



**ADDIS ABABA UNIVERSITY  
ADDIS ABABA INSTITUTE OF TECHNOLOGY  
SCHOOL OF CHEMICAL AND BIO ENGINEERING**

**MSC THESIS ON  
CHARACTERIZATION AND USE OF SEWAGE SLUDGE ASH  
AS SUPPLEMENTARY CEMENTITIOUS MATERIAL FOR  
CONCRETE**

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This is to certify that thesis prepared by Mohammed Ali, entitled: characterization and use of sewage sludge ash as supplementary cementitious material for concrete, submitted in partial fulfillment of the requirement for the degree of Master of Science in environmental engineering complies with the regulation of University and meets the accepted standards with respect to originality and quality.

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## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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### **DECLARATION**

I declare that this thesis entitled “characterization and use of sewage sludge ash as supplementary cementitious material for concrete” has not been submitted in any form for another degree, diploma or award at any university or other institution of the ternary education. Whenever contribution of other are involved, every effort is made to indicate this clearly, with the reference to the literature and discussion. Information taken from published and unpublished work of other has been acknowledged in text and list of references is given. The thesis work was under guidance of Dr.-Ing. Shimelis Kebede (Assistant professor) chairman of Environmental engineering stream, and instructor in School of Chemical and Bio engineering, Addis Ababa University institute of Technology.

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## Characterization and use of sewage sludge ash as supplementary cementitious material for concrete

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### ABSTRACT

Disposal of sewage sludge is becoming a serious environmental problem. The incineration process is able to reduce the volume of sewage sludge. It is not a proper solution, so the sewage sludge ash generated after the incineration process, and it must be disposed to landfill. The general objective of this study was to investigate the properties and use of sewage sludge ash as supplementary cementation material for concrete. Sewage sludge was incinerated at temperature of 600 °C and 800 °C for 3 & 2.5 hours. The incinerated sewage sludge ash was sieved through size of 75 µm and investigates physical properties, and chemical compositions of sewage sludge ash were carried out. SSA is pozzolanic material satisfied the requirement of ASTM C618. Chemical composition of SSA incinerated at 600 °C and 800 °C were determined by Atomic Absorption spectrometer. AAS test has shown that the sewage sludge ash contained primarily these oxides such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO. The concrete specimens were prepared using 5 %, 10 %, 15 %, and 20 % of the two temperatures incinerated sewage sludge ash that is 600 °C and 800 °C, and design mix ratio of 1:2:4 with w/c ratio was 0.82. The cubic mold size of 10 cm x 10 cm x 10 cm was used to produce a concrete specimen, and the concrete tests have then been measured both at the fresh and hardened concrete. In the hardened concrete, the results of a concrete have shown that 600 °C SSA 5% & 800 °C SSA 10% replacement of OPC by sewage sludge ash achieved a higher compressive strength than the others percentage of set in this research at the age of the 28 days as compared with the control concrete (0 % SSA). The percentage of water absorption increased with increase in sewage sludge ash that incinerated at temperature of 600 °C & 800 °C, and all the blended (% SSA) concretes have shown a higher amount of water absorbed as compared with the control concrete (0 % SSA).

**Keyword:** Incineration, Sewage sludge ash, Characterization, Percentage of replacement, Concrete

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**LIST OF ABBREVIATIONS**

AAS	Atomic Absorption Spectrometer
ACI	American Concrete Institute
ASTM	American Society for Testing and Material
AASHTO	American Association of State Highway and Transportation Officials
BS	British Standard
C-S-H	Calcium Silicate Hydrates
cm <sup>3</sup>	cubic centimeter
ES	Ethiopian Standards
GHG	Green House gas
GGBF	Ground Granulated Blast Furnace Slag
LOI	Loss on Ignition
kg	Kilo gram
MPa	Mega Pascal
mg	milligram
PPC	Portland pozzolana Cement
PC	Portland cement
SCMs	Supplementary Cementation Materials
SS	Sewage sludge
ISSA	Incinerated sewage sludge ash
SSA	Sewage sludge ash
W/C	Water Cement ratio
OPC	Ordinary Portland cement
UK	United Kingdom

## **1 INTRODUCTION**

### **1.1 Background**

Sewage sludge is a by-product of suspended or dissolved organic and inorganic materials from the treatment process of municipal wastewater treatment plant. It contains mineral, organic and biological impurities in soluble, insoluble and colloidal form (Doh et al., 2017). The purpose of sewage treatment is to produce treated sewage water which is suitable for safe discharge into the environment and for re-use purposes (Seiw et al., 2016).

Sewage sludge is usually treated by several common methods which include anaerobic digestion, aerobic digestion and composting. Sewage sludge tends to accumulate heavy metals existing in the wastewater. The origin of sludge and treatment options affects the composition of sewage sludge and its content of heavy metals. In the past decades, sewage sludge was mainly disposed in landfills and the seas. However, space limitation on existing landfills as well as increasing environmental concerns such as ground water pollution from landfill leaching, odors emission and soil contamination have urged further investigation on various disposal methods. According to the working document of sewage sludge, two main types of pollution by sewage sludge were identified; heavy metals and certain groups of organic compound (Rizzardini & Goi, 2014).

In general, Cd, Pb and Zn represent the biggest concentrations of heavy metals in sewage sludge from domestic waste waters which can be a source of pollution for soils. Faeces contribute 60 % - 70 % of the Cd, Zn, Cu and Ni load in domestic wastewater and >20 % of the input of these elements in mixed wastewater from domestic and industrial premises. Other principal sources of metals in domestic wastewater include body care products, pharmaceuticals, cleaning products as well as liquid wastes (Seiw et al., 2016).

Sewage sludge ash (SSA) is a waste material obtained from the incineration of wastewater sludge (Seiw et al., 2016). Sewage sludge ash has been identified for potential use in various construction products, such as brick manufacture, artificial

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aggregate and cement replacement in concrete ( Dhir et al., 2012). The construction industry is a great consumer of resources and materials, which makes it a sector with an enormous potential for the use of waste materials generated by its own activities and those from other sectors. The use of such waste materials allows decreases the energy consumption, to preserve on renewable natural re-sources, and to reduce the higher amount of material that goes to landfills. However, in the cement industry, which has always been among the largest CO<sub>2</sub> emission sources, technical, economic, and legal challenges still play as remarkable obstacles against the widespread implementation of procedures to help mitigate this CO<sub>2</sub> emission ( Paya et al., 2014). In the present work, it was evaluated the potentialities of the sewage sludge, after calcinations process, as a partial substitute of Portland cement to produce mortars and concretes. The substitution content, in mass, of cement by SSA was 10 - 30 % for the mortars and 5 - 10 % for the high performance concrete (Barbosa & Filho, 2004).

Sewage sludge ash is primarily a silty material with some sand-size particles. The specific size range and properties of the sludge ash depend to a great extent on the type of incineration system and there are two major incineration systems such as, multiple hearth and fluidized bed, are employed in the United States. Approximately 80 percent of the incinerators used in the United States are multiple hearth incinerators. And sludge ash from multiple hearth incinerators will usually consist primarily of silty material mixed with some larger sand-sized particles. The formation of larger particles is normally the result of higher operating temperatures and the formation of clinkers. Fluidized bed furnaces produce only a very fine (silt-sized) ash. It is known the effect of these wastes as cement substitutes in matrices with the conventional binders. For example, previous work has shown that mortars fabricated with 10% of replacement by SSA meet the mechanical requirements of European standard in terms of early age compressive strength and nominal compressive strength (Garc esetal, 2008), and mixtures containing 30 % of SSA as cement replacement showed a higher compressive strength at the age of 7 days compared to the reference mixture.

## **1.2 Statement of problem**

Recently, continuing population growth of Ethiopia has greatly increased freshwater consumption. The result of this consumed freshwater increases the volume of wastewater and sewage sludge. The disposal of domestic sewage sludge is a challenging issue due to the lack of financial and technological burden for many municipalities especially in developing countries.

Sewage sludge originated from the process of wastewater treatment plant contains organic materials, heavy metals, and pathogens that are harmful to the environment and human healthy. This problem associated with landfill disposal of sewage sludge, from a management and materials handling perspective, landfill is perhaps the simplest solution. However, as landfill spaces become more limited and contaminate the ground water and surface water due to the landfill leachate.

The utilization of sewage sludge as fertilizers can be a solution to disposal problems. But increasing the use of sewage sludge as a fertilizer on agriculture that is causing a health problem from accumulation of heavy metals in the soil, heavy metal accumulate in the topsoil (long-term build up in the soil) due to sewage sludge application on farmland, and their uptake by crop plants, and the food systems have not been free from this heavy metal.

The incineration of sludge seems to be an interesting and acceptable solution because of the incineration of sewage sludge is a common technique that allows conversion of bulky sludge into practically inert, odorless ash. However, this incineration technique has one of the disadvantages that are producing the sewage sludge ash. Therefore, this study focus on the main components of sewage sludge incinerated ash and the use of SSA in a concrete material as a replacement of cement. The contribution of this study was regarded from the environment and economic perspective, which are minimized landfill leachate, odor, and space requirements. Not only this, also reduce cement contents by replacing with by the SSA during the concrete making process, and minimize the sewage sludge disposal cost due to reducing the volume of sewage sludge by incineration process. It is stated that the use of sewage sludge for cement

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manufacturing found to conserve the resources, reduce CO<sub>2</sub> emissions, and preserve energy and that the cement matrix can immobilize the SSA contents.

### **1.3 Scope of study**

The sewage sludge used in this research was obtained from Kality wastewater treatment plant in the Addis Ababa municipality. The sewage sludge was dried with by an oven at 105 °C for 24 hours. The oven dried sewage sludge was incinerated by using a furnace at the temperature of 600 °C and 800 °C for 3 & 2.5 hours respectively to produce sewage sludge ash and investigated physical properties and chemical composition of sewage sludge ash. The concrete specimens were tested at 3, 7 and 28 days. For each curing age, two concrete specimens were tested to get the average result of compressive strength, and the water absorption test, and leaching test were carried out that after the 28 days of curing age in a concrete.

### **1.4 Significance of the Study**

The results of this study could provide information that may encourage the use of Kality wastewater treatment plant sewage sludge ash as a partial replacement of Portland cement in concrete.

The advantages resulting from sewage sludge incineration include the decrease in weight and volume of wastes, the thermal destruction of toxic organic compounds, the recovery of energy and the minimization of odor ( Jolanta and Jarosław, 2014).

This study also important for the reduction of the landfill space requirements during disposal of sewage sludge from the plant, and also minimize other environmental impacts such as leachate. In generally, it has advantages for economic and environmental aspects, which was saved on the cement and also saving the environment from accumulation of sewage sludge when it was disposed.

## **1.5 Objectives**

### **1.5.1 General objective**

The general objective of this study was to investigate the properties and use of sewage sludge ash as supplementary cementation material for concrete.

### **1.5.2 Specific objectives**

- To prepare sewage sludge ash thermally temperatures at 600 °C and 800 °C.
- To investigate physical properties and chemical composition of sewage sludge ash.
- To evaluate the workability of a mixture of fresh SSA- cement concrete.
- To investigate the compressive strength, water absorption, and leaching test of hardened SSA- cement concrete.
- To find the optimum percentage of sewage sludge ash to be blended with cement.

## **2 LITERATURE REVIEW**

### **2.1 Concrete**

Concrete is the most commonly used modern construction materials. It forms the basis of the modern construction system. Many of our activities directly or indirectly are affected by concrete structures; the buildings we live and work in, the roads we drive on, the dams from which we get water and energy, etc. can be an example. The ability of concrete to be cast into any desired shapes and configurations is the reason for its versatility. The word concrete comes from a Latin word *concretus* which means to grow together, which implies that it is a composite of different materials. It is composed of coarse granular material called aggregate or filler which is embedded in a hard matrix of material (cement or binder with water) binding the aggregates together and filling the space formed between them. When the constituents are mixed with water the concrete solidifies and hardens due to a chemical reaction between the water and the cement called hydration, which finally forms a stone like material by binding the aggregates together. Concrete is mainly composed of cement, aggregate and water. Cementation materials, pozzolanic materials, filler materials, chemical admixtures, and some other additives may also be the constituents of concrete depending on the need and their availability. All the constituents have their own purpose in the concrete. Cement with water acts as a binding medium in which the aggregates which accounts 70 % to 80 % of the concrete are bound together to form the concrete. Economy, dimensional stability and wear resistance are the main reasons behind using aggregates. Different types of admixtures are used to modify the properties of ordinary concrete so as to make it suitable for any situation. If a concrete is to be suitable for a particular purpose, it is necessary to select the constituent materials and combine them in such a manner as to develop the special qualities required as economical as possible. Therefore the selection of constituents of concrete depends on the quality and economy of the particular concrete required (Hailu, 2011).

## **2.1.1 Constituent of Concrete**

### **2.1.1.1 Cement**

Cements, are finely ground inorganic materials with adhesive and cohesive properties which make them capable of uniting or bonding together fragments or particles of solids matter into a compact whole. In engineering purpose, cements are materials which form a paste which sets and harden by means of hydration reactions, and which after hardening retain its strength and stability even under water and can be molded or deformed but later it sets and hardens to a rigid mass (Adise, 2017).

#### **a. Chemical composition of Portland cement**

Portland cement is made by grinding together its principal raw materials, which are:

1. Argillaceous, for example silicates of alumina in the form of clays and shale's, and
2. Calcareous, for example calcium carbonate in the form of limestone, chalk, and marl which is a mixture of clay and calcium carbonate.

The mixture is then burned in a rotary kiln at a temperature between 1300 and 15000 °C. The material partially fuses into a clinker which is taken from the kilns, cooled and then passed on to ball mills where gypsum was added and it was ground to the required fineness. The result of a cement is allowed to contain small strictly limited percentages of materials not required that any disadvantages for some uses, such as iron oxide, and sulfur trioxide. Table 2.1 shows oxide composition ranges of Portland cement to indicate a general idea of the composition of cement.

Table 2.1: Oxide content of Portland cement raw materials (Adise, 2017)

<b>Oxides</b>	<b>CaO</b>	<b>SiO<sub>2</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>SO<sub>3</sub></b>	<b>MgO</b>	<b>Na<sub>2</sub>O</b>	<b>K<sub>2</sub>O</b>
<b>Ranges</b>	60-79%	17-25%	3-8%	0.5-6%	1-3%	0.1-4%	0.5-1.3%	0.5-1.3%

### **2.1.1.2 Aggregates**

Aggregates are the materials basically used as filler with binding material in the production of concrete and provide concrete with better dimensional stability and wear resistance. They are derived naturally from igneous, sedimentary and metamorphic rocks or manufactured from blast furnace slag, etc. It is therefore significantly important to obtain right type and quality of aggregates (fine and coarse) because aggregates occupy 60 % to 75 % of the concrete volume (70 % to 85 % by mass) and strongly influence the concrete's freshly mixed and hardened properties, mixture proportions, and economy. So that to proportion suitable concrete mixes, certain properties of aggregate which influence the paste requirement of fresh concrete such as shape and texture, size graduation, moisture content, specific gravity and bulk unit weight must be known. In addition to these, aggregates should be hard, strong, dense, durable, clear and free from veins and adherent coating; and free from injurious amounts of disintegrated pieces, alkali, vegetable matter and other deleterious substances.

Aggregates can be classified based on their origin, size, shape and unit weight, but most specifications such as Ethiopian, Indian and South African standards classified aggregates based on their sizes as fine and coarse aggregate depends on their most particles retained or passed on 4.75 mm sieve. But British standard uses 5 mm sieve size for classification. In addition to this, aggregates can be classified as light weight, normal weight and heavy weight depending on their unit weight (Kahsay, 2014).

### **2.1.1.3 Water**

Water is an important constituent in concrete. It chemically reacts with cement to produce the desired properties of concrete. Mixing water is a quantity of water that comes in contact with cement, impacts slump of concrete, and is used to determine water to cementations materials ratio of concrete mixtures. Strength and durability of concrete is controlled to a greater extent by water cement ratio. Concrete strength increases when less water is used during preparation of the mix. Although the hydration process consumes a certain amount of water, wet concrete actually contains more water than required for the hydration reactions. The excess water is added to

provide the wet mix with sufficient workability. Concrete needs to be workable so that it can be molded into the desired shape and consolidated to the required density. The quantity of water used divided by the amount of cement gives the water to cement ratio. Low water-cement ratio leads to high strength but low workability while a high water to cement ratio produces a low strength concrete but good workability. A careful balance of cement to water is therefore required when preparing the mix. Good quality water is required for the mixing of concrete. Natural water that is drinkable and has not pronounced taste or odor can be used as mixing water for making concrete. Salt water should not be used for mixing concrete as it causes a significant reduction in strength and large variations in setting time (Degefa, 2017).

### **2.1.2 Properties of Fresh Concrete**

Fresh concrete is defined as concrete at the state when its components are fully mixed but its strength has not yet developed. The properties of fresh concrete directly influence the handling, placing and consolidation, as well as the properties of hardened concrete. Some of important properties of fresh concrete are discussed as follows:-

#### **2.1.2.1 Workability**

Workability is a general term to describe the properties of fresh concrete. Workability is often defined as the amount of mechanical work required for full compaction of the concrete without segregation. This is a useful definition because the final strength of the concrete is largely influenced by the degree of compaction. A small increase in void content due to insufficient compaction could lead to a large decrease in strength. The primary characteristics of workability are consistency (or fluidity) and cohesiveness. Consistency is used to measure the ease of flow of fresh concrete (Adise, 2017).

Water content is the most important factor affecting the workability of concrete. Increasing the amount of water will increase the workability of the concrete. However the increase in water content of the mix will decrease the strength and also result in segregation and bleeding. When considering the effect of aggregate, the amount of

aggregate, the proportion of coarse and fine aggregate, and the shape, and texture of the aggregate particles affect the workability of concrete. Keeping the water content and cement content constant increasing the amount of aggregate reduces the workability of concrete. Spherical and smooth aggregate result in a more workable mix, whereas flat, elongated and rough aggregate particles will result in reduction of workability. The increase in the ambient temperature will reduce the workability of the concrete, due to increase of evaporation and rate of hydration caused by the higher temperature. The cement content and cement replacing materials also affect the workability. Higher cement content reduces workability. The effect of cement replacing materials depends on their nature. Finer materials result in reduction of workability while spherical materials increase it (Hailu, 2011).

### **2.1.3 Properties of Hardened Concrete**

#### **2.1.3.1 Compressive Strength**

Of the various strength properties of concrete, it is generally the compressive strength which attracts the greatest interest since it is this property which is made use of in designing building units of structural or of simple load bearing quality. In addition, it has a great practical and economic significance because the sections and sizes of the concrete structures are determined by it. Since most concrete structures are designed to resist compressive stress, it is this property which is usually prescribed by codes or standards (Azaria, 2005)

Of all, compressive strength of concrete is the most important one because the best quality of concrete is in its compression resistance or capacity. More over other strengths like flexural and tensile can be correlated to this property. The 3, 7 and 28 days compressive strength of concrete will be determined by a standard uniaxial compression test is accepted universally as a general index of concrete strength (Adise, 2017)

**a. Factors Affecting Compressive Strength**

Strength of concrete depends on the strength of aggregate, cement paste, the bond between the aggregate and the surrounding cement paste and the overall adhesion and compaction of the concrete particles. The cement paste characteristics depend on the characteristic of the cement, water/cement ratio, and the completeness of hydration. Aggregate properties like strength, shape and texture affect the concrete strength by the strength of aggregate, its effect on aggregate – cement paste bond and compaction.

*i. Water/Cement Ratio*

Water is mandatory for hydration reaction to proceed. But if the amount of water added is exceedingly much, the excess water remains free in the concrete forming capillary pores. These pores remain the concrete to be permeable and weak in strength. The strength of concrete increases as the water/cement ratio decreases provided that there is sufficient water for hydration of cement

*ii. Degree of Compaction*

Immediate upon placement of concrete in the forms, it should be compacted to assure close contact of the constituent materials with themselves, as well as with other forms. This consolidation may be accomplished with the use of hand tools, but vibrators are much preferred. This consolidation eliminates air pores and achieved maximum density which gives the concrete higher strength and durability (Azaria, 2005).

*iii. Curing of Concrete*

The last step, and exceedingly important one in the manufacturing of concrete, is curing. As hydration of cement takes place only in the presence of moisture and at favorable temperatures, these conditions must be maintained for a suitable time interval called curing time.

The degree of completeness of hydration which is an important factor in cement paste characteristic depends on the ability of maintain the moisture in concrete which is called curing of a concrete. At the time concrete is mixed, sufficient water is added to give workability. The amount of mixing water actually used is ordinarily in excess. Therefore, if the original water can be retained, there is more than sufficient for curing purpose of concrete gains strength most rapidly at early ages so that the greatest

benefit from curing is secured during this period and each additional day is lesser importance than the preceding one. Specifications usually require that the surface of concrete be protected to prevent loss of moisture for at least 7<sup>th</sup> days where normal cement is used, and some specifications require curing for 14<sup>th</sup> days. Where high – early – strength cements are used, the curing may be reduced by half, while for slow – hardening cements the time should be longer than for normal cements (Azaria, 2005).

Curing temperature affects the hydration of cement and hence the duration of strength gains. Cubes kept at about 10 °C will have their 7<sup>th</sup> day strength reduced by 30 % and their 28 days strength by 15 % (Hailu, 2011).

## **2.2 Sludge Treatment Process**

There are three different process parts in the sludge treatment, sludge digestion, sludge thickening, Sludge drying and sludge dewatering. These three parts are the most important treatments in the sludge treatment process. Transportation is an alternative process, depending on wastewater and incineration plant's location (Vitorio et al., 2007).

### **2.2.1 Sludge Digestion**

The first treatment is sludge digestion. In this process sludge is transported by pipes and pumps to the digestion chamber. The purpose of digestion is to reduce the amount of organic matter and disease causing microorganisms. Anaerobic digestion is still a long process; it lasts minimum twelve days to maximum thirty days in the chamber. In the digestion chamber heat is utilized among bacteria which are in the sludge to digest the sludge anaerobically. The anaerobically digested sludge produces methane gases. Microorganisms which are methanogens utilize carbon sources and generate these gases as the result of the process. The methane gas can be utilized as an energy source in sludge digestion. Stored methane gas runs fuel generators and fuel pumps in the wastewater plant sludge digestion. It is a circulation of the energy in the sludge digestion of a wastewater plant to utilize energy and heat from sludge digestion (Vitorio et al., 2007).

Aerobic digestion is a process occurring in oxygen. Under oxygen bacteria rapidly consumes organic material converting it to carbon dioxide. Aerobic digestion occurs faster than anaerobic digestion but consumes more energy. Aerobic digestion requires blowers and pumps to add oxygen in this process. These days high technology enables to use natural air in this aerobic process and energy can be saved. It's up to a user whether time is more precious than money while choosing digestion process between aerobic or anaerobic (Vitorio et al., 2007).

### **2.2.2 Sludge Thickening**

Sludge thickening is a process to reduce amount of water in sludge, making sludge thicker. There are common types of sludge thickeners, which are called DAFs (dissolved air flotation) or flow type of thickeners. Those types of thickeners are inside huge chambers pushing air to sludge in very high pressure. Pressure is released to sludge and sludge will flow out of the chamber while floating on water. The result is solid and thick sludge leaving watering parts in the bottom. Usually sludge thickening occurs before sludge digestion, but it is after digestion treatment. Sludge thickening is a preliminary process of sewage sludge treatment which is either used for increasing the concentration of biological sludge removed from activated sludge process, or obtaining a denser primary sludge. Sludge thickening process benefits from the effect of gravity as well as flotation (Vitorio et al., 2007).

### **2.2.3 Sludge dewatering**

It is applied after one of the sludge stabilization processes. The aim of dewatering is to reduce water content of sludge which yields lower transportation cost and reduces produced sludge mass. It is also applied to increase the mass loadings to sludge drying process, since it is easier to dry sludge which has lower moisture content. Stabilized sewage sludge which has 1- 4 % dry solids is transformed into a material which is called as 'cake' having 20 - 30 % dry solids by dewatering. Pressure, vacuum or centrifugal forces are used in sludge dewatering process, belt filters and vacuum filters are used as mechanical devices for dewatering process (Atak, 2013).

#### **2.2.4 Sludge drying**

Sludge drying is the last step in sewage sludge treatment. It is used to achieve further volume reduction of sewage sludge, to obtain biologically stable material which allows for easier transportation or handling and to make sewage sludge usable in thermal process which requires fully dried material such as thermal power plants or cement kiln. Solar energy as well as thermal energy from combustion processes are used for sewage sludge drying. Use of solar energy is carried by spreading dewatered sewage sludge to a controlled land where high solar energy is available. Thermal drying of sewage sludge consists of two different mechanisms; direct drying is a high rate operation where high temperatures are reached. Indirect drying is achieved by countercurrent introduction of low temperature air to sewage sludge which is sprayed in a filter medium to obtain a thin film. Regardless, both methods of thermal drying enables sewage sludge having more than 90 % dry solids content (Atak, 2013).

#### **2.3 Disposal and Beneficial Use of Sewage Sludge**

Eventhough sludge goes through a treatment during which its unpleasant characteristics is amended and its volume is reduced significantly; at the end of treatment there is still a high quantity of material left behind. This part of sludge management is challenging since there are not so many options. After treatment of sewage sludge, most typical application is to send to land filling. It is the simplest operation which offers to handle sludge with least amount of cost. On the other hand, huge quantities of sludge could become a limiting factor where land is scarce. Therefore, in order to achieve sustainable sewage sludge management, alternatives to landfilling of sewage sludge have been used. Composting, agricultural usage and thermal treatment of sewage sludge are the most frequently used sewage sludge management alternatives. However, agricultural use of sewage sludge has been gradually restricted since it is regarded as risky due to the presence of pathogens, heavy metals and trace organic compounds in sludge. Also, there are some technical difficulties of agricultural use of sewage sludge since sewage sludge is being produced continuously, while fertilizers are applied in certain periods of a year in

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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agriculture. In addition, public opinion regarding sewage sludge is generally negative, therefore it brings a negative impact on agricultural use of sewage sludge (Atak, 2013).

Use of sewage sludge in thermal processes becomes an attractive solution since it enables both handling of sludge and substitution of primary fuels. Sewage sludge is an organic substance; in fact it has a calorific value close to brown coal in dry basis. Therefore, when it is dried, it is considered as a biomass, an alternative to primary fuels, to be used in thermal processes (Atak, 2013).

### **2.3.1 Use of sludge in agriculture and other land use**

Dewatered, stabilized and sanitized sludge can be used as a fertilizer in agriculture. The sludge is often stored temporary before it can be transported away for use in agricultural areas or other land use. It is important that the storage do not lead to any re-growth of pathogens or pollution of different substances. The use of sludge as a fertilizer is only possible during certain times of the year, due to legislation and practice in agriculture, but the sludge is produced continuously so the design of the temporary storage can be of great importance. Transportation of sludge is almost always needed due to the fact that the wastewater treatment plants are situated in urban areas and the sludge is spread in rural areas. In Sweden for example trucks perform most of the transportation of sludge. The sludge is transported in open containers and the loading is performed by loading trucks or from silo. The transport as well as the handling and storage should be performed in such a way that the risks for personnel handling the sludge are minimized. The usage of sludge as a fertilizer enables the recycling of all the nutrients in the sludge. There are several potential negative consequences of the spreading of sewage sludge at agricultural land, for example the risk of spreading pathogens, organic pollutants and metals. There are also other compounds in the sludge that need to be more investigated as for example hormones and pharmaceutical substances. The risk can be minimized by the choice of suitable crop, methods of spreading and by regulating the time between fertilize and harvest. Sludge can also be used as a fertilizer at existing forests and this would increase growth which would enable a higher withdraw of raw material in the future. Energy forest is another option for the usage of sludge as a fertilizer. It has been

shown in experiments in Sweden that *Salix* accumulate more metals than is provided in the sludge. Only 2 % of the cadmium that is accumulated in the plant comes from the sludge and the rest from the land. The land is then consequently cleaner from cadmium after the growth. The water content of the sludge is reduced to one third if the sludge is dried and pelletized. These pellets can then be used in agriculture or in forest industry. Energy forest can also be irrigated with sludge directly through a pipe system. The cultivation is then a part of the treatment plant ( Nilsson and Hanna, 2005).

#### **2.4 Incinerated sewage sludge ash (ISSA)**

ISSA is an inorganic ash derived from the incineration of the residual stream of fine organic and inorganic solids (sewage sludge). This arises from municipal wastewater treatment works. The incineration process thermally destroys the organic matter. The total quantity of ISSA produced in the UK (through nine incinerators throughout England and one in Northern Ireland), is approximately 100,000 tons per annum, Less than a quarter of the sewage sludge produced is incinerated and there is a potential to increase to 450,000 tons if all the UK sludge were incinerated in the future. Recycling and disposal routes for sewage sludge itself are agriculture(54%), incineration - producing ISSA (22%), landfill (11%), land reclamation (5%) and other (8%). There is anecdotal evidence for a significant geographic variation in the composition of ISSA (Coutand. et al., 2012).

##### **2.4.1 Properties of incinerated sewage sludge ash (SSA)**

###### **2.4.1.1 Proximate Analysis**

Proximate analysis for the combustible components of SS includes the following tests: Moisture (drying at 105 °C for 24 hrs), Volatile combustible matter (ignition at 950 °C in the absence of oxygen), fixed carbon (combustible residue left after Step 2) and ash (weight of residue after combustion in an open crucible). Fixed carbon is the carbon remaining on surface as charcoal. A waste with high fixed carbon requires a longer detention time on the surface of the furnace to achieve complete combustion

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than the waste with a low fixed carbon load. Typical proximate analysis values are showed in below Table 2.2.

Table 2.2: Proximate analysis result of SS (Lumley et al., 2014)

Parameters	Result (%)
Moisture content	80
Ash content	19.67
Volatile matter	71.3
Fixed carbon	9.03

### **2.4.1.2 Physical properties**

Sludge ash is a silty-sandy material. The particle size distribution ranges between 1 and 100  $\mu\text{m}$ , with a mean diameter around 26  $\mu\text{m}$ . A relatively large fraction of the particles (up to 90 percent in some ashes) are less than 0.075 mm (No. 200 sieve) in size. 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  $\text{cm}^2/\text{g}$ , whereas the ASTM C 150 standard recommends a minimum of 2800  $\text{cm}^2/\text{g}$  (Hailu, 2011). Sludge ash has a relatively low organic and moisture content. Permeability and bulk specific gravity properties are not unlike those of natural inorganic silt. Sludge ash is a no plastic material (Yusuf et al., 2010). The specific gravity is typically in the range between 2.3 and 3.2  $\text{g}/\text{cm}^3$  and SSA density has been found to increase with increasing incineration temperature from 2.67  $\text{g}/\text{cm}^3$  at 800  $^\circ\text{C}$  to 2.83  $\text{g}/\text{cm}^3$  at 1000  $^\circ\text{C}$  (Vouk et al., 2017). The LOI data for SSA obtained from the sourced literature yielded an average value of 3.5 %, though occasionally very high values up to 13 % have been reported. Thus, it is possible for SSA, as presently produced, to be generally able to comply with the LOI limit of 5 % set for cement in EN 197 (2011) and fly ash for concrete in EN 450 (2012). Where SSA is earmarked for specific use in concrete, a thorough burn during incineration should be able to control the LOI. The major

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elements present in SSA are Si, Ca, Fe, Al, P, and O and the main crystalline phases present are quartz ( $\text{SiO}_2$ ), calcium phosphate ( $\text{Ca}_3(\text{PO}_4)_2$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ). SSA may also be amorphous when high temperature combustion is used.

### **2.4.2 Use of ISSA in cementations materials**

The cement industry has three main options for using waste materials. These are the beneficial recycling of wastes as alternative raw materials to form clinker, use of wastes as alternative fuels and use of wastes as supplementary materials in blended cements, effectively substituting for Portland cement. Given that ISSA possesses no calorific value, use as an alternative fuel is not appropriate. The major elements present in Portland cement are Ca, Si, Al and Fe. These compare reasonably well to the major elements in ISSA, with the notable exception of P. Thus ISSA could be used to a limited extent as an alternative raw material for cement manufacture.

It is worth noting that the use of dried or even dewatered sewage sludge has received considerable attention in the literature, as this can simultaneously use the calorific value of sewage sludge to reduce fuel requirements and the inorganic content of the sludge to reduce cement raw material requirements (Donatello & Cheeseman, 2013).

Review of different wastes (wood ash, fly ash, bagasse ash, volcanic ash, waste paper ash, sewage sludge ash with eggshell powder, sewage sludge ash) used for supplementary cementation material as a construction and building, and those waste materials were summarize in the below Table 2.2.

Table 2.3: Review of different waste materials used as supplementary construction materials

Authors	Titles( materials used)	Parameters	Products
(Sajad et al.,2013)	Study of Concrete Involving Use of Waste Paper Sludge Ash as Partial Replacement of Cement	Temperature	concrete
HailuB. 2011	Bagasse ash as cement replacing material	temperature	mortar

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(Muhammad et al.,2017)	A comparative study on different burning method of sewage sludge ash in mortar brick with eggshell powder as additive	Temperature	Mortar bricks
(Zigeng et al.,2017)	Quantitative analysis of fly ash in hardened cement paste	Temperature	Cement paste
(Mudasiru I.,2015)	Durability characteristics of Portland cement/volcanic ash concrete exposed to chemically aggressive environment	Durable strength	Concrete
(Esben H. 2016)	Wood ash as a resource in lightweight concrete blocks and lightweight aggregate	Temperature	Block and lightweight aggregate
(Marta K. 2010)	Application of SSA as Partial Replacement of Aggregate in Concrete	Temperature	concrete
In this research	Characterization and use of sewage sludge ash as supplementary cementation material for concrete	Temperature	Concrete

### 2.4.2.1 Use of ISSA in the PC manufacturing process

Cement is manufactured by firing a combination of limestone (80 wt. %) and clay (20 wt. %). Small amounts of quartz sand, bauxite and/or hematite may be added to optimize the Si, Al and Fe contents. In the cement kiln, all organic material is combusted and inorganic compounds, including those from any ISSA used as an alternative raw material, fuse into molten clinker phases at around 1450 °C, with flame temperatures reaching 1800 – 2000 °C. In cement raw meal blended with ISSA it was shown that when the P<sub>2</sub>O<sub>5</sub> content increased above 0.46 wt. %, the belite content of clinkers increased at the expense of alite and this caused longer setting times and lower strength development in cement pastes. Showed that clinkers produced containing 2 wt. % ISSA were satisfactory but that when the ISSA content increased to 8 wt. %, a significant reduction in alite content and increase in free lime

content was observed. This was attributed to the elevated phosphate and possibly sulphate contents of ISSA inhibiting alite formation. Pre-treatment of ISSA to remove phosphates prior to use as a raw material in the production of cement clinker was suggested. From a practical point of view, significant quantities of ISSA could in theory be diverted from landfill by use in cement kilns without reaching the problematic phosphorus levels reported by various authors. Global cement production was estimated at 3.6 billion tons in 2011 whereas a reasonable estimate of global ISSA production is approximately 1.7 million tons, over 2000 times less. However, where sewage sludge is not incinerated, the direct use of dried sewage sludge in the cement industry is generally preferred because this avoids the need for investment in a dedicated mono-incineration plant for sewage sludge, although investment would still be required in a thermal drying facility. The use of dried sewage sludge instead of ISSA allows the calorific value of the organic matter to be exploited, reducing kiln fuel requirements (Donatello & Cheeseman, 2013).

#### **2.4.2.2 Use of ISSA as an additive to PC**

Wastes can be recycled in cement based materials as either active pozzolanic materials, partially replacing cement, or as inert filler, replacing sand and/or aggregates. A considerable number of papers have reported the use of ISSA as a partial replacement for Portland cement and the effect on the workability and strength development of pastes, mortars and/or concretes and a selection of the data available in the literature is presented in increasing ISSA contents causes a decrease in compressive strength and milling of ISSA generally improves strengths at a given percentage of ISSA addition. When attempting to compare results from different authors using the same cement replacement rate, it is clear that large differences in relative strengths exist. For example, when replacing 20 % of Portland cement with ISSA. While obvious differences will arise due to factors such as mortar specimen dimensions and water/binder ratio used, an important factor not generally considered has been the processes used to produce the ISSA. For example, the pioneering work was carried out using a dewatered digested sludge fired in a laboratory oven at 550 °C for an unspecified period. Used ash produced by a multiple hearth furnace, used dewatered primary sludge fired at 700 °C for 3 hrs and the ISSA was sourced from

industrial scale fluidized bed incinerators where ash residence times in the combustion zone were of the order of seconds at 800 - 900 °C. Each of the mentioned methods of ash preparation will impart a specific thermal history to the ash and may affect the physical and chemical properties of the ISSA formed. Regardless of the combustion history of ISSA, the irregular particle morphology causes a decrease in workability when replacing cement, even at low percentage additions. To some extent, poor workability can be overcome by milling (Donatello & Cheeseman, 2013).

#### **2.4.2.3 Pozzolanic activity of ISSA**

Despite the generally negative effects on compressive strength of ISSA in blended cements, many authors have attributed a certain degree of “pozzolanic activity” to ISSA. The definition of a “pozzolanic” material as given in (ASTM C618, 2008) is: A siliceous and aluminous material which, in itself, possesses little or no cementation value but which will, in finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementation properties. The requirement of significant  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  content suggest that ISSA may have potential as a pozzolan. Another important consideration is that any clay present in sludge fed to the incinerator may be thermally activated and this can contribute pozzolanic properties to ISSA. Such a phenomenon is well known in paper sludge ash in which kaolin may be converted to pozzolanic metakaolinite many methods are available to determine the pozzolanic activity of a material. These can be broadly classified as either direct or indirect methods. The pozzolanic reaction involves  $\text{Ca}(\text{OH})_2$  reacting with silicate or aluminosilicate phases to form amorphous C-S-H or C-A-S-H type gel products. Thus direct methods measure the change in  $\text{Ca}(\text{OH})_2$  concentration as the pozzolanic reaction proceeds. Examples of direct methods are the Frattini test (EN196-5), the saturated lime test and analysis of pastes with hydration reactions being inhibited after specific times. Indirect methods measure a physical property of pastes that is linked to the pozzolanic reaction. Examples of indirect methods are the strength activity index (ASTM C311, 2007) and electrical conductivity methods. The majority of work involving the assessment of ISSA pozzolanic activity has used indirect methods, monitoring the effect of replacing cement with ISSA on compressive strength of pastes and mortars.

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Effects were generally negative but this could be strongly influenced by the increased water demand caused by the irregular particle morphology of ISSA. In a comprehensive assessment of ISSA pozzolanic activity, it was found that ISSA gave highly positive results in the saturated lime test, negative results in the strength activity index test and positive or negative results depending on the percentage cement replacement in the Frattini test. The authors concluded that ISSA was not pozzolanic but instead possessed a limited affinity for  $\text{Ca}^{2+}_{(aq)}$  ions via an ion exchange mechanism. They also concluded that the saturated lime test method was biased in favor of positive results with ISSA due to the lower total quantity of  $\text{Ca}^{2+}_{(aq)}$  present in this system compared to the Frattini or strength activity index tests. Increasing the fineness of ISSA has also been shown to increase setting times, which was attributed to the improved ability of finer ISSA to adsorb  $\text{Ca}^{2+}$  ions from the liquid phase during cement hydration, inhibiting the massive precipitation of C–S–H gel responsible for setting of Portland cement. Given the general lack of pozzolanic activity of ISSA, another option for recycling in cement-based materials is as a fine aggregate. Very little work has been presented on this aspect. In one set of experiments, concretes containing up to 30 % replacement of sand by ISSA showed a 22 % reduction in 28 day compressive strength (Donatello & Cheeseman, 2013).

### **3 MATERIALS AND METHODS**

#### **3.1 Materials**

The materials used for this study works are sewage sludge, Ordinary Portland Mughher cement, fine aggregate, coarse aggregate, and water.

##### **3.1.1 Cement**

Ordinary Portland cement is designed for general use of construction works throughout in the world. From locally produced Mughher Ordinary Portland Cement has been used in this study, OPC available at market in Addis Ababa and it was purchased from their sub -branch of Ordinary Portland Mughher Cement Industry in Addis Ababa city, cement conformed to requirements of Ethiopia Standard, ES C.D5 201 and ES C.D5 202.

##### **3.1.2 Fine aggregate**

Locally available sand was used as fine aggregate and physical properties of fine aggregate: specific gravity & absorption capacity (ASTM C 128), fineness modulus (ASTM C 136), & moisture content (ASTM C566) are determined at Appendix A.

##### **3.1.3 Coarse aggregate**

Basaltic crushed rock was used as coarse aggregate. The maximum aggregate size of coarse aggregate is 37.5 mm and physical properties of coarse aggregate: specific gravity & absorption capacity (ASTM C 127), moisture content (ASTM C 566) & sieve analysis (ASTM C136). Thus are determined at Appendix A.

#### **3.2 Methods**

In laboratory analysis were carried out to characterize the sample and the concrete. General experimental work flow is given figure 3.1

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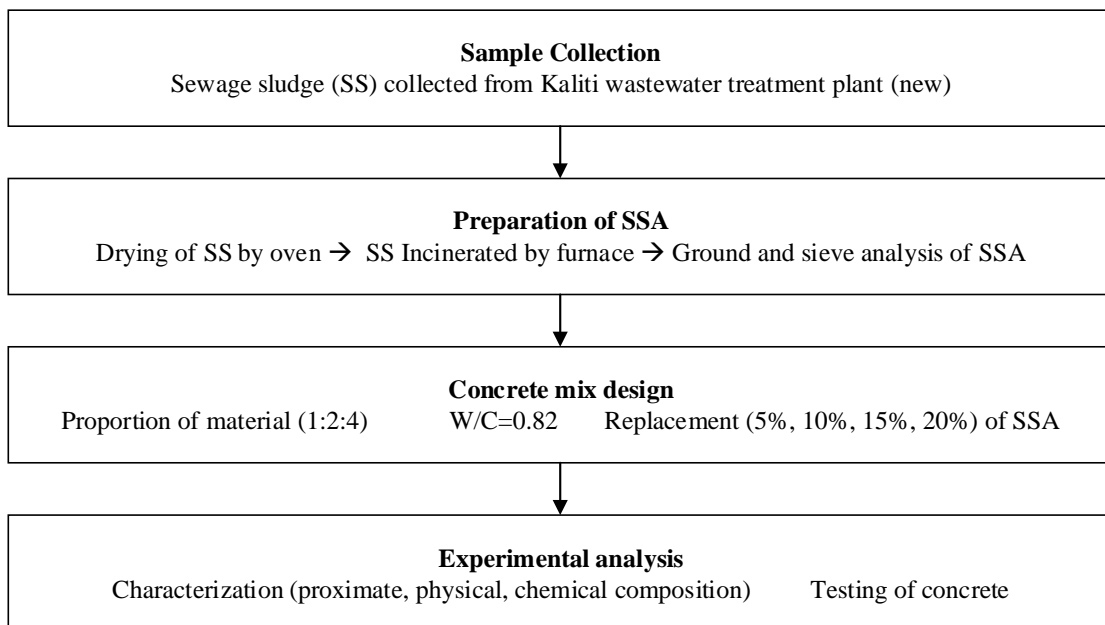


Figure 3.1: Experimental work flow

### 3.2.1 Preparation of SSA

The wet sewage sludge was collected from Kaliti wastewater treatment plant. The preparation of SSA was performed in School of Chemical and Bio-Engineering laboratory. The sewage sludge was dried by an oven at 105 °C for 24 hours to reduce the moisture content of the sample before incineration process. Next, the oven dried sewage sludge was incinerated by using a furnace at the temperature of 600 °C and 800 °C for 3 & 2.5 hours respectively to produce sewage sludge ash. These two temperatures was selected to remove the organic materials and increases the pozzolanic activity (reaction) in the SSA with water to get the expected compressive strength.

Heat treatment process was done on the sludge for up to 650°C, the reason for using this temperature relates to the fact that it was enough to remove the organic matter from the dry sludge ( Jamshidi et al, 2011). Cements having sewage sludge calcined at the lowest temperatures will have slow and low strength development properties because of the low CaO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> contents (Naamane et al., 2016). Incineration of sewage sludge at temperature higher than 800 °C leads to decrease in the pozzolanic activity of sewage sludge ash due to crystallization of amorphous silica.

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Hence, incineration of sewage sludge ash must be optimized at 800 °C to preserve the pozzolanic activity of the resultant ash (Tantawy et al., 2012). Sewage sludge ash was then taken and ground to make a powdered form by a grinder and was sieved by using sieve shaker with 75 µm sieves, which means the particle size of SSA was below 75 µm but it is not exactly 75 µm. The SSA that passed through the 75 µm sieve was used to prepare the concrete mix in this study.

### 3.2.2 Concrete mix design

A concrete mix design is usually expressed as based on mass of ingredients per unit volume. The basic constituents of a concrete are cement, coarse aggregate, fine aggregate, water, and in addition of SSA that incinerated at 600 °C and 800 °C.

The proportional of material for concrete were 1:2:4 of cement, fine aggregate and coarse aggregate, respectively with water to cement ratio of 0.82. Also, four different types of replacement of Portland cement- sewage sludge ash concrete are produced; 5%, 10%, 15% and 20% with 0% as a control to determine the most appropriate replacement. Weight measurement was used for the preparation of constituent to determine the amount of materials (aggregates, cements and SSA) to be used for making the concrete cubes and were mixed dry for one minute. After the addition of water, all materials were mixed for another two minute. Then, the mixed fresh concrete was placed on a firm, and level of the surface of the prepared mould (10 cm x10 cm x 10 cm). The cubes were vibrated for 45 minutes and after vibration the top surface is finished by using a trowel. The concrete removed from the mould after 24 hours and were cured in the water baths. The batching and mixing of fresh concrete was conducted using 1:2:4 nominal mix ratios in accordance to BS 1881-125(1986)

Table 3.1: Mix proportion for cubes

Replacements SSA (%)	of cubes	No of Cement, g	SSA, g	Fine aggregate, g	Coarse aggregate, g	Water, g
0%	6	1920	0	3840	7680	1574.4
600°C5%	6	1824	96	3840	7680	1574.4

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600°C10%	6	1728	192	3840	7680	1574.4
600°C15%	6	1632	288	3840	7680	1574.4
600°C20%	6	1536	384	3840	7680	1574.4
800°C5%	6	1824	96	3840	7680	1574.4
800°C10%	6	1728	192	3840	7680	1574.4
800°C15%	6	1632	288	3840	7680	1574.4
800°C20%	6	1536	384	3840	7680	1574.4



Figure 3.2: Concrete casted from 10cm x 10cm x 10cm cubes

### 3.2.3 Experimental Analysis

#### 3.2.3.1 Characterization of SSA

##### a. Proximate analysis

The proximate analysis of sewage sludge was determined when the sludge sample was heated under specified condition. All these proximate analysis were conducted in School of Chemical and Bio-Engineering laboratory. Proximate analysis that includes moisture content (ASTM D, 3173) by using the Oven, ash content (ASTM D3174) by using the Furnace, volatile matter (ASTMD 3175) by using the Furnace, and fixed carbon (ASTM D3172). Proximate analysis was computed by using the method of

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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ASTM (ASTM, 1989). Those proximate analyses were detailed to discuss in Appendix B.

### **b. Physical properties**

Physical properties of SSA included moisture contents (ASTM D, 3173) by using the Oven, specific gravity (ASTM C311- 05, 2008) by using Le Chatelier's flask, loss on ignition (LOI) ASTM C311 (ASTM C311- 13 2013) by using Furnace, and specific surface area (ASTM C 204) by using Blain air permeability. Those physical properties of SSA were conducted in School of Chemical and Bio-Engineering laboratory. All These physical properties were detailed to discuss and set the calculation in Appendix C.

### **c. Chemical composition**

The chemical composition tests of the SS and SSA were conducted in Geological survey of Ethiopia at geochemical laboratory directorate by using Atomic Absorption Spectrometer (AAS). Chemical composition of SSA consisted of oxides and trace element (heavy metal content). Oxides are Silicon dioxide( $\text{SiO}_2$ ), Aluminum oxide ( $\text{Al}_2\text{O}_3$ ), Iron oxide( $\text{Fe}_2\text{O}_3$ ), calcium oxide( $\text{CaO}$ ),Magnesium oxide( $\text{MgO}$ ),Sodium oxide( $\text{Na}_2\text{O}$ ), Potassium oxide( $\text{K}_2\text{O}$ ),Manganese oxide( $\text{MnO}$ ),Phosphorus oxide( $\text{P}_2\text{O}_5$ ),Sulfur oxide( $\text{SO}_3$ ),Titanium oxide( $\text{TiO}_2$ ), and Chlorine( $\text{Cl}$ -).

0.1 g of a sample was taken in the platinum crucible and mixed with 0.5 g of  $\text{LiBO}_2$ . Then, the mixture has placed in the muffle furnace at temperature  $950\text{ }^\circ\text{C}$  for 45 minutes to melt the sample. After the 45 minutes reached it was removed from a furnace and cool, 5 ml of  $\text{HNO}_3$  into the fused sample and warmed by hot plate and stirred until the fused sample disintegrated and removed from platinum crucible. After that, the sample transferred to the 500 ml of volumetric flasks until the flask filled with distilled water up to the mark and then the samples were analyzed to determine the oxides by AAS. AAS has read automatically to display the results of oxides on the computer screen due to there is no free state in nature.

Trace elements are Cobalt (Co), Cupper (Cu), Chrome (Cr), Nickel (Ni), Lead (Pb), and Zinc (Zn). 5 g of a sample was taken into a 250 ml beaker and 10 ml of  $\text{HNO}_3$

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was added, and mixed together. The mixture was boiled for 45 minutes to remove organic materials. After 45 minutes reached, it was cool, 5 ml of  $\text{HClO}_4$  was added and the mixture was boiled until white fume was observed to remove remaining impurities. Then, 50 ml of distilled water was added. After digestion, the solution was filtered by 0.45  $\mu\text{m}$  filter paper and trace elements in the solution were determined by AAS.



Figure 3.3: Atomic absorption spectrometry (AAS) at Geological survey of Ethiopia

### 3.2.3.2 Testing of concrete

#### a. Workability test of fresh concrete

Slump test was performed in School of Civil and Environmental Engineering laboratory. Slump test method is used to test the workability of the fresh Portland cement sewage sludge ash concrete. Slump test is conducted following ASTM C143 procedures. All these procedures were set each and every step on Appendix D. The figure 3.4 set in below.



Figure 3.4: Slump test of fresh concrete mix

**b. Compressive strength test of hardened concrete**

Compressive strength test was done in School of Civil and Environmental Engineering laboratory. Compressive strength was conducted following ASTM C 39 procedures. Those procedures of compressive strength test were detailed discusses on Appendix D. Set the figure 3.5 in the below.



(a)



(b)

Figure 3.5: (a) Automatic compressive strength measuring machine and (b) some of crushed sample

**c. Water absorption test**

The water absorption test was conducted in School of Chemical and Bio-Engineering laboratory. The concrete samples were removed from the curing tank and allowed to merely surface dry, and so placed in the electronic oven to dry at 105 °C for 72 hours. The samples were taken out from the oven and allowed to cool at room temperature, then weighed to determine the initial weights. The final weights were determined after immersing of the concrete samples in the curing medium for 30 minutes, then removed specimens to surface dry by a cloth and re-weighed again. The obtained values were recorded and the results were calculated to measure the rate of water absorption from the concrete specimens in the accordance with British Standard (BS1881-122 (1983)). Lastly, the calculation was done by the following formula.

$$\text{Water absorption (\%)} = \frac{A - B}{B} * 100$$

Where: A = after immersion mass of a concrete, g

B = before immersion mass of a concrete, g



(a)



(b)

Figure 3.6: Oven dried concrete cubes (a) and (b) weighted of the concrete sample

**d. Leaching test of concrete**

Leaching tests are used for determining the concentration of elements (mainly heavy metals) that are released within a certain time from the solid phase into the liquid phase, which are then considered to be potentially dangerous (Vouk et al., 2017). The

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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leaching test for the environmental study (liquid–solid extractions) was carried out at 28 days of age in order to evaluate the quantity of pollutant released by cement based materials containing SSA (Coutand et al., 2007). Leaching test refer to test method for leaching of trace elements that test was performed for the purpose of determining the amount of trace elements leached from a concrete specimen that contained the SSA into the water that has been immersed in the water bath. Leaching test was conducted in Horticoop Ethiopia (Horticulture) PLC by inductively coupled plasma optical emission spectrometry (ICP - OES).

The water sample was delivered in closed plastic container and analyzed within 48 hours. When not used immediately, the water sample was stored in the refrigerator between 2 °C and 5 °C until to analyses the samples (identify each (12 ml) sample clearly upon arrival). Shake the sample bottle well and a sample was filtered by 0.45 µm filter paper. After the filtration, the sample was placed into ICP test tube and then analyzed for heavy metals by ICP.

## **4 SRESULTS AND DISCUSSION**

In this section, the results of the characterization of SSA that are including the proximate analysis of SS, physical properties, and chemical composition and also the concrete test, such as test on fresh and hardened concrete. Those are discussed in the following ways.

### **4.1 Characterization of SSA**

#### **4.1.1 Proximate analysis**

The proximate analysis of sewage sludge determined only the moisture content, ash content, volatile matter and fixed carbon content. These proximate analyses of the SS were done by according to the method of ASTM. Proximate analyses of sewage sludge (SS) results were presented with other results available in the literature as shown below (Table 4.1).

Table 4.1: Proximate analysis of SS

Parameters	Results (%)	Literature data
Moisture content	46 %	80%
Ash content	26 %	19.67%
Volatile matter	24%	71.3%
Fixed carbon	4%	9.03%

Source: (Lumley et al., 2014)

In this study, the results of a sample were obtained percentages of 46 % of moisture content, 26 % of ash content, 24 % of volatile matter, and 4 % of fixed carbon. The moisture content was 46 %, which indicated that the SS have 46 % content of water and 54 % amounts of SS.

Higher moisture content requires a longer drying time and much more heat energy, causing a lower temperature in the furnace; and vice versa. If the moisture content is too high, the furnace temperature will be too low for combustion (Shiferaw, 2014)

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As a result shown, the composition of sewage sludge was contained 50 % of ash content and volatile matter from the proximate analyses. The results were obtained in a proximate analysis (moisture content, ash content, volatile matter and fixed carbon) were as compared with the literature data. The ash content of SS higher than the literature information about on ash content which means 26 % of ash content greater than 19.67 %. While the percent of moisture content, volatile matter, and fixed carbon were lower than the literature data. The variation in results may be depends on the composition of the wastewater and the treatment process used.

### 4.1.2 Physical properties of SSA

The results of physical properties of 600 °C SSA and 800 °C SSA are given in the Table 4.2 with the ASTM C618 and other results available in the literature.

Table 4.2: physical properties of SSA

Physical properties	Results			ASTMC618 Requirements	Literature data
	SS	600°CSSA	800°CSSA		
Moisture C. (%)	2.51	0.46	0.22	3%	
LOI (%)	55.34	2.49	1.65	6%	
Particle size	-	< 75µm	< 75µm	-	1-100µm(Coutand et al, 2007).
Specific gravity(g/cm <sup>3</sup> )	-	2.36	2.54	3.15 g/ cm <sup>3</sup>	2.3–3.2g/cm <sup>3</sup> (Vouk et al., 2017)
Specific surface area, (cm <sup>2</sup> /g)	-	3419.4	3475.1	Mini.2800 cm <sup>2</sup> /g ASTMC150	

The moisture content was 2.51 % of the SS, 0.46 % of 600 °C SSA, and 0.22 % of 800 °C SSA. The three samples of moisture content results were met the requirement of 3% maximum as stated in ASTM C618. The low moisture contents of a sample are preferred and neglected which is not accounted for it when determining the water to cement ratio.

## Characterization and use of sewage sludge ash as supplementary cementitious material for concrete

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The loss on ignition expresses the amount of organic material in the SSA (Rem, 2014). In this study, LOI of SS was 55.34 %, 600 °C SSA and 800 °C SSA were 2.49 % and 1.65 % respectively. Therefore, the results of LOI in the two samples (2.49 % 600 °C SSA & 1.65 % 800 °C SSA) were met the value (6 %) of requirement stated by ASTM C 618, which means the percentage value of the sample under the stated of the standard. However, sewage sludge (SS) of LOI was 55.34 %, which was in the result of SS is more than 6 % that stated for pozzolanic materials in a maximum LOI and this indicated it has high carbon content.

The largest drawback to having a pozzolan with high LOI is that it is typically indicative of high carbon content. High carbon contents tend to interfere with the adsorption of air-entraining admixtures and water-reducing admixtures; the higher specific surface area of carbon preferentially adsorbs the air-entraining agent rendering it less effective ( Paris, 2016).

Sludge ash has a relatively low organic and moisture content (Yusuf et al., 2010). Therefore, the SSA has shown a low moisture and organic content according to obtain the results from this study.

The particle sizes of SSA were below 75  $\mu\text{m}$  that is incinerated at the two temperatures such as 600 °C and 800 °C. The results of specific gravity were 2.36  $\text{g}/\text{cm}^3$  600 °C SSA and 2.54  $\text{g}/\text{cm}^3$  800 °C SSA and the specific gravity of SSA which have indicated to somewhat increase with increasing incinerated temperature at 600 °C and 800 °C. So, the specific gravity of SSA was between the ranges of literature data set in the Table 4.2.

The 600 °C SSA & 800 °C SSA has a low specific gravity that was 2.36 & 2.54  $\text{g}/\text{cm}^3$  as compared to 3.15  $\text{g}/\text{cm}^3$  of OPC specific gravity. Specific surface area of 600 °C SSA and 800 °C SSA were 3419.4 & 3475.1  $\text{cm}^2/\text{g}$  that is fulfills 2800  $\text{cm}^2/\text{g}$  of greater than or minimum requirement that stated by ASTM C150.

The material fineness should give an indication to reactivity as higher surface area and fineness is related to higher reactivity (Rem, 2014).

## Characterization and use of sewage sludge ash as supplementary cementitious material for concrete

### 4.1.3 Chemical composition of SSA

The chemical composition of sewage sludge (SS) and sewage sludge ash (SSA) incinerated at 600 °C and 800 °C was determined by Atomic Absorption spectrometer (AAS) and the results of oxides were presented in Table 4.3 with the ASTM C618 and European standards DS/ EN 450 -1 : 2012.

Table 4.3: Chemical Composition in SS and SSA

Oxides	Percentage composition (%)			Result	ASTM C618	European standards
	SS	600°CSSA	800°CSSA			
SiO <sub>2</sub>	22.82	55.02	55.62	<u>600°CSSA:</u>	(SiO <sub>2</sub> ) +(	
Al <sub>2</sub> O <sub>3</sub>	7.04	15.88	16.81	(SiO <sub>2</sub> )+(Al <sub>2</sub> O <sub>3</sub> )	Al <sub>2</sub> O <sub>3</sub> )	
Fe <sub>2</sub> O <sub>3</sub>	4.08	8.88	8.52	+(Fe <sub>2</sub> O <sub>3</sub> )	+(Fe <sub>2</sub> O <sub>3</sub> )	
CaO	3.52	7.60	8.78	= 79.72 %	= ≥ 70 %	≤ 10 %s
MgO	0.82	1.96	2.44	<u>800°CSSA:</u>		≤ 4 %
Na <sub>2</sub> O	0.50	1.44	2.06	(SiO <sub>2</sub> )+(Al <sub>2</sub> O <sub>3</sub> )		Total
K <sub>2</sub> O	0.52	1.70	<0.01	+(Fe <sub>2</sub> O <sub>3</sub> ) =		content of
				80.95%		alkalis≤5%
MnO	0.14	0.32	0.28			
P <sub>2</sub> O <sub>5</sub>	1.27	2.44	2.50			≤ 5 %
TiO <sub>2</sub>	0.24	0.58	0.69			-
SO <sub>3</sub>	0.56	<0.01	1.78		Max. 5%	≤ 3.00 %
Cl <sup>-</sup>	0.78	<0.10	<0.10			≤ 0.10 %

Source: (Rem, 2014)

AAS test has shown that the sewage sludge ash contained primarily these oxides such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO in this study. The other remaining oxides (MgO, Na<sub>2</sub>O, K<sub>2</sub>O, MnO, P<sub>2</sub>O<sub>5</sub> TiO<sub>2</sub>, SO<sub>3</sub> and Cl<sup>-</sup>) are also presented in the sample to a lesser degree.

The ASTM C618 states that for materials to be a pozzolana the summation of composition of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> are must be greater than or minimum of 70 %.

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The sum of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$  contents in sewage sludge, 600 °C SSA and 800 °C SSA were 33.94 %, 79.72 %, and 80.95 % respectively.

The summation results of these oxides ( $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ) in SSA have incinerated those temperatures, which were used in this research work achieved 79.72 % and 80.95 % that is fulfills ASTM C618 requirement, which is the percentage of SSA greater than 70 %. So, the SSA possible to use the replacement of cement to produce a concrete by according to this specification, the SSA classified to be a class F pozzolana.

A pozzolana is defined in ASTM C618 as 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 cementing compounds.

There was high content of silicon dioxide ( $\text{SiO}_2$ ) in the SSA sample after the incinerated at a temperature of 600 °C and 800 °C. The raw sewage sludge consists of 22.82 % silicon dioxide ( $\text{SiO}_2$ ) and the content of silicon dioxide ( $\text{SiO}_2$ ) in sewage sludge ash increased to 55.02 % and 55.62 %, which was about 2.4 times the silicon dioxide content in the SSA greater than as compared with silicon dioxide in raw sewage sludge based on the obtained results in this study.

The process of incineration is able to produce more silicon which is the main component that is responsible for pozzolanic activity in cementations material (Siew et al. 2016).

Silica is one of the most important chemical compounds that control the durable and strength of the concrete. Thus the silica is required in the pozzolan reaction to produce calcium – silica – hydroxide (C – S – H) so that higher amount of the silica oxide tends to create higher durable and strength of concrete (Yeong et al., 2016).

The percentage of calcium oxide (CaO) in SS, 600 °C SSA and 800 °C SSA were 3.52 %, 7.60 % and 8.78 % respectively. The result of CaO in SSA have incinerated at temperature 600 °C and 800 °C, which was used in this study work achieved 7.60 % and 8.78 % that fulfill European standards requirement. Those, the result of calcium oxide less than 10 % limit that set by European standard.

## Characterization and use of sewage sludge ash as supplementary cementitious material for concrete

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The function of the calcium oxide is one of the compounds that responsible in formation of silicate and aluminates of calcium. However, excess calcium oxide will make the concrete unsound and cause the cement to expand and disintegrate (Yeong et al., 2016).

Cements having sewage sludge calcined at the lowest temperatures will have slow and low strength development properties because of the low CaO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> contents. However, for an addition rate of 10 %, the amounts of these three oxides increase with the increase of the temperature of calcinations from 300 °C to 800 °C (Naamane et al., 2016).

The percentage results of P<sub>2</sub>O<sub>5</sub>, SO<sub>3</sub>, and Cl<sup>-</sup> in the SSA were lower than the requirement expressed by the European standard, and that fulfilled European standard requirements. So, which did not performed acid attack test for a concrete based on the obtained results from this study.

Heavy metal analyses were carried out that is Cu, Zn, Ni, Co, Cr, & Pb in SS & SSA. These heavy metals were determined by AAS. The concentration results of trace (heavy) metals in SS, 600 °C SSA and 800 °C SSA were presented in Table 4.4 with USA – EPA standards for heavy metal concentration.

Table 4.4: Heavy Metal in SS and SSA.

Heavy metals	Results (mg/kg)			USA - EPA limits ( mg / kg )
	SS	600°CSSA	800C°SSA	
Co	21.20	48.80	46.00	-
Cr	40	39	39	3000
Cu	19.80	182.40	264.40	4300
Ni	19.00	100.20	92.00	420
Pb	<0.01	20.20	19.80	840
Zn	151.20	1,640.00	2,020.00	7500

Table 4.4 compared the result of heavy metal contents of SSA with USA – EPA standards to know heavy metal concentration. The Zinc, Copper, and Nickel was

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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shown a high amounts in 600 °C SSA and 800 °C SSA due to those metals could be volatilized at a higher temperature because they have lower thermal stability( which are easily volatile), but other heavy metal, such as lead, cobalt, and chromium was shown small amounts in 600 °C SSA and 800 °C SSA from the heavy metal analysis results because of these metals have thermally stable( which are low volatile) with increases the temperature according to obtained results in the SSA. The obtained results in the SSA that indicated all these heavy metals (Cu, Zn, Ni, Co, Cr, & Pb) were below the allowable limit a given in USA – EPA standards.

### **4.2 Concrete test**

#### **4.2.1 Workability of fresh concrete (slump test)**

The slump test results of 9 mixtures were presented in Table 4.5. The trial of slump test was designed to be in the range between 25 mm and 100 mm by according to ACI 211-1-91. The table 4.5 indicated the workability decreased with increase sewage sludge ash (SSA) content in the concrete mixture that SSA was burnt at temperature 600 °C and 800 °C due to the high water demand of SSA. So that, sewage sludge ash particles have absorbed more water due to the irregular surface of the SSA particles as compared to cement and decreasing the workability of concrete mix. It was observed from the test result that there was a 20 % reduction in slump test as the SSA content to vary from 0 % to 20 %.

Table 4.5: Slump test results at 600 °C & 800 °C SSA-Cement mixture (mm).

S.No	Percentage replacement of SSA with OPC	Slump ( mm )
1	0% SSA	60
2	600°C SSA 5%	56
3	600°C SSA 10%	52
4	600°C SSA 15%	47
5	600°C SSA 20%	40
6	800°C SSA 5%	58

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7	800°C SSA 10%	51
8	800°C SSA 15%	44
9	800°CSSA 20%	39

Confirmed that workability decreased with increased SSA addition (an average decrease of workability was about 20% when 20% of cement was replaced by SSA) (D. Vouk et al., 2017).

The slump test values of 600 °C SSA were almost the same as the 800 °C SSA by comparing with the 600 °C and 800 °C SSA due to the SSA have the same particle sizes.

### 4.2.2 Compressive strength result of hardened concrete

Compressive strength was determined the following by ASTM C 39 procedures. The test was done on 10 x 10 x 10 cm<sup>3</sup> cubic specimens. The compressive strength test results for each SSA-cement concrete were obtained from an average of two cubic specimens determined at the curing age of 3<sup>rd</sup>, 7<sup>th</sup>, and 28<sup>th</sup> days. The compressive strength result from 25 % and 30 % of SSA was 0.21 MPa & 0.17 MPa, and this result indicated very low. Therefore, 25 % and 30 % of SSA were dropped. Both compressive strength results of the SSA concrete were presented in Table 4.6.

Table 4.6: Average compressive strength of cubes

Specimens	Compressive strength, MPa		
	3 days	7 days	28 days
0% SSA	4.5	8.08	14.86
600 °C SSA5%	3	6.49	13.45
600°C SA10%	2.56	6.23	11.43
600°C SSA15%	1.56	5.27	9.95
600°C SSA20%	0.61	4.72	8.775

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800°C SSA5%	2.95	7.07	13.88
800°C SSA10%	3	6.35	14.01
800°C SSA15%	1.79	5.69	10.04
800°C SSA20%	0.46	4.80	9.03

The compressive strength result of control concrete (0 % SSA) was 4.5 MPa at the age of 3 days, which is the highest among all the others SSA concrete. The concrete specimens 600 °C SSA(5%, 10%, 15%, 20%) and 800 °C SSA(5 %, 10%, 15%, 20%) were as recorded as 3MPa, 2.56 MPa, 1.56 MPa, 0.61 MPa and 2.95 MPa, 3 MPa, 1.79 MPa, 0.46 MPa, respectively. Those results indicated that the early strength of the SSA concretes were weak as compared to the control concrete except 600°C SSA5% and 800°C SSA 10 %.

The compressive strength of the 600 °C SSA ( 5%, 10%,15% ,20%) & 800 °C SSA (5%, 10%, 15%, 20%) were as recorded 6.49 MPa, 6.23 MPa, 5.27 MPa, 4.72 MPa and 7.07 MPa, 6.35 MPa, 5.69 MPa, 4.80 MPa respectively at the age of 7 days.

On the other hand, the control concrete was 8.08 MPa. The compressive strength of reference concrete was 8.08 MPa which is greater than the obtained values all of SSA- cement concrete in the age of 7 days.

Somewhat the result of 800 °C SSA 10 % greater than 800 °C SSA 5 % in the compressive strength at 28 days, which may be this difference comes from the vibration effect of fresh concrete that placed in the metallic mold cube.

The results of concrete work have shown that 600 °C SSA 5 % & 800 °C SSA 10 % replacement of OPC by sewage sludge ash achieved a higher compressive strength than the others percentage of set in this research at the age of 28<sup>th</sup> days as compared with control concrete (0 %SSA) and the compressive strength results of 0 % SSA, 600 °C SSA 5 % & 800 °C SSA 10 % at 28<sup>th</sup> days were 14.86 MPa, 13.45 MPa, & 14.01 MPa respectively.

As it had been described about 89.67-100 % of compressive strength of concrete would be attained after 28<sup>th</sup> days. As the result saw the concrete test taken on 28<sup>th</sup> day

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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of 600 °C SSA 5 % & 800 °C SSA 10 % replacement were 13.45 MPa & 14.01 MPa of compressive strength which is less than by 9.4 % & 5.66 % as compared with the control but it was expected to more than control.

The formation of calcium silicate hydrate, C-S-H at the early stage becomes retarded as the replacement of SSA increases where C-S-H is responsible for the early strength development in cement based materials (Siew et al. 2016).

### **4.2.3 Water absorption result of hardened concrete**

Water absorption test was carried out for 9 mixtures and percentage of water absorption of the concretes were measured by using British standards (BS 1881-122(198)). The water absorption results of SSA concrete were presented in Table 4.7.

Table 4.7: Water absorption test results at 28 day

S. No.	Sewage sludge ash, %	Dry wt. of cubes, g	Wet wt. of cubes, g	Water absorbed, g	Percentage of water absorption, %
1	0%SSA	8334	8465	131	1.57%
2	600C SSA5%	8354	8496	142	1.69%
3	600CSSA10%	8355	8500	145	1.74%
4	600CSSA15%	8351	8507	156	1.86%
5	600CSSA20%	8188	8352	164	2%
6	800C SSA5%	8313	8456	143	1.72%
7	800CSSA10%	8214	8365	151	1.83%
8	800CSSA15%	8306	8466	160	1.92%
9	800C SSA20	8218	8390	172	2.09%

The percentage of water absorption increased with increase the replacement of sewage sludge ash (SSA) in the concrete that incinerated at temperature 600 °C & 800

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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°C, and all the blended (% SSA) concretes have shown a higher amount of water absorbed as compared with the control concrete (0 % SSA).

Water absorption of concrete increases together with the increase in SSA content (Kosior, 2011).

The lowest value of water absorption was observed for concrete mix with 5 % of (600 °C & 800 °C) SSA that is 1.69 & 1.72 % and highest value of water absorption in 20 % of SSA that is 2 %.

### **4.2.4 Leaching test of hardened concrete**

The leaching test was carried out of heavy metals that are Cu, Fe, Zn, Ni, Co, Mn, Cr, Cd, Hg, Pb, As and B in SSA- cement concrete specimens cured after 28<sup>th</sup> day. These heavy metals were determined by inductively coupled plasma optical emission spectrometry (ICP - OES). The leaching test results were presented in Table 4.8 with USEPA concentration limit. The results these elements Pb, As, and Ni have shown relatively maximum concentration, whereas the other metals namely Cu, Fe, Zn, Co, Mn, Cr, Cd, & Hg have shown the minimum leachability from concrete specimens. However, the obtained results of leaching test in the concrete specimens that shows all these heavy metals (Cu, Zn, Ni, Mn, Cr, Cd, Hg, Pb, and As) were below the allowable limit a given in US EPA standards. So that, the sewage sludge ash possible to use as supplementary cementation material for concrete.

When the concrete specimen was contacted with moisture (water) to consider that was released more metal ions from the concrete. That is way, the table 4.8 differs from the table 4.4.

Table 4.8: Leaching test of heavy metal concentration in SSA- cement concrete

Heavy metal	Results , (mg/l)	USEPA concentration limit, (mg/l)
Copper(Cu)	0.05	100
Iron (Fe)	0.060	-
Zinc (Zn)	0.009	500

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Nickel (Ni)	0.107	1.3
Cobalt (Co)	0.057	-
Manganese (Mn)	0.018	260
Chromium (Cr)	0.078	5.0
Cadmium (Cd)	0.013	1.0
Mercury ( Hg)	0.094	0.2
Lead (Pb)	0.285	5.0
Arsenic (As)	0.112	5.0
Boron (B)	0.058	-

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## **5 CONCLUSION AND RECOMMENDATION**

### **5.1 Conclusion**

Based on the experimental investigation for the following conclusions are drawn:

The results of LOI in the two sample (2.49 % 600 °C SSA & 1.65 % 800 °C SSA) were met the value (6 %) of requirement stated by ASTM C 618, which means the percentage value of sample under the stated of the standard . However, sewage sludge (SS) of LOI was 55.34 %, which was in the result of SS is more than 6% that stated for pozzolanic materials in a maximum LOI and this indicated it has high carbon content. The SSA has shown low moisture and organic content according to obtain results from this study.

AAS test has shown that the sewage sludge ash contained primarily these oxides such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO in this study. The summation results of these oxides in SSA have incinerated those temperatures, which was used in this research work achieved 79.72 % and 80.95 % that is fulfills ASTM C618 requirement, which is the percentage of SSA greater than 70 %. The obtained results in the SSA that shows all these heavy metals (Cu, Zn, Ni, Co, Cr, &Pb) were below the allowable limit a given in USA – EPA standards.

The workability decreased with increase sewage sludge ash (SSA) content in the concrete mixture that SSA was burnt at temperature 600 °C and 800 °C due to the high water demand of SSA. Those result indicated that the early strength of the SSA concretes were weak as compared to the control concrete except 600 °C SSA5 % and 800 °C SSA 10 %.

The results of concrete work have shown that 600 °C SSA 5% & 800 °C SSA 10% replacement of OPC by sewage sludge ash achieved a higher compressive strength than the others percentage of set in this research at the age of 28 days as compared with control concrete and the compressive strength results of 0 % SSA, 600 °C SSA 5 % & 800 °C SSA 10 % at 28 days were 14.86 MPa, 13.45 MPa, &14.01 MPa respectively.

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The percentage of water absorption increased with increase the replacement of sewage sludge ash (SSA) in the concrete that incinerated at temperature 600 °C & 800 °C. The obtained results of leaching test in the concrete specimens that shown all these heavy metals (Cu, Zn, Ni, Mn, Cr, Cd, Hg, Pb, and As) are below the allowable limit a given in US EPA standards.

### **5.2 Recommendation**

In this research, the recommendations are set in the following way:

Kality wastewater treatment plants will use the incineration method in order to reduce the volume, organic content, and odor of the sewage sludge with the flue gas treatment technology.

Use the sewage sludge ash (SSA) to partial replace of Ordinary Portland cement (OPC) in concrete production to reduce the environment impacts.

The optimum of replacement of sewage sludge ash (SSA) is 600 °C SSA 5 % & 800 °C SSA 10 % of OPC to show higher compressive strength at 28 days as compared with the other percentage.

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**APPENDIX A: Physical properties of coarse and fine Aggregate**

**a. Physical properties of fine Aggregate**

**Moisture content of fine aggregate**

A = Weight of Original sample = 500 g

B = Weight of dry sample @ 105 °C for 24 hrs = 480 g

$$MC (\%) = \frac{A - B}{B} * 100 = \frac{500 - 490}{490} * 100 = 2.04 \%$$

**Specific gravity and absorption capacity of fine aggregate**

Bulk specific gravity

$$\text{Bulk sp gr} = \frac{A}{B+50-C}$$

Where

A=weight of oven dry sample in air (g)

B=weight of pycnometer filled with water (g)

C=weight of pycnometer with sample and water to calibration mark (g)

Bulk specific gravity (saturated-surface-dry basis)

$$\text{Bulk sp gr (saturated-surface-dry basis)} = \frac{50}{B+50-C}$$

Apparent specific gravity

$$\text{Apparent sp gr} = \frac{A}{B+A-C}$$

Absorption

$$\text{Absorption (\%)} = \frac{50-A}{A} * 100$$

$$C=0.9976Va + 500 + W,$$

Where;

C= weight of picnometer filled with sample plus water (g)

Va=volume of water added to pycnometer (cm<sup>3</sup>)

W=Weight of pycnometer empty (g)

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$$B=0.9976V + W$$

B= weight of flask filled with water (g)

V= volume of flask (cm<sup>3</sup>)

W= weigh of the flask empty (g)

Results of Specific gravity and absorption capacity of sample fine aggregate

$$A = 44 \text{ g}$$

$$B = 0.9976 (100) + 46 = 99.76 + 46 = 145.76 \text{ g}$$

$$C = 0.9976 (80) + 50 + 45 = 175.808 \text{ g}$$

$$\text{Bulk specific gravity} = \frac{44}{145.76+50-175.808} = 2.21$$

$$\text{Bulk specific gravity (SSD)} = \frac{50}{145.76+50-175.808} = 2.5$$

$$\text{Apparent specific gravity} = \frac{44}{145.76+44-175.808} = 3.15$$

$$\text{Absorption capacity (\%)} = \frac{50-44}{44} * 100 = 1.5$$

### Sieve Analysis of fine aggregate

Sieve size (mm)	Weight of sieve (g)	Wt. of sieve & Retained (g)	Weight of Retained (g)	Percentage Retained (%)	Cumulative Coarser (%)	Cumulative Passing (%)
9.5 mm	583	590	7	1.4	1.4	98.6
4.75 mm	430	443	13	2.6	4	96
2.36 mm	380	400	20	4	8	92
1.18 mm	350	410	60	12	20	80
600 $\mu\text{m}$	328	438	110	22	42	58
300 $\mu\text{m}$	315	480	165	33	75	25
150 $\mu\text{m}$	290	390	100	20	95	5
Total					245.4	

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Calculation: Fineness Modulus (F.M) of fine aggregate

$$F.M = \frac{\sum \text{cumulative coarse } (\%)}{100} = \frac{245.4}{100} = 2.454\%$$

### b. Physical properties of Coarse Aggregate

#### i. Moisture Content of Coarse Aggregate

Test Results:           Where: w = Moisture content, %

A = 2000 g                   A = weight of original sample, g

B = 1970 g                   B = weight of oven dry sample, g

$$W (\%) = \frac{A - B}{B} * 100 = \frac{2000 - 1970}{1970} * 100 = 1.52 \%$$

#### ii. Specific gravity and water absorption of coarse Aggregate

Test Results:Where:

A = 4790 g                   A = weight of oven dry sample in air, g

B = 4870 g = weight of saturated- surface –dry sample in air, g

C = 3130 g = weight of saturated sample in water, g

**Calculation:**

$$\text{Bulk Specific Gravity} = \frac{A}{B - C} = \frac{4790}{(4870 - 3130)} = 2.75 \text{ g/cm}^3$$

$$\text{Apparent Specific Gravity} = \frac{A}{A - C} = \frac{4790}{4790 - 3130} = 2.88 \text{ g/cm}^3$$

$$\text{Absorption Capacity} = \frac{B - A}{A} * 100 = \frac{4870 - 4790}{4790} * 100 = 1.67\%$$

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**iii. Sieve Analysis of coarse aggregate**

Sieve size (mm)	Weight of sieve (g)	Wt. of sieve & Retained (g)	Weight of Retained (g)	Percentage Retained (%)	Cumulative Coarser (%)	Cumulative Passing (%)
37.5 mm	1387	1387	0	0	0	100
19 mm	1389	2289	900	45	45	55
13.2 mm	1155	1555	400	20	65	35
9.5 mm	1170	1480	310	15.5	80.5	19.5
4.75 mm	1265	1455	190	9.5	90	10
Pan	750	950	200	10	-	-
Total					280	

**Calculation:** Fineness Modulus (F.M) of coarse aggregate

$$F.M = \frac{\sum \text{cumulative coarse } (\%)}{100} = \frac{280}{100} = 2.8 \%$$

**APPENDEX B: Proximate Analysis of SS**

*i. Moisture content*

The moisture content of sample was determined by weighing 1g of sample into pre-weighed crucible and drying it in an oven at a temperature of 105 °C for 24 hours till the constant weight dry matter was obtained. The percentage of moisture content computed by this formula:

$$\text{Moisture content}(\%) = \frac{W_1 - W_2}{W_1} * 100 \quad (0.1)$$

Where:  $W_1$  = weight of sample before drying (gram) without crucible weight

$W_2$  = weight of sample after drying (gram) without crucible weight

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Calculation of M.C:

$$\text{Moisture content}(\%) = \frac{1 - 0.54}{1} * 100 = 46 \%$$

### *ii. Ash content*

Ash is an inorganic residue remaining after the materials has been completely burnt at a temperature 800 °C in furnace. About 1g of dried sample was weighed with crucible ( $W_1$ ) and incinerated at 800 °C for 3 hrs. The crucible was cooled in desiccators and reweighed ( $W_2$ ).

$$\text{Ash content}(\%) = \frac{W_2}{W_1} * 100 \quad (0.2)$$

Where:  $W_1$  = the weight of sample before incineration (gram) without crucible weight

$W_2$  = the weight sample after incineration (gram) without crucible weight

Calculation of ash content:

$$\text{Ash content}(\%) = \frac{0.26}{1} * 100 = 26 \%$$

### *iii. Volatile content*

About 1g of dried sample was weighed with crucible ( $W_1$ ) and incinerated at 950 °C for 15 minute in furnace. The crucible was cooled in desiccators and reweighed ( $W_2$ ).The percentage of volatile content computed by this formula and set calculation.

$$\text{Volatile matter}(\%) = \frac{W_1 - W_2}{W_1} * 100 \quad (0.3)$$

Where:  $W_1$ = weight of sample before incineration (gram)

$W_2$  = weight of sample after incineration (gram)

Calculation of V.M:

$$\text{Volatile matter}(\%) = \frac{1 - 0.76}{1} * 100 = 24 \%$$

## **Characterization and use of sewage sludge ash as supplementary cementitious material for concrete**

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### *iv. Fixed carbon*

Fixed carbon is calculated value and it is the resultant of summation of percentage of ash content and volatile matter content subtracted from 100 (ASTM, 1989).

$$\text{Fixed carbon} = 100 - [\text{moisture} (\%) + \text{ash}(\%) + \text{volatile matter}(\%)] \quad (0.4)$$

Calculation of F.C:

$$\text{Fixed carbon} = 100 - [(46 \%) + (26\%) + (24\%)] = 4 \%$$

## **APPENDEX C: Physical properties of SSA**

### *v. Specific gravity*

Specific gravity of 600 °C and 800 °C incinerated SSA were determined at inorganic lab in school of chemical and bio engineering by using Le Chatelier's flask. Fill the flask with kerosene to point on the stem between the 0 and 1 ml mark. Record the first reading of volume after the flask has been immersed in the water bath with 20 °C then 50 g of SSA added in the flask and taken final reading of volume after the flask has been in water bath (ASTM C311- 05, 2008). The difference between the first and the final reading volume of liquid displaced by the mass of SSA used in the test and the specific density computed by this formula and set calculation.

$$\rho \left( \frac{g}{cm^3} \right) = \frac{M}{\Delta V} * 100 \quad (0.1)$$

Where: M= mass of SSA (gram)

$\Delta V$  = difference between the first and the final reading of volume (cm<sup>3</sup>)

Calculation of specific gravity at 600 °C SSA:

$$\rho \left( \frac{g}{cm^3} \right) = \frac{50 g}{[22.10 - 0.90]cm^3} * 100 = 2.36 g/cm^3$$

Calculation of specific gravity at 800°C SSA:

$$\rho \left( \frac{g}{cm^3} \right) = \frac{50 g}{[20.40 - 0.70]cm^3} * 100 = 2.54 g/cm^3$$



**Figure C1:** Le Chatelier's flask

*vi. Loss on ignition (LOI)*

About 1gm of SS and SSA were weighed with crucible (A) and ignited at  $750 \pm 50$  °C for 15 minute in furnace. The crucible was cooled in desiccators and reweighed (B) according to ASTM C311 (ASTM C311- 13 2013). The percentage of LOI computed by this formula and set calculation.

$$LOI (\%) = \frac{A - B}{A} * 100 \quad (0.2)$$

Where: A= weight of SSA without crucible before ignited (gram)

B = weight of SSA without crucible after ignited (gram)

Calculation of LOI at SS:

$$LOI (\%) = \frac{1 - 0.4466}{1} * 100 = 55.34 \%$$

Calculation of LOI at 600 °C SSA:

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$$LOI (\%) = \frac{1 - 0.9751}{1} * 100 = 2.49 \%$$

Calculation of LOI at 800 °C SSA:

$$LOI (\%) = \frac{1 - 0.9835}{1} * 100 = 1.65\%$$

### *vii. Specific surface area*

Blain air permeability was used to determine the specific surface area of SSA. It consists of a stainless steel cell, perforated disc and plunger. A U- tube glass manometer is fit to the steel stand. The set is supplied complete with rubber aspirator. Put filter paper into the cell then weight 2.2 and 2.35 gm of SSA sample in to it and put another filter paper on it then compress with the plunger. Attach the cell on top of U-tube manometer and opened side valve to evacuate the air in manometer through the side tube using the aspiration bulb until the oil reached level1 then closed the side valve and monitor the oil as it start to fall. Using stop watch, measure the taken for the oil to fall from level2 to level3. Blain fineness test of SSA was conducted in mugger cement industry.



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Figure C2: Blain fineness

The specific surface area of SSA computed by this formula and set calculation in the following ways.

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}}$$

Where: S = Specific surface area of the tested sample, cm<sup>2</sup> / g (SSA)

S<sub>s</sub> = Specific surface area of standard (reference) sample used in calibration, 3774 cm<sup>2</sup> / g (cement)

T<sub>s</sub> = Measured time interval, second of manometer drop for standard sample used in calibration of the apparatus, 37.8 s.

T = Measured time interval, second of manometer drop for tested sample.

$$w = gv (1 - e)$$

Where: w = weight of SSA, g

v = volume, 1.852 cm<sup>3</sup>

e = porosity, 0.5

g = specific gravity of SSA

At 600 °C SSA: T = 31.03 s

$$w = gv (1 - e) = 2.36 \frac{g}{cm^3} * 1.852 cm^3 (1 - 0.5) = 2.2 g$$

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}} = \frac{3774 cm^2/g \sqrt{31.03 s}}{\sqrt{37.8 s}} = 3419.4 cm^2/g$$

At 800 °C SSA: T = 32.05 s

$$w = gv (1 - e) = 2.54 \frac{g}{cm^3} * 1.852 cm^3 (1 - 0.5) = 2.35 g$$

$$S = \frac{S_s \sqrt{T}}{\sqrt{T_s}} = \frac{3774 cm^2/g \sqrt{32.05 s}}{\sqrt{37.8 s}} = 3475.1 cm^2/g$$

## **APPENDEX D: Test of concrete**

### **a. Workability test of fresh concrete**

Slump test is conducted following ASTM C143 procedures:

This test was carried out in metallic mold also known as slump cone, which is open at both ends and has attached handles. Cone was placed on a hard-non-absorbent surface and was filled in three layers with fresh concrete by tamping each layer 25 times.

The concrete was struck off at top of the cone to smooth out the surface and the mold was lifted carefully vertically upwards. Finally, the slump of the concrete is measured by measuring the distance from the top of the slumped concrete to top of the cone.

### **b. Compressive strength test of hardened concrete**

Compressive strength was conducted following ASTM C 39 procedures:

Two moist cured specimens were prepared for each sample that met the specific requirements mentioned in ASTM C39. The compressive strength of each sample was measured for 3 days, 7 days and 28 days respectively in a moist condition, i.e. immediately after it was taken out of the curing tank. The bearing surface of the sample was cleaned, and bottom and the top bearing plate was placed on top and bottom of the sample. The sample was aligned with the axis of the testing machine. Testing machine was turned on and different properties of samples were entered, and the rate of loading was fixed at 15 MPa which was kept constant for all samples. The load was then applied till the sample failed and the maximum load taken by the sample was recorded