

# **USE OF DYEING INDUSTRY SLUDGE THROUGH PRODUCTION OF BRICKS**

**S. M. HABIB ULLAH**



**DEPARTMENT OF CIVIL ENGINEERING  
BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY  
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# **USE OF DYEING INDUSTRY SLUDGE THROUGH PRODUCTION OF BRICKS**

By

**S. M. HABIB ULLAH**  
(Student ID: 0413042304P)

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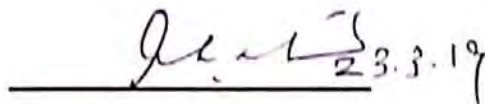
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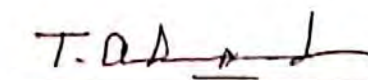
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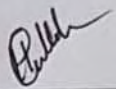
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## DECLARATION

It is hereby declare that this thesis has been performed by the author under the supervision of Dr. Syed Ishtiaq Ahmad, Professor of the Department of Civil Engineering, BUET. Neither this thesis nor any part of it has been submitted elsewhere for the award of any degree or diploma.

March 2019

  
\_\_\_\_\_  
S. M. HABIB ULLAH

**DEDICATED  
TO  
MY BELOVED PARENTS**

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### **List of Abbreviations**

AAS	Atomic Absorption Spectrophotometer
AASHTO	American Association of State Highway and Transportation Officials
ASTM	American Society for Testing and Materials
BDS	Bangladesh Standard
BEPZA	Bangladesh Export Processing Zone Authority
BOD	Biological Oxygen Demand
CETP	Central Effluent Treatment Plant
COD	Chemical Oxygen Demand
DO	Dissolved Oxygen
ETP	Effluent Treatment Plant
DOE	Department of Environment
GFAAS	Graphite furnace Atomic Absorption Spectrophotometer
LL	Liquid Limit
OMC	Optimum Moisture Content
PI	Plasticity Index
PL	Plastic Limit
DS	Dyeing Sludge
TCLP	Toxicity Characteristics Leaching Procedure
TDS	Total Dissolved Solid
TOC	Total Organic Carbon
UNEP	United Nations Environment Program
UNIDO	United Nations Industrial Development Organizations
USEPA	United States Environmental Protection Agency
XRF	X-ray fluorescence

SRDI	Soil Resource Development Institute
SDF	Savar Dyeing and Finishing Ind. Ltd.
WTO	World Trade Organization
RMG	Ready-Made Garments
BTMA	Bangladesh Textile Mill Association
DWA	Department of Women Affairs
3R	Reduce, Reuse and Recycle

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## **Abstract**

Dyeing Sludge (DS) has the potential to become a serious environmental burden for Bangladesh in future with very limited safe disposal options. One potential way for the disposal and stabilization of DS is by incorporating it in clay bricks. However, it must be ensured first that the essential engineering properties of bricks are not diminished and leaching of harmful materials does not occur because of incorporation of sludge as a brick constituent. In this study, laboratory experiments have been performed to assess the engineering properties of sludge-amended clay bricks as well as to determine the leaching potential of toxic heavy metals from such bricks. The goal was to explore the viability of DS as a brick constituent and determine the effectiveness of such bricks as a safe option for sludge management.

Clay bricks were prepared with different proportions of sludge (10%, 20%, 30%, 40% and 50% by weight) in a brick kiln and its suitability as a construction material was assessed based on its strength, water absorption, firing shrinkage, weight loss on ignition and bulk density. Results of the tests indicated that the sludge proportion is the key factor in determining the brick quality. Increasing the sludge content in bricks resulted in a decrease in compressive strength. The compressive strength of DS bricks reduced considerably from 25.6 MPa to 9.93 MPa when DS content increased from 0% to 50%. The addition of 50% sludge into the mixture reduces strength approximately 61.2% compared with the control bricks though all the DS bricks have been found to meet both the Bangladesh and ASTM requirements for building construction material.

Water absorption of bricks depends on sludge content also. Water absorption was increased with the increase of sludge addition raw 11.4% bricks absorbed 12.8% of dry weight where brick that is made of 50% DS absorb 25.2% of water.

Also, total shrinkage and weight loss in burning were compared with raw brick which shows that total shrinkage did not vary that much however the weight loss in burning varied from 10.8% to 24.3% in case of raw brick to 50% DS brick.

The TCLP test results indicated that the leaching behavior of several targeted heavy metals (As, Cd, Cr, Cu, Hg, Ni, Pb and Zn) analyzed in this study for all samples have been found to be insignificant and far below the USEPA regulatory limits. the



leaching concentration of As, Cr, Cd, Cu and Pb are reduced by 92.6%, 97.5%, 91.3%, 85.6% and 96.9%, respectively in case of 10% sludge amended bricks with respect to the original sludge.

The recommended proportion of sludge in brick making can be 10% (by dry weight) for producing good quality bricks. Also 20% and 30% sludge brick can be a viable option for light weight brick. Results from this study indicate that textile dyeing sludge can be sustainably stabilized in clay bricks.

## **Chapter 1**

### **Introduction**

#### **1.1 Background**

The textile and clothing industries provide the single source of growth in Bangladesh's rapidly developing economy. (UNFPA, 2012) Exports of textiles and garments are the principal source of foreign exchange earnings. By 2002 exports of textiles, clothing, and ready-made garments (RMG) accounted for 77% of Bangladesh's total merchandise exports (WTO, 2008). In 1972, the World Bank approximated the gross domestic product (GDP) of Bangladesh at US\$6.29 billion, and it grew to \$173.82 billion by 2014, with \$31.2 billion of that generated by exports, 82% of which was ready-made garments. As of 2016, Bangladesh held the 2nd place in producing garments just after China (Latif et al., 2016). Bangladesh is the world's second-largest apparel exporter of western (fast) fashion brands. Only 5% of textile factories are owned by foreign investors, with most of the production being controlled by local investors (Textiles Intelligence, 2003). In the financial year 2016-2017 the RMG industry generated US\$28.14 billion, which was 80.7% of the total export earnings in exports and 12.36% of the GDP; the industry was also taking on green manufacturing practices (Hossain et al., 2017).

There are 1461 mill members in Bangladesh where, yarn manufacturing mills are 425, fabric manufacturing mills are 796 and 240 dyeing-printing-finishing mills. (BTMA, 2018)

The effluents from almost all industries of the country are directly or indirectly discharged into canals and rivers. Due to continuous disposal of wastewater into water bodies, the surface water quality throughout the country is getting gradually deteriorated because of the mixing of various chemical pollutants of the effluent with water. (Jolly et al., 2009)

Many different groups of chemical substances are used in the textiles sector, including dyes, solvents, optical brighteners, crease-resistance agents, flame retardants, heavy metals, pesticides, and antimicrobial agents. They are used in dyeing, printing, finishing, bleaching, washing, dry cleaning, weaving slashing/sizing, and spinning. Respiratory and skin sensitizers can be found in the textiles industry, for example textiles fibres, reactive dyes, synthetic fibres, and formaldehyde. The exposure of

workers to dusts from material such as silk, cotton, wool, flax, hemp, sisal, and jute can occur during weaving, spinning, cutting, ginning, and packaging. (Islam et. al., 2011)

Heavy metals frequently found in sludge include Cd, Zn, Cu, Cr, Co, Pb, Mn, Ni, Hg, etc. (Islam et al., 2009). These metals are very harmful, because of their non-biodegradable nature, long biological half-lives and their potential to accumulate in different body parts (Manahan, 2005; Wilson et al., 2007; Singh et al., 2004).

The only option practiced in Bangladesh for sludge disposal is landfilling. The environmental impacts of landfill are the pollution of air, water and soil. The uncontrolled production of landfill gases such as methane, carbon dioxide, traces of non-methane and volatile organic carbons leads to global warming. The most serious environmental impact of sludge disposal to landfill is contamination of local groundwater by the generated leachate. The leaching potential of the raw sludge from the effluent treatment plant is very high (Iqbal et.al. 2013, Abed et.al. 2004)

The land-filling scenario is now under increased scrutiny, mainly because of highly-polluting emissions, the low recovery of energy and the limited capacity of available land-fill sites (Swarnalatha et al., 2006; Kirk et al., 2002).

Moreover, the thermal treatment of sludge involves incineration, gasification, and pyrolysis as a means of disposal, whilst also recovering energy from waste. These are costly and may contribute to air pollution and the residues of the process still have to be disposed of, but this is more easily achieved than with untreated sludge (Basegio et al., 2002).

Techniques were adopted by several researcher to treat or stabilize hazardous waste of textile industry by using as partial replacement of clay in fired clay brick and cement in concrete and concrete block. (Baskar et al., 2006, Balasubramaniam et al., 2006, Jahagirdar, et al., 2012)

Stabilization indicates the process or techniques which reduce the harmful effect of waste chemically and release the residual in a safe and environment friendly manner. This means the treated or converted product should have the heavy metal and other hazardous parameters in allowable limit.

The solidified product can be disposed off to a secured landfill site or it can be reused as construction materials if the resulting product meets the specific strength

requirement and the leaching of toxic pollutants within acceptable limits (Rouf et al., 2003; Rahmat, 2001).

Several studies have shown that textile dyeing sludge can be effectively stabilized in construction materials such as brick, concrete and ceramic tiles (Jahagirdar, et al., 2013). Here in this study, experiments are demonstrated to show that dyeing sludge can be stabilized in clay bricks, one of major construction materials in Bangladesh.

In Bangladesh, around 7,000 brick kilns produce 23 billion pieces of burnt clay bricks from around 3 billion cubic feet of topsoil annually (DoE, 2017).

Due to inadequate supply of stone, brick has become the major building material for construction industry of the country and the demand will continue to rise in future. So, exploring the brick manufacturing industry as a potential option to stabilize the huge amount of dyeing sludge in Bangladesh can be a feasible alternative for commercial application.

The characteristics of sludge-incorporated clay bricks were evaluated with respect to its engineering properties as well as its environmental implications. Clay brick specimens were prepared with different proportions of sludge in field conditions and its suitability as an engineering material was assessed based on its strength, deformation, shrinkage, absorption and Leaching characteristics to determine the efficiency of the stabilization technique against the discharge of heavy metals in the environment.

## **1.2 Objectives With Specific Aims of The Thesis:**

Specific objectives of the thesis are:

- i. To assess the environmental viability of stabilization of harmful chemicals and heavy metals present in dyeing industry sludge by solidification with clay mix in brick production.
- ii. To assess the effect of using sludge (with the clay mix) on the strength and other physical properties of brick.
- iii. To propose an optimum clay-sludge mix proportion that can be used for brick manufacturing without compromising quality of bricks and thereby establish effective way to recycle sludge in environmentally friendly way.

### **1.3 Outline of Methodology/ Experimental Design**

Raw materials (Dyeing sludge) were collected from a dyeing effluent treatment plant.

Upon collection, sludge sample was oven dried for 24 hours at 105°C. Basic physicochemical characteristics, including moisture content, pH, and organic compound were analyzed. Heavy metal content i.e. Arsenic, Cadmium, Chromium, Copper, Mercury, Nickel, Lead and Zinc was determined by acid digestion with a HNO<sub>3</sub>: HCl volume of ratio of 1:3 followed by analysis using AAS (Shimadzu AA 6800). The Toxicity Characteristics Leaching Procedure (TCLP) in accordance with USEPA Method 1311 (USEPA, 1992) was used to determine toxic characteristics of sludge.

Clay sample was collected from a typical brick manufacturing industry. XRF test was conducted to determine the chemical composition of the DS.

Total 1000 bricks samples (length 254 mm. width 127 mm and height 76 mm) of sludge-clay mixture in varying proportions (10%, 20%, 30%, 40% and 50%) at field condition prepared in commercial brick factory (Brick kiln). A clay-only mixture sample was prepared as a reference specimen. After 7 Days of natural drying, these samples were burnt in brick kiln for 3 weeks at 2100 °F (Approximately). The produced bricks then went through a number of tests including firing shrinkage, weight loss on ignition, water absorption, and compressive strength according to ASTM C67 to determine a suitable condition for producing qualified bricks that meets the BDS 208 (2009) standards.

Leaching test of all these burnt brick samples were carried out by TCLP in accordance with USEPA 1311.

### **1.4 Organization of the Thesis**

This study shows the potential of dyeing sludge to be incorporated in the clayey bricks in Bangladesh. Chapter 2 introduces a brief review of dyeing industry including a short description regarding techniques of wastes management and disposals. The latter part of Chapter 2 summarizes the previous work that was done regarding utilization of different types of sludge in the form of ceramic tiles, bricks and concrete. Chapter 3 describes the detail methodology that was adopted in this study. Chapter 4 deals with the results obtained in this study. Chapter 5 is on conclusion where overall findings, limitation and directions for future work have been

discussed. At last, Appendix A contains the average results of different mechanical and physical properties of DS bricks. Appendix B contains BDS 208 (2009) standards for clay bricks.

## **Chapter 2**

### **Literature Review**

This chapter presents sludge management in Bangladesh, characterization of Dyeing wastewater, treatment technology of dyeing wastewater, dyeing sludge management and disposal practices, brick manufacturing technology and previous studies on stabilization of sludge through fired clay bricks.

#### **2.1 General Considerations and Requirements for Sludge Management**

The major priority of these standards and guidelines for sludge management in Bangladesh are to ensure that human health and the environment are strictly protected from any negative impacts of sludge management.

According to the “National 3R Strategy” and the recommendations by the “Textile Sludge Study in Bangladesh” as well as the DWA mission report of phase 1 this guidance will follow the required principles in waste management. An important principle for guidance is the waste hierarchy of reducing, reusing and recycling (3R strategy) followed by disposal. This means that these measures shall be executed according to the state of technology and ecological feasibility before the waste has to be disposed in an end-of-pipe facility. Another central concept in this waste management guideline is to separate and segregate generated waste streams at source and, if possible, treat them directly on site which is also established in the European and German law and executed accordingly.

Challenges for sludge management in Bangladesh, among others, are the following:

- i. Waste streams are not separated. Complex mixtures are difficult to classify and in consequence, it can be a problem to define an appropriate treatment/disposal route.
- ii. An analysis for all potential pollutants, especially in complex mixtures of wastewater from industry, presents a huge challenge. Besides, the high cost of analysis can result in difficulties guaranteeing the compliance with limit values for certain disposal routes.
- iii. Reusing and recycling technologies may not be available, or may be uneconomic or not having sufficient capacities for the rising amount of waste, specifically sludge being produced.

- iv. Some types of disposal facilities, e.g. incineration or land fill sites do not exist or do not have the necessary capacities or standards for some types of waste (e.g. hazardous) in Bangladesh; suitable disposal facilities are a prerequisite for sustainable sludge management and before a guideline for sludge management can be implemented such facilities or other disposal routes guaranteeing a safe and final disposal need to be planned and built.

## 2.2 Classification of Sludge

Depending on the origin of the wastewater as per department of environment Bangladesh, sludge can be classified as

Category A: Municipal sludge including comparable sludge

Category B: Sludge from industry including sludge from CETP

Category C: Sludge from industry including sludge from CETP belonging to the category of hazardous waste.

In cases where wastewater producing a sludge classified as Category C is mixed with other types of wastewater and treated together, for example in a CETP, the resulting sludge is to be classified as Category C. In cases where wastewater producing a sludge classified as Category B is mixed with wastewater producing sludge classified as Category A and treated together, the resulting sludge is to be classified as Category B. If different classes of sludge are mixed during collection, transport, treatment or other stages of sludge management the method of classification described above is to be applied.

Category A + Category B = Category B

Category A + Category C = Category C

Category B + Category C = Category C

Category A + Category B + Category C = Category C

Depending on the classification of the sludge different management options are permissible. In view of the protection of the environment, it is forbidden to mix Category A sludge with Category C sludge.

## 2.3 Sludge Management Options

Sludge can be treated in different way depending on the source of sludge and the chemical content. Before the sludge is disposed of, it is required to apply pre-treatment in order to implement the “3R-principle” (reduce, reuse and recycle) which



is a central component of the National 3R Strategy for Bangladesh (DoE). The main goal of pre-treatment is to minimize the volume and the organic matter of the sludge in order to reduce waste volume that must be disposed of and to enable a safer disposal. If the waste has to be transported e.g. to a central disposal facility, a pre-treatment would ensure better characteristics.

Possible treatment types for industrial wastewater and sludge from industrial wastewater are the following:

- i. mechanical treatment: e.g. sedimentation, thickening
- ii. Physical/chemical treatment: e.g. use of ferrous sulphate, lime and polyelectrolyte in coagulation, flocculation,
- iii. ozonation, chemical oxidation (wet oxidation or wet peroxidation), adsorption of non- biodegradables on activated carbon
- iv. biological treatment: e.g. (aerobic) activated sludge treatment, anaerobic digestion
- v. further sludge treatment: e.g. dewatering and drying by use of several aggregates (filter presses and centrifuges)

### 2.3.1 Overview of Management Options

Depending on the waste class, as per department of environment Bangladesh, sludge can be managed by different alternatives. Table 2.1 summarizes the overview of the sludge management options based on the waste class.

Table 2.1. Sludge Management Options as per Waste Class

Management option	Waste class		
	A	B	C
Anaerobic digestion (co-fermentation)	X1	X1	**
Aerobic digestion (composting)	X1		
Agricultural use	X		
Controlled landfill *	X	X	X
Thermal incineration	X1	X1	X1
Land application (filling material e.g. for flood prevention)	X	X2	**
Recycling in brick, cement or asphalt making	X	X3	**

1Residues will remain that have to be disposed of, fulfilling the requirements applicable to the category, on an alternative route. E.g. By landfill.

2Inert material (low organic matter required)

3 Availability and capacity limited by local conditions. Accepted sludge volume limited due to a loss of compressibility of the product

\* Requirements for the landfill class vary depending on category of the sludge.

\*\*If the producer provide evidence that sludge categorized as category C sludge and it does not possess any hazardous characteristics; in this case it may be categorized as category B sludge and the management options of anaerobic digestion (co-fermentation), land application ( filling material e.g. for flood prevention), recycling in brick, cement or asphalt making are permissible.

## **2.4 Description of Management Options Including Design Parameter**

Depending on the waste class, different sludge management options applied to manage the sludge as mentioned in Table 2.1. Different options are described here with the respective design parameters.

### **2.4.1 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.

Especially when sludge from industries is 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.

For reasons of climate protection, the organic matter in the residue should be as low as possible to prevent uncontrolled biodegradation leading to emissions and leachate when it is used in landfill application. Requirements for landfill application are described in Table 2.3.

Anaerobic digestion is not permissible for Category C 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.

### **2.4.2 Aerobic digestion (composting)**

Composting can be used to produce fertilizer for application in agriculture. To gain 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, co-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 (phyto-) toxic substances.

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

#### 2.4.3 Agricultural use:

Sludge can be used in agricultural land if it fulfills certain criteria. For this option sludge should contain minimum of the heavy and toxic metals to avoid pollution. Table 2.2 shows the limiting values of heavy metals up to which sludge can be used in agricultural purposes.

Table 2.2. Parameter limits of sludge for use as compost/fertilizer<sup>2</sup>

Parameter	in sludge mg/kg dry substance	in soil* mg/kg dry substance
Pb (Lead)	900	100
Cd (Cadmium)	10	1.5
Cr (Chromium)	900	100
Cu (Copper)	800	60
Ni (Nickel)	200	50
Hg (Mercury)	8	1
Zn (Zinc)	2500	200

\*Soil of the agricultural land before application of sludge  
The quantity is limited:

< 3 t dry substance sewage sludge per ha in 3 years

< 10 t dry substance sludge compost per ha in 3 years

<sup>2</sup> Sources: German Sewage Sludge Ordinance, July 2002

#### 2.4.4 Controlled landfill

Controlled landfill describes the possibility to deposit the sludge in the ground. To establish a controlled landfill site, it is necessary to obtain prior approval from the DoE, which is responsible for granting an environmental clearance certificate.

The sludge has to be stabilized by reducing the organic fraction to prevent uncontrolled degrading processes. 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 different kinds of waste depending on its hazardous potential or pollutants, which have different requirements for the construction, the pollutants (measure in leachate) and monitoring.

Basic requirements for the location of a landfill site:

- a) The over flooding level should be  $> 2.0$  m of the maximum expected water level of the surrounding water bodies
- b)  $> 500$  m distance to populated areas
- c) no construction in protected areas
- d) no construction in flood plains and areas with a high risk of natural disasters
- e) the underground has to resist mechanical stresses, has to hold back or prevent leachate and pollutants
- f) water impermeability
- g) buoyancy safety has to be considered

Controlled landfill sites in Bangladesh are proposed to be categorized in 3 classes, inert landfill (class 0), non-hazardous landfill (class 1 and class 2) and hazardous landfill (class 3) depending on requirements for the acceptance of waste and for the construction. The definition of the landfill classes is based on the types of waste (i.e. inert, non-hazardous and hazardous) that may be accepted and the binding limit values the waste in question must comply with. These are listed in Table 2.3. It is recommended that the operator shall maintain a list of approved wastes that can be disposed.

Table 2.3. Landfill classes<sup>3</sup>

Designation	Unit	Landfill class 0 (inert)	Landfill class 1 (non-hazardous)	Landfill class 2 (non-hazardous)	Landfill class 3 (hazardous and non-hazardous)
Investigation on original substance					
water soluble part	%	0.4	3	6	10
extractable lipophilic substances	%	0.1	0.4	0.8	4
total BTEX <sup>4</sup>	mg/kg d.s.	6			
total PCB's <sup>5</sup>	mg/kg d.s.	1			
total PAK's <sup>6</sup>	mg/kg	30			

MKW7 C10 -C40	d.s. mg/kg d.s.	500			
acid neutralization capacity	mmol/kg				
caloric	kJ/kg	6000	6000	6000	6000
breathability ( <sup>8</sup> AT <sub>4</sub> )	mg/g O <sub>2</sub> d.s.	5	5	5	5

3Landll class 0, 1, 2 and 3 are over ground sites characterized by types of wastes (i.e. inert, non-hazardous and hazardous) and its limiting values allowed to be disposed as provided in the table. Detailed information about construction and maintenance of landfill of each class can be found in German Landfill Ordinance

4BTEX = Benzol, Toluol, Ethylbenzol and ortho-xylol

5PCB = Polychlorinated biphenyl

6PAK = Polycyclic aromatic hydrocarbons

7MKW = Petroleum-derived hydrocarbon

<sup>8</sup>AT<sub>4</sub> = Breathability according to DIN ISO 16072

Designation	Unit	Landfill class 0 (inert)	Landfill class 1 (non-hazardous)	Landfill class 2 (non-hazardous)	Landfill class 3 (hazardous and non-hazardous)
Investigation on dry residue					
total organic carbon (TOC)	%	1	1	3	6
loss on ignition 550°C	%	3	3	5	10
Leachate with distilled water					
pH*	mg/l	5.5 - 13	5.5 - 13	5.5 - 13	4.0 - 13
weak acid dissociable cyanide	mg/l	0.01	0.1	0.5	1
Fluoride (F)	mg/l	1	5	15	50
Phenols	mg/l	0.1	0.2	50	100
dissolved organic carbon (DOC)**	mg/l	50	50	80	100
Arsenic (As)	mg/l	0.05	0.2	0.2	2.5
Lead (Pb)	mg/l	0.05	0.2	1	5
Cadmium (Cd)	mg/l	0.004	0.05	0.1	0.5
Copper (Cu)	mg/l	0.2	1	5	10
Nickel (Ni)	mg/l	0.04	0.2	1	4
Mercury (Hg)	mg/l	0.001	0.005	0.02	0.2
Zinc (Zn)	mg/l	0.4	2	5	20
Barium (Ba)	mg/l	2	5	10	30
Chromium (Cr), total	mg/l	0.05	0.3	1	7

Molybdenum (Mo)	mg/l	0.05	0.3	1	3
Antimony (Sb)***	mg/l	0.006	0.03	0.07	0.5
Antimony C0 value	mg/l	0.01	0.12	0.15	1
Selenium (Se)	mg/l	0.01	0.03	0.05	0.7
Chloride (Cl)	mg/l	80	1500	1500	2500
Sulphate (SO4)****	mg/l	100	2000	2000	5000

\*Divergent pH values alone shall not represent an exclusion criterion. Where pH values are too high or too low, the cause shall be examined.

\*\* The allocation value for DOC shall also be satisfied if the waste or the landfill replacement construction material fails to satisfy allocation value at its own pH value, but does satisfy the allocation value at a pH value between 7.5 and 8.0. With the approval of the competent authority, excessive values of DOC up to 200 mg/l shall be permissible if the public welfare is not impaired and up to max. 300 mg/l if they are based on inorganically bound carbon.

\*\*\* Antimony values that exceed the values given for “Antimony (Sb)” shall be permissible if the Concentration value of the percolation test provided for “Antimony C0 Value” is not exceeded.

\*\*\*\* Excessive sulphate values up to 600 mg/l shall be permissible if the Co value of the percolation test does not exceed 1,500 mg/l where the liquid/solid ratio = 0.1 l/kg.

#### 2.4.5 Thermal (co-) incineration

Currently available and applied co-incineration technologies<sup>9</sup> are listed below. The sludge coming from the biological treatment with volatile moisture content is added to the incineration chamber.

<sup>9</sup>Reference Document on the Best Available Techniques for Waste Incineration, August 2006

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.

A range of incineration methods, as described below, may be applied.

- a) Dried sewage sludge (~90% d.s.) is blown as dust into the furnace.
- b) Drained sewage sludge (~20 - 30% d.s.) is supplied separately through sprinklers into the incineration chamber and distributed on a grate. The sludge is integrated into the bed material by overturning the waste on the grates. Operational experiences show up to 20 mass-% sludge (at 25% d.s.). Other

experiences have shown that if the sludge ratio is too high (e.g. >10 %), high fly ash content or un-burnt material in bottom ash may occur.

- c) Drained, dried or semi-dried (~50 - 60% d.s.) sludge is mixed with the remaining waste or fed together into the incineration chamber. This can occur in the waste bunker through targeted doses by the crane operator or controlled in a feeding hopper by pumping dewatered sludge into the hopper or by spreading systems into the bunker.

The average dry matter content identified by the Textile Sludge Study in Bangladesh is about 30 to 40%. So it can be characterized as drained sewage sludge and an appropriate incineration technique can be chosen.

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 ( $\text{Cr}^{+3}$ ) to the carcinogenic chromium ( $\text{Cr}^{+6}$ ). These have a harmful impact on human health and the environment and therefore, the installation of expensive pollutant filters are required. In co-incineration in existing plants these filter systems need to be given as well.

The temperature has to be at least over 800°C to avoid noxious smells. The water content of the sludge has an impact on the efficiency and in some cases an additional drying step is required. An absence of control systems would only lead to a shift of pollutants which violates the principle of sustainability.

The following value limits to exhaust emissions by incineration plants need to be observed to guarantee a safe incineration and to avoid harmful impacts on human health.

Table 2.4. Limiting values for exhaust parameters of incineration plants

Substances, or group of substances	Concentration g/m <sup>3</sup>	Averaging period for the calculation of the value*	Permissible excess frequency per year**
Benzene	5	Year	-

Lead and inorganic compounds contained in suspended particulate matter (PM10), to be indicated as Pb	0.5	Year	-
Particulate matter (PM10)	40	Year	-
	50	24 hours	35
Sulphur dioxide	50	Year	-
	125	24 hours	3
	350	1 hour	24
Nitrogen dioxide	40	Year	-
	200	1 hour	18
Tetrachloroethylene (PER)	10	Year	-

\* Integrated time used to calculate the standard. For example, for particulates, the lter will collect material for 24 hours anthen analyzed. The annual integration will be an integration of the daily values.

\*\* Refers to the number of times the concentration measured can exceed the standard limiting value. Similar permission is allowed for ambient air in the Bangladesh Air Quality Standards (Bangladesh Govt. Gazette S.R.O. No: 220-Law/2005 of 16 July 2005).

These averaging times reflect exposure periods of public health concern. Stack sampling duration may be different from averaging period. Stack tests only provide a snapshot of emissions during the period of the test. The methods may include continuous monitoring to measure actual emissions; or extrapolation of results from short-term emissions tests.

- For periodic emission tests, the sampling period should ideally be of one hour.
- For 24 hour averaging period under continuous measurement, average of all valid hourly averages will be used to calculate concentration. For 24 hour averaging period under periodic measurement, average of three one-hourly averages taken on the same day will be used to calculate concentration.
- For calculating yearly concentration, at least 3 samplings should take place each year. The following table 2.5 outlines specific instructions for each of



the substances. As an example, yearly concentration of Sulphur Dioxide can be calculated using average of three 24-hour sampling averages or using average of three one-hour sampling averages taken at three different times over a year.

Table 2.5. Specific instructions to determine short-term emission for different substances.

Substances, or group of substances	Using this average time (year)
Benzene	3-run average (1 hour minimum sample time per run)
Lead and inorganic compounds contained in suspended particulate matter (PM <sub>10</sub> ), to be indicated as Pb	3-run average (collect a minimum volume of 1 dry standard cubic meters)
Particulate matter (PM <sub>10</sub> )	3-run average (collect a minimum volume of 1 dry standard cubic meters)
Sulphur dioxide	3-run average (1 hour minimum sample time per run)
Nitrogen dioxide	3-run average (1 hour minimum sample time per run)
Tetrachloroethylene (PER)	3-run average (1 hour minimum sample time per run)

Other techniques like pyrolysis or gasification to generate useful products are under development and may be used as and when suitable.

#### 2.4.6 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. Requirements for land application depend on the specific use and should be decided on an individual basis only with permission of the

responsible authority in agreement with the Soil Resource Development Institute (SRDI) and the Department of Environment (DoE).

#### **2.4.7 Recycling**

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 clay is deficient 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 use of sludge from physico-chemical treatment seems more suitable than from a biological treatment as the organic content from biological treatment may be too high for optimal use in production of bricks when replacing high amount of raw material.

The amount of replaced raw material can vary from 10 to 70% but a decrease in compressive sludge depending on the sludge content has to be considered. High sludge content may be possible if the product is used in non-structural-building.

The application of this disposal route depends on the demand of brick making companies. If they cannot match with the quantities of the sludge production this alternative is limited and additional options need to be found.

The recycling of sludge from class C needs a specific evaluation whether the harmful components are stable and long term bound into the final material.

#### **2.5 Available Techniques for Wastewater Treatment of Textile Industry**

A wide range of waste streams and appropriate treatment has been covered and described by experts. The treatment options named below are examples; other methods not described here may be equally suited.

- i. Origin of wastewater: Mixed textile wastewater with partial recycling of water and on-site treatment:

Wastewater from textile dyeing industry must be treated by the effluent treatment plant (ETP). The treated wastewater can be recycled.

The wastewater treatment contains the following stations:

- a) Equalization/neutralization,
- b) activated sludge treatment with added lignite coke powder for bio-degradation,

- c) absorption stage with added lignite coke powder to remove dyestuffs and other hardly or non-biodegradable compounds,
- d) flocculation/precipitation and removal of the sludge by flotation (aluminium sulphate and an anionic polyelectrolyte are added as flocculants),
- e) filtration to remove suspended solids,
- f) reverse osmosis plant (option)

Sludge is taken from the activated sludge treatment and from the flotation. It gets dewatered in a thickener and decanter and is then thermally regenerated in a rotary kiln (kiln is about 450 °C) and the flue-gas is submitted to post-combustion (about 850 °C).

- ii. Origin of wastewater: Recycling of residual dyestuff from padding liquors and printing paste residues

In the dyeing and printing process printing pastes and padding liquors (for dyeing) with a high organic content are used. The residues can be treated in anaerobic digesters preferably in co-fermentation together with the sludge from the biological treatment from municipal wastewater treatment plants.

In the process the Azo-groups are irreversibly destroyed but will still absorb light and a slight yellowish colour remains.

The water-soluble cleavage products (the ones with sulphonic groups) are present in the water phase and reach the activated sludge treatment both as overflow from the anaerobic digester and as filtrate from sludge dewatering.

The more-substituted naphthalene derivatives are hardly biodegradable and may still be present in the final effluent. For this reason, the supernatant needs to be subsequently treated in an activated sludge system.

Separation of the residual padding liquors from other streams at source in order to keep them concentrated.

Dosage of reactive printing paste should not exceed 10 g/kg sludge (inhibition effects).

Padding liquors and printing pastes with heavy metal-containing dyestuffs should be separated unless the sludge resulting from the anaerobic treatment is incinerated or

disposed of in appropriate landfill, which is also recommended for all kinds of residues from dyestuff treated in anaerobic digestion.

iii. Origin of wastewater: Wastewater containing pigment paste

The technique is membrane based and allows complete reuses of the resulting permeate. The wastewater contains pigment printing pastes with high organic compounds. In the cleaning process coagulation, a precipitation and microfiltration of the precipitation need to be done. The suspended solids in the concentrate are removed in a tube settler by dosage of flocculants.

The produced sludge needs to be treated in a physico-chemical plant before it is sent for incineration.

iv. Origin of wastewater: Mixed and coloured wastewater

As a first pre-treatment step a flocculation/precipitation needs to be done. For a maximized COD elimination and colour removal the precipitation can be removed by a dissolved air flotation after equalization. Therefore, the dosage of flocculants (e.g. for a mixed effluent with COD of ca. 1000 mg/l) is about:

aluminium sulphate:	400 - 600 mg/l
cationic organic flocculants:	50 - 200 mg/l
anionic polyelectrolyte:	1 - 2 mg/l

The use of sulphates is preferred to chloride when the sludge is about to be incinerated. Also, sulphates are easier to remove from water.

**Operational data:**

- a) The expected specific sludge production is 0.7 - 1 kg of dry matter per m<sup>3</sup> of treated wastewater
- b) The expected specific sludge production is 0.5 kg of removed COD per 3 kg sludge produced
- c) The sludge will be dewatered in a chamber filter press up to a dry matter content of 35 - 40%

The resulting sludge is supposed to be incinerated.

v. Origin of wastewater: Partial stream containing organic solvents from a solvent recovery system

For the treatment of textiles organic solvents are sometimes used. In the solvent recovery system, a stream with a high content of PER (Perchloroethylene) (150 – 250 mg/l) is produced and gives rise to an emission of 75 - 125 g/h PER. This substance is known to be carcinogenic (category 2a according to IARC) and therefore a hazardous waste. The solvent is not biodegradable and should not be mixed with the effluent stream to be treated in the biological stage because it would accumulate and last indefinitely. Thus, a segregation and treatment by stripping and absorption through active charcoal cartridges that is periodically changeable and rechargeable is possible. The system is able to ensure a residual PER content into the draining water not higher than 1 mg/l (emission in the water  $\leq 0.5$  g/h PER).

For small effluent streams with a wastewater flow up to 0.5 m<sup>3</sup>/h, an advanced oxidation process (e.g. Fenton process) on site would also be an appropriate treatment.

The remaining sludge has high water content and a high PER concentration which makes it very difficult to manage the waste. Landfill is almost no option because it creates soil or aquifer contaminations and PER is released to the atmosphere in landfill gas.

A solution would be to redesign the distilling group (forced circulation type) and especially the sludge distiller (thin layer evaporator) so that the solvent residue is reduced to 1% and a thick, dry sludge is produced.

A thermal co-incineration is possible when the incineration plant contains filters that can absorb the PER emissions and furan and dioxin emissions that appear in the thermal treatment.

vi.     Origin of wastewater: Wool scouring

In the wool scouring process the wool fibers are washed in a series of bowls with detergent and a high organic effluent stream is produced.

**Evaporator:**

Concentrate/sludge from the evaporator contains suint, a natural grease of sheep that dries on the wool, consisting of fatty matter and potassium salts, as well as dirt and other grease. The presence of suint alters the physical properties of the sludge. It can be liquid at high temperatures and solid at low temperature, which makes it difficult

to handle and dispose evaporator sludge. One option can be to transport the sludge in a heated tank to its final disposal location.

### **Coagulation/Flocculation:**

Sludge from coagulation/flocculation contains only dirt and grease because suint is highly water-soluble and is not flocculated. Depending on the water content the consistency varies from resembling moist earth to semi-liquid mud.

### **Composting and application to agricultural land:**

For this disposal route a pre-treatment, such as composting or anaerobic digestion, is required.

For composting an optimized C:N ratio would be 25 – 30:1 so that wool scour sludge needs an addition of carbon-rich material e.g. green waste, sawdust, woodchip and straw with a grain size up to 50 mm to allow the ready ingress of air. Optimum moisture content of the material for composting is 50 – 60%.

The composting process happens in 2 stages:

- i. earlier thermophilic (45 – 60°C)
- ii. later mesophilic (20 – 45°C)

and is controlled by the aeration.

For industrial sludge enclosed or in-vessel composting systems are recommended.

Advantages:

- a) better control of process
- b) higher temperature → higher rate
- c) control of odors, dust and leachate

The disadvantage is higher investment costs. Additionally, the product must be allowed to mature for several weeks to make it more suitable compost.

Use of wool scouring sludge in brick-making:

Clay for brick-making should contain a certain amount of organic material. The oxidation of this material during the brick-firing process improves the quality of the resulting bricks. Some clay is deficient in organic content and an addition is desirable. Wool scour sludge is excellent in this application.

Besides these methods described above, any other suitable method (if available) could be followed to treat wastewater.

## **2.6 Treatment Methods for Dyeing Wastewater**

Textile dyeing industry is one of the major industries of Bangladesh with the problem of proper treatment and disposal of the produced wastes. Dyeing wastewater effluent can be treated in many different ways. Some of the dyeing industries used ETP on site some others execute pre-treatment or a part pre-treatment or no treatment at all, sending the effluent to a centralized effluent treatment plant. However, a standard treatment process is necessary to prevent the adverse effect of the toxic substances on the environment caused by untreated textile dyeing effluents and sludge's.

One standard treatment process that used by Youngone group to treat their waste water will be described in more detail here:

Youngone Dhaka facility has different Dyeing Section (Jigger, Polyester) and Printing Section whereas produce a huge amount of Effluents. The dyeing plant requires about 1000 m<sup>3</sup> of water per day of which almost 93% drains to the ETP as wastewater for treatment. The wastewater from the dyeing plant is usually found to be with high concentrations of various chemicals and other contaminants, including high levels of pH, BOD, COD, TDS and heavy metals.



Figure 2.1. Aerial view of ETP facility

Youngone Dhaka facility particularly deals with Dyeing, Printing, and Laboratory operations. The capacity of these areas is about 10 M tons per day of which around

1000 M3 water is used in these processes. To treat the wastewater Dhaka facility has an ETP entire quantity of wastewater is passed and treated at this ETP.

There are three types of ETP Process:

- i. Physical Treatment Process;
- ii. Bio-Chemical Treatment Process;
- iii. Chemical Treatment Process;

Steps that involved in the entire process are as follow:

- a) Screening
- b) Homogenization
- c) Coagulation
- d) pH Control
- e) Flocculation
- f) Flotation
- g) Scum storage
- h) Sludge thickening
- i) Sludge mixing
- j) Pressure filter
- k) Aeration
- l) Sedimentation
- m) Filtration

The entire process flow is shown below:

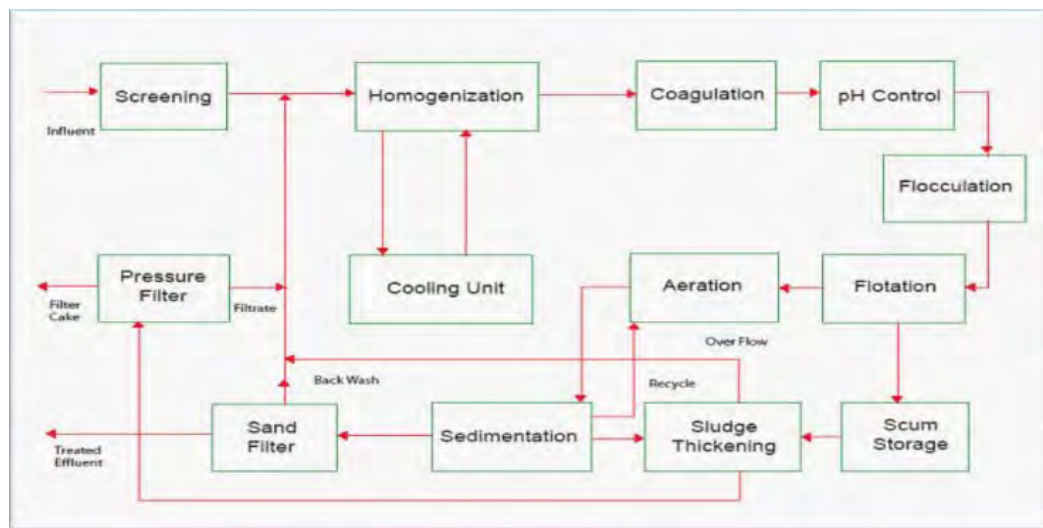


Figure 2.2. Effluent treatment plant flow diagram of SDF Ind. Ltd.



### 2.6.1 Screening

Raw effluent is initially passed through screen to trap all the floating material like wooden plank, plastic bags, paper etc. Initially the effluent is passed through coarse screen and later on through fine screens. All the material trapped in the screen is removed manually with the help of scoop. The screened materials are disposed suitably. Many industrial units discharge oil and grease in waste water due to leakage /spillage from the system. The oil & grease are to be removed in oil/Grease removing chamber. As the effluent with oil and grease enters into the oil and grease removing chamber - due to availability of more cross sectional area the velocity of effluent suddenly drops down. Since the specific gravity of oil & grease is lower than water, it floats on the top of the surface. From top surface oil & grease are removed by manual skimmer.



Figure 2.3. Screening Tank

### 2.6.2 Homogenization

The flow of effluent varies from time to time. At the same time quality of effluent changes during different period of the day. Since the treatment plant is designed for a particular flow rate and quality, it is essential to have an equalization tank prior to main treatment units. In this equalization tank a detention period for effluent is given to take care of surge load and also the flow during lean period. The effluent in the equalization tank is pumped at constant flow rate to the treatment plant.



Figure 2.4. Equalization Tank

### 2.6.3 Coagulation

This treatment process includes several different operations. There are suspended solid particles of such size that they will not naturally agglomerate. Hence, chemical coagulation of these suspended solids is made so that agglomeration of these particles takes place. The chemicals added are alum and lime so that surface water with turbidity resulting from colloidal particles to cluster, or flocks that are large enough to be removed by gravity settling. The wastewater is then mixed (rapid mix) as to ensure that the chemicals are equally distributed in the water (so that it can be well treated). During or after mixing, certain chemical reactions occur. Some of the reaction products are insoluble and will begin to precipitate as solid particles. The term coagulation is sometimes used to refer specifically to these chemical reactions, which begins the formation of flocks. The first step destabilizes the particle's charges. Coagulants with charges opposite those of the suspended solids are added to the water to neutralize the negative charges on dispersed non – settle-able solids such as clay and color-producing organic substances.



Figure 2.5 Coagulation Tank

Once the charge is neutralized, the small suspended particles are capable of sticking together. The slightly larger particles formed through this process and called microflocs, are not visible by naked eye. The water surrounding the newly formed microflocs should be clear. If it is not, all the particles' charges have not been neutralized, and coagulation has not been carried to completion. More coagulant may need to be added. A high-energy, rapid-mix to properly disperse the coagulant and promote particle collisions is needed to achieve good coagulation. Over-mixing does not affect coagulation, but insufficient mixing will leave this step incomplete. Coagulants should be added where sufficient mixing will occur. Proper contact time in the rapid-mix chamber is typically 1 to 3 minutes.

#### **2.6.4 pH Control**

At this tank Sodium Hydroxide is added to control the pH. The value of pH should be kept in the range of 6.00-6.50 for better flocculation. As the Alum (Aluminum Sulfate) has slightly acidic in characteristic the pH value of wastewater (after passing the Coagulation Tank) may decrease slightly.



Figure 2.6. pH Control Tank

If the value of pH goes beyond the set value (6.00-6.50), NaOH is added into the pH Tank. This process is controlled by an automatic system. A pH sensor from the coagulation tank sense pH value, if the value of pH goes beyond the set value then the NaOH dosing pumps will run automatically for adjusting the pH value.

#### **2.6.5 Flocculation**

Flocculation, a gentle mixing stage, increases the particle size from submicroscopic microfloc to visible suspended particles. Again, the word flocculation may have two slightly different meanings. Theoretically it may mean the growth of the flock

particles. In practice, it more often refers to the gentle agitation of the water, which brings about this growth. The micro-flocks are brought into contact with each other through the process which refers to gentle agitation of the treated water for a period of time (slow mix). Collisions of the micro-flock particles cause them to bond to produce larger, visible flocks called pin-flocks. This favors the collision of the small flock particles with each other with other suspended particles in the water. This causes them to stick together, or agglomerate, and grow into large, readily settle able masses.



Figure 2.7 Flocculation Tank

#### **2.6.6 Flotation**

This is mainly separation process. Separate the flocks from water. By applying air & water pressure the flocks are made to float and send the sludge to the scum storage tank and the treated clean water goes to pre-treatment tank.



Figure 2.8. Flotation Tank

### **2.6.7 Scum storage**

The air diffuser is used in this step to aerate the sludge.



Figure 2.9.Scum storage tank

### **2.6.8 Sludge thickening**

Thickening tank is used to thicken liquid sludge from industrial waste water treatment plants. By slow stirring of the sludge water, sludge is separated and sinks down to the bottom of the tank.

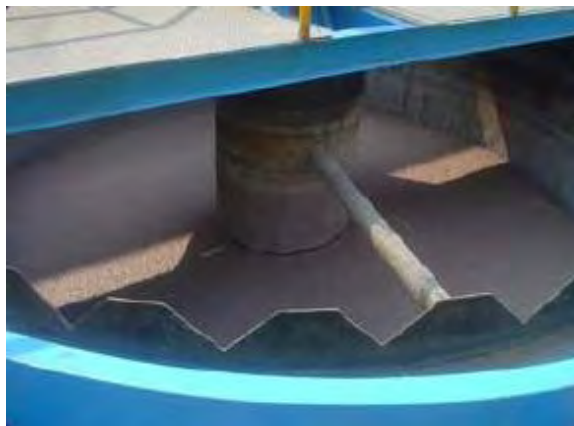


Figure 2.10. Sludge Thickener Tank

The water can be removed continuously or discontinuously from the tank and subsequently discharged into additional treatment stages. The operational mode is chosen according to the costumer's needs.

### **2.6.9 Sludge mixing**

The sludge is continuously stirred for easy access to Dehydrator Filter Press.



Figure 2.11. Sludge Mixing Tank

#### 2.6.10 Pressure filter

To make solid filter cake from liquid sludge by applying required pressure and the filtrate water goes back to the storage tank.



Figure 2.12. Filter Press

#### 2.6.11 Aeration

The primary-treated wastewater is then passed to an aeration chamber. Aeration provides oxygen to the activated sludge and at the same time thoroughly mixes the sludge and the wastewater. Aeration is by either bubbling air through diffusers at the bottom of the aeration tank, or by mechanically agitating the surface of the water. In the aeration tank, the bacteria in the activated sludge consume the organic substances in the wastewater. The organic substances are utilized by the bacteria for energy, growth and reproduction. The wastewater spends a few hours in the aeration chamber before entering a second sedimentation tank to separate the activated sludge from the treated wastewater. The activated sludge is returned to the aeration tank. There is an



increase in the amount of activated sludge because of growth and reproduction of the bacteria.



Figure 2.13. Aeration Tank

#### **2.6.12 Sedimentation**

Sedimentation, or clarification, is the processes of letting suspended material settle by gravity. Suspended material may be particles, such as clay or silts, originally present in the source water. More commonly, suspended material or flock is created from material in the water and the chemical used in coagulation or in other treatment processes, such as lime softening. Sedimentation is accomplished by decreasing the velocity of the water being treated to a point below which the particles will no longer remain in suspension. When the velocity no longer supports the transport of the particles, gravity will remove them from the flow.



Figure 2.14. Sedimentation Tank

### 2.6.13 Filtration

Here the treated water comes from Sedimentation Tank and filtrated to reuse or discharge to the environment. The treated water is tested regularly and the quality is monitored regularly. At Youngone Dhaka the treated water is recycled and used for toilet flush, saving around 2000 \$ per month in water utility bill.



Figure 2.15. Effluent Tank

### 2.6.14 Disposal of Sludge Cake

Youngone Dhaka unit produces 20,000 Kg of sludge cake per Month. These solid wastes packed into small polybag (Capacity of per polybag 10-15 kg) taking appropriate safety provisions. After then 5-6 polybags again packed into a large plastic bag. Finally the plastic bag is disposed into BEPZA designated area by trolley.



Figure 2.16. Sludge Cake Disposal



## 2.7 Manufacturing of Bricks

The fundamentals of brick manufacturing have not changed over time. However, technological advancements have made contemporary brick plants substantially more efficient and have improved the overall quality of the products. A more complete knowledge of raw materials and their properties, better control of firing, improved kiln designs and more advanced mechanization have all contributed to advancing the brick industry.

### 2.7.1 Raw materials

Clay is one of the most abundant natural mineral materials on earth. For brick manufacturing, clay must possess some specific properties and characteristics. Such clays must have plasticity, which permits them to be shaped or molded when mixed with water; they must have sufficient wet and air-dried strength to maintain their shape after forming. Also, when subjected to appropriate temperatures, the clay particles must fuse together.

#### Types of Clay

Clays occur in three principal forms, all of which have similar chemical compositions but different physical characteristics.

- a) *Surface Clays*: Surface clays may be the up thrusts of older deposits or of more recent sedimentary formations. As the name implies, they are found near the surface of the earth.
- b) *Shales*: Shales are clays that have been subjected to high pressures until they have nearly hardened into slate.
- c) *Fire Clays*: Fire clays are usually mined at deeper levels than other clays and have refractory qualities.

Surface and fire clays have a different physical structure from shales but are similar in chemical composition. All three types of clay are composed of silica and alumina with varying amounts of metallic oxides. Metallic oxides act as fluxes promoting fusion of the particles at lower temperatures. Metallic oxides (particularly those of iron, magnesium and calcium) influence the color of the fired brick. The manufacturer minimizes variations in chemical composition and physical properties by mixing clays from different sources and different locations in the pit. Chemical composition varies within the pit, and the differences are compensated for by varying manufacturing

processes. As a result, brick from the same manufacturer will have slightly different properties in subsequent production runs. Further, brick from different manufacturers that have the same appearance may differ in other properties.

### 2.7.2 Method of Manufacturing

The manufacturing process has six general phases:

- i. Mining and storage of raw materials,
- ii. Preparing raw materials,
- iii. Forming or molding the brick,
- iv. Drying,
- v. Firing and cooling and
- vi. De-hacking and storing finished products.

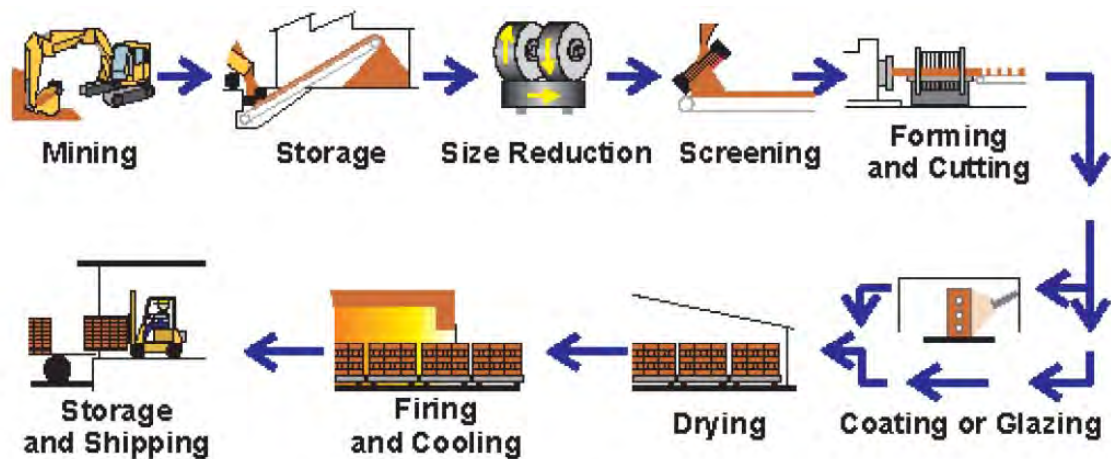


Figure 2.17. Diagrammatic representation brick manufacturing process. Source:  
(Brick Industry Association 2006)

#### 2.7.2.1 Mining and Storage

Surface clays, shales and some fire clays are mined in open pits with power equipment. Then the clay or shale mixtures are transported to plant storage areas (see Photo 1). Continuous brick production regardless of weather conditions is ensured by storing sufficient quantities of raw materials required for many days of plant operation. Normally, several storage areas (one for each source) are used to facilitate blending of the clays. Blending produces more uniform raw materials, helps control color and allows raw material control for manufacturing a certain brick body.

### 2.7.2.2 Preparation

To break up large clay lumps and stones, the material is processed through size-reduction machines before mixing the raw material. Usually the material is processed through inclined vibrating screens to control particle size.



Figure 2.18. Clay is thoroughly mixed with water in pug mill extrusion. Source: (Brick Industry Association 2006)



Figure 2.19. Clay is extruded through a die and trimmed to specified dimension before firing. Source: (Brick Industry Association 2006)

### **2.7.2.3 Forming or Molding.**

Tempering, the first step in the forming process, produces a homogeneous, plastic clay mass. Usually, this is achieved by adding water to the clay in a pug mill (Figure 2.7) a mixing chamber with one or more revolving shafts with blade extensions. After pugging, the plastic clay mass is ready for forming. There are three principal processes for forming brick: stiff-mud, soft-mud and dry-press.

#### **a) Stiff-Mud Process**

In the stiff-mud or extrusion process (Figure 2.8), water in the range of 10 to 15 percent is mixed into the clay to produce plasticity. After pugging, the tempered clay goes through a de-airing chamber that maintains a vacuum of 15 to 29 in. (375 to 725 mm) of mercury. De-airing removes air holes and bubbles, giving the clay increased workability and plasticity, resulting in greater strength. Next, the clay is extruded through a die to produce a column of clay. As the clay column leaves the die, textures or surface coatings may be applied. An automatic cutter then slices through the clay column to create the individual brick. Cutter spacing and die sizes must be carefully calculated to compensate for normal shrinkage that occurs during drying and firing. About 90 percent of brick in the United States are produced by the extrusion process.

#### **b) Soft-Mud Process**

The soft-mud or molded process is particularly suitable for clays containing too much water to be extruded by the stiff-mud process. Clays are mixed to contain 20 to 30 percent water and then formed into brick in molds. To prevent clay from sticking, the molds are lubricated with either sand or water to produce “sand-struck” or “water-struck” brick. Brick may be produced in this manner by machine or by hand. This process is followed in Bangladesh

#### **c) Dry-Press Process**

This process is particularly suited to clays of very low plasticity. Clay is mixed with a minimal amount of water (up to 10 percent), then pressed into steel molds under pressures from 500 to 1500 psi (3.4 to 10.3 MPa) by hydraulic or compressed air rams.

#### **2.7.2.4 Drying**

Wet brick from molding or cutting machines contain 7 to 30 percent moisture, depending upon the forming method. Before the firing process begins, most of this water is evaporated in dryer chambers at temperatures ranging from about 100 °F to 400 °F (38 °C to 204 °C). The extent of drying time, which varies with different clays, usually is between 24 to 48 hours. Although heat may be generated specifically for dryer chambers, it usually is supplied from the exhaust heat of kilns to maximize thermal efficiency. In all cases, heat and humidity must be carefully regulated to avoid cracking in the brick.

#### **2.7.2.5 Firing**

Brick are fired between 10 and 40 hours, depending upon kiln type and other variables. There are several types of kilns used by manufacturers. The most common type is a tunnel kiln, followed by periodic kilns. Fuel may be natural gas, coal, sawdust and methane gas from landfills or a combination of these fuels. In a tunnel kiln brick are loaded onto kiln cars, which pass through various temperature zones as they travel through the tunnel. The heat conditions in each zone are carefully controlled, and the kiln is continuously operated. A periodic kiln is one that is loaded, fired, allowed to cool and unloaded, after which the same steps are repeated. Dried bricks are set in periodic kilns according to a prescribed pattern that permits circulation of hot kiln gases. Firing may be divided into five general stages:

- i. Final drying (evaporating free water);
- ii. Dehydration;
- iii. Oxidation;
- iv. Vitrification; and
- v. Flashing or reduction firing.

All except flashing are associated with rising temperatures in the kiln. Although the actual temperatures will differ with clay or shale, final drying takes place at temperatures up to about 400 °F (204 °C), dehydration from about 300 °F to 1800 °F (149 °C to 982 °C), oxidation from 1000 °F to 1800 °F (538 °C to 982 °C) and vitrification from 1600 °F to 2400 °F (871 °C to 1316 °C).

Clay, unlike metal, softens slowly and melts or vitrifies gradually when subjected to rising temperatures. Vitrification allows clay to become a hard, solid mass with relatively low absorption. Melting takes place in three stages:

- i. Incipient fusion, when the clay particles become sufficiently soft to stick together in a mass when cooled;
- ii. Vitrification, when extensive fluxing occurs and the mass becomes tight, solid and nonabsorbent; and
- iii. Viscous fusion, when the clay mass breaks down and become molten, leading to a deformed shape. The key to the firing process is to control the temperature in the kiln so that incipient fusion and partial vitrification occur but viscous fusion is avoided.

The rate of temperature change must be carefully controlled and is dependent on the raw materials, as well as the size and coring of the brick being produced. Kilns are normally equipped with temperature sensors to control firing temperatures in the various stages. Near the end, the brick may be “flashed” to produce color variations (Brick Industry Association 2006)

#### **2.7.2.6 Cooling**

After the temperature has peaked and is maintained for a prescribed time, the cooling process begins. Cooling time rarely exceeds 10 hours for tunnel kilns and from 5 to 24 hours in periodic kilns. Cooling is an important stage in brick manufacturing because the rate of cooling has a direct effect on color.

### **2.8 Previous Studies on Sludge Stabilization through construction materials**

Brick is one of the major construction materials in Bangladesh. So many researchers and environment specialists attempted to stabilize different types of wastes such as water treatment sludge (Coagulant sludge), sewage sludge, desalination sludge, textile dyeing sludge, ceramic sludge, cigarette Butt, petroleum ETP sludge, tannery shredded sludge etc.

From their researched and experimental result it was clear that, incorporation of sludge with clay brick has a positive output in terms of sludge management and engineering properties of the product. This is a practical solution to face the environmental pollution problem.

### **A. Textile Sludge**

Jahagirdar et al. (2013) discussed the reuse of textile mill sludge in fired clay bricks. The textile mill sludge was mixed together with different proportion (5% to 35%) as the raw material in this study. The brick was fired at 600°C to 800°C and for 8, 16 and 24 hours. Based on the results, textile sludge can be added up to 15% as it gives compressive strength above 3.5MPa and the water absorption ratio is also less than 20%.

According to Herek et al. (2012) the investigation on the incorporation of textile laundry sludge into a brick showed that sludge can be incorporated up to 20% in terms of the mechanical properties. The compressive strength of the control brick was 3.73MPa and 4.62MPa for the sludge brick. On the other hand, the water absorption result obtained has shown that 15.73% and 10.10% for control brick and sludge brick respectively. Besides, the produces brick is safe according to applied leaching and do not exceed the standard limits.

Baskar et al. (2006) also discussed about characterization and reuse of textile effluent treatment plant waste sludge in clay bricks. In his study, the sludge composition was from 3% to 30% and the firing temperature is about 200°C to 800°C. The compressive strength was between 4.24MPa to 3.54MPa which satisfies the Bureau of Indian Standard (BIS). The maximum amount range of sludge that can be added is from 6% to 9%.

### **B. Water Treatment Sludge**

Babu et. al. (2013) in their research investigated on bricks durability of cast brick with industrial sludge. The results show that the earth brick can be replaced with sludge up to 40% by weight with satisfactory value in strength. The compressive strength of brick without sludge and 5% of sludge were 11.7MPa and 17.6MPa respectively. The compressive strength was decreasing with addition of sludge beyond 5% from 17.6MPa to 10.5MPa. For water absorption result, when the sludge added more than 10% by weight, the water absorption was gradually increased. In this study, addition of sludge into brick gives dual benefits of safe disposal of sludge from industry and also conservation of brick making.

As for Hegazy et al. (2012), they discussed the incorporation of water treatment sludge and rice husk ash in clay bricks. In this study, 25%, 50% and 75% by weight of

water treatment sludge was added to produce clay bricks. Each brick series was fired at 900°C, 1000°C, 1100°C, and 1200°C. The compressive strength of brick value were 5.7 MPa to 6.8 MPa for the control brick and 2.82 MPa to 7.84 MPa for Sludge- RHA brick. Meanwhile, for the water absorption test, the results were 9.94% to 11.18% of control brick and 17.41% to 73.33% for Sludge-RHA brick respectively. From the obtained results, it was concluded that by common temperature, 75% addition was the optimum sludge to produce the bricks.

Hegazy et al. (2012) also discussed the incorporation of water sludge, silica fume (SF) and rice husk ash (RHA) in brick making. Three different series of sludge to SF and RHA proportion which were (25:50:25%), (50:25:25%) and (25:25:50) were incorporated. Each brick was fired at 900°C, 1000°C, 1100°C and 1200°C. For the compressive strength and water absorption the results obtained 5.03MPa to 8.12MPa and 16.24% to 52.11% respectively. The operating at the temperature commonly practiced in brick klin could be concluded that mixture consists of 50% of sludge, 25% of SF and 25% of RHA was the optimum materials proportions that demonstrated obvious superior properties to the 100% clay control-brick.

Victoria et al. (2013) developed bricks from water works sludge with five different mixing ratio of sludge at 0%, 5%, 10%, 15% and 20% of the total weight of sludge-clay. Each brick has been molded by hand and been fired into furnace at elevated temperature of 850°C, 900°C, 950°C, 1000°C and 1050°C. The result of compressive strength of sludge clay brick are between 0.97MPa to 12.98MPa. Increasing the sludge content result in decreased compressive strength decreased density and increased water absorption. Results for density and water absorption are 1g/cm<sup>2</sup> to 2g/cm<sup>2</sup> and 14.07% to 31 % respectively. Toxicity characteristic leaching procedure (TCLP) result showed that the metal leaching level is within the acceptable limits of NESREA and USEPA limits.

According to Saijun et al. (2011) in their investigation, the incorporation of three sludge percentages which are 6%, 8% and 10% shows that the compressive strength was decreased to 20.22% but the flexural strength increased. The compressive strength of 10% of sewage sludge obtained 21.8MPa and flexural strength of 4.6MPa. Autoclaved sludge fly ash was incorporated in brick when pH 6.9 was obtained which is close to normal pH. The heavy metals were solidified during the curing process and it will not pollute the environment.



In the year 2008, Ramadan et al. (2008) discussed the reuse of sludge from water treatment plant. The results of water absorption ranged between 4.84% and 17.34% which comply with the requirement of the Egyptian standard specification. There were five brick types that exhibited water absorption less than 7% and met the requirement for the British standard to be classified as Engineering Brick B. According to Ramadan et al. (2008) compressive strength is usually affected by the porosity, pore size and type of crystallization. The results show compressive strength values between 2.3MPa and 11.66MPa. Compressive strength values more than 7.35MPa met the requirement of British standard for engineering brick. A. Ramadan et al. (2008) also concluded that all bricks tested in this investigation are superior compared to the commercial clay brick types available in the Egyptian market.

### **C. Sewage Sludge**

Ingunza et al. (2011) used 5%, 10%, 15%, 20%, 25% and 30% of sewage sludge incorporated into soft-mud brick with 12 specimens for each sludge percentages. From the result obtained there is no sign of alteration in color or odor. Brick with 35% sludge were very brittle and there are some of dimension reduction changes between 1mm to 7mm. Based on the result, the brick mass significantly loss according to the percentage of sludge. Weng et al. (2003) also reported the same conclusion. Inaganza also claim that bricks manufactured with 20%, 25% and 30% are above the limit proposed. In terms of properties the water absorption result shows there were increment for each brick compared to control brick. With 25% of sludge used, the brick absorbing capability increased to an average of 160% more than control brick. The sludge brick with 25% and 30% inclusion do not meet minimum standard required but other percentages comply with the minimum standard strength.

Liew et al. (2004) study the incorporation of dry weight of sludge into brick with 10% to 40% and fired at 985°C. In this study, the utilization of sludge more than 40% still complied with the standard based on physical and chemical properties. However, the researchers concluded that the maximum percentage of sludge used should not be more than 30% by weight due to its fragility. The water absorption value increased by up to 37% compared to the control brick (23.6%) and the compressive strength decreased to 2MPa against 15.8MPa for the control brick. During the firing process, the gases included steam and CO<sub>2</sub> were released, also the cross section revealed black coring due to the combustion of the organic content in the sludge. The bricks were

only appropriate for use as common bricks because of the entire weak and poor exterior surface.

According to Lin et al. (2001) the results obtained demonstrated that the appropriate percentage of ash sludge to produced good quality bricks is in the range of 20% to 40% by weight with a 13% to 15% optimum moisture content prepared in the molded mixture. Firing is conducted at 1000°C for 6 hours. Utilization of 10% sludge ash exhibited higher compressive strength than normal brick.

Sidrak (1996) research on the Biofly brick is by reuse of fly ash and sewage sludge. The results show the brick incorporated with 50% to 70% indicated that the average of compressive strength ranged between 21.4MPa to 49.7MPa for Biofly brick and 39.1MPa for ordinary brick. This research used firing temperature of 200°C, 400°C, 600°C, 800°C and 1100°C. The water absorption result shows averaged of cold and hot water absorption values of 15% and 15.3% for Biofly brick and 3.9% and 4.9% for clay/shale brick. As for the leachate studies there were three different size fractions were undertaken for all the bricks made. All leachate samples were analyzed for copper, iron, manganese, nickel, lead, zinc, cadmium, chromium and aluminium concentration. All metals were tested by using Atomic Absorption Spectrophotometry. The results shown the concentration of heavy metals was within the limits standard by Victoria EPA and US Code of Federal Regulation (CFR). Nevertheless, the heavy metals were detected still inside the brick even in the low concentration. The gas consumption and gas emission study indicated that the brick process uses less energy and produced a smaller amount of air pollution compared to standard brick. Biofly brick also saves energy up to 44%, produces 20% to 24% lighter brick and 10% to 30% stronger compared to the conventional bricks.

#### **D. Tannery Sludge**

Juel et.al. (2016) used tannery sludge in brick production. Clay bricks were prepared with different proportions of sludge (10%, 20%, 30% and 40% by dry weight) in both laboratory-controlled and field conditions. For the sludge incorporated bricks, the compressive strength ranged from 10.98 MPa to 29.61 MPa and water absorption ranged from 7.2% to 20.9%, which in most cases met the criteria for bricks as a construction material as per Bangladesh Standards and ASTM. Volumetric shrinkage, weight loss and efflorescence properties of sludge-amended bricks were found to be

favorable and it was estimated that an energy saving of 15–47% could potentially be achieved during firing with 10–40% tannery sludge-amended bricks.

### **E. Other Sludge**

Stone sludge was another sludge that studied by Rajgor et al. (2013) to be used in clay bricks. Varying percentages of stone sludge 10%, 20%, 30%, 40%, 50% and 60% were incorporated in the clay bricks. All samples were fired at 1050°C. The results for compressive strength are 2.11MPa to 4.2MPa and water absorption ratio is from 8% to 12%.

Hii et al. (2013) discussed the reused of desalination sludge for brick. Desalination sludge has been dried and ground into fine powder before being mixed with clay with mixing ratios 0%, 10%, 20%, 30%, and 40% content by weight. The average of compressive strength was decreasing from 8MPa, 3MPa and 2MPa for 0%, 10%, and 20% sludge bricks respectively. Water absorption results showed that increasing sludge will increase the water absorption with 8.1kg/m<sup>2</sup>min for control brick to 14.6kg/m<sup>2</sup>.min for 40% of the dried desalination sludge.

Ferez et al. (2011) investigated on how to manufacture ceramic bricks by using recycled brewing spent kieselguhr sludge. The result obtained demonstrated an increases value in the porosity and decreases the bulk density around 1919kg/m<sup>3</sup> to 2090kg/m<sup>3</sup> at 900°C to 1000°C respectively. Water absorption was increased with the increasing of the sludge and decrease with firing temperature. For the mechanical properties, strength shows 8.3MPa, 17.1MPa, 17.5MPa and 18.4MPa at 105°C, 900°C, 950°C, and 1000°C respectively. In this research, the results shown no constrain concerning mechanical properties. Furthermore, the ecotoxicity evaluation also shows the safety of the brewing spent kieselghur incorporation in ceramic product is complied with the standard.

According to Sengupta et al. (2002), petroleum sludge was hazardous sludge containing high amount of hydrocarbons. The petroleum sludge contains oil, water and inorganic material. The major constituents of the sludge are SiO<sub>2</sub>, CaO, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. The result shows that, the quality of brick sludge is better than the standard bricks due to color and less fuel of firing. Compressive strength results shown the Soil: Sand: Water (SS) and Soil: Sand: Sludge (SSS) brick produced 16.45MPa and 16.02MPa respectively higher than commercial brick with 9.06MPa.

All bricks complied with all requirements according Indian standard. Most of the metals (Mn, Cr, Sb, Ni, Co, and Hg) are emitted during firing. By using this sludge, it will reduce the requirement of water and fuel in brick manufacture and could be one of the disposal methods for the hazardous sludge.

Tay et al. (2001) used 2% to 16% of industrial sludge in clay bricks and fired with temperature 1050°C. The compressive strengths of sludge clay bricks with various mix proportions fall within range of 12MPa to 31MPa. The experimental study indicates that the reuse of marine clay-industrial sludge mixes as brick making material offers a technically feasible alternative for disposal of the wastes as well as resource recovery.

Unlike the previous studies, the current study was focused on brick preparation in field conditions in conventional brick kiln in large scale where other studies were mainly lab based brick preparation. As brick were prepared in brick kiln the firing temperature were higher than the lab conditions. In most of the previous studies the brick were fired in lower temperature. The temperature varies from 600 °C to 1250 °C for the previous studies whereas the firing temperature was around 2100 °C to 2400 °C for this study. Due to the elevated temperature produced bricks showed better engineering properties than the bricks from previous studies.

Bricks produced by incorporating dyeing industry sludge met the requirements of standards regarding strength and other mechanical and durability parameters as per relevant codes. Additionally, leach toxic pollutants from these bricks was within the acceptable limits. This study showed that water absorption increased with the increase in percentage of sludge. A lower amount of sludge showed the greatest dry density of all. This study recommended that a material containing 10% dyeing sludge can be used safely considering the environmental aspects. These papers reported the results based on laboratory-based studies as well as on field condition.

In Bangladesh, around 7,000 brick kilns produce 23 billion pieces of burnt clay bricks from around 3 billion cubic feet of topsoil annually (DoE, 2017). The brick-manufacturing industry contributes to about 1 percent to the country's gross domestic product (GDP) (BUET 2007). Due to unavailability of stone aggregate, brick has become the principal building material for the country's construction industry and will continue to be so in future. Therefore, exploring the brick manufacturing industry

as a potential avenue for stabilization of the huge amount of dyeing sludge in Bangladesh can be a viable option for commercial application.

## Chapter 3

### Methodology

This chapter narrates the working procedure which was followed in this study. The methodology for collection of samples (soil and sludge), analytical procedures to determine characteristics of sludge and sludge – clay mix, preparation and tests for bricks using sludge-clay mixtures and leaching test of bricks are discussed in a sequential manner.

#### 3.1 Collection of Sludge Samples

Sludge samples were collected from SDF Ltd, Plot# 4 (half)-8, 12 (half)-16, DEPZ, Ganakbari, Savar, Dhaka, (Figure 3.1). SDF Ind. Ltd. is a renowned textile dyeing factory in Bangladesh. A bio-chemical Effluent Treatment Plant (ETP) with capacity of 10000 m<sup>3</sup> is employed to treat the wastewater coming from the dyeing factory. Alum [ $\text{Al}(\text{SO}_4)_2 \cdot 18 \text{H}_2\text{O}$ ], Lime [ $\text{Ca}(\text{OH})_2$ ] are used as coagulants and polymer is used as flocculent. Sludge generated from chemical and biological treatment is transferred to sludge thickener then to pressure filter produce sludge cake. Raw sludge cake was collected from dump zone shown in Figure 3.2. The samples were stored and conveyed using polythene bags.



Figure 3.1. Location of SDF Ltd. (From Google map)



Figure 3.2. Raw sludge Filter press and storage

### 3.2 Collection of Clay Samples

Soil samples are collected from Ms. Mir Bricks situated at Sujabad, Shahjanpur, Bogura. There are numbers of brick field in this area and meet the partial demand of clay bricks of Bogura city as well as supply huge amount of brick in other districts. Soil sample was collected from different collection site at varying depth. Sample was collected in plastic sandbags. Soil sample was transported up to BUET laboratory.

### 3.3 Characteristics of Sludge and Soil

The entire flow chart of this study has been presented in Figure 3.3. The analytical method adopted in this study of samples has been shown in Table 3.1. Physical characteristics like moisture content, presence of organic compound were tested.

**Moisture Content:** Wet method was followed in determining moisture content of sludge samples. Both soil and Sludge samples of 2.5 gm. respectively were taken at ambient temperature. Thereafter samples were oven dried at 105°C for about 24 hours. After oven drying the samples were placed in a desiccator until the sample cooled. Then the weights of the samples ( $W_d$ ) in gm. were taken again. Then, the Moisture content of the sample was calculated using equation (3.1)

$$\text{Moisture Content, \%} = \frac{(2.5 - W_d)}{2.5} \times 100\% \quad (3.1)$$

Where,

$W_d$  = dry weight of the specimen.

**pH:** The pH of the sludge sample was analyzed with colorimetric methods using litmas paper. A colorimetric method is a simple and practical procedure. The strip of the litmus paper was taken in and emerged in the raw sample. The color of the paper changed within a second into dark brown. The color of the litmus paper was matched with the colors of the color chart to determine the pH of the sample.

**Organic Content:** The organic matter sludge was measured using the Dry combustion Technique adopted for measuring organic carbon in soil (Alam et al., 1991). Approximately 100 gram of sludge sample was oven dried at 100°C (Until constant weight) and from there about 25-30 gram of dried sample was taken in a crucible. The initial weight ( $W_1$ ) was then recorded. The dried sample was then burnt in a muffle furnace (CARBOLITE) at 440°C- 450 C for six hours (AASHTO 267, equivalent to ASTM 2974) Final weight ( $W_2$ ) was taken to calculate the percentage of organic matter in the sludge using equation (3.2)

$$\text{Organic Content, \%} = \frac{(W_1 - W_2)}{W_1} \times 100\% \quad (3.2)$$

Where,

$W_1$  = Initial dry weight of the specimen, and

$W_2$  = Final weight of the specimen after burnt in muffle furnace.

**Chemical Composition:** X-Ray Fluorescence (XRF-1800, Shimadzu) was used to determine the chemical composition of dyeing sludge and soil.

Table 3.1. Analytical methods for characterization of clay, sludge and bricks.

Parameters	Test method
For Raw Sludge	
pH	BS 1377 (1990): Part 2
Moisture content	BS 1377 (1990): Part 2
Organic content	BS 1377 (1990): Part 2
Chemical composition	X-Ray Fluorescence (XRF)



Total Heavy metal	EPA 3050B & AAS
TCLP	USEPA 1311
<b>For Sludge- Clay mix</b>	
Plastic limit	ASTM D 4318
Liquid limit	ASTM D 4318
Plasticity Index	ASTM D 4318
Compaction test	AASHTO T-99 (1982)
<b>For Sludge- Brick Properties</b>	
Compression strength	ASTM C 67
Water absorption	ASTM C 67
Weight loss on ignition	Thermal method
Bulk Density	ASTM C 20-00
Firing shrinkage	ASTM C 210-95
TCLP	USEPA 1311

### 3.4 Heavy Metal Determination

After collection of sludge, some portion of sludge samples were dried in a vacuum oven at 105°C until constant weight, lightly ground for homogenization and to pass 2-mm sieve. For heavy metal analysis, sample preparation was carried out according to EPA 3050B with a slight variation. 5 gm. of dried sample was digested with acid (HNO<sub>3</sub>: HCl =1:3 volume ratio) for 24 hour. After adding 350-400ml distilled water, sample was boiled for 2.5 hour and prepared a 500ml solution. Then, solution was filtered through 0.45 µm pore size filter paper and filtrate was collected to determine the concentration of seven heavy metals (As, Cd, Cr, Cu, Hg, Ni, Pb and Zn) by using Atomic Absorption Spectrophotometer (AAS) (Shimadzu AA 6800) in the environmental engineering laboratory, BUET. In this method, a light beam of appropriate wavelength for particular metal is directed through a flame. The flame atomizes the sample, producing atoms in their ground (lowest) electronic energy state. These are capable of absorbing radiation from the lamp. Although the equipment appears completely different from other forms of absorption spectrometry, the law by

which absorption of light is related to concentration is similar to that used for absorption of light related to concentration.

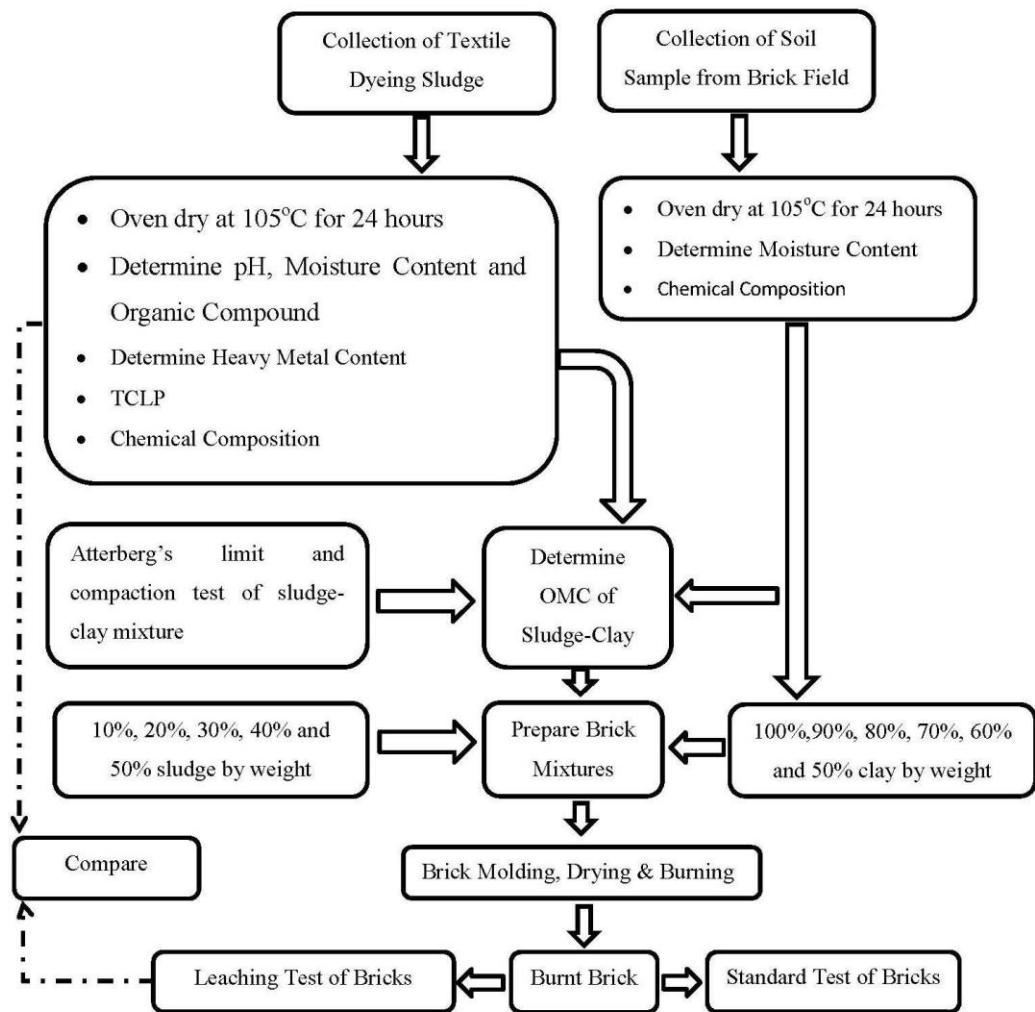


Figure 3.3. Flow chart of the total process

The AAS is extremely sensitive technique and for common ions dilution prior to analysis is preferred. The concentration range over which the law applies for Atomic adsorption Spectrometry (AAS) is usually 0-5 mg/L.

### 3.5 Characteristics of Clay – Sludge Mixture

Different compositions of clay-sludge mix were characterized by plasticity behaviors, optimum moisture content and dry density. Atterberg's limit test and compaction test of different sludge – clay mixture were carried out as follow.

### 3.5.1 Atterberg's tests

The moisture content, in percent, at which the transition from solid to semisolid state takes place, is defined as the shrinkage limit. The moisture content at the point of transition from semisolid to plastic state is the plastic limit, and from plastic to liquid state is the liquid limit. These limits are also known as Atterberg limits.

The Atterberg's limits are a basic measure of the critical water contents of a fine-grained soil: its shrinkage limit, plastic limit, and liquid limit.

Depending on its water content, a soil may appear in one of four states: solid, semi-solid, plastic and liquid. In each state, the consistency and behavior of a soil is different and consequently so are its engineering properties. Thus, the boundary between each state can be defined based on a change in the soil's behavior. The Atterberg's limits can be used to distinguish between silt and clay, and to distinguish between different types of silts and clays.

Air-dry sludge and clay samples were grind into finer particles and passed through number 40 sieve. Clay-sludge mixtures were made with 0%, 10%, 20%, 30%, 40% and 50% sludge (by dry weight). Atterberg limit test of these samples were carried out following ASTM D 4318 (2000) to determine liquid limit and plastic limit. The amount of water which must be added to change a soil from its plastic limit to its liquid limit is an indication of the plasticity of the soil. The plasticity is measured by the "plasticity index" which is calculated by the equation (3.3)

$$PI = LL - PL \quad (3.3)$$

### 3.5.2 Compaction test

In order to determine the optimum moisture content (OMC), which is an important factor affecting the properties of brick, a standard AASHTO T- 99 (1982) compaction test was used in this study. Air dry samples were grind to pass through sieve number 4. Sample amounting 2.5 kg for each type of mix proportion was taken to determine individual parameters of optimum moisture content (OMC) and dry density corresponding to OMC. The experimental setup is as below:

- a) The soil is compacted in a mold that has a volume of 943.3 cm<sup>3</sup>.

- b) The diameter of the mold is 101.6 mm. During the laboratory test, the mold is attached to a base plate at the bottom and to an extension at the top (Figure 4.2a).
- c) The soil is mixed with varying amounts of water and then compacted (Figure 4.3) in
- d) Three equal layers by a hammer (Figure 4.2b) that delivers 25 blows to each layer.
- e) The hammer weighs 24.4 N (mass  $\approx$  2.5 kg), and has a drop of 304.8 mm. For each test,

### 3.6 Preparation of Bricks

Sludge and clay samples were mixed in five mix ratios of clay-sludge mixtures.

A set of sludge-incorporated bricks was prepared in a commercial brick kiln as per their usual procedure (Figure 3.7). There are two types of brick molding process mostly used in Bangladesh, by hand molding and by automatic pressed molding. In this study the sludge-amended clay bricks were hand-molded and the regular wooden forma used for brick molding. Then molded brick samples were then kept for 7 days for natural drying. Total 1000 bricks sample (length 254 mm, width 127 mm and height 76 mm) of sludge-clay mixture in varying proportion (10%, 20%, 30%, 40% and 50%) and 200 nos of 100% clay samples (as a reference specimen) were prepared in the field.

After the bricks were manufactured in the kiln, they were subjected to the different test to determine the engineering properties as well as the leaching behavior of heavy metal to the environment.



Figure 3.4. Brick mold



Figure 3.5. Prepared raw bricks in the field



Figure 3.6. Brick Kiln



Figure 3.7. Brick burning in tunnel kiln (using coal as fuel)





Figure 3.8. Burnt brick unloading from kiln

### 3.7 Standard Test of Bricks

Certain some tests which indicate the quality of bricks were conducted to check whether it the bricks are suitable to be used as construction materials or not. These tests include compressive strength, water absorption and weight loss on ignition, firing shrinkage and bulk density. All these test methods are described as follows.

#### 3.7.1 Compressive strength of bricks

The compressive strength test was conducted according to ASTM C 67 method. There are two methods viz. Sulfur-Filler Capping and Gypsum capping method. Here Sulfur-Filler Capping method was followed. The load is applied up to one half of the expected maximum load, at any convenient rate, after which, the controls of the machine were adjusted so that the remaining load is applied at a uniform rate. The compressive strength is computed using equation (3.4).

$$C = \frac{W}{A} \quad (3.4)$$

Where,

$C$  = compressive strength of the specimen,  $\text{N/mm}^2$ .

$W$  = maximum load,  $\text{N}$ .

$A$  = average of the gross areas of the upper and lower bearing surfaces of the specimen,  $\text{mm}^2$ .



Figure 3.9. Compressive strength test machine  
(Macklow-Smith Ltd. Camberly, London)

### 3.7.2 Water absorption of bricks

Water absorption test of bricks was carried out according to ASTM C 67 method. According to this method brick samples were oven dried at 105°C for 24 h. After drying, the specimens were cooled at room temperature. Dry weight ( $W_d$ ) of brick samples were notes. Then the dry, cooled specimens were submerged in distilled

water at 26 - 30°C for 24 h. Then the specimens were wiped off the surface water with a damp cloth and the saturated weights the specimen ( $W_s$ ) were taken. Then, water absorption of the sample is calculated using equation (3.5)

$$\text{Water Absorption, \%} = \frac{(W_s - W_d)}{W_d} \times 100\% \quad (3.5)$$

Where,

$W_d$  = dry weight of the specimen (kg), and

$W_s$  = saturated weight of the specimen after submersion in cold water (kg).



Figure 3. 10. Arrangement for conducting water absorption test.

### 3.7.3 Weight loss on ignition

The raw brick samples were dried naturally for 7 days. The weights of air dry samples were measured. Loss of Ignition (LOI) was determined by measuring the weight loss of the sample between the drying and firing stage using equation (3.6).

$$LOI(\%) = \frac{(W_d - W_b)}{W_d} \times 100\% \quad (3.6)$$

Where,

$W_d$  = Mass of air dried brick sample (kg)

$W_b$  = Mass of burnt brick sample (kg)





Figure 3.11. Oven drying of burnt brick.



Figure 3.12. Dry brick weight measurement

### 3.7.4 Firing shrinkage of bricks

Total volumetric shrinkage values were obtained by measuring the volume of the samples with a caliper before and after the firing stage using equation (3.7).

$$\text{Firing shrinkage, (\%)} = \frac{(V_d - V_b)}{V_d} \times 100\% \quad (3.7)$$

Where,

$V_d$  = volume of dry bricks before Burning ( $\text{mm}^3$ ).

$V_b$  = volume of bricks after burning ( $\text{mm}^3$ ).



Figure 3. 13. Brick measurement (Air dry and burnt brick )

### 3.7.5 Bulk density

The weight of fired brick samples and volume of the samples was also measured. Volume was accounted from multiplying the average length, width and height of the sample. Bulk density was measured by the following equation (3.8).

$$\text{Bulk density (g/cm}^3\text{)} = \frac{M}{V} \quad (3.8)$$

Where,

$M$  = dry mass of burnt brick (g)

$V$  = volume of burnt brick (cm<sup>3</sup>)

### 3.8 Leaching Test of Building Materials

The toxicity and leachability tests were carried out to ensure the environmental compatibility of these fired bricks to be used as building materials. In this study, the toxicity characteristic leaching procedure (TCLP) test of the sludge samples was carried out according to USEPA 1311 method (USEPA, 1992). In TCLP test, dried samples are ground and passed through 9.5 mm standard sieve. An acetic acid solution (0.57% v/v) was added to samples at a constant ratio of liquid: solid (20:1). The pH of the extraction fluid was  $2.88 \pm 0.05$ . After 18 h rotating with rotary mixture at  $30 \pm 2$  rpm, the leachate was filtered with 0.45  $\mu\text{m}$  pore size filter paper and analyzed for As, Cd, Cr, Cu, Hg, Ni, Pb and Zn using FLAAS (Shimadzu AA 6800).



Figure 3.14. Arrangement for conducting TCLP test according to USEPA 1311

## Chapter 4

### Result and Discussion

The results obtained from this study will be discussed in this chapter. This chapter presents laboratory experiments to determine the characteristics of sludge and clay - sludge mixtures, engineering properties of sludge-amended bricks and leaching test results of both sludge and sludge incorporated fired bricks.

#### 4.1 Characteristics of Sludge

##### 4.1.1 General Characteristics

The characteristic of raw dyeing sludge sample is shown in Table 4.1. The pH of sludge samples was found 6.9 and so, the sludge can be regarded as neutral. The moisture content of the sludge sample was 72%. The organic contents of sludge were 4.3%.

Table 4.1. Characteristics of Dyeing sludge.

Parameters	Sludge
pH	6.9
Moisture Content (%)	72
Organic content (%)	4.3

##### 4.1.2 Heavy Metal Content in Sludge

The selected heavy metal concentrations for the sludge sample found in this study are shown in Table 4.2. The total heavy metals content found in the sludge were compared with national and international regulatory limits of heavy metal content for sludge utilization.

Table 4.2. Heavy metal content in dyeing sludge and regulatory their limits

	Heavy Metal Concentration (mg/kg)							
	As	Cd	Cr	Cu	Hg	Ni	Pb	Zn
Dyeing Sludge	1.52	13.2	365.4	188.5	0.2	87.5	125.3	386.6
USEPA Limit	75	85	3000	4300	57	420	840	7500

BDS for Land application <sup>a</sup>	40	10	900	800	8	200	900	2500
SEPAC Limit in China <sup>b</sup>	-	0.6	250	100	-	26600	350	300
Limit in India	-	3-6	250	135-270	-	26600	250-500	300-600

<sup>a</sup> Source: DoE 2015, <sup>b</sup> Source: SEPA (1995), <sup>c</sup>Bhatnagar & Awasti (2000)

In this study it was found that the concentration of Arsenic, Cadmium, Chromium, Copper, Mercury, Nickel, Lead and Zinc were well below the US EPA and BDS Land Disposal Limits. But Cd exceeds BDS limit and China and Indian limit and Chromium crossed the China and Indian limit whereas Copper is higher than China limit.

Though the maximum of the heavy metal content is under allowable limit set by USEPA and BDS for land application however due to the above higher value of certain metal presence in the sludge based on different regulatory limit, the sludge is not suitable for application in agricultural land.

#### 4.1.3 Chemical Composition of Sludge and Soil

The chemical composition of dyeing sludge and soil was determined using X-Ray Fluorescence (XRF-1800, Shimadzu) in Glass and Ceramic Engineering department, BUET. The chemical composition found as percentage of major oxides was shown in Table 4.3.

Table 4.3. Chemical composition (dry weight, %) of dyeing sludge and soil.

Analyte	Result in %		
	SDF Sludge	Soil Sample	Standard Brick Clay
Al <sub>2</sub> O <sub>3</sub>	40.2	15.3	30.0
SiO <sub>2</sub>	26.2	63.0	55.0
SO <sub>3</sub>	22.0	0.10	0.00
CaO	3.82	1.59	1.00
P <sub>2</sub> O <sub>5</sub>	2.66	0.20	0.00
Fe <sub>2</sub> O <sub>3</sub>	2.20	8.79	8.00
MgO	1.42	3.02	5.00
TiO <sub>2</sub>	0.43	1.28	0.00
ZnO	0.32	0.02	0.00
BaO	0.27	0.00	0.00
Cr <sub>2</sub> O <sub>3</sub>	0.18	0.00	0.00
K <sub>2</sub> O	0.16	5.34	0.00
MnO	0.10	0.09	0.00

SrO	0.00	0.02	0.00
ZrO <sub>2</sub>	0.00	0.04	0.00
Na <sub>2</sub> O	0.00	1.19	0.00
Rb <sub>2</sub> O	0.00	0.03	0.00

aDr. M.A. Aziz, Engineering Materials, 1995.

Clay that was used for brick manufacturing consist of similar components present in the dyeing sludge but in different quantities. So, clay can be partially replaced by dyeing sludge as raw material for brick production

#### 4.1.4 Leaching Characteristics:

Toxicity Characteristics Leaching Procedure (TCLP) test results of the dyeing sludge is given in Table 4.4. The concentrations of all the metal leaching is lower than the USEPA regulatory limit however even these low concentration of the hazardous metal may lead to the surface and ground water pollution in long term and affect the marine life.

Table 4.4. Leaching of heavy metals from original sludge samples.

<b>Metal</b>	<b>Leached from Sludge (mg/L)</b>	<b>USEPA Regulatory level (mg/L)</b>
As	0.23	5
Cd	0.15	1
Cr	1.8	5
Cu	0.61	100
Hg	0.002	0.2
Ni	0.22	11
Pb	0.26	5
Zn	0.5	500

## 4.2 Characteristics of Sludge-Clay Mix

### 4.2.1 Atterberg's Limit

The behavior of soil is related to the amount of water in the system. The Liquid Limit is the boundary between the liquid and plastic states whereas the Plastic Limit is the boundary between the plastic and semi-solid states. The effect of moisture on the plastic behaviour of the pulverized materials is evaluated by the Atterberg Limit test. Panjaitan (2014) showed that plasticity index states the properties of soil. Sand has a Plasticity Index of 0 whereas clay has greater than 17 (Table 4.5). A small Plasticity

Index such as 5 shows that a small change in moisture content will change the soil from semi-solid to liquid.

Table 4.5. Plasticity Index and properties of soil (Panjaitan, 2014)

PI range	Plastic nature	Soil properties
0	Non plastic	Sand
< 7	Low plastic	Silt
7 – 17	Medium plastic	Silt clay
> 17	High plastic	Clay

The Plasticity Index also gives a good indication of compressibility. The greater the PI refers to the greater the soil compressibility. The results of Atterberg tests of sludge-clay mixture shown in Figure 4.2 indicate that the value of Plasticity Index decreases when sludge content increases in the mixture. Plastic Limit values have revealed that with the addition of sludge, the plastic nature of the mixture is lowered and the bonding ability of the mixture is also decreased. When the mixture contains high amounts of sludge, the bonding capacity of the mixture will decrease.

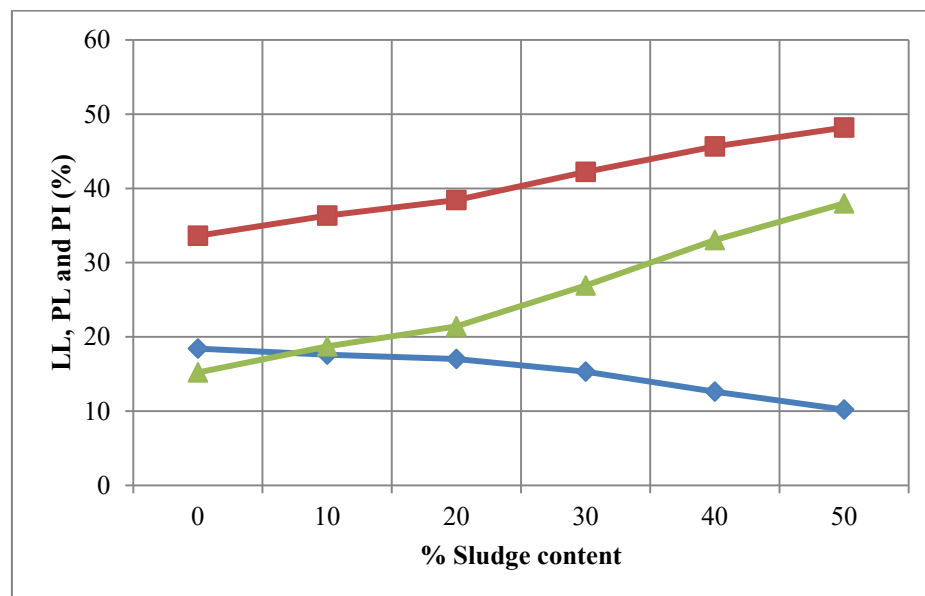


Figure 4.1. Relationship between Plastic Limit, Liquid Limit and Plasticity Index with sludge content.

The Plasticity Index of the soil used in brick manufacturing process is 18.42 (Table 4.6) and can be classified as highly plastic material. The Plasticity Index of sludge – clay mixture ranged from 17.6, 17.01, 15.32, 12.63 and 10.21 for 10%, 20%, 30%,

40% and 50% sludge-clay mixture respectively. According to the soil properties stated in Table 4.5, 10% as well as 20% of sludge can be used in brick making without losing the major plastic behavior.

Table 4.6. Effect of moisture content on OMC, Dry density, Liquid Limit, Plastic Limit, and Plasticity Index of sludge-clay mixtures

Parameters	Sludge content (% by dry weight)					
	0	10	20	30	40	50
Optimum Moisture Content (%)	17.1	22	25	28	32	38
Dry density (gm/cm <sup>3</sup> )	1.77	1.69	1.64	1.58	1.51	1.45
Liquid Limit (%)	33.63	36.33	38.42	42.23	45.66	48.19
Plastic Limit (%)	15.21	18.73	21.41	26.91	33.03	37.98
Plasticity Index (%)	18.4	17.6	17.01	15.32	12.63	10.21

#### 4.2.2 Compaction test

Moisture content has the potential to affect the properties of bricks. Up to a certain point additional water replaces air from the soil voids, but, after a relatively high degree of saturation is reached, the water occupies space which would otherwise be occupied with soil particles (Lambe, 1993). Therefore, an optimum amount of mixing water in brick manufacturing process is usually sought which would give the maximum level of compaction for a given amount of soil sample. This will result in increased bulk density, lower tendency of water absorption and eventually the highest compressive strength. The results of the compaction test from Figure 4.2 shows that OMC increases with the increase of sludge incorporation into the mixture. On the other hand dry density of sludge – clay mixture decreased with the increase of sludge content to the mixture.

#### 4.3 Characteristics of Burnt Bricks

After brick burning, the produced bricks were received a series of tests including firing shrinkage, weight loss on ignition, water absorption and compressive strength following standard methods.

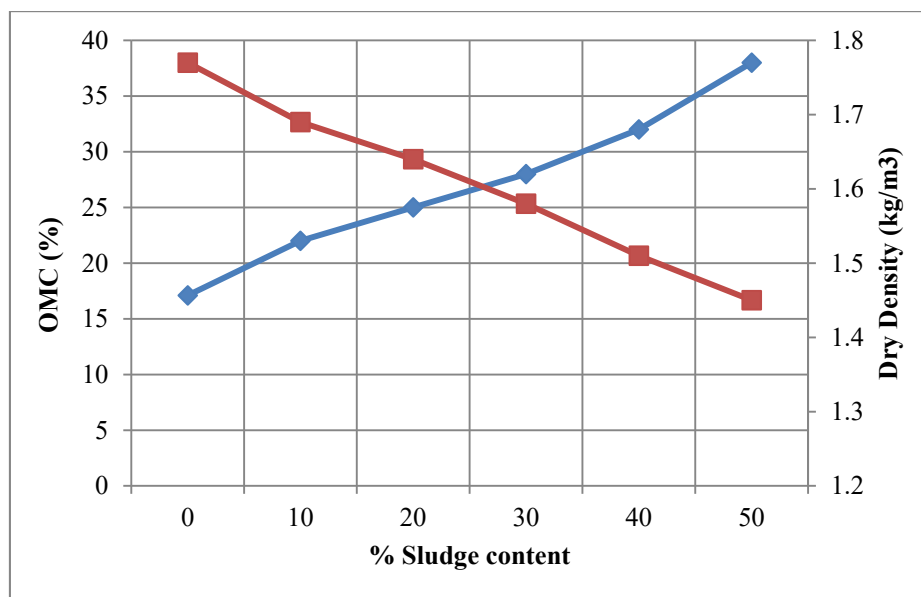


Figure 4.2. Effect of sludge addition on optimum moisture content and bulk density of mixtures.

#### 4.3.1 Loss on brick drying and burning

200 number of brick from each sludge-clay ration were molded including 200 number of clay bricks. During the drying phase it was observed that some of the brick were broken apart. Total number of brick that were wasted during drying was noted. Same phenomenon observed during burning. Numbers of brick broken during burning were noted also. The number of bricks lost during drying and burning is given in table 4.7 and graphical representation is given in figure

Table: 4.7.: Brick quantity lost during drying and burning

Sludge Content (%)	Loss in drying (%)	Loss in Burning (%)	Total Loss (%)
0	3	5.67	8.67
10	4.5	7.33	11.8
20	5.5	8.47	14.0
30	8	10.3	18.3
40	9.5	19.3	28.8
50	11.5	24.9	36.4



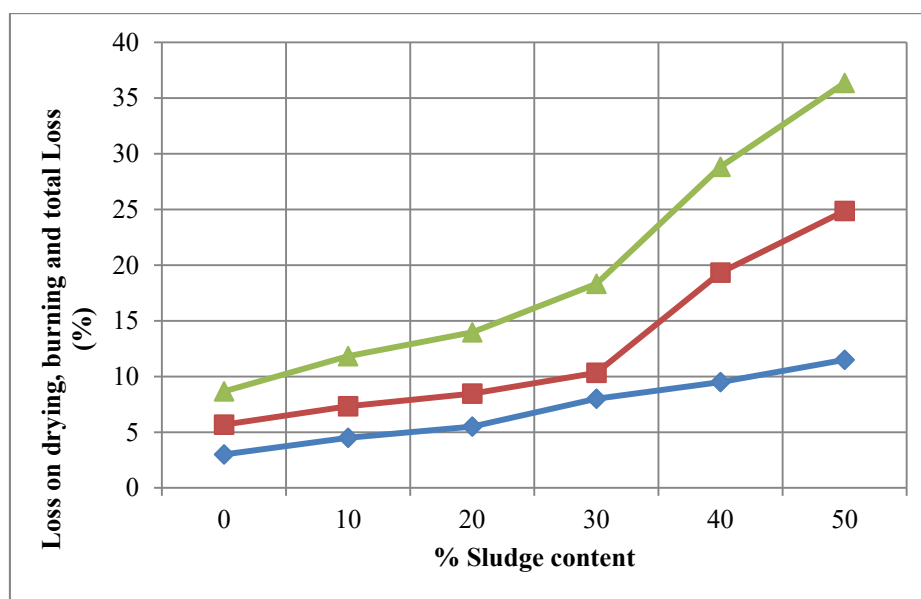


Figure 4.3 Quantity of bricks lost during drying and burning with different sludge-clay composition.

From table 4.7 and figure 4.3 it can be deduced that the loss of brick during drying and burning increase with the sludge content. This might be due to the less plastic nature of the sludge-clay mixture. From the plasticity index of the different sludge-clay mix table 4.6 it was found that the plastic behavior of the mix reduces with respect to the sludge content. Bricks with non-plastic mixture have less bonding capacity results bricks splitting. From the table 4.7, total loss of bricks varies from 8.67% to 36.4% when the DS proportion was increased from 0% to 50%.

#### 4.3.2 Compressive strength of bricks

The compressive strength test is the key test for ensuring the engineering quality of a building material. The strength of a material, in general terms, is its ability to resist forces at failure. The results of the average compressive strength test of 3 bricks from each composition were shown in Figure 4.4. The compressive strength of DS incorporated bricks ranged from 101.281 kg/cm<sup>2</sup> (9.93 MPa) to 249.92 kg/cm<sup>2</sup> (24.5 MPa). It appears that the content of sludge has a profound effect on the compressive strength of bricks.

Compressive strength has been found to be inversely proportional to the sludge content. This proportional trend may be due to increase in porosity.

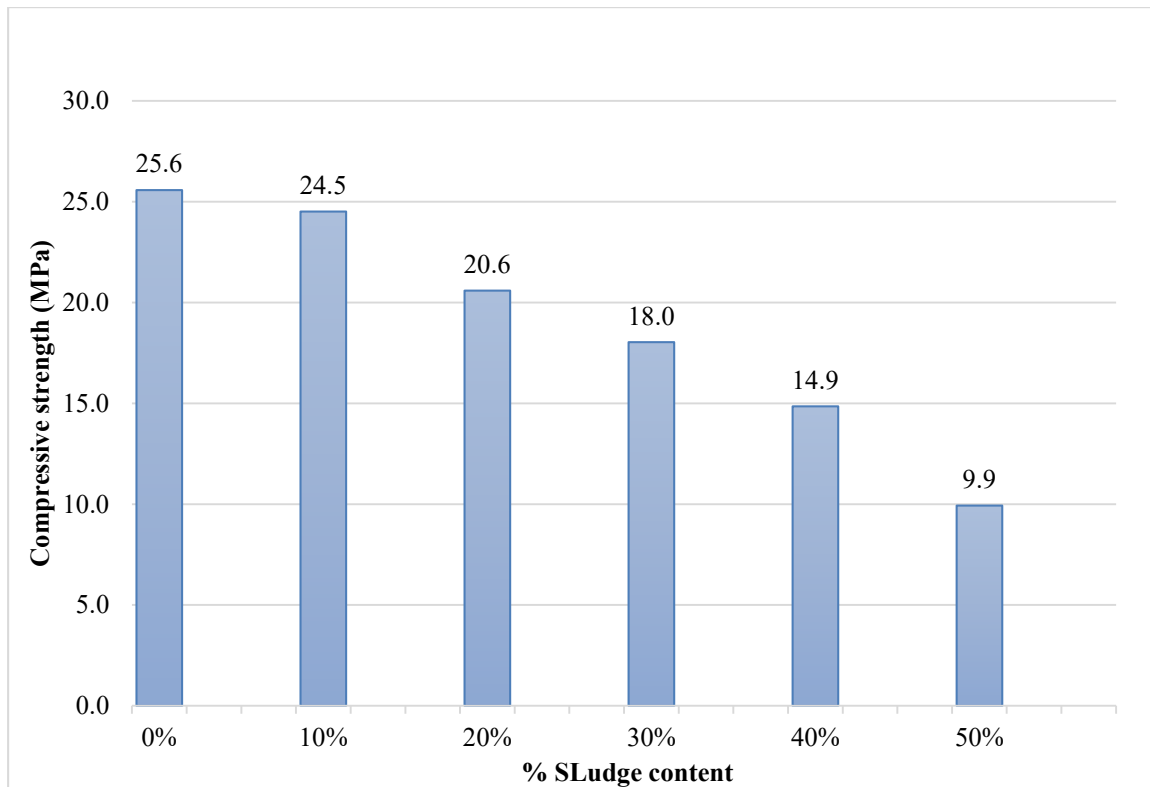


Figure 4.4. Compressive strength of bricks at different clay-sludge ratio.

Table 4.8. Criteria of bricks in Bangladesh (BDS 208, 2009)

Name of Parameters	Grade S	Grade A	Grade B
Compressive strength (kg/cm <sup>2</sup> )	≥ 245	≥ 154	≥ 105
Water absorption (%)	≤ 10	15	≤ 20
Weight loss on ignition (%)	≤ 15	≤ 15	≤ 15
Firing shrinkage (%)	≤ 8	≤ 8	≤ 8

**Note: Grade S:** This type of bricks may be used for breaking into aggregate for plain and reinforced concrete and for making base course of pavement. **Grade A:** This type of bricks may be used in construction of buildings of long duration. **Grade B:** This type of bricks may be used for one storied building, temporary shed, where intended durability is not very long.

The comparison of the properties of textile dying sludge bricks found in this study with that found in other similar studies are shown in Table 4.9. It shows Similar pattern of compressive strength reduction with sludge content in other studies using

textile industries sludge (Jahagirdar et al. 2013, Baskar et al. 2006, Begum et al., 2013)

Table 4.9. Performance comparison for sludge incorporated bricks with previous attempts involving textile sludge. The % increase/reduction in the parenthesis indicates how much the specific parameter changed compared to the reference brick (i.e. 0% sludge).

Physical and Mechanical Properties	Present study 10% Textile dyeing sludge in bricks	Previous Study on Textile Sludge in Brick		
		9% Textile sludge in bricks (Baskar et al. 2006)	10% Textile sludge in bricks (Jahagirdar et al. 2013)	10% Textile sludge in bricks (Begum et al., 2013)
Firing temperature °C	2100-2400	800	800	800
Compressive strength (MPa)	24.5 (4.3% reduction)	3.65 (44% reduction)	6.5 (44% reduction)	5.5 (30% reduction)
Water absorption (%)	12.8 (12% increment)	15.8 (52 % increment)	18 (11 % increment)	13
Firing shrinkage (%)	13.2 (14 % increment)	-	-	-
Weight loss on ignition (%)	13.3 (23% increment)	7.54 (55% increment)		4
Bulk density (kg/m <sup>3</sup> )	1530 (Not affected)		1600 (13% reduction)	2100

From table 4.9 it seems that the compressive strength varies from 3.65 MPa to 6.5 MPa for the previous study whereas the compressive strength found 24.5 MPa in current study. For the current study the compressive strength is much higher than the previous study mentioned above. This higher value in current study may results due to the higher firing temperature applied in brick manufacturing process. So it seems that the firing temperature affects the quality of bricks that is the compressive strength of bricks are proportional with the firing temperature.

Table 4.10 shows that the addition of 50% sludge into the mixture reduces strength approximately by 61.2% compared with the control bricks (i.e. 0% sludge). The addition of 10%, 20%, 30%, 40% and 50% sludge into the mixture reduces strength

approximately by 4.2%, 19.5%, 29.5%, 41.9% and 61.2% respectively compared with the control bricks (i.e. 0% sludge).

Table 4.10. Compressive strength of fired bricks

<b>% Sludge content</b>	<b>Avg. Compressive Strength (MPa)</b>	<b>Avg. Compressive Strength (Kg/cm<sup>2</sup>)</b>	<b>% Strength Reduced with respect to 100% Clay brick</b>
0%	25.6	260.79	0.00
10%	24.5	249.94	4.20
20%	20.6	209.99	19.5
30%	18.0	183.87	29.5
40%	14.9	151.45	41.9
50%	9.93	101.28	61.2

According to BDS 208 (2009) Standards (Table 4.8) 10% DS bricks can be considered as Grade S category where as 20% and 30% DS bricks can be considered as Grade A category. 40% DS bricks can be considered as Grade B category. 50% DS brick samples have failed to meet the both ASTM (ASTM C 62) and Bangladesh BDS 208 (2009) standards which set out that the value of compressive strength should be greater than 87.69 kg/cm<sup>2</sup> (8.6 Mpa) and 105 kg/cm<sup>2</sup> respectively.

### 4.3.3 Water absorption of bricks

The degrees of firmness and compaction of bricks, as measured by their water absorption characteristics, vary considerably depending on factors such as the type of clay and methods of production used (BS 1377, 1990). Water absorption is a key factor affecting the durability of brick. The less water infiltrates into brick, the more durability of the brick and resistance to the natural environment are expected (Weng et al. 2003). Thus, the internal structure of the brick must be intensive enough to avoid the intrusion of water.

Figure 4.5 shows the average results of the water absorption of 10 brick samples from each type of clay- sludge composition as function of sludge content. It has been found

that the water absorption of the bricks increased with increased sludge addition thereby potentially increasing its susceptibility to weathering action. Generally, sludge contains high amount of organic content which generates pore spaces within the brick during firing and these pore spaces favor water absorption.

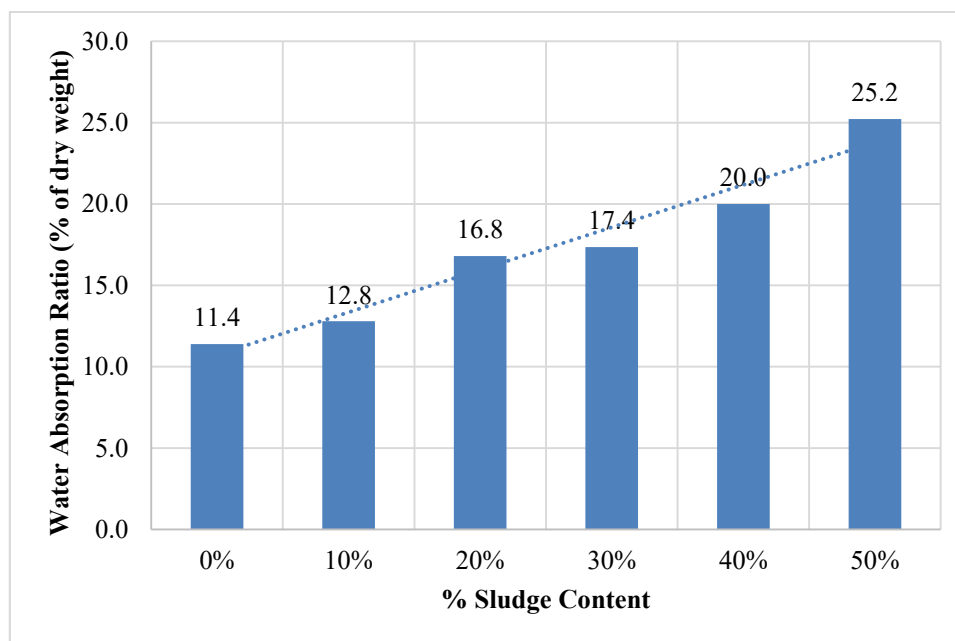


Figure 4.5. Water absorption of bricks as function of sludge content.

The Dyeing sludge used in these experiments contained low amount of organic content (4.3%, see Table 4.1) however it was found that the quantity of absorbed water increased with the increase of DS proportion. In case of bricks containing 10% (by wt.) sludge, water absorption was 12.8%. On the other hand water absorption increased from 11.4% to 25.3% when DS proportion was increased from 0% to 50% (Fig. 4.5). Similar trends in water absorption with sludge fraction in bricks and other construction materials have been observed in other studies (Chiang et al. 2009, Liew et al. 2004, Weng et al. 2003).

According to the criterion of water absorption of bricks stated in BDS 208 (2009) Standards (Table 4.8), bricks made with 10% sludge can be regarded as Grade A category bricks and 20%, 30% and 40% DS incorporated brick lies in category B.

#### 4.3.4 Weight loss on ignition

The brick weight loss on ignition is not only attributed to the organic matter content in the clay, but it also depends on the inorganic substance in both clay and sludge being burnt off during the firing process (Karaman et al., 2006). The effect of sludge content

on weight loss of 10 bricks is shown in Figure 4.6. It was found that the weight loss of bricks increased as percent of sludge increased. The control bricks showed the lowest weight loss of 10.8%, while the 50% sludge incorporated bricks showed the highest weight loss of 24.7%. This weight loss could be due to the combustion and decomposition of the organic and inorganic matter present in both the sludge and clay during the firing process (Weng et al.2003, Liew et al., 2004).

Normally, the weight loss criterion for a clay brick is 15% (AASHTO T- 99, 1982). In this regard, bricks incorporating 10%, 20% & 30% sludge have met weight loss criteria.

#### 4.3.5 Brick firing shrinkage

Firing shrinkage is also an important parameter for ensuring the quality of bricks. Shrinkage in bricks occurs as chemically and mechanically bound water is lost during firing (Karaman et al., 2006). It is very similar to the weight loss in ignition parameter; the only difference is that firing shrinkage measures the volumetric deformation of bricks during firing or drying stages of production while the other is a measure of weight loss though the reasons for shrinkage can also cause weight loss of bricks.

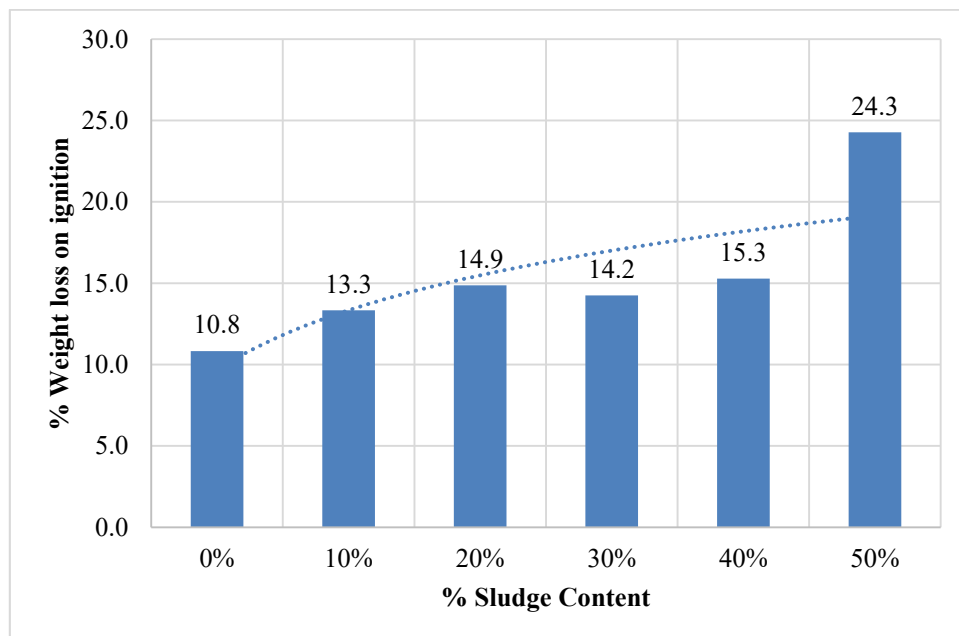


Figure 4.6. Weight loss of bricks on ignition as function of sludge content.

High shrinkage is an undesirable property for any kind of engineering material. Figure 4.7 shows the average volumetric shrinkage of 10 bricks due to firing as a function of different sludge content.

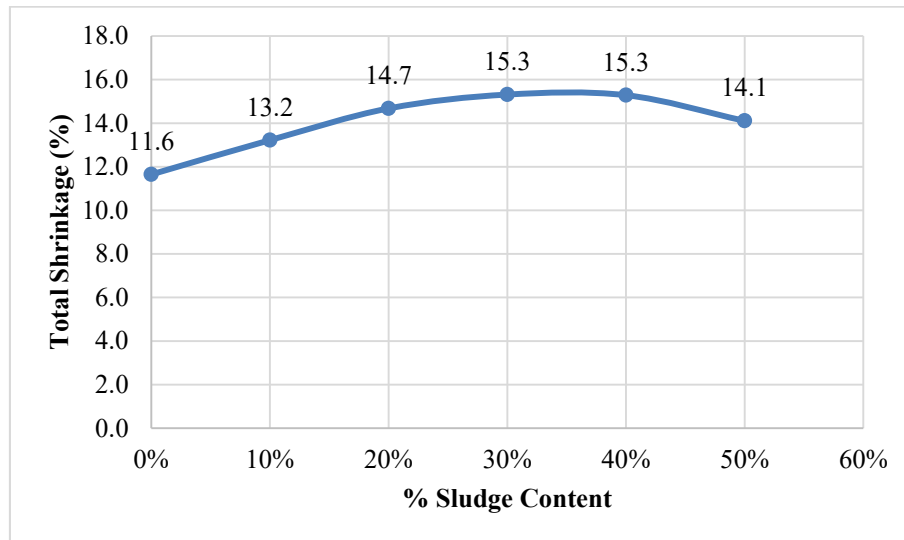


Figure 4.7. Shrinkage of bricks as function of sludge content.

Total shrinkage in this study increase with the increase of sludge addition up to 30% but decreased with higher sludge content. For 20% sludge amended bricks the volumetric shrinkage reduced by up to 15.3% where the 100% clay bricks total shrinkage was 11.6%. There are two possible reasons for this. Firstly, the decreasing trend obtained here may be due to non-plastic nature of dried sludge. Rhodes and Hopper (2000) reported that low plastic soil shrinks less than high plastic soil. Secondly, the decreasing trend obtained here may be due to expansion in the bricks during the firing stage. Bricks incorporated with sludge could release more gases compared to control bricks due to burning of organic matter, generating voids which caused a net expansion within the sludge-clay matrix and eventually resulting in a lower net shrinkage (Ukwatta et al., 2016).

#### 4.3.6 Bulk density

The average bulk density at different percentage of 10 DS-amended bricks is shown in Figure 4.8. From the experimental results it an inverse relationship found between the bulk density of the DS-amended bricks and the amount of sludge added in the mixture. In this study, bulk density decreased from 1.53 to 1.35 gm/cm<sup>3</sup> (reduced of 11.5%) as the DS content increased from 0% to 50%. This finding is closely related to the quantity of water absorbed as shown in Figure 4.5.

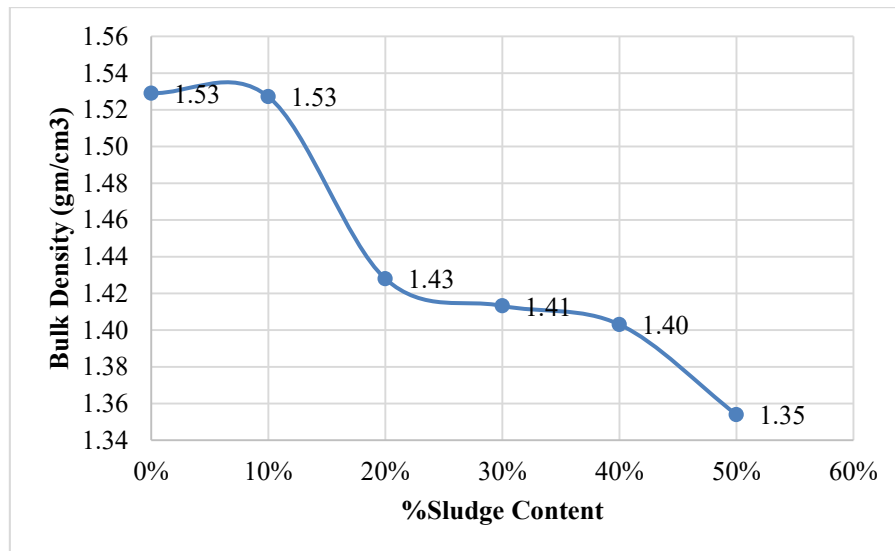


Figure 4.8. Bulk density of the bricks as function of sludge content.

Higher water absorption ratio indicates larger pore size than the one with lower water absorption, which results smaller bulk density. Though with the lower bulk density corresponds higher water absorption which is related with the quality of brick however the light weight bricks has a good potential in construction for their lower dead load, ease in handling, and savings in cost in transportation. Bricks with low bulk density also possess good thermal insulation property. Internal structure of bricks is shown in Figure 4.9. From Figure 4.9, it can be seen that the porosity of bricks increase with the increase of sludge content as well as the colour of the brick changes from dark reddish to light reddish. This is one of the reasons for varying different properties of sludge brick such as compressive strength, water absorption and bulk density with the sludge content.



Figure 4.9. Internal structure of different DS brick



#### 4.4 Environmental Aspects of Sludge Amended Bricks

##### Heavy metal leaching

Leaching of heavy metal is the prime concern as heavy metal contaminated sludge is introduced to the finished products. It should be ensured that leaching of heavy metal from bricks does not exceed the maximum permissible limit even under extreme condition. TCLP leaching test was carried out in this study.

The Toxicity Characteristics Leaching Procedure (TCLP) test is designed to identify wastes that are likely to leach hazardous concentrations of particular toxic constituents into the groundwater. During the TCLP test, constituents are extracted from the waste to simulate leaching actions that actually occur in landfills. If the concentration of the toxic constituents exceeds the regulatory limit, the waste is classified as hazardous.

The leaching analysis according to USEPA 1311 from sludge amended bricks is given in Table 4.11.

Table 4.11. TCLP test result of DS amended bricks

Metal	Raw Sludge	0% DS Sludge Brick	10% DS Sludge Brick	20% DS Sludge Brick	30% DS Sludge Brick	40% DS Sludge Brick	50% DS Sludge Brick	USEPA
As (mg/L)	0.23	0.02	0.017	0.014	0.014	0.013	0.01	5
Cd (mg/L)	0.15	0.01	0.013	0.013	0.019	0.02	0.027	1
Cr (mg/L)	1.8	0.031	0.044	0.045	0.071	0.08	0.082	5
Cu (mg/L)	0.61	0.08	0.088	0.196	0.515	0.669	0.106	
Hg (mg/L)	0.002	n.d	n.d	n.d	n.d	n.d	n.d	0.2

Ni (mg/L)	0.22	n.d	n.d	n.d	n.d	n.d	n.d	
Pb (mg/L)	0.26	0.008	0.008	0.041	0.041	0.041	0.046	5
Zn (mg/L)	0.5	0.38	0.34	0.33	0.26	0.21	0.168	

---

“n.d” = not detected; Detection limit > 0.001 mg/l; <sup>a</sup> United States Environmental Protection Agency (USEPA) (1996).

Hg and Ni did not leach from the sludge amended bricks as their concentrations were far below detectable levels. Though other metals such as Cr, Cd, Pb, Cu and As leached from sludge amended bricks, but although the concentrations are far below the US-EPA regulatory limits. From Table 4.10 it is revealed that the leaching concentration of As, Cr, Cd, Cu and Pb are reduced by 92.6%, 97.5%, 91.3%, 85.6% and 96.9%, respectively in case of 10% sludge amended bricks with respect to the original sludge.

From above leaching test results, it can be concluded that sludge incorporated bricks can be widely used in construction without causing any significant environmental hazard even after end of life cycle of these bricks.

## Chapter 5

### Conclusion and Recommendation

#### 5.1 Conclusion

In this study the effect of incorporation sludge generated from ETP of dyeing industry in fired-clay bricks has been investigated based on their physical and mechanical properties as well as on their environmental aspects. The bricks were manufactured incorporating 0%, 10%, 20%, 30%, 40% and 50% (by dry weight) of dyeing sludge and the relevant engineering properties of the sludge incorporated fired-clay bricks were assessed.

The major findings of this study are as follows:

- a) Atterberg's limit test indicates that Plasticity Index of clay – sludge mixture reduces with increase of % sludge content and up to 20% of sludge can be applied to brick making without losing the plastic behaviour.
- b) Number brick loss (broken apart) during the manufacturing process depends on the sludge content in the mixture. Total loss of bricks varies from 8.67% to 36.4% when the DS proportion was increased from 0% to 50%. This might be due to the change in plastic behaviour of the sludge-clay mix from plastic to non-plastic for sludge addition.
- c) The compressive strength of DS incorporated bricks ranged from 249.9 kg/cm<sup>2</sup> (24.5 MPa) to 101.3 kg/cm<sup>2</sup> (9.93 MPa) to when DS content increased from 0% to 50%. The addition of 50% sludge into the mixture reduces strength approximately by 61.2% compared with the control bricks (i.e. 0% sludge). The addition of 10%, 20%, 30%, 40% and 50% sludge into the mixture reduces strength approximately by 4.2%, 19.5%, 29.5%, 41.9% and 61.2% respectively compared with the control bricks (i.e. 0% sludge).
- d) Water absorption increased from 11.39% to 25.23% when DS proportion was increased from 0% to 50%. According to the criterion of water absorption of bricks stated in BDS 208 (2009) Standards (Table 4.8), bricks made with 10% sludge can be regarded as Grade A category bricks and 20%, 30% and 40% DS incorporated brick lies in category B.

- e) The clay bricks showed the lowest weight loss on ignition 10.83%, while the 50% sludge incorporated bricks showed the highest weight loss of 24.27%. This weight loss could be due to the combustion and decomposition of the organic and inorganic matter present in both the sludge and clay during the firing process.
- f) Firing shrinkage increases with increase of sludge content up to 30% sludge incorporation then decreases with increase of sludge content.
- g) Bulk density decreased from 1.53 to 1.35 gm/cm<sup>3</sup> (reduced of 11.5%) as the DS content increased from 0% to 50%. Though with the lower bulk density corresponds higher water absorption which is related with the quality of brick however the light weight bricks has a good potential in construction for their lower dead load and good thermal insulation
- h) TCLP results for leaching concentration of targeted heavy metals analyzed in this study for all samples found to be negligible compared to the USEPA regulatory limits. Considering other properties 10% sludge amended brick is suitable for the brick masonry construction.

Based on the results obtained from the different test for the DS incorporated bricks, 10% sludge content by dry weight good quality bricks which comply all the desirable mechanical and physical properties of ASTM and BDS standards. Also the leaching of toxic metals in the environment is negligible cause no concern for environmental hazard. Textile Dyeing sludge incorporation can be a potential approach supplement the demand of raw soil for brick production and stabilize waste into construction materials.

## **5.2 Limitation of the Study**

- a) In this study only one textile dyeing sludge was investigated where there are a lot of dying industries around. Sludge from different dyeing industries may have a wide range of constituents (including toxic metals).
- b) In field condition with large amount of brick manufacturing the clay-sludge mixing may not be uniform.
- c) OMC, which is a very important factor, may not be maintained exactly on the field.
- d) As the bricks were manufactured in a conventional brick kiln, temperature may not be controlled uniformly.

- e) Results may vary in case of different aged sludge which was not considered in this study.

### **5.3 Recommendation for Future Work**

- a) This research can be carried out with the other available textile dyeing industries sludge in Bangladesh.
- b) Different researchers showed that sludge generated from sewage, ternary can be successfully utilized through concrete, pavement blocks and ceramics. So, similar studies can be carried out for dyeing sludge.
- f) Similar approach using the advance auto brick manufacturing system can be assessed.
- g) Similar approach can be carried out using incinerated sludge ash.
- h) Similar approach can be carried out using admixtures with clay-sludge mix.

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## Appendix A

Appendix A contains the average results of different mechanical and physical properties of bricks and also contains the respective calculations of different test of DS bricks.

Table A- 1. Compressive strength of DS bricks

<b>Compressive Strength Test</b>					
<b>% Sludge content</b>	<b>Designation</b>	<b>Area (mm<sup>2</sup>)</b>	<b>Calibrated load (KN)</b>	<b>Avg. Compressive Strength (MPa)</b>	<b>Avg. Compressive Strength (Kg/cm<sup>2</sup>)</b>
<b>0%</b>	01	13338	348.321	25.575	260.791
	02	13452	363.141		
	03	14625	345.357		
<b>10%</b>	11	13776	335.477	24.511	249.940
	12	13334	341.405		
	13	13685	322.633		
<b>20%</b>	21	13340	286.077	20.593	209.986
	22	14040	267.305		
	23	13110	279.161		
<b>30%</b>	31	12656	239.641	18.031	183.867
	32	13447	228.773		
	33	12880	233.713		
<b>40%</b>	41	14152	210.001	14.853	151.455
	42	13572	207.037		
	43	13221	191.229		
<b>50%</b>	51	13338	138.865	9.932	101.281
	52	13924	143.805		
	53	13804	125.033		

Table A- 2. Average water absorption of 10 samples from each type.

<b>Sample Type</b>	<b>% Sludge Content</b>	<b>Water Absorption (%)</b>
Type - 0	0%	11.39
Type - 1	10%	12.80
Type - 2	20%	16.80
Type - 3	30%	17.36
Type - 4	40%	20.00
Type - 5	50%	25.23

Table A- 3. Average weight loss on ignition of 10 samples from each type.

<b>Sample Type</b>	<b>% Sludge Content</b>	<b>Wt. loss on ignition (%)</b>
Type - 0	0%	10.83
Type - 1	10%	13.33
Type - 2	20%	14.87
Type - 3	30%	14.25
Type - 4	40%	15.28
Type - 5	50%	24.27

Table A- 4. Average firing Shrinkage of 10 samples from each type.

<b>Sample Type</b>	<b>% Sludge Content</b>	<b>Total Shrinkage (%)</b>
Type - 0	0%	11.65
Type - 1	10%	13.22
Type - 2	20%	14.68
Type - 3	30%	15.32
Type - 4	40%	15.29
Type - 5	50%	14.11

Table A- 5. Average bulk density of 10 samples from each type.

<b>Sample Type</b>	<b>% Sludge Content</b>	<b>Bulk Density</b>
Type - 0	0%	1.529
Type - 1	10%	1.527
Type - 2	20%	1.428
Type - 3	30%	1.413
Type - 4	40%	1.403
Type - 5	50%	1.354

## **Appendix B**

### **Bangladesh Standard**

#### **Specification for Common Building Clay Bricks (3<sup>rd</sup> Revision)**

### **1. Scope**

This standard lays down requirements for dimensions, strength and other quality requirements of common burnt clay building bricks.

### **2. Terminology**

For the purpose of this standard following definitions shall apply

#### **2.1 Bricks**

The burnt clay building bricks which are commonly used in building and civil engineering construction purposes.

### **3. Dimensions**

3.1 Size- The standard dimensions of common burnt clay bricks shall be of following:

Table 1: Size of Bricks

<b>Dimension</b>	<b>cm</b>
Length	24
Width	11.5
Height / Depth	7

NOTE- One bedding face of each brick shall have a recess, panel or frog. The size of the frog for the standard brick shall not exceed 13cmx 5cmx 1cm as shown in fig. 1. This would not apply to brick manufactured by extrusion process or any special bricks required by the purchaser.

3.2 Variation - Small variation for Grade A and B in the dimension shall be permissible to the following extent only:

**Table 2: Maximum Permissible Variation of Bricks**

<b>Specified Dimension</b>	<b>Maximum Permissible Variation</b>
Over 5 cm and up to 7.5cm	± 2.0 mm
Over 7.5 cm and up to 10 cm	± 3.0 mm
Over 10 cm and up to 15 cm	± 5.0 mm
Over 15 cm and up to 25 cm	± 6.0 mm

#### 4. Classification

Three grades of bricks have been incorporated based on their strength properties. This will include reasonable variation in the quality of clays available locally.

**Table 3: Classification Of Bricks**

<b>Grade</b>	<b>Mean for twelve halved bricks</b>	<b>Minimum for individual halved bricks</b>
S	280 kg <sup>f</sup> /cm <sup>2</sup>	245 kg <sup>f</sup> /cm <sup>2</sup>
A	175 kg <sup>f</sup> /cm <sup>2</sup>	154 kg <sup>f</sup> /cm <sup>2</sup>
B	140 kg <sup>f</sup> /cm <sup>2</sup>	105 kg <sup>f</sup> /cm <sup>2</sup>

**Grade S:** This type of bricks may be used for breaking into aggregate for plain and reinforced concrete and for making base course of pavement.

**Grade A:** This type of bricks may be used in construction of buildings of long duration.

**Grade B:** This type of bricks may be used for one storied building, temporary shed, where intended durability is not very long.

#### 5. Water Absorption

Water absorption by weight shall not exceed 20% for bricks of Grade B and 15% for bricks of Grade A and 10% for bricks of Grade S.

#### 6. Measurement of Dimension and Size

6.1 Number of Specimens- 24 bricks, for carrying out dimensional tests as per procedure set out in clauses 6.3 to 6.5 of this standard shall be taken.

6.2 Preparation of specimens - Any blister or other small project together with any loose particles of clay which might have adhered to the face of brick shall be removed before the bricks are assembled for measurement.

6.3 Length- a) 24 Bricks laid end to end on a level surface in contact in a straight line shall measure between 568 cm to 588 cm when all frog face upwards as shown in Figure 2.

b) 24 Bricks laid in a similar manner as in clause 6.3 (a) above but having alternate frogs facing upwards and downwards shall also measure between 568 cm to 588 cm as shown in Figure 3. The difference between the length noted in clause 6.43 (a) above and this system will not be more than 17 mm

6.4 Width- a) 24 Bricks laid side by side on level surface in contact in a straight line shall measure between 281 cm to 291 cm when all frogs face upwards as shown in Figure 4.

b) 24 Bricks laid in a similar manner as in clause 6.4 (a) above but having alternate frogs facing upwards and downwards shall also measure between 281 cm to 291 cm as shown in Figure 5. The difference between the length noted in clause 6.4 (a) and this system will not be more than 8 mm.

6.5 Depth- 24 bricks laid on edge bedding surface to bedding surface on a level surface in contact in a straight line will measure 165 cm to 171 cm

NOTE: - When bricks without frogs are to be measured the system for clauses 6.3 and 6.4 shall be arranged by reversing sides of alternate bricks.

6.6 Measurement- The overall length of the assembled bricks shall be measured with a steel tape of other suitable inextensible measure long enough to measure the whole row at once. Measurement by repeated application of a short rule or measure shall not be considered satisfactory. If, for any reason, it is found impracticable to measure 24 bricks in one row the samples may be divided into two rows of 12 bricks which shall be measured separately to the nearest of 1.5 mm to their measurement added,

6.7 Compliance - If the measurements in clauses 6.3 to 6.5 are fulfilled, the whole consignment from which the sample is drawn shall be deemed to comply with the specification and these specimens shall thereafter be used for other specified tests. If



the specimens do not comply, the whole consignments from which the specimens are drawn shall be rejected and no further tests in regard to crushing strength, water absorption and efflorescence are needed to be carried out.

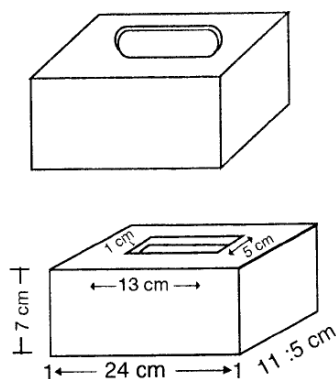


FIG NO. 1

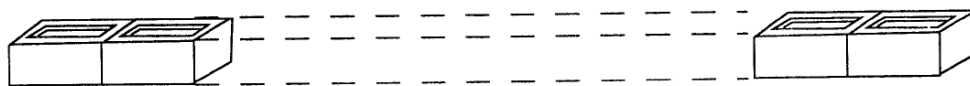


FIG NO. 2

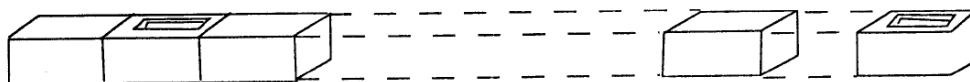


FIG NO. 3



FIG NO. 4



FIG NO. 5

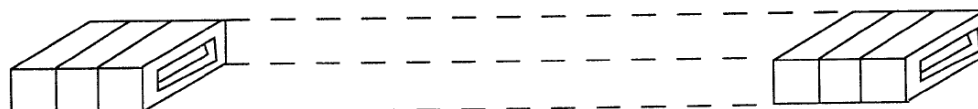


FIG NO. 6

## 7. Determination of Crushing Strength

7.1 Specimens – Twelve bricks taken at random from sample shall be halved and one half from each whole brick used for determining the crushing strength as detailed in 7.2. The overall dimension of each bedding face shall be measured to the nearest of 1.3 mm and the area of the face having smaller area shall be taken as the area of the bricks for testing the crushing strength.

Sample sizes for crushing strength are as follows:

**Table 4: Sample Size for Crushing Strength**

Lot size	Sample size
2.001 to 10.000	5
10,001 to 35,000	10
35.001 to 50.000	15

## **7.2 Procedure**

Bricks without frogs - These shall be immersed in water at 15°C to 20°C for 24 hours. They shall then be removed and allow to dry at room temperature for about 5 minutes. Their frogs shall be filled with cement sand mortar of 1: 1 ½, sand being clean and well graded passing 3.2 mm sieve. The mortar shall be trowelled off flush with surface of the bricks. The bricks shall then be stored under the damp sacks for 24 hours after filling the frogs and shall then be immersed in water for six days before bricks are considered ready for testing\*. After seven days of filling the mortar, specimens will be taken out wiped dry with damp cloth and used plaster of Paris or sulfur capping to ensure a uniform surface of brick for crushing strength test and carefully centered between the plates of the compression testing machine. The compressing plates of the testing machine shall have a blasting in the form of a portion of sphere, the center of each coinciding with the center of the face of the plate. The load shall be applied axially at a uniform rate of 140 kg/cm<sup>2</sup> per minute, until failure.

**7.3 Results-** Maximum load in kilogram at failure divided by the minimum area of the bedding surface of the half bricks in square centimeter shall be taken as the crushing strength whole limiting values are given in 7.4. The main crushing strength of twelve half bricks along with that of each individual half bricks shall be noted.

**7.4 Limiting value** - The value of crushing strength in kilogram per square centimeter shall not be less than the figures given in table 3.

**7.5 Compliance** - If the mean crushing strength of twelve bricks falls below the limiting crushing strength given 7.4 above, the batch from which the sample is taken shall be deemed not to comply. If however, any individual brick gives the crushing strength less than the minimum value for an individual brick given in 7.4 above, the

test will be repeated as detailed in Para 7.2 over twice the number of bricks that have failed provided the number of individual brick not complying of any does not exceed clause 4. If on retest the crushing strength of any individual bricks still falls below the minimum value given in para 7.4 the sample as a whole shall be deemed not to comply with the crushing strength values.

## **8. Determination of Water Absorption**

8.1 Specimen- The specimens shall consist of whole bricks. Six specimens shall be tested for water absorption as detailed in 8.2.

8.2 Procedure - The test specimen shall be dried in a ventilated oven at 110°C-115°C for 48 hours or more until constant weight. The specimen shall be deemed to have reached the constant weight when after 2 hours drying in the same over the loss in weight does not exceed 0.1%. Each specimen shall immediately be weighed which shall be called the dry weight of the specimen. The dry specimen shall then be cooled in air at room temperature for about 2 hours after which they shall be immersed completely in clean soft water at \*\* 15°C for 24 hours. Each specimen shall then be removed from the water, the surface wiped off with a damp cloth and the specimen weighed. Weighing of any one specimen shall be completed within 3 minutes after removing the specimen from the water. This shall be called the wet weight.

\* When the test on the mortar is cubes shows that the compressive strength of the mortar is not less than 280 kgf/cm<sup>2</sup> and not exceeds 420 kgf/cm<sup>2</sup>. The failure shall be deemed to have occurred when no further increase in the load is registered with unchanged rate of moving head travel.

\*\* Specimen noticeably warm to the touch shall not be immersed water.

8.3 Result- The percentage of water absorption by weight shall be calculated as,

$$\frac{W_2 - W_1}{W_1} \times 100$$

Where,

W<sub>1</sub> = Dry weight of the specimen

W<sub>2</sub> = Wet weight of the specimen

8.4 Limiting value - The average value of the six specimens shall be taken as the water absorption of the batch shall be given in art 5.

8.5 Compliance - In case the average absorption value of the specimen exceeds the limiting value given in clause 8.4 above the test will be repeated on the same number of bricks. If after the test the absorption value exceeds the prescribed limit the batch from which the samples taken shall be deemed not to comply with this clause.

## 9. Initial Rate of Absorption (Suction)

### 9.1 Apparatus:

9.1.1 Trays or Containers - Watertight trays or containers, having an inside depth of not less than 12.7 mm and of such length and width that an area of not less than 1935.5cm<sup>2</sup> of water surface is provided. The bottom of the tray shall provide a plane, horizontal upper surface, when suitably supported, so that an area not less than 203.2 mm in length by 9.2.4 mm in width will be level when tested by a spirit level.

9.1.2 Supports for Brick/Block – Two non-corrodible metal supports consisting of bars between 127 mm and 152.5 mm in length, having triangular, half round, or rectangular cross section such that the thickness (height) will be approximately 6.35 mm. The thickness of the two bars are rectangular in cross section, their width shall not exceed 1.94 mm.

9.1.3 Means for Maintaining Constant Water Level – Suitable means for controlling the water level above the upper surface of the supports for the brick/block within + 0.25 mm (see note 3) , including means for adding water to the tray at a rate corresponding to the rate of removal by the brick/block undergoing test (see note 4). For use in checking the adequacy of the method of controlling the rate of; flow of the added water, a reference brick/block or half brick/block shall be provided whose displacement in 3.18mm of water corresponds to the brick/block or half brick/block to be tested within + 2.5 %. Completely submerge the reference brick/block in water for not less than 3 h preceding its use.

Note - A suitable means for obtaining accuracy in control of the water level may be provided by attaching to the end of one of the bars two stiff metal wires that project upward and return, terminating in points; one of which is 3.18 – 0.25 mm and other 3.18 – 0.25 mm above the upper surface or edge of the bar. Such precise adjustment is obtainable by the use of depth plates of a micrometer microscope. When the water

level with respect to the upper surface of edge of the bar is adjusted so that the lower point dimples the water surface when viewed by reflected light and the upper point is not in contact with the water, the water level is within the limits specified. Any other suitable means for fixing and maintaining a constant depth of immersion may be used if equivalent accuracy is obtained. As an example or such other suitable means, there may be mentioned the use of rigid supports movable with respect of the water level.

Note -A rubber tube leading from a siphon of gravity feed and closed by a spring clip will provide a suitable manual control. The so-called “chicken-feed” devices as a rule lack sensitivity and do not operate with the very small changes in water level permissible in this test.

9.1.4 Balance, having a capacity of not less than 3000 g. and sensitive to 0.5 g.

9.1.5 Drying Oven that provides a free circulation of air through the oven and is capable of maintaining a temperature between 110 and 115° C.

9.1.6 Constant – Temperature Room, maintained at a temperature of  $21 \pm 1.4^{\circ}\text{C}$ .

9.1.7 Timing device - A suitable timing device, preferably a stop watch or stop clock, which shall indicate a time of 1 min to the nearest 1 s.

9.2 Test specimen, consisting of whole brick/block. Five specimens shall be tested.

9.3 Procedure:

9.3.1 Drying- Dry the test specimens in a ventilated oven at 110 to 115° C for not less than 24 h and until two successive weighing at intervals of 2 hours show an increment of loss not greater than 0.2 % of the last previously determined weight of the specimen.

9.3.2 Cooling – After drying, cool the specimens in a drying room at a temperature of  $24 \pm 8^{\circ}\text{C}$ , with a relative humidity between 30 and 70 %. Store the units free from drafts, unshackled, with separate placement for a period of at least 4 h. Do not use specimens noticeably warm to the touch for any test requiring dry units.

9.3.2.1 An alternative method of cooling the specimens to approximate room temperature may be used as follows: Store units, unstacked, with separate placement, in a ventilated room for a period of 4 h, with a current of air from an electric fan passing over them for a period of at least 2h.

9.3.2 Measure to the nearest 1.27mm the length and width of the flatwise surface of the test specimen of rectangular units or determine the area of other shapes to similar accuracy that will be in contact with the water. Weigh the specimen to the nearest 0.5 g.

9.3.3 Adjust the position of the tray for the absorption test so that the upper surface of its bottom will be level when tested by a spirit level, and set the saturated reference brick (9.1.3) in place on top of the supports. Add water until the water level is  $3.18 + 0.25$  mm above the top of the supports. When testing tile with scored bed surfaces, the depth of water level is  $3.18 + 0.25$  mm plus the depth of scores.

9.3.4 After removal of the reference brick/block, set the test brick/block in place flatwise, counting zero time as the moment of contact of the brick/block with the water. During the period of contact (1 min + 1 s) keep the water level within the prescribed limits by adding water as required. At the end of 1 min + 1 s, lift the brick/block from contact with the water, wipe off the surface water with a damp cloth, and reweigh the brick/block to the nearest 0.5 g. Wiping shall be completed within 10 s of removal from contact with the water, and weighing shall be completed within 2 min.

Note – Place the brick/block in contact with the water quickly, but without splashing. Set the brick/block in position with a rocking motion to avoid the entrapping of air on its under surface. Test brick/block with frog of depression uppermost.

#### 9.4 Calculations and Report:

9.4.1 The difference in weight in grams between the initial and final weighings is the weight in grams of water absorbed by the brick/block during 1-min contact with the water. If the area of its flatwise surface (length times width) does not differ more than  $+ 4.84 \text{ cm}^2$  (+ 2.5%) from  $193.55 \text{ cm}^2$ , report the gain in weight in grams as the initial rate of absorption in 1 min.

9.4.2 If the area of its flatwise surface differs more than  $+ 4.84 \text{ cm}^2$  (+ 2.5%) from  $193.55 \text{ cm}^2$  as follows:

$$X = \frac{193.55W}{LB}$$

Where:

$X$  = Gain in weight corrected to basis of  $193.55 \text{ cm}^2$  flatwise area,

$W$  = actual gain in weight of specimen, g

$L$  = length of specimen, cm and

$B$  = width of specimen, cm.

9.4.3 Report the corrected gain in weight,  $X$ , as the initial rate of absorption 1min.

9.4.4 If the test specimen is a cored brick calculate the net area and substitute for  $LB$  in the equation given in 9.4.2. Report the corrected gain in weight as the initial rate of absorption in 1 min.

9.4.5 If specimen is non-prismatic, calculate the net area by suitable geometric means and substitute for  $LB$  in the equation given in 9.4.2.