Addis Ababa University
Addis Ababa Institute of Technology
School of Chemical and Bio Engineering

Textile Sludge Based Bricks Production from Hawassa Industrial Park Zero Liquid Discharge Facility

By:

Tsegay Gebremedhin

A Thesis submitted to the School of Chemical and Bio Engineering, Addis Ababa Institute of Technology, in Partial Fulfillment of the Requirements for the Degree of Master of Science in Environmental Engineering

Advisor: Dr. Eng. Shimelis Kebede (Assistant Professor)

December, 2018

Addis Ababa, Ethiopia

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DECLARATION

I hereby declare that the thesis work which is entitled as “Textile Sludge-Based Bricks Production from Hawassa Industrial Park Zero Liquid Discharge Facility” submitted by me, is my original work, and has not previously been presented for a degree of any other university and all the resource materials used for the thesis have been duly acknowledged.

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I certify that the above declaration provided in this thesis is accurate to the best of my knowledge.

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ABSTRACT

Textile sludge is an inevitable byproduct discharged from wastewater treatment plants and becoming a great challenge in today's industrial parks. Presently, the sludge generated from the ZLD facility is sent and collected inside nearby big constructed shed without any treatment. Since, the sludge is characterized by high level of organic and inorganic components including heavy metals, its accumulation is a burden to the industry and affects the environment and human well-being adversely. Hence, it demands an alternative sludge disposal method. Therefore, the main objective of this study was to investigate the physio-chemical sludge characteristics and suitability of utilizing sludge in the manufacture of bricks replacing natural clay the case of Hawassa industrial park. The test results revealed that concentration of BOD, COD and the principal heavy metals viz. Pb, Zn, Cr & Mn of the textile sludge were 535, 9820 mg/l and 2.25, 30.45, 8.08, 6.75 mg/kg respectively. Additionally, the proximate analysis of the sludge moisture content, VOM, ash and fixed carbon was 82.5, 48.4, 44 and 7.6 percent respectively. Similarly, the quantity of chemical constituents SiO_2, Al_2O_3, CaO and Fe_2O_3 conducted by using chemical analysis methods, were 16.10, 21.40, 2.56 and 2.32 in percent respectively. The presence of these compounds indicates the potential use of this sludge as partial replacement of building materials. In this work, the effects of sludge proportion 10%, 15% and 20% by dry weight and firing temperature of 800℃, 900℃ and 1000℃, on the quality of the bricks (Water Absorption, and Compressive Strength) were examined and the results were compared with those of controlled bricks. From the results, the minimum values of compressive strength obtained was 2.93 N/mm^2 at 20% and 800℃ whereas the maximum amount is 4.2 N/mm^2 at 10% sludge mix and 1000℃. Again, the Water Absorption of 21.33% and 12.4% was gained at 20% and 10% sludge content. Moreover, the sludge amended up to 20% mixing ratio shows minimum leachability of heavy metals as performed by ICP-OES and compatible as compared to the USEPA standard. Consequently, the experiment work indicates the potential of textile sludge in the manufacturing of bricks.

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List of Abbreviation’s

AAS: Atomic absorption spectroscopy

AOAC: Association of Official Agricultural Chemists

APHA: American public health and association

ASTM: American society for testing and materials association

AWWA: American Water Works Association

BOD: Biological oxygen demand

CaO: Calcium oxide

CETP: Common Effluent Treatment Plant

COD: Chemical oxygen demand

CS: Compressive Strength

CV: Calorific Value

EPA: Environmental Protection Agency

ETIDI: Ethiopian Textile Industry Development Institute

ETP: Effluent Treatment Plant

HIP: Hawassa Industrial park

ICPS: Inductively Coupled Plasma Spectroscopy

IPDC: Industrial Park Development Corporations

LOI: Loss of Ignition

VOM: Volatile Organic Matter

WA: Water Absorption

WEF: Water Environment Federation

XRF: X-ray Fluorescence Spectroscopy

ZLD: Zero Liquid Discharge

CHAPTER ONE

1 INTRODUCTION

1.1 Background

The world’s ever-increasing population and the progressive adoption of an industrial based life style has inevitably led to an increased anthropogenic impact on the biosphere. This is due to the fact that extensive utilization of chemicals including dyes, pigments, and aromatic molecular structural compounds for several industrial applications such as textiles, printing, pharmaceuticals, food, toys, paper, plastics, and cosmetics manufactured and used in day-to-day life (Karthik & Rathinamoorthy, 2015). Generally speaking, environmental pollution is a serious global environmental issue due to an unavoidable consequence of economic development and people’s desire to improve their standard of living (Jahagirdar *et al*., 2013).

As long as there is an industry there is a waste generated from the plant either in the form of solid, semisolid, liquid or gaseous form. However, the amount and quality of waste produced is different for different industries. This may be due to difference in the manufacturing processes and treatment given to the wastewater. More specifically, textile industries involve processing or converting of raw materials into finished cloth materials by employing various processes, operations and consumes large quantity of water and produces extremely waste effluents (Balasubramanian *et al*., 2006; Palanisamy, 2011; Sandesh *et al*., 2014). While treating the waste water released from textile industries huge volume of sludge is generated. This sludge must be managed in an environmentally acceptable way.

As mentioned previously, textile industry is one of the prominent industries that uses a variety of chemicals and a large amount of water for all of its manufacturing steps. This discharges a corresponding wastewater effluents containing various pollutants (Palanisamy, 2011). For purifying this waste water to an acceptable discharge limit, various treatment processes have to be employed. As a result of this, a large quantity of semisolid by product known as sludge is generated each year from the treatment plant. The quantity of the sludge produced depends upon the amount of wastewater and the type of treatment adopted for treating the wastewater (Guha *et al*., 2015).

Recently, Ethiopia has inaugurated one of the first and largest industrial park in Africa named Hawassa Industrial Park, which was built on 300 hectares of land and has been dedicated to host mainly textile and garment products (Arkebe, 2016). The benefit of this industrial park can be seen in one of the following perspectives. From an economic point of view, it plays an important role in the economic development of the country. More importantly, it is amongst the top foreign exchange earning industries for the country that can generate about 1 billion U.S. dollars per annum. From the stand point of job creation, it provides employment and business opportunity to a significant number of people (Helen, 2017). On the other hand, it is one of the main sources of environmental pollution. One of the major concerns amongst these is safe and sound disposal of sludge wastes (Lissy & Sreeja, 2014).

Currently, eighteen leading global apparel and textile companies from America, China, India, Sri Lanka as well as six local manufacturers are operating within the park. These industries have established Zero liquid discharge facility (ZLD) that enables to recycle 90 percent of sewerage disposal water and fulfills international standards (Arkebe Oqubay, 2016). However, the main problem encountered within the industrial park is sludge disposal problem. The sludge is generated during the treatment process as a result of chemical coagulation (by addition of aluminum/iron/ magnesium salts and lime), flocculation and liquid/solid separation (Helen, 2017).

Therefore, the disposal of sludge is an inescapable by-product of textile wastewater treatment process due to the use of various dyes & chemicals in different wet processing steps. The textile sludge includes a cluster of organic (having high BOD and COD load) and inorganic complex with high concentrations of heavy metals such as Iron, Copper, Cadmium, Zinc, lead, Chromium and so on (Guha *et al.*,2015). In addition, the sludge contains pollutants and unstable pathogen content, therefore leading to potential health and environmental hazards. The inorganic salts and toxic metals in the sludge pose a threat to residents (Palanisamy, 2011; Shivanath *et al*., 2011). It is categorized under toxic substances by statutory authorities. Besides, even though the amount of heavy metals in the sludge is negligible they may have cumulative effect on the environment. The problem of sludge disposal will be intensified as the amount of sludge produced increases (Karthik & Rathinamoorthy, 2015; Tay, 1987). Compositions of sludge vary considerably depending on the wastewater composition and the treatment processes used (Sandesh *et al*., 2014).

Nowadays, the treatment of industrial effluents invariably results in the generation of large volume of the sludge transferring pollutants from liquid phase to solid phase. This indiscriminate disposal of sludge from the waste water treatment plant is a burning issue because it has a significant impact on the environmental media specifically contamination to soil, surface water and groundwater. Eventually, these wastes cause alteration of the physical, chemical, and biological properties of aquatic environment that is harmful to public health, livestock, wildlife, fish, and other biodiversity (Adyel *et al.*, 2013; Shivanath *et al.*, 2011).

Presently, the sludge generated from the ZLD facility is sent and collected inside nearby big constructed shed without any treatment. Since, the sludge contains the chemicals removed by the purification processes it is considered to be a non-biodegradable (recalcitrant) waste material. As a consequence, sludge accumulation is a burden to the industry and affects the environment adversely (Adyel *et al.*, 2013).

Hence, it is now a global concern to find a socio, techno-economic and ecofriendly solution to dispose industrial solid wastes. There are several sludge management options available. Those are like use of the sludge as fertilizer for agriculture, incineration, sanitary land filling, mixing with cement, biogas generation, as additive in construction materials such as concrete, bricks, tiles and so forth (Guha *et al*., 2015). The conventional disposal methods like landfilling and incineration may not be suitable because the leachate from the landfilling sites and the residues from the incinerators induce secondary pollution (Baskar *et al*., 2006). Common method adopted for disposing the sludge is land filling. Landfill disposal of the sludge has drawbacks like high cost of transportation, difficulty in getting suitable sites for land filling, heavy metal contamination of the land, emission of foul gases etc., (Shivanath *et al.*, 2011). Therefore, it is crucial to find an effective and sustainable solution for the management of textile sludge. The use of Textile sludge in construction materials could serve as an alternative solution to disposal and reduce pollution (Rahman *et al*., 2017).

Utilization or reuse of textile sludge as building materials is a win-win strategy for the reason that it not only converts the wastes into useful materials but also alleviates the disposal problem. The prospective benefits of using sludge or sludge ash as the brick or tile additive include immobilizing heavy metals in the fired matrix, oxidizing organic matter and destroying any pathogens during the high temperature firing process, and reducing the frost damage based on the results of several full or bench scale studies (Weng *et al*., 2003). The recycling of industrial solid wastes as substitute for building materials is not only environment friendly but also cost-effective alternative way to sustain a cleaner and greener environment (Patel *et al*., 2017). Efforts are being made to utilize the sludge for making useful materials. Considering the sludge characteristics and the demand of construction material in Ethiopia bricks production is the best option.

This study focuses on the possibility of using sludge as a brick material. The chemical composition of the sludge waste contains significant amount of silica, alumina, magnesia and so on. Thus, this indicates it is possible to use it as partial replacement of building materials or brick manufacturing. The utilization of textile sludge merely depends on its chemical composition and physico chemical characteristics. In other words, reusing sludges as a partial replacement of clay for making bricks is one of the most feasible solution to sludge disposal problem unless the final product pollutes the environment over a longer period. For this reason, brick construction from sludge is effective enough to manage and utilize the sludge for commercial purpose. It is estimated that at present demand in the form of building materials (brick) would be around above million pieces per year in Ethiopia (Ethiopian Embassy, 2010).

The usage of sand, stone, gravel, lime, clay, gypsum, etc. as building materials and manufacture of other building materials such as brick, cement, etc. depletes the existing natural resources and damage the environment due to continuous exploitation. Hence the usage of industrial solid wastes as building materials will exhibit environmental benefits such as conservation of natural resources/raw materials, decrease mining activity, reduce landfill capacity, minimize global warming (Shivanath *et al.*, 2011).

So far, there is no significant work done on the textile sludge for management and utilization in HIP. So that, it is vital to recycle the sludge into construction material. This paper, assesses the effect of mixing proportion and temperature on suitability of textile sludge for bricks. Moreover, laboratory scale clay brick specimens were prepared with different proportions of sludge and their suitability as an engineering material was assessed based on their strength and water absorption characteristics. Lastly, leaching test of sludge amended bricks was also carried out to verify the possibility of leaching of heavy metals to the environment in the long run.

1.2 Statement of the Problem

Lately, Textile Industry is considered as number one priority sector by the Government‘s Industrial Development Strategy (ETIDI, 2014). As a result, many Industrial Parks are evolving at an alarming rate from time to time. More specifically, HIP is one of the largest manufacturing industries producing textile and garment products having a great impact on the economic development. On the contrary, it generates a massive quantity of sludge to the environment after purifying the effluent waste water through zero liquid discharge facility. Presently, the Industrial Park generates sludge at a rate of 500kg/day (personal communication with the Deputy manager of the ZLD facility). This sludge is thrown away as worthless material which leads to environmental pollution. Some researchers have linked the sludge impact to the environment in such away. It has an impact on the soil composition by the input of compounds enriched in the sludge. Similarly, the impact on the percolating water and consequently on the ground water quality by the immobilization of the compounds accumulated in the soil as well as on the neighboring environment by eventual problems of odour nuisance are the main problems (Sulthana *et al.*, 2013). On top of that, the inorganic salts and toxic metals in the sludge pose a threat to residents (Balasubramanian *et al*., 2006; Shivanath *et al*., 2011). Therefore, considering the impacts of textile wastewater sludge on the environment, it is an important issue and should be considered along with the wastewater management in an industry.

To reduce the adverse environmental impacts of the sludge no satisfactory attention and action is taken up to the present. Therefore, effective management and utilization of sludge becomes important. There are various methods to handle sludge waste materials for sustainable development. Reuse of the sludge as building material is the most environmentally friendly and economically viable as it generates resources, which are in high demand and eliminates problems associated with sludge disposal. In addition, building construction sector consumes conventional materials such as clay, which is generated directly from natural resources. Fast growth of building construction industry is putting enormous load on the natural resources leading to environmental degradation (Lissy & Sreeja, 2014). So that, sludge waste recycling has a great potential for natural resource conservation like clay because it has the potential for use as substitute in building material. This resource conservation goal is supportive enough to sustainable development, where rapid industrialization and economic development is putting a lot of pressure on it.

1.3 Objectives of the Study

1.3.1 General objective

The principal aim of this study was to assess the suitability of Textile Zero Liquid Discharge Facility Sludge utilization in the manufacturing of bricks.

1.3.2 Specific objectives

- To carry out the characterization of the textile sludge sample.
- To investigate the feasibility of textile sludge as ingredient in brick making and recommend as an alternative to conventional bricks.
- To investigate the appropriate raw material mix proportion.
- To characterize the property of the final brick's product.
- To examine the effect of key operating parameters (mixing proportion and temperature) determining the brick quality (Weng *et al*., 2003).
- To perform the leachate analysis of the bricks.

To answer the aim of this study, it's vital to use the following research questions.

- ◊ What is the characteristics of the sludge generated from Hawassa industrial park?
- ◊ How can the sludge be managed and utilized?

1.4 Significance of the study

This research will have a paramount significance in providing concrete evidence for development policy makers in assuring sustainability by mitigating the environmental threat caused by unsafe generation of sludge. In the same manner, it will provide baseline information to Ethiopian industrial parks as well as environmental protection agencies and other interested researchers in the future.

Moreover, this study contributes in management and utilization of the textile sludge which is important to protect the harmful effect of the sludge and to keep the sustainability of the environment. Additionally, the use of the textile sludge has a great contribution in conservation of natural resources like clay and in providing low cost building materials and generation of income to the company.

Generally, recycling of such waste as raw material alternatives may contribute in the conservation of natural resources; improvement of the population health, protection of the environment and in providing baseline information to concerned bodies and future researchers.

CHAPTER TWO

2 LITERATURE REVIEW

2.1 Background

The economic history and development experience of countries around the globe show that the pace of economic growth is greatly linked to the rate of waste generation. Waste may be defined as an unwanted material generated after the manufacturing process of industrial, or from agricultural, or from house hold activity. It is the discarded material which essential requirement of disposal. Waste causes many nuisances in the environment. It produces many types of viral or bacterial infection for the human and animal which create bed effect on health (Ashani, 2015).

The industrial sector has long been considered the main engine of economic growth and structural transformation. Particularly, textile industry is among the vital industrial sectors that plays a crucial role for development of any country as far as it produces one of the basic needs of human being clothe materials. However, it produces a great deal of sludge during its treatment of waste water. Specifically, Hawassa Industrial Park uses an advanced wastewater treatment plant the so-called ZLD facility for recycling process.

2.2 Zero Liquid Discharge Technology

The literal meaning of ZLD is zero discharge of wastewater from Industries. A ZLD system involves a range of advanced wastewater treatment technologies to recycle, recovery and re-use of the ‘treated’ wastewater and there by ensure there is no discharge of wastewater to the environment (Hussain, 2014). In other words, Zero Liquid Discharge (ZLD) is an ambitious wastewater management strategy that eliminates any liquid waste leaving the plant or facility with the majority of water being recovered for reuse. ZLD obviates the risk of pollution associated with wastewater discharge and maximizes water usage efficiency, thereby striking a balance between exploitation of fresh water resources and preservation of aquatic environments. Achieving ZLD, however, is generally characterized by intensive use of energy and high cost.

The Zero Liquid Discharge system comprises of the following components.

- Pretreatment
- Reverse osmosis

- Evaporator and Crystallizer

The ZLD Technology is highly recommended for industries generating a wastewater with high TDS/ salinity such as pharmaceutical, pulp and paper, tanneries and textile plants. This is because the inability of removing the salinity in a waste effluent by the conventional treatment methods i.e., physio - chemical and biological methods (Hussain, 2014).

ZLD technology is beneficial for the plant's water management; encouraging close monitoring of water usage, avoiding wastage and promotes recycling by conventional and far less expensive solutions. Generally, there are three principal benefits of this type of facility. Firstly, it recycles greater than 98% of the water so as to reduce the natural water demand. Secondly, recovery of over 90% of salt by products. Finally, it has a lion's share of contribution in the protection of the environment from wastewater discharge pollution by meeting the stringent regulatory limits.

2.3 Challenges of Zero Liquid Discharge Facility

One of the main challenges of ZLD facility is the generation of hazardous solid/semisolid wastes that create disposal challenges need to think of Zero Waste Disposal (ZWD) Plants. Similarly, in association to this Economic viability- cost and availability of water, regulatory pressures are the real driving force. In addition, High Carbon footprint as well as high Operating cost and financial impact on the industry and its Regional/ National/Global competitiveness (Hussain, 2014).

Waste generation and management is becoming a global challenge resulting into increased environmental concern. Waste management and recycle into a sustainable construction material as proved to be an alternative for waste disposal helping out in the area of environmental pollution and economics. In recent years various type of waste has been used/reused in the development of sustainable construction materials (Johnson *et al.*, 2014).

2.4 Sludge generation

Sludges are the solids, liquid or semisolid residuals (concentrated contaminants) generated as a byproduct of wastewater treatment. Sludge generated in effluent treatment plants is not only troublesome to that industry but also affects the environment adversely. Usually sludge contains 0.25-12 % solids by weight, depending upon the operations and the processes used (Karthik & Rathinamoorthy, 2015).

Sludge is a hazardous material that harms both human health and the environment. Currently, growing volume of sludge from industrial and municipal wastes is disposed of haphazardly. Environmental protection is our prime concern for our existence and for the future generations as well. Though difficult, but all relevant authorities need to take necessary measures for management and safe disposal of sludge.

Textile effluent contains both inorganic and organic substances in dissolved, colloidal or suspended forms and is typically coloured due to the presence of residual dyestuffs. The inorganic substances contain hazardous materials like Chromium, Cadmium, Beryllium, Lead, Mercury, Nickel, Aluminum and the toxic organic substances have high BOD and COD load. Hence, various physical, chemical and biological methods are used for the treatment of textile wastewater depending upon its characteristics. The suspended and dissolved solids and ones, which are added during the wastewater treatment process, are separated in the form of settleable solids called sludge.

Depending upon the physical, chemical and biological treatment given to the wastewater, they are classified as chemical sludge and biological sludge. Generally, physico-chemical (primary) treatment is most commonly used for satisfactory removal of colour from textile effluent. Different coagulants like alum, ferrous sulphate and lime are effective for colour and organic removal from textile wastewater. So that, coagulation prior to biological treatment might be advantageous for alkaline wastewaters. The physico-chemical treatment leads to the generation of large quantity of chemical sludge, which is inorganic and non-biodegradable in nature (Patel & Pandey, 2008).

The chemical sludge is an unwanted residual solid generated in the textile wastewater treatment plant and its management is a critical environmental issue (Iqbal *et al.*, 2014; Karthik & Rathinamoorthy, 2015; Lissy & Sreeja, 2014). The safe disposal of these types of wastes has received a considerable attention in recent years to protect the environment (Bhalerao *et al.* 1997).

2.4.1 Chemical sludge

As stated previously, in a textile effluent treatment plant, the major types of sludge are generated as a result of physico-chemical and the biological processes. The chemical sludge is generated in the form of solids and semi-solid residuals of the concentrated contaminants. The dye-stuff present in the effluent gets precipitated in the form of settleable sludge slurry in the presence of chemical additives like ferrous sulphate and lime.

The chemical sludge from the textile industry contains inert solids, polymer solids, precipitated dye products, metal salts and other chemicals. It is considered as a hazardous waste as per Hazardous Waste management and handling Amendment Rules 2003 (Schedule I, category No. 24) due to the presence of objectionable materials such as metals and toxic chemicals such as surfactants and chlorinated organics. The two aspects that are of great environmental concern are the quantity and toxicity of chemical sludge. The composition of wastewater, chemicals used, and treatment units mainly determine the amount and properties of chemical sludge (Patel & Pandey, 2008).

2.4.1.1 Characteristics of chemical sludge

Characterization of sludge is a crucial step towards management of waste. It provides useful information primarily about the chemical substances present in it at the point of generation and the extent and quantity of substances of concern being emitted to the environment. In addition, it also provides useful data on the type of treatment and disposal practices to be considered for its environmentally sound management (Metcalf & Eddy, Inc. *et al*., 2001).

Few researchers have been involved to characterize the chemical sludge from textile waste water treatment plants. Patel & Pandey, (2008) obtained semi-dried textile sludge samples generated by physico -chemical treatment of textile effluent from drying beds of effluent treatment plant of a local textile unit engaged in the production of bleached and dyed/printed, cotton, polyester cotton and viscose fabrics. The sludge pH was found to be alkaline due to the addition of lime during the primary treatment (chemical coagulation) of the textile effluent. Electrical conductivity values were also high (1.15 - 1.16 S/m). The total solid content was 20 - 25 % out of which 30-40 % was volatile solids. The calorific value was very low (496.6 kcal/kg) and the specific gravity in the range of 1.11-1.12. They also found traces of heavy metals like cadmium, chromium, cobalt, copper, lead, nickel and zinc in the sludge samples. Conversely, Heavy metals like arsenic and mercury were rarely present.

Additionally, other researchers like Balasubramanian *et al.* (2005) and Baskar *et al*., (2006) have attempted to characterize chemical sludge from common effluent treatment plants in textile industry. Balasubramanian *et al.* (2005) found the specific gravity of sludge as 2.4 and the volatile content as 32% which resulted in very high ash content as a result of incineration and therefore it was not recommended as a sludge disposal option. They also analyzed the heavy metal concentrations existed in the sludge such as Cd, Cu, Total Cr, Zn, Ni and Pb. Furthermore, they performed physico-chemical characterization of oven dried sludge at 105°C.

The sludge generated from textile industry is both organic and inorganic with measurable quantities of metals and other compounds. The sludge can contribute to low dissolved oxygen concentration in areas where sludge solids are concentrated. Land application of sludges leads to leachates problem, ground water pollution, erosion, runoff and odour. It may also affect the food chain. Some knowledge of the sludge characteristics is required to select the best appropriate means of sludge handling and processing.

2.5 Impact of textile sludge disposal

Sludge can become a problem unless improperly managed or disposed of. The substantial amount sludge generated from textile industry such as HIP is getting rid of into the nearby constructed shed that leads to environmental pollution. The potential effect of textile sludge can be seen from three main perspectives. Those are, environmental, economic and public health. Again, it may also have classified as primary and secondary impact. It can induce three adverse impacts on the environment.

Primarily, it causes land degradation by the input of compounds enriched in the sludge to the soil such as potentially toxic elements and compounds, pathogens and parasites having a secondary effect on the natural vegetation and plant growth. Secondly, it causes the surface water and ground water contamination by the immobilization of the compounds accumulated in the soil and consequently health hazard. Thirdly, it has an impact on the aesthetics of the environment by creating problems of odour nuisance that can lead to public reaction & adverse impact on health. Therefore, taking in to consideration the toxic characteristics of the sludge and considerable amount, management of chemical sludge is a demanding issue. So, to address this crucial problem in an economical and environmentally sound manner careful selection of sludge management and disposal method is required (Karthik & Rathinamoorthy, 2015;Patel & Pandey, 2008).

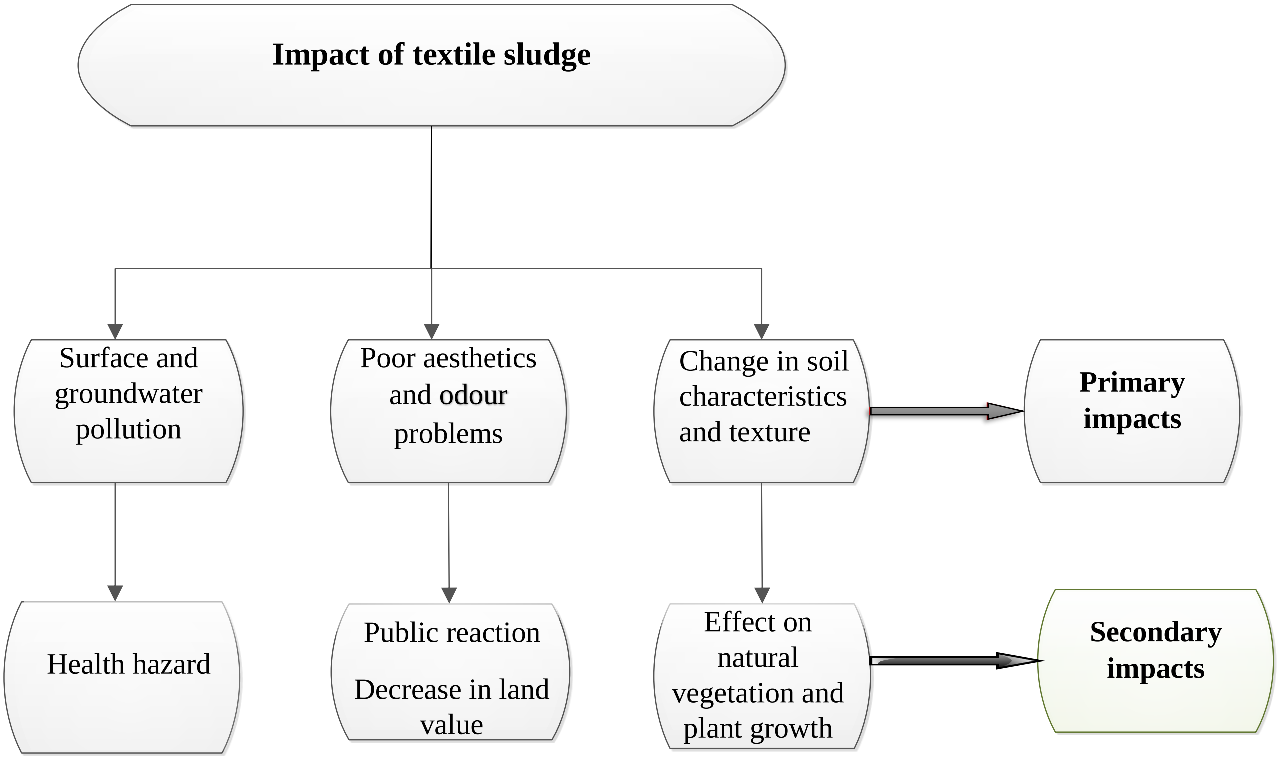

Figure 2.1 Impact identification network for dumped textile sludge.

[Adapted from]: (Karthik & Rathinamoorthy, 2015)

2.6 Sludge management options

The following sludge management options are available.

- Anaerobic digestion
- Aerobic digestion (composting)
- Agricultural use
- Controlled landfill
- Thermal incineration
- Land application (filling material e.g. for flood prevention)
- Recycling in brick, cement or asphalt making

2.6.1 Description of the management options

2.6.1.1 Anaerobic digestion (biogas recovery)

It may be beneficial to add sludge from the biological treatment based on activated sludge treatment to an anaerobic digestion plant or conduct co-fermentation with municipal sewage sludge and other suitable materials to collect biogas for energy production and save emissions. In addition, nutrients in the residue can be used as a fertilizer if the input materials all may be used in agriculture. However, when sludge from industries are used, it is necessary to keep in mind, that some substances may have an inhibiting effect on the microorganisms to optimize the digestion process. The inhibitors commonly present in anaerobic digesters include ammonia, sulfide, light metal ions, heavy metals, and organics.

Anaerobic digestion is not permissible for sludge from hazardous industries CETP in any case as the risk of toxic substances causing emissions that are harmful for human beings and the environment is high.

2.6.1.2 Aerobic digestion (composting)

Composting can be used to produce fertilizer for application in agriculture. To gain a suitable compost, carbon-rich material is required and an optimized C: N ratio would be 25 – 30:1. As not all sludge ensures this ratio, so composting material like green waste, sawdust, woodchip, rice and straw can be added. The major advantages of promoting composting are an increase of the C: N, a reduction of salt, heavy metal and leaching of hazardous and toxic substances.

Therefore, this option should be prohibited for the use of hazardous waste and even for non-hazardous waste from industries when the product is intended to be used in agriculture.

2.6.1.3 Controlled Landfill

Controlled landfill describes the possibility to deposit the sludge in the ground. To have a better control of greenhouse emissions (methane) and leachate, the waste (sludge) has to be deposited in dedicated landfill sites. Categories exist for deferent kinds of waste depending on its hazardous potential or pollutants, which have different requirements for the construction, the pollutants (measure in leachate) and monitoring. This is very costly and difficult for acquisition of land.

suitable waste disposal landfill sites are becoming more and more difficult to locate due to expanding urbanization, poor landfill capacity and unsuitable buffer zones (Sarkar *et al.,* 2017).

2.6.1.4 Thermal incineration

Currently available and applied co-incineration technologies are listed below. The sludge coming from the biological treatment with volatile moisture content is added to the incineration chamber. Before energy can be produced the sludge has to be dried in the chamber. It is possible that the incineration consumes more energy for drying than it produces.

Although this technique enables a recovery of energy, it is only discussed as an alternative if other disposal routes are restricted or too expensive. In the use of incineration, the observation of emission limits is very important. Besides furans, dioxins and a number of other flue gas components about 5 to 10 % of the total chromium is converted from chromium to the carcinogenic chromium. These have a harmful impact on human health and the environment and therefore, the installation of expensive pollutant filters is required. In incineration existing plants these filter systems need to be given as well.

2.6.1.5 Land application

Land application includes a wide variety of uses such as Filling material for flood prevention, material/ substrate for re - cultivation of mining sites or covering landfill sites. Land application does not include agricultural use it can be assumed that sludge appropriate for agricultural use is also suitable for land application, but when using large amounts, nutrient content must be taken into consideration to minimize leaching.

Land application of textile sludge can be a good solution, whereas it is cost-effective disposal method for treatment plants and also can provide a favorable fertilizer for agricultural lands. It provides an economical alternative for the final disposal of the textile sludge, but heavy metals in textile sludge is always an issue restricting its general use. Therefore, removal of heavy metals prior to land application is likely to be a possible and practical means for reducing metal content in textile sludge (Maddumapatabandi *et al*., 2014).

2.6.1.6 Recycling

Recycling has become an essential alternative method for management and utilization of textile sludge because it has dual importance of the need to control pollution and economic value (Karthik & Rathinamoorthy, 2015). Recycling the wastes by incorporating them into building materials is a practical solution to pollution problem. The utilization of wastes in clay bricks usually has positive effects on the properties, although the decrease in performance in certain aspects has also been observed. The positive effects such as lightweight bricks with improved shrinkage, porosity, thermal properties and strength can be obtained by incorporating the recycled wastes. Most importantly, the high temperature in clay brick firing process allows: (a) volatilization of dangerous components, (b) changing the chemical characteristics of the materials, and (c) incorporation of potentially toxic components through fixation in the vitreous phase of the waste utilized (Kadir & Mohajerani, 2011).

The increase in the popularity for using environmentally friendly, low-cost and lightweight construction materials in the building industry has brought about the need to investigate how this can be achieved by benefiting the environment as well as maintaining the material requirements affirmed in the standards. Brick is one of the important materials used in construction industries (Sulthana *et al.*, 2013).

So far, some studies were reviewed by many researchers on the potential of sludge applications. It is reported that Tay (1984;1987) used municipal wastewater sludge and pulverized sludge ash mixed with clay to produce bricks respectively. the addition of 10% to 50%) pulverized sludge ash was carried out and it was concluded that 50% was the maximum to produce a good quality brick. Leaching tests conducted on the sludge product also showed positive results with no sign of potential contamination problems for similar applications. Another sludge that was recycled by Tay *et al.* (2001) was the industrial sludge. Bricks were manufactured from industrial sludge from 30% up to 100%. The firing temperature employed was 1050°C. During the observation, cracks were prone to occur during the firing with 100% sludge and 90% sludge.

2.6.2 Utilization of textile sludge for bricks

Major research is being conducted in the field of utilization of sludge in brick manufacturing. The quality and type of brick made depends on various factors like composition of sludge, additives used, temperature at which the brick is fired, water content etc. The sludge is detrimental because of its recalcitrant nature and long biological half-lives.

Brick is one of the oldest manufactured building materials in the world and it is extensively used even at present because of its durability, strength, reliability, low cost, easily availability etc. Burnt clay bricks are commonly been used as a solid matrix mainly due to their characteristics, such as a good mechanical resistance, satisfactory stability (Jahagirdar *et al.*, 2013). In many areas of the world, conventional bricks are produced from clay as a basic building material for construction of houses with high temperature kiln firing which result in scarcity of natural resource material for production of the conventional bricks.

Still now, bricks are one of the most demanding masonry units. The main raw material for bricks is clay. Clays used for brick making vary broadly in their composition and are dependent on the locality from which the soil originates. Different proportions of clays are composed mainly of silica, alumina, lime, iron, manganese, Sulphur and phosphates. For example, the chemical composition of Ethio bricks factory clay samples were shown as in Table 2.1below.

Table 2.1The chemical composition of clay samples collected from Ethio bricks factory

Components	White soil (%)	Red soil (%)
CaO	<0.01	<0.01
SiO_2	66.61	55.30
Al_2O_3	11.56	16.40
Fe_2O_3	8.16	9.62
MgO	0.16	0.60
Na_2O	2.60	1.16
K_2O	3.06	1.48
MnO	0.32	0.34
P_2O_5	0.09	0.15
TiO_2	0.57	1.12
H_2O	2.17	4.32
LOI	4.04	8.15

Source: Adapted from Ethio brick factory laboratory report

The worldwide annual production of bricks is currently in billion units and the demand for bricks is expected to be continuously rising. Quarrying operations for obtaining the clay are energy intensive, adversely affect the landscape, and generate high level of wastes. It is also noted that there is a shortage of clay in many parts of the world. To protect the clay resource and the environment, some countries such as China have started to limit the use of bricks made from clay.

The increase in demand for construction materials in the recent years as a result of development has called for an alternative way to develop or derive construction materials from other sources. In order to meet the increased demand, attention has been given to the development of sustainable construction material. The usage of improved construction materials in construction industry has been on increase daily which has led to the investigation of its environmental impact and meeting required standards when waste is used in developing sustainable construction material. The treatability studies using solidification/stabilization indicate that chemical sludge generated from treatment of textile dyeing waste water has the possibility to be used as the construction material (Patel and Pondey, 2009).

For environmental protection and sustainable development, extensive research has been conducted on production of bricks from waste materials. Therefore, industrial waste and by-products could be valuable alternative resources for building construction and other applications. Numerous attempts have been made to incorporate industrial waste in the production of bricks. So far, a wide variety of waste materials have been studied to produce bricks such as paper, tannery, textile industry generated wastes. For illustration, the usage of textile effluent treatment plant sludge, fly ash, cotton waste, rice husk ash, granulated blast furnace slag, processed waste tea, construction and demolition waste, mine tailings, wood sawdust, petroleum effluent treatment plant sludge, craft pulp production residue and waste paper pulp (Johnson *et al.*, 2014; Shakir *et al.*, 2013; Kadir & Mohajerani, 2011;Balasubramanian *et al.*, 2006).

The utilization of these wastes will help to reduce the negative effects of their disposal. However, the potential wastes can only be recycled if the properties and the environmental pollutant of the new manufactured brick meet the specific requirements and comply with the relevant standards (Kadir & Mohajerani, 2011). Many scholars in different disciplines have studied how to convert waste sludge’s from domestic and industrial operations into useful products, such as retrieving the organic components in sludge to improve farm land, palletized aggregate for concrete, replacing cement in concrete, production of ceramic bricks and tiles, mixing sludge with clay to produce building bricks (Baskar *et al.*, 2006).

More importantly, The use of sludge as construction and building material not only converts waste into useful products but also eliminates disposal problems. Natural resources like clay are also conserved (Baskar *et al.*, 2006).

The research is focused merely to textile sludge generated from zero liquid discharge treatment plant. Although much research has been conducted, the commercial production of bricks from waste materials is still very limited. For wide production and application of bricks from waste materials, further research and development is needed, not only on the technical, economic and environmental aspects but also on standardization, government policy and public education related to waste recycling and sustainable development.

Solidification is such a technique that stabilizes and solidifies components of waste materials. The solidified products can be disposed of to a secure landfill site or recycled and reused as construction materials, namely, bricks, concretes, roofing materials, tiles, building blocks, and so forth if meet the specific requirements. Although there are related works on the sludge of different industries to make constructional materials. Present study systematically investigates the partial replacement of clay or soil by sludge in the manufacturing of building materials such as bricks. In order to get quality products, the influence of sludge proportion and firing temperature on water absorption, and compressive strength were investigated..

If the utilization of the waste like sludge in clay bricks usually has positive effects on the properties such as lightweight bricks with improved shrinkage, porosity, thermal properties, and strength. The reuse of textile sludge as substitute for clay will reduce clay content in the fired clay brick, and then reduce the manufacturing cost and become economical for construction to use it in a more efficient and sustainable manner.

Recycling of sludge is an alternative method to avoid the problem posed by the excessive sludge production from textile industries. An accepted recycling option is to replace raw materials with dried sludge (or sludge from thermal treatment) in the production of cement, bricks, tiles, ceramics, glass and asphalts. Some clays are decent in organic content which is why an addition of sludge is desirable. The oxidation of this material during the brick firing process improves the quality of the resulting bricks. The amount of replaced raw material can vary from 10 to 70% but a decrease in compressive strength depending on the sludge content has to be considered.

Many studies have been conducted in this area and reported that the pollution level is high in ground water and nuisance due to dumping in the treatment plant area premises. There is a growing need to find alternative solutions for the sludge management. In the present study, an attempt has been made to utilize the textile ETP sludge in making of construction materials. According to (Palanisamy, 2011) textile sludge was incorporated in fly ash brick manufacturing and it was observed that with increase in sludge content, there is a decrease in compressive strength of bricks. Pulverized and sieved bricks show better compressive strength when compared with pulverized form and grinded form.

Baskar *et al.*, (2006) reported that addition of textile Sludge with clay up to 30 % by weight and mixed well to form a homogeneous mixture, led to results indicating that the compressive strength is greatly dependent on the amount of waste in the brick and the firing temperature. The compressive strength of bricks decreases with the increase of waste mix in the bricks and increases with the increase of firing temperature. Similarly, Balasubramanian *et al*., (2006) have reported suitability of textile ETP sludge as partial replacement of cement in structural and non-structural building materials. It is found that the use of textile sludge in partial replacement of cement results in reduction of strength of the materials. It is finally recommended that the substitution of textile ETP sludge for cement, up to a maximum of 30 %, may be possible in the manufacturing of non-structural building materials such as in flooring tiles, solid and pavement blocks, and bricks.

On the other hand, (Weng et al., 2003) found that brick were produced by direct substitution of clay with dried sludge collected from industrial waste water plant without further treatment and they were taken sludge proportion and the firing temperature as the key factors determining the brick quality. It was observed that with up to 20% sludge added to the bricks, the strength measured at temperatures 960 and 1000 ℃ met the requirements of the Chinese National Standards and increasing the sludge content results in a decrease of brick shrinkage, water absorption, and compressive strength. Again, toxic leaching characteristic tests of brick also showed that the metal leaching level is low. Finally, they were recommended to utilize 10% sludge with 24% of moisture content prepared in the molded mixtures and fired at 880 – 960^{o}C for manufacturing of good quality bricks. In the same manner, (Ravikrishnan & Senthilselvan, 2014) investigated that production of bricks through the combination of textile sludge, clay and lime. In this study, the compressive strength, water absorption and efflorescence test were conducted. Consequently, it was suggested that the substitution of textile sludge up to 25% by weight of clay materials gives satisfactory results and the optimum amount of textile Sludge waste was found to be 10% by weight of clay which gives better bonding, higher compressive strength and lower water absorption. This recycling of sludge converts waste into useful materials and eliminates problems associated with sludge disposal. Therefore, sludge recycling is considered to be a better alternative than land disposal in the environmental point of view.

Additionally, Jahagirdar *et al.,* (2013) discussed the reuse of textile mill sludge in fired clay bricks. The textile mill sludge was mixed together in different proportions (5–35 %) as the raw material in this study. The brick was fired at 600 - 800 °C for 8, 16, and 24 h. Another research carried out by (Sulthana et al., 2013), assessed production of bricks by mixing textile sludge with clay with 3% sludge increment up to 30% that is satisfactory for 15% addition as first class quality bricks and 20-30% addition as sludge mix ratio satisfies for second class bricks requirements.

However, investigating the effects of using textile sludge with some admixtures in clay bricks has not been duly explored in the open literature. Therefore, the objective of the present study was to investigate the incorporation of textile sludge in the manufacture of bricks and to examine the influence of textile sludge proportion and firing temperature in raw materials in relation to the brick quality. In this case the sludge characteristics were performed and textile sludge were added at three different dosages 10, 15 and 20% by weight of clay to produce the bricks. Eventually, the mechanical and physical properties of the sludge substituted bricks namely compressive strength, water absorption were analyzed and compared with the control bricks. All the brick specimens investigated were produced on a small scale in a maffle furnace.

CHAPTER THREE

3 MATERIALS AND METHODS

3.1 Materials

The main raw materials used for the brick making purpose consists of textile sludge, clay (both Red and White type), sand and water. Analytical grade reagents were also used such as nitric acid for digestion.

3.1.1 Analytical instruments

The major instruments that was used during the laboratory analysis are mentioned as below. The chemical composition of raw materials was determined using X-ray fluorescence (XRF); pH value was measured using digital pH meter (Hl5221, HANNA, Italy); BOD apparatus and some reagents were used to measure BOD of the sludge samples. COD reactor (Hl 839800, HANNA, Italy), to measure the proximate analysis plates, crucible, drying oven (Beschickung, loading model 100 - 800, MEMMERT, Germany), muffle furnace, grinding machine, desiccators, ASTM compression strength machine to measure compressive strength. and digital weighing balance (SARTORIUS AG, BP110, Germany) were used. Besides, AAS and ICPS were used for analysis of heavy metals in the textile sludge and leachate in the bricks respectively.

3.2 Methods

The methodology for this study mainly comprises of sample collection, sample preparation, characterization, mixing, brick making, testing procedure, and lastly leachate analysis to determine the feasibility of the textile sludge in the production of bricks. Generally, these methods are briefly explained in detail as below.

3.2.1 Sample collection

The textile sludge for this study was collected by random sampling method from the Zero liquid discharge facility of Hawassa industrial park, Hawassa city, Southern part of Ethiopia. It is about 275kms away from Addis Ababa.The sample was collected from the temporary sludge storage area found in the ZLD treatment plant premise at different time duration in the months of march and may respectively. The samples were collected from all corners of the storage area and mixed so as to attain representative sample. The sample was packed in 25 kg of plastic container and brought to Addis Ababa for laboratory analysis. It was then, preserved and kept in a deep freezer below 4℃ and transferred to place of characterization within 24hrs. In addition to this, the other raw material clay has been obtained from a local brick manufacturing plant known as Ethio brick factory. Similarly, sand was used collected from nearby construction areas. All the materials have been collected personally.

3.2.2 Sample preparation of the raw materials

The sludge sample was dried in an oven with aluminum foil covered plate in some consecutive days for 24h at 105℃. Then, the size reduction of dried sample was carried out until it gets fine powder using laboratory electrical mill. After this the sieve analysis was done by a sieve of 1mm mesh size by a sieve machine and finally packed into airtight sealed polyethylene bags and kept in the storage area for further analysis. In the same manner, the clay sample was sun dried for 24h and crushed using the electrical mill and sieved by a sieve of 1mm size. The sand preparation was also conducted following the same procedure.

3.2.3 Characterization of textile sludge

The chemical properties of ZLD sludge used in this study were analyzed as per standard methods.

3.2.3.1 pH value determination

The pH of the textile sludge was determined using digital pH meter according to (H. Patel & Pandey, 2008).

3.2.3.2 Calorific Value determination

The calorific value of textile sludge was determined from the dried sludge at 105°C and then was measured using an adiabatic oxygen Bomb calorimeter (1241EF, PARR MOLINE ILLINOISE, USA) according to (H. Patel & Pandey, 2008).

3.2.3.3 Proximate Composition Analysis

The proximate analysis basically includes the determination of, moisture content, volatile organic matter, fixed carbon and ash content present in the sludge sample. The laboratory methods for conducting the proximate analysis of this research were carried out based on standard procedures set by APHA, 1998. Each of them was conducted as indicated in Appendix A. The moisture content was determined by drying a weighed quantity of the sludge sample in an oven at 105℃ for 24 hours and taking its difference. It was determined according to APHA method (APHA 2540 G). The amount of VOC was determined by igniting the oven dried textile sludge sample in a muffle furnace according to (ASTM D 3175). The ash content was determined by burning the sample in a muffle furnace according to AOAC (2000) using the official method 923.03 and applying a simple formula as shown in Appendix A. The percentage fixed carbon was determined directly by deducting the sum total of volatile matter and ash percentage from 100. The experiment was made at Addis Ababa Institute of Technology, School of Chemical and Bio Engineering laboratory area. For further information the experimental procedure for proximate analysis and other physico chemical characteristics are given in Appendix A.

3.2.3.4 BOD, and COD determination

The quantity of organic compounds in wastewater is generally evaluated by chemical oxygen demand (COD) and biological oxygen demand (BOD) test. BOD is defined by the amount of oxygen expressed in mg/l or ppm that bacteria take from water (waste water) when they oxidize organic matter. The carbohydrates (cellulose, starch, sugars), proteins, petroleum hydrocarbons and other materials that comprise organic matter get into water from natural sources and from pollution. They may be dissolved, like sugar or suspended as particular matter, like solids in sewage.

Generally, BOD the basic means to determine the degree of pollution or the efficiency of waste water treatment plant knowing that the influent BOD and effluent BOD.

More specifically, the BOD is determined by BOD apparatus and some analytical reagents. The formula used to compute the BOD_5 was taken from the difference of initial and final dissolved oxygen according to standard methods for examination of waste water (APHA 5210 B).

$BOD_5 = (D_5 – D_1)$ mg/l x dilution factor ………………………………………………………. (A)

Where; D_1 = initial amount of dissolved oxygen in mg/l

D_5 = final amount of sample dissolved oxygen in mg/l

Figure 3.1 BOD test experiment of the sludge

Chemical oxygen demand (COD) is termed as the amount of a specific oxidizing agent that reacts with sample under controlled conditions and it is expressed as oxygen equivalence. This parameter indicates the extent of organic matter contamination of water and is always higher than the biochemical oxygen demand (BOD). It is used to indicate organic matter contamination and it helps in knowing overall organic load to the receiving body.

The chemical oxygen demand (COD) values are a measure of the amount of oxygen required to completely convert organic compound into carbon dioxide and water. COD determination is used to measure the organic and inorganic matter content of a water or waste water sample by digesting it in a COD reactor (Benis *et al.*, 2010). Therefore, COD is determined by digestion in a COD reactor followed by spectrophotometer measurement. Detailed description is given in Appendix A.

Figure 3.2 COD test using spectrophotometry

3.2.3.5 Concentration of heavy metal analysis

Digestion of the textile sludge were the primary task in determination of heavy metals. This is conducted to reduce interferences by organic matter and to convert the metals associated with particulates to a form (usually the free metal). The heavy metal analysis for Cr, Cd, Mg, Ca, Mn, pb, Fe, Cu, Ni and Zn was conducted using flame Atomic Absorption Spectrophotometry (PG 990, FAAS Hydride and cold Hg technique, Germany). This analysis was performed at JIJE LABOGLASS Pvt. Limited company laboratory.

3.2.3.6 Chemical composition determination

The chemical compositions of raw materials are well correlated with their mineralogical composition. The textile sludge constitutes mainly of the major oxides such as SiO_2, Al_2O_3, CaO and Fe_2O_3. Thereby, it can be used in the production of a construction material such as bricks. The Chemical composition of the sludge used in this study were analyzed in the Chemical Industry Corporation Mugher Cement Factory Laboratory, Mugher town, as per standard methods.

To determine the chemical constituents in the textile sludge X- ray fluorescence (Thermo Fisher ARL 9900, United states) and chemical analysis method was used. The step by step procedures to determine those predominant oxides were reported in Appendix A.

3.2.4 Production of brick sample

For this study, three principal raw materials were used to produce bricks i.e., clay, textile sludge and sand. There are several fundamental steps involved in the formation of bricks. Those are mixing, moulding, drying and firing. In this study, two different series of mixing proportions were attempted as shown in Table 3.1.

Table 3.1 Raw material proportions prepared for brick making by dry weight

Trial 1	a	b	c	**Trial 2**	a	b	c
Textile sludge	10	25	40	Textile sludge	10	15	20
Clay	90	75	60	clay	90	85	80

The case of trial 1, the 25 and 40% mixing ratios were not given the required brick strength. However, the case of trial 2, all proportions were satisfied the requirement of brick strength excluding the 20% mixing ratio. It was also tried with the addition of 10 percent sand assuming to increase bricks strength nevertheless it didn’t give as it was supposed to be.

As mentioned previously, the mix proportion of the raw materials required to produce the bricks were given in Table (3.1). Furthermore, Ethio brick factory uses two types of soils for brick manufacturing with the ratio of three white soil to one red soil. So that, for simplicity it is decided to use similar proportions for this study.

In the beginning, sludge free bricks were made (without addition of sludge) as a control specimen, then textile sludge is used to replace both soils by weight. Firstly, the aforementioned raw materials are mixed and homogenized with each other in proportion before the addition of water to produce sample bricks. As can be seen in Table (3.1) trial 2 shows, 10%, 15% and 20% proportion of the sludge by weight was replaced for both soil types. Thereafter, the mixture was then mixed properly by adding sufficient amount of water. Finally, filled in to an oil lubricated mould to form the desired shape. Mould size of 10 x 10 x 5 cm^3 were used to mould the bricks or 10cm length, 10cm width and 5cm height mixed the materials very well and the mixtures were moulded manually.

Figure 3.3 Air drying in the lab room before and after firing in the furnace

The moulded brick specimens has then been maintained at room temperature for uniform drying conventionally inside the laboratory as depicted in Figures 3.3.

Before the firing process begins, most of this water is evaporated in this process aiming to prevent cracking. After a week and 2days of air-drying period the bricks were ready to be fired in a furnace. Finally, the dried bricks were burned in a maffle furnace (Nabertherm, LH 60/40, Germany) in the range of varying temperatures 800, 900 and 1000℃ and were held in a constant time period of 3h respectively, to observe the effect of temperature on its quality. Consequently, the bricks were allowed to cool down and transferred for physical and mechanical property as well as leachate analysis test to assess and compare the engineering quality of the brick with the controlled bricks.

3.2.4.1 Experimental procedures for brick making

The experimental procedure for the brick making work was carried out in the following steps:

1. First, a dried powder raw material was prepared in different containers.
2. Then, the dried clay soil (25% red soil and 75% white soil) was mixed with homogeneous mixtures of textile sludge at various proportions.
3. The mixing proportion of the sludge was prepared as, 10%, 15% and 20% and 10% sand has been used as additive. Control bricks of pure clay with 3:1 of white and red was also prepared and kept as a reference for this study.

4. The mixtures of the sample have been mixed and moulded and kept at room temperature. Solid bricks having different size were molded and prepared.
5. Then, the samples were dried in the open atmosphere for 11 days, Before the brick is fired, it must be dried to remove excess moisture. If this moisture is not removed, the water will burn off too quickly during firing, causing cracking.
6. Next to this the dried sample were fired in an electric furnace (Nabertherm, LH 60/14, Germany) at high temperature in the range of (800 - 1000℃) with an interval of 100℃ for about 3hours. The samples were taken out from the furnace and kept for cooling.
7. Water absorption, and compressive strength were evaluated for assessing the mechanical and physical properties of the manufactured bricks. The test methods were carried out using standard methods. Then, the compressive strength of the samples was measured to know their quality and compare to the control sample.
8. The water absorption capacity of the building material was determined as follows: each of the building materials were immersed in water for 24 h, then taken out and wiped dry; each unit is weighed immediately after wiped; and the percentage was taken.
9. Finally, Leachate analysis investigation for the leachability of the metals from the bricks was performed.

As stated earlier, three 0% sludge clay sample were prepared as a control specimen. All The produced sample bricks are then tested for compressive strength and water absorption as per standards. Since the prime objective of this research is to be utilized the textile sludge waste as a source for making bricks, it needs to be tested to check its quality. Since, the compressive strength and water absorption are the most dominant properties that are used to determine the quality of building materials (Jahagirdar *et al*., 2013; Baskar *et al.*, 2006). Both of them are selected for testing the different specimens of bricks. In the end, leachability test was also carried out. The average value of the test results was taken from the two replicate tests.

3.2.5 Testing the property of brick sample

3.2.5.1 Compressive strength test

The compressive strength is the key parameter used for ensuring the engineering quality of building materials. Then, the compressive strength of the bricks was calculated from the recorded findings and the dimensions of the bricks using the formula given below.

Compressive strength = $\frac{Maximum\ load\ at\ failure\ (N)}{Area\ of\ the\ bricks\ (mm2)}$ ………………………………………. (B)

The value of compressive strength should be > 3.5 N/mm^2 for good quality bricks.

3.2.5.2 Water absorption test

Water absorption is the most important factor to test building materials next to compressive strength. It measures the amount of water embedded by the brick after a 24h submersion in water. It can be expressed as a percentage and defined as ratio of the weight of water that is taken up into its body divided by dry weight of the unit.

Mathematically, it can be calculated by the following formula.

Water Absorption (%) = $\frac{W2-W1}{W1} * 100$ ……………………………………………………….. (C)

Where, W_2 and W_1 are weight of the brick after and before immersion in water.

In any case, it should be < 20% to fulfill the requirement of bricks.

3.2.5.3 Leachability test of the bricks

Leaching is a process by which soluble constituents are dissolved from a solid material (solid or waste) into a contact water phase (McIsaac & Matko, 2016). The leachate can impact the local environmental media.

The leachate analysis test is highly important to know the concentration of the toxic heavy metals leached from the textile sludge-based bricks. In this case, the bricks were immersed in water for certain period of time and the leachate analysis was conducted at Horticoop Ethiopia (Horticulture) PLC, Addis Ababa, Ethiopia using ICP-OES Analysis examination method.

CHAPTER FOUR

4 RESULTS AND DISCUSSION

This part presents the results of some characteristics of basic parameters of textile sludge and the final bricks property in a tabular or graphical format. The parameters investigated include pH, calorific value, proximate components, heavy metals and chemical constituents. The results of properties of the textile chemical sludge are presented as below in the following Table 4.1& 4.3. Moreover, the test results of the bricks are also included in the subsequent Figures.

4.1 Characteristics of the textile sludge

4.1.1 pH

The pH value of the textile sludge was alkaline which shows a range from 8.1 – 8.4 with a mean value of 8.2. The result of some researchers shown that the pH value is very close to the value of this study. For instance, (Weng *et al.*, 2003; Patel & Pandey, 2008) found that it is in the range of 7.8 – 8.5 and 8.02 – 9 respectively. This is due to the addition of coagulating chemicals during the pre-treatment stage. Moreover, (Balasubramanian *et al*., 2006) also found very close value to the pH range mentioned earlier. The above analyses were conducted according to (H. Patel & Pandey, 2008).

4.1.2 Calorific value

The heating value of the textile sludge was found to be 1880.32 cal/gm. In other studies, such as (Patel & Pandey, 2008) it was found to be as low as negligible to as high as 2066.33 cal/gm. Thus, the calorific value of the present study doesn't show a significant variation as compared to the aforementioned result. This might be happened due to the existence of high level of organic chemical compounds in the sludge. On the other hand, as reported by Bhalerao *et al*, (1997) and Ansari and Thakur (2001) the calorific value was in the range of 495 to 498 kcal/kg. In addition, according to Anwar *et al.*, (2018) the calorific value was found to be 4,261.91 kcal/kg which is higher than ever reported values. As a result of this, the calorific value obtained in this study was neither too small nor too greater, so it has its own contribution in the utilization of sludge for brick manufacturing especially in saving energy during the firing process. The experiment was performed at Geological Survey of Ethiopia, Hydrocarbon Laboratory Analysis section, Addis Ababa.

4.1.3 BOD and COD Characterization

The experimental result that was obtained for BOD, COD and TOC were 535 mg/l, 9820 mg/l and 51.3% respectively. This indicates that the textile sludge has high amount of organic and inorganic pollutants due to the addition of various chemicals in the production process as well as in the wastewater treatment stage. When compared this value to the literature data values studied by Patel & Pandey, (2008) the Total Organic Carbon (TOC) ranging from 1.23 % to 17.82% was gained. This shows the TOC of this study is much higher than that given in the literature and the great variation in TOC is due to the variation of chemical doses and treatment processes available from one industry to another industry. For the COD and BOD, the researcher didn’t get any results in the literature but comparing to the allowable discharge limits as listed in EPA the result shows it was a bit higher and dumping this to the environment without any treatment is above the permissible limit and very harmful.

4.1.4 Proximate analysis result

Resulting the sludge characteristics analysis, the proximate analysis of ZLD facility textile sludge sample from HIP is given in Table 4.1.

Table 4.1 Proximate analysis of textile sludge sample

Parameters (%)	**Percentage value**
Moisture content	82.5
Ash content	44
VOM	48.4
Fixed carbon content	7.6

As stated earlier, proximate analysis was made to determine the presence of the four essential components namely fixed carbon, ash content, moisture content and volatile matter in the textile sludge. As indicated in Table 4.1, the mean value of the proximate analysis is revealed, so the moisture content of the textile sludge was 82.5% and the ash content, organic matter and fixed carbon contents were 44%, 48.4%, and 7.6% respectively. This result shows that the sludge get rid of from the ZLD facility has high amount of moisture content and ash content as well as having voluminous amount of organic content (volatile matter), whereas it has low amount of fixed carbon.

The quantity of moisture content obtained in the literature was found to be fluctuating from 4.6 – 94.6%. For instance, (Balasubramanian *et al.*, 2006) was found 28.72% moisture content whereas (Anwar et al., 2018) was obtained more than 80% moisture content. Similar moisture content was also reported by Rahman *et al*., therefore the results of this study are closer to those later findings. While the volatile content are found to be in the range of 24.2 – 80 %, and in one case the volatile content was found to be high as 80 % as stated by Patel & Pandey, 2008. This result suggested that the organic matter significantly contributed to the high value of volatile matter. Therefore, except the moisture content the value of proximate components of this study are comparable to literature results gained by Balasubramanian *et al.*, (2006). The ash content was determined to be 44% in the sludge showing a significant amount of inorganic component.

4.1.5 Heavy metal content determination

Table 4.2 Heavy metal analysis of the textile sludge sample

Heavy Metals	**Quantity (mg/kg)**	**Literature range (Patel & Pandey, 2008)**
Cu	5.23	9.9 – 57.48
Zn	30.45	0.65 - 306
Fe	323.63	
Pb	2.25	0.50 - 27
SiO_2	279.60	
Ca	0.28	
Mg	3.58	
Cd	ND	0.10 - 396
Cr	8.08	4.70 - 199
Mn	6.75	
Ni	ND	0.68 – 0.42

ND: refers to not detectable

In this part, the inorganic elements of the textile sludge sample, particularly the heavy metals were investigated. As can be shown above, the heavy metals in the sludge were analyzed and their value of concentrations were found to be, Zn (30.45 mg/kg), Fe (323.63 mg /kg), Cr (8.08 mg/kg), Cu (5.23 mg/kg), Mn (6.75 mg/kg), and so on. The result shows iron is found as leading constituent and the most harmful metals Cd and Ni concentration is below detectable limit. The high concentration of iron could be owing to the addition of chemical coagulants such as iron chloride in the treatment process. On top of that, the concentration of lead is below the regulatory limits of the united states EPA.

On the contrary, from the well-known toxic heavy metals existed in textile sludge only Cu, Zn and Cr are found to be a little bit above the permissible limit value. The heavy metal concentration obtained in the literature are quite different from researcher to researcher, this is due to the fact that the presence of heavy metals in the sludge are highly dependent on the dosage of dye molecule, water and chemical impurities utilized in the manufacturing process. Furthermore, except Zn and Cr most of the heavy metals concentrations are below the literature range results described in Table 4.2.

At the same time, Baskar *et al.*, (2006*)* found Cu, Zn, Cd and Cr concentration in common effluent treatment plant sludge to be 119,190, 5.6 and 358 mg/kg respectively. Similarly, Balasubramanian *et al*., (2006) obtained 57.48, 91.6, 3.96, 108.2, 154.3, 180.5, 12.2, 2.98, and 0.68 mg/kg concentration of heavy metals for Cu, Zn, Cd, Ca, Mg, Fe, Pb, Cr and Ni. Again, according to Anwar *et al.*, (2018) data results the metal content of Cu, Cd, Cr, and Pb were 57.5, 4, 3 and 12.1 mg/kg. Comparing all the heavy metal analysis or characterization results to the present study indicates there is a great variation due to the previously mentioned reason. The literature results show the concentration of the heavy metals are quite high while the findings of this study are not as such significant. However, the disposal of sludge to the environment causes a considerable risk. So, it is better to reuse it for manufacturing of bricks.

4.1.6 Chemical composition determination

The chemical analysis results of the sludge are given in Table 4.3 as below.

Table 4.3 Chemical composition of textile sludge

Minerals	**Chemical analysis content by weight (%)**	**XRF analysis content by weight (%)**
CaO	2.56	9.8
SiO_2	16.10	18.15
Al_2O_3	21.40	25.3
Fe_2O_3	2.32	3.12
MgO	1.51	3.11
SO_3	0.94	5.88
LOI	51.3	
Na_2O		2.75
K_2O		0.27

As can be seen in the Table above, the chemical analysis data result shows, the textile sludge composed of high alumina (Al_2O_3) and silica (SiO_2) content relative to the other components. This chemical composition data suggests that the textile sludge has a potential to be used as partial substitute of clay soil for making building materials such as bricks. However, as revealed before it is hard to use it alone due to the less quantity of mineral oxides that do not fulfill the average amount of oxide requirements.

Clearly, the characteristics of good bricks are largely depending on the main chemical constituents' silica, alumina, lime, ferric oxide and Magnesia with an average quantity of 50% to 60%, 20% to 30%, 2% to 5%, 5% to 6% and less than 1% by weight respectively (Altayework, 2013).

The existence of these components plays highly important role in the property of the bricks. The presence of silica component prevents cracking, shrinking and warping of raw bricks. It imparts uniform shape to bricks. If silica is in excess, it makes the brick brittle. In the same manner, the occurrence of alumina imparts plasticity to earth so that it can be moulded easily. If alumina is present in excess, raw bricks shrink and warp during drying and burning. Moreover, lime causes grains of sand to melt and bind the particles of clay together. It prevents shrinkage of raw bricks. If lime is excess, it will cause the brick to melt and hence shape is lost. Simultaneously, Iron oxide act as a flux to cause the grains of sand to melt and this helps to bind the particles together. It imparts red colour to brick on burning. Excess amount of iron oxide makes the brick dark blue. Finally, the role of Magnesia is to impart yellow colour to brick and decreasing shrinkage. Excess magnesia leads to decay of bricks.

Obviously, the chemical composition of the sludge is very much related to the minerals found in clay but with lower amount. As given in Table 4.3, the main components of textile sludge were CaO (2.56%), Al_2O_3 (21.4%), SiO_2 (16.1%), and Fe_2O_3 (2.32%). This result shows that the textile sludge has similar components to clay, which means a likeness in terms of composition to clay, unlike percentage quantities. On the contrary, as can be seen in the stated table, the quantity of the silica content in the sludge is exceptionally low which is 16.10 to 18.5 percent according to the chemical analysis and XRF analysis test result that implies it has much lower amount of silica than that found to be in the standard range 50 – 70 percent in the material for building materials. As shown in the Table 2.1, both types of soils have 66.61 and 55.30% silica content for white and red soils respectively, which is extremely high and in the required range. Class III bricks are possible with these soil samples.

Several researchers described the great role of silica content in the strength of bricks. For instance, Hegazy *et al.*, (2012) revealed that the bricks strength is highly dependent on the amount of silica in the raw materials which implies the higher the silica content the stronger the bricks. Moreover, the less amount of silica in the sludge indicates it lacks in binding property if it is used as additive in building material. On the other hand, the quantity of the remained principal chemical compounds satisfies more or less the minimum percentage requirement. Also, the high value of Loss on ignition (L.O.I), with weight loss of approximately 51% indicates the availability of high volatile and organic content in the sludge, which suggested that the removal or reduction of the organic content was required at high temperature.

The present study contained the same chemical composition as compared to Rahman *et al.*, (2017). However, it is not quit consistent to other literature data results that reported by (Baskar *et al.*, 2006; Patel *et al*., 2017; Sandesh *et al*., 2014). This might be due to variation of the chemicals and other compounds utilized in the wastewater treatment plant and the manufacturing processes.

Therefore, it is so difficult to use textile sludge to produce bricks solely but suitable to make bricks by partially replacing some percentage of other materials such as clay material as raw material so as to get the required amount of chemical composition in the mixture.

4.1.7 Testing the bricks property

The textile sludge-based sample bricks were tested for compressive strength, water absorption and finally leaching test. The compressive strength and water absorption test results of the bricks produced from the mixture of clay and oven dried textile sludge are depicted in Figure 4.2 and Figure 4.3.

4.1.7.1 Compressive strength test results

In this study, the sludge proportion was varied from zero to twenty percent by weight and the firing temperature was conducted from 800 to 1000℃ at three levels. In other words, the maximum quantity of sludge used in the production of bricks is 20% because the trial made above this proportion have poor bonding property and it results in an easily breakable brick. From Figure 4.2, it can be observed that except for the 20% sludge mix the compressive strength of textile sludge amended bricks is more or less satisfied the minimum requirement of compressive strength for bricks which is 3.5 MPa according to ASTM.

4.1.7.2 Water absorption test results

Although there are numerous factors that affect the durability of bricks such as type of raw material, mixing proportion and method of manufacturing, compressive strength and water absorption are predominantly two important factors in the characterization of bricks. This study investigated the influence of sludge mixing proportion and burning temperature on the brick water absorption. The water absorption obtained is a minimum of 12.4% and maximum of 21.33% at high temperature and low sludge proportion for the former and vice versa for the later value.

4.1.7.3 Leachate test results

Table 4.4 Leachate Analysis Test Results

Parameters		**Content (mg/l)**	**USEPA Concentration limit (mg/l)**
Copper	Cu	0.003	100
Iron	Fe	0.006	-
Zinc	Zn	0.003	500
Nickel	Ni	0.048	1.3
Cobalt	Co	0.004	-
Manganese	Mn	0.006	260
Chromium	Cr	0.053	5.0
Cadmium	Cd	0.001	1.0
Mercury	Hg	0.045	0.2
Tin	Sn	0.066	-
Lead	Pb	0.003	5.0
Arsenic	As	<0.001	5.0
Boron	B	0.0295	-

Table 4.4 shows the leachability result for the textile sludge-based bricks. The results indicate that the elements Tn, Cr, Ni and Hg have shown relatively maximum concentrations in descending order whereas other metals such as Cd, As, Pb, Zn and Cu have shown that minimum leachability from the brick sample. However, as the result data obtained indicates the amount of all the parameters of heavy metals is still below the allowable limit given in USEPA standard. This is due to the phenomenon of heavy metals in sludge is completely vaporized at high firing temperature during sintering process, therefore could reduce the heavy metals inside the brick.

4.1.7.4 Effect of the sludge addition on the quality of bricks

The mixing ratio of sludge have a great influence on the compressive strength of the bricks. Compressive strength has been found to be inversely proportional to the sludge content and directly proportional to the firing temperature. This may be due to the removal of the organic compounds from the internal and peripheral structure of the bricks which has a possibility of creating pores and voids and increase in bulk density resulting from increased firing temperature. To repeat, the compressive strength decreases as the sludge proportion increases.

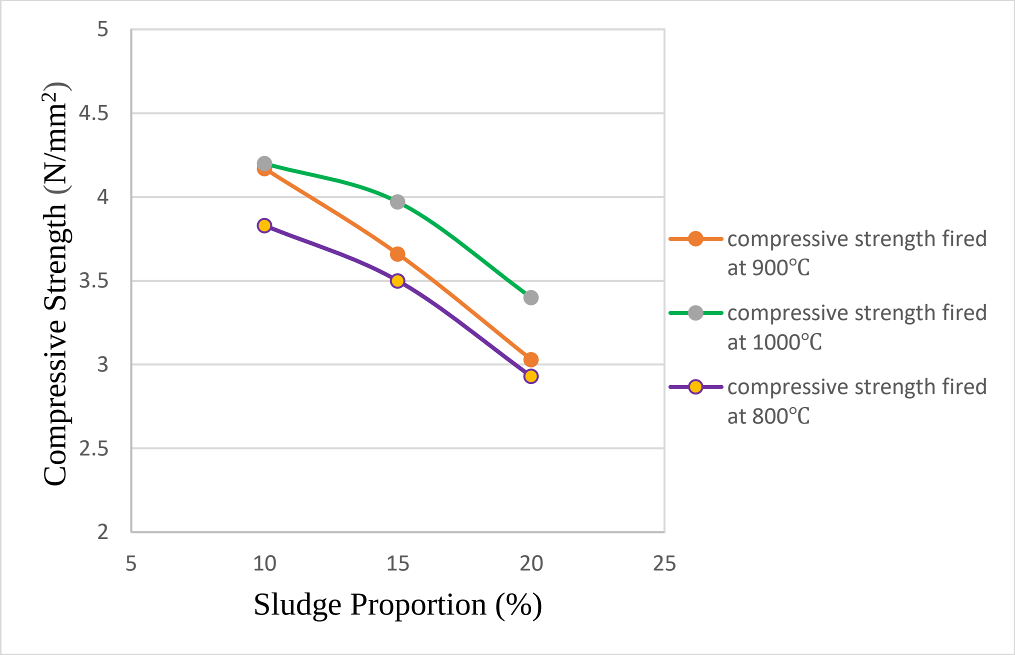

Figure 4.1 effect of textile sludge proportion on the compressive strength of bricks

Moreover, since the sludge has much less amount of chemical compositions especially silica that implies an increase in sludge content creates low binding power with the clay. So, the percentage of maximum 20 sludge content gives quite good result from the investigation and the compressive strength of 20% sludge content burned at 1000℃ is 3.40 N/mm^2. The minimum values of compressive strength obtained was 2.93 N/mm^2 at 20% and 800℃ whereas the maximum amount is 4.2 N/mm^2 at 10% sludge mix and 1000℃. Therefore, the addition of 10% and 15% sludge content can be considered or classified as second-class bricks based on ASTM standards.

Although, the relationship between compressive strength and sludge percentage shows same trend that is an increase in sludge content results in a corresponding decrease of compressive strength. The mixing proportion in this study is found to be less than as compared to other studies. Some of them used up to 30% of textile sludge as a substitute of clay to produce bricks but in this case 15% that gives successful results. The addition of sludge beyond this amount gives unsatisfactory results or poor-quality bricks. This is because the variation in property and composition of the sludge from one industry to another industry.

Based on Balasubramanian *et al*., (2006) findings it is recommended to use up to 30% textile sludge to replace Portland cement in the preparation of building materials and reported that as the sludge mix is getting higher the compressive strength of the brick drops gradually. In the same manner, Rahman *et al*., (2017) reported same results. To conclude, the effect of sludge content in the bricks on the compressive strength, the result shows that the compressive strength of the bricks gradually decreases with increasing percentage of sludge content.

As can be observed from Figure 4.3, water absorption increases with increase in sludge content because of increased number of pores in the brick mass resulting from burning of organic matter present in the sludge. As the sludge composition increases so does the percentage of water absorption. The higher the percentage of sludge added the higher the water absorption it becomes. As a result, the lower the quality of the bricks.

In other words, the increase in water absorption results in drastically decrease the quality of the brick. So that, the optimal sludge content that gives good results is the 10% fired at 900℃. Based on the results obtained by Baskar *et al.*, (2006) the addition of textile sludge up to 9% is effective enough to make brick material at a temperature of 800℃ and obtained a compressive strength of 3.48N/mm^2. Besides, according to Jahagirdar *et al*., (2013) the textile sludge content up to 15% is recommended to obtain 3.5 N/mm^2 exceeded values of compressive strength.

The water absorption for the control sample is as high as 10.47% and as low as 9.69 whereas for brick samples made with the textile sludge shows a significant increment to the lower level of 12.4 and higher level of 21.33%. Generally, the lower water absorption bricks are more durable than those with higher water absorption. This indicates, bricks with lower water absorption has the possibility of resisting the destructive environmental conditions and its effects. In this case, water absorption of bricks is good with maximum sludge proportion of 15% at all level of temperatures to satisfy the standard requirement which is less than 20%.

4.1.7.5 Effect of temperature on the quality of brick's

Unlike, the sludge proportion the compressive strength has found to be directly proportional with burning temperature. As can be noticed from the investigation the compressive strength has been increased with temperature holding the mixing proportion of the sludge constant. Nevertheless, the 20% sludge content fired at 1000℃ was cracked and deformed its shape exceptionally. The burning temperature of 900℃ is the optimum value at a time period of 3hrs which gives good results. About 70 to 80% increase in compressive strength is found when firing beyond 400℃ (Baskar *et al.*, 2006).

The burning process of bricks has a direct impact on the final product quality of the brick's material. In addition, the firing process essentially involves the oxidation of organic matter, transformation of inorganic components in to less harmful compounds and elimination of the pathogens that exist in the sludge so as to reduce public health impact. The mineral compositions are transformed to increase the durability and strength of the bricks because the durability and strength of the bricks are highly related to the mineral composition. During firing these minerals are fused and undergone possible chemical reactions forming complex compounds at high temperature. For this reason, firing temperature is an important factor that greatly affects the final bricks property which is mainly subjected to compressive strength and water absorption tests. The effect of temperature on compressive strength and water absorption is given in Figure 4.2 and 4.3.

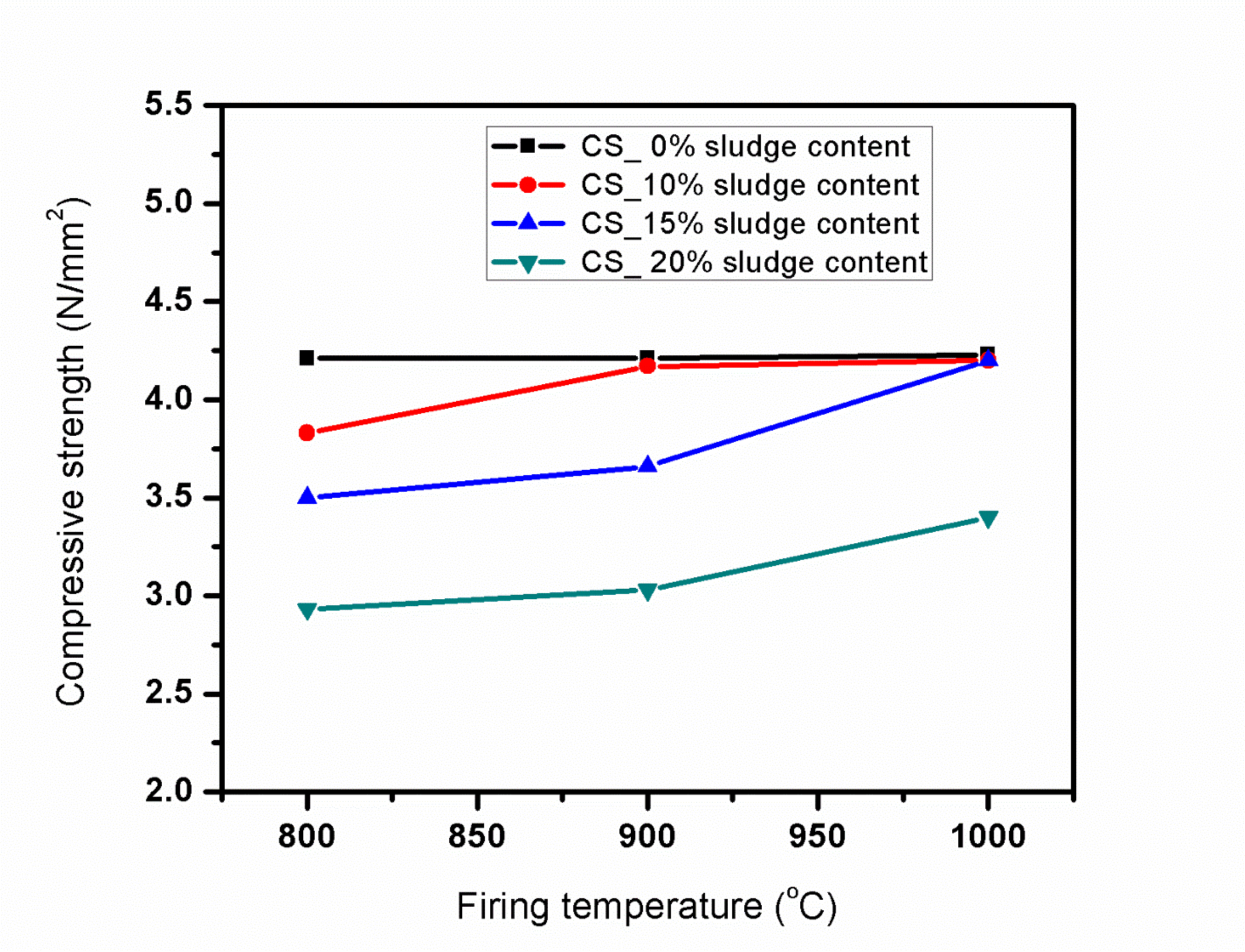

Figure 4.2 The effect of temperature on compressive strength

The relationship between firing temperature and compressive strength can be observed in Figure 4.2, Which shows that higher temperature leads to higher compressive strength. The compressive strength increases from 2.93 to 4.20 N/mm^2 as the temperature increased and sludge content decreased. More specifically, the compressive strength of 10% sludge content fired at higher temperature nearly reaches the compressive strength of the control bricks made by 0% sludge proportion fired at the same temperature. As firing temperature increases porosity sharply decreases. During the sintering process the atomic bonding of particles increase by the mechanism of diffusion creating a denser material. Conversely, this leads the brick to shrink progressively.

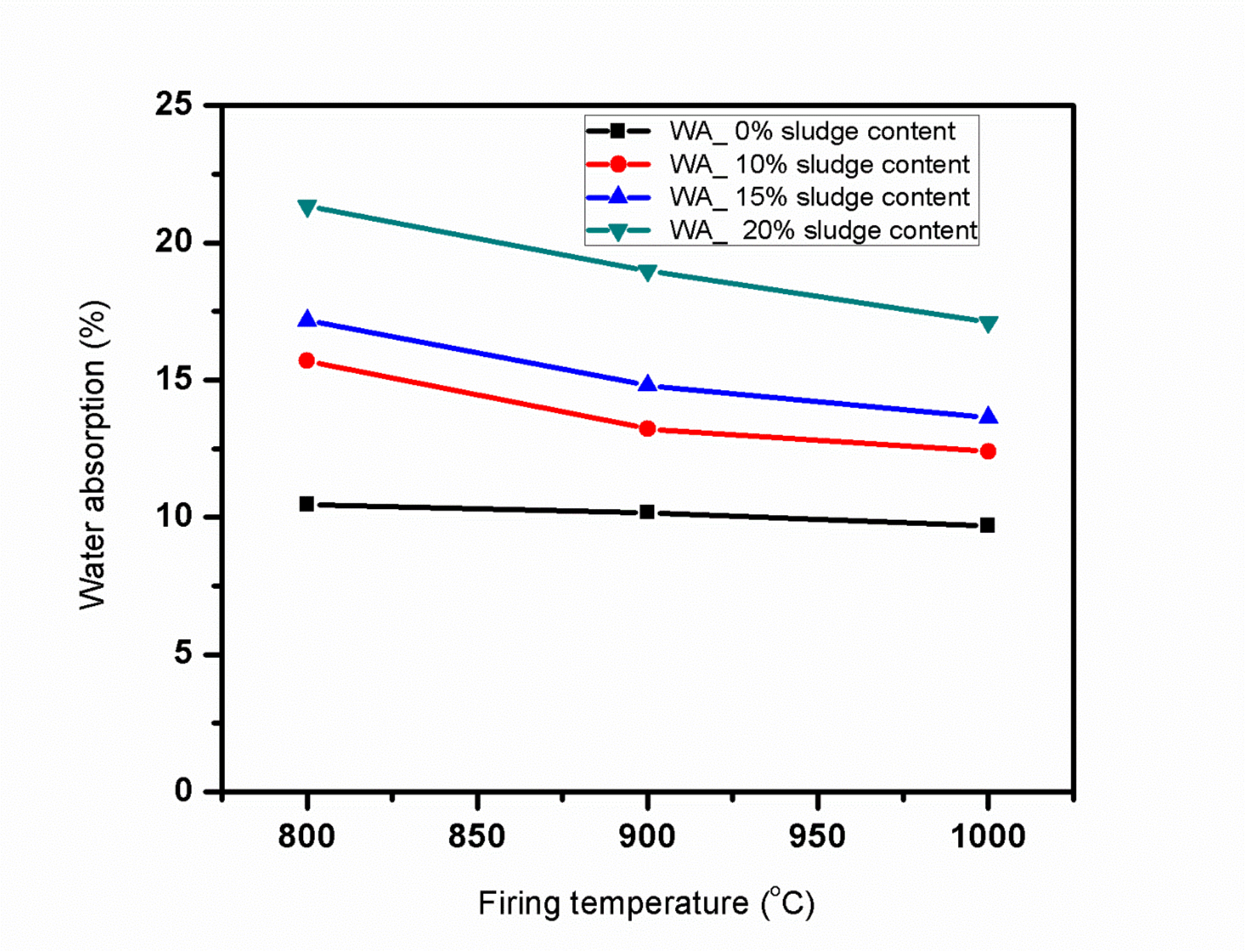

Figure 4.3 The effect of firing temperature on water absorption

Figure 4.3 shows the variation of water absorption graphs of bricks operating at the three levels of temperature and sludge values. The effect of firing temperature causes the water absorption to lower as it is getting greater. For this reason, the water absorption decreased from 21.33% to 12.4%. This is because as the temperature increased the organic matter eliminated and resulted in the durability of the bricks. In other words, the water absorption value at high temperature is more important brick than the low firing temperature brick.

The water absorption of bricks occurred at a higher temperature in between 900 and 1000°C gives good results. On the other hand, the water absorption of textile sludge-based bricks reached high value in a reduced range of temperature and, with a high content of sludge. Generally, the water absorption was heavily reduced as firing temperature increases and drastically increase as sludge content increases.

CHAPTER FIVE

5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The sludge discharged from currently evolving Ethiopian industrial parks treatment plant is the existing practical problem. In the study area, Hawassa industrial park, a significant quantity of sludge is generated that has a potential of negative impact on public health as well as the environment. This study addressed the issue through partial replacement of clay to develop a building material brick which is an alternative method of management and utilization of sludge.

This study was carried out in order to establish baseline sludge characterization data for recycling and management of the sludge to produce bricks. To do so, the physico- chemical characteristics including the heavy metals concentration and chemical composition in the textile sludge was determined using AAS and XRF analysis. The analysis revealed that it has high fraction of organic compounds and considerable quantity of heavy metals that have a potential of altering the environmental condition but lower than previously studied results. Similarly, the chemical composition result obtained indicates that the potential of textile sludge as partial replacement of building materials. Comparing the sludge chemical composition data with the clay composition more or less similar values was obtained. Ultimately, the influence of mixing proportion at 10%, 15% and 20% of the sludge and at temperature of 800℃, 900℃ and 1000℃ have been performed to assess the quality of the brick. Then, the brick was examined for compressive strength, water absorption and leachate analysis tests and it was found that, the compressive strength drops gradually as sludge proportion increases and increases progressively with temperature.

Consequently, a good compressive strength result can be achieved at higher temperature and at minimum sludge proportion. Hence, the sludge proportion as much as 15% and less are effective to produce good brick material with a corresponding compressive strength of 3.5 and above. In addition, the addition of sludge content has an effect on the water absorption to rise gradually. Finally, from the leachability test results it can be concluded that the addition of textile sludge up to 20% composition into clay to produce bricks is possible and the heavy metals concentration were reduced significantly when incorporated into fired clay bricks. Therefore, textile sludge could be used as an alternative raw material for the production of bricks but recommended with quite small quantity which is up to 15%.

5.2 Recommendations

From the result of the studied research, partial substitution of clay with textile sludge for making bricks is possible and the following recommendations are raised for the production of building materials in the future.

- ✓ Further research to convert this waste to make other building materials or a means to increase the percentage of sludge by adding other mixtures.
- ✓ Further studies on mixing proportion of the sludge with the clay to increase its durability.
- ✓ Further investigation on the characterization and chemical composition of the sludge.
- ✓ Economic analysis of the sludge amended bricks.
- ✓ Further research needs to be carried out to investigate the leachate analysis of the sludge-based bricks over a long period of time.

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APPENDICES

Appendix A: General Information About Analysis Procedures

A.1 Chemical Analysis Procedures to Determine the Major Oxides

To analyze the chemical composition of the major oxides the chemical analysis method was followed the following procedures.

First, 2.5g of $NaKCO_3$ (fusion mixture) and 0.5g sample were measured in a platinum crucible and the surface of the mix was Covered by 0.5g of $NaKCO_3$. Then, the covered mixture has been placed in a maffle furnace at a temperature of 1000℃. After 20 minutes of burning time it was removed from the furnace and gently swirled in order to spread the melt to the sides of the crucible. Next, it was placed on a porcelain basin and holden it in about 50ml cold distilled water of the basin.

Thereafter, the crucible and the lid were removed from the basin and placed in a clean dry porcelain dish. Then, the fuse was loosened with about 20ml of concentrated HCl and rinsed with distilled water. Subsequently, the contents of the dish were evaporated on sand bath to dryness and washed with another 20ml concentrated HCl while it was dried and stirred until the water evaporates.

Again, it was then Placed in sand bath for dryness and baked for an hour in a drying oven at 105℃ respectively. Afterwards, the oven dried sample was cooled and 20ml of 1+1 HCl was added. Latterly, it was digested for 10 minutes and the contents was filtered in 500 ml volumetric flask while hot with coarse filter paper (blank band). Then, it was washed repeatedly with hot water, cooled and filled up to the mark. Eventually, the precipitate was used for SiO_2 analysis, while the filtrate was used for CaO, Al_2O_3, Fe_2O_3 and MgO determination.

Determination of Silica (SiO2)

- Place the precipitate obtained in a previously weighed platinum crucible (W1)
- Dry, char (until the carbon from the filter paper is completely consumed without inflaming) and ignite in muffle furnace at 975 ± 25℃ for an hour.
- Cool in a desiccator and check for a constant mass (W_2)
- Place the precipitate obtained in a previously weighed platinum crucible (W1)
- Dry, char (until the carbon from the filter paper is completely consumed without inflaming) and ignite in muffle furnace at 975 ± 25℃ for an hour.
- Cool in a desiccator and check for a constant mass (W_2)
- Add about 0.5 – 1 ml of distilled water and 0.5 – 1 ml 1+1 H_2SO_4 (5 drops of water, 5 drops of sulfuric acid) [add water, H_2SO_4 and HF cautiously evaporated to dryness].
- Evaporate the content at low heat to dryness (use sand bath or hot plate)
- Continue the crucible with the evaporation residue in an electric furnace 1175 ± 25℃ for 20 minutes.
- Leave to cool to room temperature in a desiccator and weigh (W_3)
- If the residue obtained exceeds 0.5% start the analysis and use Na_2O_2 for decomposition. The difference between first and second weight is weight of SiO2.

% pure SiO2 = $(w_2 – w_1) – (w_3 - w_1)$ x200 while %HF residue = $(w_3 - w_1)$ x200

Determination of the major oxides

Consider the filtrate obtained above inside the 500ml volumetric flask

For CaO:

- Pipette out 25ml of the filtrate in 300ml Erlenmeyer flask
- Dilute to about 100ml with distilled water
- Add 13ml of 30% Tri ethanol amine
- Adjust the pH between 12 & 13 with 10% KOH (with out formation of precipitate and check it by pH meter)
- Add about 50mg of the mixed fluorexone indicator
- Titrate with 0.01 M EDTA from light green to pink

Expression of results

% CaO = 2.2432 x F_{EDTA} x V_{EDTA}

Where, F_{EDTA} = is factor of EDTA and V_{EDTA} = the volume of EDTA used to end point

For MgO

- Pipette out 25ml of the filtrate
- Dilute to about 100ml with distilled water
- Add 10 ml of 30% Tri ethanol amine (T.E.A)
- Adjust the pH between 10 & 11 with pH = 10 buffer
- Add 5 drops of par and 5 drops of copper complex mate
- Titrate with 0.01 M EDTA from red to orange yellow

Expression of results

% MgO = 1.6128 x F_{EDTA} x V_{EDTA}

For Fe_2O_3

- Pipette out 25ml of the filtrate
- Add Sulpho – Salicilic indicator about 50mg
- Add drop wise 1+4 NH4OH solution till the yellow color is observed
- Return back the color to violet by adding 1+4 HCl solution

- Check the pH of the solution to be between 1 & 2
- Heat gently to body temperature, do not exceed 50℃
- Titrate with 0.01 M EDTA from violet to colorless

Expression of results

% $\mathbf{Fe_2O_3}$= 0.7985 x F_{EDTA} x V_{EDTA}

For Al_2O_3

- Pipette out 100 ml of the filtrate
- Take the volume of EDTA consumed for Fe_2O_3
- Add 15 – 20 ml of pH = 3 buffer
- Heat just to boiling
- Add exactly 3 drops of copper complex mate
- Add 10 drops of PAN indicator
- Boil and titrate while hot form red – violet to yellow
- Repeat boiling and titrating till yellow color is stable

% $\mathbf{Al_2O_3}$ = 0.5098 x F_{EDTA} x V_{EDTA}

Determination of Loss on Ignition

Procedure

- Weigh 1g of the sample in to a crucible which has been previously ignited and tarred
- Place the covered crucible in the electric furnace controlled at 1000℃
- After heating for 5 min remove the lid and leave the crucible in furnace for further 20 min.(for an hour)
- Cool the crucible to room temperature in desiccator
- Weigh and determine the mass

Expression of results

The observed LOI is calculated in % from the formula (A - B) x100, where,

A = the mass of crucible and sample before ignition
B = the mass of crucible and sample after ignition

Sulphates (SO_3) determination

Procedures:

- Measure 1g of dry textile sludge and place it in a beaker
- Add 25 ml of distilled water and 13 ml of concentrated HCl
- Heat to boiling
- Add 100 ml of boiled distilled water
- Filter immediately with a medium (red – band) filter paper and wash it several times with hot distilled water.
- The filtrate is used for SO_3 analysis
- Heat the filtrate to boiling, while placing a glass rod and piece of filter paper in it
- Add 10 ml of $BaCl_2$ solution slowly
- Remove from the hot plate and stir very well
- Add 50 – 60 ml of Congo red carefully and stir very well
- Leave it for some minutes 20 – 25 for settlement. Clear upper layer shows enough amount of Congo red is added, otherwise additional amount might be needed.
- Filter with fine (blue – band) filter paper after the settlement
- Wash the residue several times with hot water
- Place the precipitate in a previously weighed porcelain crucibles
- Place the crucibles on one of the holes of a rapid incinerator or on a hot plate until the paper blacken
- Ignite in a muffle furnace controlled at 1000℃ for an hour.
- Cool in a desiccator and weigh

Remember: wash the precipitate several times with hot distilled water until it is free from Cl^- ions. Check it with the $AgNO_3$ test.

Expression of results

The sulphate content, expressed as SO_3 is calculated in percent from the formula.

$\% \, SO_3 = \frac{(A - B) x \, 0.343}{W} \, x \, 100$, where, A = weight of crucible and the precipitate

B = weight of empty crucible

C = weight of the sample

0.343 factor between BaSO4 and SO_3 i.e. $\frac{\mathrm{SO3}}{\mathrm{BaSO4}} = \frac{80\ g/mol}{233.33\ g/mol} = 0.343$

A.2 Detailed procedure to determine the physico – chemical characteristics

pH value determination

One to five dilution factors of the textile sludge to distilled water was used and continuously stirred the solution for one hour in a shaker and then it was allowed to settle and the supernatant was used for the pH measurements (H. Patel & Pandey, 2008). The pH of the textile sludge was determined using digital pH meter. The experimental determination of pH was performed in Hawassa industrial park zero liquid discharge facility laboratory section.

Calorific Value Determination

The calorific value of textile sludge was determined from the dried sludge at 105°C and then was measured using an adiabatic oxygen Bomb calorimeter (1241EF, PARR MOLINE ILLINOISE, USA) according to (H. Patel & Pandey, 2008). The experiment was performed at Geological Survey of Ethiopia, Hydrocarbon Laboratory Analysis section, Addis Ababa.

More importantly, the procedure for calorific value determination of the sludge using bomb calorimeter can be described in the following manner. The dried sludge used for calorific value analysis has to be finely grinded. It was prepared using a sieve of 60 mesh. Then, 1 g of sample was taken in a crucible and made into a pellet, and the initial weight was noted. It was placed in the bomb, which was pressurized to 18.2385 bar of oxygen. The bomb was placed in a vessel containing 2000 g of water. The ignition circuit was connected and the water temperature noted. After ignition a temperature rise was noted every minute till a constant temperature was reached. The pressure was released and the length of unburned fuse wire was measured. Benzoic acid was used to standardize the calorimeter. The calorific value of the sample is then calculated as shown in equation (1):

$$CV = [(E \times \Delta T) - e_1 - e_2] / g \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Where: CV = calorific value, Btu/lb.

E = energy equivalent, Btu/°F**.** This is a constant factor of benzoic acid equivalent to 2420.

T = corrected temperature rise,℃; e_1 = correction for heat of combustion of firing wire, Btu which is equivalent to 20.9; e_2 = correction for the heat of formation titrant, Cal, equivalent to the volume. g = weight of sample, g

Moisture content determination

The moisture content was determined by weighing 1 kg and 1.2 kg of textile sludge samples into a pre-weighed aluminum foil covered plate and were dried in an oven to drive off water in the sample. The sample containing plates were removed and kept in a room temperature to cool, then the final sample weight was measured. Finally, the moisture content was conducted by taking the difference of the sample in weight before and after drying. The procedures to determine the moisture content of textile sludge were replicated three times and the average values were taken.

It was calculated as a percentage using the following formula as indicated in equation (2).

Moisture content (%) = $\frac{W1-W2}{W1} * 100$ …………………………………………………………. (2)

Where, W$_1$ = weight of wet sludge sample

W$_2$ = final weight of dry weight sample

Volatile Organic Matter Determination

The amount of VOC was determined by igniting the oven dried textile sludge sample at 950℃ in a muffle furnace for 7 minutes (ASTM D 3175). After digestion, the dishes were removed and kept in a desiccator for about 30 minutes. and finally, taking the difference in weight of before and after ignition. The percentage of VOM is determined by the following equation as shown below.

VOM (%) = $\frac{DS - AS}{DS} * 100\%$ ………………………………………………………………… (3)

Where, DS: Dry sample weight

AS: Ash sample weight

Ash content determination

It was determined from the oven dried sample. First, the residues were cooled and weighed. Then, a dry porcelain crucible containing 2g sample was placed in a muffle furnace (Hersbruck, Model VMK 72 158, Germany), set at 550℃ for 4hr and by allowing to cool in a desiccator and weighing it. Ash content of sludge is defined as a portion of residue remained after the sludge is burnt, which represents the non-combustible materials or the natural substances after carbon, oxygen, sulfur and water. The ash content was determined by AOAC (2000) using the official method 923.03 and applying a simple formula:

Ash content (%) = $\frac{(Wt\ of\ Ash + Crucible\ in\ g) - (Wt\ of\ empty\ Crucible\ in\ g)}{Wt\ of\ sample\ in\ g}$ x 100 ……………………(4)

Fixed carbon content

Fixed carbon is defined by carbon found in the material which is left after volatile test. Fixed carbon is determined by removing the mass of volatile from the original mass of the sample (Kalanatarifard, Yang, & Korea, 2012).

It is calculated using the following formula:

Fixed carbon content = 100 – (Ac(%)+ VM(%)) ……………………………………………….. (5)

Where, Ac is ash content and VM is volatile matter respectively.

COD determination

The procedure used to determine the COD of the sludge was carried out as per the standards in the following manner. First, the COD reactor was Preheated for 30 minutes. Then, 2ml volume of sample was added into a COD reagent (CSB 4000) contained teste tube. Soon after that, the test tubes was closed and the sample was shaken to mix the content. Thereafter, the temperature and time of the COD reactor were set to 150℃ and 2hrs respectively. Then, the solution was put in the COD reactor at the specified temperature and time. Take the tubes out from the reactor after oxidation of the sample for about two hours and was allowed to cool to room temperature for 10 minutes. Eventually, the amount of COD was measured using spectrophotometer.

BOD determination

The BOD of the textile sludge was determined by preparing a solution which involves dissolving the textile sludge into distilled water in (1:5 w/v proportion) according to the expected BOD5 ranges. After adjusting the pH of the solution into neutral condition between 6.5 to 7.5 (APHA 5210 A) using H_2SO_4 and NaOH. It was then poured into a 300ml BOD bottle and mixed well and added some analytical grade reagents to the sample. The reagents are 3 drop of nitrification inhibitor and 3 - 4 drop of potassium hydroxide (KOH) solution into the seal gasket and insert gasket in the neck of the bottle, screw the BOD sensors to the sample bottle and then place the bottle in the bottle rack or automatically inserted to BOD apparatus for successive 5 days of incubation period. During the five-day period of a BOD test @ 20℃, the micro organism oxidizes mainly the soluble organic matter present in the water.

Heavy metal analysis determination

To do so, the Organic matrix was destroyed by ashing in muffle furnace. Remaining ash was dissolved in diluted acid or 5mL 1N $HNO_{3,}$ then, warming the solution on hot plate 2.5 min to aid in solution. Next to this, the solution was added to 50mL volumetric flask and repeat with 2 additional portions of 1N HNO_3. Lastly, it was Diluted to 50mL with 1N HNO_3. After digestion, the solution was filtered via 0.45μm filter paper and the analyte were determined by different principles of determination. Total Calcium and Magnesium were determined by EDTA titration which forms a complex with calcium and magnesium ions.

Compressive strength determination

To do this, the Compression Testing Machine was used. All the brick samples produced with various proportions of textile sludge were subjected to a Compressive strength testing machine to obtain the compressive strength result, and the average strengths were obtained. To sum up, the compressive strength test was made by applying a uniform load to the bricks till the breakage of the brick occurs. The reading value obtained from the testing machine were recorded.

Table 1 Compressive Strength calculated value from the load and dimension of the bricks

Mix prop (%)	**T(℃)**	**L(cm)**	**W(cm)**	**A (cm^2)**	**LOAD (kN)**	**COMPRESSIVE STRENGTH(N/mm^2)**
10	800	10	9.5	95	36.45	3.83
10	900	10	9.5	95	39.70	4.17
10	1000	10	9.5	95	39.90	4.20
15	800	9	8.5	76.5	27	3.53
15	900	9.5	8.5	80.75	29.62	3.66
15	1000	9	8	72	28.61	3.97
20	800	9.5	8	76	22.3	2.93
20	900	9	9.2	82.8	25.12	3.03
20	1000	8.5	9	76.5	26.08	3.40

Water absorption determination

The procedure used to test the water absorption are described as follows. First, brick specimens were weighed dry. Then, they were immersed in water for a period of 24 hours and the specimens were taken out and wiped with cloth. The weight of each specimen in wet condition was measured. Ultimately, the percentage water absorption is the ratio of water absorbed to the dry weight multiplied by 100.

Table 2 Water absorption calculated values from weight of the bricks before and after immersion

Mix prop (%)	**T(℃)**	Weight before WA(g)	Weight after WA(g)	Percentage of WA (%)
10	800	130.5	151	15.70
10	900	131.6	149	13.22
10	1000	129	145	12.40
15	800	127	148.8	17.17
15	900	131	150.4	14.81

15	1000	129.7	147.39	13.64
20	800	132.7	161	21.33
20	900	130	154.68	18.98
20	1000	129.8	152	17.10

Appendix B: Laboratory work photographic images

Figure A1. Sample collection area

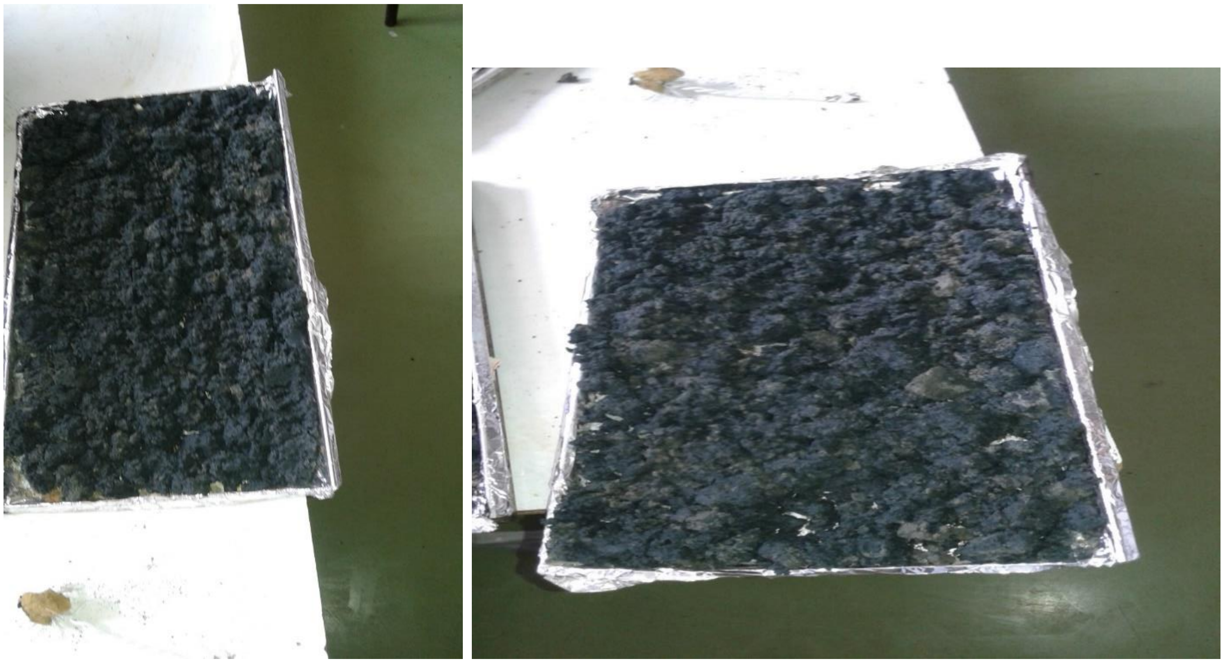

Figure A2. Collected wet sludge sample before drying in oven

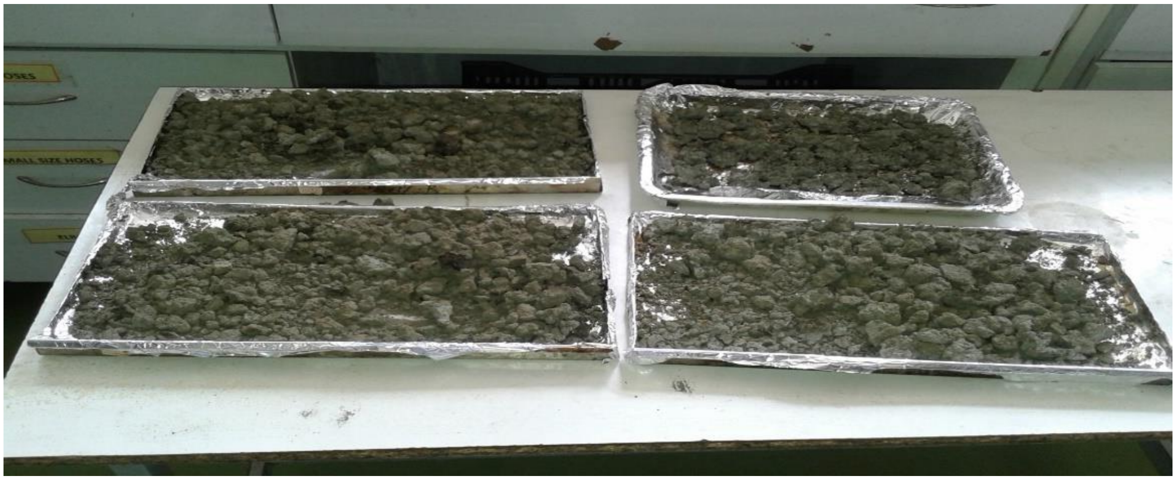

Figure A3. Oven dried textile sludge

Figure A4. Oven dried textile sludge sample was crushed and sieved respectively

(a) (b) (c)

FigureA5.Brick moulding (a) during air drying in the lab room (b) bricks after fired in a furnace (c)

(a) (b)

Figure A6. Compressive Strength test (a) Water Absorption test (b)