

SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
UNIVERSITI SAINS MALAYSIA

STUDY ON RECYCLING OF INDUSTRIAL STEEL WASTE IN THE PRODUCTION OF
PORCELAIN TILES

by

Afif Hafizhan bin Azman

Supervisor : Dr. Shah Rizal Kasim
Co-Supervisor : Dr. Khairul Anuar Shariff

Dissertation submitted in partial fulfillment
of the requirements for the degree of Bachelor of Engineering with Honours
(Materials Engineering)

Universiti Sains Malaysia

August 2022

**STUDY ON RECLYING OF INDUSTRIAL
STEEL WASTE IN THE PRODUCTION OF
PORCELAIN TILES**

AFIF HAFIZHAN BIN AZMAN

UNIVERSITI SAINS MALAYSIA

2022

DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled 'Study On Recycling Of Industrial Steel Waste In The Production Of Porcelain Tiles'. I also declare that it has not been previously submitted for the award of any degree and diploma or other similar title of this for any other examining body or University.

Name of Student: Afif Hafizhn Bin Azman

Signature:

Date: 18 August 2022

Witness by

Supervisor: Dr Shah Rizal Kasim

Signature:

Date: 18 August 2022

ACKNOWLEDGEMENT

I would like to express my deep gratitude to School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia (USM) led by our present Dean, Profesor Ir. Dr. Mariatti Jaafar for the given opportunities to further increase my knowledge related to materials engineering in undergraduate programme. My honest and sincere appreciation to my supervisor, and co-supervisor Dr. Shah Rizal Bin Kasim and Dr. Khairul Anuar Shariff for their patient guidance and regular supervision and encouragement in successfully completing this research especially in providing and sharing multiple extra knowledge and information in ceramic tile industry.

Thank you to Ceramic Research Company (CRC), Guocera Tile Industry (Meru) Sdn. Bhd., and Dr Chin Chee Lung for giving me an opportunity to do my final year project with CRC. A special thanks goes to my field supervisor, Dr Wei Tze Mook, for her advice and assistance in keeping my progress on schedule. She helped me in completing the project and exchanged her useful ideas and thoughts with me. Thank you, my parents, Mr Azman Bin Abd Razak and Mrs. Salmah Binti Said for their constant support and leading me to end of the road of this study life. They deserve special mention for their inseparable support and prayers. I also want to thank all my family members and friends who motivated me in this project. On the other hand, I would like to deliver my appreciation to all lecturers, technicians, staffs and members of the School of Materials and Mineral Resources Engineering and all staff for providing the facilities and supports during the progress of the final year project.

TABLE OF CONTENTS

ACKNOWLEDGEMENT	ii
TABLE OF CONTENTS	iii
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	x
LIST OF SYMBOLS	xi
KAJIAN KITAR SEMULA SISA BUANGAN INDUSTRI KELULI DALAM PENGHASILAN JUBIN PORSELIN ABSTRAK	xii
STUDY ON RECLYING OF INDUSTRIAL STEEL WASTE IN THE PRODUCTION OF PORCELAIN TILES	xiv
CHAPTER 1 INTRODUCTION	1
1.1 Research Background.....	1
1.2 Problem Statement	3
1.3 Objectives.....	5
1.4 Scope of Research	5
CHAPTER 2 LITERATURE REVIEW	8
2.1 Introduction of Porcelain Tiles.....	8
2.1.1 History of Ceramic Tiles	9
2.1.2 Raw Materials	12
2.1.2(a) Clay.....	12
2.1.2(b) Silica	13
2.1.2(c) Feldspar.....	14
2.1.3 Production Process of Porcelain Tiles.....	15
2.1.4 Different Types of Tiles	17
2.1.4(a) Porcelain Tiles	17
2.1.4(b) Wall Tiles.....	18

2.1.4(c)	Floor Tiles.....	19
2.2	Introduction to Steel Making.....	21
2.2.1	Basic Oxygen Furnace (BOF).....	22
2.2.2	Electric Arc Furnace (EAF).....	23
2.2.3	Secondary Steelmaking or Ladle Furnace (LF).....	24
2.2.4	Production of EAF Slag and LF Slag.....	25
2.3	Recycling Steel Slag Waste into Ceramic Products.....	26
2.3.1	Steel Slag in Ceramic Tiles.....	26
2.3.2	Steel Slag in Concrete and Cement.....	27
CHAPTER 3 MATERIALS AND METHODOLOGY.....		28
3.1	Introduction.....	28
3.2	Raw Materials.....	31
3.2.1	Clay.....	31
3.2.2	Silica.....	31
3.2.3	Feldspar.....	31
3.2.4	LF Steel Slag.....	31
3.2.5	EAF Steel Slag.....	32
3.3	Characterization of Raw Materials.....	32
3.3.1	X-Ray Fluorescence (XRF).....	32
3.3.2	X-Ray Diffraction (XRD).....	33
3.3.3	Particle Size Analyzer (PSA).....	33
3.3.4	Scanning Electron Microscope (SEM).....	34
3.4	Fabrication of Porcelain Tile.....	34
3.4.1	Raw Material Preparation.....	34
3.4.2	Milling-Mixing.....	37
3.4.3	Drying and Grinding.....	37
3.4.4	Pressing.....	37

3.4.5	Drying	37
3.4.6	Firing	38
3.5	Characterization of Fired Body	38
3.5.1	Appearance Observation	39
3.5.2	Linear Shrinkage	39
3.5.3	Modulus of Rupture (MOR).....	40
3.5.4	Water Absorption, Apparent Porosity, and Bulk Density	42
3.5.5	X-Ray Diffraction for Phase Analysis	43
3.5.6	Scanning Electron Microscope (SEM) morphology Analysis	43
CHAPTER 4 RESULTS AND DISCUSSION		44
4.1	Characterization of Raw Materials.....	44
4.1.1	X-ray Fluorescence (XRF).....	44
4.1.2	Particle Size Analysis (PSA).....	46
4.1.3	X-Ray Diffraction (XRD)	47
4.1.3(a)	Clay.....	47
4.1.3(b)	Silica	48
4.1.3(c)	Potash Feldspar.....	49
4.1.3(d)	EAF Slag.....	50
4.1.3(e)	LF Slag.....	52
4.1.4	Scanning Electron Microscope (SEM).....	54
4.2	Characterization of Fired Sample.....	56
4.2.1	Appearance Observation	56
4.2.2	Linear Shrinkage	58
4.2.3	Water Absorption, Apparent Porosity, and Bulk Density	60
4.2.4	Modulus of Rupture (MOR).....	64
4.2.5	X-Ray Diffraction (XRD)	66
4.2.6	Scanning Electron Microscope (SEM).....	71

CHAPTER 5	CONCLUSION AND RECOMMENDATION	73
5.1	Conclusion.....	73
5.2	Recommendation.....	74
REFERENCES		75
APPENDICES		

LIST OF TABLES

		Page
Table 2.1	Percentage of Water Absorption according to of types of tiles (Standards Australia, 2003).....	19
Table 2.2	Minimum value of Modulus of Rupture (MOR) according to types of tiles (Bechthold et al., 2015).....	20
Table 3.1	Composition of Steel Slag added in Porcelain Tiles.....	36
Table 4.1	Percentage of Chemical Composition for Each Raw Materials.....	45
Table 4.2	Particle Size Analysis of Raw Materials.....	46
Figure 4.1	XRD pattern of clay powder	
	Table 4.3 Percentage of mineral phases in clay powder	48
Table 4.4	Percentage of mineral phase in silica powder	49
Table 4.5	Percentage of mineral phase in feldspar powder.....	50
Table 4.6	Percentage of mineral phase in EAF slag powder	52
Table 4.7	Percentage of mineral phase in LF slag powder	54
Table 4.8	Appearance comparison between control, EAF slag, and LF slag at different firing temperature	57
Table 4.9	Percentage of mineral phase in the porcelain tiles.....	70

LIST OF FIGURES

	Page
Figure 2.1	The example of colourful wall tiles (Bernardin et al., 2006)..... 11
Figure 2.2	The porcelain tiles used at the mosque (Toplicic-Curcic et al., 2018) 11
Figure 2.3	The summary of production of ceramic tiles (Toplicic-Curcic et al., 2018a)..... 17
Figure 2.4	Blast Furnace (Bayer Ozturk & Eren Gultekin, 2015) 23
Figure 2.5	Electric Arc Furnace (Penteado et al., 2019) 2 4
Figure 2.6	Ladle Furnace (Penteado et al., 2019)..... 25
Figure 3.1	The summarize of three stages involve in the research work 30
Figure 3.2	Firing profile for sample of porcelain tiles 38
Figure 3.3	Schematic diagram of three-point bending test (Curkovic et al., 2010) 41
Figure 4.1	XRD pattern of clay powder Table 4.3 Percentage of mineral phases in clay powder 48
Figure 4.2	XRD pattern for Silica powder 49
Figure 4.3	XRD pattern of feldspar powder 50
Figure 4.4	XRD pattern of EAF slag 51
Figure 4.5	XRD pattern of LF slag powder 53
Figure 4.6	SEM microstructure of raw materials powder at 500x magnification (a) clay, (b) feldspar, (c) silica, (d) EAF slag, and (e) LF slag..... 55
Figure 4.7	Percentage of linear shrinkage by using LF slag at firing temperature 1200°C and 1210°C 59

Figure 4.8	Percentage of linear shrinkage by using EAF slag at firing temperature 1200°C and 1210°C	59
Figure 4.9	Percentage of water absorption for LF slag at firing temperature 1200°C and 1210°C	61
Figure 4.10	Percentage of water absorption for EAF slag at firing temperature 1200°C and 1210°C	62
Figure 4.11	Percentage of apparent porosity for LF slag at firing temperature 1200°C and 1210°C	62
Figure 4.12	Percentage of apparent porosity for EAF slag at firing temperature 1200°C and 1210°C	63
Figure 4.13	Bulk density for LF slag at firing temperature 1200°C and 1210°C	63
Figure 4.14	Bulk density for EAF slag at firing temperature 1200°C and 1210°C	64
Figure 4.15	Modulus of Rupture (MOR) for LF slag at firing temperature 1200°C and 1210°C	65
Figure 4.16	Modulus of Rupture (MOR) for EAF slag at firing temperature 1200°C and 1210°C	66
Figure 4.17	XRD pattern of control sample at firing temperature 1200°C and 1210°C	67
Figure 4.18	XRD pattern of optimum sample (20LF/20F fired at 1210°C).....	67
Figure 4.19	XRD pattern for tile with addition 40wt% EAF slag.....	69
Figure 4.20	SEM morphology of fired samples at 100x magnification (a) control fired at 1200°C, (b) control fired at 1210°C, (c) optimum tiles (20LF/20F) fired at 1210°C, and (d) tile with addition 40wt% EAF slag.....	71

LIST OF ABBREVIATIONS

Al ₂ O ₃	Alumina
AP	Apparent porosity
BD	Bulk density
BF	Blast furnace
BO	Basic oxygen
CaO	Calcium Oxide
CRC	Ceramic Research Company
EAF	Electric arc furnace
ICDD	International Centre for Diffraction Data
ISO	International Standard Organization
K ₂ O	Potassium Oxide
LF	Ladle Furnace
MOR	Modulus of rupture
SEM	Scanning electron microscope
SiO ₂	Silica
TiO ₂	Titanium Dioxide
WA	Water absorption
XRD	X-ray diffraction
XRF	X-ray Fluorescence
ZnO	Zinc Oxide

LIST OF SYMBOLS

°C	Degree Celsius
%	Percentage
MPa	Mega pascal
μm	Micron meter
g	gram
nm	nano meter
μ	Micron
wt%	Weight percent
g/cm ³	Gram per cubic centimeter
mm	millimeter

KAJIAN KITAR SEMULA SISA BUANGAN INDUSTRI KELULI DALAM PENGHASILAN JUBIN PORSELIN

ABSTRAK

Sisa buangan keluli industri juga dikenali sebagai slag keluli merupakan bahan sampingan semasa penghasilan keluli. Lambakan slag keluli di tapak pelupusan sampah akan memberi kesan terhadap flora dan fauna selain boleh menyebabkan pencemaran tanah di kawasan sekitar. Oleh itu, kitar semula sisa buangan keluli merupakan salah satu cara untuk mengurangkan lambakan slag keluli di kawasan pembuangan sampah. Dalam kajian ini, keluli slag EAF dan LF telah digunakan. Tujuan kajian ini untuk menghasilkan jubin porselin dengan komposisi yang berbeza keluli slag (LF dan EAF) pada suhu pembakaran 1200°C dan 1210°C. Kajian ini juga bertujuan untuk menilai kesan komposisi keluli slag yang berbeza kepada jubin porselin yang terhasil terhadap sifat mekanikal dan fizikal. Dalam kajian ini, 9 komposisi berbeza dihasilkan, salah satu daripadanya adalah kawalan. Komposisi keluli slag digunakan dalam kerja kajian ini adalah 40wt%, 30wt%, 20wt% dan 10wt%. Kesemua bahan mentah (tanah liat, feldspar, silica, dan keluli slag) dicampur menggunakan kaedah campuran dan pengisaran basah dan diikuti proses pengeringan. Selepas itu, campuran kering dikisar sehingga menjadi serbuk dan ditekan kepada bentuk segi empat tepat dengan dimensi 103mm X 39mm X 3.6mm pada tekanan 37 MPa, seterusnya, jubin porselin dibakar pada suhu 1200°C dan 1210°C. Jubin porselin melalui pencirian rupa bentuk, kecutan linear, penyerapan air, liang ketara, ketumpatan pukal, modulus kepecahan (MOR), pembelauan sinar-X (XRD), dan analisa morfologi elektron mikroskop (SEM). Keputusan diperolehi daripada kajian ini menunjukkan jubin porselin dengan komposisi 20wt% keluli slag LF dan 20wt% feldspar pada suhu pembakaran 1210°C mempunyai penyerapan air terendah (0.01%), liang ketara

terendah (0.03%), ketumpatan pukal tertinggi (2.6g/cm^3) dan nilai modulus kepecahan tertinggi (33.68 MPa).

STUDY ON RECLYING OF INDUSTRIAL STEEL WASTE IN THE PRODUCTION OF PORCELAIN TILES

ABSTRACT

Industrial steel waste also known as steel slag is a by-product from the steel making industry. The dumping of steel slag in the landfill are affecting the flora and fauna and can causes for land pollution around the area. So, recycling of the steel waste into another product is one of the best methods to reduce the steel waste in the landfill. In this research work, the Electric Arc Furnace and Ladle Furnace slag was used. The objective of this experiment is to fabricate porcelain tiles with various composition of steel slag (LF and EAF slag) at firing temperature 1200°C and 1210°C. This study also to evaluate the effect of different composition of steel slags at different firing temperature 1200°C and 1210°C to physical and mechanical properties of porcelain tiles. In this study, nine compositions are set, and one of them is set as a control. The composition of the steel slag used in this research work is 40wt%, 30wt%, 20wt%, 10wt%. Firstly, the raw materials (clay, feldspar, silica, steel slag) were mixed and milled, followed by drying. Next, the dried mixture was pressed for a rectangular shape (103 mm X 39 mm X 3.6 mm) at pressure 37 MPa and then the tiles were fired at temperatures 1200°C and 1210°C. The fired tiles were characterized by appearance observation, linear shrinkage, water absorption, apparent porosity, bulk density, modulus of rupture (MOR), X-Ray Diffraction (XRD), and scanning electron microscope (SEM) for morphology observation. Result obtained from this research work show that the tiles with composition 20wt% LF slag and 20wt% feldspar at firing temperature 1210°C has lowest water absorption (0.01%), apparent porosity (0.03%), highest bulk density (2.6g/cm³) and has highest value of MOR (33.68 MPa).

CHAPTER 1

INTRODUCTION

1.1 Research Background

Porcelain tiles are one of the ceramic products that produced from clay, feldspar and silica which are fired at high temperatures to form a glassy and crystalline phase (Sánchez et al., 2010). Clay is a primary raw material used in producing porcelain tiles because it gives plasticity and binding characteristics to the porcelain tiles. Feldspar also important component to produce porcelain tiles since it acts as a fluxing agent to the porcelain body (Sánchez et al., 2010). Fluxing agent is used to reduce the firing temperature during firing process to form a glassy phase. During the drying and firing process of porcelain tiles, silica acts as a diffuser for escaping gases and it is also used to increase the whiteness of the fired tiles and reducing the drying shrinkage of the tiles (Correia et al., 2004).

Based on the previous study by Teo et al., (2014), the steel slag is introduced to replace partially the feldspar as a fluxing agent. The electric arc furnace (EAF) steel slag can partially replace the feldspar in production of porcelain tiles with addition of different ratio of feldspar and steel slag. The composition of 40 wt% EAF steel slag, 30 wt% ball clay, 10 wt% feldspar, and 20 wt% silica is the optimum of composition of tiles produced with high flexural strength, lowest water absorption and lowest apparent porosity (Teo et al., 2014).

EAF steel slag are one of the by-products from the steel fabrication process when steel scrap is melted in an electric arc furnace (Shi, 2004). In the EAF steel making process, the molten steel will be transferred to the ladle furnace (LF) for further secondary refining process (Penteado et al., 2019). During secondary refining process,

the steel slag also produced as a by-product that called LF steel slag. In the steel making process, one tonne steel will produce around 0.13 to 0.2 tonne of steel slag as their by-product (Yu & Wang, 2011). Basically, all steel slag consists of Calcium Oxide (CaO), Magnesium Oxide (MgO), Silicon Dioxide (SiO₂), and Iron Oxide (FeO) as their chemical elements (Shi, 2004). However, the composition of each chemical elements in all steel slags are different based on the type of steel being produced and type of furnace used during steel making process. For example, based on study by Shi (2004), for production of alloy and stainless steels by using EAF furnace, the EAF steel slag have low content of FeO whereas when producing the carbon steel, the content of FeO is higher (Shi, 2004). In addition, the LF steel slag also have significant different in chemical composition compared to EAF steel slag. This is because, the composition of LF steel slag is depending on refining process. For example, in some steel making process, Calcium Fluoride (CaF₂) is used for further refining purpose, therefore the composition of CaO and SiO₂ is higher in the LF steel slag compared to EAF steel slag (Shi, 2004).

Therefore, in this research work, two types of steel slag which are Ladle Furnace (LF), and Electric Arc Furnace (EAF) are used. The steel slag wastes are chosen because the steel slags contain of fluxing elements such as Calcium Oxide (CaO) and Magnesium Oxide (MgO) that can replace feldspar. This research work is carried out to fabricate the porcelain tiles with varies composition of LF steel slag with feldspar also EAF steel slag with feldspar at firing temperature 1200°C and 1210°C. This study also will evaluate the effect of different ratio of steel slag waste with feldspar at different firing temperature 1200°C and 1210°C to physical and mechanical properties of porcelain tiles.

1.2 Problem Statement

Steel is the one of the important materials that used in construction and manufacturing industries. It is the main materials in every engineering design or structure such as vehicles and machinery manufacturing as well as for buildings construction. The finished steel consumption is increasing between 2018 to 2019 in Malaysia which is from 10.6 million tons to 10.8 million tons respectively (World Steel Association, 2020). This trend causes the steel production of hot rolled will also be increasing in Malaysia between 2018 to 2019 from 3.8 million tons to 5.3 million tons respectively (World Steel Association, 2020). In 2019, almost 89.4% of steel making in Malaysia are from electric arc process. Since the production of steel in Malaysia is growing over a year, this causes the quantity of steel slag produced as a waste product of the steel making process would also rise. Basically, the steel slag that produced from the steel making process will be end up in the landfill and incineration (Koros, 2003). Over a year, the dumping of steel slag in the landfill are affecting the flora and fauna and can causes for land pollution around the area. In addition, the rising amount of steel waste demands the usage of a great deal of land, which increases the expense of disposing of steel slag (Das et al., 2007). Moreover, the steel slag disposal by using incineration also can causes a problem to the steel maker industry since it is costly disposal technique and uses extremely high energy to dispose the steel slag. Since the steel slag is considered as toxic waste, this disposal technique is not recommended because it can cause the hazardous ash to enter the human lung during the disposal process of steel slag (Aguirre et al., 2009).

In order to reduce the dumping of steel slag wastes, it can be recycled into another product as a raw material. Recycling is one of the efficient techniques to reduce the waste in the world. According to the study by Teo et al., (2014), the EAF steel slag

can be recycled to use as a fluxing agent in production of heavy-duty ceramic tiles. In production of ceramic tiles, usually feldspar is used as a raw material for the fluxing agent. In addition, based on study by Sarkar et al., (2010), the composition of 30-40wt.% of EAF steel slags, 40-50wt.% of clay, 10-20wt.% feldspar and 10wt.% quartz is used to produce the ceramic tiles. The addition of 30-40wt.% EAF steel slag in the ceramic tiles shows relatively higher density and shorter firing range with good strength properties to the final product (Sarkar et al., 2010). In another study by Dong et al., (2021), the steel slag aggregate also can be recycled into Portland cement concrete. Because of the steel slag aggregate have high density, rough surface, and contain of cementitious ingredients compared to natural aggregates, it causes the recycling of steel slag aggregates can improve the strength in concrete (Dong et al., 2021).

The purpose of this research work is to fabricate the porcelain tiles with addition of varies composition between feldspar and steel slags waste by using two types of steel slag. This study also to evaluate the effects to properties of the porcelain tiles when steel slag is added in the composition of porcelain tiles at different firing temperatures.

1.3 Objectives

The objectives of this study are:

1. To fabricate porcelain tiles with varies composition of LF steel slag with feldspar also EAF steel slag with feldspar at firing temperature 1200°C and 1210°C.
2. To evaluate the effect of different composition of steel slags with feldspar at different firing temperature 1200°C and 1210°C to physical and mechanical properties of porcelain tiles.

1.4 Scope of Research

In this study, nine compositions are set, and one of them is set as a control. The composition of clay and silica is fixed at 50wt% and 10wt%, respectively, for all the sets of experiments. The samples with a composition 40wt% of feldspar are set as a control, whereas for another eight sets of experiments, the composition between feldspar and steel slag are varies. This study consists of 3 stages, first stage is raw materials characterization, second stage is porcelain tiles fabrication, and third stage is characterization of fired samples.

In the first stage, all the raw materials including clay, silica, feldspar, EAF steel slag, and LF steel slag are characterized by using X-Ray Fluorescence (XRF), Particle Size Analyzer (PSA), Scanning Electron Microscope (SEM), and X-Ray Diffraction (XRD). All the tests are performed in order to verify and validate the raw materials received before the next stage can be proceed. In the second stage, the porcelain tiles are fabricated through the process, batching, mixing, milling, pressing, drying and firing. In this stage, the rectangular tiles with dimension 103 mm X 39 mm X 3.6 mm are fabricated with pressing pressure 37 MPa. The tiles are fired at temperatures

1200°C and 1210°C. For the third stage, the fired porcelain tiles are subjected for characterization analysis. The analysis including, appearance observations, percentage of linear shrinkage, percentage of water absorption, percentage of apparent porosity, bulk density, SEM observation, XRD analysis and Modulus of Rupture (MOR). Figure 1.1 show the flowchart of this research work.

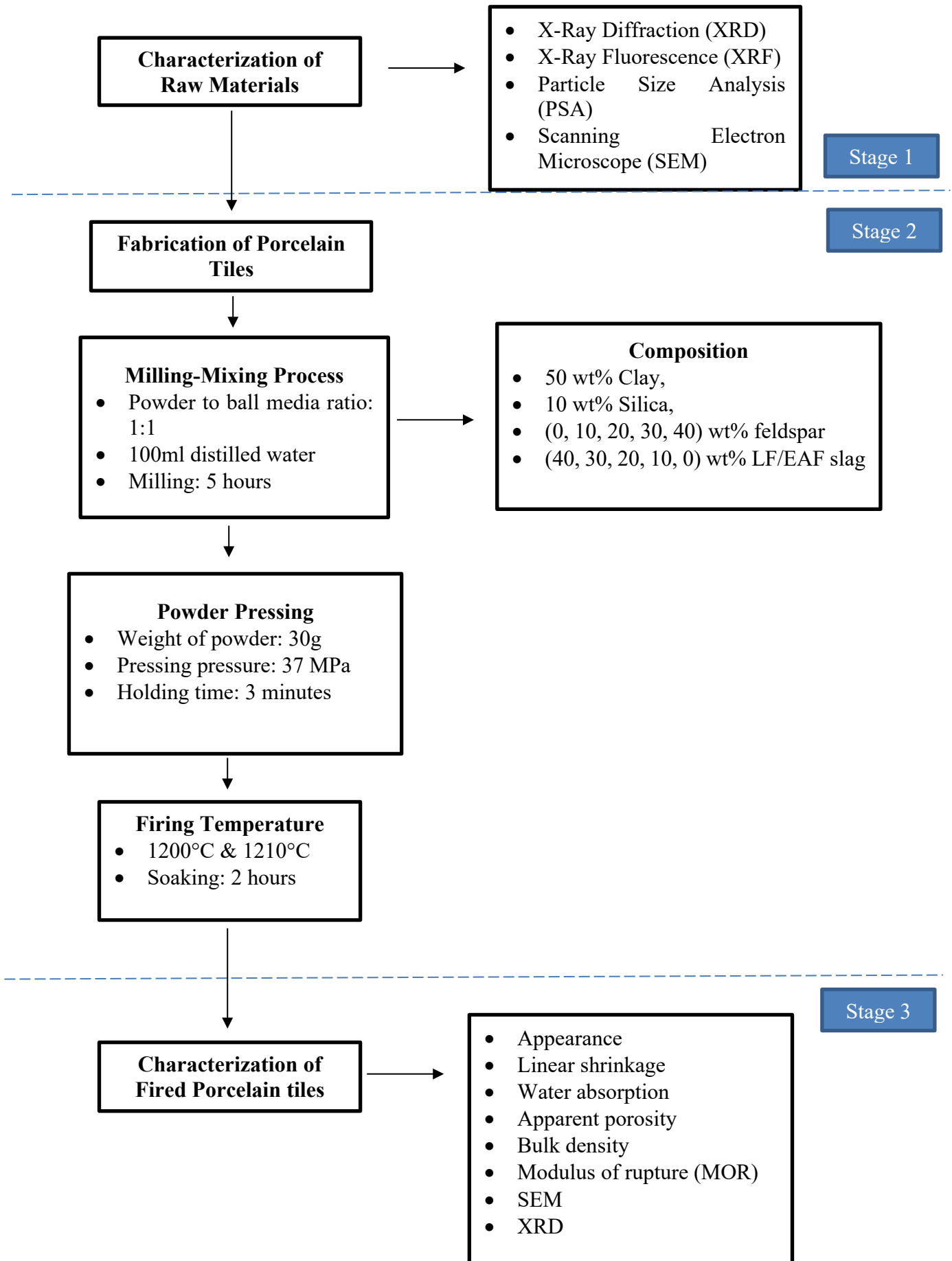


Figure 1.1: The summarize of three stages involve in the research work

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Porcelain Tiles

Porcelain tiles are one of the ceramic materials that produced from clay, feldspar, and silica which are fired at high temperatures to form a glassy and crystalline phase. Unlike any other ceramic product, porcelain tile first appeared in Italy in the late 1970s as a high performer with a natural look and that are more comparable to natural rock or stone. As a result of its high breaking threshold, great resistance to abrasion, and ease of cleaning, it is an excellent choice for industrial and heavy-trafficked paving applications (Sánchez et al., 2010).

Clay or kaolin, quartz, and feldspar are combined in a triaxial combination to provide the basic composition. The clay fraction that aids in shaping by giving plasticity and dry mechanical strength during processing, and it also generates mullite and glassy phase after firing. It is possible to obtain almost no open porosity which is less than 0.5% and a very low degree of closed porosity which is less than 10% when using feldspars even at low temperatures firing temperature. Because of its high melting point, quartz is good for thermal and dimensional stability (Sánchez et al., 2010).

In the manufacture of porcelain tiles, firstly, wet milling and homogenization of raw materials are followed by spray drying of the resulting suspension. The spray-dried powder is then subjected to 35–45 MPa uniaxial pressing and a fast firing at 1180–1220°C for 40–60 minutes to achieve maximum densification (Sánchez et al., 2010).

2.1.1 History of Ceramic Tiles

Ancient civilizations including Egypt, Mesopotamia, China, and the Islamic Empire are thought to be the first user of ceramic tile (Van Lemmen, 2013). Tiles from Tunisia, Kashan Iran and many mosques in the Middle East depict Koranic letters in relief by utilising brightly coloured relief tiles. Europe's churches were embellished with tiles in the 13th and 14th centuries. At the same period, several sites in England, such as York and Winchester, have yielded early European tiles. Figure 2.1 shows the example of porcelain tiles. Alhambra Palace and Cordoba's Great Mosque are two of the best places to see some of the most beautiful ceramic tiles from that era because they were considered a costly building material at the time. Glazed tiles are said to have been employed in ancient civilisations like Egypt and Mesopotamia to embellish homes and buildings (Toplicic-Curcic et al., 2018).

To this day, the mosque, madrasa, and palace walls in Muslim architecture have been decorated with mosaic or ceramic tile. When Islamic culture predominated, wall tiles became a popular decorative element. When it comes to the art of Islamic tilework, 16th and 17th century was the golden period. The use of brilliantly coloured tile in the interiors of buildings was common at the time. Figure 2.2 shows the porcelain tiles used at the mosque. Glazed tiles are said to have been employed in ancient civilisations like Egypt and Mesopotamia to embellish homes and buildings (Toplicic-Curcic et al., 2018).

It was common practise in northern Europe throughout the 13th century to paint the floors of churches, royal palaces, and private homes with a variety of floral design and figurative motifs and religious symbols. Traditionally, tiles for hot wax painting were produced by hand using local clays and by smoothing and cutting the clay into shape (Toplicic-Curcic et al., 2018a). From the 12th to 16th centuries, these tiles were

made and then lost sight of until the Industrial Revolution of the 19th century, when production of wall and floor tiles reached its peak. Until the middle of the 18th century, ceramic tiles were made and painted by hand. However, with the advent of the Industrial Revolution in the 19th century, transfer printing on tiles became a common method for producing wall and floor tile (Sánchez et al., 2010). Ceramic tile became more commonly available and utilised in household items like kitchenware and bathroom fixtures as we moved into this modern era.



Figure 2.1 The example of colourful wall tiles (Bernardin et al., 2006)



Figure 2.2 The porcelain tiles used at the mosque (Toplicic-Curcic et al., 2018)

2.1.2 Raw Materials

In general, ceramic raw materials are categorised according to their purposes in ceramic production as well as their fundamental features. Usually, the ceramic raw materials classify into two fundamental categories which is plastic and non-plastic.

2.1.2(a) Clay

For the most part, clay is the most significant constituent of a ceramic tile's body. Chemically pure alumina silicate known as clay is the primary ingredient in natural clay. A wide range of mineral concentrations and purity levels may be found in clay. Clay can serve as a binder, a suspension aid, and a cost-effective source of alumina and silica in a variety of applications (Baccour et al., 2009).

According to a geologist, kaolin is generated from feldspar as a result of weathering or acidic water. Water, wind, and glaciers are all examples of weathering, which is the process of reducing the land surface to sea level. A mechanical or physical method can be used to accomplish this. Water in the form of rain or waves is a mechanical process that, over time, erodes the surface of rocks. Wind at a high velocity can also cause harm. Clay formation is a chemical process aided by the fragmentation and separation of coarse grains into fine particles (Baccour et al., 2009).

Granite, feldspar, mica, and quartz, among other materials, were altered to produce clays. At their source, they are referred to as primary or residual clays. Because neither wind nor water moved the clays, they stayed at the position of the parent rock. Unaltered rocks, such as china clays, are typically discovered in irregular pockets. Since the clay was not transported by water, these deposits are coarse-grained and inflexible. Therefore, there was no sifting of different particle sizes in the process. Because most fundamental clays are made of pure feldspar, they are usually free of non-clay minerals. Most kaolins are made from primary clay. The white colour of the

burnt china clay is due to its purity. Because of this, it may be used to produce porcelain (Baccour et al., 2009).

Aluminous rocks, particularly those containing feldspar, are the primary sources of clay-forming minerals. Orthoclase, KAlSi_3O_8 , and albite, $\text{NaAlSi}_3\text{O}_8$, are the primary kaolin-forming minerals in the ore (Baccour et al., 2009).

2.1.2(b) Silica

On the Earth's surface, silica is the most common oxide. Quartz is the most common type of free silica, while other forms of silica, such as the silicate minerals, account for most of the silica found in nature (Cam & Senapati, 1998). There are 3 types of silica found in nature that are rock, granular and powdered.

1. Rock type - Quartz is a common name for this sort of stone. This form of silica is not often utilised in the ceramic industry because of the impurities it contains.
2. Granular Type - It's called silica sand, and it's the most prevalent type of silica. As a result of its purity, this form of silica is frequently utilised in pottery.
3. Powder Type – Known as diatomaceous earth, this form of silica contains a significant number of contaminants. Thermal insulation is the primary application, not ceramics.

Silica may take a few shapes and forms. Quartz, tridymite, and cristobalite are the three primary crystalline forms. Quartz is a low-cost raw material compared to other ceramic body components. Silica sand or flint is a source of silica in the human body. The use of silica sand reduces the unfired strength and flexibility, but it facilitates gas escape after drying and firing. Reduces drying shrinkage and enhances the whiteness of the fired body (Cam & Senapati, 1998).

2.1.2(c) Feldspar

Ceramic bodies and glazes commonly use feldspar as a fluxing ingredient, and it is also one of the three primary raw elements required to create the triaxial body. By producing the glassy phase, Feldspar is utilised to minimise the firing temperature of ceramic bodies. In the feldspar mining procedure, bulldozers or backhoes are used to remove the overburden, which is then drilled and blasted. Crushing is done with a jaw or a cone crusher, and then varied apertures such as 5mm or according to market demand are used to screen out the material. In addition, magnetic separators are used to remove other pollutants (Ochen et al., 2021).

Na-Feldspar, K-Feldspar, and Ca-Feldspar are the three kinds of feldspar minerals found in nature (Correia et al., 2008). Feldspar is not found in nature in its pure form, but rather in a variety of intermediate compositions. As a result, sodium feldspar also contains orthoclase and anorthite. There are various minerals such as quartz, iron complex, and magnesia. Feldspars serve a crucial part in achieving the vitreous appearance of the ceramic tile body, especially porcelain tile. This is especially for the porcelain tiles. Feldspars, like other non-plastics, operate as a flux while simultaneously facilitating drying and gas release after fired (Cam & Senapati, 1998).

2.1.3 Production Process of Porcelain Tiles

The first step in preparing the ceramic body is to measure out and batching the individual raw materials into the precise amounts. After the raw materials have been coarsely crushed, the raw materials will be mixed according to the formulation to make the final product (Cam & Senapati, 1998).

To reduce the particle size of the solid raw materials, several procedures are performed in the grinding process. Smaller particles can be made from coarse particle via grinding process. In addition, it improves the reactivity of minerals and allowing for more efficient firing and a more uniform particle size distribution, resulting in higher packing densities. Wet grinding and dry grinding are the two most common ways of grinding. Materials must be finer in both circumstances to remove contaminants that might result in spots or defects (Alves et al., 2012a). When using the wet grinding procedure, each of the raw components that make up the body composition is kept in a distinct bay of the mill. The raw components are weighed in accordance with the formulation and mixed with water and a deflocculant to facilitate dispersion in ball mills. The ball mill slip particle size distribution is designed to produce a variety of coarse, medium, and small particles, with the goal of achieving high compaction in the body powder (Alves et al., 2012a). Wet batch milling involves loading the mill, running it for a period of time, and then refilling it with a fresh charge. For 24 hours, the slip is placed in the storage tank when it has reached the proper particle size. The moisture percentage of raw materials might be as low as 8% or as high as 10%, depending on the kind of mill used (Alves et al., 2012a).

Next, the spray drying is the procedure by which the body slip from the mill is transformed into granules of the right size and moisture content for pressing. High

pressure atomizes the body slide, and hot air evaporatively removes water from the little droplets (Alves et al., 2012a).

After that, the spray dried powder is pressed into a green body. When a powder or granular substance is compressed and shaped at the same time, it is referred to as "pressing." Presses can be divided into two categories which is hydraulic and friction. Ceramic tile manufacturers rely on hydraulic presses the most. They have the benefit of being able to maintain a high-pressure level that can be readily managed. Single-fired products, such as vitrified stoneware tiles and large-size tiles, require a steady pressure for dimensional correctness, and these machines are ideal for this purpose. Varying tiles require different amounts of pressure to be applied. It is required to apply pressures of 350-400MPa for vitrified tiles of the same size while working with white-body floor tiles, as opposed to pressures lower than 300MPa (Martín-Márquez et al., 2008b).

Between the pressing step and the firing process, the ceramic tile will dry first. In order to strengthen the strength of the unfired tile and to limit the possibility of tile loss due to deformation as the steam quickly evolves in the kiln, this procedure has been done. Once the green body has been heated, chemical and physical processes take place that turn the previously weak and unfired piece of tile into an extremely resilient and long-lasting product. Following a fired, the ceramic tile body takes on the mechanical and aesthetic characteristics of the completed item. Single layer firing offers homogeneous temperature distribution and high-quality tiles as seen below (Martín-Márquez et al., 2008b).

In roller kilns, mass vitrification and firing temperature interval dimensional stability are the primary objectives. Sorting is the final step in the production of porcelain tiles. Planarity and piece quality are the most important considerations in the

selection line (Martín-Márquez et al., 2008b). In Figure 2.2 shows a summary of the entire procedure in production of ceramic tiles.

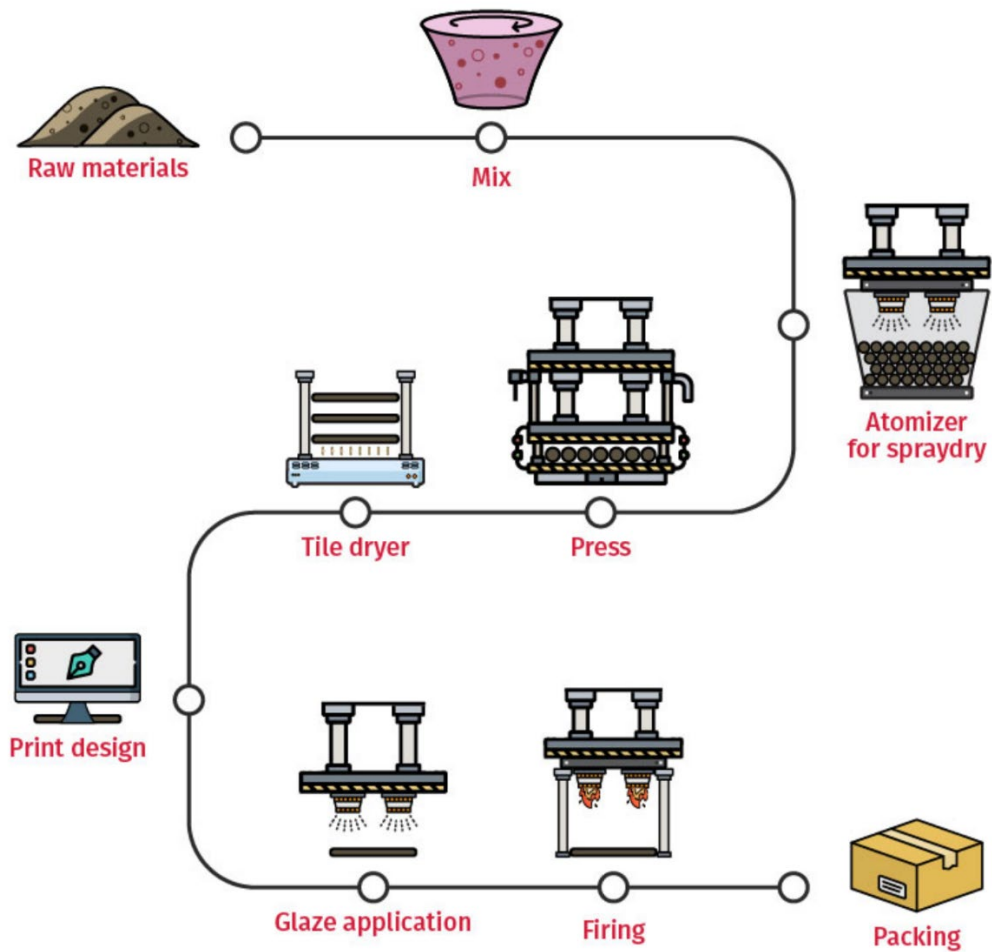


Figure 2.3 The summary of production of ceramic tiles (Toplicic-Curcic et al., 2018a)

2.1.4 Different Types of Tiles

2.1.4(a) Porcelain Tiles

After being fired at a high temperature, kaolin clays containing silica and feldspar are combined with water to make porcelain tiles. Porcelain tile has a denser and harder body than most other tiles because of the fine-grain clays and other minerals that make it resistant to moisture, stains, and damage for many years. They used to cover floors and walls with a water absorption rate of less than 0.5% (Cam & Senapati,

1998). Pressing clay into a desired shape and firing it at a high temperature result in porcelain tiles. To make porcelain tile, basic materials such as clay, bentonite (from bentonite clay), and kaolin (from bentonite clay) combine with mullite, a mineral created by the chemical reaction of quartz and feldspar crystallisation. Vitreous phase is often 45–80%, with the quartz phase typically greater than 30%, whereas mullite is confined to just 8%. Unglazed porcelain tile and glazed porcelain tile are the most common types of porcelain tile in use today. Additional impermeable layers on glazed porcelain tiles provide colour and patterning on their surface. The glaze protects the ceramic body from liquids and moisture absorption by acting as a protective barrier. Fluxing agents, such as feldspar, are employed to create a glassy phase and make quartz and clay easier to melt. There is less porosity and water absorption in porcelain tile, which increases the mechanical strength and density of the ceramic tile. This is due to the higher feldspar content of porcelain tile, which also increases the glassy phase present (Bernardin et al., 2006).

2.1.4(b) Wall Tiles

By the naked eyes, the wall tiles and floor tiles are looking similar. Due to their modest weight and lack of requirement to withstand intense traffic or abrasion from large weights, wall tiles are less durable than floor tiles. A semi-gloss or matte finish is the most common appearance of glazed wall tile. When wet, the glazed surface loses most of its traction and becomes dangerously slick. In comparison to porcelain and floor tiles, wall tile has a water absorption value of 10% to 20%. For the kitchen and bathroom, where they are regularly exposed to stains and chemicals, wall tiles must be stained and chemical resistant (Bernardin et al., 2006).

2.1.4(c) Floor Tiles

A process of pressing or extruding raw materials into a thick body with a distinctively textured look is used to create floor tiles (Bernardin et al., 2006). A beautiful, wear-resistant, and visually appealing surface is created by applying them to the floor. However, floor tiles, whether it is coated or not, it must be strong enough to withstand the weight of furniture and foot activity without cracking or breaking. In order to handle significant weight and foot activity, floor tiles are thicker, denser, heavier and more durable than wall tiles. Since it can take greater loads, it is typically used on floors, although it may also be used on walls (Sánchez et al., 2010). Table 2.1 and 2.2 shows the difference between porcelain tiles, wall tiles, and floor tiles in terms of water absorption value and the value of minimum modulus of rupture (MOR).

Table 2.1 Percentage of Water Absorption according to of types of tiles (Standards Australia, 2003)

Types of Tiles	Percentage of Water Absorption (%)
Porcelain Tiles	≤ 0.5
Floor Tiles	1.0-3.0
Wall Tiles	10-20

Table 2.2 Minimum value of Modulus of Rupture (MOR) according to types of tiles
(Bechthold et al., 2015)

Types of Tiles	Modulus of Rupture (MPa)
Porcelain Tiles	28
Floor Tiles	23
Wall Tiles	17.5

2.2 Introduction to Steel Making

Iron (Fe) is the primary component of steel, with a little amount of carbon (C) (Das et al., 2007). Other alloying elements may also be present in variable quantities. The proportions of alloying elements have a significant impact on the characteristics of steel. The heat treatment of steel influences its properties as well. Iron ore and steel scrap are used in the steelmaking process. Blast furnaces are the equipment used to make iron. The crucial aspect to decrease losses and supply effective raw material for iron manufacture is the sintering process. From properly selected grades of coal, coke is created. The coal is heated in the ovens until it forms coke. The coke is then taken out of the oven, cooled, and graded before being put into the blast furnace. Coke, ore, and sinter are all fed into the blast furnace from the top, where they are combined with limestone. A hot air blast is pumped through the nozzles, called tuyeres, in the base of the furnace. Coal or oil may be pumped into the blast air to give extra heat and decrease the need for coke (Yu & Wang, 2011).

There is a pool of molten metal at the bottom of the furnace from the iron in the ore and sinter. The melted iron ore and sinter are combined with limestone to produce a slag, which floats on top of the molten metal because it is lighter than the metal. The iron manufacturing process is continuous. Molten iron from the hearth of the blast furnace is tapped out into ladles when the amount of molten iron in the hearth is adequate for steel manufacture (Koros, 2003).

A second notch or top-hole is used to tap off the slag that accumulates on the surface of the molten metal. Meanwhile, warm air is blown into the furnace from the bottom and raw material is added to the top. Until the heat-resistant brick lining begins to degrade, the process continues indefinitely (Koros, 2003).

2.2.1 Basic Oxygen Furnace (BOF)

Steel scrap and molten metal from a blast furnace are the main materials used in BOF. High-purity oxygen is forced onto the metal at extremely high pressure using an oxygen lance that is water-cooled and lowered inside the converter. Carbon and other undesirable atoms are removed from the molten charge when oxygen is introduced. The temperature of the metal may be regulated by the amount of scrap that is introduced to the oxidation process. Carbon, in the form of carbon monoxide, is expelled from the converter. Lime is used as a flux during the 'blow' to assist remove the oxides as a floating layer of slag from the furnace. For the right steel temperature and composition, the amounts of scrap, hot metal, lime, and other fluxes are calculated. Gases, including argon, nitrogen, and carbon dioxide, are injected through the furnace's base to aid in the refinement process. Ladles are tapped into the steel after it has been refined. After refining, carbon content in the steel is typically less than 0.04%. Alloy additions can be introduced during tapping to alter the steel's final composition. It is then flipped over and the remaining slag, which has been poured into a waiting ladle, is removed to cool in a slag cooling pit. The slag undergoes further processing (Bayer Ozturk & Eren Gultekin, 2015).

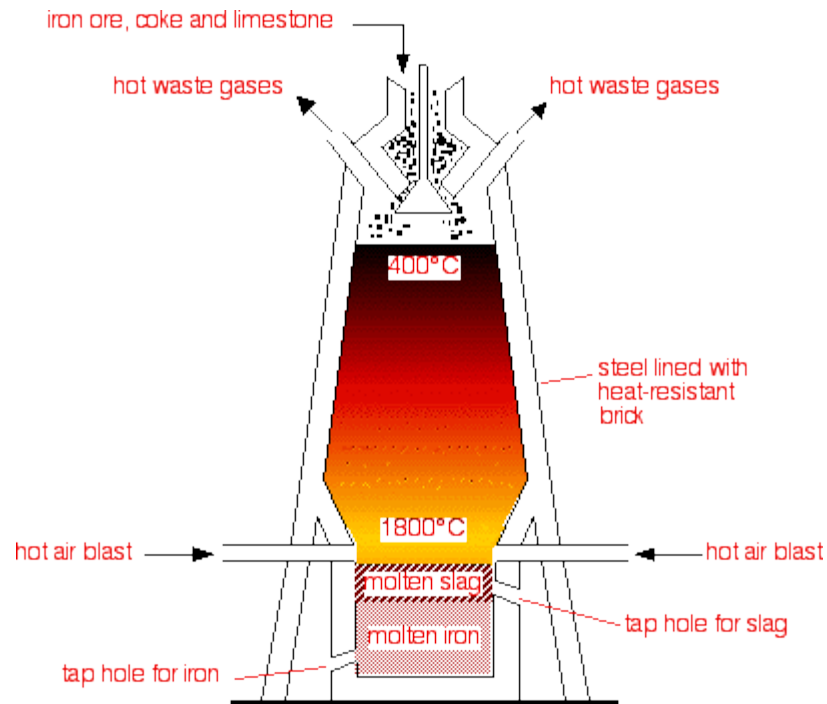


Figure 2.4 Blast Furnace (Bayer Ozturk & Eren Gultekin, 2015)

2.2.2 Electric Arc Furnace (EAF)

Cold scrap metal is the only source used for the Electric Arc Furnace (EAF). The initial application of the method was to produce high-quality steel. However, it is also employed in the production of other extensively used steels, such as alloy and stainless steels, as well as some specific carbon and low-alloy steel grades (Aguirre et al., 2009). Three graphite electrodes can be raised or lowered through a moveable ceiling in an EAF circular bath. At the beginning of the operation, the electrodes are removed, and the ceiling is flung open. A big steel basket is dropped from an overhead crane to charge the furnace with steel scrap. When the battery is fully charged. The electrodes are dropped into the furnace when the roof has been swung back into place. Fluxes like lime and fluorspar are blown in to melt the melt, which is then mixed with oxygen. Slag is formed because of the metal's impurities combining. A sample is obtained, and the steel's composition is tested to determine its quality. The furnace is quickly tapped into a ladle when the necessary composition and temperature are

obtained. In order to meet exacting client specifications, alloys might be added during tapping (Yu & Wang, 2011).

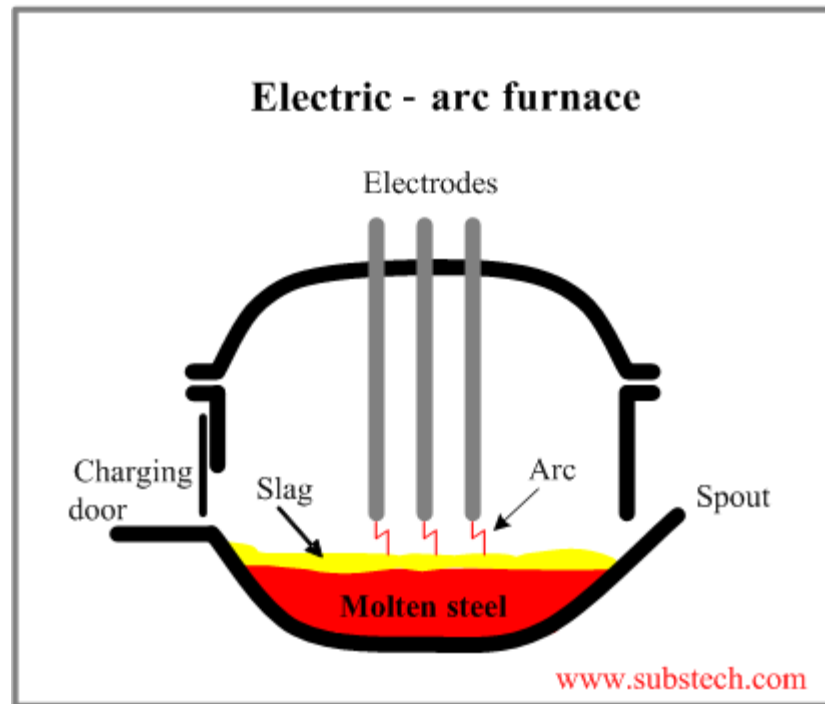


Figure 2.5 Electric Arc Furnace (Penteado et al., 2019)

2.2.3 Secondary Steelmaking or Ladle Furnace (LF)

Depending on the necessary steel quality, the molten metal from BOF or EAF is given one or more further treatments once it is tapped into a ladle. Vacuum degassing, powder injection, argon stirring in the ladle, and ladle arc heating are all steps in the refinement process. Using these methods, the temperature and composition are more evenly distributed, allowing for more exact trimming, removing potentially dangerous gases like hydrogen, and drastically lowering the concentrations of potentially toxic materials like sulphide (Manso et al., 2005).