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ADDIS ABABA INSTITUTE OF TECHNOLOGY
SCHOOL OF GRADUATE STUDIES
CHEMICAL AND BIO ENGINEERING DEPARTMENT

**PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS A
PARTIAL SUBSTITUTE FOR CLAY**

By: -Haregewoin Nida

A Thesis Submitted to the School of Graduate Studies of
Addis Ababa Institute of Technology in Partial Fulfilment of the
Requirements for the Degree of Master of Science in Chemical Engineering
(Process Engineering)

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October, 2021
Addis Ababa, Ethiopia

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October, 2021

ADDIS ABABA, ETHIOPIA

DECLARATION

I hereby declare that the thesis work which is entitled as “Production of Eco Bricks Using Tannery Sludge as a Partial Substitute for Clay” which is submitted to School of Chemical and Bio Engineering, Addis Ababa Institute of Technology, Addis Ababa University in partial fulfilment of the requirements for the Degree of Master of Science in Process Engineering, complies with the regulations of the university and meets the accepted standards with respect to originality and quality.

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Date: _____

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

Advisor:

Signature: _____

Date: _____

Place of Submission: School of Graduate Studies, Addis Ababa University, Addis Ababa, Ethiopia. October, 2021.

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ABSTRACT

Tannery sludge is an unavoidable by-product of waste water treatment plants of leather industries and with no safe disposal options; it has the potential to become a significant environmental burden for Ethiopia in the future. Presently, the sludge generated from the leather industries will be land filled without any kind of treatment. Because sludge contains a high concentration of organic and inorganic components, including heavy metals, its accumulation is a burden on the industry and has negative consequences for the environment and human health. As a result, an alternative sludge disposal strategy is required.

The major goal of this research was to see if using TS with certain proportions in the manufacturing of bricks may be a viable alternative to natural clay.

In laboratory-controlled conditions, clay bricks were made with various proportions of sludge (10%, 15%, 20%, and 25% by weight) with firing temperature of 800°C, 900°C and 1000°C and its potential as a construction material was evaluated based on its Compressive strength, water absorption, weight loss on ignition, bulk density, firing shrinkage, electrical conductivity and heavy metal leaching properties.

As a result, increasing the sludge content in bricks resulted in a decrease in compressive strength and increase in water absorption. When TS content was increased from 0% to 25%, the compressive strength of TS bricks decreased significantly, from 30.24 MPa to 20.15 MPa. Water absorption increased from 7.03% to 16.03% when sludge proportion was increased from 0% to 25% at firing temperature of 1000°C.

In addition, total shrinkage and weight loss in burning were compared to raw brick, revealing that total shrinkage did not differ significantly, while weight loss in burning varied from 12.25 % to 30.8 % in raw brick to 25% TS brick at firing temperature of 1000°C.

Furthermore, the sludge altered up to a 15% mixing ratio demonstrates minimal heavy metal leachability as analyzed by TCLP and is compatible with the USEPA standard. As a result of the research, tannery sludge has the potential to be used in the production of sludge bricks with sludge proportion of 10% to 15% and firing temperature of 900°C to 1000°C.

TABLE OF CONTENTS

DECLARATIONII

ACKNOWLEDGEMENT III

ABSTRACT..... IV

LIST OF FIGURES VIII

LIST OF TABLES IX

LIST OF ABBREVIATIONS..... X

CHAPTER ONE 1

Introduction..... 1

 1.1 Background of the Study 1

 1.2 Statement of the Problem 4

 1.3 Objectives..... 5

 1.3.1 General Objective 5

 1.3.2 Specific Objectives 5

 1.4 Significance of the Study 5

 1.5 Scope of the Study..... 6

CHAPTER TWO 7

Literature Review..... 7

 2.1 Background of the Tannery Sludge..... 7

 2.1.1 Overview of the Tannery 7

 2.1.2 Definition of Sludge..... 9

 2.1.3 Generation of Sludge 10

 2.1.4 Characteristics of Tannery Sludge 10

 2.2 Sustainable Disposal and Reuse of Tannery Sludge 11

2.2.1	Solidification and Stabilisation.....	12
2.3	Review of Previous Studies on Sludge Stabilization Using Fired Clay Bricks	13
CHAPTER THREE		16
Materials and Methods.....		16
3.1	Materials.....	16
3.2	Methods.....	17
3.2.1	Collection of Samples.....	17
3.2.2	Study Location.....	17
3.2.3	Characterization of the Raw Material.....	17
3.2.4	Preparation of Sample.....	20
3.2.5	Characterization of Clay – Sludge Mixture	21
3.2.6	Making of Brick.....	22
3.2.7	Quality Characterization of the Final Product	23
CHAPTER FOUR.....		27
Result and Discussion.....		27
4.1	Characteristics of Raw Material.....	27
4.1.1	pH Value, Moisture Content, Ash Content, and Organic Content.....	27
4.1.2	Heavy Metal Content	28
4.2	Characterization of Clay – Sludge Mixture.....	29
4.2.1	Atterberg Limit Tests.....	29
4.2.2	Compaction Test.....	31
4.3	Quality Characterization of the Final Product.....	32
4.3.1	Compressive Strength of Bricks	32
4.3.2	Water Absorption of Bricks	33

4.3.3	Firing Shrinkage.....	35
4.3.4	Weight Loss on Ignition.....	36
4.3.5	Bulk Density	37
4.3.6	Soluble Salt Content and Electrical Conductivity of Bricks.....	38
4.3.7	Leaching Test of Bricks	39
CHAPTER FIVE		41
Conclusion and Recommendation		41
5.1	Conclusion.....	41
5.2	Recommendation.....	43
REFERENCES		44
APPENDICES		49

LIST OF FIGURES

Figure 1: Schematic diagram of Polluting waste resulting from various phases of cattle hide processing 9

Figure 2: Sample Collection 17

Figure 3: Flow Chart of the Total Process 18

Figure 4: Air Dried Samples 20

Figure 5: Oven Dried, Grinded and Sieved Samples 21

Figure 6: Result of sludge addition on OMC and Dry Density 32

Figure 7: Compressive Strength of Brick at Different Clay-Sludge Ratio and Firing Temperature 33

Figure 8: Water Absorption of Brick at Different Clay-Sludge Ratio and Firing Temperature..... 35

Figure 9: Firing Shrinkage of Brick at Different Clay-Sludge Ratio and Firing Temperature..... 36

Figure 10: Weight Loss on Ignition of Brick at Different Clay-Sludge Ratio and Firing Temperature 37

Figure 11: Bulk Density of Brick at Different Clay-Sludge Ratio and Firing Temperature 38

LIST OF TABLES

Table 1: List of Chemicals and Materials 16

Table 2: Composition of Raw Materials by Percent and Mass..... 23

Table 3: Characteristics of the Sludge and Clay Sample 27

Table 4: Concentration of Heavy Metals in Sludge and Standards for Sludge Utilization 28

Table 5: Plasticity Index (PI) and Properties of Soil 29

Table 6: Effect of Moisture Content on Sludge Clay Mixture Liquid Limit, Plastic Limit, and Plasticity Index..... 30

Table 7: Effect of Moisture Content on OMC and Dry Density of Sludge Clay Mixture..... 31

Table 8: Ethiopian Standard for Fired Clay Bricks (ECAE) 34

Table 9: Electrical Conductivity and Soluble Salt Content of Sludge and TS Bricks 39

Table 10: Leaching Test of Tannery Sludge Bricks Fired at 1000°C..... 40

LIST OF ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Standard of Testing and Materials
CETP	Central Effluent Treatment Plant
CSA	Central Statistics Agency
ETP	Effluent Treatment Plant
GFAAS	Graphite furnace Atomic Absorption Spectrophotometer
ICP-OES	Inductively Coupled Plasma Optical Emission
IPPC	Integrated Pollution Prevention and Control
LIDI	Leather Industry Development Institute
LL	Liquid Limit
LOI	Loss of Ignition
OMC	Optimum Moisture Content
PI	Plasticity Index
PL	Plastic Limit
SLTC	Society of leather technologists and chemists
SS	Suspended Solids
S/S	Solidification or Stabilization
TCLP	Toxicity Characteristics Leaching Procedure
TDS	Total Dissolved Solids
TS	Tannery Sludge
UNEP	United Nations Environment Program
UNIDO	United Nations Industrial Development Organizations
USEPA	United States Environmental Protection Agency
WA	Water Absorption

CHAPTER ONE

Introduction

1.1 Background of the Study

The leather industry is one of the world's most polluting industries. The processing of leather is to blame for the unfavorable impacts on the environment [1, 2]. The leather industry generally uses hides and skins as raw materials, which are the by-products of the meat processing industry. In this respect, the leather industry could have easily been distinguished as an environmentally friendly industry; since it processes waste products from meat production [1]. However, the tanning industry has been categorized as one highly polluting because different chemicals have been used during the tanning process and different solid, gaseous, and high amounts of liquid wastes are generated which harm the environment [3].

The leather manufacturing sector in Ethiopia is one of the priority sectors due to the availability of resources for leather manufacturing, its potential for the export market, for generating massive employment opportunities [4]. The resource endowment of the country illustrates the considerable potential of the country in the leather industry [5]. In 2012, in Ethiopia, there were 26 tanneries in operation and most of them are located in the vicinity of Addis Ababa.

Annually, all leather industries put together use 2.3million pieces of hides and 44.3 million pieces of skins as an input for processing at full capacity operations [4]. The leather industry in developing countries is facing lots of solid wastes problems. Out of 1000kg of raw hide, nearly 700-750kg is generated as solid wastes in leather processing [6]. Raw hides and skins are processed into leather in tanneries through a number of chemical and mechanical operations that lead to the discharge of hazardous chemicals into effluent treatment plants (ETP) [7].

In the leather industries, chromium is used in the processing of about 80-90% of the hides. The most often used tanning material for converting putrescible collagen fibers into a non-putrescible leather matrix is basic chromium sulphate which also protects the leather from the environmental effect such as heat, moisture, and microbial degradation because of the excellent property of chromium salt for tanning [8, 9].

About 60% of the chromium salts react with the source materials, with the remainder remaining in the exhaust tanning bath and being discharged into the wastewater [10, 11]. Chemical precipitation with alum, lime, or ferric chloride is used to remove dissolved chromium and other types of wasted chemicals from wastewater, such as sulfide, salt, proteins, polyphenolic compounds, surfactants, dyes, syntans, and so on. The chromium that has precipitated, as well as other organic substances, is released as sludge [12, 13].

As a result of the tanning process, TS contains high amount of heavy metals such as Cr, As, Pb, Ni, Cu, Zn, and Cd. In the tanning process, basic chromium salt, various syntans, dyes, pigments, retanning agents, and other substances are used [14]. Due to their inability to biodegrade, prolonged biological half-lives, and capacity to accumulate in biological systems, these heavy metals are extremely dangerous [15, 16]. If not properly disposed of, chromium-rich sludge has the ability to pollute soil, surface water, and groundwater through produced leachate, posing a hazard to the environment and natural resources [17].

Sludge is a severe concern in many nations because of its high treatment costs and the risks it poses to the environment and human health. Because of the shortage of acceptable disposal locations, rising labor costs, and environmental concerns, sludge is becoming an increasingly difficult challenge for towns of all sizes [18].

Moreover, in Ethiopia, most of the tanneries do not have treatment facilities and neither environmental management systems. Thus, they simply discharge their wastes into the environment together with other solid municipal wastes and some tanneries are using the nearby river for dumping their solid wastes because there is no designated site for the disposal of hazardous waste causing critic environmental and health problems in specific areas (including urban and rural areas) [19, 20].

To meet the demands of a growing population, the volume of waste generated by daily activities, production, and industry continues to rise significantly. Furthermore, environmental rules are becoming increasingly stringent [21].

Therefore, alternate techniques to manage and utilise wastes have to be determined [21]. Improper disposal of these leather wastes causes environmental pollution; therefore, properly optimized utilization of these wastes into valuable end products will be a promising solution [6].

Environmentally friendly waste recycling has been one of the extremely important research subjects for many decades. Various recycling technologies have been utilized to convert sludge into valuable products, including fertilizer, adsorbents, cement, brick, and others, as a countermeasure for sludge disposal problems. Of these proposed technologies, the production of bricks for construction material can be a useful option for sludge recycling, because the thermal process involved in producing bricks from sludge immobilizes toxic and hazardous materials by heat treatment [22, 21].

The use of waste such as sludge in clay bricks lowers the negative consequences of waste disposal while also improving qualities such as lightweight bricks with increased shrinkage, porosity, thermal characteristics, and strength [21]. The lightweight bricks will lower the amount of time it takes to deliver them and manufacturing costs. Furthermore, by incorporating waste, the clay content of burnt clay bricks will be reduced, lowering manufacturing costs and making construction more affordable [18].

Because of its extensive qualities, brick has been one of the most commonly utilized conventional construction materials all over the world from old period [23, 21]. The earliest sun-dried clay bricks were discovered 8000 years ago, and the history of brick making dates back to that time [24, 25]. It is long-lasting and has evolved over time. It remains technically and economically competitive with alternative structure and field systems.

Clay, lime, sand/flint, concrete, and natural stone are some of the materials used to make bricks. Fired clay bricks are made by molding appropriate clay into standard-size blocks [26]. Apart from soft slate, clayey soils, and shale, which are primarily extracted from open pits with the resultant disruption of drainage, flora, and wildlife habitat, clay is the main raw material for bricks [21]. The strength, fire resistance, durability, attractiveness, and excellent bond and performance with mortar are the main characteristics of bricks that make them superior building units.

Furthermore, bricks do not cause indoor air quality problems. The thermal mass effect of brick masonry can be a useful component for fuel-saving, natural heating, and cooling strategies such as solar heating and night time cooling [21, 24]. They have moderate insulating characteristics, therefore brick houses are cooler in the summer and warmer in the winter than houses made of other materials. Clay bricks are also non-combustible and conduct electricity poorly [2, 13].

For the past thousands of years, the bricks were made out of clay. Due to their ceramic properties, clays are highly regarded as a raw material for clay bricks [21]. The sludge from waste treatment plant is almost similar to brick's clay. Its chemical composition is also similar. Therefore, this sludge can be used efficiently as a replacement for clay brick [1, 27].

1.2 Statement of the Problem

In the treatment process, approximately 100–150 kg of sludge is produced per ton of hides/skins treated [28], which is primarily composed of chemically precipitated dissolved chromium, various types of spent chemicals, surfactants, dyes, syntans and others [29]. The improper management of tannery sludge is one of the environmental challenges facing urban cities worldwide with particular emphasis on developing countries [19].

There is no secure landfill in Ethiopia, solid wastes generated from leather manufacturing industries dumped into an open landfill site, no attention is given to waste management practices as heaps of wastes are dumped indiscriminately in open dumps space [30]. This has contributed not only to the spread of communicable diseases in the affected areas; it has also resulted in flooding and the emission of greenhouse gases [31].

Ultimately, these harmful metals can eventually find their way into the human body, causing bioaccumulation and biomagnification. Excessive heavy metal deposition in agricultural soils due to wastewater irrigation and sludge disposal may cause soil contamination as well as food quality and safety issues [31, 32]. Besides, some of the tanneries are burning it in the surrounding area to minimize the volume of wastes and transportation cost this in turn, causes the oxidation of Cr (III) to Cr (VI) which causes a carcinogenic effect to the human body through the food chain [33].

Consequently, the tanning industry demands more research, innovative and practical solutions to ensure its sustainable development. As industrialization progresses, the environmental problem becomes more noticeable. For that reason, it becomes very important to enhance the protection of the environment in the initial step of the industrialization phases. In order to mitigate these problems, remediation techniques need to be implemented; these wastes can be recovered and processed to produce value added products. Accordingly, this study aims to use tannery sludge as a partial substitution for preparation of bricks.

1.3 Objectives

1.3.1 General Objective

The main objective of this study was to evaluate the partial substitution of clay with tannery sludge in the production of eco bricks.

1.3.2 Specific Objectives

- To characterize the raw materials.
- To provide an optimal clay-sludge mix proportion for brick manufacturing paving the way towards environment-friendly recycling of toxic tannery sludge.
- To see how sludge (in combination with clay) affects the strength and other physiochemical properties of brick.

1.4 Significance of the Study

In this study tannery sludge was used to provide alternative ways to manage wastes and minimize the number of wastes dump into the environment. Other researchers will use the baseline data for further study. From a society point of view, this process provides a clean environment around the tanneries. This tannery sludge recycling technique will markedly reduce environmental pollution; public health problems associated with improper tannery waste management, and create a great opportunity to establish an industry that produces all value-added products on an industrial scale whereby the country can conserve its environment. Therefore, this research helps to bring a clean environment by making a value-added product.

In addition to this, the public image of the tanning industry will be enhanced and the competition of sectors in the world market will be increased. Also, the demand gap on the construction materials will be minimized.

1.5 Scope of the Study

The scope of the research is limited to tannery sludge characterization; investigate the possibility of brick production from tannery sludge mixed with clay and characterization of the final product. This study doesn't consider other sludge sources, rather than tannery sludge from wastewater treatment plants.

CHAPTER TWO

Literature Review

2.1 Background of the Tannery Sludge

2.1.1 Overview of the Tannery

The variety of leather that can be produced is reflected in the variety and complexity of processes required. However, the vast majority of hides and skins are chrome tanned in rotating process vessels. Processing is by batch and the size of batches is governed by the capacity of processing vessels. A large amount of chemicals are used during the tanning processes. The main chemicals will be: Lime, Sodium Bisulphite, Sodium Sulphide, Ammonium Sulphate, Sodium Chloride, Calcium Formate, Sulphuric Acid, Sodium Carbonate, Bating Agent, Bactericides, Vegetable Tans and Chrome Salts. [34]

The production operation in a tannery can be divided into four categories: [35]

a) Pre-tanning (Beam house Operations)

The majority of the effluent load is generated by cleaning and conditioning hides and skins.

- Soaking: is the first step involved in tanning where the preserved raw skins or salted skins are treated with water to make the skin dirt free and soft. The main purpose of soaking is to remove salt, rehydrate the dry skin and also to remove unwanted materials like blood, soil, dung, etc. [35, 37].
- Liming: The second operation is liming which involves the removal of hair and unwanted materials which are not transferred to leather. It also loosens the epidermis and also removes soluble skin proteins. It uses lime and sodium sulphide as liquor. The hair is loosened due to increase in pH. The higher pH also causes splitting and swelling of fiber bundles. Dehairing and fleshing is also done in order to remove extra flesh and allow tannins to penetrate easily [35, 36, 20].
- Deliming and Bating: Deliming is the process of adjusting pH between 8-9 which enhances the enzyme activity and converts proteins into soluble forms. It uses ammonium sulphate and results in de-swelling of pelts. Bating makes the grain surface soft and flexible. It prepares skin for tanning. It is an enzymatic operation which removes unwanted

proteins and increases the degree of stretch. It imparts flexibility and softness towards the leather. This results in the major part of the ammonium load in the effluents [35, 36].

- Pickling: the introduction of acid liquor and salts raises the acidity of the hide to a pH of 3, allowing chromium tannins to penetrate the hide. To keep the hide from inflating, salt is used. Fungicides and bactericides are commonly used at a concentration of 0.03-2 % for the ease of preservation [20, 37].
- Degreasing: Degreasing is a process used to remove extra fat and oils which allows the tannin to penetrate easily through the skin [36, 34].

b) Tanning (Tanyard Operations):

Tanning is the main operation which converts skin or hide to stable material called leather. In this step tannins are allowed to interact with the prepared skin which act on collagen and make it stable. The tanning process stabilizes the proteins (collagen) network of the hides or skins [35, 20, 37].

c) Wet Finishing (Post-Tanning):

In some cases, the wet finishing procedures are carried out in a single float. To achieve the right fullness, smoothness, and color, chromium tanned hides or Wet blue are usually retanned (through which procedure the good features of more than one tanning agent are united) and treated with dye and fat. The excess water is removed before the skins are allowed to dry completely, making them ideal for splitting and shaving. To achieve the necessary thickness of the hide, it is split and shaved [36, 34, 37, 35].

d) Finishing:

Following retanning and drying, the crust goes through a series of finishing processes, including surface coating, polishing, seasoning, plating, embossing, spraying, and final sorting and measuring. The goal of these procedures is to soften the hide and conceal minor flaws. The hide is dyed and varnished using an organic solvent or a water-based dye [36, 35, 34, 20].

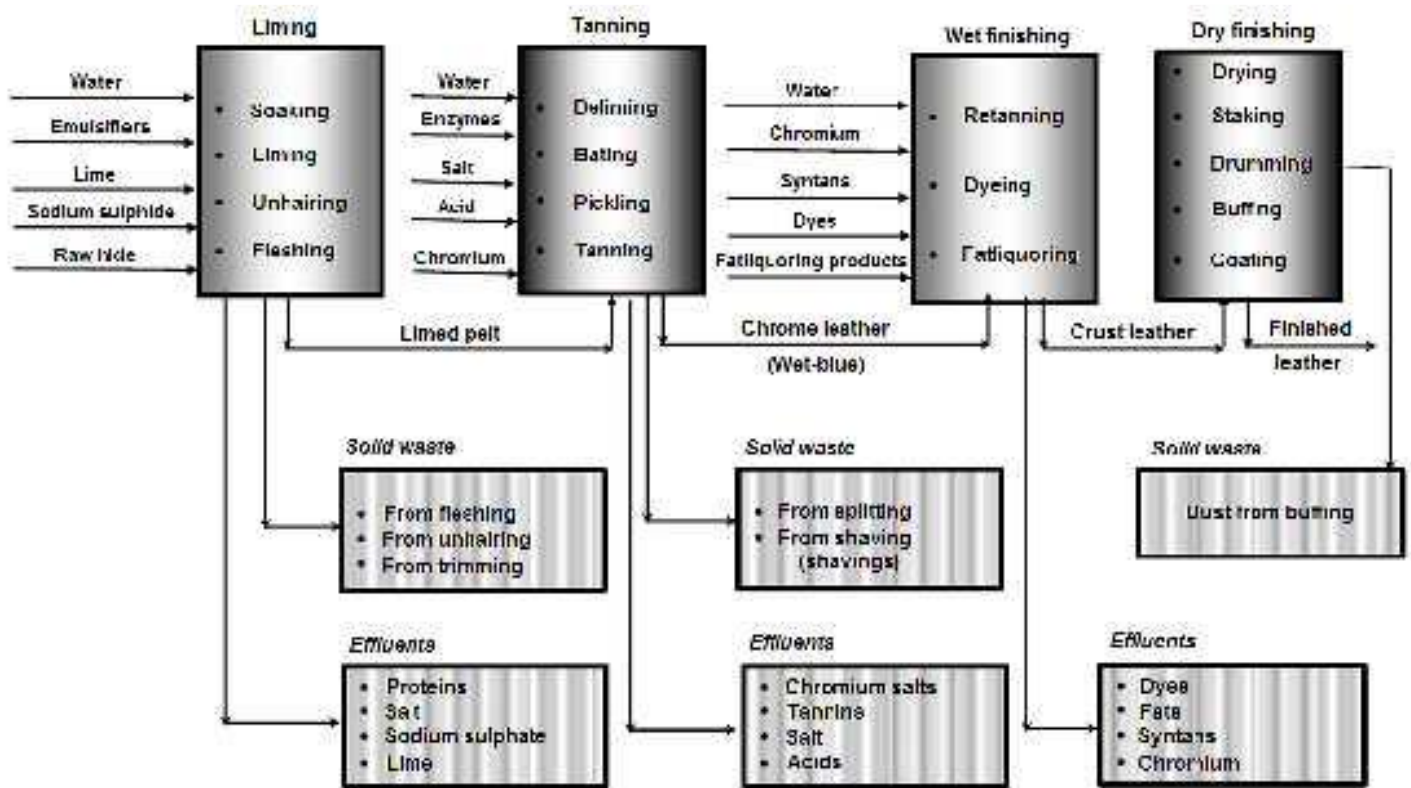


Figure 1: Schematic diagram of Polluting waste resulting from various phases of cattle hide processing

2.1.2 Definition of Sludge

Sludge is semi-solid slurry that can be generated by a variety of industrial applications, such as water treatment, wastewater treatment, or on-site sanitation systems, according to Field M. It can be made from a settled suspension from typical drinking water treatment, sewage sludge from wastewater treatment operations, or fecal sludge from pit latrines and septic tanks, for instance. The term is also frequently used to denote solids that have been separated from their liquid suspension; this 'soupy' material typically retains significant volumes of 'interstitial' water (between the solid particles) [38, 27, 26].

For Manish K. and Lokesh, S. Sludge is a result of wastewater treatment that consists of solids, liquids, or semisolids residuals (concentrated contaminants). Depending on the activities and methods utilized, sludge typically contains 0.25-12 % particles by weight [39, 40, 27].

Tannery sludge is semi-solid slurry that is formed as a by-product of tannery effluent. This residue is classified as Primary & Secondary Sludge. The primary sludge contains precipitated solids that

occur during the primary treatments which are chemical precipitation, sedimentation, and other primary processes in the primary clarifiers, whereas the secondary sludge separated in the secondary cleaners contains the sewage sludge purified from the secondary treatment bioreactors [41, 27].

2.1.3 Generation of Sludge

Tannery sludge (TS) production is rising around the world [42]. Only 250-300kg of the raw material is converted into leather [6]. About 30 – 40 m³ of wastewater is generated when processing one tonne of raw hide/skin to finished leather. The wastewater is treated either in individual or common effluent treatment plants to meet standards prescribed for discharge to different recipients. Suspended solids in the wastewater, generally in the range of 2000 to 5000 mg/l, are precipitated by the application of chemicals such as alum, lime, and/or polyelectrolyte, at the stage of primary treatment. Though sludge is separated at different stages of treatment in a treatment plant, it is the primary sludge, which accounts for the bulk of it [43]. In conventional Physico-chemical and biological treatment systems, the primary treatment produces 70-80% of the sludge, while the secondary biological treatment produces the remaining 20-30% [28].

Tannery generates a huge amount of solid waste as follows: fleshing, 50-60%; chrome shaving/chrome splits/buffing dust, 35-40%; skin trimmings, 5-7%; and hair, 2-5% [6]. While factors such as the nature of the effluent, method of treatment employed and effectiveness of equipment used for clarification/settling are responsible for the quantity of sludge generated. It is generally in the range of 300 – 350 kg at 30-40% dry solids per tonne (1000kg) of raw hide/skin processed. Sludge thus is yet another solid waste of the tanning industry requiring appropriate utilization or safe disposal [43].

2.1.4 Characteristics of Tannery Sludge

To treat and dispose of the sludge that is produced in a wastewater plant, it is crucial to know the characteristics of the sludge that will be processed. Besides the conventional disposal of sludge, there is a growing interest in the utilization of sludge (bio solids) as a raw material to produce various value-added products, including bio pesticides, bio plastics, bio surfactants, enzymes, etc.

Therefore, it is also essential to investigate the physical, chemical, and biological characteristics of various types of sludge (bio solids) [44].

Wastewater sludge (bio solids) management options require extensive sludge characterization since wastewater sludge exhibit wide variations in their properties depending on the origin of the solids, the amount of aging that has taken place, and the type of processing to which they have been subjected [45].

Conventional sludge characteristics can be grouped in physical, chemical, and biological parameters. Physical parameters give general information on sludge processability and handleability. Chemical parameters are relevant to the presence of nutrients and toxic/dangerous compounds, so they become necessary in the case of utilization in agriculture. Biological parameters give information on microbial activity and organic matter/pathogen presence, thus allowing the safety of use to be evaluated. The chemical and biological characteristics of sludge play a major role in value-added products when the sludge is used as the raw material [45, 46].

2.2 Sustainable Disposal and Reuse of Tannery Sludge

Sludge from industry might be organic or inorganic. To avoid pollution, the inorganic content of industrial sludge, like heavy metals, should be treated separately. Furthermore, industrial sludge has become a critical problem as a result of public concern and insufficient land availability. [39]. Costs associated with WAS management may constitute as much as 30–50% of the total cost of wastewater treatment processes [45, 43, 40].

Tannery industries are continuously creating more environmental problems, particularly in polluting organic matters and dangerous solid waste from skin/hides processing [1]. Tannery sludge has a higher inorganic matter level, a higher heavy metal content, primarily chromium, and a higher sulfur compound content than sanitary sludge [47]. More than 40% of the chromium used in the leather making process will join the wastewater treatment plant and finally end up with wastewater treatment sludge [48].

Therefore, a simple method of discharging sludge directly into nearby hydric bodies or dumping in landfill sites is not a sustainable solution. It is a need to develop suitable sludge management strategies for sustainable development [46, 45]. Simply discharging the dewatered WTS into

drains or landfilling is not a sustainable disposal option. Hence, there is a need to find some environmentally friendly, sustainable, and at the same time economical solution [2].

Landfilling, hydrometallurgical processes to recover chromium, incineration, gasification, and other ways are used to dispose of tannery sludge or alternatively, if the chromium content can be lowered, it can be used as a soil coordinator [49]. Recycling the sludge in the building and construction industry could be a safe disposal option [46].

2.2.1 Solidification and Stabilisation

Solidification in construction materials such as brick or concrete has been used to manage hazardous waste in a variety of scenarios [50, 51, 52, 13, 53]. Solidification, also known as cementation, is a method of transforming waste into an insoluble rock-like material by mixing it with promising applications to produce a solid outcome [54, 29, 55]. It does not always require a chemical reaction between the pollutants and the solidifying additives. Solidification suggests the production of a solid, monolithic mass with sufficient structural integrity to be transported in conveniently-sized pieces without requiring any secondary container. Solidification, therefore, is the act of tying-up free water in waste to improve its handling characteristics or to makes it acceptable for landfills. To minimize the mobility of pollutants, additives are used in the solidification and stabilization of sludge [55].

The primary objective of the solidification/stabilization (S/S) process is to convert (potentially) toxic waste into an inert, dry, transportable, physically stable mass having very low leachability and with acceptable environmental properties and sufficient mechanical strength to allow for non-bearing construction, road foundations, land reclamation, landfilling, etc. [55, 49, 29].

The solidified product may be disposed of at a secured landfill site since the mobility of the wastes is greatly reduced or it can be recycled as construction materials. The qualities of the sludge-stabilized building material must meet local building material requirements in order to propose tannery sludge as a viable recycling option in the construction sector. Moreover, heavy metal leaching from the material must not create long-term harm to the environment [54, 55, 29].

Tannery sludge solidification/stabilization is a promising emerging treatment/disposal technology. Tannery sludge may be efficiently stabilized in construction materials, according to several studies [49, 56, 55, 29].

When it comes to hazardous waste disposal, S/S is essentially cost-effective solution than landfill disposal. S/S often uses readily available and low-cost raw materials as well as simple technologies. It hasn't received the same level of attention as safe landfilling. Additionally, it must be proven that S/S does not offer a solution to every disposal issue [49, 55, 29].

2.3 Review of Previous Studies on Sludge Stabilization Using Fired Clay Bricks

The leather industry is one of the world's most polluting industries. Leather processing has an unfavorable impact on the environment. Tannery wastes in the form of sludge must be managed responsibly in order to protect the environment. Clay has been used to make bricks for thousands of years. Tannery sludge could be used as a partial replacement for clay [2].

Due to the high demand and flexibility of a brick, various types of waste have been successfully incorporated into fired clay brick. The use of these wastes in clay bricks generally has a favorable effect on the characteristics, while there has been some evidence of a decline in performance in some areas. By using recycled wastes, good effects like lightweight bricks with better shrinkage, porosity, thermal characteristics, and strength can be achieved. The lightweight bricks will save money on shipping and production.

Moreover, this waste incorporation will reduce clay content in the fired clay brick, and then reduce the manufacturing cost. Most importantly, the high temperature used in the clay brick firing process allows for (i) the volatilization of hazardous components, (ii) the modification of chemical characteristics of the materials, and (iii) the incorporation of potentially toxic components through fixation in the waste's vitreous phase of the waste utilized. This has prompted many researchers to look into the possibility of incorporating different types of sludge into bricks [26, 21, 29, 39].

M. A. Swarna et al. (2014) incorporates 20% and 30% tannery sludge in different ratio of sand and cement and found that in cement bricks, tannery sludge can replace up to 20% of the cement and quarry dust can replace up to 100% of the sand [57].

G. Divya et al. (2018), in this study, an attempt has been taken to mix tannery sludge with clay in different proportions (5%, 10%, 15%, 20%, and 25%) in order to make porotherm block. The tannery sludge in powdered form is mixed with clay and hand cast in a mold. The mold is manufactured in such a way that it fits the dimension of the porotherm block (16"X6"X8"). Based on the experimental investigation it is observed that the inclusion of tannery sludge with clay results in the same value in compressive strength as normal porotherm block with the optimum percentage of tannery sludge with clay is 20%. Also, the introduction of tannery sludge in powdered form doesn't affect the environment as various tests are done to know its properties [2].

Md. Ariful et al. (2017), prepared clay bricks with various ratio of sludge (10%, 20%, 30%, and 40% by dry weight) and a curing temperature of 900⁰c, 950⁰c, and 1000⁰c in both laboratory-controlled and field conditions. Compressive Strength, water absorption, shrinkage, weight-loss on ignition and bulk density were used to evaluate their viability as a construction material. The findings of this study show that tannery sludge can be solidified in clay bricks sustainably with an optimum proportion of 10% [58].

In mortar samples, 3-12 % tannery sludge by weight of cement and 3-9% tannery sludge by weight of aggregate were tested. Flowability, density, ultrasonic pulse velocity (UPV), flexural and compressive strength, water absorption, and sorptivity were all tested on the mortar. It was discovered that by substituting 6% of cement or sand with TS, it is feasible to use TS in construction mortars. The addition of up to 6% TS to the mortar reduced porosity and water absorption while enhancing strength of up to 26%, as well as a 4% increase in density and ultrasonic pulse velocity. Cement, which is a costly reinforcing agent, can be saved in this fashion [42].

In a study done by P. Amsayazhi et al., (2018) the sludge brick (SB) blends were combined with various proportions (20%, 30%, and 50%) of swage waste (SW). Fineness, specific gravity, water absorption, and compressive strength were among the tests carried out. In the conclusion, bricks made with 20% SW are appropriate for producing good quality bricks [1].

S. Ramesh et al. (2017) blended soil and sludge samples in different proportions, such as 90: 10, 80: 20, 70: 30, 60: 40, and 50: 50. These samples are now put through a series of tests to

determine their strength, including the Sieve analysis test, California Bearing Ratio test, standard proctor test, and direct shear test. When compared to other mixed proportions (60:40, 50:50), the study suggests that using 20% and 30% tannery sludge in the soil proportion gives better strength [59].

Tannery sludge can be used in brick manufacturing for up to 10% of the dry weight of bricks, according to a laboratory and pilot plant study. The porosity of sludge containing bricks is higher than that of control bricks. Bending strength and frost resistance are satisfactory at 10% sludge, but are reduced to unacceptable levels in 15% sludge bricks. [60].

Prof. S.S. Razvi et al. (2016) looked at the use of clay-infused water treatment sludge. In the research, sewage sludge additions of 20, 25, 30, and 40% by dry weight, were used to make bricks and the results were compared to conventional brick. Sludge bricks containing up to 40% sludge were able to meet the technical requirements. Bricks with more than 30% sludge, on the other hand, should not be utilized since they are brittle and easily break even when handled carefully, and their color does not fit the specifications [18].

Literature review shows that utilizing the sludge in brick manufacturing is not only an economical method but also environment friendly. Also, if sludge is used on large scale for manufacturing the bricks, clay can be avoided from depletion. Besides literature shows that the chemical composition of the water treatment plant (WTP) sludge is similar to the clay. WTP sludge is a suitable material for mixing in the clay for brick manufacturing [26, 23, 61].

CHAPTER THREE

Materials and Methods

3.1 Materials

The primary raw material employed in this study were Tannery sludge, clay (white [75% from the total mass of clay] and red [25% from the total mass of clay]), and water.

The test parameters and the corresponding chemicals and the instruments used are shown in the table below.

Table 1: List of Chemicals and Materials

No		Test parameters	Chemicals	Material
1.	Characterization of raw material	PH	Buffer solution PH 4 and PH 7	PH meter
		Moisture content	-	Oven Crucible
		Ash content	-	Oven Crucible
		Heavy metals	HNO ₃ H ₂ O ₂ Heavy metal standards (Cd, Cr, Ni, Cu, Zn, As, and Pd)	ICP-OES
2.	Characterization of the clay sludge mixture	Atterberg tests	-	
		Compaction test	-	Compaction tester
3.	Characterization of the final product, bricks	Compressive strength of bricks	-	Strength machine
		Water absorption of bricks	-	
		Weight loss on ignition	-	Furnace
		Firing shrinkage of bricks	-	Shrinkage tester
		Bulk density	-	Density tester
		Heavy metals	HNO ₃ H ₂ O ₂ Heavy metal standards (Cd, Cr, Ni, Cu, Zn, As)	ICP-OES

3.2 Methods

3.2.1 Collection of Samples

The sludge was collected from the Addis Ababa tannery waste treatment plant which is located at the Kolfe Keraniyo sub-city Addis Ababa, Ethiopia. The sample was taken from the sludge storage facility that was temporarily set up after it has passed through many processing steps and the filter press, during the months of April and May, at various times. The sample was gathered and blended from all ends of the storage area to generate a representative sample. The sample was transported to the research site in a 25 kg plastic container. Furthermore, the other raw material, clay, was obtained from a local brick manufacturing company known as Ethiopian bricks manufacturing Sh. Co., which is also located in Kolfe Keraniyo Sub-city, Addis Ababa, Ethiopia.



A Tannery sludge

B. White Clay

C. Red Clay

Figure 2: Sample Collection

3.2.2 Study Location

The research work was conducted at Leather Industry Development Institute (LIDI) which is located in Akaki Kality Sub-city on the road to Bishoftu; the experimental analysis was conducted in LIDI Research and Testing Laboratory and Core consultancy engineering PLC.

3.2.3 Characterization of the Raw Material

The sludge sample, after it was collected and brought to the LIDI laboratory, was preserved and kept in a deep freezer and the characterization was done within 24hrs. To determine the physicochemical characteristics of sludge the following parameters were conducted; PH, moisture content, Ash content, organic content and heavy metal content.

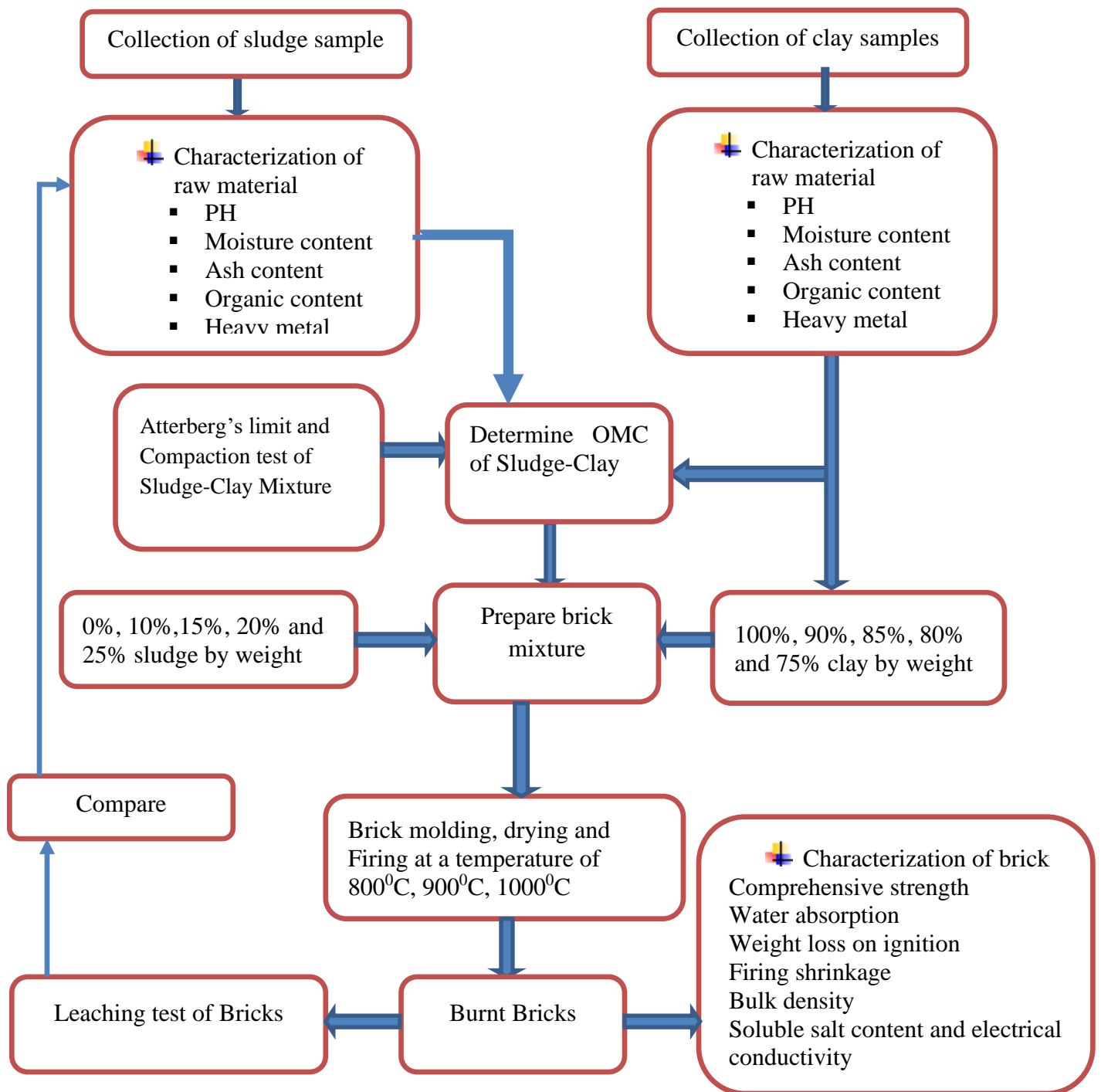


Figure 3: Flow Chart of the Total Process

3.2.3.1 Determination of the pH Value

The pH of the samples was determined by soaking 5grams of the sample in 100ml distilled water and keep in an orbital shaking device for 16-24 hours followed by direct measurement of the pH (Digital pH meter HANNA 210) according to the standard methods of SLC 13. (SLC, 1996)

3.2.3.2 Determination of Moisture Content

The moisture content of the sludge and the clay was measured by weighing 3 grams of the sample by crucible and put it in an oven (High Performance Oven FLLI GALLI, Model 2100) for 5 to 6 hours and the moisture content was determined according to the standard method of SLC 3. (SLC, 1996)

3.2.3.3 Determination of Ash Content

The ash content of the dried samples was determined by weighing 2.5 gram from the sludge and the clay sample separately by crucible and put it in a muffin furnace (GEFRAN 1001) for 2 to 3 hours at 500°C and the ash content was determined according to standard method of SLC 6.(SLC, 1996)

3.2.3.4 Determination of Organic Carbon Content

The organic content was determined by weighing 1.0gram from each sample separately into 500ml conical flask and then 10ml of $K_2Cr_2O_7$ solution and concentrated 20ml of H_2SO_4 was added. The flask was then gently swirled and allowed to stand for 30min on an asbestos sheet for the reaction to complete.

To this, 200ml distilled water was added to dilute the suspension and filtered. It was expected that the end point of the titration will not be obvious. To achieve this, 10ml orthophosphoric acid and 1-2ml of the diphenylamine sulfonate indicator were added and followed by back titration against 0.5N ferrous ammonium sulphate solution till the color flashes from violate through blue to bright green. Then the volume of the titrant was recorded.

3.2.3.5 Determination of Heavy Metal

The chromium metal content of the sludge and the clay was determined by using ISO17072-2:2012 test method. The samples were ground with a grinding machine (Ret Sch SM 300 grinder).

1gram of the ground sample, 8ml of concentrated nitric acid, and 2ml concentrated hydrogen peroxide was added in the digestion tube, including blank and the microwave digester with a maximum temperature of 250°C and pressure of 1200 psi was turned on for 30 minutes. Then the digested samples were poured into a 25ml Erlenmeyer flask and made up to the mark with 2% nitric acid solution and filtered through 0.45µm pore diameter membrane filter to avoid possible contamination. Finally, the sample was analyzed for the amount of chromium by using ICP-OES (Agilent 700 Series).

The calibration solutions were prepared from ultra-high purity grade ICP-OES heavy metal (Cd, Cr, Ni, Cu, Zn, As, and Pd) standard (99.99% pure). Six calibration solutions were prepared (1ppm, 2ppm, 3ppm, 5ppm, 7ppm and 10ppm) by plotting calibration curve with the specific wavelength (ISO17072-2, 2012).

3.2.4 Preparation of Sample

The clay and sludge samples collected were air-dried for 2 weeks. The air-dried samples were then subjected to an oven for 24 hours with a temperature of 105°C until constant weight was achieved and the dry samples were ground using a grinder (Ret Sch SM 300). The ground samples were sieved with a fine mesh sieve making ready for homogenization and preparation of brick.

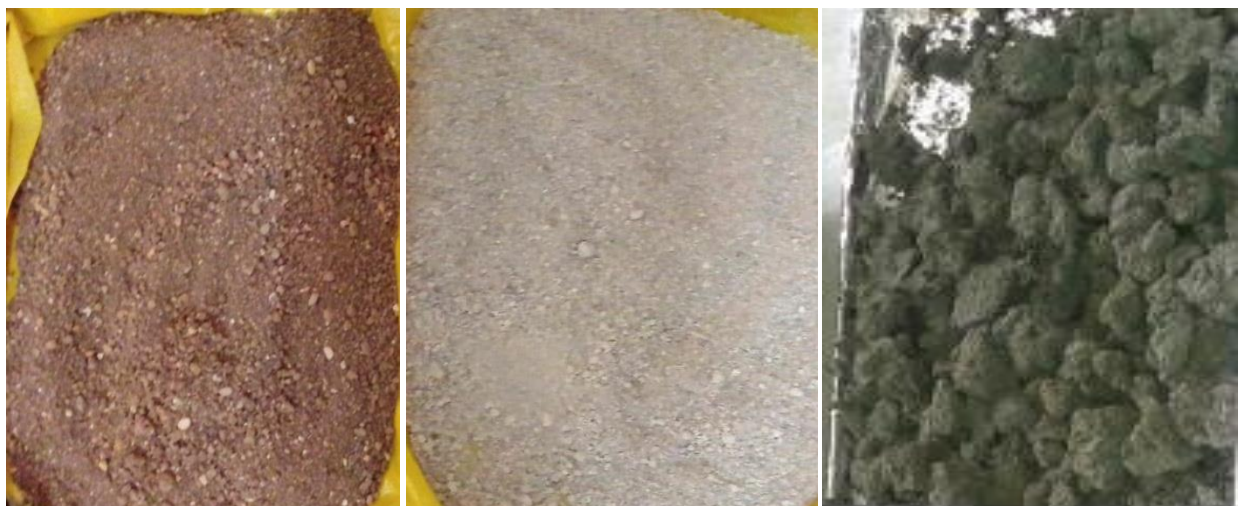


Figure 4: Air Dried Samples



Figure 5: Oven Dried, Grinded and Sieved Samples

3.2.5 Characterization of Clay – Sludge Mixture

This heading includes the Atterberg limit test and compaction test of sludge – clay combination.

3.2.5.1 Atterberg Tests

As the moisture content of a soil decreases the soil passes from the liquid state to the plastic state to the solid state. The range of moisture contents over which the soil is plastic is used as a measure of the plasticity index. The points at which a soil changes from one state to another are arbitrarily defined by simple tests called the liquid limit test and plastic limit test.

The liquid limit of a soil is the moisture content, expressed as a percentage of the weight of the oven-dried soil, at the boundary between the liquid and plastic states of consistency. The plastic limit of a soil is the moisture content, expressed as a percentage of the weight of the oven-dry soil, at the boundary between the plastic and semisolid states of consistency. These tests are known as the Atterberg limits. The plasticity index of a soil is the numerical difference between its liquid limit and its plastic limit, and is a dimensionless number.

Both air-dry sludge and clay samples were crushed into smaller particles and sieved through 40 number mesh sizes. Sludge concentrations of 0%, 10%, 15%, 20%, and 25% (by dry weight) were used to make clay-sludge mixtures. The liquid and plastic limit of these samples was determined using the Atterberg limit test, which was conducted in accordance with ASTM D 4318(2000).

The amount of water needed to transform soil from its plastic to liquid state is a measure of its plasticity. The "plasticity index," which is equal to the liquid limit minus the plastic limit, is used to determine plasticity (ASTM, 2010).

3.2.5.2 Compaction Test

Mechanical compaction is one of the most common and cost-effective means of stabilizing soils. Soil compaction is defined as a method for mechanically increasing the density of soil by reducing the air volume from the pore spaces [62]. Most engineering properties, such as the strength, stiffness, resistance to shrinkage, and imperviousness of the soil, will improve by increasing the soil density. The optimum water content is the water content that results in the greatest density for a specified compactive effort [13].

A standard method, AASHTO T- 99, was used to obtain the optimum moisture content (OMC), which is a key component impacting the characteristics of brick. Samples were air dried and crushed to pass through a 90m sieve. Individual OMC and dry density characteristics relating to OMC were determined using 2.5 kg samples for each type of mix proportion (AASHTO T-99, 1982).

3.2.6 Making of Brick

Mixing, molding, drying, and burning are the four basic phases in the production of bricks. In this research, a total of 45 samples were prepared by varying the sludge ratio and the firing temperatures. The amount of sludge in the mixture and the temperature at which it is fired are the two most important factors that affect the quality of the brick [18].

The proportions of raw components that must be mixed to make bricks with the respective firing temperature were given in Table (2). The sludge percentages were selected by referring to different literature [57, 2, 58, 1, 59, 60, 18]. Also, the ratio of white to red clay i.e., 3:1 was adopted from Ethio bricks manufacturing Sh. Co. Total mass taken was 400grams.

Table 2: Composition of Raw Materials by Percent and Mass

Trial	By Percentage [%]			By Mass[gm]			Firing Temperature [°C]
	Tannery Sludge	White Clay	Red Clay	Tannery Sludge	White Clay	Red Clay	
Control	0	75	25	0	300	100	800,900&1000
Exp1	10	67.5	22.5	40	270	90	800,900 &1000
Exp2	15	63.75	21.25	60	255	85	800,900 &1000
Exp3	20	60.0	20.0	80	240	80	800,900 &1000
Exp4	25	56.25	18.75	100	225	75	800,900 &1000

First, raw materials (sludge and clay) were mixed and homogenized with each other in the proportion mentioned above. Then a sufficient amount of water was added to the mixed samples to mold the brick. The sludge amended clay bricks were molded by hand using a steel rectangular sheet material of size 8 x 6 x 4 cm³ to give the desired shape. If excess moisture is not removed before the brick is fired, the water will burn off too quickly during the firing, causing cracking.

So, the produced brick blocks were then left for 10 days to allow for natural uniform drying before being dried in an oven for additional 24 hours at 105°C. Finally, the dried bricks were heated in a muffle furnace (GEFRAN 1001, Italy) to the designed temperatures of 800, 900, and 1000°C and were held in a constant time of 6hr. After the required time was elapsed the samples were removed from the furnace and set aside to cool (**See Appendix B**).

3.2.7 Quality Characterization of the Final Product

Some tests indicate the quality of bricks, allowing for the determination of whether or not they are suitable for use as construction materials. Compressive strength, water absorption, bulk density, firing shrinkage, and weight loss on ignition are among them. The most important parameters used to determine the quality of building materials are compressive strength and water absorption [63]. All of these quality parameters are described as follows.

3.2.7.1 Compressive Strength of Bricks

The ASTM C 67 method was used to conduct the compressive strength test. The load was applied at any suitable rate up to one-half of the estimated maximum load, following which the machine's settings were altered so that the remaining weight was applied uniformly in not less than 1 min and not more than 2 minutes. Using the equation below, the compressive strength was calculated.

$$\text{Compressive strength, (kg/cm}^2\text{)} C = \frac{W}{A}$$

Where,

C = compressive strength of the specimen, kg/cm².

W = maximum load, kgf.

A = average of the gross areas of the upper and lower bearing surfaces of the specimen, cm².

3.2.7.2 Water Absorption of Bricks

The ASTM C 67 method was used to conduct the water absorption test on bricks. Brick samples were oven-dried for 24hrs at 105°C using this method. The specimens were cooled to room temperature after drying. The dry, cooled specimen was immersed in distilled water at 26 -30°C for 24 h without any preliminary partial immersion. After that, the specimen was wiped clean of surface water with a damp cloth, and the weight of the specimen was collected within 5 min of removing it from the bath. Then, the water absorption of the sample was calculated using the equation below:-

$$\text{Water Absorption, (\%)} = \frac{100 (W_s - W_d)}{W_d}$$

Where,

W_d = dry weight of the specimen, and

W_s = saturated weight of the specimen after submersion in cold water.

3.2.7.3 Weight Loss on Ignition

The raw brick samples were dried in an oven at 105°C until they achieved a constant mass. After the samples had cooled, the weights of the samples were measured. After that, the samples were burned at various temperatures (800°C, 900°C, and 1000°C). The weight loss of the sample during the drying and firing steps was used to measure the Loss of Ignition (LOI).

$$\text{Loss of Ignition, LOI, (\%)} = \frac{100 (W_d - W_f)}{W_d}$$

Where,

W_d = Mass of oven-dried specimens (g)

W_f = Mass of fired specimens (g)

3.2.7.4 Firing Shrinkage of Bricks

The following equation was utilized to compute total volumetric shrinkage by taking the volume of the samples with a calliper before and after the firing stage.

$$\text{Firing shrinkage, (\%)} = \frac{100 (V_p - V_f)}{V_p}$$

Where,

V_p = volume of bricks before firing (cm³).

V_f = volume of bricks after firing (cm³).

3.2.7.5 Bulk Density

The weight of burnt brick samples, as well as their volume, was analysed. The average length, width, and height of the sample were multiplied to calculate volume. The following equation was used to calculate bulk density.

$$\text{Bulk Density (g/cm}^3\text{)} = \frac{M}{V}$$

Where,

M = dry mass of fired brick (g)

V = volume of fired brick (cm³)

3.2.7.6 Soluble Salt Content and Electrical Conductivity of Bricks

BS 1377 was used to determine the soluble chloride and sulphate content (1990). In a 500 ml container, a 100 gm sample passed through a 2 mm sieve and was wetted with 200 ml distilled water. After that, the sample was shaken for 18 hours. The sample was filtered with a 0.45 m filter paper after shaking. The chloride concentration of the collected filtrate was determined using a silver nitrate titration, whereas the sulphate content was assessed using a DR 4000 Spectrophotometer (HACH, USA).

A 50-gm broken sample was placed in a beaker with 150 ml distilled water to determine Electrical Conductivity (EC). The sample was first stirred and maintained for 24 hours, after which the EC was determined by dipping probe of the conductivity meter in the sample.

3.2.7.7 Leachable Chromium Test

The leachability of chromium from bricks was determined based on the Toxicity Characteristics Leaching Procedure (TCLP) test which is developed to find wastes that are likely to leach dangerous levels of specific harmful elements into groundwater. Dried brick samples fired at 1000⁰C were pulverized using a grinder and allowed through a 90mm mesh size sieve in the TCLP test. The samples were treated with an acetic acid solution (0.57%t v/v) at a constant ratio of liquid: solid (20:1). The fluid's pH was also evaluated. The leachate was collected with 0.45m pore size filter paper and tested for Cu, Ni, Pb, Cr, and Zn using an ICP-OES apparatus after 18 hrs. of rotational mixing at 32 rpm.

CHAPTER FOUR

Result and Discussion

This chapter includes laboratory tests to assess the properties of sludge and sludge-clay mixtures, engineering parameters of sludge-amended bricks, and leaching test findings for both sludge and sludge-amended burned bricks.

4.1 Characteristics of Raw Material

4.1.1 pH Value, Moisture Content, Ash Content, and Organic Content

The pH value of the raw materials was neutral which shows a range from 6.7– 7.6. According to the Ethiopian environmental protection the waste disposal standard pH should be between 6.5 and 8.0. Low pH sludge (less than approximately pH 6.5) promotes leaching of heavy metals, while high pH sludge (greater than pH 11) kills many bacteria and, in conjunction with soils of neutral or high pH, can inhibit movement of heavy metals through soils [45], so the results are within allowable limit.

The moisture content of the sludge, the white clay and the red clay was 34.9%, 4.06%, and 7.7% respectively. The ash content was founded to be 3.55% and 2.9% for the white and red clay respectively. The sludge's ash level was determined to be 37.1 %, indicating that there is a significant amount of inorganic component. The organic contents of sludge were 33.04%. The relatively low level of organic matter in sludge decreases the water infiltration and water-holding capacity of the clay. The lower water infiltrates into bricks, the higher is the durability [45].

Table 3: Characteristics of the Sludge and Clay Sample

Sample type	pH	Moisture content (%)	Ash content (%)	Total organic carbon (%)
Red Clay	6.7	7.7	2.9	3.77
White Clay	7.2	4.06	3.5	3.60
Sludge	7.6	34.9	37.1	33.04

4.1.2 Heavy Metal Content

The following heavy metal concentrations were found in the sludge sample: Cd: 0.069 mg/kg; Cr: 451.1 mg/kg; Cu: 3.02 mg/kg; Ni: 0.84 mg/kg; Pb: 2.27 mg/kg; and Zn: 4.65 mg/kg. The total heavy metal concentration of the sludge was compared to heavy metal content regulatory limits for sludge utilization. The average concentration of Cadmium, Chromium, Copper, Nickel, Lead, and Zinc was found to be considerably below the US Land Disposal Restriction Limits, China's SEPAC limit, and India's limit.

But the mean concentration of Cr crossed the SEPAC limit in China and the Limit in India. Because most hides and skins are tanned using basic chromium salt, tannery sludge usually contains significant levels of chromium, and during wastewater treatment, over 60% of the chromium is wasted and subsequently moved to sludge. [64].

Though the highest heavy metal content is below the USEPA's permissible limit for land application, the sludge will not be acceptable for use as a soil conditioner in agricultural land or household vegetation due to the greater value of Chromium present in the sludge based on different regulatory limits.

Table 4: Concentration of Heavy Metals in Sludge and Standards for Sludge Utilization

Heavy metals	Concentration (mg/kg)	Limit in Ethiopia	USEPA limit (mg/kg) ^a	SEPAC limit in China (mg/kg) ^b	Limit in India (mg/kg) ^c
Cd	0.069	NA	0.6	0.6	3-6
Cr	451.1	NA	3000	250	250
Cu	3.02	NA	4300	100	135-270
Ni	0.84	NA	420	26600	26600
Pb	2.27	NA	840	350	250-500
Zn	4.65	NA	7500	300	300-600

NA: Not Available

Source:^a<http://www3.epa.gov/npdes/pubs/sludge.pdf>,^b Source: SEPA(1995),^c Bhatnagar&Awasthi (2000)

4.2 Characterization of Clay – Sludge Mixture

4.2.1 Atterberg Limit Tests

The Atterberg limits are empirical tests which are used to indicate the plasticity of fine-grained soil by the differentiation between highly plastic, moderately plastic and non-plastic soils. The tests enable classification and identification of the soil to be carried out and give a rough guide to the engineering properties.

Panjaitan (2014) showed that plasticity index states the properties of soil (Table 5). A low Plasticity Index indicates that a little variation in moisture content will turn the soil from semi-solid into liquid. The Plasticity Index can also be used to determine compressibility. The higher the PI indicates the higher the compressibility of the soil. Both the liquid and plastic limits are moisture contents [65].

Table 5: Plasticity Index (PI) and Properties of Soil

PI Range	Plastic Nature	Soil Properties
0	Non plastic	Sand
<7	Low plastic	Silt
7-17	Medium plastic	Silt clay
>17	High plastic	Clay

Figure 6 shows the results of Atterberg tests on a sludge-clay mixture, which show that the Plasticity Index drops as the sludge proportion in the mixture rises. The addition of sludge reduces the plastic character and also the bonding ability of the mixture, according to Plastic Limit values. When a mixture has a large amount of sludge, the mixture's bonding capacity is reduced.

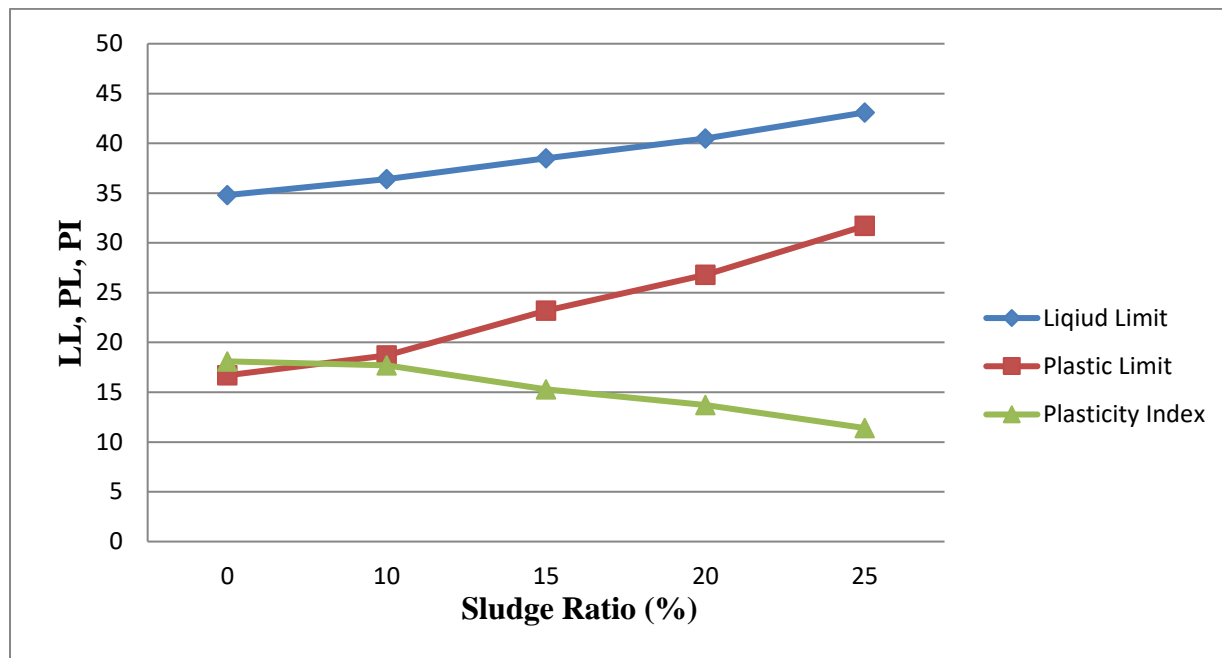


Figure 6: Plastic Limit, Liquid Limit, and Plasticity Index in Relation to Sludge Content

Table 6: Effect of Moisture Content on Sludge Clay Mixture Liquid Limit, Plastic Limit, and Plasticity Index

Parameters	Sludge proportion (% by dry weight)				
	0%	10%	15%	20%	25%
Liquid Limit (%)	33.7	35.3	37.4	39.4	43.0
Plastic Limit (%)	16.2	18.6	22.9	27.1	32.0
Plasticity Index (%)	17.9	18.0	15.8	13.1	11.4

The soil utilized in the brick making process has a Plasticity Index of 17.9 (Table6), indicating that it is a highly plastic material. The Plasticity Index of sludge–clay mixture was 18.0, 15.8, 13.1, and 11.4 for 10%, 15 %, 20 %, and 25 % sludge–clay mixture, respectively. As per the soil characteristics listed in Table 5, sludge can be utilized in brick manufacturing at a percentage of 10% to 20% without losing its major plastic behavior.

4.2.2 Compaction Test

The amount of moisture in bricks has the potential to affect their properties. Additional water replaces air in the soil voids up to a point, but after a certain degree of saturation is reached, the water takes up space that would otherwise be occupied by soil particles [66].

Compacting at water contents higher than (wet of) the optimum water content results in a relatively dispersed soil structure (parallel particle orientations) that is weaker, more ductile, less pervious, softer, more susceptible to shrinking, and less susceptible to swelling than soil compacted dry of optimum to the same density. The soil compacted lower than (dry of) the optimum water content typically results in a flocculated soil structure (random particle orientations) that has the opposite characteristics of the soil compacted wet of the optimum water content to the same density [13].

Table 7: Effect of Moisture Content on OMC and Dry Density of Sludge Clay Mixture

Parameters	Sludge proportion (% by dry weight)				
	0%	10%	15%	20%	25%
Dry Density (gm/cm³)	1.77	1.63	1.56	1.50	1.44
Optimum Moisture Content (%)	18.0	21.0	25.0	28.0	32.0

The dry density of the sludge-clay mixture dropped as the sludge content of the mixture increased, according to the results of the compaction test shown in Figure 6. On the other hand, when the amount of sludge in the mixture increases, OMC increases. The amount of moisture in bricks has the potential to impact their properties.

As a result, in the brick manufacturing process, an optimal amount of mixing water is usually sought to achieve the highest level of compaction for a particular amount of soil sample. As a consequence, the bulk density will improve, the water absorption tendency will decrease, and the compressive strength will be the most [58, 66].

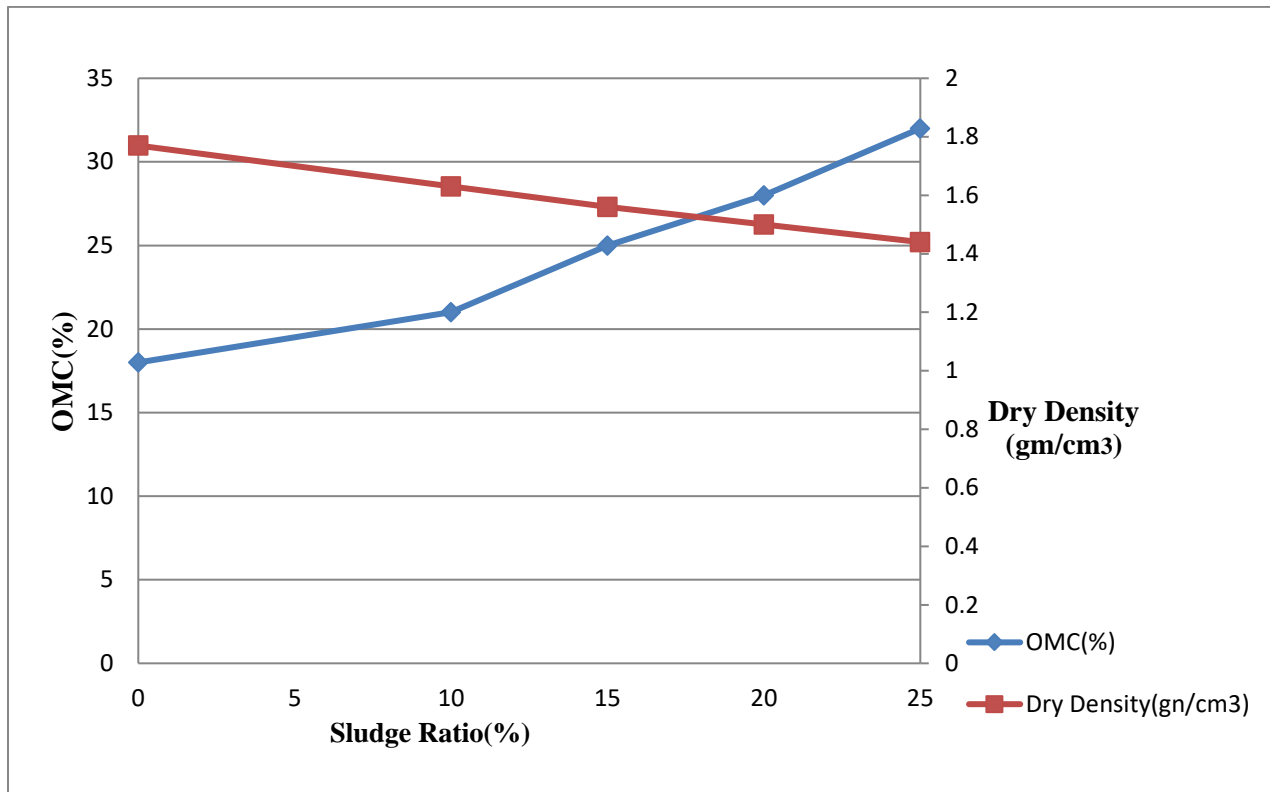


Figure 6: Result of sludge addition on OMC and Dry Density

4.3 Quality Characterization of the Final Product

4.3.1 Compressive Strength of Bricks

Compressive strength of bricks is the capacity of brick to resist or withstand under compression when tested on Compressive testing machine [CTM]. The Compressive strength of a material is determined by the ability of the material to resist failure in the form of cracks and fissure.

Figure 7 shows the results of the bricks' compressive strength test. Tannery sludge integrated bricks had compressive strengths ranging from 9.21 MPa to 27.14MPa. Sludge concentration and firing temperature appear to have a significant impact on the compressive strength of bricks. The comparative strength of bricks is directly proportional to the firing temperature, but inversely proportional to the concentration of sludge supplied which is in agreement with the finding in other studies [49, 54, 67, 68].

According to a laboratory test on compressive strength of bricks, adding 25% sludge to the mix decreases strength by 33.4 %, 38.3 %, and 58.9 % for firing temperatures of 1000⁰C, 900⁰C, and 800⁰C, respectively, in comparison with sludge-free brick. As 10%, 15%, 20%, and 25% sludge is added to the mixture, the strength is reduced by 10.3%, 14.6%, 23.4 %, and 33.4%, respectively, when compared to control bricks fired at 1000⁰C. The rise in porosity could be the cause of this proportionate trend.

The test was completed at three distinct temperatures and sludge concentrations, and all of the data from the lab test is organized in (Appendix D). According to Ethiopian standard for fired clay bricks (ECAE) all the sludge incorporated bricks meet the minimum compressive strength value and also can be considered as Grade A category.

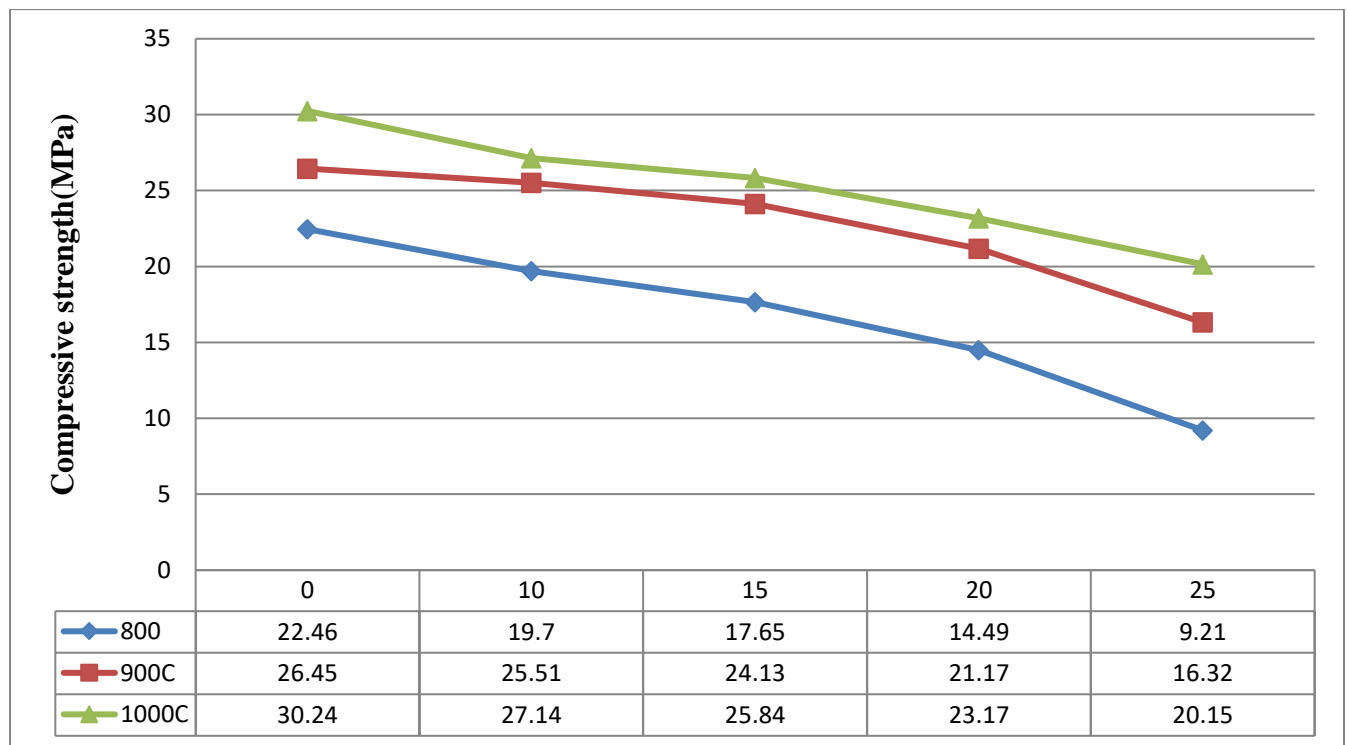


Figure 7: Compressive Strength of Brick at Different Clay-Sludge Ratio and Firing Temperature

4.3.2 Water Absorption of Bricks

Water absorption tests on bricks are used to measure the bricks' durability properties, such as degree of burning, quality, and weathering behavior. Water absorption tests can be used to

determine the degree of compactness of bricks, as water is absorbed through pores in bricks. Unlike compressive strength, water absorbency is inversely proportional to temperature this could be due to the amorphous phase forming at high firing temperatures [69].

Whereas sludge concentration is directly connected, this is due to the fact that tannery sludge includes a high concentration of organic chemicals, which give the brick a high porosity, resulting in a higher water absorbency rate perhaps making them more susceptible to weathering. In terms of water absorption capabilities, the findings demonstrate that as the amount of TS decreases, so does the percentage of water absorption.

Water absorption was 13.83 % in bricks containing 10% sludge fired at 800°C, but it was reduced to 11.07 % when the firing temperature was increased to 900°C and even to 9.17 % when the firing temperature was increased to 1000°C. Water absorption, on the other hand, increased from 7.03 % to 16.03 % when the sludge fraction was increased from 0% to 25% at 1000°C firing temperature (Fig. 8). Other investigations [13, 70, 71] found same water absorption tendencies in bricks with sludge amended.

The average findings of water absorption of samples bricks of every form of clay-sludge combination as a function of sludge content and temperature are shown in figure 8. According to the data gathered, the water absorbance ranges from 7.03% to 21.03%, which falls within the permitted limit for Ethiopian standard for fired clay bricks.

Table 8: Ethiopian Standard for Fired Clay Bricks (ECAE)

Class	Minimum Compressive Strength (N/mm ²)		Water Absorption (%)	
	Average of 5 Bricks	Individual Brick	Average of 5 Bricks	Individual Brick
A	20	17.5	21	23
B	15	12.5	22	24
C	10	7.5	No Limit	No Limit
D	7.5	5.0	No Limit	No Limit

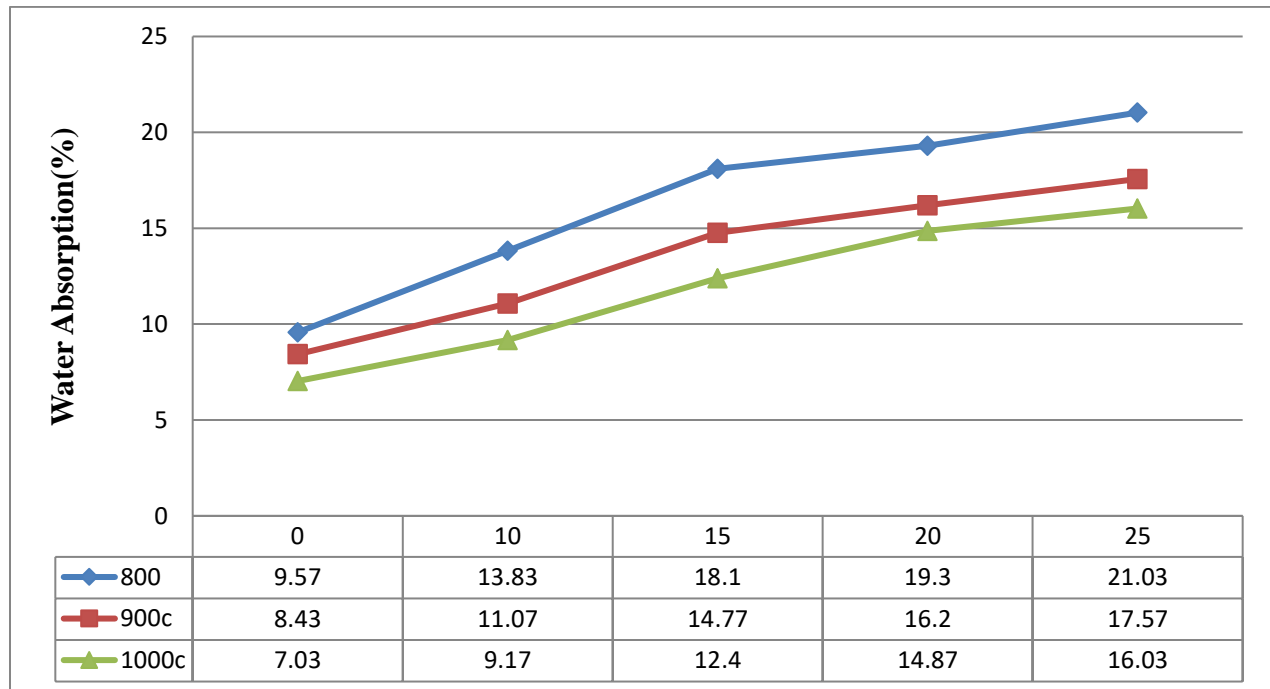


Figure 8: Water Absorption of Brick at Different Clay-Sludge Ratio and Firing Temperature

4.3.3 Firing Shrinkage

Another key factor in determining the quality of bricks is firing shrinkage. The volumetric variation of bricks during the drying stages of manufacture is known as shrinkage. While heating, the chemically and mechanically attached water is removed, resulting in shrinkage [72]. High shrinkage is not a good characteristic for any engineering material, and bricks are no exception.

The firing shrinkage in this experiment increased as the temperature increased, but dropped as the sludge concentration increased. In case of 10% sludge incorporated bricks shrinkage increased from 5.20%, 7.70% and 8.93% as firing temperature increased from 800°C, 900°C and 1000°C respectively. The volumetric shrinkage of 25% sludge amended bricks fired at 1000°C was reduced by up to 50.96% in comparison to 0% sludge bricks.

The non-plastic character of dried samples accounts for the decrease in shrinkage for the sample with high sludge concentration. Soil with a low plastic content shrinks less than soil with a high plastic content. With increasing sludge percentage (Table 6), the plasticity index of tannery sludge - clay mixtures dropped, causing the mixture to become more and more non-plastic [73]. Also, the

declining tendency observed here is related to the expansion in the bricks during the firing process.

Due to the combustion of organic matter, sludge-incorporated bricks released more gases than control bricks, resulting in voids that produced a net expansion inside the sludge-clay matrix and followed by a decreased net shrinkage [68].

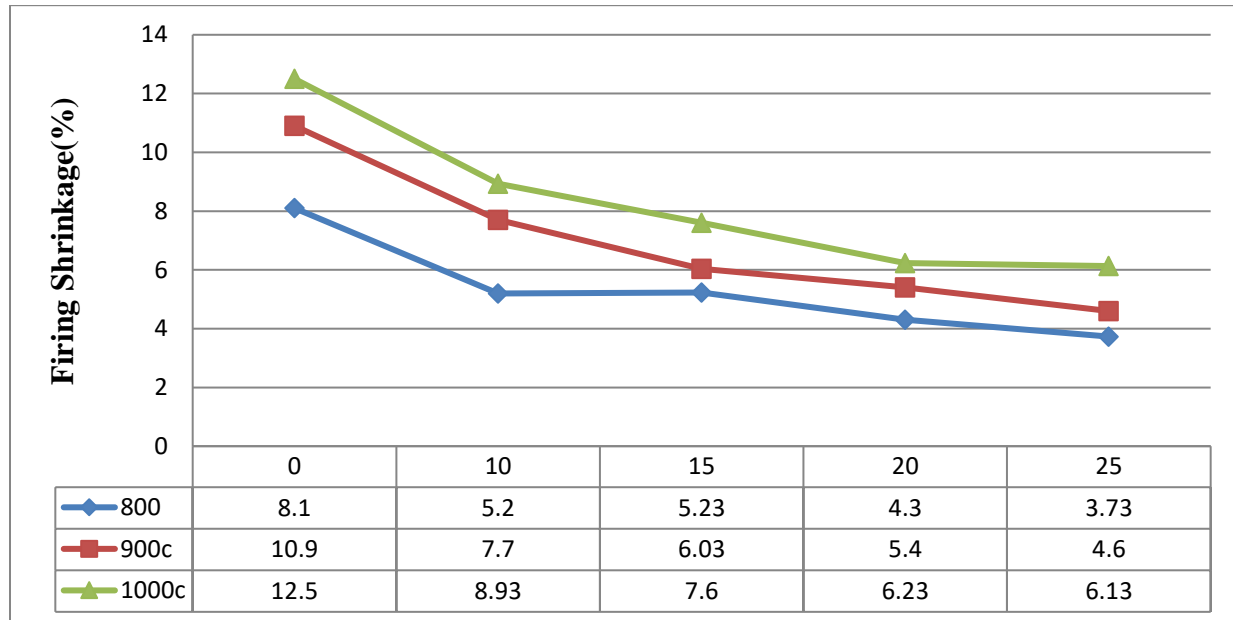


Figure 9: Firing Shrinkage of Brick at Different Clay-Sludge Ratio and Firing Temperature

4.3.4 Weight Loss on Ignition

Weight loss due to ignition does not only reflect the amount of organic matter consumed, but also considers the number of inorganic materials consumed during ignition [72]. Temperature and sludge content have a big impact when it comes to weight loss on ignition. It is very similar to the firing shrinkage parameter; the sole difference is that weight loss is a measurement of weight loss, whereas firing shrinkage is a measurement of volumetric deformation of bricks during the firing stages of the process; however the reasons for shrinkage can also result in weight loss of bricks.

Figure 10 depicts the influence of sludge content and fire temperature on brick weight loss. The weight loss of bricks is shown to rise as the percentage of sludge increased. Weight loss similarly increased as the firing temperature was raised. A linear relationship was observed between weight

loss during burning and the percentage sludge content of the bricks. The final weight of the brick decreases as the percentage of sludge utilized increases. This is a favorable consequence because it reduces the overall dead weight of the brickwork which in turn eases in handling, and savings in cost in transportation.

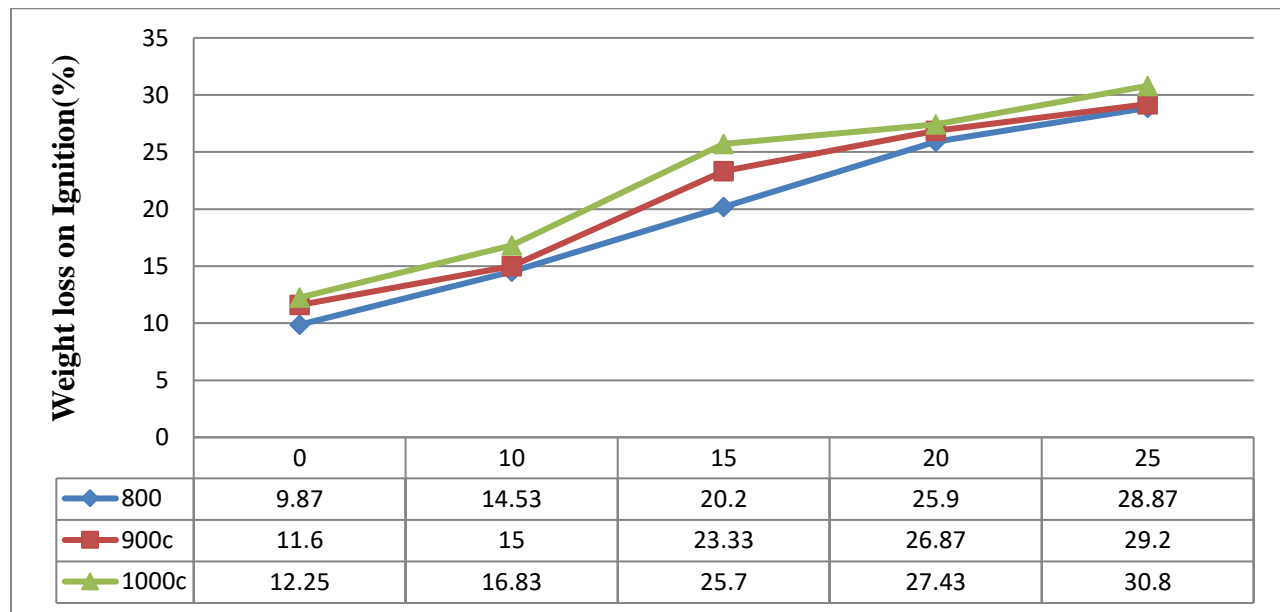


Figure 10: Weight Loss on Ignition of Brick at Different Clay-Sludge Ratio and Firing Temperature

4.3.5 Bulk Density

Bulk density is a measure of how compacted materials are. It is computed by dividing the material's dry weight by its volume. This volume includes the volume of particles and the volume of pores among the particles.

According to the experiment results, the bulk densities of the TS-added bricks and the quantity of sludge introduced in the combination show an inverse relationship. As the sludge content grew from 0% to 25% in this experiment, the bulk density declined from 1.91 to 1.46 at 1000°C firing temperature. This is due to the fact that when the TS % in the mixture rises, so does the amount of water absorbed through the pores. More water absorption implies a larger pore size than less water absorption, which results in a lower bulk density. The bulk density of the bricks can also be affected by the firing temperature. The analysis revealed that raising the temperature causes a

modest increase in bulk density. The thermal insulating properties of bricks with a low bulk density are good.

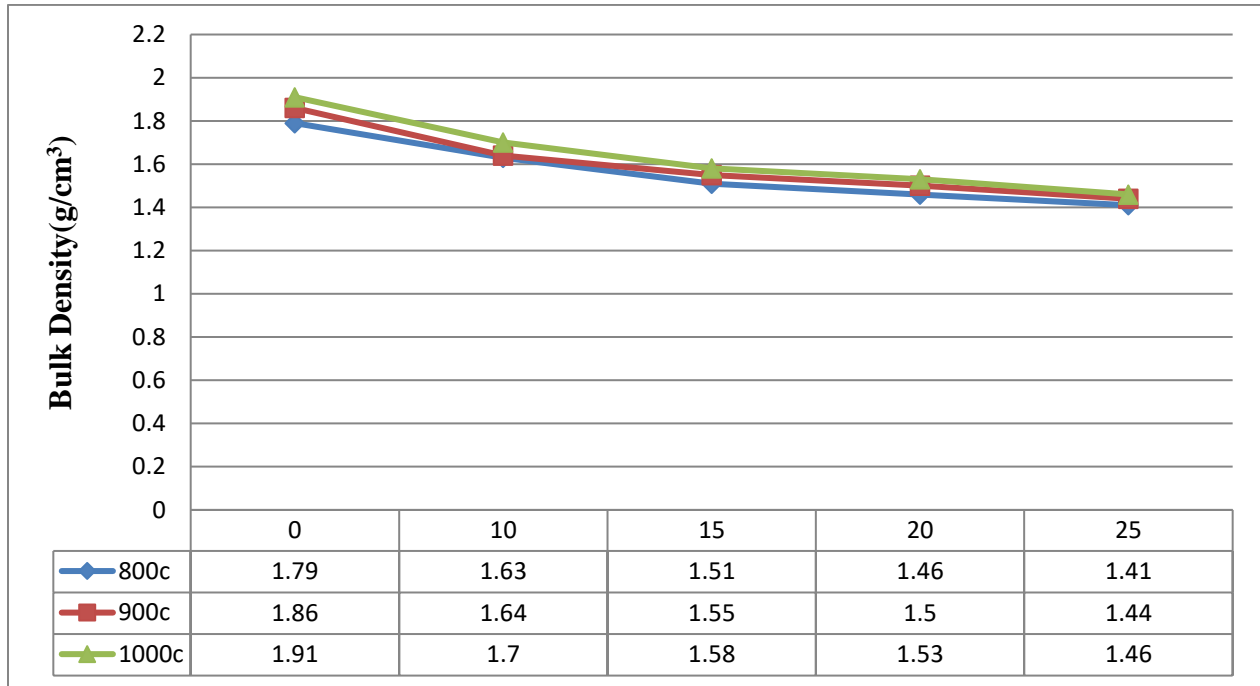


Figure 11: Bulk Density of Brick at Different Clay-Sludge Ratio and Firing Temperature

4.3.6 Soluble Salt Content and Electrical Conductivity of Bricks

The amount of salt in a construction material has a significant impact on its durability. Sulphates, chlorides, and nitrates are the most prevalent salts found in masonry. When salts in brick, such as calcium, magnesium, sodium, and potassium, dissolve in water, they generate efflorescence on the brick surface. Bricks with an excessive amount of salt are less resistant to weathering and have a lower compressive strength [74].

Decomposition of bricks and mortar due to salt-induced deterioration, and damage to paint or plaster adhesion due to precipitating salts on the surface are all possible consequences of this concentrating phenomenon.

Sludge and TS bricks' soluble chloride content, sulphate content, and electrical conductivity are shown in Table 9. Even the 25% TS brick had a soluble chloride content of 0.024 %, which was lower than the European limit of 0.03 %. In both raw sludge and TS-clay amended bricks,

sulphate concentration was higher in percentage than chloride content. This could be related to the use of basic chromium sulphate in the leather tanning process.

As can be shown in table 9, electrical conductivity increased with the concentration of sludge. Electrical conductivity of sludge was determined to be 8052 s/cm, but for 10% and 15% TS bricks, it was reduced to 1337 s/cm and 1465 s/cm, respectively. This result showed that raw sludge had a larger concentration of free electrolytes, resulting in increased electrical conductivity. Because of its thermal insulating properties, thermal conductivity is an important factor in determining energy savings. A material with low heat conductivity has better insulating qualities [68].

Table 9: Electrical Conductivity and Soluble Salt Content of Sludge and TS Bricks

Parameters	Raw Sludge	10 % TS Bricks	15 % TS Bricks	20 % TS Bricks	25 % TS Bricks	European Limit
Sulphate Content (%)	1.16	0.31	0.73	1.25	1.89	0.50
Chloride Content (%)	0.2	0.003	0.009	0.015	0.024	0.03
Conductivity (µs/cm)	8052	1337	1465	1594	1710	-

4.3.7 Leaching Test of Bricks

As heavy metal-contaminated sludge is introduced into finished products, heavy metal leaching becomes a major concern. Even under extreme conditions, it should be assured that heavy metal leaching from bricks does not exceed the maximum allowable limit. The waste is considered as hazardous if the number of toxic components surpasses the permissible limit.

Table 10 summarizes the resultant leachability readings of six heavy metals from sludge included and control bricks burnt at 1000°C, which were compared to the USEPA Standards of leaching restriction limits for construction materials.

The leachate assessment as per the USEPA 1311 from sludge altered bricks fired at 1000°C temperature and after 83 days showed that metals like Cd, Cr, Cu, Ni, Pb and Zn leached from

sludge amended bricks, even though the concentrations are well below the US-EPA acceptable range.

Table 10: Leaching Test of Tannery Sludge Bricks Fired at 1000°C

	Cd	Cr	Cu	Ni	Pb	Zn	
Raw sludge (mg/kg)	0.07	439.9	3.07	0.82	2.39	4.35	
White Soil	0.03	0.21	3.45	6.12	7.89	65.75	
Red Soil	0.04	0.53	2.13	4.23	1.55	11.84	
Leached	0%	0.007	0.015	0.024	0.009	0.078	0.016
	10%	0.002	0.270	0.014	0.003	0.021	0.012
	15%	ND	0.412	0.008	ND	0.040	0.009
	20%	ND	0.503	0.003	ND	0.054	0.004
	25%	ND	0.596	0.003	ND	0.062	0.003
US-EPA limits	1.0	5.0	100.0	11.0	5.0	500.0	

Table 10 shows that the leaching concentrations of Cr, Pb, and Cu for the 10% sludge introduced bricks burnt at 1000°C are reduced by 99.9%, 99.1%, and 99.5 %, respectively, when compared to the original sludge. These metals are thought to be locked as metal oxide inside the burnt bricks [70].

The leaching of Cr increases as the sludge content in the brick increases, while the leaching of other metals does not appear to be affected by the amount of sludge in the bricks. This could be because, apart from Cr, the quantities of the other heavy metals measured in the raw sludge were fairly low, and hence heavy metal stabilization from sludge by integration in brick was more prominent for Chromium (Table 10).

Based on the results of the leaching tests, it is possible to declare that sludge altered bricks can be utilized for building without causing any environmental issues, and even after the end-of-life cycle of these bricks, heavy metal leaching will be insignificant.

CHAPTER FIVE

Conclusion and Recommendation

5.1 Conclusion

The sludge released from the leather industry's treatment plant is currently a practical issue. A large amount of sludge is produced, which has the potential to harm public health and the environment. This research looks into the physical, mechanical, and environmental effects of partially substituting clay with tannery sludge in the development of a construction material brick that is an alternative form of sludge management and utilization.

As per the major findings of this study, the Plasticity Index of clay – sludge mixtures decreases as the amount of sludge added increases, and 10% and 15% of sludge can be utilized in brick manufacturing while keeping the major plastic properties. For all brick samples, the compressive strength improved when the firing temperature was increased. In comparison to the control brick, adding 25% sludge to the combination decreases strength by 33.4 %, 38.3 %, and 58.9 % for fire temperatures of 10000C, 9000C, and 7000C, respectively. Compressive strength of tannery sludge bricks, on the other hand, decreased as tannery sludge content increased. When the TS content was increased from 0% to 25% and burnt at 1000°C, the compressive strength of TS bricks decreased significantly, from 30.24 MPa to 20.15 MPa.

Water absorption increased from 7.03% to 16.03% when sludge proportion was increased from 0% to 25% at firing temperature of 1000°C. When the sludge proportion was increased from 0 to 25% at a firing temperature of 1000°C, water absorption rose from 7.03% to 16.03%. Water absorption rises as sludge content rises, but falls as the fire temperature rises. According to Ethiopian standard for fired clay bricks (ECAE) all the sludge incorporated bricks meet the minimum compressive strength value and the water absorption limit and also can be considered as Grade A category. As the firing temperature rises, so does the shrinkage, but as the sludge content rises, firing shrinkage decreases.

On ignition at a burning temperature of 1000°C, the control brick lost the least weight (12.25%), whereas the 25 % sludge amended bricks lost the most weight (30.8%). Due to the burning of organic matter, sludge addition and higher firing temperatures resulted in increased weight loss in

manufactured bricks. The combustion and degradation of organic and inorganic substances contained in both sludge and clay in the process of burning could be the cause for the weight loss.

As the TS content increased from 0% to 25%, the bulk density reduced from 1.91 to 1.46 gm/cm³. Though lower bulk density corresponds to higher water absorption, which is connected to brick quality, light weight bricks have good construction potential due to their lower dead load and good thermal insulation. The TCLP values for leaching concentrations of selected heavy metals tested in this study were determined to be negligible in comparison to USEPA permissible limits for all samples. The findings of TCLP studies revealed that increasing the firing temperature leads heavy elements like chromium to become even more immobilized.

To conclude, considering other properties 10% to 15% sludge amended brick and fired between 900⁰c to 1000⁰c is suitable for the brick masonry construction. According to the findings of the various tests conducted on the TS integrated bricks, a 10% sludge concentration by dry weight produces good quality bricks that meet all of the ECAE standards' mechanical and physical requirements. Furthermore, the amount of harmful metals leached into the environment is negligible, posing little risk to the environment. Tannery sludge incorporation could be a viable way to supply raw soil demand for brick manufacturing while also stabilizing waste in construction materials.

5.2 Recommendation

- I. This research might be used to convert tannery sludge into other building materials, as well as to look into ways to enhance the percentage of sludge by mixing it with other materials.
- II. Because mechanical properties and leaching behavior developed as firing temperature increased, a large-capacity furnace can be utilized to explore the impact of burning at a higher temperature on brick property and heavy metal leaching behavior.
- III. Additional research is needed to look at the long-term leachate analysis of sludge-based bricks.
- IV. Feasibility study on the TS amended bricks

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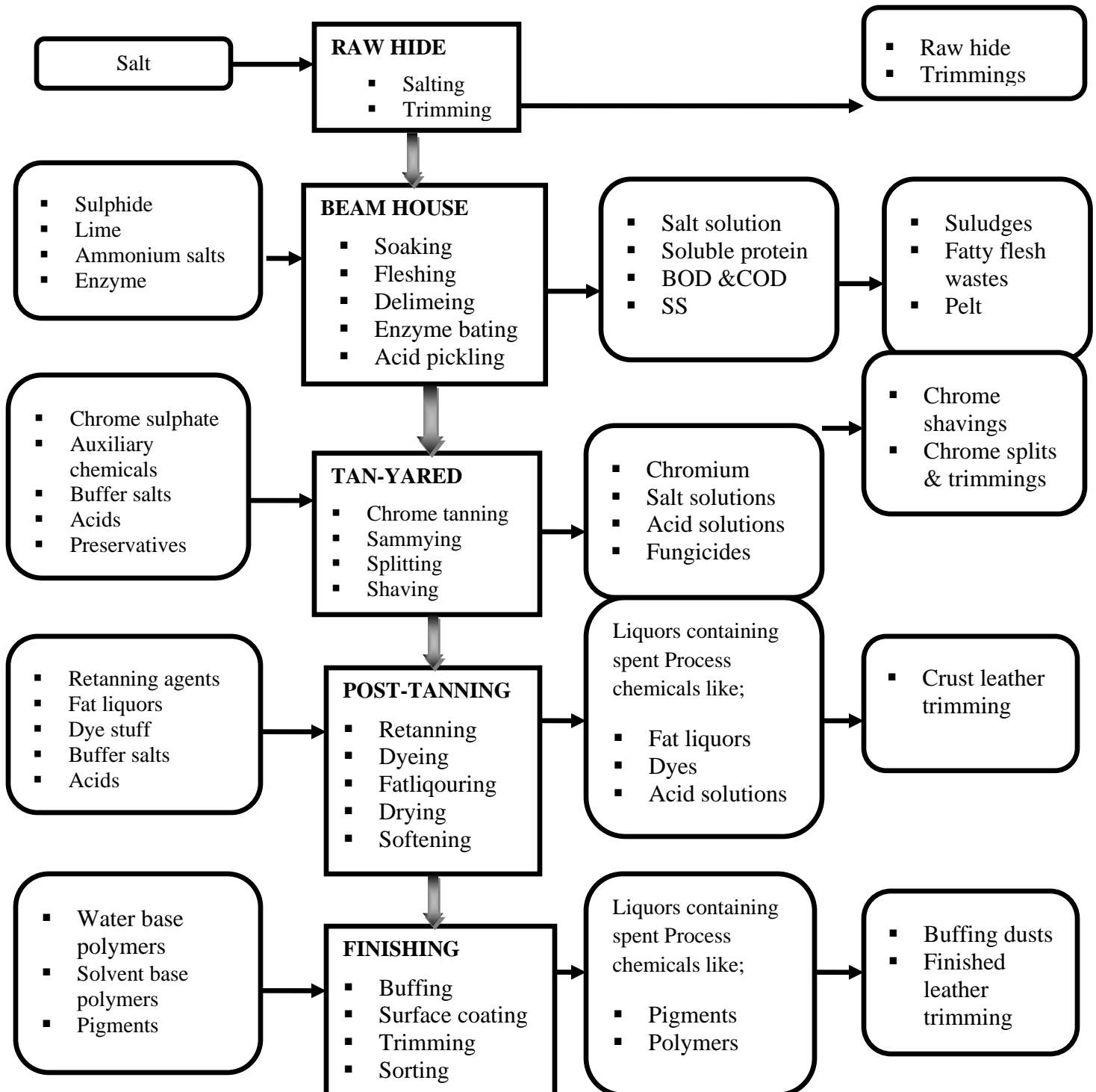
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APPENDICES

Appendix A: Flow Diagram of Leather processes

CHEMICAL INPUTS PROCESS STAGE LIQUID WASTES SOLID WASTES



Appendix B: Images for the Bricks Preparation Process



A. Weighing and preparing different proportion of white clay, red clay, and sludge



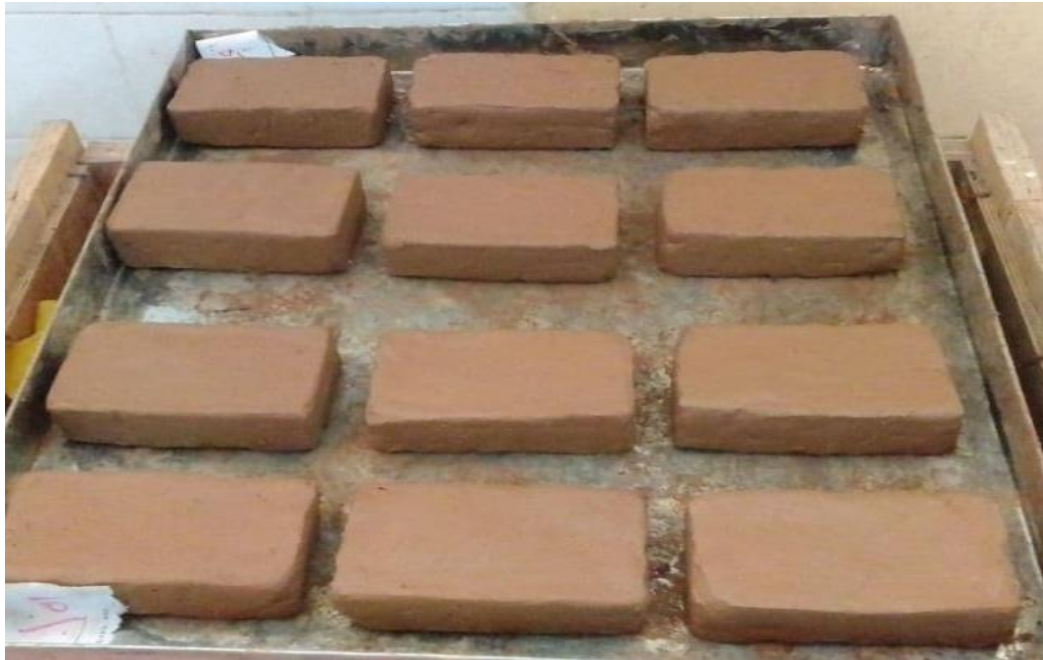
B. Preparation of sample and equipments for sample mixing and molding



C. Mixed sample for molding



D. Molded sample with different sludge to clay proportion

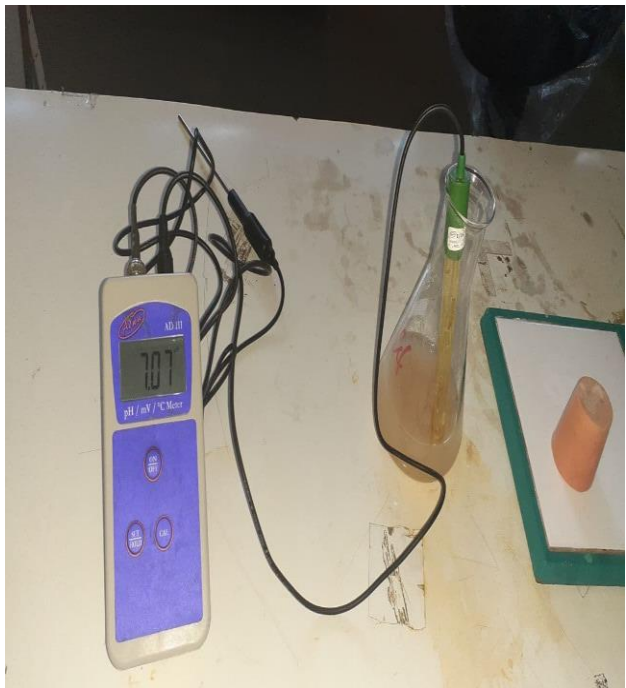


E. Natural air dried samples



F. Fired samples with different proportion and firing temperature

Appendix C: Images for the laboratory work



a. pH Measuring Instrument



b. Weighing Balance



c. Curcible



d. Muffen Furnance



e. Micro Wave Digester



f. High Performance Oven



g. Shaking Device



h. ICP-OES

PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS A PARTIAL SUBSTITUTE FOR CLAY | 2021

Appendix D: Results of the Final Product Characterization

100% brick at 800°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	20.41	10.0	9.1	374.1	332.8	11.0	1.78
2	23.24	9.2	8.0	371.2	334.7	9.8	1.80
3	23.73	9.5	7.2	376.0	342.9	8.8	1.78
Average	22.46	9.57	8.10	373.77	336.80	9.87	1.79

100% brick at 900°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	26.07	8.5	10.7	369.8	323.6	12.5	1.84
2	25.84	8.6	11.1	372.5	330.1	11.4	1.82
3	27.45	8.2	10.9	367.5	327.4	10.9	1.92
Average	26.45	8.43	10.90	369.93	327.03	11.60	1.86

100% brick at 1000°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	29.79	7.1	12.5	375.4	327.9	12.7	1.9
2	30.41	7.4	12.9	368.2	320.4	13.0	1.88
3	30.53	6.6	12.1	363.3	323.2	11.04	1.95
Average	30.24	7.03	12.50	368.97	323.83	12.25	1.91

PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS A PARTIAL SUBSTITUTE FOR CLAY | 2021

10% sludge brick at 800°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	19.87	13.9	5.0	359.2	308.1	14.2	1.66
2	19.39	13.5	4.6	368.1	317.3	13.8	1.65
3	19.83	14.1	6.0	361.9	307.5	15.0	1.57
Average	19.70	13.83	5.20	363.07	310.97	14.33	1.63

10% sludge brick at 900°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	25.61	10.9	7.5	364.1	310.1	14.8	1.6
2	25.63	10.5	7.2	356.3	303.9	14.7	1.70
3	25.30	11.8	8.4	358.2	302.7	15.5	1.62
Average	25.51	11.07	7.70	359.53	305.57	15.0	1.64

10% sludge brick at 1000°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	27.24	9.1	8.9	358.3	296.3	17.3	1.70
2	27.05	9.6	9.3	354.2	301.4	14.9	1.65
3	27.13	8.8	8.6	362.0	295.9	18.3	1.75
Average	27.14	9.17	8.93	358.17	297.87	16.83	1.70

**PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS
A PARTIAL SUBSTITUTE FOR CLAY**

2021

15% sludge brick at 800°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	17.62	17.2	4.7	350.4	279.8	20.1	1.53
2	17.98	18.3	5.3	353.5	277.9	21.4	1.76
3	17.35	18.8	5.7	356.9	288.8	19.1	1.24
Average	17.65	18.10	5.23	353.60	282.17	20.2	1.51

15% sludge brick at 900°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry(g)	Weight after ignition in muffle furnace(g)	WLOI (%)	Bulk Density (g/cm ³)
1	24.10	14.1	5.7	347.6	264.5	23.9	1.54
2	24.13	14.7	6.1	345.0	267.8	22.4	1.55
3	24.17	15.5	6.3	355.1	271.0	23.7	1.56
Average	24.13	14.77	6.03	349.23	267.77	23.33	1.55

15% sludge brick at 1000°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	25.87	12.5	7.3	344.7	256.2	25.7	1.56
2	25.84	12.4	7.8	348.2	258.9	25.6	1.57
3	25.82	12.3	7.7	341.7	253.4	25.8	1.60
Average	25.84	12.40	7.60	344.87	256.17	25.7	1.58

PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS A PARTIAL SUBSTITUTE FOR CLAY | 2021

20% sludge brick at 800°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	14.46	19.0	4.3	335.4	250.1	25.4	1.37
2	14.54	19.4	4.4	336.7	248.3	26.3	1.48
3	14.47	19.5	4.2	333.6	246.8	26.0	1.54
Average	14.49	19.30	4.30	335.23	248.40	25.9	1.46

20% sludge brick at 900°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	21.14	15.7	5.4	329.7	243.2	26.2	1.58
2	21.01	16.8	6.0	334.7	238.9	28.6	1.42
3	21.37	16.1	4.8	324.9	241.1	25.8	1.50
Average	21.17	16.20	5.40	329.77	241.07	26.87	1.50

20% sludge brick at 1000°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry(g)	Weight after ignition in muffle furnace(g)	WLOI (%)	Bulk Density (g/cm ³)
1	23.16	15.6	6.5	320.3	226.9	29.2	1.47
2	23.26	14.2	5.8	319.3	238.7	25.2	1.59
3	23.10	14.8	6.4	322.5	232.6	27.9	1.53
Average	23.17	14.87	6.23	320.70	232.73	27.43	1.53

PRODUCTION OF ECO BRICKS USING TANNERY SLUDGE AS A PARTIAL SUBSTITUTE FOR CLAY | 2021

25% sludge brick at 800°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace(g)	WLOI (%)	Bulk Density (g/cm ³)
1	9.20	21.5	3.8	316.5	221.1	30.1	1.40
2	9.14	21.1	4.2	320.7	225.6	29.7	1.36
3	9.29	20.5	3.2	312.5	228.7	26.8	1.48
Average	9.21	21.03	3.73	316.57	225.13	28.87	1.41

25% sludge brick at 900°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	16.34	17.5	4.5	310.2	219.3	29.3	1.40
2	16.30	17.7	4.6	314.4	219.4	30.2	1.45
3	16.32	17.5	4.7	305.6	219.8	28.1	1.48
Average	16.32	17.57	4.60	310.07	219.50	29.2	1.44

25% sludge brick at 1000°C for 6hr

Bricks samples	Comprehensive strength (MPa)	Water absorption (%)	Firing Shrinkage (%)	Weight after oven dry (g)	Weight after ignition in muffle furnace (g)	WLOI (%)	Bulk Density (g/cm ³)
1	20.12	16.0	6.1	304.7	213.2	30.0	1.43
2	20.16	15.9	6.4	305.5	211.2	30.9	1.51
3	20.17	16.2	5.9	304.0	208.6	31.5	1.43
Average	20.15	16.03	6.13	304.73	211.00	30.8	1.54