

ADDIS ABABA UNIVERSITY
ADDIS ABABA INSTITUTE OF TECHNOLOGY
SCHOOL OF CIVIL AND ENVIRONMENTAL ENGINEERING



**TEFF STRAW ASH AS PARTIAL CEMENT
REPLACEMENT MATERIAL**

A Thesis in Structural Engineering

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December 03, 2020

Addis Ababa

A Thesis

Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science in
Structural Engineering

The undersigned have examined the thesis entitled ‘**Teff Straw Ash as partial cement replacement material**’ presented by **Selamawit Woldemichael**, a candidate for the degree of **Master of Science** and hereby certify that it is worthy of acceptance.

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DECLARATION

I, the undersigned declare that this thesis entitled “**Teff Straw Ash as partial cement replacement material**” is my original work, prepared under the guidance of Girma Zerayohannes (Dr.-Ing.). All sources of material used for this research have been dually acknowledged. Moreover, this thesis has not been presented by any other person in this or any other university or Higher Educational Institute for an award of a degree.

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ABSTRACT

Efficient utilization of agricultural residue is the need of today's environment to optimize the production cost of materials with the same nature and use. Teff Straw is one of such an agricultural residue which is available in high amount in Ethiopia but didn't get that much attention. Some researchers have indicated that Brown Teff Straw Ash contains high silica which is a potential agent for pozzolanic reaction. In the present study, heat treatment has been used to extract Teff Straw Ash for the partial replacement of cement.

This paper aims to enhance the compressive strength of cement mortar by partially replacing the cement with amorphous Teff Straw Ash. Experiments were conducted for replacement ratios of 10% and 20% by volume of cement. Teff Straw Ash (TSA) used for this study is prepared at Material Engineering Laboratory of Addis Ababa University Institute of Technology using muffle furnace at combustion temperatures of 600°C, 750°C and 900°C. Physical and chemical analysis tests, such as: XRD, SEM and complete silicate analysis are performed, in this research, to investigate the properties of Teff Straw Ash (TSA).

The results of the mortar cubes have shown that, up to 10% replacement of the ordinary Portland cement by Brown Teff Straw Ash (BTSA) achieved a higher compressive strength at combustion temperature of 600°C, whereas the 20% replacement has shown a lower strength as compared to the control specimen made of cement mortar.

Keywords: Teff Straw Ash (TSA); Compressive strength; XRD; SEM; Complete silicate analysis

ACKNOWLEDGMENTS

First of all, I praise God for giving me the courage and strength to carry out this thesis work in particular.

My sincere gratitude goes to my advisor Dr.-Ing. Girma Zerayohannes for his useful suggestions, kind support, guidance, constant encouragement and valuable comments since the beginning of the study. My gratitude is also extended to Dr. Esayas G/Yohannes, for his valuable comments, kind support, constant encouragement and cooperation throughout the study.

I am very grateful for the kind of cooperation and support provided by the staff members of Material Engineering laboratory of Addis Ababa University Institute of Technology, Chemical Engineering laboratory of Addis Ababa University Institute of Technology and Geological survey of Ethiopia laboratory.

I am grateful to honor my family for their parental advice, encouragement, continuous support and help during my postgraduate study entire path.

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CHAPTER ONE

1. INTRODUCTION

1.1. General

With the ever increasing demand and consumption of cement and in the backdrop of waste management, scientists and researchers all over the world are always in quest for developing alternate binders that are environment friendly and contribute towards sustainable management.[1]

Cement which is one of the components of concrete plays a great role, but is the most expensive and environmentally unfriendly material. Therefore requirements for economical and more environmental-friendly cementing materials have extended interest in other cementing materials that can be used as partial replacement of the normal Portland cement. [2] Ground granulated blast furnace slag, fly ash, silica fume, rice Husk, egg shell powder, sugarcane bagasse etc. have been used successfully for this purpose.

Throughout the world, concrete is being widely used for the construction of most of the buildings, bridges etc. Hence, it has been properly labeled as the backbone to the infrastructure development of a nation. Development of a nation not only depends upon the technology but also depends upon the infrastructure. Without concrete infrastructure is not possible. Thus concrete is indispensable material in every construction. The major element of concrete is cement. [3] Since cement price is volatile and demand is so high, finding an alternate material for replacement of cement become mandatory. Several replacement experiments were done by different researchers.

At present, for a variety of reasons, the concrete construction industry is not sustainable. Firstly, it consumes huge quantities of virgin materials which can remain for next generations. Secondly, the principal binder in concrete is Portland cement, the production of which is a major contributor to greenhouse gas emissions that are implicated in global warming and climate change. Thirdly, many concrete structures suffer from lack of durability which may waste the natural resources. A major component of concrete is cement and it is one of the three primary producers of carbon dioxide, a major greenhouse gas. 900kg of CO₂ are emitted for every ton of concrete. As of 2001, the production of Portland cement contributed 7% to global CO₂ emission, largely due to the sintering of limestone and clay at 1500°C. The CO₂ emission of concrete is directly proportional to the cement content used in the concrete mix. Cement

manufacture contributes greenhouse gases both directly through the production of carbon dioxide when calcium carbonate is thermally decomposed, producing lime and carbon dioxide and also through the use of energy, particularly from the combustion of fossil fuels. There is a growing interest in reducing carbon emission related to cement production from both academic and industrial sectors. Recycling of waste components contributes to energy savings in cement production, to conservation of natural resources and to protection of the environment. Hence, currently, the entire construction industry is in search of a suitable and effective waste product that would considerably minimize the use of cement and ultimately reduce the construction cost. On the other hand The price of building materials now a days is very high in different parts of the nation. This high and uncontrolled rising cost can be reduced to minimize by use of different building materials that are cheap, locally available and bring about a reduction in the overall self-weight of the building. [3]

The cement industry was reported to produce 5% of global man-made CO₂ emissions, of which 50% are from chemical processes and 40% from burning fuel (World Business Council for Sustainable Development, 2002). In addition, it is commonly known that the manufacturing of conventional cement is extremely CO₂ intensive; production of one ton of cement clinker results in approximately one ton of CO₂ released into the Earth's atmosphere. The concrete industry worldwide has realized this matter and has been seeking new solutions. Pozzolans, natural or industrial by-products, have been used extensively as cement replacing materials in the past decades. Cement replacing materials help reducing the proportion of cement in concrete, and at the same time they also can improve the properties of concrete in several ways. Besides pozzolans, finely ground inert material known as filler is also used widely. [4]

Building construction serves as an essential role in human life with the construction of housing, offices, factories, buildings, hotels, and hospitals. One proof of the progress of the construction industry is the optimal use of materials in economic aspects. [5]

The increasing demand for cement and concrete is conformed to by partial replacement of cement. Significant cost savings can result when by-products are used as a partial replacement for the energy acute Portland cement. The use of by-products also reduces the pollution and proved as an environmental friendly method of disposal of large quantities of waste materials that would otherwise pollute land, air and water. [6]

As the world's population increases and consumes more of the natural resources, it is incumbent upon the civil engineer to use building materials that contribute to sustaining development instead of satisfying only the short-term need. [7]

Despite the high availability of Teff Straw Ash in Ethiopia, nothing has been done so far to check the feasibility of the Teff Straw Ash related to construction use. This research will investigate the use of Teff Straw Ash as cement replacing material and the physical and chemical properties of the ash will be studied.

1.2. Scientific Relevance

Cement production consumes natural resources and emits high amount of CO₂ during the production process. Furthermore, currently the price of cement is increasing significantly in Ethiopia. This research attempts to find a partial cement replacement material by using agricultural residue to reduce the price of cement, emission of CO₂ and consumption of natural resources.

The scientific relevance of this study is to discover new pozzolanic cement replacement material by using Teff Straw Ash (TSA).

1.3. Objectives of the Thesis

1.3.1. General Objectives

This thesis aims to assess the use of Teff Straw Ash (TSA) by processing the Teff Straw which is collected from local farmers in Ethiopia as a cement replacement material.

1.3.2. Specific Objectives

- Checking the physical and chemical properties of Teff Straw Ash (TSA).
- Evaluating the performance of mortar by replacing cement with Teff Straw Ash (TSA) at different proportions (10% and 20% replacement by volume).
- Checking the environmental impact of replacing cement with Teff Straw Ash (TSA).

1.4. Methodology

- Collecting the Teff Straw (TS) from the local farmers.
- Thoroughly wash the collected sample.
- Oven dry Teff Straw (TS) at a temperature of 110°C for twelve hours to remove the moisture.
- Burn the oven dried sample at a temperature of 600°C , 750°C and 900°C for six hours
- After cooling the burnt sample for 24 hours grind the sample until the required size attained.

- Perform tests to check the chemical and physical properties of the Teff straw ash (TSA)
- Perform tests on TSA and mortar at hardened state. The major tests are tests for determination of fineness for Teff straw ash (TSA), Complete Silicate Analysis, XRD and SEM test on TSA and compressive strength at 1, 3, 7, 14 and 28 days for the mortar on the hardened mortar cube.

1.5. Scope and Limitations

1.5.1. Scope

This paper studies the effect of TSA as cement replacing materials by analyzing the effect on the compressive strength of mortar mixes.

1.5.2. Limitations

Due to limited resources in the laboratory, that makes the production of Teff Straw Ash difficult, only two cubes were tested for one sample of mortar cube. In addition other properties such as compressive & flexural strength of concrete cubes and water permeability were not performed in this research paper.

1.6. Organization of the Thesis

The first part of this chapter is introduction: basic principles, history, and applications of cement replacement materials are outlined in order to better define specific aspects on properties and performance of pozzolanic cement replacement material in this thesis. The second part of this chapter provides an overview of literature review on previous experiences. Chapters 3- 4 explain and discuss the various steps and experimental results performed. Chapter 5 discusses conclusions, and offers recommendations for improvements to this work. Complete silicate analysis test results and ASTM C 109 Compressive strength of hydraulic cement mortar cubes are contained within the Appendices.

CHAPTER TWO

2. LITRATURE REVIEW

2.1. Introduction

From the 20th century, there had been an increase in the economic consumption of mineral admixtures by the cement and concrete industries. The increasing demand for cement and concrete is conformed to by partial replacement of cement. Significant cost savings can result when by-products are used as a partial replacement for the energy acute Portland cement. The use of by-products also reduces the pollution and proved as an environmental friendly method of disposal of large quantities of waste materials that would otherwise pollute land, air and water. [6]

As of 2001, the production of Portland cement contributed 7% to global CO₂ emission, largely due to the sintering of limestone and clay at 1500°C. The CO₂ emission of concrete is directly proportional to the cement content used in the concrete mix. Cement manufacture contributes greenhouse gases both directly through the production of carbon dioxide when calcium carbonate is thermally decomposed, producing lime and carbon dioxide and also through the use of energy, particularly from the combustion of fossil fuels. There is a growing interest in reducing carbon emission related to cement production from both academic and industrial sectors. Recycling of waste components contributes to energy savings in cement production, to conservation of natural resources and to protection of the environment. Hence, currently, the entire construction industry is in search of a suitable and effective waste product that would considerably minimize the use of cement and ultimately reduce the construction cost. On the other hand, the price of building materials now a days is very high in different parts of the nation; particularly developing areas. This high and uncontrolled rising cost can be reduced to minimize by use of different building materials that are cheap, locally available and bring about a reduction in the overall self-weight of the building. Some industrial and other products that would otherwise dump the environment as waste or at best be put into only limited use could gainfully be employed as building material. [3]

As the world's population increases and consumes more of the natural resources, it is incumbent upon the civil engineer to use building materials that contribute to sustaining development instead of satisfying only the short-term need. [7] Different studies have been made worldwide to use wastes from agricultural residues by converting them in to ash for cement replacement purpose at different proportions. Even if Teff is one of the

highly produced cereals in Ethiopia, a Little attention was given on the use of the Teff Straw other than using it as cattle fodder.

2.2. Cement

Cements are adhesive substances, which when mixed with water form paste and the paste is temporarily plastic and may be molded or deformed. But later it sets and hardens to a rigid mass. Cements of this kind are known as calcareous cements. Calcareous cements contain lime. Calcareous cements can be classified as Non-hydraulic and Hydraulic. Non-hydraulic cements are cements which are not able to set and harden in water (e.g. non-hydraulic lime) or which are not stable in water (e.g. gypsum plasters). Hydraulic cements are cements that are able to set and harden in water, and give a solid mass that does not disintegrate, i.e., remain stable in water (e.g. Portland cement). [9]

In the manufacture of cement, the raw materials are first mined and then ground to a powder before blending in predetermined proportions. The blend is fed into the upper end of a rotary kiln heated to 2600 to 3000F by burning oil, gas, or powdered coal. Because cement production is an energy-intensive process, reheaters and the use of alternative fuel sources, such as old tires, are used to reduce the fuel cost. (Burning tires provide heat to produce the clinker and the steel belts provide the iron constituent.)[7]

The most common hydraulic cement used in construction today is Portland cement. There are several varieties of hydraulic portland cement, as recognized by the American Society for Testing and Materials (ASTM), which vary in their properties. Hydraulic cement is defined as cement that sets and hardens by chemical reaction with water and is capable of doing so underwater. [9]

2.2.1. Chemical composition of cement

Raw materials used in the manufacture of Portland cement consist of mainly of lime, silica, alumina and iron oxide. These compounds interact with one another in the kiln to form a series of more complex product. The resulting pulverized cement is a heterogeneous mixture of several fine-grained minerals. [14]

Cement type	ASTM C150/ C150M-12	C ₃ S, % by mass	C ₂ S, % by mass	C ₃ A, % by mass	C ₄ AF, % by mass	Fineness, m ² /kg
I	General purpose	57	15	9	8	384
II	Moderate sulfate resistance	57	17	7	10	377
III	High early strength	56	16	8	9	556
IV	Low heat of hydration	42	32	4	15	340
V	Sulfate resistant	58	18	4	12	389

*Shorthand notation CaO= C; SiO₂=S; Al₂O₃=A; Fe₂O₃=F; SO₃=S; and H₂O=H.

Table 2.1—Typical compound composition of Portland cement [10]

2.2.2. Types of Cement

There are many varieties of cements and numerous ways of classification. One of the simplest classifications is by the chemical constituent that is responsible for the setting or hardening of the cement. On this basis, the silicate and aluminate cements, wherein the setting agents are calcium silicates and aluminates, constitute the most important group of modern cements. Included in this group are the portland, aluminous, and natural cements. [7]

There are different types of cement depending on their composition, method of manufacturing (grinding, burning, etc.) and also the relative proportion of the different compounds. One of these types and the most commonly used one is Portland cement, which in turn is divided into many types. The other common type of cement is a blended type of cement called Portland pozzolana cement which contains some amount of pozzolanic materials. [2]

Other cementitious materials, such as polymers, fly ash, and silica fume, may be used as a cement replacement in concrete. Polymers are plastics with long-chain molecules. Concretes made with them have many qualities much superior to those of ordinary concrete. [8]

2.2.2.1. Portland cement

Portland cement, the most common of the modern cements, is made by carefully blending selected raw materials to produce a finished material meeting the requirements of ASTM C150 for one of eight specific cement types. Four major compounds [lime (CaO), iron (Fe_2O_3), silica (SiO_2), and alumina (Al_2O_3)] and two minor compounds [gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and magnesia (MgO)] constitute the raw materials. The calcareous (CaO) materials typically come from limestone, calcite, marl, or shale. The argillaceous (SiO_2 and Al_2O_3) materials are derived from clay, shale, and sand. The materials used for the manufacture of any specific cement are dependent on the manufacturing plant's location and availability of raw materials. Portland cement can be made of a wide variety of industrial by-products. [7]

The basic types of portland cement covered by ASTM C150 are as follows: [7]

Type I, general-purpose cement, is the one commonly used for many structural purposes. Chemical requirements for this type of cement are limited to magnesia and sulfur-trioxide contents and loss on ignition, since the cement is adequately defined by its physical characteristics.

Type II is a modified cement for use in general concrete where a moderate exposure to sulfate attack may be anticipated or where a moderate heat of hydration is required. These characteristics are attained by placing limitations on the C_3S and C_3A content of the cement. Type II cement gains strength a little more slowly than Type I but ultimately will achieve equal strength. It is generally available in most sections of the country and is preferred by some engineers over Type I for general construction. Type II cement may also be specified as a low-alkali cement for use where alkali reactive aggregates are present. To do so requires that optional chemical requirements be included in the purchase order. Type II low-alkali cement is commonly specified in California.

Type III cement attains high early strength. In 7 days, strength of concrete made with it is practically equal to that made with Type I or Type II cement at 28 days. This high early strength is attained by finer grinding (although no minimum is placed on the fineness by specification) and by increasing the C_3S and C_3A content of the cement. Type III cement, however, has high heat evolution and therefore should not be used in large masses. Because of the higher C_3A content, Type III cement also has poor sulfate resistance. Type III cement is not always available from building materials dealers' stocks but may be obtained by them from the cement manufacturer on short notice.

Ready-mix concrete suppliers generally do not stock Type III cement because its shorter set time makes it more volatile to transport and discharge, especially in hot weather.

Type IV is a low-heat cement that has been developed for mass concrete construction. Normal Type I cement, if used in large masses that cannot lose heat by radiation, will liberate enough heat during the hydration of the cement to raise the temperature of the concrete as much as 50 or 60 F. This results in a relatively large increase in dimensions while the concrete is still soft and plastic. Later, as the concrete cools and is hardening, shrinkage causes cracks to develop, weakening the concrete and affording points of attack for aggressive solutions. The potential-phase compounds that make the largest contribution to the heat of hydration are C_3S and C_3A ; so the amounts of these are permitted to be present are limited. Since these compounds also produce the early strength of cement, the limitation results in a cement that gains strength relatively slowly. This is of little importance, however, in the mass concrete for which this type of cement is designed.

Type V is a portland cement intended for use when high sulfate resistance is required. Its resistance to sulfate attack is attained through the limitation on the C_3A content. It is particularly suitable for structures subject to attack by liquors containing sulfates, such as liquids in wastewater treatment plants, seawaters, and some other natural waters. Both Type IV and Type V cements are specialty cements. They are not normally available from dealer's stock but are usually obtainable for use on a large job if arrangements are made with the cement manufacturer in advance.

2.2.2.2. Blended cements

Portland cement or clinker can be blended or interground with other materials to achieve certain properties. These cements are specified under ASTM Specification C 595 and C 1157. There are five classes of blended cement in C 595:[9]

- Portland blast-furnace slag cement (Type IS);
- Portland-pozzolan cement (Type IP and Type P);
- Pozzolan-modified portland cement (Type I (PM));
- Slag cement (Type S); and
- Slag-modified portland (Type I (SM)).

a) Portland Pozzolana cement

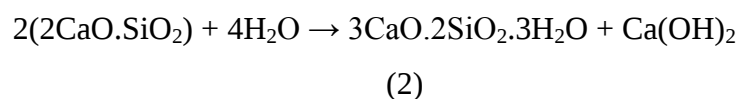
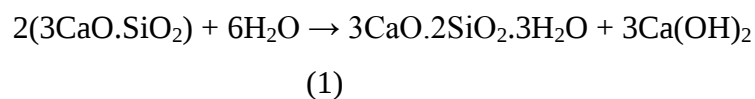
Portland-pozzolan cement includes four types: IP, IP-A, P, and P-A, which must meet the requirements of ASTM C 595. The first two types may be used for general concrete construction and the latter two when high strength at an early age is not required. The letter "A" designates the air-entraining counterparts. These cements are produced by

either intergrinding portland cement clinker and pozzolan, by blending portland cement and finely divided pozzolan, or by a combination of both intergrinding and blending. Portland pozzolan cements contain between 15 and 40% pozzolan in the blended cement. In addition, the cements may be designated low or moderate heat of hydration (LH or MH), as well as moderate sulfate resistant (MS) for Types IP and IP-A. [9]

When pozzalanic materials are added to cement the silicon dioxide (SiO_2) present in these materials reacts with free lime released during the hydration of cement and forms additional CSH (calcium-silicate hydroxide) as new hydration products, which improve the mechanical properties of concrete formulation. The capacity of pozzalanic materials of enhancing the strength of concrete is more closely associated with physical than chemical effects. It is reported that cement blended with pozzolan would produce 65 to 95 % strength of OPC concrete in 28 days and at later ages their strength normally improves because pozzolan like fly ash react more slowly than cement due to different composition and at one year about the same strength is obtained. [15]

2.2.3. Hydration of Cement

This is the product of reaction of cement with water. In the presence of water the silicates and aluminates of Portland cement form products of hydration and hydrates, which in time produce a firm and hard mass, the hardened cement paste. The two calcium silicates (C_3S and C_2S) are the main cementitious compounds in cement, the former hydrating much more rapidly than the latter. The product of hydration of C_3S is the microcrystalline hydrate $\text{C}_3\text{S}_2\text{H}_3$ with some lime separating out as crystalline $\text{Ca}(\text{OH})_2$. C_2S behaves similarly but clearly contains less lime. Nowadays, the calcium silicate hydrates are described as C-S-H. The hydration of C_3S and C_2S are shown in Eq. (1) and (2). [8]



2.2.4. Quality control

Cement is regularly sampled and tested by the producer. Tests include both chemical analysis and physical tests such as strength, fineness, and setting behavior. Tests on cement are done for quality control and to verify that the cement meets the requirements of standards such as ASTM C 150. [9]

2.2.5. Properties of Cement

a) Physical Properties

Fineness:

It is about the grinding of cement and affects the rate of hydration: the finer the cement, the faster the strength development takes place. [14]

The surface area is measured by the Blaine air-permeability test (ASTM C 204 or AASHTO T 153) that indirectly measures the surface area of the cement particle per unit mass. According to the Ethiopian standard ordinary Portland cement shall have a specific surface area of not less than 2250 cm²/g, whereas the ASTM C 150 standard recommends a minimum of 2800 cm²/g. [2]

Strength:

The quality of cement for its strength in compression is judged by finding the compressive strength of cement – sand mortar. [14]

2.3. Aggregates

Aggregate is granular material such as sand, gravel, crushed stone, blast-furnace slag, and lightweight aggregates that usually occupies approximately 60 to 75% of the volume of concrete. Aggregate properties significantly affect the workability of plastic concrete and also the durability, strength, thermal properties, and density of hardened concrete. Aggregates may be broadly classified as natural or artificial, both with respect to source and to method of preparation. Natural sands and gravels are the product of weathering and the action of wind or water, while manufactured crushed fine aggregate and crushed stone coarse and fine aggregate are produced by crushing natural stone. [16]

2.3.1. Fine Aggregates

Fine aggregate shall consist of natural sand obtained from the natural disintegration of rock, sand obtained from crushed stones or both.

2.3.1.1. Properties of Fine Aggregates

a) Grading Requirement

Fine aggregate for standard sand shall fulfill the grading requirement given below as per ASTM, C778.

Percentage passing			
Sieve size(mm)	Average	Lower range	Upper range
1.18	100	100	100
0.6	97	96	100
0.425	69	65	75
0.3	26	20	30
0.15	1	0	4

Table 2.2—Grading requirement for Standard Sand as per ASTM, C778

b) Silt content

The material in fine aggregates which is finer than 75µm is generally regarded as silt. This silt in the sand for the concrete has a severe effect on the quality of the concrete. It mainly affects the workability of the concrete, and also results in the reduction of strength. [8]

According to ASTM C117 maximum allowable silt content for concrete subjected to abrasion is 3% and for all other concrete is 5%.

2.4. Water

Water fit for drinking is generally suitable for making concrete. Substances in water that, if present in large amounts, may be harmful are: salt, oil, industrial wastes, alkalies, sulphates, organic matter, silt, sewage etc. Tests by the sense of smell, sight or taste should reveal such impurities; however water of doubtful quality should be submitted for laboratory analysis and test.

Water-used in concrete mixes has two functions, the first is to react chemically with the cement which will finally set and harden, and the second function is to lubricate all other materials and make the concrete workable.

The quality of cement paste is determined by the proportion of water to cement (Water cement ratio). Too much water prevents proper setting: Too little water prevents complete chemical reaction called hydration; the water and cement combine chemically to form a new compound. [17]

2.5. Pozzolans

A pozzolan is a siliceous or aluminosiliceous material that, in finely divided form and in the presence of moisture, chemically reacts with the calcium hydroxide released by the hydration of portland cement to form calcium silicate hydrate and other cementitious compounds. Pozzolans and slags are generally categorized as supplementary cementitious materials or mineral admixtures. They may be used in addition to or as a partial replacement of portland cement or blended cement in concrete, depending on the properties of the materials and the desired effect on concrete. [12]

Fly ash, ground granulated blast-furnace slag, silica fume, and natural pozzolans, such as calcined shale, calcined clay or metakaolin, are materials that, when used in conjunction with portland or blended cement, contribute to the properties of the hardened concrete through hydraulic or pozzolanic activity or both. [12] The addition of pozzolanic material will help to rapidly tie up alkalis and inhibit their mobilization. [11]

2.5.1. Classification of Pozzolans

ASTM C618-12 outlines the physical and chemical requirements of pozzolanic materials. Pozzolanic materials include natural pozzolans (Class N) and by-product materials. Natural pozzolans are notably volcanic ashes, diatomaceous earth, calcined clay, metakaolin clay, and rice husk ash. By-product material is most typically fly ash, classified as either Class F or Class C, reflecting a difference in chemical composition. Class F fly ashes possess largely pozzolanic properties. Class C fly ashes generally possess cementitious and pozzolanic properties. ASTM C618-12 differentiates Class C and Class F fly ashes based on the sums of the SiO_2 , Al_2O_3 , and Fe_2O_3 . For Class C fly ash, $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ must be greater than or equal to 50%. For Class F fly ash, $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ must be greater than or equal to 70%. Class C fly ashes essentially contain 15 to 25% calcium, which makes their performance characteristics different from a low-calcium Class F fly ash. [10]

	Class F fly ash	Class C fly ash	Ground slag	Silica fume	Calcined clay	Calcined shale	Metakaolin
SiO ₂ , %	52	35	35	90	58	50	53
Al ₂ O ₃ , %	23	18	12	0.4	29	20	43
Fe ₂ O ₃ , %	11	6	1	0.4	4	8	0.5
CaO, %	5	21	40	1.6	1	8	0.1
SO ₃ , %	0.8	4.1	9	0.4	0.5	0.4	0.1
Na ₂ O, %	1.0	5.8	0.3	0.5	0.2	—	0.05
K ₂ O, %	2.0	0.7	0.4	2.2	2	—	0.4
Total Na eq. alk, %	2.2	6.3	0.6	1.9	1.5	—	0.3
Loss on ignition, %	2.8	0.5	1.0	3.0	1.5	3.0	0.7
Blaine fineness, m ² /kg	420	420	400	20,000	990	730	19,000
Relative Density	2.38	2.65	2.94	2.40	2.50	2.63	2.50

Table 2.3— Chemical Analysis and Selected Properties of Typical Fly Ash, Slag, Silica Fume, Calcined Clay, Calcined Shale, and Metakaolin[12]

2.5.2. Fly ash as cementitious material

Fly ash, the most widely used supplementary cementitious material in concrete, is a byproduct of the combustion of pulverized coal in electric power generating plants. [12]

The most common sources of fly ash are electric power-generating stations. Fly ash has become the predominant pozzolan in use throughout the world due to performance and economic factors. Extensive literature is available on the effects of fly ash in both fresh and hardened concrete. [10]

The main constituent of fly ash is silica. Glassy noncrystalline forms of silica, alumina, and iron are principally responsible for the pozzolanic reaction with calcium hydroxide (lime). In concrete, lime results from the hydration of portland cement. Other components of fly ash are calcium, magnesium, sulfur, potassium, and sodium. Class C

fly ashes contain less silica, alumina, and iron than the Class F fly ashes, and Class C fly ashes usually have elevated levels of calcium. [10]

Although the constituents are not necessarily present in the oxide form, it is customary to express chemical analysis results in terms of the oxides of the elements: SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , and SO_3 . Carbon is seldom determined directly but is often assumed to be approximately equal to the LOI. [10]

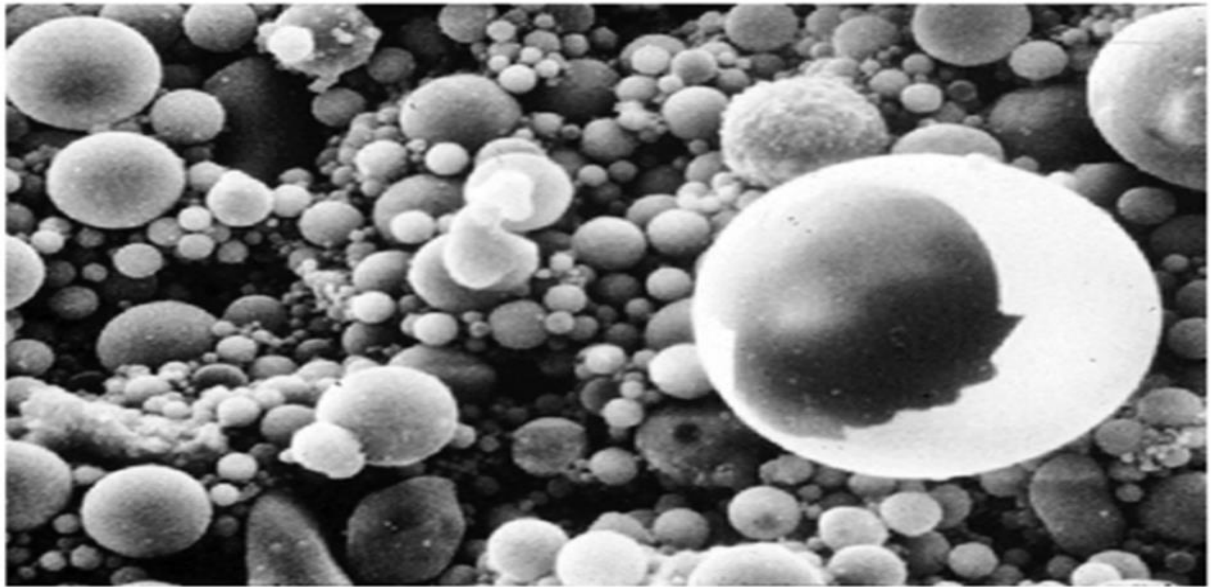


Figure 2.1— Fly ash particles viewed at 1000× magnification [10]

2.5.3. Other cementitious materials

2.5.3.1. Rice Husk Ash (RHA)

There are various efforts to utilize waste material as a primary material for composing concrete. One such waste material is rice husk ash (RHA) as a residual material in rice production. Rice husk ash, is the result of the residual combustion of rice husk, has a dominant silica content of 93% and almost the same silica content found in factory-made micro silica. [5]

RHA is a good pozzolanic material and can be used in a big way to make special concrete mixes. [6]

S.No.	Constituents	Percentage value
1	Fe ₂ O ₃	0.54
2	K ₂ O	0.1-2.54
3	SiO ₂	62.5 - 97.6
4	CaO	0.1 - 1.31
5	MgO	0.01 - 1.96
6	Na ₂ O	0.01 - 1.58
7	P ₂ O ₅	0.01 - 2.69
8	SiO ₃	0.1 – 1.23
9	Carbon	2.71 – 6.42

Table 2.4— Chemical composition of RHA [6]

2.5.3.2. Sugarcane Bagasse Ash (SCBA)

AJAY Goyal et al. studied SCB which was obtained by burning SCB at 600°C for 5 hours under controlled conditions and its physical, chemical, and mineralogical characterization was done to evaluate the possibility of its use as binder partially replacing cement in the mortar applications. [1]

Chemical properties									Physical properties			
Chemical composition (wt. %)									Density	Blaine surface area	Particle size	color
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	LOI	g/cm ³	cm ² /g	µm	
OPC	18.4	5.6	3.0	66.8	1.4	2.8	0.5	2.0	3.15	3250	36.2	Dark grey
SCBA	62.43	4.38	6.98	11.8	2.51	1.48	3.53	4.73	2.52	5140	28.9	Reddish grey

Table 2.5— Physical and chemical properties of OPC and SCBA [1]

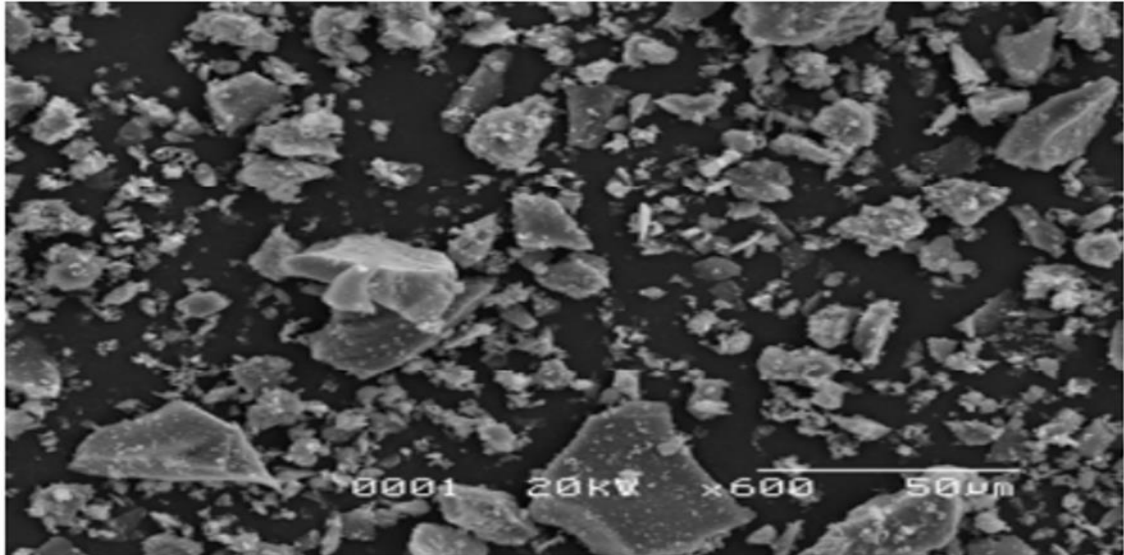


Figure 2.2— SEM photograph of SCBA (sugarcane bagasse ash) [1]

2.5.3.3. Egg shell

Anand Parkash et al. 2017 confirmed that Egg shell Powder can be used as a partial replacement of cement. [3]

2.6 Teff Straw Ash (TSA)

Teff is major cereal crop used for human consumption in Ethiopia. It is highly valued for its injera making quality. The composition of Teff shows that it has good mineral content and generally higher amount of the essential amino acids. Besides, the crop can grow well in moisture stress and waterlogged conditions; can also grow from sea level up to 2800m, under various rainfalls, temperature and soil conditions. Teff can grow under wide and diverse agro-ecologies. Even though there are areas where the crop is grown during the short rainy season (Belg), Teff is mainly cultivated during the main rainy season (Meher). The length of the growing period (LGP) ranges from 60 to 180 days (depending on the variety and altitude) with an optimum of 90 to 130 days. [18]

Efficient utilization of agricultural residue is the need of today's environment. Teff Straw is one of such an agricultural residue which is available in high amount in east African continent particularly in Ethiopia. [13]

Teff Straw Ash (BTSA) is the solid residue obtained from the Teff cereal. Despite the high availability of Teff Straw, it has received little attention.

2.6.1 Teff Straw Ash (TSA) Composition

Silicon is the second most abundant element in soil which accounts for approximately 30-35% of the total mass of soil. Therefore, plants also contain silicon in their tissues. Silica (SiO_2) constitutes 65-70% of total minerals present in the plants. Many authors have previously used heat and chemical treatment in different sequence to produce silica material from bagasse, rice, wheat and other agricultural residues, and achieved up to 98% SiO_2 purity and further used it for glass ceramics and other applications. [13]

X-ray fluorescence (XRF) analysis on Brown Teff showed presence of high amount of silicon dioxide ($\approx 52\%$) in raw Teff Straw. Thermal treatment at 600°C for 4 hour increased the SiO_2 concentration to $\approx 92\%$ in its ash. Further acid treatment increased the concentration to $\approx 97\%$. Fourier transform infrared (FTIR) spectroscopy also confirmed increase in SiO_2 after thermal and acid treatment. X-ray diffraction (XRD) analysis showed the silica of amorphous nature in Teff Straw Ash before acid treatment (S-BAT) whereas crystallinity increased after acid treatment (S-AAT). Transmission electron microscopy (TEM) showed presence of uniform nano-disks shaped particles of 50 nm average particle size in acid treated Teff Straw Ash. Compared to other agricultural residues high amount of silica availability as raw and its small diameter anatomy structure will make Teff Straw better source of silica material at lower heat and chemical treatment. [13]

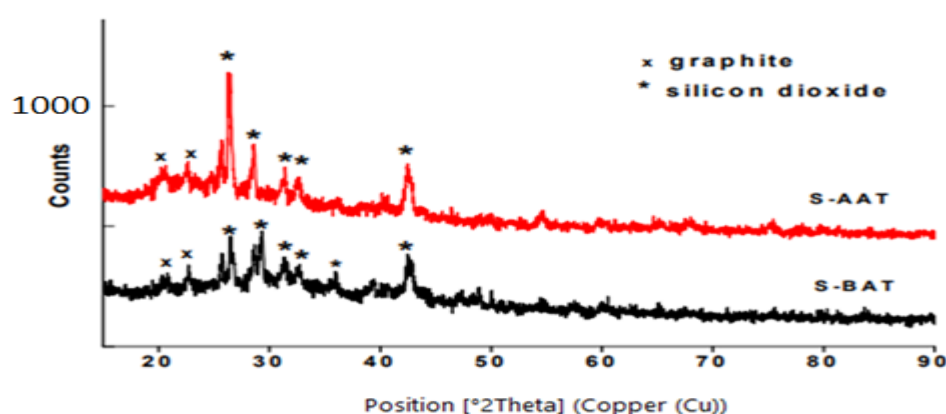


Figure 2.3— XRD of Brown Teff Straw Ash S-BAT and S-AAT. [13]

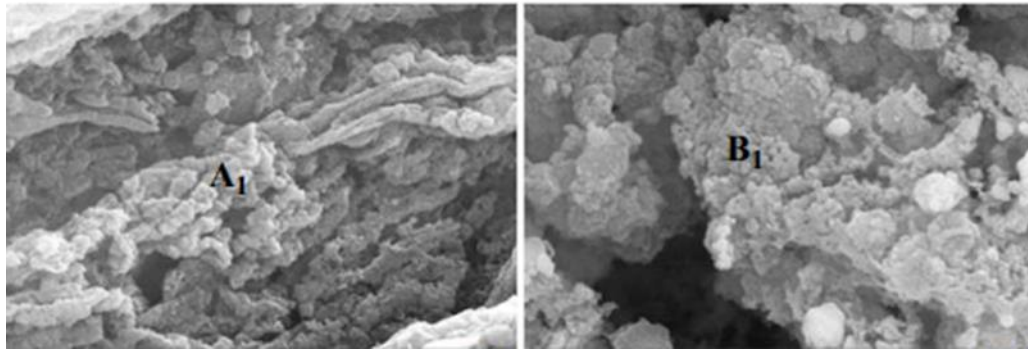


Figure 2.4— SEM results of S-BAT (A1) and S-AAT (B1). [13]

2.6.2 Availability of Teff Straw in Ethiopia

Teff is one of the most important and dominant staple cereal crops in Ethiopia. Cereal crops grown on 71% of the total area cultivated and about 61% of total agricultural production shared. Though Teff is untouched cereal crop; it is the second largest share of cereal crops production following maize. Teff accounted approximately 28.4% of the total cereal crop's cultivated area and 50% of total cereal production quantity. Teff production area continues to expand; and a greater number of farmers are producing Teff. Approximately 6.3 million farmers were growing Teff in 2013; compared with 4.4 million farmers in 2001/2002. Similarly; the cultivated area which is allocated to Teff is increased from 1.8 million hectares in 1997 to 2.7 million hectares in 2013. [19] Ethiopia is the largest Teff producer in the world. In 2017, Teff accounts for 24% of the grain area, followed by maize 17% and sorghum 15%. [20]

In the year 2013-14, Teff accounted for 22.6% (about 3.2 million hectares per year) of the grain crop area in Ethiopia. With average straw yield of 5000 kg/ha, the annual Teff straw yield is estimated to be ≈ 16 million tonne/year. Thus, Teff straw is available in high quantity may be used for making nano-silica. [13]

CHAPTER THREE

3. EXPERIMENTAL PROGRAM

3.1. Introduction

The main objective of the experimental program is to study the effect of Teff Straw Ash (TSA) as a cement replacing material.

3.2. Teff straw ash (TSA) production

White and Brown (mixture of red and white) Teff Straw used in this study are collected from Bishoftu (Debre Zeyit), Denbi Hulet wuhalemat Woreda, Ethiopia.



Figure 3.1—Teff Straw (TS) at the farm land

3.2.1. Washing, drying and grinding of Teff Straw

The first step done on the collected sample is to thoroughly wash the samples using tap water. The washed sample is then dried in electric oven at 110 °C for 12 hours.

The washed and dried Teff Straw sample is then grinded to pass through 1 mm sieve mesh as shown in the picture below.



Figure 3.2— Grinding machine used for grinding the washed Teff Straw (BTS)



(A)



(B)

Figure 3.3—Teff Straw **(A)** Oven dried sample **(B)** Grinded Sample to pass through 1 mm sieve

3.2.2. Teff Straw Combustion

Combustion of the grinded Teff Straw is performed using a muffle furnace under controlled Temperature and time. The Teff Straw sample is then combusted in the muffle furnace at temperatures of 600°C, 750°C and 900°C for 6 hours.

3.2.3. Grinding of Teff Straw after Combustion

After combustion the Teff Straw is turned in to an ash. To obtain the required size of particles in the ash, the combusted Brown Teff Straw Pass grinding process using a ball mill grinder for 4 hours.



Figure 3.4— Ball mill grinder

3.3. Physical and chemical analysis of Teff Straw Ash (TSA)

3.3.1. Chemical analysis

White and Brown Teff Straw Ash samples are taken to the Geological survey of Ethiopia laboratory to perform complicate silicate analysis of the ash. The complete silicate analysis is used to determine the amount of the oxide content as weight percent of the major oxides as SiO_2 , Al_2O_3 , Fe_2O_3 , Fe_2O_3 , CaO , MgO , P_2O_5 and Na_2O .

3.3.2. Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) was carried out to find the shape and size of Brown Teff Straw Ash (BTSA) particles in dry form. The scanning Electron Microscopy (SEM) is performed on the ash passing through the 63 μm sieve. Brown Teff straw ash (BTSA) samples are taken to the Addis Ababa Science and Technology University laboratory to perform SEM analysis of the ash.

3.3.3. X-ray diffraction (XRD)

X-ray diffraction is done to examine amorphous and crystalline structures of Brown Teff straw ash (BTSA) particles in dry form. XRD test is performed on the ash passing through the 63 μm sieve. Brown Teff Straw Ash (BTSA) samples are taken to the Addis Ababa University, College of Science, department of chemistry to perform XRD analysis of the ash.

3.3.4. Fineness (Blaine air) of Brown Teff Straw Ash (BTSA)

The fineness of Brown Teff straw ash (BTSA) is measured as specific surface area by measuring the time taken for air to flow through a compacted Brown Teff straw ash (BTSA) by using a standard Blaine air-permeability apparatus in accordance with ASTM C207.

The weight of the sample needed for the fineness test is determined using the following formula.

$$W = \rho v (1 - \epsilon) \quad (3)$$

Where,

W = weight of sample (in grams),

ρ = density of the sample (in g/cm^3),

v = volume of bed of cement the apparatus is calibrated with (in cm^3),

ϵ = porosity of bed of cement the apparatus is calibrated with

The density of Brown Teff straw ash (BTSA) is measured at Geological survey of Ethiopia laboratory and the result is 2.3 g/cm^3 . The volume of calibration of the apparatus used is 1.852 g/cm^3 . The porosity determined for the specific apparatus is 0.5. The time for the manometer mercury drop is taken using a stopwatch. The specific surface area of the samples is finally calculated using the following formula taken from the ASTM standard mentioned above.

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}} \quad (4)$$

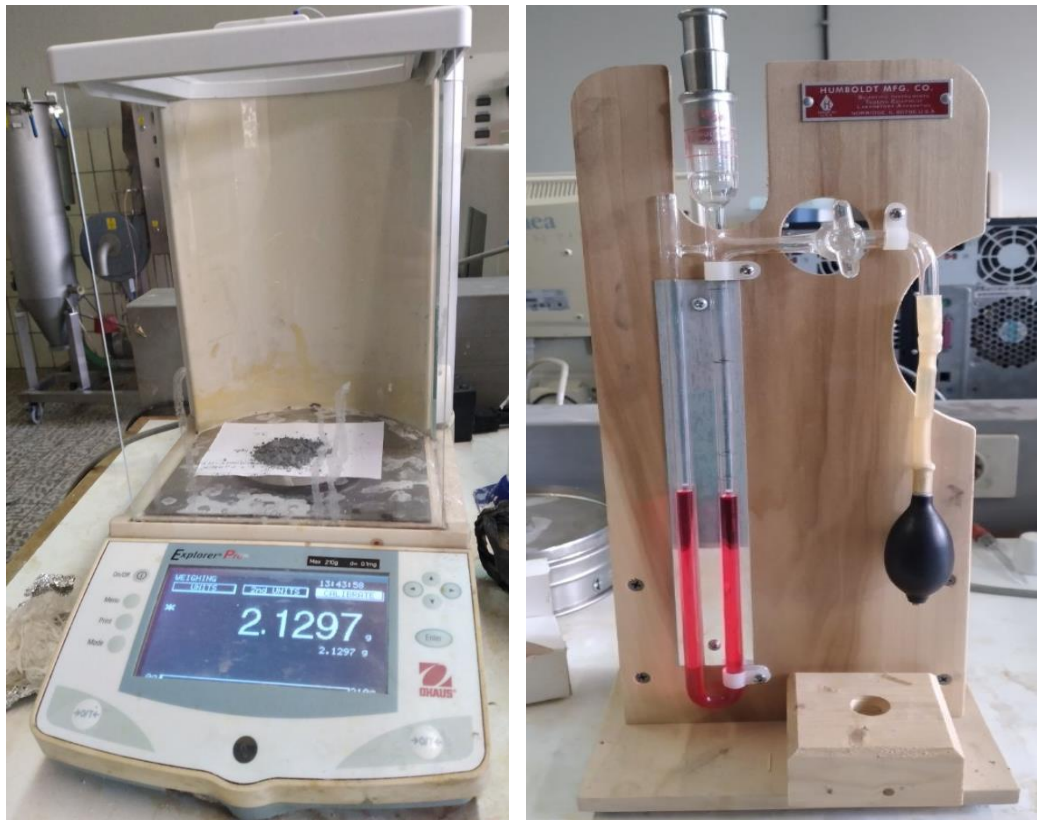
Where,

S = the specific surface area of the sample (in cm^2/g),

S_s = the specific surface area of the standard sample used to calibrate the apparatus (in cm^2/g) which is $3774 \text{ cm}^2/\text{g}$,

T = time measured for the manometer drop for the sample (in seconds),

T_s = time measured for the manometer drop for the standard sample used to calibrate the apparatus (in seconds) which is 37.8 sec.



(A) (B)
Figure 3.5— (A) Weighting Balance (B) Blaine air-permeability apparatus

3.4. Tests on Mortar

This experimental program consists of preparing different mortar mixes with different percentage of Brown Teff Straw Ash (BTSA) replacing the cement.

3.4.1. Cement

The types of cement used for this experiment is Messebo ordinary Portland cement. Messebo cement for this experiment is purchased from the local market available at Addis Ababa, Ethiopia.

3.4.2. Sand

The first step is preparation of standard sand as per ASTM C778. The sieve analysis of the standard sand prepared in the laboratory is as shown in the table below.

Percentage passing				
Sieve size(mm)	Average	Lower range	Upper range	Standard sand prepared in the lab.
1.18	100	100	100	100
0.6	97	96	100	99.5
0.425	69	65	75	53.37
0.3	26	20	30	17.53
0.15	1	0	4	1.153

Table 3.1— Gradation of standard sand used

The prepared standard sand is checked for silt content in accordance to ASTM C117. According to ASTM C117 the maximum allowable silt content for should not me greater than 5%. From the jar test the measurement sand+ silt= 265ml and for sand is 8ml.

$$\text{Therefore, silt content} = \frac{8}{265} * 100 = 3.01\% \text{ which is less than } 5\%$$

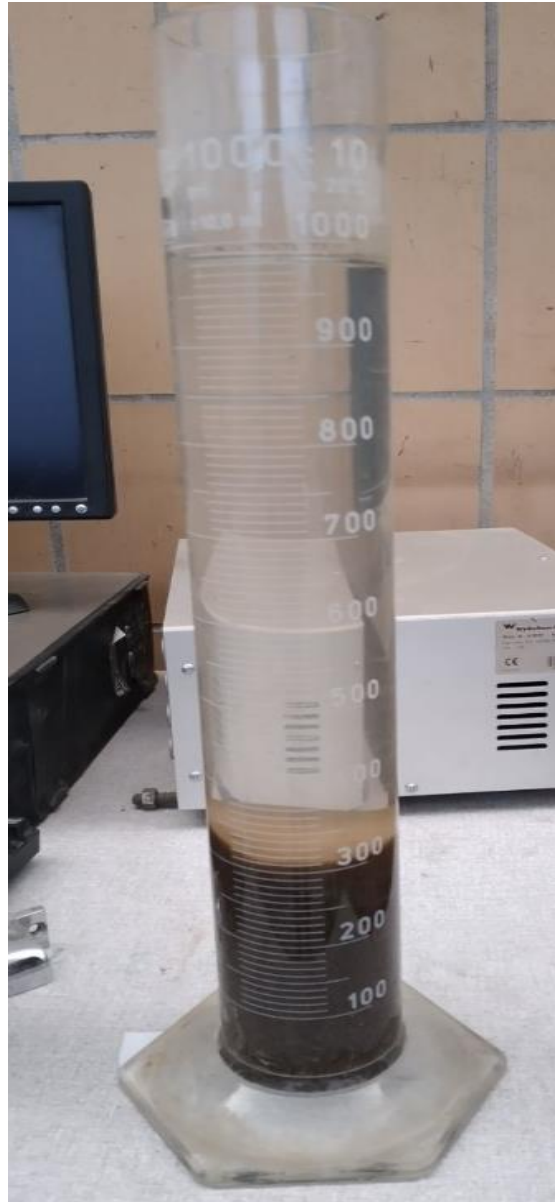


Figure 3.6— Jar test for Silt content

3.4.3. Preparation of Mortar Cubes

The test is performed for testing the Compressive strength of mortar using Brown Teff Straw Ash (BTSA). Various cubes were made with various percentage of Brown Teff Straw Ash (BTSA) by volume of cement and tested. The specific gravity of Brown Teff straw ash (BTSA) is 2.3 g/cm^3 . Two concrete cube specimens for the test is made for each mortar test with 0%, 10% and 20% Brown Teff Straw Ash (BTSA) composition. Compressive strength test is conducted on hardened concrete as it is an easy test to perform and also most of the suitable characteristic property of concrete. The strength tests are conducted curing ages of 1 day, 3 days, 7 days , 14 days and 28 days . The total

amount of mortar cubes tested is 90 cubes. The cube specimen is of size 50×50×50mm. Composition values of components of cement mortar is based on ASTM C109.

Number of specimens	9
Cement, grams	740
Sand, grams	2035
Water, mL (Portland -w/c- 0.485)	359

Table 3.2— Composition values of components of cement mortar based on ASTM C109
The step by step procedure is described below-

- First of all the moulds of cast iron, thick enough to prevent deformation, is used to prepare the specimen of size 50×50×50mm.
- placing of the mortar in the moulds
- Then these moulds are placed on the vibrating table and are compacted until the specified condition is attained.
- The test specimens are stored in dry place free from vibration
- After 24 hrs period, the specimen are submerged in water for curing and kept there until taken out just prior to test. The specimens should not to be allowed to become dry at any time until they have been tested.
- The cube is then taken out of the curing tank and placed in the compression testing machine so to find the Maximum load at which the concrete fails by compression.

Mix code	Cement (gm)	Brown Teff straw ash (BTSA) (gm)	No. of mortar cubes	Water (ml)	Sand (gm)
BTSA 0%	822.22	0	10	399	2261.11
BTSA 10 %	740	60.4	10	399	2261.11
BTSA 20 %	657.78	120.07	10	399	2261.11

Table 3.3— Mix proportions used in the preparation of BTSA mortar specimens



Figure 3.7— Mortar casted in to moulds



Figure 3.8— Compressive strength test machine

CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.1. Combustion of Brown Teff Straw

During Combustion of the grinded Brown Teff Straw sample the sample starts to release smoke to the atmosphere at 200°C. The amount of smoke increases gradually as the combusting temperature increases until the combusting temperature reaches 600°C. After 600°C the amount of smoke emission decreases and stops around 700°C.

Due to the release of CO₂ from Brown Teff Straw sample while combustion process takes place the color of the ash for different combusting temperature shows significant change. As the combustion temperature increases the color of the ash tends to be whiter in color until it reach 750°C. After 750°C the color of the ash starts to change its color to a very light yellowish color.

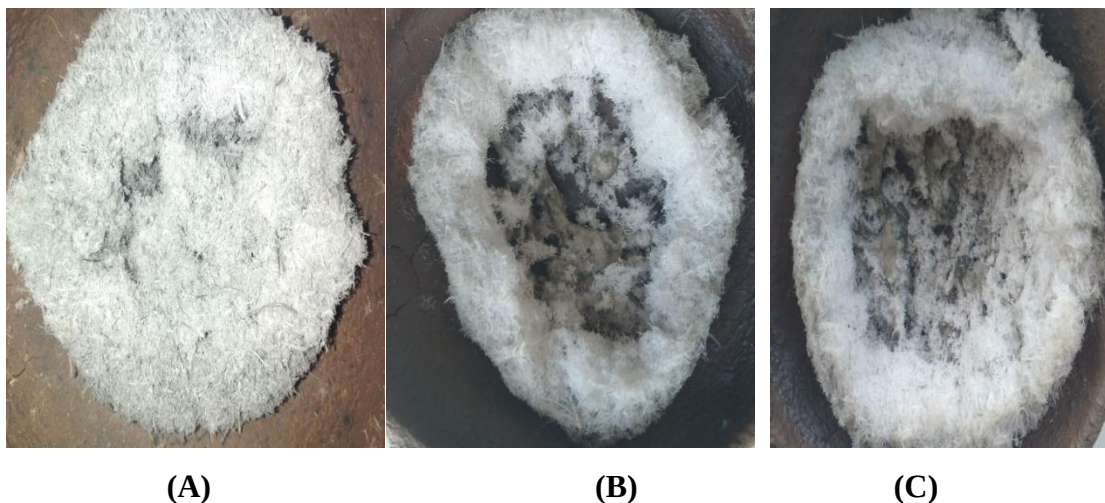


Figure 4.1— BTSA samples at combustion temperature of (A) 600°C, (B) 750°C & (C) 900°C

As it can be seen in the above picture the Brown Teff Straw Ash formed after combustion starts to form a small amount of solid object at a combustion temperature of 750°C. The solid object formed at this stage is very soft and can easily be broken using hand but as the temperature increases the solid object becomes a litter stiffer than before. The amount of solid object formation increases as the temperature increases from 750°C to 900°C and it became constant after 900°C.



Figure 4.2— BTSA sample after grinding of the ash for the combustion temperature of 750°C



Figure 4.3— Solid object formed during combustion of BTS samples at a temperature of 900°C

4.2. Mass retained of Brown Teff Straw Ash

During Combustion of the grinded Brown Teff Straw sample in a muffle furnace at different temperatures a significant mass loss occurred. The mass retained curve shows a slight decrease in mass up to 200°C and a steep decline in mass occurred until it reaches 450°C. From 450°C - 900°C there is a gradual change in mass of the sample and finally it becomes constant after it reaches 900°C.

sample s No.	Combustion temperature										
	0 ⁰ C	100 ⁰ C	200 ⁰ C	300 ⁰ C	400 ⁰ C	500 ⁰ C	600 ⁰ C	700 ⁰ C	800 ⁰ C	900 ⁰ C	1000 ⁰ C
S1	12	11.7	11.4	8.6	5.6	4	3.1	2.3	1.8	1.1	1.1
S2	12	11.5	11.1	7.4	4.4	3.4	2.7	2.1	1.5	1	1
S3	12	11.6	11.3	8.3	5.2	3.8	2.9	2.2	1.7	1.1	1.1
Average	12	11.6	11.27	8.1	5.07	3.73	2.9	2.2	1.67	1.07	1.07

Table 4.1— Mass retained during combustion of Brown Teff Straw (BTS)

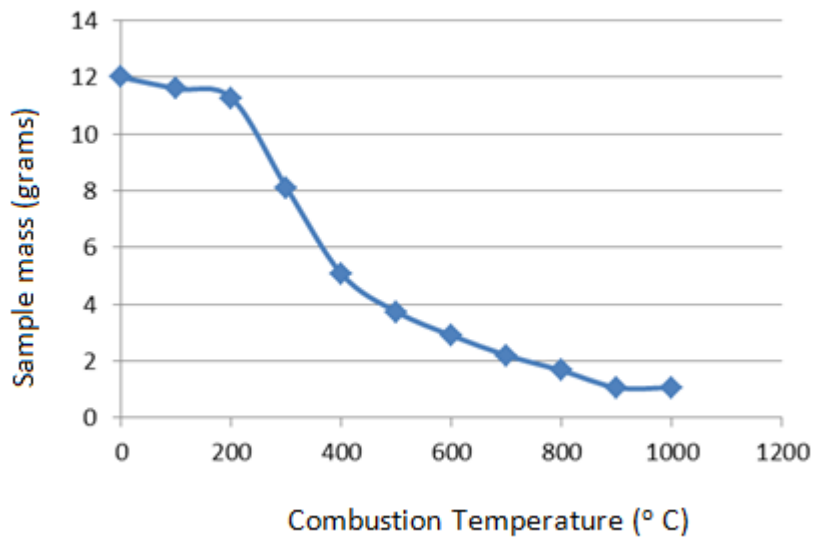


Figure 4.4— Average mass retained curve of Brown Teff straw ash (BTSA)

4.3. Chemical analysis

The results of complete silicate analysis for White Teff Straw Ash (WTSA) and Brown (BTSA) samples from Geological survey of Ethiopia laboratory are presented below

Chemical properties WTSA for Combustion time of 6hrs.												
Chemical composition (wt. %)												
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
BTSA-600°C	30.32	<0.01	0.68	0.94	1.12	0.1	10.38	<0.01	1.89	0.03	4.84	51.04

Table 4.2— Chemical composition of WTSA for combustion temperatures of 600°C

The combined chemical composition for WTSA-600°C; $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 31 < 50\%$. Therefore, WTSA is not a pozzolanic material and cannot be used for cement replacement purpose because it does not show the characteristics to be categorized as pozzolanic materials as per ASTM C- 618 specifications.

Chemical properties BTSA for Combustion time of 6hrs.												
Chemical composition (wt. %)												
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
BTSA-600°C	56.18	7.80	1.14	6.04	1.88	<0.01	16.8	0.10	0.96	0.03	1.03	7.24
BTSA-750°C	74.92	2.05	0.62	5.06	3.52	<0.01	7.84	0.14	6.59	0.02	<0.01	<0.01
BTSA-900°C	69.6	<0.01	1.04	4.70	3.50	<0.01	13.78	0.08	6.48	0.02	0.14	1.27
BTSA-900°C	70.90	3.24	0.84	5.72	3.98	0.64	7.56	0.20	5.80	0.04	0.54	2.02
BTSA-900°C	66.06	<0.01	0.64	4.18	3.24	0.60	17.08	0.08	6.27	0.05	<0.01	1.27

Table 4.3— Chemical composition of BTSA for combustion temperatures of 600°C, 750°C & 900°C

The combined chemical composition for BTSA-600°C; $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 65.12 > 50\%$ will be categorized under Class C pozzolanic materials as per ASTM C- 618 specifications.

The combined chemical composition for BTSA-750°C; $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 77.59 > 70\%$ will be categorized under Class F pozzolanic materials as per ASTM C- 618 specifications.

The combined chemical composition for BTSA-900°C; $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 70.64 > 70\%$ will be categorized under Class F pozzolanic materials as per ASTM C- 618 specifications.

The silicate content of the ash increases when temperature increases from 600°C to 750°C and decreases from 750°C to 900°C. This decrease in silicate content at 900°C occurred because of decomposition of minerals at high temperature as it can be seen from the Loss on ignition Value. Two additional chemical analysis tests are performed at

900°C to confirm the decomposition/ mass loss at this temperature. The LOI at 900°C is greater than that of 750°C due to loss of weight or decomposition of major oxides at higher temperature.

4.4. Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) carried out to find the shape and size of Brown Teff Straw Ash (BTSA) particles in dry form are presented below

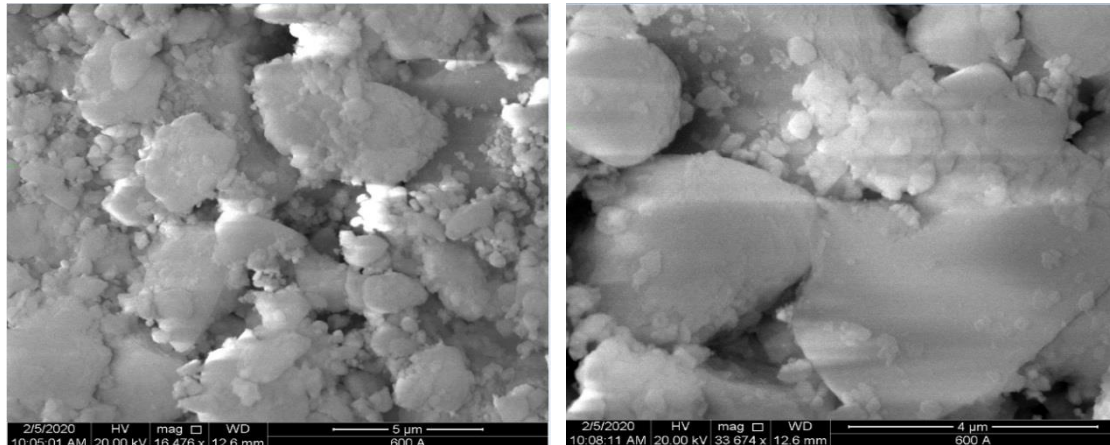


Figure 4.5— SEM result for combustion temperature of 600°C

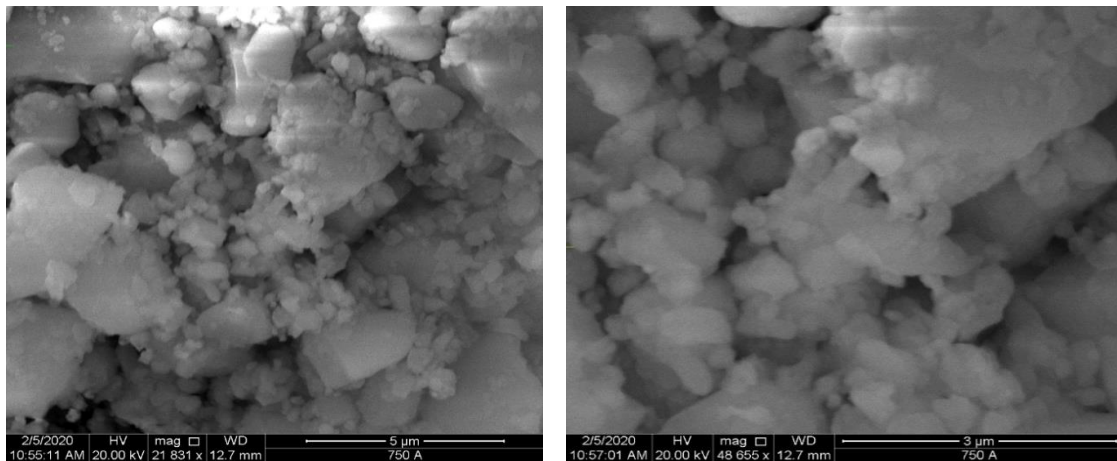


Figure 4.6— SEM result for combustion temperature of 750°C

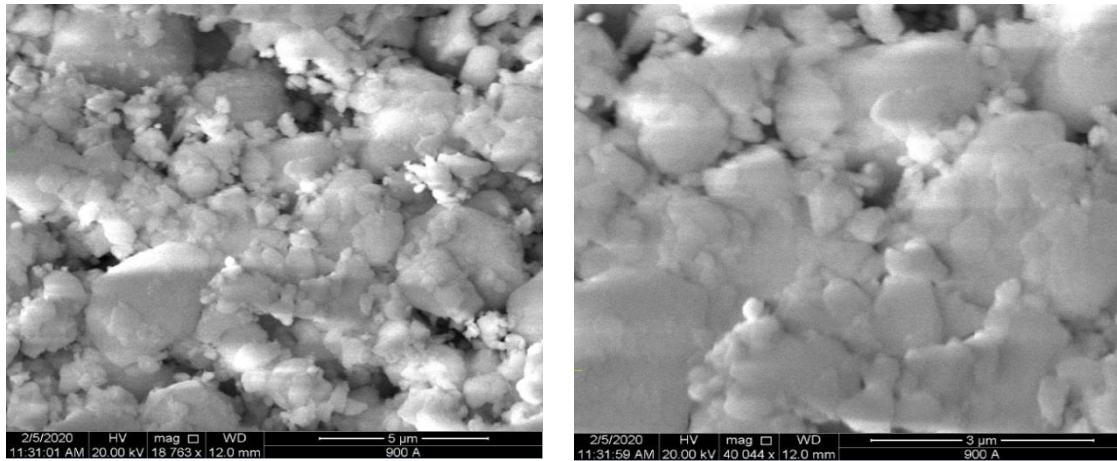


Figure 4.7— SEM result for combustion temperature of 900°C

The Scanning Electron Microscopy (SEM) result shows that Brown Teff Straw Ash (BTSA) particles have flaky and oval type irregular shapes and most of the particles of the ash are below 3 µm in size.

The flaky and oval type irregular morphology of Brown Teff Straw Ash in the SEM result images indicates that the surfaces are more loosely bound which make them highly amorphous and reactive. The SEM images also show that many residual pores are distributed within the ash samples, indicating that the silica is an active and highly porous material.

4.5. X-ray diffraction (XRD)

X-ray diffraction is done to examine amorphous and crystalline structures of Brown Teff straw ash (BTSA) particles in dry form.

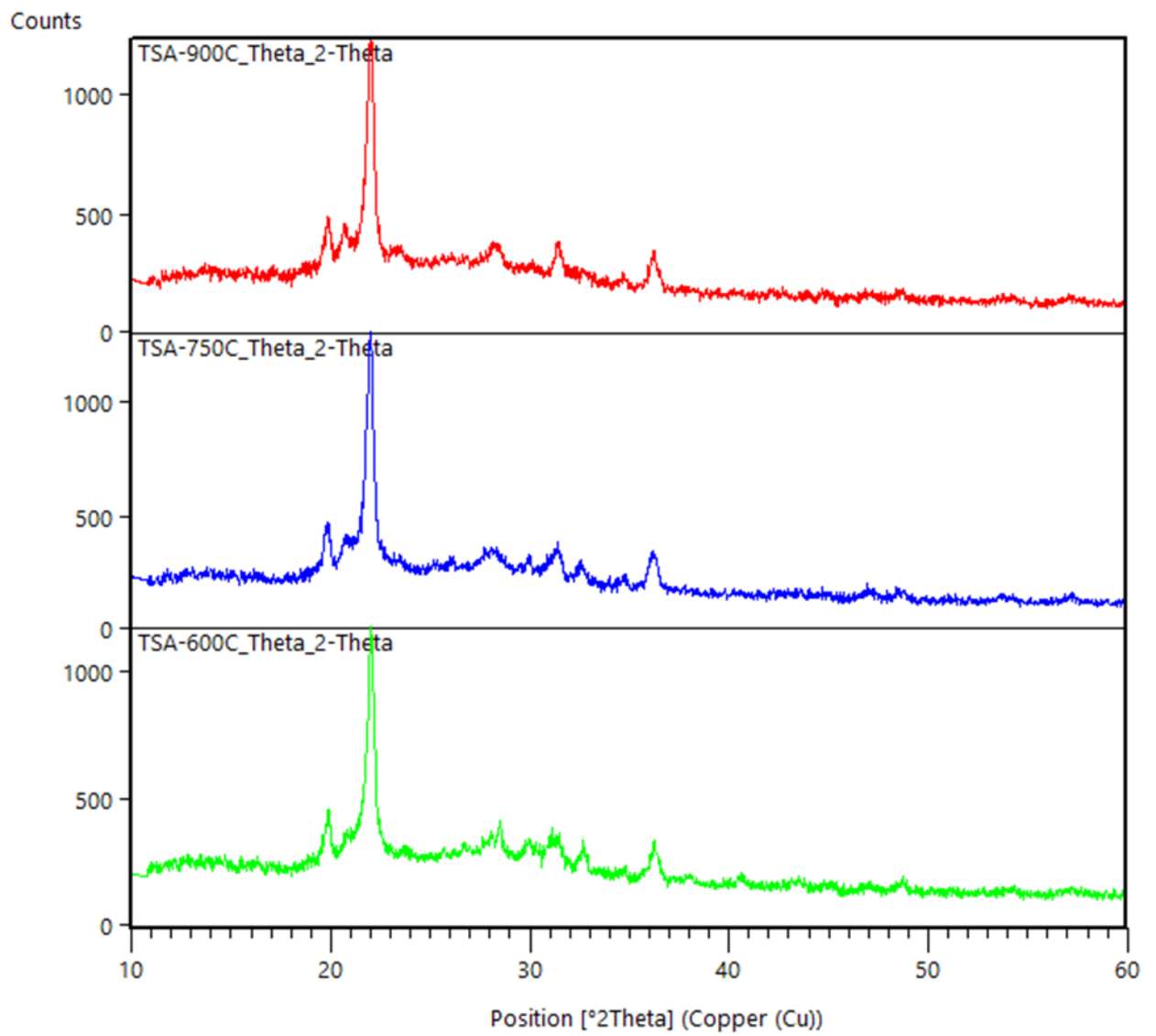


Figure 4.8— 2D-XRD result for combustion temperature of 600°C, 750°C & 900°C

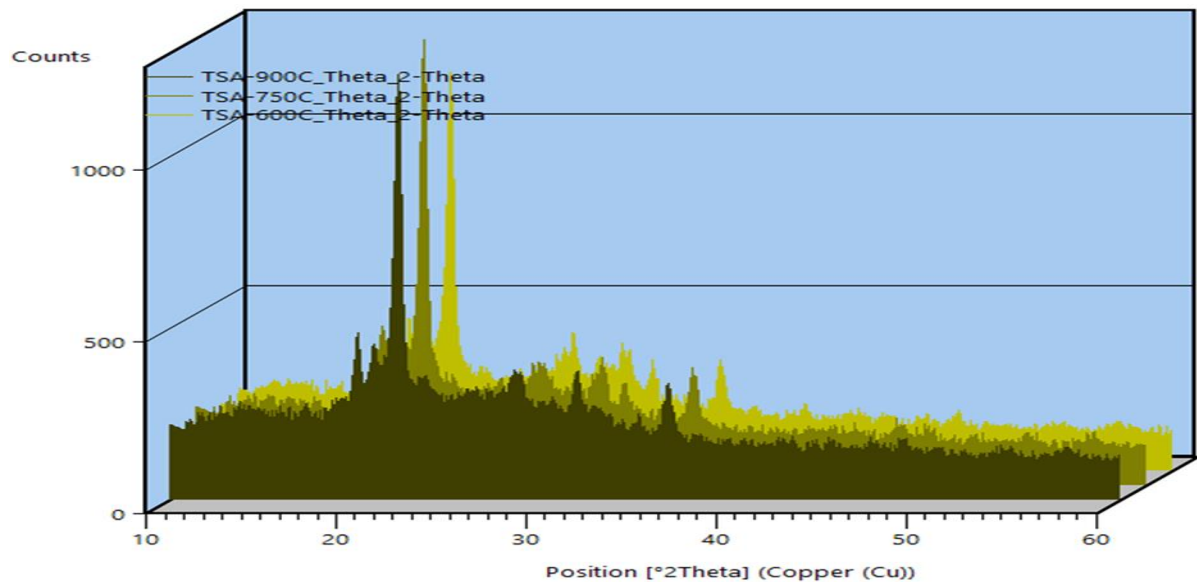


Figure 4.9— 3D-XRD result for combustion temperature of 600°C, 750°C & 900°C

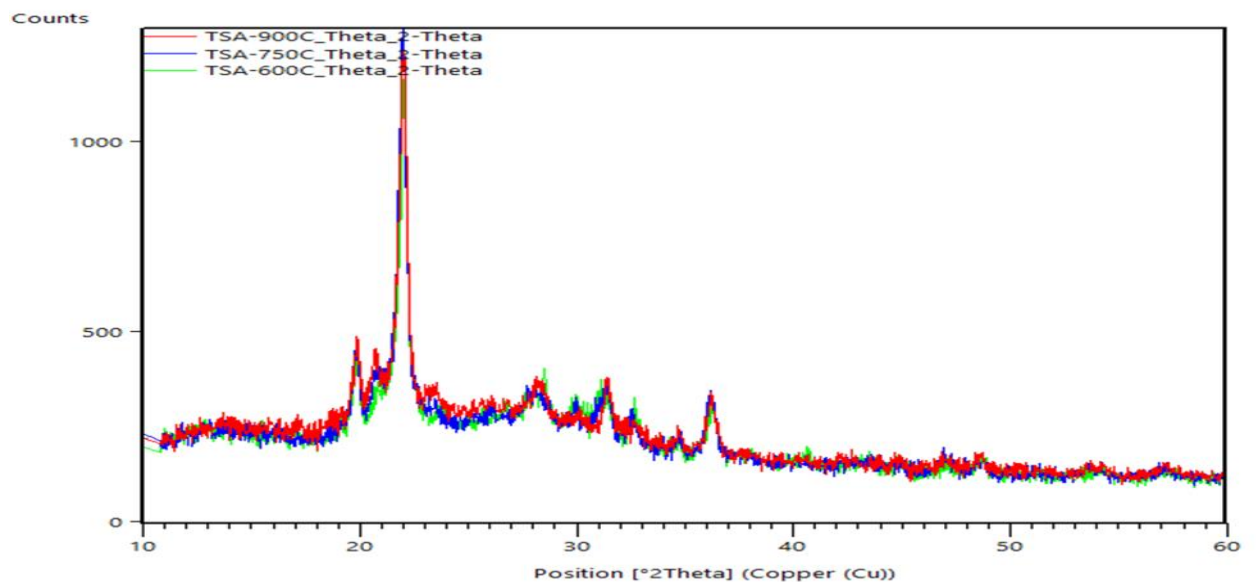


Figure 4.10— Comparison of XRD result for combustion temperature of 600°C, 750°C & 900°C

From X-ray diffraction result an increase of peak intensity of various peaks was observed. The highest pick value for all temperatures is around 2θ and as temperature increases the value of the pick increases due to formation of crystalline silica.

Overheating of amorphous silica above 700°C may transform it to crystalline forms of silica. The presence of these crystalline phases reduces the reactivity of silica in the

synthesis of other industrial products. [13] Therefore, as it can be seen from the above graphs the amount of crystalline silica increases with the increasing temperature.

4.6. Fineness (Blain air) of Brown Teff straw Ash (BTSA)

The fineness of Brown Teff straw Ash (BTSA) is calculated using the formula provided in chapter 3.

The weight of the sample needed for the fineness test is determined using the following formula provided in chapter 3.

$$\begin{aligned} W &= \rho v (1 - \epsilon) \\ W &= 2.3 \frac{g}{cm^3} * 1.852 cm^3 (1 - 0.5) \\ w &= 2.1298 \text{ grams} \end{aligned}$$

The specific surface area of the samples is calculated using the following formula provided in chapter 3.

$$\begin{aligned} S &= \frac{S_s \sqrt{T}}{\sqrt{T_s}} \\ S &= \frac{3774 cm^2 / g \sqrt{32.14 sec}}{\sqrt{37.8 sec}} \\ S &= 3478 cm^2 / g \end{aligned}$$

The surface area of the ash is smaller than the surface area of cement. Therefore, Brown Teff straw ash (BTSA) particles are fine relative to that of cement.

4.7. Compressive strength of cement mortars

Mortar, which is a mixture consisting of fine aggregate (standard sand for this study), adhesive material (Brown Teff Straw Ash, Portland cement) and water, were cured and tested at curing ages of 1day, 3days, 7days and 28 days age. The compressive strength values of mortar increased by 12.94% at the age of 28days for the sample with 10% of BTSA-600°C replacement. The compressive strength values of mortar increased by 5.94% at the same proportion of BTSA replacement and test date of BTSA-750°C replacement. But for 900°C the compressive strength values of mortar decreased by 10.45% at the same proportion of BTSA replacement and test date as above. Maximum compressive strength values of 32.3 Mpa for 28 days curing day were achieved when BTSA were added to mortars at 10% replacement by volume.

Load (KN)							
Percentage of replacement	Code	Cube No.	Test days				
			1day	3days	7days	14days	28 days
0%	BTSA0%	Cube 1	11.05	28.58	43.55	55.68	69.88
		Cube 2	12.38	32.98	49.70	64.28	73.13
			Average Value				
			11.71	30.78	46.63	59.98	71.50
10%	BTSA-600°C	Cube 1	13.90	34.48	49.43	60.95	76.88
		Cube 2	15.20	38.58	57.50	65.38	84.63
			Average Value				
			14.55	36.53	53.46	63.16	80.75
20%	BTSA-600°C	Cube 1	10.73	25.60	36.88	48.10	58.95
		Cube 2	12.33	29.80	39.18	55.90	67.55
			Average Value				
			11.53	27.70	38.03	52.00	63.25
10%	BTSA-750°C	Cube 1	11.45	30.48	54.08	56.30	70.45
		Cube 2	13.30	33.98	58.08	64.13	81.05
			Average Value				
			12.38	32.23	56.08	60.21	75.75
20%	BTSA-750°C	Cube 1	8.95	22.48	34.43	44.88	55.68
		Cube 2	10.25	24.13	39.73	47.83	61.33
			Average Value				
			9.60	23.30	37.08	46.35	58.50
10%	BTSA-900°C	Cube 1	8.85	30.58	37.63	52.33	62.65
		Cube 2	10.25	34.73	43.08	56.93	65.35
			Average Value				
			9.55	32.65	40.35	54.63	64.00
20%	BTSA-900°C	Cube 1	7.28	24.00	34.63	39.18	51.28
		Cube 2	8.15	25.45	35.33	42.93	55.23
			Average Value				
			7.71	24.73	34.98	41.05	53.25

Table 4.4— Maximum failure load of mortar cubes

Compressive strength test result (N/mm ²)							
Percentage of replacement	Code	Cube No.	Test days				
			1day	3days	7days	14days	28 days
0%	BTSA0%	Cube 1	4.42	11.43	17.42	22.27	27.95
		Cube 2	4.95	13.19	19.88	25.71	29.25
			Average Value				
			4.69	12.31	18.65	23.99	28.60
10%	BTSA-600°C	Cube 1	5.56	13.79	19.77	24.38	30.75
		Cube 2	6.08	15.43	23.00	26.15	33.85
			Average Value				
			5.82	14.61	21.39	25.27	32.30
20%	BTSA-600°C	Cube 1	4.29	10.24	14.75	19.24	23.58
		Cube 2	4.93	11.92	15.67	22.36	27.02
			Average Value				
			4.61	11.08	15.21	20.80	25.30
10%	BTSA-750°C	Cube 1	4.58	12.19	21.63	22.52	28.18
		Cube 2	5.32	13.59	23.23	25.65	32.42
			Average Value				
			4.95	12.89	22.43	24.09	30.30
20%	BTSA-750°C	Cube 1	3.58	8.99	13.77	17.95	22.27
		Cube 2	4.10	9.65	15.89	19.13	24.53
			Average Value				
			3.84	9.32	14.83	18.54	23.40
10%	BTSA-900°C	Cube 1	3.54	12.23	15.05	20.93	25.06
		Cube 2	4.10	13.89	17.23	22.77	26.14
			Average Value				
			3.82	13.06	16.14	21.85	25.60
20%	BTSA-900°C	Cube 1	2.91	9.60	13.85	15.67	20.51
		Cube 2	3.26	10.18	14.13	17.17	22.09
			Average Value				
			3.09	9.89	13.99	16.42	21.30

Table 4.5— Average compressive strength values of mortar

The compressive strength using 10% replacement of cement by BTSA for the code BTSA-600°C (Brown Teff Straw Ash at combustion temperature of 600°C) shows increase in values from that of 0% (mortar with no ash replacement added) mortar mix . For 20% replacement of cement by BTSA for the code BTSA-600°C (Brown Teff straw Ash at combustion temperature of 600°C) shows decrease in values from that of 0% (mortar with no ash replacement added) mortar mix.

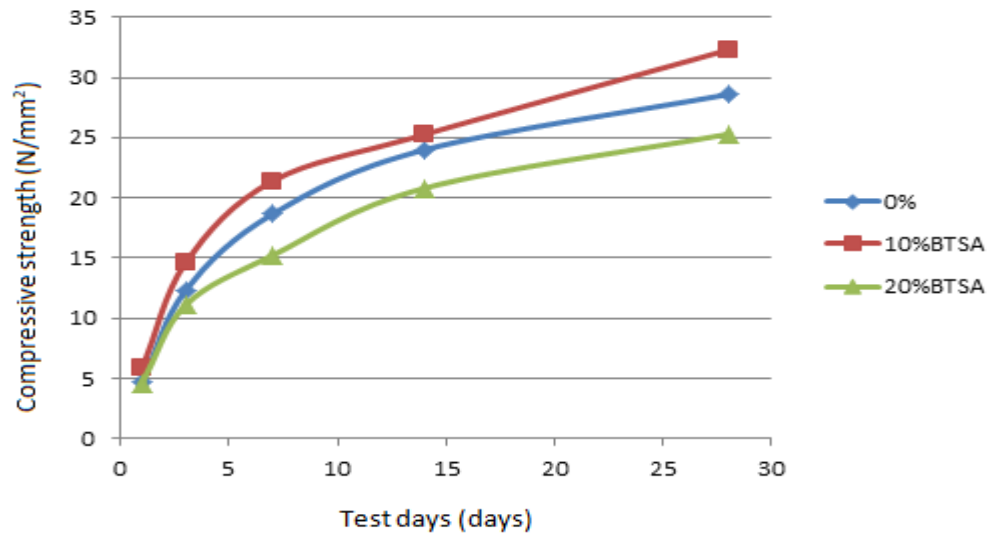


Figure 4.11 — Average compressive strength values of mortar for 10% & 20% BTSA-600°C relative to BTSA0%

Similar to the test made on combustion temperature of 600°C, The compressive strength using 10% replacement of cement by BTSA for the code BTSA-750°C (Brown Teff Straw Ash at combustion temperature of 750°C) shows increase in values from that of 0% (mortar with no ash replacement added) mortar mix . For 20% replacement of cement by BTSA for the code BTSA-750°C (Brown Teff straw Ash at combustion temperature of 750°C) shows decrease in values from that of 0% (mortar with no ash replacement added) mortar mix.

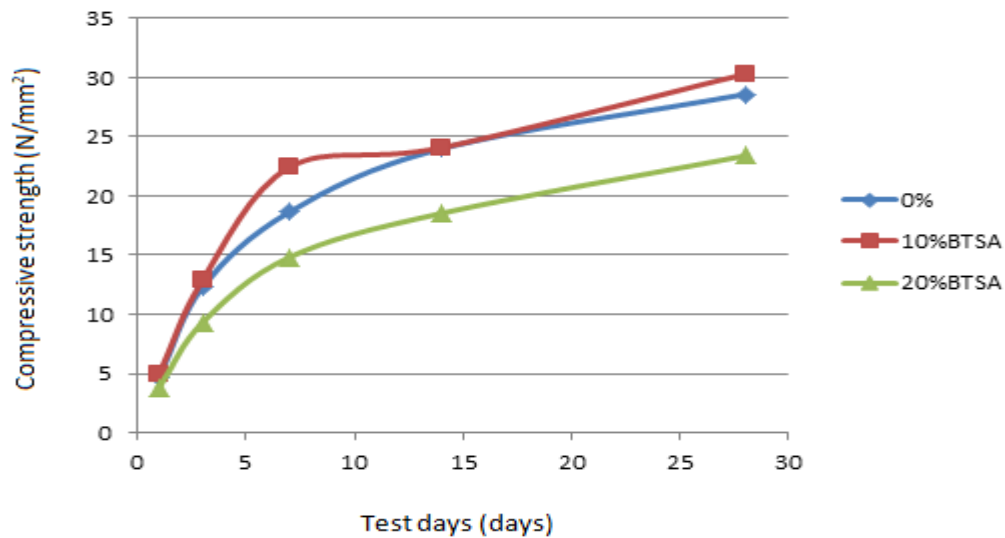


Figure 4.12— Average compressive strength values of mortar for 10% & 20% BTSA-750°C relative to BTSA0%

At combustion temperature of 900°C, The compressive strength using 10% replacement of cement by BTSA for the code BTSA-900°C (Brown Teff Straw Ash at combustion temperature of 900°C) shows decrease in values from that of 0% (mortar with no ash replacement added) mortar mix for all test dates except for the 3rd day . For 20% replacement of cement by BTSA for the code BTSA-900°C (Brown Teff Straw Ash at combustion temperature of 900°C) shows decrease in values from that of 0% (mortar with no ash replacement added) mortar mix. 10% replacement of cement by BTSA for the code BTSA-900°C (Brown Teff Straw Ash at combustion temperature of 900°C) shows increase in values from that of 20% replacement of cement by BTSA for the code BTSA-900°C (Brown Teff Straw Ash at combustion temperature of 900°C).

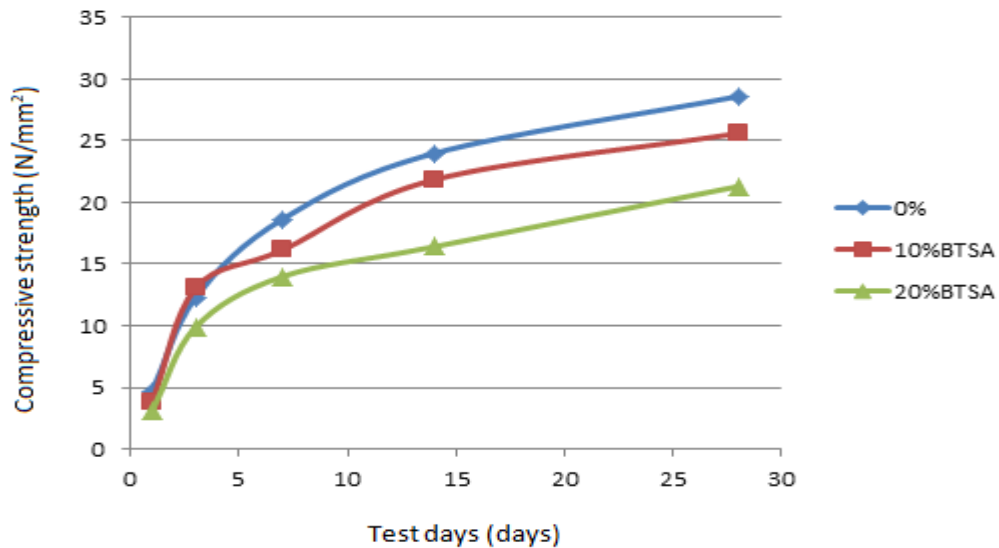


Figure 4.13— Average compressive strength values of mortar for 10% & 20% BTSA-900°C relative to BTSA0%

As it can be seen in the graph below for 10% replacement of cement by BTSA for the ashes produced using combustion temperature of 600°C, 750°C and 900°C the highest values of compressive strength occur for the concrete cubes tested for BTSA- 600°C except at the 7th day test the test result of BTSA-750°C increased by 4.86% from that of BTSA-600°C. 3days compressive strength for 900°C is 1.32% greater than that of 750°C.

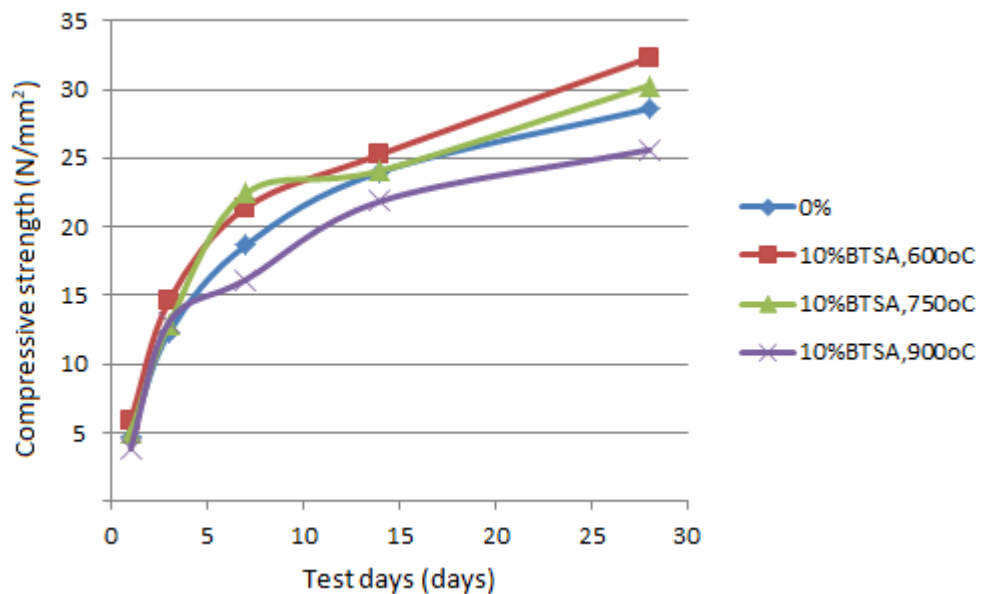


Figure 4.14— Comparison of Average compressive strength values of mortar at BTSA-600°C, BTSA-750°C & BTSA-900°C for 10% replacement

As shown in the graph below for 20% replacement of cement by BTSA for the ashes produced using combustion temperature of 600°C, 750°C and 900°C all values of compressive strength result are lower than that of 0% mortar mix. Comparing the compressive strength results of only 600°C, 750°C and 900°C, the maximum result occur for the concrete cubes tested for BTSA- 600°C. 3days compressive strength for 900°C is 6.12% greater than that of 750°C.

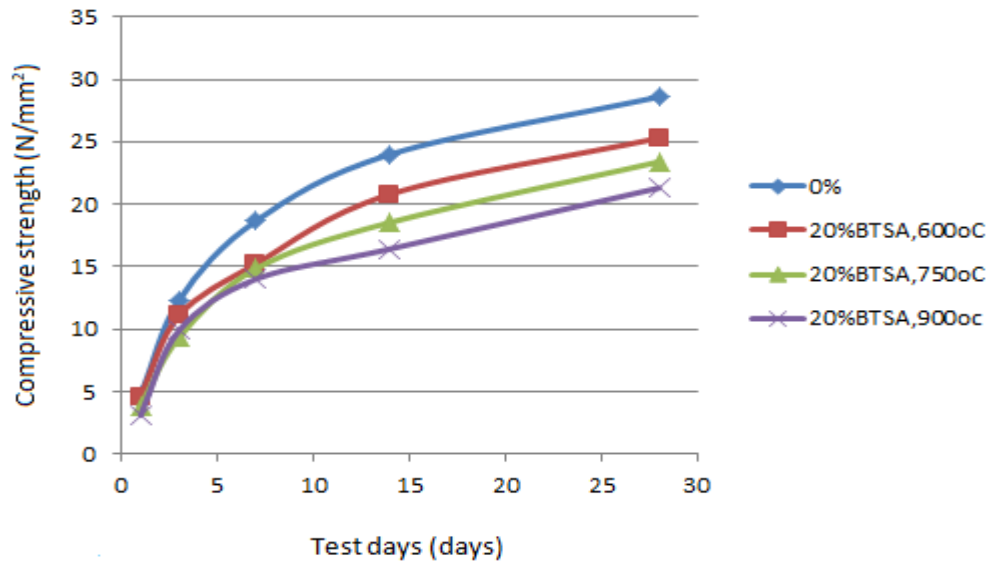


Figure 4.15— Comparison of Average compressive strength values of mortar at BTSA-600°C, BTSA-750°C & BTSA-900°C for 20% replacement

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATION

5.1. Conclusion

Brown Teff Straw Ashes obtained after control burning of BTS at 600°C for 6 hours were reasonably reactive given by the fact that little crystallization of minerals occurred but after 750°C up to 900°C the reactivity of the ash decreased due to increase in crystalline form of the major oxides.

SEM study of the blended pastes confirmed the Brown Teff Straw Ash (BTSA) particles have oval type irregular shapes and most of the particles of the ash are below 3 µm in size.

XRD study of the blended pastes confirmed the amount of crystalline silica increases with the increasing temperature.

Compressive strength tests confirmed the actual behavior of BTSA blended mortars and it suggested that up to 10% substitution by volume of OPC with BTSA can be made with better strength results than that with pure cement. Therefore, BTSA shows a good potential to be used as a pozzolanic material.

Replacement of cement by up to 10% in concrete production was found to enhance the compressive strength by 12.94% at the age of 28days for the sample with 10% of BTSA-600°C. The use of BTSA saves production cost of concrete and reduces emission of CO₂ to the environment due to high energy use while production of cement.

5.2. Recommendation

Since cement price is volatile and demand is so high, an alternate material can be used for replacement of cement.

This paper studies the effect of BTSA as cement replacing materials by analyzing the effect on the compressive strength of mortar mixes. In addition to the compressive strength of mortar cubes, other properties such as compressive & flexural strength of concrete cubes and water permeability can be improved using BTSA. Further research is recommended in this regard. Also, further studies are recommended to investigate the effect of washing BTS with chemicals.

Because cement production is an energy-intensive process it is necessary to reduce both the cost of production and emission of CO₂ to the atmosphere by use of alternative building materials that are locally available and use low combustion temperature and

have the capacity enhance the strength of cement by replacing it partially. Therefore, BTSA shows a good potential to be used efficiently as cement replacing material.

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APPENDIX

➤ Specific Gravity of Brown Teff Straw Ash (BTSA)

Geological Survey of Ethiopia
Mineralogy & Geotechnical Laboratory Directorate
Result Form



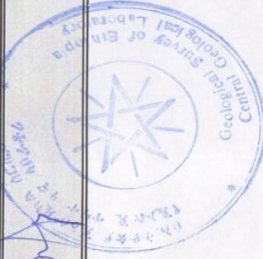
Directorate: - Mineralogy and Geotechnical Laboratory Directorate
Client / Originator Name: - Selamawit W/Michael
Client Category: - Survey Gov. ☐ Pvt. ☒
File name: - 0015/20 PVT
Sample Type: - Ash Powder
Type of Analysis: - Specific gravity
Lab section: - Mineralogy ☐ Physical ☒
No of Samples: - 1
Sample No.
Date Submitted: - 31/12/19
Preparation required: -

Coll.No.	Lab. No.	Pycnometer No.	m ₂ Mass of pycnometer in g	m ₃ Mass of test solution in the pycnometer without test sample in g	Q ₂ Density of test solution in g/cm ³	m ₄ Mass of pycnom. plus test sample in g	m ₄ -m ₂ mass of test sample in g	m ₅ mass of pycnom. test sample and solution in g	m ₅ +m ₄ -m ₅ volume of test sample in g/cm ³	Specific Gravity in g/cm ³	Average
TSA-600 °c	60/60	29/29	29.246	78.9452	1 g/cm ³	37.3191	8.0731	83.4711	3.5472	2.27	2.30
	0015/20		28.19	78.0636	1 g/cm ³	37.053	8.863	83.1313	3.7953	2.33	

Described By / Analysts: -
Aregahagn Kefelegn 

Checked by: -
Girma Asemu 

Date Completed: - 08/01/20



- Complete silicate analysis results for White Teff Straw Ash at 600°C

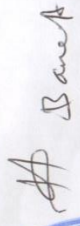
		GEOLOGICAL SURVEY OF ETHIOPIA GEOCHEMICAL LABORATORY DIRECTORATE Complete Silicate Analysis Report		GLD/F5.10.2 Page 1 of 1
Document Title:		Effective date:		May, 2017

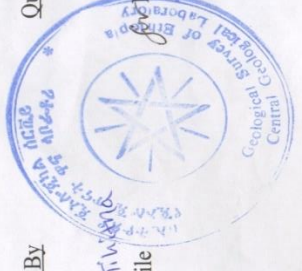
Customer Name:- Selamawit W/michael
 Issue Date: -26/12/2019
 Request No:- GLD/RN/865/19
 Report No:- GLD/TR/803/19
 Sample Preparation:- 200 Mesh
 Number of Sample:- One (1)

Sample type:- Ash
 Date Submitted: - 03/12/2019
 Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₃ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
TSA 600c0, 6hrs	30.32	<0.01	0.68	0.94	1.12	0.10	10.38	<0.01	1.89	0.03	4.84	51.04

Note: - This result represent only for the sample submitted to the laboratory.

Analysts Yirgalem Abriham Tihitna Beletkachew Tizita Zemene Nigist Fikadu	Checked By  Yohannes Getachew	Approved By  Gosa Haile	Quality Control  Negash Worku
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- Complete silicate analysis results for Brown Teff Straw Ash at 600°C, 750°C and 900°C respectively

	GEOLOGICAL SURVEY OF ETHIOPIA		Doc Number: GLD/F5.10.2	Version No: 1
	GEOCHEMICAL LABORATORY DIRECTORATE			Page 1 of 1
	Document Title: Complete Silicate Analysis Report		Effective date:	May, 2017

Issue Date: 26/12/2019
 Request No:- GLD/RN/916/19
 Report No:- GLD/TR/809/19-A
 Sample Preparation: - 200 Mesh
 Number of Sample:- One (1)

Customer Name:- Selamawit W/michael
 Sample type:- Powder
 Date Submitted: - 19/12/2019
 Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₂ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
TSA 600c0	56.18	7.80	1.14	6.04	1.88	<0.01	16.80	0.10	0.96	0.03	1.03	7.24

Note: - This result represent only for the sample submitted to the laboratory.

Analysts

Yirgalem Abriham

Tihina Beletkachew

Tizita Zemene

Nigist Fikadu

Checked By



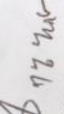
Yohannes Getachew

Approved By



Gosa Haile

Quality Control



Negash Worku



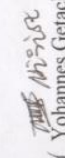
	GEOLOGICAL SURVEY OF ETHIOPIA		Doc. Number: GLD/F5.10.2	Version No: 1
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Document Title: Complete Silicate Analysis Report		Effective date: May, 2017		

Customer Name:- Selamawit W/Michael
 Issue Date: -19/02/2020
 Request No:- GLD/RQ/133/20
 Report No:- GLD/RN/100/20
 Sample Preparation:- 200 Mesh
 Number of Sample:- One (1)
 Sample type:- Ash powder
 Date Submitted: - 03/02/2020
 Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₂ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
TSA 750c	74.92	2.05	0.62	5.06	3.52	<0.01	7.84	0.14	6.59	0.02	<0.01	<0.01

Note:- This result represent only for the sample submitted to the laboratory.

Analysts
 Yirgalem Abriham
 Tizita Zemene
 Nigist Fikadu

Checked By

 Yohannes Getachew

Approved By

 Gosa Haile

Quality Control

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Customer Name: Selamawit W/Michael
 Issue Date: 16/03/2020
 Request No:- GLD/RQ/232/20
 Report No:- GLD/RN/190/20
 Sample Preparation: - 200 Mesh
 Number of Sample:- One (1)

Sample type:- Ash Powder
 Date Submitted:- 28/02/2020
 Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₂ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
TSA900c	69.96	<0.01	1.04	4.70	3.50	<0.01	13.78	0.08	6.48	0.02	0.14	1.27

Note: - This result represent only for the sample submitted to the laboratory.

Analysts
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 Nigist Fikadu

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 Yohannes Getachew

Approved By

 Gosa Haile



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Issue Date: -20/02/2020
 Request No:- GLD/RQ/138/20
 Report No:- GLD/RN/116/20
 Sample Preparation: - 200 Mesh
 Number of Sample:- One (1)

Customer Name:- Selamawit W/Michael
 Sample type:- powder
 Date Submitted: - 05/02/2020
 Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₂ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
TS900c	70.90	3.24	0.84	5.72	3.98	0.64	7.56	0.20	5.80	0.04	0.54	2.02

Note: - This result represent only for the sample submitted to the laboratory.

Analysts
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 Gosa Haile

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		GEOLOGICAL SURVEY OF ETHIOPIA GEOCHEMICAL LABORATORY DIRECTORATE		GLD/F5.10.2	Page 1 of 1
Document Title:		Complete Silicate Analysis Report			
Effective date:		May, 2017			

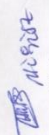
Customer Name: Selamawit W/Michael
 Issue Date: 29/05/2020
 Request No:- GLD/RQ/419/20
 Report No:- GLD/RN/349/20
 Sample Preparation: 200 Mesh
 Number of Sample:- One (1)

Sample type:- Ash Powder
 Date Submitted:- 18/03/2020

Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides
 Analytical Method: LiBO₂ FUSION, HF attack, GRAVIMETRIC, COLORIMETRIC and AAS

Collector's code	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
T-S-900 0c	66.06	<0.01	0.64	4.18	3.24	0.60	17.08	0.08	6.27	0.05	<0.01	1.27

Note:- This result represent only for the sample submitted to the laboratory.

<u>Analysts</u> Yirgalem Abriham Tizita Zemene Nigist Fikadu	<u>Checked By</u>  Yohannes Getachew	<u>Approved By</u>  Gosa Haile	<u>Quality Control</u>  Negash Worku
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➤ ASTM C 109 Compressive strength of hydraulic cement mortar cubes

ASTM C 109/C 109M – 07^{e1}

10.1.2 The quantities of materials to be mixed at one time in the batch of mortar for making six and nine test specimens shall be as follows:

	Number of Specimens	
	6	9
Cement, g	500	740
Sand, g	1375	2035
Water, mL		
Portland (0.485)	242	359
Air-entraining portland (0.460)	230	340
Other (to flow of 110 ± 5)	...	...

10.2 Preparation of Mortar:

10.2.1 Mechanically mix in accordance with the procedure given in Practice C 305.

10.3 Determination of Flow:

10.3.1 Determine flow in accordance with procedure given in Test Method C 1437.

10.3.2 For portland and air-entraining portland cements, merely record the flow.

10.3.3 In the case of cements other than portland or air-entraining portland cements, make trial mortars with varying percentages of water until the specified flow is obtained. Make each trial with fresh mortar.

10.3.4 Immediately following completion of the flow test, return the mortar from the flow table to the mixing bowl. Quickly scrape the bowl sides and transfer into the batch the mortar that may have collected on the side of the bowl and then remix the entire batch 15 s at medium speed. Upon completion of mixing, the mixing paddle shall be shaken to remove excess mortar into the mixing bowl.

10.3.5 When a duplicate batch is to be made immediately for additional specimens, the flow test may be omitted and the mortar allowed to stand in the mixing bowl 90 s without covering. During the last 15 s of this interval, quickly scrape the bowl sides and transfer into the batch the mortar that may have collected on the side of the bowl. Then remix for 15 s at medium speed.

10.4 Molding Test Specimens:

10.4.1 Complete the consolidation of the mortar in the molds either by hand tamping or by a qualified alternative method. Alternative methods include but are not limited to the use of a vibrating table or mechanical devices.

10.4.2 **Hand Tamping**—Start molding the specimens within a total elapsed time of not more than 2 min and 30 s after completion of the original mixing of the mortar batch. Place a layer of mortar about 1 in. or [25 mm] (approximately one half of the depth of the mold) in all of the cube compartments. Tamp the mortar in each cube compartment 32 times in about 10 s in 4 rounds, each round to be at right angles to the other and consisting of eight adjoining strokes over the surface of the specimen, as illustrated in Fig. 1. The tamping pressure shall be just sufficient to ensure uniform filling of the molds. The 4 rounds of tamping (32 strokes) of the mortar shall be completed in one cube before going to the next. When the tamping of the first layer in all of the cube compartments is completed, fill the compartments with the remaining mortar and then tamp as specified for the first layer. During tamping of the second layer, bring in the mortar forced out onto the tops of the molds after each round of tamping by means of the gloved fingers and

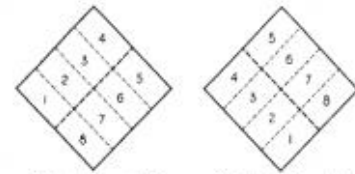


FIG. 1 Order of Tamping in Molding of Test Specimens

the tamper upon completion of each round and before starting the next round of tamping. On completion of the tamping, the tops of all cubes should extend slightly above the tops of the molds. Bring in the mortar that has been forced out onto the tops of the molds with a trowel and smooth off the cubes by drawing the flat side of the trowel (with the leading edge slightly raised) once across the top of each cube at right angles to the length of the mold. Then, for the purpose of leveling the mortar and making the mortar that protrudes above the top of the mold of more uniform thickness, draw the flat side of the trowel (with the leading edge slightly raised) lightly once along the length of the mold. Cut off the mortar to a plane surface flush with the top of the mold by drawing the straight edge of the trowel (held nearly perpendicular to the mold) with a sawing motion over the length of the mold.

10.4.3 **Alternative Methods**—Any consolidation method may be used that meets the qualification requirements of this section. The consolidation method consists of a specific procedure, equipment and consolidation device, as selected and used in a consistent manner by a specific laboratory. The mortar batch size of the method may be modified to accommodate the apparatus, provided the proportions maintain the same ratios as given in 10.1.2.

10.4.3.1 Separate qualifications are required for the following classifications:

Class A, *Non-air entrained cements*—for use in concrete, such as sold under Specifications C 150, C 595, and C 1157.

Class B, *Air-entrained cements*—for use in concrete, such as sold under Specifications C 150, C 595, and C 1157.

Class C, *Masonry, Mortar and Stucco Cements*—such as sold under Specifications C 91, C 1328, and C 1329.

10.4.3.2 An alternative method may only be used to test the cement types as given in 10.4.3.1 above, for which it has been qualified.

10.4.3.3 It can also be used for Strength Activity Index determinations for fly ash and slag, such as sold under Specifications C 618 and C 989, provided the alternative method has qualified for both Class A and Class C cements.

10.4.4 **Qualification Procedure**—Contact CCRL to purchase cement samples that have been used in the Proficiency Sample Program (PSP). Four samples (5 Kg each) of the class to be qualified will be required to complete a single qualification (See Note 7).

10.4.4.1 In one day, prepare replicate 6-cube or 9-cube batches using one of the cements and cast a minimum of 36 cubes. Complete one round of tests on each cement on different days. Store and test all specimens as prescribed in the sections below. Test all cubes at the age of 7-days.



11. Calculation

11.1 Record the total maximum load indicated by the testing machine, and calculate the compressive strength as follows:

$$f_m = P/A \quad (1)$$

where:

f_m = compressive strength in psi or [MPa],
 P = total maximum load in lbf or [N], and
 A = area of loaded surface in² or [mm²].

Either 2-in. or [50-mm] cube specimens may be used for the determination of compressive strength, whether inch-pound or SI units are used. However, consistent units for load and area must be used to calculate strength in the units selected. If the cross-sectional area of a specimen varies more than 1.5 % from the nominal, use the actual area for the calculation of the compressive strength. The compressive strength of all acceptable test specimens (see Section 12) made from the same sample and tested at the same period shall be averaged and reported to the nearest 10 psi [0.1 MPa].

12. Report

12.1 Report the flow to the nearest 1 % and the water used to the nearest 0.1 %. Average compressive strength of all specimens from the same sample shall be reported to the nearest 10 psi [0.1 MPa].

13. Faulty Specimens and Retests

13.1 In determining the compressive strength, do not consider specimens that are manifestly faulty.

13.2 The maximum permissible range between specimens from the same mortar batch, at the same test age is 8.7 % of the average when three cubes represent a test age and 7.6 % when two cubes represent a test age (Note 10).

NOTE 10—The probability of exceeding these ranges is 1 in 100 when the within-batch coefficient of variation is 2.1 %. The 2.1 % is an average for laboratories participating in the portland cement and masonry cement

reference sample programs of the Cement and Concrete Reference Laboratory.

13.3 If the range of three specimens exceeds the maximum in 13.2, discard the result which differs most from the average and check the range of the remaining two specimens. Make a retest of the sample if less than two specimens remain after discarding faulty specimens or discarding tests that fail to comply with the maximum permissible range of two specimens.

NOTE 11—Reliable strength results depend upon careful observance of all of the specified requirements and procedures. Erratic results at a given test period indicate that some of the requirements and procedures have not been carefully observed; for example, those covering the testing of the specimens as prescribed in 10.6.2 and 10.6.3. Improper centering of specimens resulting in oblique fractures or lateral movement of one of the heads of the testing machine during loading will cause lower strength results.

14. Precision and Bias

14.1 *Precision*—The precision statements for this test method are listed in Table 2 and are based on results from the Cement and Concrete Reference Laboratory Reference Sample Program. They are developed from data where a test result is the average of compressive strength tests of three cubes molded from a single batch of mortar and tested at the same age. A significant change in precision will not be noted when a test result is the average of two cubes rather than three.

14.2 These precision statements are applicable to mortars made with cements mixed, and tested at the ages as noted. The appropriate limits are likely, somewhat larger for tests at younger ages and slightly smaller for tests at older ages.

14.3 *Bias*—The procedure in this test method has no bias because the value of compressive strength is defined in terms of the test method.

15. Keywords

15.1 compressive strength; hydraulic cement mortar; hydraulic cement strength; mortar strength; strength

ANNEX

(Mandatory Information)

A1. ANALYSES OF TEST RESULTS FOR QUALIFICATION OF ALTERNATE COMPACTION METHODS

A1.1 *Calculation of Average Within-Batch Standard Deviation and Elimination of Outliers*—Tabulate the results for each cement sample (or round) in separate spreadsheets. In the spreadsheet, list results of each batch in columns and complete the calculations as shown in Table A1.1.

A1.1.1 Eliminate any outliers from the test data and repeat the calculations until none of the values lie outside the normal range.

A1.1.2 Tabulate the cube strengths with all the outliers eliminated and complete the calculations as shown in Table A1.2.

A1.2 *Summary of Results*—Compile the results of the four

rounds and complete the calculations as shown in Table A1.3. The number of outliers shall not exceed 5 % of the total number of tests when rounded to the nearest whole number (for example, 4 rounds $\times$ 4 batches $\times$ 9 cubes = 144 tests $\times$ (5%/100) = 7.2 or 7).

A1.3 *Precision Qualification*—Calculate the relative within batch error (RWBE %) as shown in Table A1.3. This value must be less than 2.1 % to comply with the limit established in Note 10 of this specification.

A1.4 *Bias Qualification*—The test results compiled in Table A1.3 are evaluated against three limits to demonstrate an acceptable qualification. The limits have been established