks. Chemical composition can also explain, to a certain extent, the brick color by indicating the presence of colorants and other additives.Based on the reinforcement property, the ingredients can be grouped in to Useful and harmful ingredients.

Chemical oxides commonly found in clay bricks are the following: silica (SiO_2), alumina (Al_2O_3), iron (Fe_2O_3). Are the main one. Silica and alumina constitute the base elements of clay and are usually found with a proportion of 50%-60% for SiO 2 and 15–20% for Al_2O_3[31]. Other components might be considered like manganese (Mn) and the like. However, these elements are always present in very small quantities and expressed in parts per million (ppm), while the proportion of the main components is expressed in percentage of the material volume [33].

2.6 Testing Mechanisms of Bricks

Structural behavior of masonry unit is influenced by constituents units like bricks and mortar individually and as composite mass together.

To modify the natural materials is required to know in detail its structural characteristics, chemical composition and properties, which can be achieved using modern analytical techniques such as Optical or electronic Microscopy, X-Ray Diffraction, Infrared Spectroscopy, among others. For the specific case of brick manufacturing, it is important not only to characterize the microstructural characteristics of the clays, but also its microstructural behavior [34, 35].

Formerly, testing mechanisms have focused on the physical, chemical and mineralogical composition of the bricks[36], deterioration and agents durability, neglecting the mechanical properties, which are more frequently retrieved in the case of the composite material. Mechanical characterization is a fundamental task for the structural works and safety assessment, where the compression strength is a key parameter in the case of masonry structures[37]. The purposes and use of additives in brick production is frequent and depends on the characteristics required. During characterizing the product the main factors determining the property are the type of raw material used and the firing temperature, both of which affect the quality of the final product[38, 39].

2.6.1 Morphological and Mineralogical Testing

The morphological of the final product observed by optical microscope slightly informs the arrangement of the defined inputs and the void formed in the brick. It is also capable in informing the general failure of the clay brick due to internal crack[40]. Scanning electron microscopy (SEM) with EDS is the best mechanism for determining the morphology of the clay bricks properly[41].

An influence of microstructure on the mechanical and physical properties of a material is primarily governed by the different defects present or absent of the structure. These defect can take many forms but the primary ones are the pores. Even if those pores play avery important role in the definition of characteristics of a material, so does its composition. In fact, for many materials, different phases can exist at the same time. These phases have different properties and if managed correctly, can prevent the fracture of material[42].

2.6.2 Physical Property Testing

The mineralogical and physical properties and mechanical strength of the fired clay bricks are generally interrelated to each other. It is reported that the firing temperature is one of the key factor to modulate the physical, sintering and mechanical properties of different types of brick samples[43]. The physical properties containing water absorption, Firing shrinkage, Mass loss on ignition, are those main properties of the brick that determine the mechanical property or load bearing capacity of the building.

2.6.2.1 Water absorption of bricks

The bricks porosity and their permeability and absorption are very important factors in influencing properties of bricks such as the bond between them and mortar, the resistance of bricks to freezing and thawing, and their chemical stability. It is depends on clay properties, manufacturing process and burning. Also water absorption determines the durability of clay bricks. High absorption capacity causes the bond strength between the slag and the bricks reduced[44]. Water absorption is the property (W.A.P) of a material to be saturated with water. It is closely associated with the porosity of the material. However, not always may all pores of material be filled with water when its water sorption is being determined. This fact is attributable to the size, volume, configuration and mutual arrangement of pores in the materials[45].

2.6.2.2 Firing shrinkage of bricks

In the drying process, clay particles draw together and shrinkage occurs and the matrix densifies and shrinkage continues. As temperature continues to rise, some of particles begin to melt and form a glassy between the others that pull them even closer. Some of the particles shrinkage themselves, Kaolin is an example. Shrinkage from dry to fire is a comparative indicator of the degree of verification. If fired shrinkages are measured over a range of temperatures for a body it is possible to create a graph to get a visual representation of the body's maturing behavior and range. As noted, fired shrinkages are relative within a system, there is no absolute of how much clay should shrink when fired. Some sintered bodies have very low fired shrinkages, because they are packed so tightly during pressing and because no glass develops. Such kind of shrinkages are not a product of temperature, but of the amount of flux present in a body to develop particle bonding glass during firing. Fluxes are available at all temperature, feldspar at higher temperature and frit is at lowest temperature[46].

Fired clay bricks are produced when clay particles bond to one another at high temperatures, forming a glassy material, which upon cooling, displays high strength and durability properties. High temperatures required to melt SiO_2 mean high energy cost associated with brick production[47].The test bricks first measured and then placed inside the furnace and fired up to needed temperature and the lie drawn across the diagonal axis of the pieces was measured to determine its final length after firing. The linear shrinkage of the materials was determined with the following equation.

$$\textbf{Weight loss on ignition} = \frac{W_{bf} - W_{af}}{W_{bf}}$$

Where, W_{bf}- is weight before firing andW_{af}- weight after firing of the brick

2.6.2.3 Mass loss on ignition

The loss of ignition is due to the elimination of the water content of the clay mineral as a consequence of dihydroxylations reaction and the elimination of the organic matter content and the carbonates contained in the clay and the residue, as well as the decomposition of other inorganic components contained in the waste[48].

The loss on ignition (LOI) is the weight reduction on the total weight of the prepared clay samples, in percentage. The loss in weight by each clay sample was determined to be the difference in their weights before and after firing[47].

When the firing temperature was below 800^0C, the carbonate in the mixture did not completely deform. When the firing temperature was higher than 950^0C, the carbonate deformed to CO_2 and caused a weight loss in the brick. Additionally, when the temperature was higher than 1000^0C, Oxygen was recaptured from the air due to the oxidation of iron and the weight loss in the brick was reduced. A normal clay bricks weight loss on ignition is 15%[42].

2.6.2.4 Efflorescence (contents of alkalis)

Efflorescence can be defined as a crystalline deposit of soluble salts of alkaline materials, generally white in color that appears on the surfaces of clay brick. The salts of efflorescence are generally potassium, sodium, magnesium, iron and calcium; silicate; or carbonates of calcium and sodium; or sodium bicarbonate. On the other hand, all soluble salt that finds its way into the materials may appear as efflorescence[29, 49].

Efflorescence needs three conditions for it to happen, the presence of soluble salts in the materials, the presence of moisture to dissolve soluble salts, and take them to the surface and hydrostatic pressure or evaporation must reason the solution to be in motion. Efflorescence cannot happen if any one of these conditions is not present[50].

The efflorescence phenomenon has a significant impact on the performance and properties of clay brick in engineering structures, the most important of which are most of the salts are salts of alkaline metals or sulphate and alkaline dust carbonate, although the chlorides sometimes appear with a small amount of efflorescence and the future effect of these salts is negative on the properties of bricks, resulting in cracking and fragmentation of the walls as a result and transform it into a fragile material.

2.6.3 Compressive strength Test

Compressive test is part of mechanical testing and is the main and important test for bricks. This test was carried by out by a compressive testing machine (CTM) that has its own capacity. Compressive Strength is defined as the resistant to compression of bricks and measured in the laboratory by applying perpendicular load to the largest face. Also compressive strength is the most universally accepted value for determining the quality of bricks. Nevertheless, it intensely related with the soil types and stabilizer content. Compressive strength is the ability of material or structure to carry the loads on its surface without any crack or deflection. Compressive strength depends on many factors such as bricks surface, strength, quality and quality control during production of bricks. Compressive strength for any material is the load applied at the point of failure to the cross section area of the face on which load was applied [33].

2.7 Gap analysis

Different researches has been conducted on the replacement of different construction materials inputs like the clay soil, cement and the like totally or partially by other materials aiming at different targets[16, 51-52]. Some of the experiments conducted on the cement and clay soil are aiming at improving the hydration property of the composite cement pest [53]. Based on the result of the experiment the researcher declare that this hydration property improved due to addition of fly ash improves the fire resistance property of the clay brick.

The research by (Vihangraj V. Kulkarni, et.al 2019) also reported that utilization of hazardous battery sludge in fired clay brick production with the conjoint used for reducing depletion of fertile clay and heavy metal fixation[54]. The modified bricks exhibited an improved ductile behavior and porosity restraining its deformation and/or distortion during firing and avoiding sudden failure of masonries. Moreover the improvement thermos-physical properties of clay brick by adding natural fiber sisal also reported by (M. Ouakarrouch, et.al, 2020)[55].

The research report was also reported by (Ertugrul Esmeray, et. Al, 2019) investigating the semi-qualitative chemical property, physical, thermal and mechanical property of sewage sludge, oven slag and fly ash added clay brick. While (K.A.M. El-Naggar, et.al, 2019) reported on the porosity and light weight clay production by adding slaked lime waste from acetylene production and waste aluminum trimmings[56]. A research reported by (Yan-bing ZONG, et.al, 2019) also indicates the influence of the particle size of iron slag on the sintering property of red clay brick [57]. Finally, as to my level of understanding no research work has been reported on the Effects of firing temperature, and ratio of steel/iron slag on the mechanical and physical properties of clay bricks.

CHAPTER THREE

3. Introduction

This experimental section was primarily concerned with the investigation of the firing temperature and potential use of iron/steel rolling slag for replacing clay soil partly in the clay brick production. Iron/steel rolling slag is usually produced as waste from Iron and steel industries in large quantities.

The use of waste slag as a partial substitute of clay soil becomes an attractive topic for researchers due to its dual advantage; economic and environmental. Adding of slag to the brick is not only reduces the consumption of clay raw materials, divert wastes from landfills, but also provides potential profit in tipping fees for manufacturers. Paying this hot issue in to consideration, the experimental program of this research was carried out to explore the effects of iron slag and the firing temperature on the water absorption behavior, physical and mechanical properties following the testing procedure of specifications of ASTM.

3.1 Materials and Methodology

The materials used in the experiment as well as the methodology to synthesize the brick with clay and iron slag has been presented in this chapter. Moreover, characterization used to examine the physical property, the chemical composition, mechanical property and morphology also stated.

3.1.1 Experimental sites

Iron/steel slag sample was collected from Bishoftu, Ethiopia, which is 45km east of the capital city, Addis Ababa. The clay was also collected from Jimma Town; Frustule kebele, which is resourceful area in white mud and clay soil. Besides, mixing of clay and Iron slag and the brick was mede in Jimma University, Materials Science and Engineering, Civil and Environmental Engineering as well as in Mechanical engineering lab. Most of the characterization has been done in Jimma Institute of Technology Laboratories.

3.1.2 Materials used in the experiment

To accomplish the objectives, the following materials has been used; mortar and pestle, electronic digital beam balance, wooden mold, sieve, glove, container to mix the proportionally arranged raw materials, table, compact machine, oven, cups to put mixed powder, electrical furnace has been used. Moreover, for of characterization purpose instruments like XRD, XRF, Research and Metallurgical Microscope and Mechanical strength (Compressive strength tester called UTEST) were used. In addition, some mathematical method applied to analyze the collected data.

3.1.3 Experimental Procedure

Samples collected from the mentioned areas were mixed together with different proportion. To manage the experiment the ratio was taken in to four parts as 100%, 90% 80% and 70% by weight of clay and the remaining part has been filled with iron slag. The mix proportions were prepared based on the dry weight of the materials. Mixture proportions were presented in Table 3.1.

Table 3.1 Designation and percentage of blending of the blended clay and slag samples

No.	**Sampled clay and slag**	**Percentage of blending proportion by weight (%)**	**Designation of the blended clay and slag**
	Clay	100%	
	Slag	0%	
	Clay	90%	
	Slag	10%	
	Clay	80%	
	Slag	20%	
	Clay	70%	
	Slag	30%	

In the processes the sample clay taken from Frustule; Jimma was Grinded properly and sieved well with 150micrometer sieve size. At the same time iron slag taken from steely RMI iron/steel rolling industry, Bishoftu area was grinded and sieved with 150-micrometer sieve and mixing process has been done.

3.1.3.1 Preparation of Clay Bricks

The raw materials were mixed mechanically for 5 minute tohave a uniform behavior. After dry mixing water was added gently until, the mix attain plastic property. After that, the mix was covered with canvas and left for 20hr to attain better plasticity property. The brick was then synthesized using the mold with dimension of 12cm x 6cm x6cm, and then the synthesized brickswere dried at room temperature for 7daysthen put in an oven at 70^0C for 24hr to remove the remaining water in it. After drying at room temperature and in an oven the drying shrinkage of pure clay bricks and iron slag clay composition bricks measured.

Table 3.2: Drying shrinkage of pure clay bricks and iron slag clay composition bricks.

Bricks types	**Before Drying**				**After Drying**			
	L_b cm	**W_b cm**	**T_b cm**	**V_b cm^3**	**L_a cm**	**W_a cm**	**T_a cm**	**V_a cm^3**
0% slag	12	6	6	432	11.7	5.8	5.7	387
10%slag	12	6	6	432	11.8	5.8	5.8	397
20%slag	12	6	6	432	11.9	5.8	5.8	400
30%slag	12	6	6	432	11.9	5.9	5.9	414

3.1.3.2 Sample Size and Firing Temperature

The research was designed in to four groups to compare the mechanical, water absorption and micro structural property of bare clay brick and different percentage proportion of iron slag added one. The mixing ration, amount of samples prepared across each groups and firing temperatures are shown in table 3.3

Table 3.3. The clay bricks prepared with respective ratio and fired temperature

No	Mixing Ratio Clay: Slag	Number of sample Prepared	Amount of Clay soil Used in (gm)	Amount prepared with the corresponding Temperature		
				900^0C	**1000^0C**	**1100^0C**
1	100%	12	7200	4	4	4
2	90%:10%	12	6480	4	4	4
3	80%:20%	12	5760	4	4	4
4	70%:30%	12	5040	4	4	4

The sample preparation consumed a total of 7.2kg of clay soil for 12 bricks with100% clay, 6.48kg of clay soil for 12 bricks with 90% clay soil, 5.76kg of clay soilfor 12 bricks with 80% clay soil and 5.04 kg has been used for 12 brick with 70% clay soil containing composite brick, the remaining was filled by iron slag. A total number of 48sample bricks were synthesized and the prepared clay bricks were grouped in to three groups with equal number of samples each and fired at a temperature of 900^0C, 1000^0C and 1100^0C.

3.2 Testing Method

For the testing purpose, twelve brick samples were prepared for each 4 groups that listed in Table3.1. These four groups of brick samples are divided into three categories, for the purpose of testing compressive strength, the other was for microstructural and morphological property and last one is to tests physical properties like water absorption, firing shrinkage. The data was used for further analysis and draw out the concluding remarks.

3.2.1 Microstructural and Morphology Testing of the Sample Brick

To understand the role played by microstructural properties, the surface roughness of the clay soil, and iron slag in the clay bricks were assessed. Surface roughness was qualitatively evaluated by observation with Research and Metallurgical Microscopy with a model of LH100-1made in china.

Similarly to assess the property of the clay brick in relation to the chemical composition in the sample raw material XRD data was collected using XRD-7000 Drawell made by china ,CuKα at 30Kv, 25mA source, with source λ=1.54A at 2θ range of $15^0$to 90^0 and step size of 0.15. Finally, the data found from XRD was analyzed using Origin 16 to identify the peaks of the diffractogram. Moreover, for identifying the expected chemical composition Analytical Xpert high score plus software has been used.

3.2.2 Physical test

3.2.2.1 Firing shrinkage Test

Firing shrinkage test was conducted to determine the shrinkage property of the prepared sample. The quality of brick can be further assured according to the degree of firing shrinkage. In the research findings, the firing temperature and time used are 900^0C, 1000^0C, 1100^0C and three-hour stay with 20min/^{0}c heating and cooling rate.

Shrinkage was obtained by measuring the Volume of the sample before and after drying or before and after firing.

$$\textbf{Firing shrinkage}(\%) = \frac{V_{dried} - V_{fired}}{V_{dried}.} x100$$

Where; V_{dried}– is the volume of dried sample before fired, V_{fired} – is the volume of dried and fired sample.

3.2.2.2 Weight loss on ignition Test

Weight loss on ignition was calculated from weight of bricks before firing and after firing measured by electronic digital beam balance. This value is important to indicate the loss during the firing of the fired-clay brick

$$\textbf{Weight loss on ignition} = \frac{W_{bf} - W_{af}}{W_{bf}}$$

Where W_{bf}- is the weight of the specimen before firing and W_{af}- the weight of the specimen after firing [58].

3.2.2.3 Water absorption Test

Water absorption test was performed mainly to measure the percentage of water absorption by the brick. In the process the sample was weighed before inserted to the water and then the sample was placed in the water for a period of (24 hours) and the weight was measured finally the percentage of water absorption was calculated using formula shown below and the data was presented in chapter 4.

$$\boldsymbol{Water\ Absorbion} = \frac{\boldsymbol{W_{asw} - W_{bsw}}}{\boldsymbol{W_{asw}}}$$

Where $\boldsymbol{W_{asw}}$- is weight of sample after soaking in water for 24hr, $\boldsymbol{W_{bsw}}$- is weight of the sample before soaking in water for 24hr.

3.2.2.4 Efflorescence Test

The presence of alkalis in bricks is harmful and they form a grey or white layer on brick surface by absorbing moisture. To find out the presence of alkalis in bricks this test performed. In this test a brick is immersed in fresh water for 24 hours and then it's taken out from water and allowed to dry in shade.

3.2.3 Mechanical Strength Test

To ensure the engineering quality of a material, especially for building construction use, mechanical testing is the essential criteria. The compression or bending strength are represent for mechanical properties. The compressive strength was measured for brick samples according to ASTM C67[59].

3.2.3.1 Compressive Strength

Compressive strength is the ability of material or structure to carry the loads on its surface without any crack or deflection. Compressive strength depends on many factors such as bricks surface, strength, quality and quality control during production of bricks. Compressive strength for any material is the load applied at the point of failure to the cross section area of the face on which load was applied.

$$\boldsymbol{Compressive\ strength}\ (\boldsymbol{MPa}) = \frac{\mathbf{Load.Max(N)}}{\mathbf{Area}\ (\boldsymbol{mm^2})}$$

3.3 Experimental Design

This experiment was designed to investigate the effect of firing temperature and iron slag addition on the mechanical strength, water absorption micro structural, shrinkage and Efflorescence of the clay bricks. The schematic diagram of the experimental design also shown below.

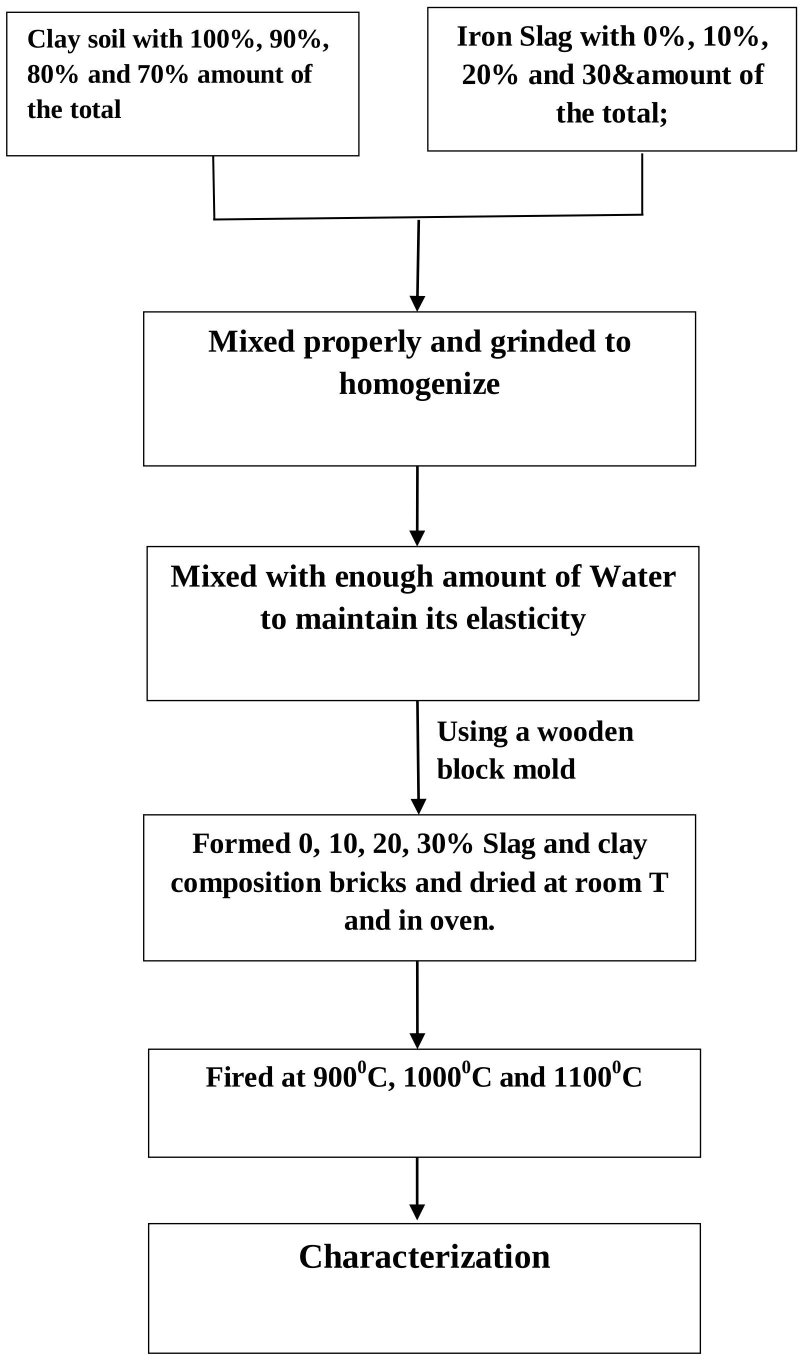

Figure 3.1 Schematic Diagram of the Experimental Design

CHAPTER FOUR

4. Result and Discussion

4.1 Introduction

The result of synthesized clay brick with variable parameters of temperature and slag to Clay soil ratio has been analyzed in this part. In this experiment three samples for each has been characterized for most of the physical characterization part and average value of the result were taken for this discussion part. Generally, this chapter is composed of the result and discussion of the characterization of the morphological and Mineralogical data, Mechanical strength (compressive strength) data, and physical characteristic data of the sample brick.

4.2 Morphological and Mineralogical composition of the bricks

4.2.1 Morphology of the bricks

Morphology of brick samples was assessed by Research and metallurgical Microscopy after etching the sample and the results are shown in figure 4.1 below

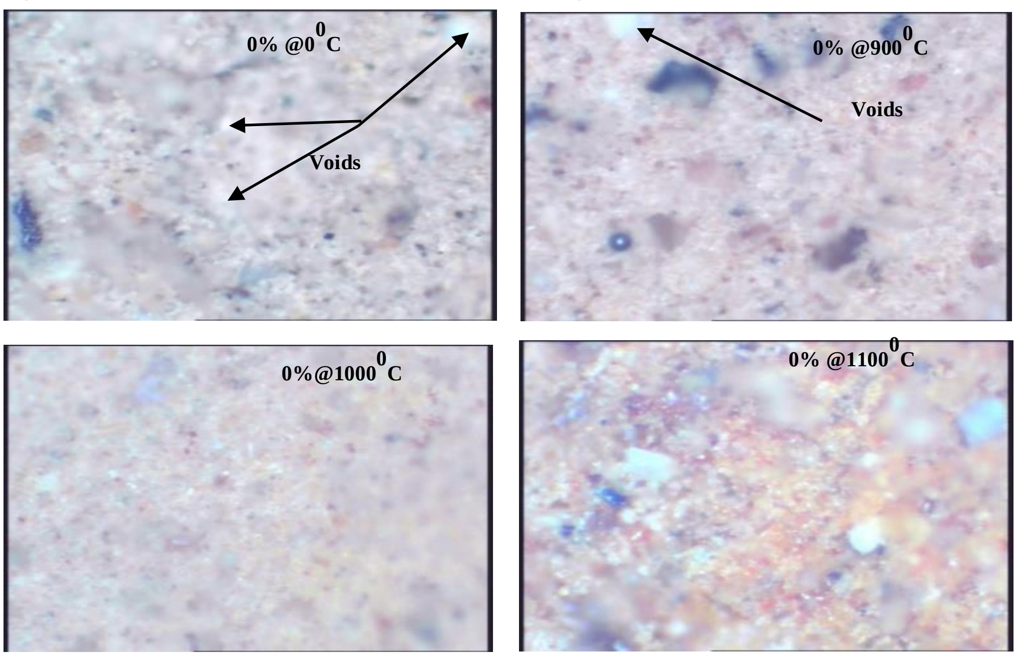

Figure 4.1: The Research and Metallurgical Microscopy pictogram of Pure Clay before and at 900, 1000 and 1100^{0}C firing

The Microscopic picture of clay brick synthesized without adding iron slag is presented in figure 4.1. The picture clearly indicates the sample create void at lower temperature as well as before firing. As the firing temperature increases, the number of void on the sample decreases and sample looks more of uniform appearance, because at higher temperature some particles in the clay soil which cannot withstand higher temperature dissociate and fill the void part as well as the particles gets compacted[60]. At the same time, the microstructure of the brick when iron slag was added at different percentage is presented Figure 4.2

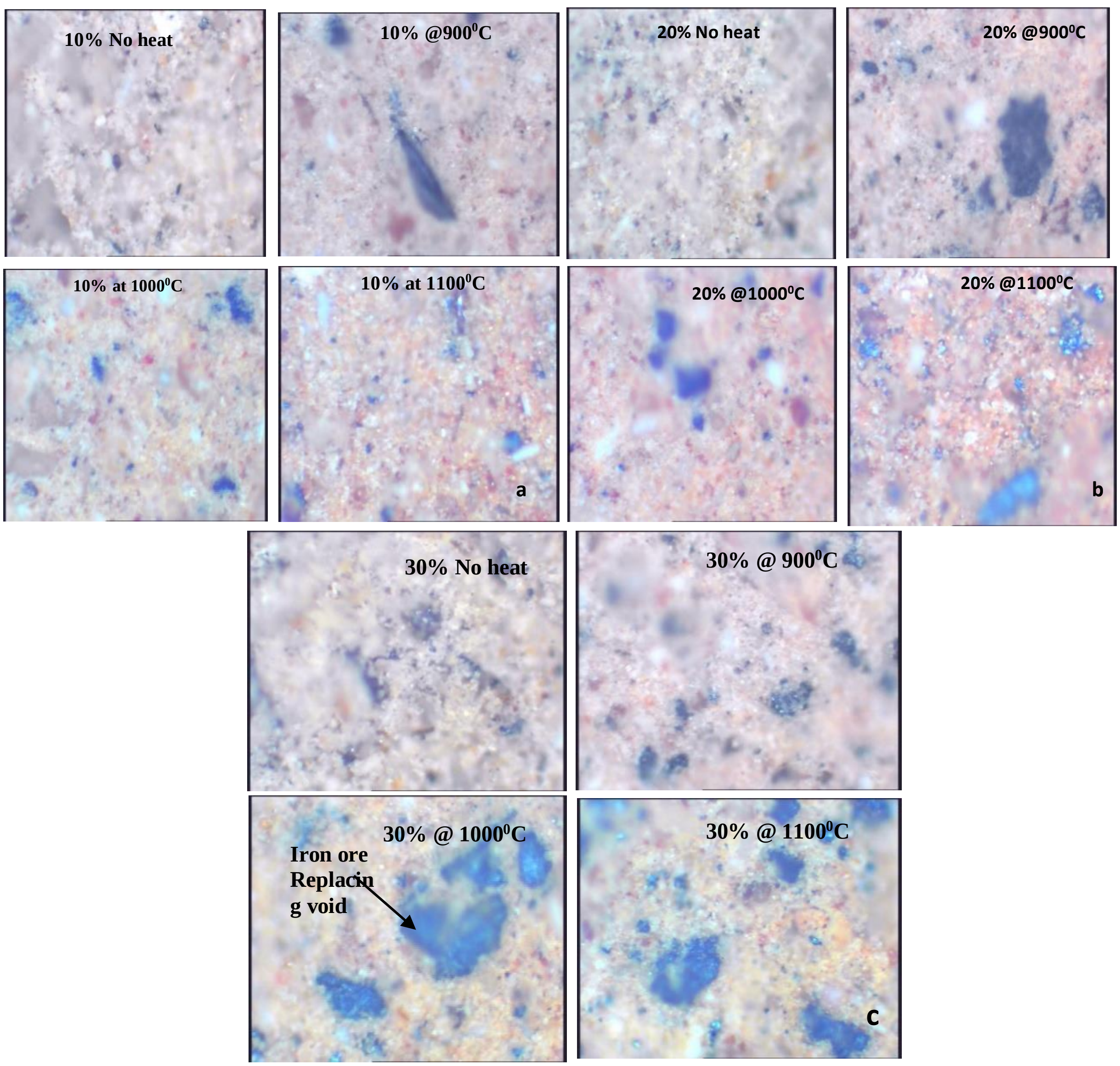

Figure 4.2: The Research and metallurgical Microscopy picture of (a) 10% (b) 20% and (c) 30% iron slag added clays after firing

The inclusion of iron slag enable to fill part of the void at the brick uniformly before firing, but when it is fired the particles in the clay soil get burned and part of the void will be filled by the slag as it appears black in color in figure 4.2. Hence as the amount of iron slag added increases, the voids in the clay brick will be filled as shown in Figure 4.2 c. (30% slag fired at 1100^0C).

4.2.2 Mineralogical composition of the clay and Iron slag

Qualitative determination of major crystalline mineralogical phases present in the clay and iron slag were achieved by using X-ray diffractometer. The results were analyzed using Origin software and is presented in the figure 4.3 shown below

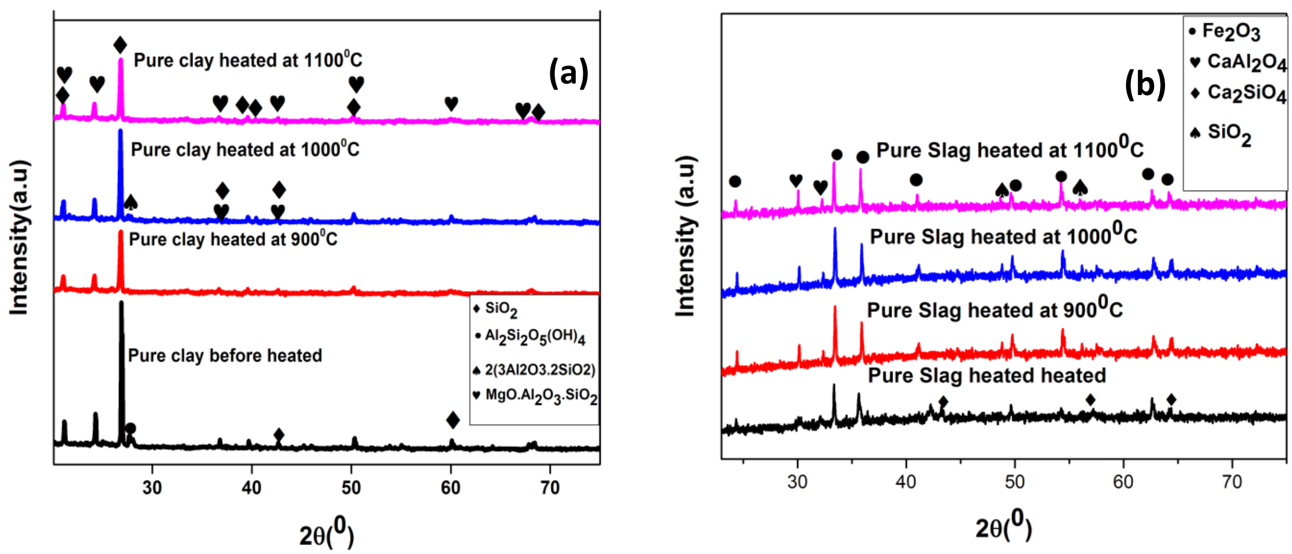

Figure 4.3: XRD Pattern of (a) Pure Clay (b) Pure slag fired at different temperature

The Mineralogical phases present in the powder form of the bare clayshown in Figure 4.3(a) indicates that the sample have almost the same peak at all firing temperature, except some peak which extinct as the temperature raises. From Xpert HighScore result the majority of the peaks are identified as quartz (SiO_2) and other minerals $CaSiO_8$, $NaAlSi_3O_8$(feldspar) and $Mg.Al_2O_3.SiO_2$,are found in it which is in agreement with literature[61].The mineralogical composition of pure slag in figure 4.3 (b)also identified as iron oxide as a major component and Ca_2SiO_4 and $CaAl_2O_4$ together.

On the other hand, Figure 4.3 (a) and (b) indicates, that the change in temperature have no effect on the hematite peak specially in figure 4.3(b), but the other minerals peak like Ca_2SiO_4 and

$Ca_2Al_2O_3$ peak decreases. This is an indication of the formation of Kaolinite $(Al_2Si_2O_3(OH)_4$ and quartz (SiO_2)after $Ca_2SiO_4$and $CaAl_2O_4$ decomposes[23].Besides, in Figure 4.3 (a) the peak intensity of quartz (SiO_2)decreases as the firing temperature increases, which is in agreement with [62].

4.2.2.1 Effect of Temperature on the Mineralogy of composite clay brick

The effect of firing temperature on the composite clay brick can help us to examine the effect of firing temperature on the mineralogy of the composite brick. Figures 4.4 and 4.5 indicates the XRD pattern of the composite sample.

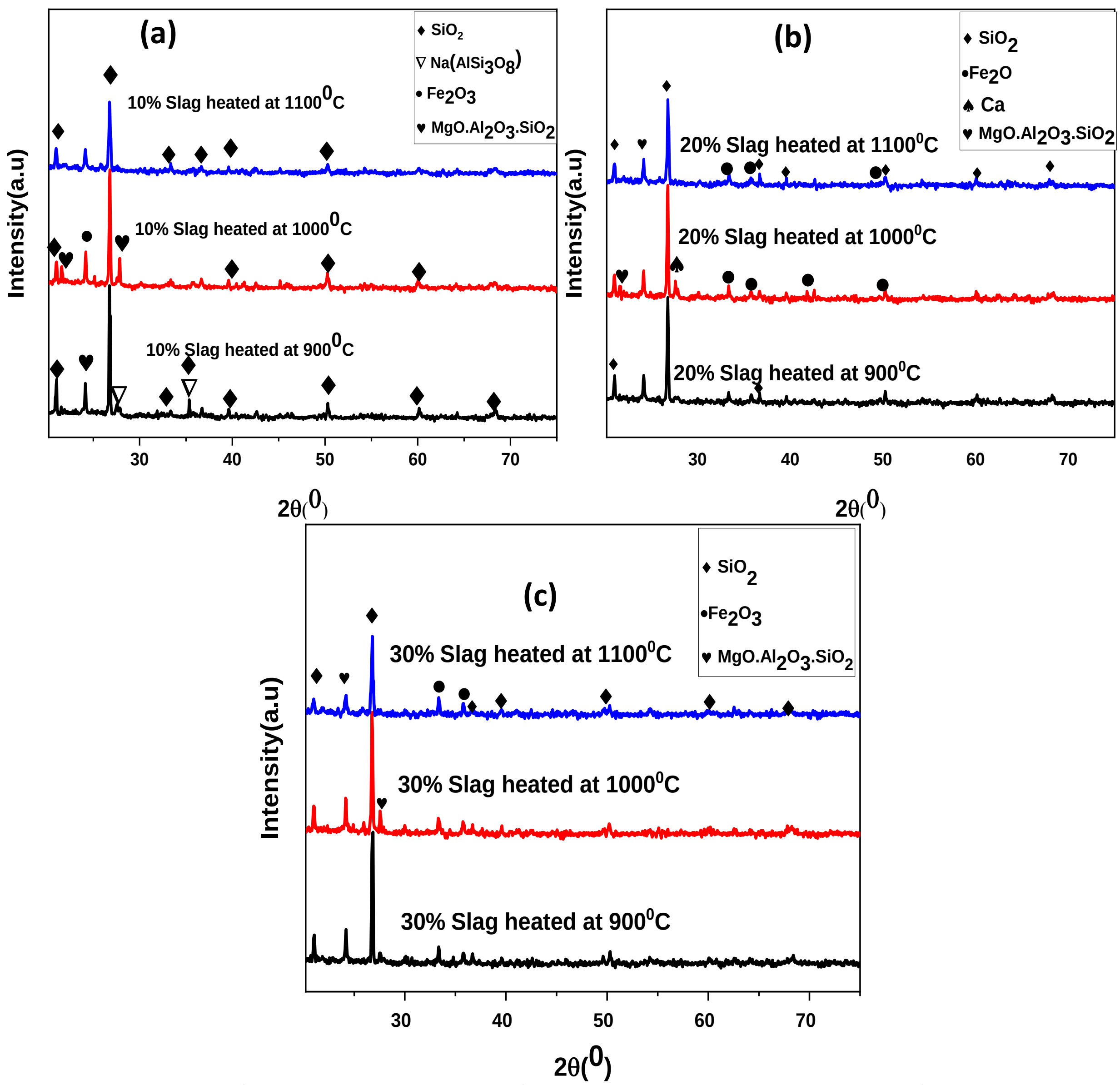

Figure 4.4: XRD of Variable Temperature for (a) 10% (b)20% and (c) 30% of slag

Figure 4.4 also clearly indicates the amount of quartz (SiO_2) peak intensity decreases with the increase in temperature, but the hematite peak remains unchanged with temperature variation. This result confirm that the effect of temperature on the transformation of hematite to other forms within the given temperature gap is negligible i.e. hematite is not transformed to other phase at this temperature range.

4.2.2.2 Effect of Iron Slag on the Mineralogy of composite clay brick

The X-ray diffraction pattern of the clay brick at fixed temperatures with a temperature of 900^0C, 1000^0C and 1100^0C independently while the iron slag mixing ratio varying is presented in the figure 4.4 and Figure 4.5.

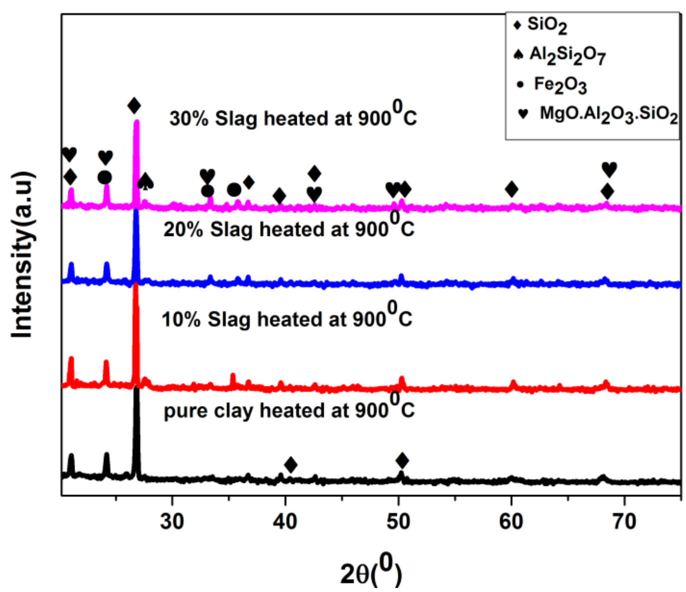

Figure 4.5: XRD Pattern of brick composed of pure clay and 10%, 20% and 30% slags fired at 900^0C

Addition of iron slag on the clay brick shown Figure 4.4 conserves the amount of SiO_2 in the sample, this is due to the contribution from iron slag. Moreover the peak of hematite in the clay sample under investigation at 900^0C shows the same at all concentration.

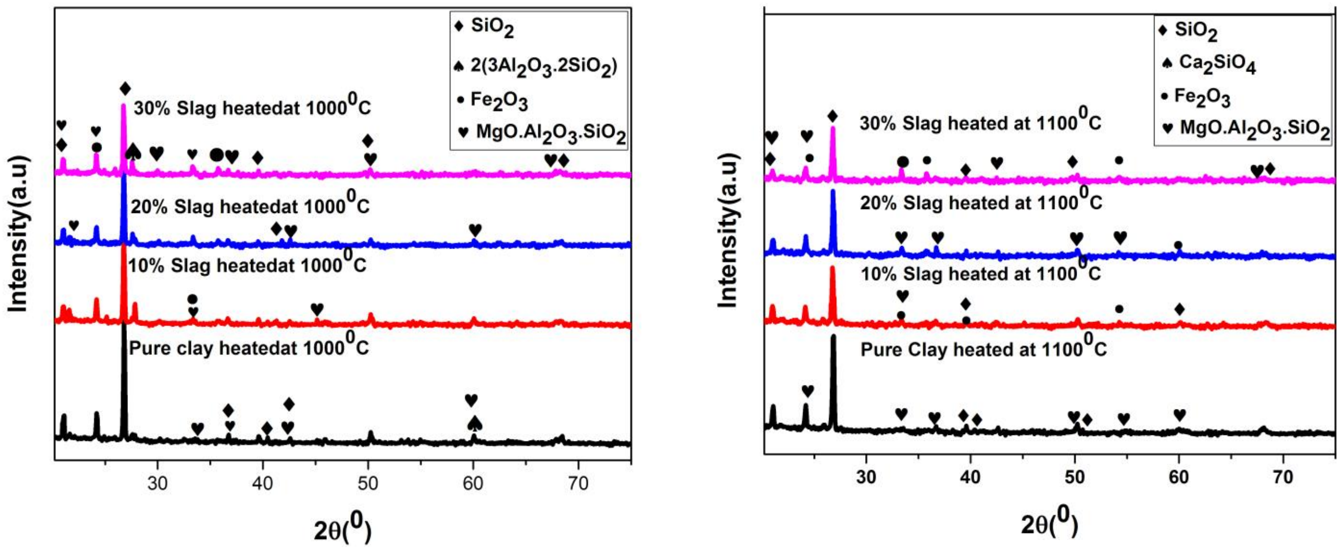

Figure 4.6:XRD Pattern of Clay brick composed of pure clay and 10%, 20% and 30% slags fired (a) at 1000^0C and (b) at 1100^0C.

Figure 4.6 (a) and (b) confirm that the peaks of hematite at elevated temperature remains unchanged. likewise, the peaks of quartz remain unchanged, this happened because the addition of iron slag and increment in temperature favor the replacement of those decomposedquartz in the sample. The two diagram (figure 4.5 and figure 4.6) also confirmed that addition of iron slag in combination with raising the firing temperature favor the formation of Kaolinite and quartz, which are the most important minerals for ceramic materials. Finally, The presence of kaolinite ($Al_2Si_2O_3(OH)_4$) and residual forms of quart and cristobite (SiO_2) indicated that these samples will have good mechanical and physico-sintering properties[63].

4.2.3Chemical Composition Microstructure of the Bricks

The chemical composition of slag has been identified using XRF shown in table 4.1 and that of clay soil was taken from literature.

Table 4.1 chemical composition of Iron slag as Identified by XRF.

Elements	**Al**	**Si**	**Ti**	**Ca**	**Fe**	**Na**	**S**	**Mn**	**Cr**
Percentage	8.31	13.64	1.01	1.58	58.3	0.39	0.06	10.60	6.11

Table 4.1 clearly indicate that the sample slag contain alumina, silica and iron which is responsible for the strength of ceramic materials. Moreover literatures indicate that most clays are composed of Kaolinite/Mullite, hematite and quartz[64, 65].

4.3 Physical Property of the sample

Physical properties of the clay bricks are the mainparameter that relies on the chemical composition, microstructure and crystalline property to determine the mechanical property of the bricks. Physical properties include; Firing shrinkage, loss on ignition, water absorption and the like[66] were investigated.

4.3.1 Firing Shrinkage:

It was determined by measuring the dimensions of the sample either before and after drying or before and after firing. During firing, especially during sintering at high temperatures, ceramic particles fuse together leading to greater proximity and thus enhancing linear shrinkage. It is considered that the reduction in firing shrinkage of brick material had a positive impact. But large shrinkage could create problems as it may cause cracks and dimensional defects.The resultsof the volume of the brick before fire and after firing are also shown in table 4.2,

Table 4.2: Volume of the sample before and after Firing

Types of bricks	**Volume of bricks before firing (cm^3)**	**Volume of bricks after firing (cm^3)**		
		900C 3hr	**1000C 3hr**	**1100C 3hr**
0%slag	387	370.4	369.6	368.4
10%slag	397	382.7	381.1	380.7
20%slag	400	393.6	392.8	392.4
30%slag	414	409.0	408.6	408.6

The results in table 4.2 shows that when the firing temperature increases, the volume of the sample brick decreases, this indicates there is a volume shrinkage during firing process. Furthermore, the weight loss of the sample brick is relatively maximum in the bare clay brick which is before firing was 387cm^3 and shrink to 370.4cm^3 at 900^{0}C, 369.6cm^3 at 1000^{0}C and 368.4cm^3 at 1100^{0}C. Whereas it attains the minimum shrinkage for the sample brick with 30% slag added. The interpreted percentage of firing shrinkage of the sample is presented in table 4.3.

Table 4.3 Percentage of firing shrinkage of the sample

Bricks	Percentage of Firing shrinkage		
	900c 3hr	**1000c 3hr**	**1100c 3hr**
0%	4.3	4.5	4.8
10%	3.6	4.0	4.1
20%	1.6	1.8	1.9
30%	1.2	1.3	1.3

Table 4.3 indicates the percentage of firing shrinkage increases with increase in firing temperature. The addition of the slag on the clay brick also improves the firing shrinkage of the sample. Based on the report of (weng, et.al 2003 and Chuanmeng Yang, et.al 2014) for a brick to be in good condition, the shrinkage of the bricks should be below 8%[62, 67].

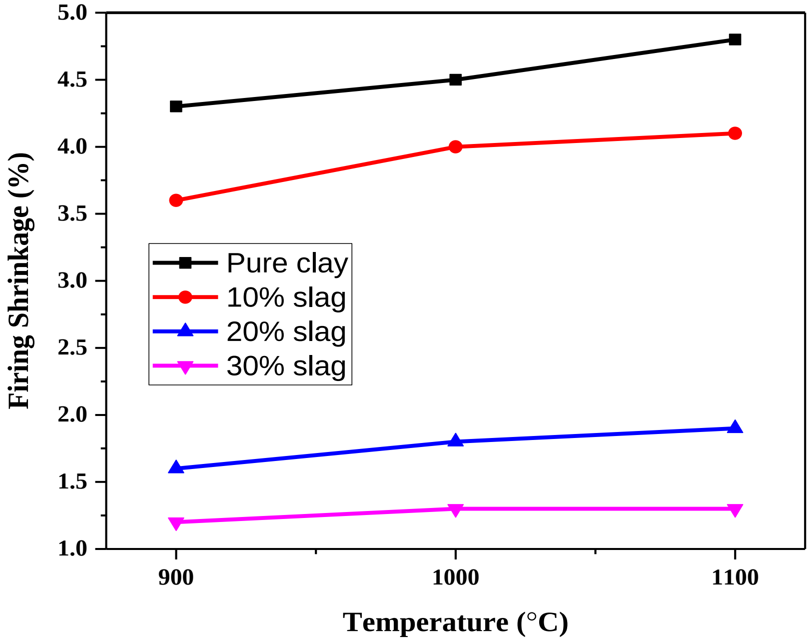

Figure 4.7 Fire shrinkage of the samples fired at three different temperature

The fire shrinkage shown in figure 4.7is also consistence with the theoretical values, that means a sample having more iron slag shrink less as temperature increases than the one with less content as well as the bare clay soil brick.

4.3.2 Weight loss on Ignition

The Loss on Ignition is used to determine the percentage of moisture loss to ignition on firing the prepared clay samples. It is determined by measuring the mass loss of the sample between the drying and firing steps. During the firing process, the organic and other volatile matter in the mix evaporated. The result of the weight of loss on ignition is presented in Table 4.4

Table 4.4 Weight of clay and iron slag clay composition bricks before and after firing.

Bricks	Weight before firing (gm.)	Weight after firing (gm.)		
		900^0C 3hr	1000^0C 3hr	1100^0C 3hr
Pure	613	586	578	555
10%slag	673	664	655	623
20%slag	702	674	682	683
30%slag	750	712	714	734

Table 4.4 also indicate that the weight loss increases with the increase in firing temperature. But in the case of 20% and 30% slag addition the weight gained by the clay as the temperature increase above 900^0C, that means when the clay brick has 20% slag its weight increase from 682gm to 683gm as the temperature raises from 1000^0C to 1100^0C. Similarly in the case of clay brick with 30% slag content the weight increases from 714gm to 734gm as its temperature increases from 1000^0C to 1100^0C. The weight loss in ignition of the clay soil brick for the three temperatures and other mixed clay bricks at the lower temperature are attributed to the dihydroxylation and carbonated decomposition [66].

The percentage of weight loss on ignition also listed in Table 4.5 below

Table 4.5: Percentage of weight loss on ignition

Bricks	Percentage of weight loss on ignition		
	900c 3hr	1000c 3hr	1100c 3hr
Pure	4.4	5.7	9.5
10%slag	1.3	2.7	7.4
20%slag	4	2.8	2.7
30%slag	5.1	4.8	2.1

The percentage of weight loss on ignition listed on Table 4.5 indicates that it is higher for bare clay bricks at 1100^0C, which is 9.5% while the least one is registered in the clay bricks at 30% percentage added clay brick, which is 2.1%. Literature recommended that the weight loss on ignition of sample clay below 15% considered as good bricks[64]. Moreover the weight loss on ignition improved by 15.7% at 1000^0C whereas at 1100^0C by 77.9% when 30% of slag is added to the clay.

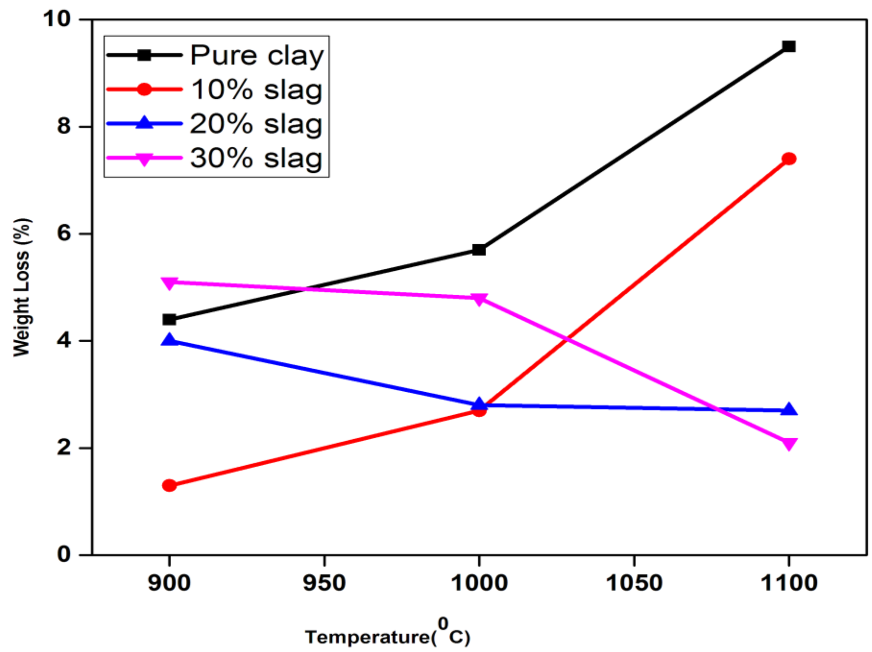

Figure 4.8: Temperature Vs Weight Loss on Ignition of the samples

The weight loss on ignition of the clay brick sample in figure 4.8 shows that pure clay has a maximum weight loss than the other while the one with higher concentration of slag is less. This is in agreement with the theory, because addition of slag can fill the void and the brick will loss its weight less.

4.3.3 Water Absorption

Water absorption is an important factor for the durability of clay bricks. When water infiltrates brick, it decreases the durability of brick. Thus, the internal structure of brick must be sufficiently dense to void the leaking of water.

Table 4.6: Water absorption before and after firing and the corresponding percentage

Bricks	Weight before immersed in water in (gm.)			Weight of bricks after immersed in water in (gm.)			Percentage of water absorption		
	900^0C	**1000^0C**	**1100^0C**	**900^0C**	**1000^0C**	**1100^0C**	**900^0C**	**1000^0C**	**1100^0C**
Pure clay	586	578	555	718	704	657	22.5	21.8	18.4
10% s	664	655	623	805	785	706	21.2	19.8	13.3
20%s	674	682	683	803	808	773	19.1	18.5	13.1
30%s	712	714	734	832	830	824	16.9	16.2	12.3

The water absorption of the pure clay brick is maximum at the lowest temperature, this is due to the formation of more voids. When the firing temperature increases the clay brick becomes more compact than the former, as a result the water intake decreases. This result is also consistence with the shrinkage result shown in table 4.6 that means as the firing temperature increase the materials become more compact. On the other hand, as the amount of slag added to the clay increases the amount of water absorbed by the sample decreases, which means most of the voids are substituted by the slag.

4.3.4 Efflorescence Test

The Efflorescence test is the most important test for assuring the engineering quality and durability of a building material because, when the brick contain soluble salts on it, it will not last for a long time with its strength. A good quality brick should not contain any soluble salts in it. If soluble salts are there, then it will cause efflorescence on brick surfaces. The results in Figure 4.9 indicated that the sample placed in a water for 24 hours and put in a plastic cover for one week to see the efflorescence effect.

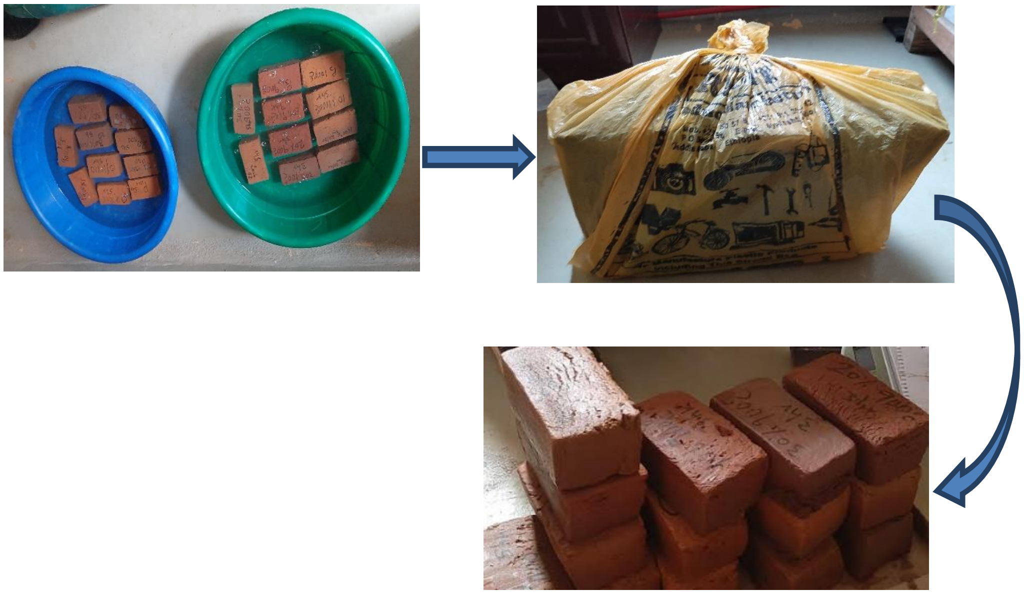

Figure 4.9: Steps showing Efflorescence test of the sample.

Figure 4.9 clearly shows that all the samples under the test of efflorescence shows no sign of a white color on it after one week test. This is an indication that both the clay soil as well as the mixture of slag and clay soil exhibits no salt in it and it is a good candidate for construction clay brick.

4.4 Mechanical Strength

Mechanical strength of the clay brick made be controlled by calcination temperature and clay soil to iron slag ratio refers the compressive strength of the brick. The compressive strength of the sintered samples is a key factor in considering their application as construction bricks. It is the most critical engineering properties for building materials.

Compressive strength is the ability of material or structure to carry the loads on its surface without any crack or deflection. The amount of load applied on the sample per cross-section of all the compositions at various firing temperatures were presented in table 4.7

Table 4.7: Average Maximum load bearing capacity of the cross section of specimen

Average Maximum load applied over the cross-section of specimen						
	900c 3hr		**1000c 3hr**		**1100 3hr**	
	L (N)	**A(mm^2)**	**L (N)**	**A(mm^2)**	**L (N)**	**A(mm^2)**
0% slag	59717	6786	90432	6669	123691	6686
10% slag	78495	6669	122312	6702	123977	6644
20% slag	86418	6762	123180	6644	140968	6612
30% slag	90827	6596	126178	6669	147448	6612

The data in Table 4.7 indicates, the average maximum load bearing capacity of the specimen made with controlled iron slag addition and fired with different temperature having almost similar cross sectional area. The result roughly indicates the load carrying capacity increases with increment in both factors; an increase in temperature and percentage of iron slag. But the data does not express the load bearing capacity in corresponding with the cross section since the cross-section of the specimen that receive the load varies slightly, hence it requires the compressive strength data. For that matter table 4.8 presents the calculated value of the Compressive strength of each specimen.

Table 4.8: Compressive strength of the sample

Bricks	**Compressive strength (MPa) of bricks at different Temperature**		
	900^{0}C 3hr	**1000^{0}C 3hr**	**1100^{0}C 3hr**
0% slag	8.80	13.56	18.50
10% slag	11.77	18.25	18.66
20% slag	12.78	18.54	21.32
30% slag	13.77	18.92	22.30

The calculated data presented in Table 4.8 indicates, the compressive strength of each specimen increases with an increase in temperature. That means a pure clay brick has a compressive strength of 8.8MPa, 13.56MPa and 18.50MPa at a temperature of 900^{0}C, 1000^{0}C and 1100^{0}C respectively. Moreover, the percentage addition of iron slag at the same temperature also raises the compressive strength of the sample clay.

The compressive strength of each specimen with respect to the firing temperature also presented in figure 4.10

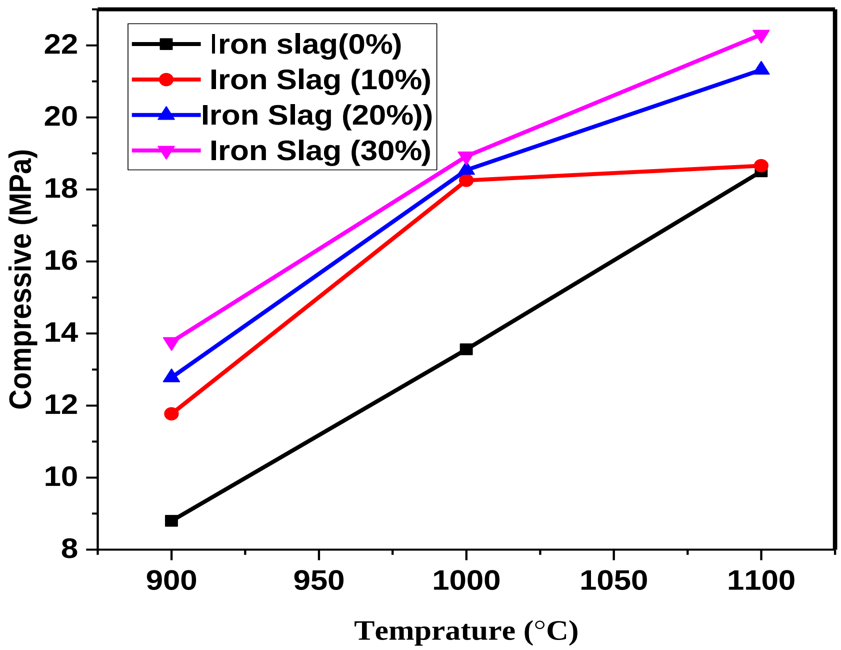

Figure 4.10: Compressive Strength (MPa) Vs Temperature (^{0}C) of sample specimen

The graph in figure 4.10 also confirmed that the compressive strength of all the sample increase with increasing temperature, but the increment in the pure clay looks linear with temperature. Similarly, the compressive strength of the pure clay is lower than those slag added specimens. This happened because, the slag added fill the void formed in the clay brick when the firing temperature increases.

CHAPTER FIVE

5. Conclusion and Recommendation

5.1 Conclusion

In this work, a clay soil taken from Jimma town, Frustale Kebele and Iron slag from Steely RMI, Bishoftu Ethiopia was used as raw material for the manufacture of clay bricks. Based on the result and discussion the physical property like, firing shrinkage, water absorption and efflorescence, the microstructural and crystalline, like optical microscopy and xrd as well as the mechanical property, like compressive strength has been investigated. The experiment was conducted by considering the variation in temperature and the clay soil to iron-slag ratio. From the experimental result the following points are drawn as main points which can be considered as conclusion.

1. The Pure clay sample brick forms more void at lower temperature than the other. But the addition of iron slag fill the void at the same time increase in firing temperature cause the brick to shrink as a result it minimize the void.
2. The XRD data confirm that the amount of SiO_2 (quartz) in the pure clay brick decreases as the firing temperature increases. But addition of slag to the clay brick favor the formation of this quartz (SiO_2) and kaolinite ($Al_2S_2O_3(OH)_4$) from the dissociation of Ca2SiOand $CaAl_2O$ at higher temperature.
3. Hematite, which is ideal for the mechanical property of the clay brick remain unaffected at higher temperature, this confirms there is no phase transformation of iron oxide within the range of 900^0C and 1100^0C
4. The firing shrinkage of the sample bricks fulfil the criteria because all are below the standard (8%), but maximum shrinkage is registered for pure clay brick and the firing shrinkage of 30% slag remain unchanged above 1000^0C.
5. The weight loss on ignition of all sample is also in the acceptable range. That is below 15%.
6. The pure clay brick at lower temperature absorbs more water than the other, it is consistent with the theory, and the more porous the material is the more the water it absorbs.

7. The result of the efflorescence test also support the mineralogical investigation of the XRD pattern, that means it shows no salts that could deteriorate the clay brick and decrease the life of the buildings.
8. Finally, the mechanical strength (compressive strength) of the sample is also consistent with all investigations. But the pure clay fired at 900^0C does not fulfil the standard as its compressive value is below 10MPa.
9. Even if addition of the slag improves the mechanical strength of the brick, addition of 30% slag has no effect on weight loss on ignition and firing shrinkage percentage above 1000^0C.

5.2 Recommendation and Future works

Further research work is recommended to be carried out in order to evaluate the potential use of iron slag in the clay brick by taking wide range of firing temperature and taking firing time into consideration. Moreover proper investigation of the chemical composition of clay soil is recommended to get the exact mineralogical composition quantitatively.

Finally further study is required on the clay brick to investigate the effect of different drying mechanisms, the effect of acidic environment in combination with iron slag addition.

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Appendices

Appendix I: Laboratory Results

1.1 XRD Result graphs

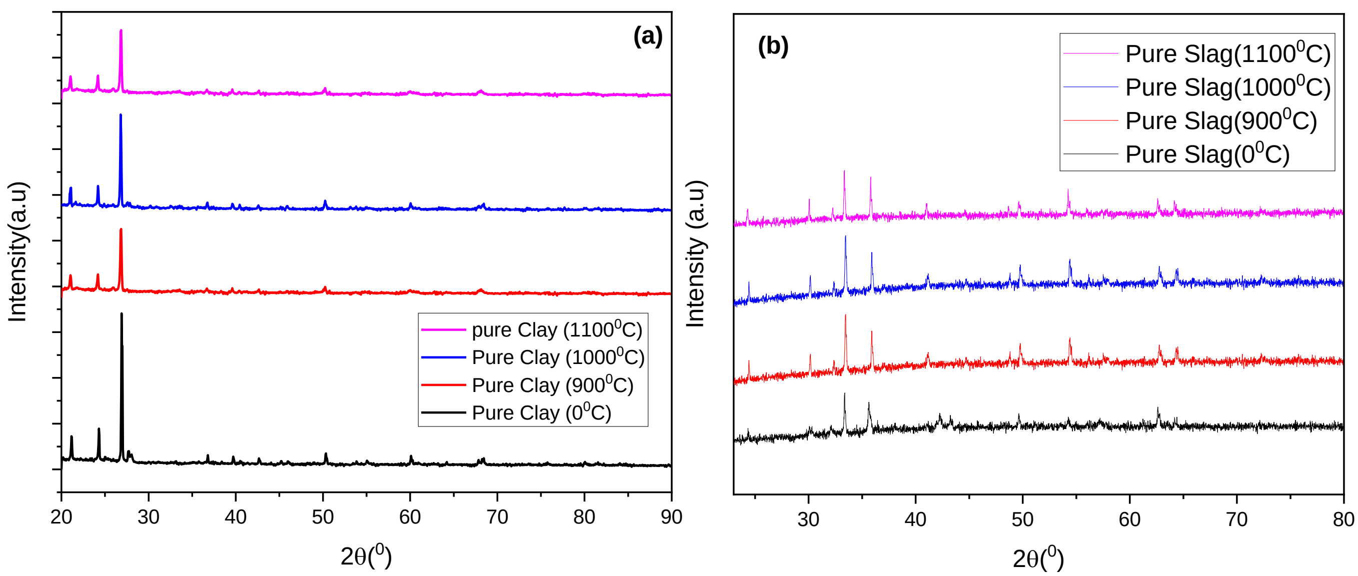

Figure A-1: The XRD data of (a) pure clay and (b) pure slag treated at four Different Temperature.

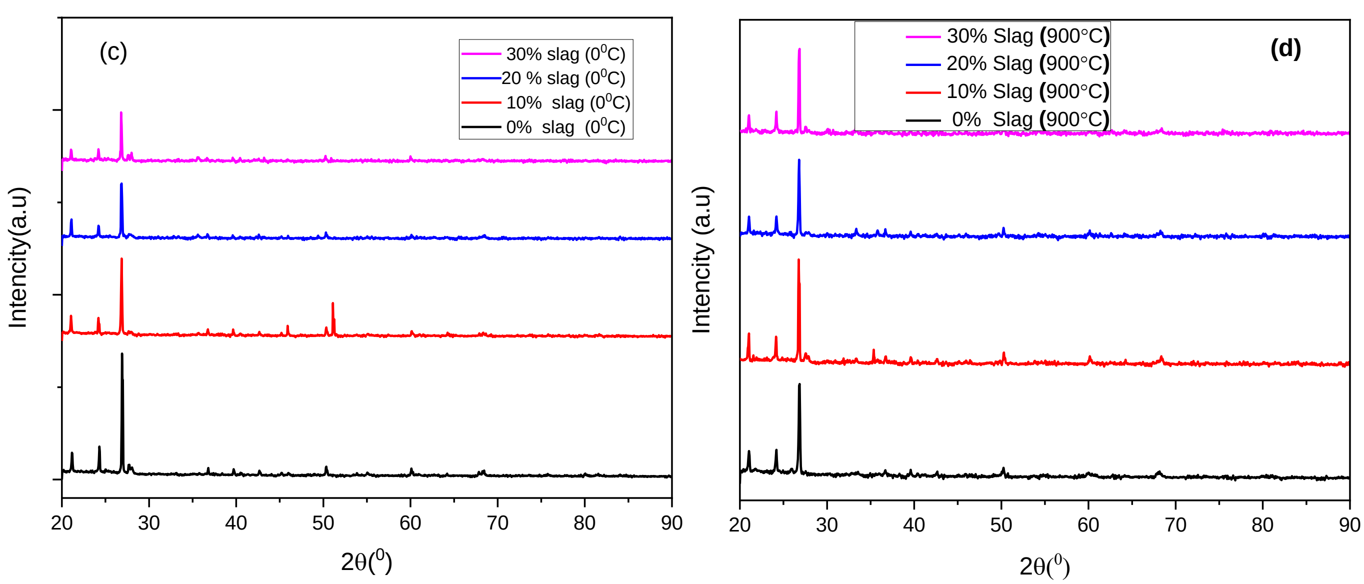

Figure A-2: The XRD graph of clay with (c) different concentration of slag with no firing

(d) different proportion of slag firing at 900^0C

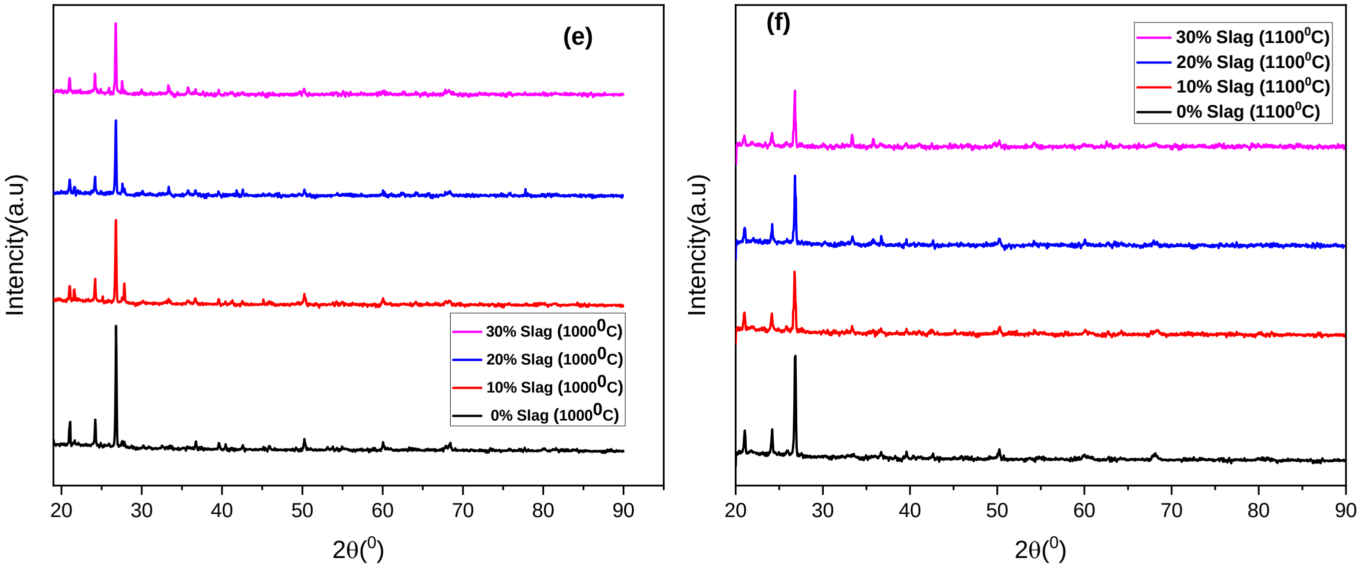

Figure A-3: The XRD graph of clay with (c) different concentration of slag firing at 1000^0C and (d) different proportion of slag firing at 1100^0C

1.2 Optical Microscopy Results

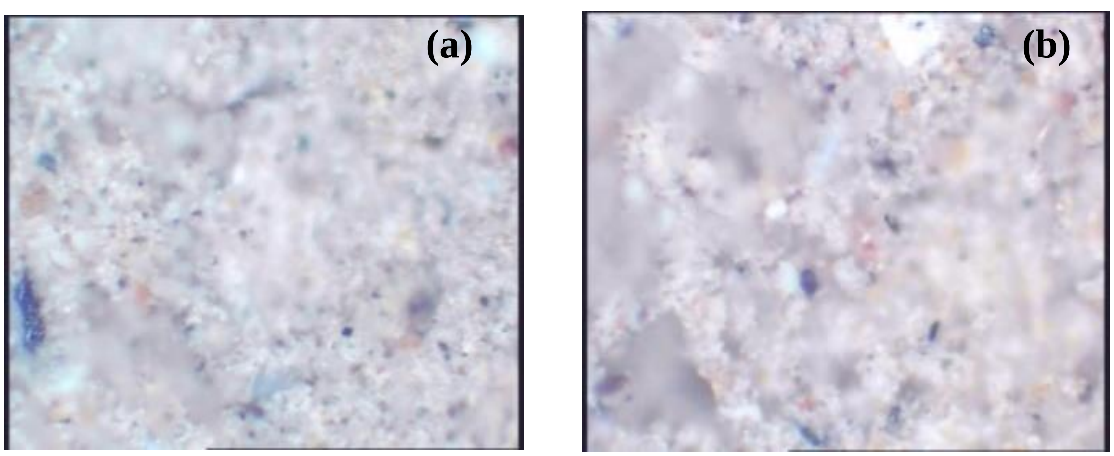

Figure B-1: Optical Microscope Image of (a) Pure Clay and (b) 10% slag added clay without firing

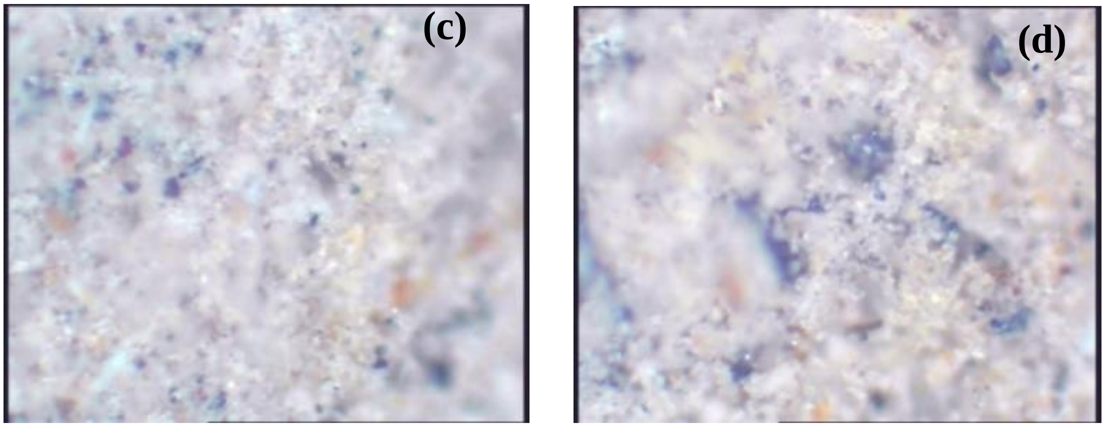

Figure B-2: Optical Microscope Image of (c) 20%slag and (d) 30% slag added clay without firing

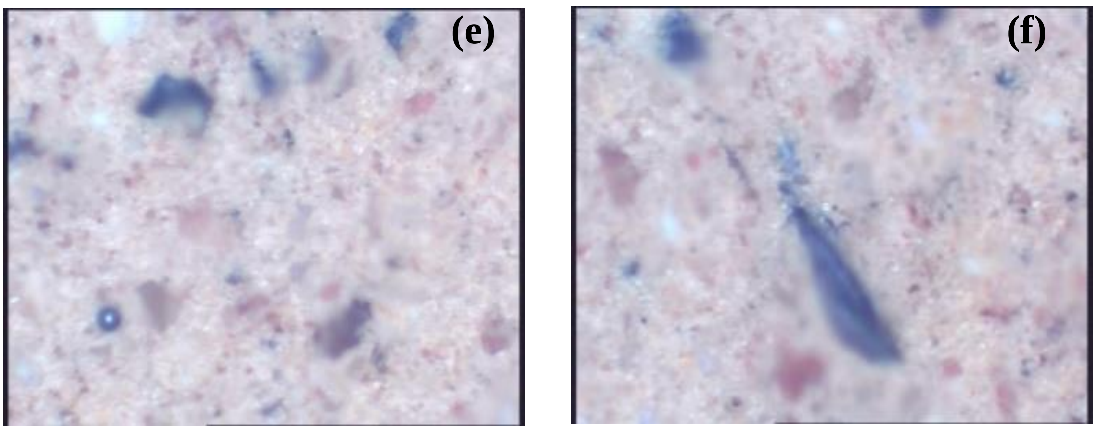

Figure B-3: Optical Microscope Image of (e) 0%slag and (f) 10% slag added clay firing with 900^0C

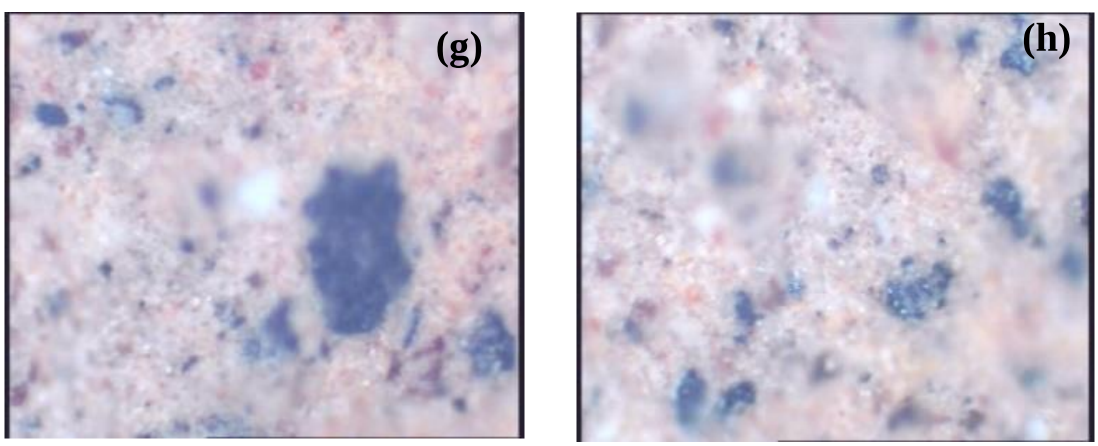

Figure B-4: Optical Microscope Image of (g) 20%slag and (h) 30% slag added clay firing with 900^0C

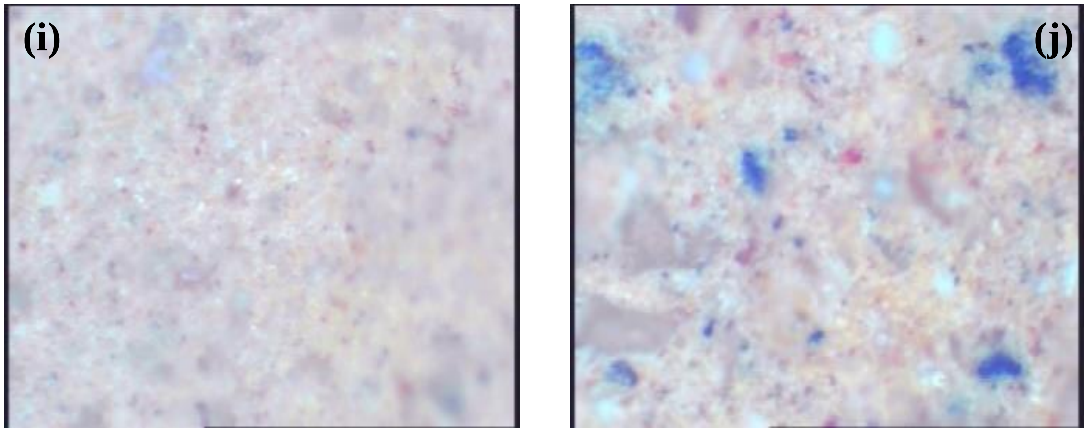

Figure B-5: Optical Microscope Image of (i) 0%slag and (j) 10% slag added clay firing with 1000^0C

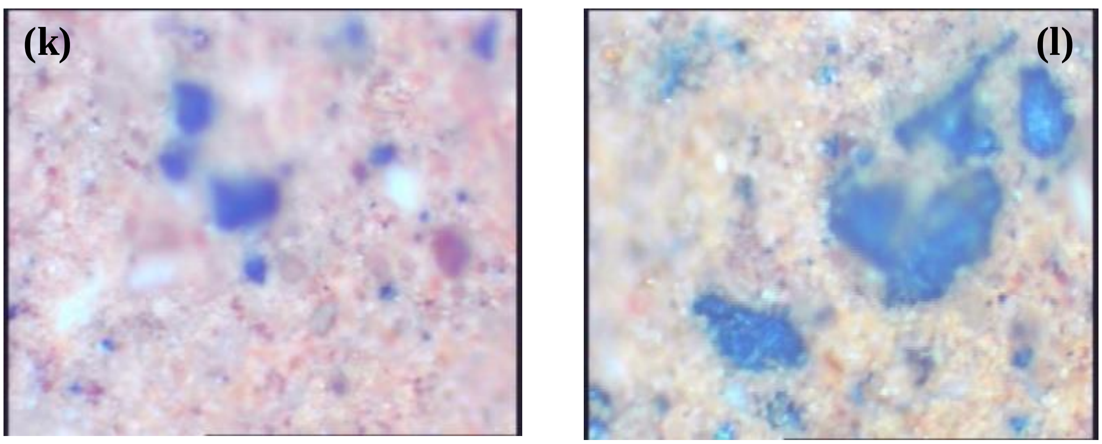

Figure B-6: Optical Microscope Image of (k) 20%slag and (l) 30% slag added clay firing with 1000^0C

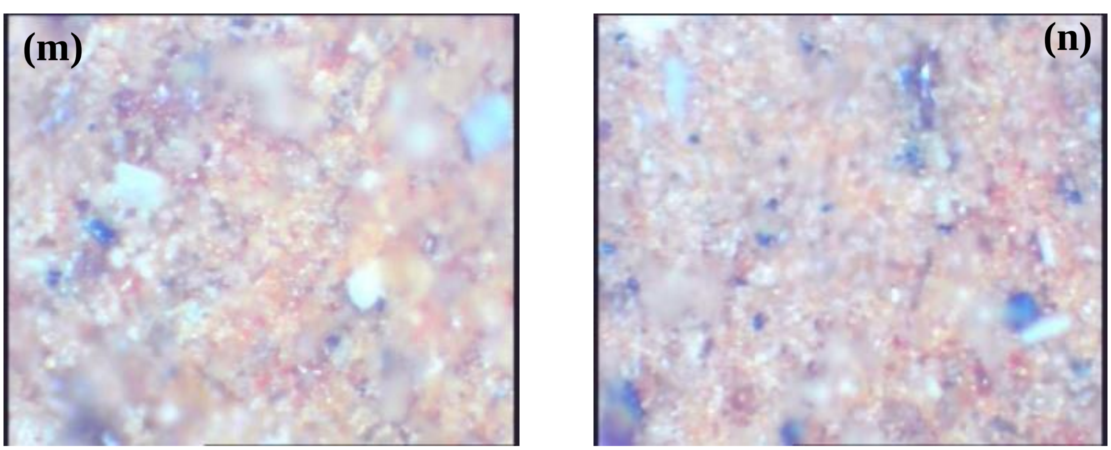

Figure B-7: Optical Microscope Image of (m) 0%slag and (n) 10% slag added clay firing with 1100^0C

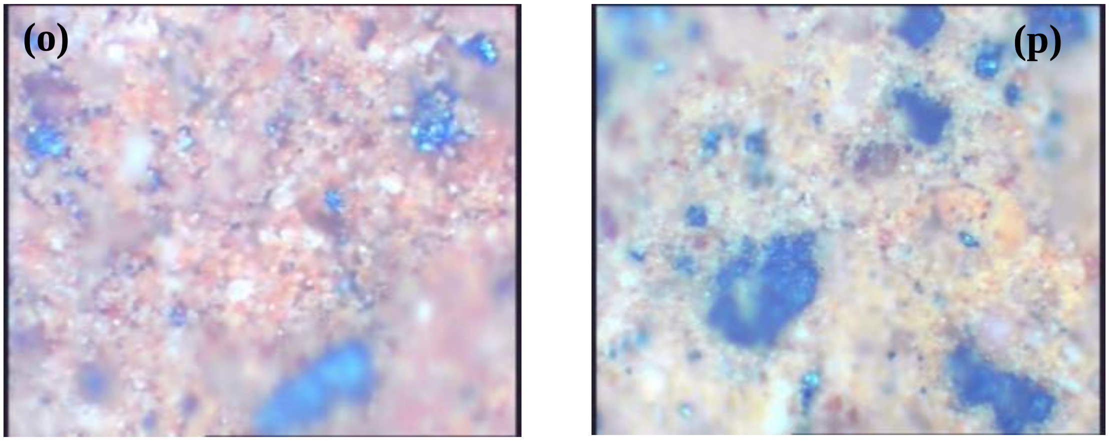

Figure B-8: Optical Microscope Image of (o) 20%slag and (p) 30% slag added clay firing with 1100^0C

1.3 Firing Shrinkage:

$$\textbf{Volume} = \textbf{L*W*T} \quad \textbf{and} \quad \textbf{\textit{Firing Shrinkage}} = \frac{V_{before} - V_{after}}{V_{before}}$$

Table C-1: Data measured to calculate the firing shrinkage

	Before firing			After firing								
				900for3hr			1000for3hr			1100for3hr		
	L	W	T	L	W	T	L	W	T	L	W	T
0%	11.7	5.8	5.7	11.6	5.6	5.7	11.7	5.7	5.54	11.6	5.7	5.57
10%	11.8	5.8	5.8	11.7	5.7	5.74	11.7	5.7	5.71	11.6	5.7	5.76
20%	11.9	5.8	5.8	11.8	5.7	5.85	11.8	5.8	5.74	11.7	5.8	5.78
30%	11.9	5.9	5.9	11.85	5.85	5.89	11.9	5.85	5.87	11.87	5.85	5.88

1.4 Water Absorption

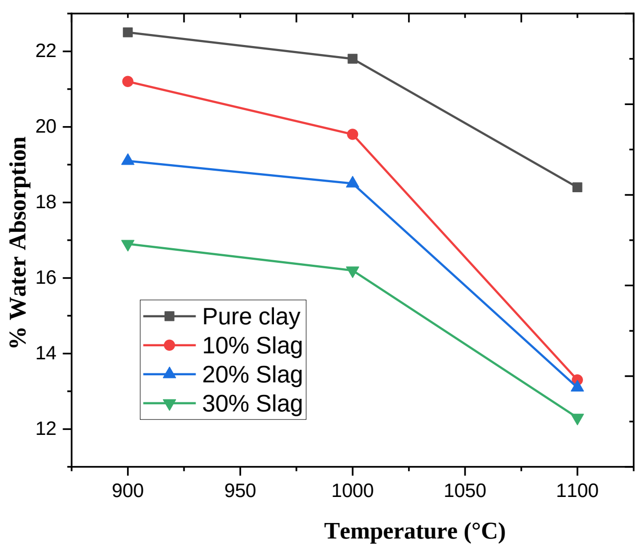

Figure B-9 Water absorption graph

Appendix II: Photographs Showing Laboratory Activities

2.1 Lab Equipment Used In the Research

Figure C-1 Mortar and pestle and Compact Machine

Figure C-2 Digital Balance and Drying Oven

Figure C-3 Electric Furnace Used for firing

2.2 Testing Machine

Figure C-4 Water Absorption Test container and Compressive Testing Machine

Figure C-5 XRD Machine and Research and Metallurgical Optical Microscopy

2.3 Raw Samples and Researcher doing the experiment

Figure D-1: Raw sample of (a) Iron slag at Steely RMI (b) clay soil at Frustalle

Figure D-2 Researcher (a) marking the samples (b) Taking the sample from water absorption test