



ADDIS ABABA UNIVERSITY
ADDIS ABABA INSTITUTE OF TECHNOLOGY (AAiT)
GRADUATE SCHOOL OF CHEMICAL AND BIO ENGINEERING

**Investigation of the Potential of Sugar Cane Bagasse Ash for
Production of Ceramic Material (Wall Tiles)**

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

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Addis Ababa, Ethiopia

09 July 2019

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DECLARATION

I hereby declare that the thesis entitled “Investigation of the Potential of Sugar Cane Bagasse Ash for Production of Ceramic Material (Wall Tiles)” has been written by me in the Addis Ababa institute of Technology, School of Chemical and Bio-Engineering under the supervision of Dr. Abubeker Yimam. The information derived from the literature has been cited in the text, and their list of references provided. No part of this thesis has been previously investigated at any university in this country.

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ABSTRACT

Utilization of sugarcane bagasse ash (SCBA) waste as a value added material in industrial process is interested from both environmental and economic perspectives. Hence, this thesis work focused on partial replacement of quartz/silica sand by SCBA which is composed of 72.02% SiO₂, 7.96% fluxing compounds (K₂O, Na₂O and MgO) and other minor components has been investigated to produce ceramic wall tiles, where ceramic raw materials (feldspar, kaolin and clay) have been remained constant. The compositional and mineralogical analysis of materials (SCBA and silica sand) have been performed by using atomic absorption (AA) method and x-ray diffraction (XRD) respectively. This result revealed the suitability of replacement of quartz by SCBA. The experiments have been conducted by replacing quartz with SCBA (20, 35 and 50 wt.%). Correspondingly the effects of pressing pressure at 15, 17, 19MPa and firing temperature at 1000°C, 1100°C, 1200°C have been investigated. The physico-mechanical properties like: green, dry and fired strength, linear shrinkage, water absorption, LOI and crazing test (for glazed) of the products have been analysed. The experimental results showed that at 1100°C firing temperature, replacement of quartz by 35% SCBA and at 17MPa pressing pressure found the best for ceramic wall tiles production. Thus, from the experimental results, it was concluded that SCBA can be utilized for production of ceramic wall tiles as partial replacement for silica sand/quartz. Hence, minimization of the SCBA environmental impacts and its economic advantages have been realized.

Keywords: SCBA, Silica sand/quartz, firing temperature, Ceramic wall Tiles.

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GLOSSARY

Blunger	A mechanical unit operation where well mixed constituents from ball mills drained and homogenised
Crazing	A hairline crack at the glazed tile surface
Cristobalite	An odd form of silica. It is composed of the same elements as Quartz but has a different crystal structure, making it a separate mineral
Beneficiated	The process of refining raw materials in order to optimize their performance or make them useful in a manufacturing process
Ceramic body	Ceramic made of one or more material designed for a special purpose.
Earthenware	A low fired form of pottery or objects made from clay, which is porous and permeable.
Extrusion	A process in which clay material is forced through a mould for the desired shape
Hydrophilic	Having a tendency to mix with, dissolve in, or be wetted by water.
Modulus of Rupture	It is a measure of the cohesive characteristics of the material forming the tile and relating to the degree of compactness and consistency.
Monoporosa	Single-fired tiles with higher porosity and water absorption levels
Mullite	A colourless mineral consisting of aluminium silicate in orthorhombic crystalline form and its chemical formula $Al_6Si_2O_{13}$.
Mono silicic acid	A form of silicon used by plants, which is found in both liquid and adsorbed phases of silicon in soils.
Plasticity	The properties of a material that allow it to be shaped and to retain its shape and moisture.
Porosity	This is the volume of pores in a fired tile relative to volume of tile body that is capable of absorbing moisture.

Shrinkage	The decrease in the size of objects due to drying and firing
Single-fired tiles	Glazed tiles produced by the single-firing method in which the raw tile body and the glaze undergo a single pass through the kiln at high temperature.
Sintering	Process of compacting and forming a solid mass of material by the action of heat which compels interstitial silica in a ceramic body to melt and help in fusing the entire body.
Vitrification	A process of fusion of ceramic body, as a result of a firing process
Water Absorption	The quantity of water an object can absorb, expressed as a percentage of the dry/wet based object weight. High water absorption corresponds to a porous structure, while compact, vitrified structures feature low water absorption

LIST OF ABBREVIATIONS

AA	Atomic absorption
AAiT	Addis Ababa institute of Technology
ANOVA	Analysis of variance
ASTU	Adam Science and Technology University
BBD	Box Behnken Design
CT	Crazing Test
EGSL	Ethiopian Geological Survey Laboratory
FWHM	Full width at half maximum
ICDD	International Centre for Diffraction of Data
MOR	Modulus of rupture
PDF	Powder Diffraction File
SCBA	Sugar Cane Bagasse Ash
SRD	Surface Response Design
TCPSC	Tabor Ceramic Products Share Company
STPP	Sodium Tri-Poly Phosphate
TST	Tensile strength Test
WA	Water absorption
W_d	Dry weight
W_{sat}	Saturated weight
XRD	X-ray diffraction

1. INTRODUCTION

1.1 Background

There have been remarkable increases in agricultural, industrial and economic growths in the recent decades that have contributed to integrated improved quality and well-being for citizens. Hence, sugar factories are one of agro- industries that use sugarcane as the main raw material and generate huge amounts of wastes worldwide. Brazil is the world's largest producer of sugarcane, followed by India, China, and Thailand. Likewise, Ethiopia is on the way to have about 10 sugar factories within a few years in which all generates huge amounts of sugarcane bagasse. The sugarcane absorbs more silicon than any other cultivated plants. Sugarcane can remove from 500 to 700kg silicon per hectare during their development (Narayan K. Savant *et al.*, 1999). Mono silicic acid (H_4SiO_4) is the form of silicon used by plants, which is found in both liquid and adsorbed phases of silicon in soils (Rodrigo H. *et al.*, 2017). The sugarcane bagasse contains fibre wastes like: cellulose, hemicellulose and lignin compositions (K.S. Shanmukharadhya, 2009). This waste material is mainly reused as fuel in boilers for energy generation (B.R. Stanmore, 2010). As a result, sugarcane factories produce huge amounts of solid waste which is characterized as a non-biodegradable material known as sugarcane bagasse ash (SCBA). The Brazilian sugarcane factories alone generates about four million tons per year of SCBA. Sugarcane bagasse ash waste is rich in silica being likely to be used as ceramic raw material (K.C.P. Faria *et al.*, 2012).

Several studies have investigated bagasse ash potential applications such as producing silica gel as, raw material for ceramics (bricks, tiles, etc.) and for concrete additives and so on depending on its characteristics. Some works on the incorporation of SCBA in ceramic bodies have shown a variation in the chemical composition of these ashes, in the crystallinity of silica and in the properties of the sintered ceramic material. According to Vieira *et al.*, (2004), the high pure crystalline silica content worsens some mechanical properties of ceramic materials. However, up to the firing temperature of 970°C the addition of ash was found to increase linear shrinkage and decrease mechanical strength of ceramic material. On the other range, the presence of alkalis (fluxes) favours the formation of the liquid phase at a temperature greater than 1000°C, where the small pores in the microstructure filled, increasing the density of the material. During the burning of kaolinite clays, two stable phases are formed: mullite and cristobalite. Their formation is very important because this phase improves the physical properties (hardness and strength) of the ceramic material. Nucleation and growth of mullite crystals can be accelerated by the presence of

additives, flux or mineralizer. For clays, the mineral fluxes are high in alkalis, which when present in a ceramic composition, act as fluxing and reduce the sintering temperature and porosity of the products. The effects of the incorporation of high concentrations (20%) of SCBA on mullite formation and on the final physical properties of ceramic products using a kaolinite clayed material from Brazil were shown by (A.E. Souza *et al.*, 2011) (A.E. Souza et al). On the other hand, ashes resulting from this process are also characterized as highly pulverized material of low density and high volume. These features mean that if transported to landfills and dispersed in the environment, SCBA can contaminate air, soil and groundwater, resulting in effects to human health and aquatic animals. The use of this material as an additive to produce clay bricks and roof tiles has also been reported. It should be noted that the chemical and mineralogical properties of SCBA depend on the industrial process from which it has been obtained.

For SCBA samples, quartz is observed as the main crystalline compound as shown by the XRD (figure 1.1) and confirmed by chemical analysis (see Table 1.1). The presence of quartz can be attributed to the sand adhering to the sugarcane at the time of harvest, due to the characteristics of soil origin. This is true even after the sugarcane has been washed (J.Torres *et al.*, 2016). SCBA can potentially replace cement for up to 20 %. However, its low bulk density would result in an additional issue for further use as cement replacement material, since higher replacement volume will bring more hydrophilic particles of sugarcane bagasse ash into the mixture (Andri Kusbiantoro et al., 2018). In similar manner Mst. Shanjida Sultana and Aminur Rahman, (2015) revealed that SCBA has proved to be a viable by-product for admixture in cement, with its intrinsic characteristic such as high content of silica in the form of quartz. However, the high carbon content is an obstacle for its use in cement/mortars. Researches all over the world today are focusing on ways of utilizing either industrial or agricultural wastes as a source of raw materials for the industry (V. S. Aigbodion *et al.*, 2010). The correct management of solid wastes from industrial and urban activities is considered a key issue on the current environmental scenario.

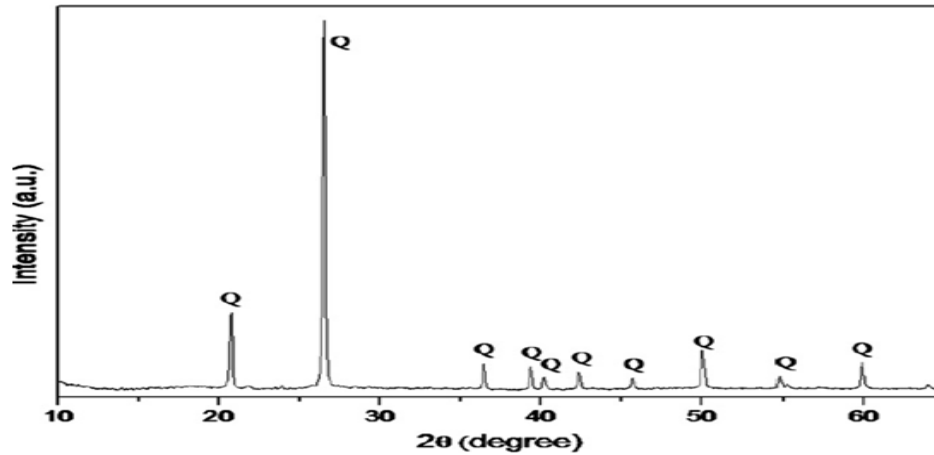


Figure 1.1: XRD of the sugar cane bagasse ash: Q- quartz (J. Torres Agredo et al., 2016)

The solid wastes are potentially pollutant, resulting in negative environmental impacts for flora and fauna. Traditional methods used in the solid waste management are considered expensive and limited. From this perspective, reuse has become a crucial issue for the solid waste management worldwide. From an economic and environmental point of view, the clay-based ceramics are highlighted successfully in the reuse of solid wastes as a source of renewable raw materials (Safiuddin Md *et al.*, 2010).

Table1.1: Chemical and physical characteristics of SCBA and Cement (OPC)

Characteristics, (%)	SCBA1	SCBA2	OPC
SiO ₂	72.8	61.3	19.4
A ₂ O ₃	6.4	5.6	4.0
Fe ₂ O ₃	5.5	5.6	3.6
CaO	3.8	3.9	64.5
MgO	2.3	3.2	1.5
K ₂ O	2.7	5.0	0.4
Na ₂ O	1.2	0.9	0.3
Loss on ignition	3.7	11.0	2.6
Average Particle size (μm)	79.8	41.5	16.1

Source: (J.Torres Agredo et al., 2016)

According to (FIESP and S.Pawulo, 2001) one ton of burned sugarcane bagasse is estimated to produce 25kg of SCBA waste. Correspondingly, (Rodrigo Heleno *et al.*, 2017) mentioned that each ton of sugarcane generates between 250 and 270kg of bagasse and 200kg burning of sugarcane bagasse generates, 1– 4% SCBA. Nevertheless, attention has never been given for the final solution of the sugarcane bagasse ash solid waste in our country. In other words, there is no safe solution for final dumping of this huge waste, as it causes significant changes in the physio-chemical characteristics of the soils and water. It needs a great effort in order to find everlasting solution for the final discarding and valorisation of this solid waste material in an ecological and economical way. In spite the fact that SCBA waste is produced in large amounts every year throughout the world. However, insufficient attention was given to manage this waste to incorporate it in the production of ceramic products.

Due to its chemical and mineralogical compositions, the sugarcane bagasse ash waste has a great potential to replace the ceramic raw materials (quartz) for the production of ceramic wall tiles. The SCBA waste is mainly composed of silicon oxide (SiO_2) and alumina with other minor components such as, iron, calcium, sodium and potassium oxides (K. C. P. Faria, 2001) (Lamego, 1985 and 2000). (G. Viruthagiri *et al.*, 2015), investigated the evaluations of different phases with various temperatures and reported the SCBA samples were composed mainly of quartz, anorthite, kaolinite, cellulose, albite and calcite as major elements as the predominant crystalline material. Similarly, (A.M. Pereira *et al.*, 2018) stated that the main crystalline compound of the SCBA is quartz/ SiO_2 (PDF #331161).

SCBA waste is very similar in terms of chemical composition to common clays and quartz that are used in the production of ceramic materials. This means that the reuse of SCBA waste as a possible raw material for ceramic material is an important technological solution (ASTM Standards, 1985a). The ceramic industries use huge amounts of natural raw materials for the fabrication of clay and quartz based products (H. Baccour, 2008). This fact, however, has taken the ceramic industry to face scarcity of good quality raw materials in locations close to the plants. SCBA has high potential in construction sectors and ceramic factories but only a few researchers studied its applications in ceramics industries.

Incorporation of SCBA in red ceramic production reveals that if the pieces fired up to 1000°C the lowest apparent density (1.51–1.70 g/cm³), which is due to the effects of sintering temperature. At 1100°C, however, a substantial increase in apparent density (1.77–1.94 g/cm³) was observed, which is in line with the verification that helps to reduce the open porosity of the red ceramic pieces. Hence the SCBA waste influenced the technological properties, microstructure, and phase evolution of the fired red ceramic pieces (K. C. P. Faria and J. N. F. Holanda, 2013).

1.2 Statement of the problem

Sugarcane bagasse is a valuable by product in sugar milling that often used as a primary fuel source to supply all the energy needed to generate power for the plants. Burning bagasse as an energy source generates ash, which is considered as a waste causing disposal to land surface and water pollution. These practices have caused undesirable environmental impacts mainly associated with soil and water. Several studies have investigated that bagasse ash potential applications such as: producing silica gel as adsorbent, raw material for ceramic products like: wall tiles, sanitary ware, table wares, porcelain and etc. Similarly, it has wide applications in cements and concrete additives, catalyst, cosmetics, paint and coating, based on its characteristics. The silica contents of bagasse and its ash differs depending on environment, harvesting period and process type and soil, (A. M. Usman *et al.*, 2014). In Ethiopia, Wonji Shoa Sugar factory (WSSF) alone generates approximately 1,625kg SCBA /day.

Moreover, sugarcane bagasse waste ash has a potential to be used as a substitute for ceramic bodies (in this case quartz/silica sand) and improve the resource usage in addition to sintering at lower temperature as Segades, (1996) mentioned. The SCBA mineralogical characterization shows that the main mineral it contained is quartz (Castaldelli, 2014). The objective of this study is to replace the quartz partially by SCBA to produce wall tile of better quality and low cost. Along with this, the environmental impact of disposing the bagasse ash waste would be reduced and energy consumption and conservation of natural resources during operations would be realized.

1.3 Objective

1.3.1 General objective

The aim of this research was to investigate the potential of sugarcane bagasse ash to produce high quality as well as lower cost ceramic wall tile by partial replacement of ceramic raw material (silica sand/quartz).

1.3.2 Specific objectives

The specific objectives of the research are to:

Collect the raw materials from available sites and prepare for compositional and mineralogical analysis.

Enhance SCBA utilization for ceramic wall tiles production with partial replacement of quartz

Investigating effects sintering temperature and pressing pressure during wall tiles productions

Characterize and evaluate the product using physicommechanical properties such as: linear shrinkage, water absorption, crazing tests and modulus of rupture of the green, dry and fired.

Characterize the mineralogical analysis of the product by using XRD.

Comparative evaluation of the products with the conventionally produced wall tiles.

1.4 Significance of the research

This thesis has focused on the partial replacement of sugarcane bagasse ash waste into ceramic wall tiles fabrication. The optimum blending of the sugarcane bagasse ash with ceramic bodies has caused remarkable changes in its character and therefore has potential application in constructions giving aesthetic value. The research has also contributed on enlightening the quality and properties of wall tiles. When sugarcane bagasse ash used for different purposes by blending with appropriate ceramic raw materials, (clay, feldspar, quartz/silica sand and kaolin) it has improved the demand and quality of ceramic products. Through incorporating sugar cane bagasse ash wastes, it would have also provided a way of converting these wastes into valued products which results in reduction of environmental problem by avoiding landfill disposal and water pollution.

Moreover, it has offered cost-effective ways of manufacturing ceramic material (wall tiles) compared to conventionally produced, that would have potential for our countries through import minimization. Thus, investigation of this research is important to evaluate the effectiveness of ceramic wall tiles product due to its better physical properties and replacement safes resource and land degradation as well as energy consumption of the ceramic factory. Generally, researches have been performed in Ethiopia regardless of sugarcane bagasse ash utilisation to enhance the concrete strength. However, nothing has been investigated the SCBA for replacement of ceramic body (quartz and feldspar) but had a significant potential.

2. LITERATURE REVIEW

2.1 Introduction to ceramic wall tiles manufacturing

The materials used for thousands of years and is still using can be divided into five main groups. These are: Metals, Polymers, Composites, Ceramics and natural materials (Paul Bormans, 2004). The term 'ceramics' is derived from the Greek word 'keramos' meaning 'burned earth' and is used to describe materials of the pottery industry. In other word 'ceramics' (ceramic products) is used for inorganic materials (with possibly some organic content), made permanently by a firing process. Traditional ceramics, such as bricks, or wall and floor tiles, generally feature high heterogeneity due to the wide range of the composition of natural clays used as raw materials in their manufacture. Therefore, there is a high tolerance for incorporating large amounts of suitable wastes as raw materials. This fact attracts further importance since ceramic industry, which is classified as heavy industry, consumes huge amounts of diminishing mineral resources (Opoku, Eric Vernon, 2015). Ceramics can be glazed or unglazed, porous or vitrified depending on its specific applications (Ceramic M. I, 2007.)

In the ceramic industry, two or more natural materials are generally mixed to prepare the ceramic mass with the desired properties. In some cases, fluxing and/or non-plastic materials (e.g., quartz) can be used to improve the sintering process and the final ceramic properties. In other words most efforts to increase strength, emphasis have been placed on minimization of quartz in the formula because of the β to α phase inversion of quartz which occurs at 573 °C during cooling. The inversion results into decrease of quartz particle volume and may lead to cracks in the body. So far, there are reports of improvements in the mechanical properties by reducing the use of quartz (Hassan Usman Jamo, 2015). According to (M.Romero and J.Perez, 2015) after the allotropic transformation of quartz which occurs at 573°C, the particles undergo pronounced shrinkage, which increases microscopic stresses. As the piece cools, the particles begin to de-bond from the matrix, giving rise to peripheral cracks. As the cooling is rapid, thermal tensile stresses formed at the surface peripheral cracks thus encounter more favourable growth conditions, which lead to an increase in natural defect size. This microstructural damage adversely affects the product's mechanical behaviour. The wide composition range of clays used for manufacturing ceramic tiles makes them good receptors of residues (Souza G. P, 2002). The possibility to recycle waste materials (rice husk ash, sugarcane bagasse ash, fly ash and etc.) in ceramic is nowadays an advantageous reality in environmental protection and in saving raw materials. SBA is widely

available in many developing countries. The bagasse ash is still disposed of as waste in landfills causing environmental problems (Sivakumar G, 2013).

2.2 Ceramic Wall Tiles Processing

Wall tiles is used for inside and outside decorations and it belongs to a class of ceramic products known as white wares. The manufacture of tile started during the ancient era and human being, including the Egyptians, the Babylonians, and the Assyrians. Later, ceramic tile was produced almost in every European countries and United States. At the early twentieth century, tile was produced in large scale. A very few wall tile producing factories are there in Ethiopia, which could not satisfy the demand of construction sectors both in quality and cost. The following table illustrates the Ethiopian wall tiles import scenario for consecutive ten years' data.

Table 2.1: Wall tiles import data from different countries and its projected demands ($\times 10^8$)

	Imported year									
	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018
Amounts(m ²)	27.6	18.5	29.3	36.9	43.1	30.3	91.3	106.3	209.6	100.6
Cost(ETB)	0.99	1.56	2.98	3.69	7.40	6.83	9.27	11.62	203.32	195.73

	Projected demand									
	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028
Amount(m ²)	154.2	169.6	184.9	200.3	215.6	230.9	246.3	261.6	276.9	292.3
Cost(ETB)	206.7	229.9	253.3	276.6	299.9	323.2	346.5	369.8	393.1	416.4

Source: Ethiopian Customs Authority

The raw materials used to produce wall tile consist of clay minerals excavated from the earth's crust, natural minerals such as feldspar and quartz that are used to lower the sintering temperature and maintain the dimensional stability and chemical additives needed for the shaping process. SCBA acts as a fluxing agent in ceramic production (Tonnyopas, 2013), (Teixeira, 2014) which reduces the temperatures at which the ceramic materials need to be fired. Thus incorporation of

SCBA can improve the energy efficiency of ceramic production (Teixeira, 2014). The following composition (Table 2.2) reveals that SCBA has a high potential in replacing ceramic raw materials to produce ceramics. The composition gives the ash properties similar to those of fine sand (non-plastic material composed mainly of quartz), used by the ceramic industry to decrease the plasticity of the clayed raw material (J. Torres Agredo *et al.*, 2016)

Table 2.2: Chemical compositions of bagasse ash and ceramic raw materials

Composition	Clay	Quartz	Feldspar	SCBA
SiO ₂	62.75	84.50	64.10	73.81
Al ₂ O ₃	29.73	5.82	13.96	7.94
Fe ₂ O ₃	3.17	3.49	0.28	5.35
CaO	0.31	2.13	0.50	3.40
MgO	0.37	1.03	0.18	2.42
K ₂ O	1.02	2.31	16.74	2.95
Na ₂ O	0.25	1.20	2.80	3.71
TiO ₂	1.52	0.02	0.15	0.42

Source: Research Journal of Recent Sciences: Vol.3 (ISC-2013), 67-70 (2014)

The desired colour for a wall tile body, in most parts of the world, is white or buff. Some producers manufacture a pink or red body, depending on the availability of local raw materials. The iron content of the clays and/or other minerals used in tile manufacture is generally the determining factor in the tile body colour. The higher the iron contents in the ceramic body composition then the ceramic product become reddish in colour. Kaolinite, feldspar minerals, quartz/silica sand and clays are well known raw materials for wall tiles production. Combinations of these materials are therefore used by the ceramist to control the final desired properties of the ceramic body during manufacture. The following are the main unit operations used in ceramic tiles production process.

2.2.1 Crusher

Hard raw materials, such as feldspars and quartz are reduced to desired size by successively grinding through one or more jaw crushers and then to either a ring mill or hammer crusher for final sizing.

These crushing basically needed for two basic reasons: a) course materials are relatively hard and supplied having large particle size, so if it charged to ball mill it will reduce the efficiency of the

mill and even damage the mill and the medias/balls, b) if it charged to the mill having large particle size, it will rotate inside the shell of mill together with balls instead of being grinded.

Oversize particles are screened after crushing and returned back through the process in a closed loop system. Most clay materials are not beneficiated at the plant site prior to blending or milling, but in some cases, especially where continuous ball milling is subsequently used, the clay portion of the body may be pre-blended in high speed blungers to insure dispersion or “slaking” of the clay particles.

2.2.2 Ball mill

Weighed materials are fed to a collector belt which transfers the materials to a ball mill. Water is manually/automatically fed to the mill for wet milling process. The control of all raw materials in the process is extremely important for continuous, smooth running of a plant (Kaloshkin, S.D *et al.*, 1997). Small to minute particles are needed to manufacture ceramic objects, especially for technical ceramics applications. There are two reasons for this: a) the raw material mass must be mixed with water and usually also with binding agents for the heat treatment. This mixing can only be optimal when the particles are small. b) During the last stage of the heat treatment, the sintering process (i.e. baking at high temperatures) it is better that the particles exhibit maximal surface contact for them to adhere to their neighbouring particles. Obviously this is achieved when there are many minute particles present. Hence milling is the process by which the above objects are achieved.

Milling may involve breaking up of hard materials (in which case individual particles retain their shape) or pulverization (which involves grinding the particles themselves to a smaller size). Ball milling is mostly used for brittle materials. When the particle size is less than 5mm, the particles exhibit a relatively large tendency to agglomerate. An agglomeration has a porous structure which can easily be moistened by a liquid (water). The particles will immediately be dispersed throughout that liquid. When there is a tendency to agglomerate, it is advisable to grind wet. Very often this is done in a mill with ceramic balls and takes place in the presence of dispersing substances, also called de-flocculants (Paul Bormans, 2004). Milling speed is an important process parameter of ball milling process, the higher the milling speed the higher would be the energy input into the combined powder. In addition, if the speed is too high, it begins to act like a centrifuge and the balls do not fall back, but stay on the perimeter of the mill. But at high speed, the temperature of

the vial increases; this increase in temperature can speed up the transformation process and resulted in the decomposition of any metastable phases formed during milling process. Similarly, diameter of the mill decides the speed of the mill. Generally, the rotational speed does not exceed 20 rpm. Diameter of cylinder is inversely proportional to the rotational speed, the larger the diameter, the slower the rotational speed.

Milling is generally done by mechanical means, including *attrition* (which is particle-to-particle collision that results in agglomerate break up or particle shearing), *compression* (which applies a forces that results in fracturing), and *impact* (which employs a milling medium or the particles themselves to cause fracturing). Compression mills include the jaw crusher, roller crusher and cone crusher. Impact mills include the ball mill, which has media that tumble and fracture the material. Shaft impacts cause particle-to particle attrition and compression to get the standard size required.

The important control parameters for the milling process are media charge and size distribution, raw material charge, mill rotation speed, type and amount of deflocculates, water content. The process of deflocculating (Common deflocculating agents used in the ceramics industry include: tri-sodium phosphate, sodium silicate, and sodium hexa-meta-phosphate.) involves the use of one or more agents whose purpose is to impart a charge to the surface of the clay particles, thereby allowing those particles to repel each other in the slurry, allowing less water (which must subsequently be removed by spray drying or some other process) in the slurry and reducing the viscosity of the slurry to facilitate pumping.

2.2.3 Blungers

Blunger is the mechanical unit operation that performs two basic tasks at the same time a) dispersing the charged clays which are in lump form and b) mixing/homogenizing the contents/compositions

2.2.4 Powders pressing

In this way of moulding, pressure is continuously of gradually exerted on a powder in a mould. A binding agent and a lubricant are added to the powder. The mass must meet the following requirements: a) suitable grain size distribution, b) sufficiently strong grain, c) correct content of binding agents and lubricants and d) bulk density which is as high as possible, i.e. as much as

possible mass per volume unit in order to obtain the densest product possible. Samples manufactured in this way are: floor tiles, wall tiles, plates, refractory stones, magnetic materials, spark plugs, high-voltage insulators and nose cones for rockets. Three kinds of techniques can be applied: uniaxial compression, biaxial compression and isostatic compression. Uniaxial method is commonly used in wall tiles manufacturing process. The controlled agglomerate which is achieved by sieving the moist powder is called granules. Granules are mechanically pressed to form the unfired/green tile. During the pressing operation, granules must deform in a controlled manner enabling the formation of a denser, shaped product with sufficient strength for subsequent handling including any surface finishing operations (James S. Reed, 2000).

2.2.5 Drying

Drying is removing the water or binder from the formed material. Spray drying is widely used to prepare powder for pressing (wall tile) operations. Other dryers are tunnel dryers and periodic dryers. Controlled heat is applied in this two-stage process. First, heat removes water, where it needs careful control, as rapid heating cause's cracks and surface defects. The dried part is smaller than the green part, and is brittle, necessitating careful handling, since a small impact will cause crumbling and breaking. In general, inexpert drying can lead to cracks in the green product, the causes of which may be: too high evaporation rate, large differences in moisture content in different places of the product and insufficient drying. Due to insufficient drying, the moisture evaporates "explosively" during the heat treatment which follows. Another negative side effect is that dissolved salts are deposited at the surface during the drying. This causes discoloration and will affect the glaze which may be applied to the product at the end of the ceramic route (Kaloshkin, S. *et al.*, 1997).

2.2.6 Glazing

Glaze act as smooth, relatively thin glassy coating of ceramic body which is effectively bonded to its substrate. The main reason for the use of glazes as surface coatings of ceramic materials is their relatively high chemical resistance and aesthetic aspects. Characterization of the interaction between glazes and ceramic bodies can provide information about application features, firing processes and range and sign of stress in the glaze layer. Glazed ceramic products often exhibit residual stresses due to manufacturing process. These residual stresses could be caused by

inhomogeneity in microstructure and by presence of microscopic defects. Glaze defects are often caused by improper stresses within glaze layers (tension, compression) or by changes in ceramic bodies (moisture expansion, rehydration) and glaze layers during usage or improper storage of ceramic products. It could be divided into primary and secondary defects. Primary (instant) defects are caused by poor glaze fit or technological processes. Secondary (delayed) defects are results of moisture expansion of the substrate or inappropriate storage conditions. All these defects can lead to the loss of utility properties or to the deterioration of the ceramic product (Mária Kavanova *et al.*, 2017).

Crazing can be caused by a manufacturing problem (the unequal contraction of the glaze and the tile body or subsequent expansion of the tile), or by the natural growth characteristics of porous tiles (delayed crazing). The colour of ceramic materials is known to be affected by strong sun light, household detergents fruit juice or even sugar, lemon or vinegar solutions. It is therefore necessary, unless guaranteed by a manufacturer, to test the stability of the glaze under the environmental effects to which the tiles will be exposed (Thomas E. Uher , 1984). Well sintered clay-ceramic objects mostly have a dull and rough appearance after firing. The reason for this lies in the fact that crystalline components are present in the top layer. You can improve the product by applying a glassy coating that are functionally important for earthen ware vessels, which would otherwise be unsuitable for holding liquids due to porosity (Mamza, Jidai and Alewai, 2014). In general, glazes are compounded in order to provide cover for ceramic bodies and important to a) serve to colorant, decorate, strengthen or waterproof a ceramic ware, b) resist top scratching/abrasion-resistant, c) resistant to crazing, peeling and e) reduce predetermined temperature ranges.

2.2.7 Firing

In traditional ceramics manufacture, from an energy viewpoint, the firing stage is the most important process stage, as it needs to supply sufficient heat for the necessary physico-chemical transformations in the material to develop and to provide the product with the desired technical and aesthetic properties. From an energy viewpoint, in the firing cycle, the material undergoes both exothermic and endothermic transformations. These need to take place gradually and in a controlled way, as they could otherwise lead to permanent defects in the end product. (S. Ferrer *et al.*, 2009).

Two basic types of kilns remain in service today with the older tunnel kilns being gradually replaced or outnumbered by roller hearth rapid fire kilns. Firing in tunnel kilns is done on refractory setters, either manually or automatically loaded. The process of manufacturing wall tiles may include tunnel kiln firing (14-hours cycle) or fast fire roller hearth kilns (40 to 60-minute cycle). The more modern roller kilns are replacing the older tunnel kilns throughout the world as the standard firing vehicle for all types of ceramic tile.

Firing cycles generally consist of three phases: gradual heating of the ceramic body to the soak temperature, which typically is approximately two-thirds of the melting point of the material at ambient pressure; a soak period of constant, peak temperature (soak temperature), where the bond between the material grains is formed; and a gradual cooling down of the fired ceramic. Maximum kiln temperatures typically range from about 900°C to 1330°C (Group A. O, 2000).

With few exceptions, the equipment and processes used in dry pressing and/or extrusion of ceramic tile are similar to those used in all parts of the ceramic industry and will not be repeated for other areas. Wall tile includes that family of glazed tile which exhibit relatively low mechanical strength and high water absorption. The figure 2.2 illustrates a typical process flow diagram for the production of wall tile process commonly used in today's wall tile industry (Baruzzo, 2006). The shape of the firing curve, temperature as a function of time shown in figure 2.1 essentially depends on the body composition, glazed/unglazed and nature of the glaze (glossy, opaque, matt, etc.). In general, every traditional ceramic firing cycle includes the following steps:

Heating step: In this step, the unfired products are heated from ambient temperature to about 800°C (depending on the raw materials composition). This is the step in which outgassing of the ceramic body needs to take place to avoid problems of bloating, bubbles, pin-holing, glaze porosity, and colour differences at higher temperatures. Various transformations take place in the material during heating, such as the removal of free water, combustion of organic matter, allotropic transformation of α -quartz to β -quartz, loss of hydroxyl (-OH) groups in the clays, and carbonate decomposition when the composition contains carbonates. All these chemical reactions lead to a series of internal stresses in the ceramic product owing to gas release, changes in volume, etc. If these stresses exceed the mechanical strength of the as yet "unfired" ceramic product, failure will occur.

Firing step: The most important changes relating to the development of ceramic properties involve the breakdown of the lattice structure of the original clay minerals, followed by the formation of new crystalline compounds and glassy phases. The temperature at which vitrification (glass formation) takes place, varies according to the mineralogy of material, it usually commences at about 900°C and is completed by about 1050°C (for many brick clays) or about 1100°C in the case of more refractory fireclays. During the verification stage of ceramic firing, many non-clay minerals such as quartz, oxides or iron, lime compounds and alkalis (oxides of sodium and potassium) become incorporated in the fired body. Some sintering and solid solution occurs, and eutectic reactions take place at the interface of mineral grains and melt phases (Reference Document , 2007).

The actual firing step runs from about 800°C to the programmed peak temperature, typically between 850°C and 1350°C, which depends on the product being made. The main physic-chemical transformations take place in this step. These reduce the ceramic material's porosity. They lead to the most important dimensional changes (shrinkage) in low porosity products (stoneware and porcelain ware) and to the formation of stable crystalline phases (calcium silicates) in porous products (earthenware wall tiles and structural ceramics). (Opoku, Eric Vernon, 2015) revealed that raw materials for ceramic formulations containing CaCO₃ and alumina silicates (Al₂O₃ SiO₂) usually form crystalline phases at relatively low temperatures.

There is evidence that under optimized conditions of firing and for a particle size of 10-30µm (Norton, F.H., 1970) (Nakagawa, Z. and Ece, O. I, 2002), quartz has a beneficial effect on the strength of tiles, in conformity with the pre-stressing theory. However, the $\alpha \rightarrow \beta$ phase transformation of quartz crystals takes place at 573°C during the heating-cooling process and to the relaxation of micro-stresses originated between quartz grains and the surrounding glassy phase by the differences in their thermal expansion coefficients (Singer and Singer, 1971). Similarly, $\beta \rightarrow \alpha$ cristobalite inversion occurs at a temperature of 225°C - 250°C similar to the quartz inversion, but produces larger volumetric change of approximately 5% (Lundin, S. T., 1964). For small particle sizes, the dissolution is more rapid leaving less quartz crystal in the glass and hence yielding a low pre-stress. For large particle sizes an interconnected matrix with favourable crack path is formed leading to low strength. Hence, quartz grain size affects bending strength in two ways, that is, directly through the induction of compressive stresses to the vitreous phase and

indirectly through the development of a favourable microstructure (Carty, W. M. and Senapati, U,1998).

Cooling step: This begins when the heat input ends. In the cooling step, product temperature decreases from peak temperature to near ambient temperature. In traditional ceramics the cooling rate is essentially constrained by quartz phase transformation at 573°C, as this produces a volume change that can cause cracking in the product if cooling does not take place uniformly throughout the material, owing to the low thermal conductivity of ceramic materials (S. Ferrer *et al.*, 2009).

The quantities of carbonates, organic matter, and other raw materials in the ceramic compositions influence kiln thermal energy consumption, as the energy required to develop the physic-chemical reactions in firing depends on the ceramic composition involved (Mezquita, A.; Monfort, E. and Zaera V, 2009). A typical traditional ceramic firing cycle is shown in figure 2.1. The figure summarises the most important physic-chemical changes that develop during traditional ceramics thermal treatment and the temperature ranges in which these changes occur (Pennisi L, 1991). A ceramic firing schedule depends on several factors, the most important are: body composition, thickness and geometry of the ceramic product, kiln loading density, and type of kiln. For instance, the schedule time in roller hearth kilns (low product thickness and low kiln loading density) is typically between 30 and 60 minutes, whereas in tunnel kilns (high loading density and thick products), this ranges from 18 to 24hours.

2.3 Over firing

All clays have a maturing point; once the temperature exceeds this point, the clay actually starts to soften or become liquid-like. Although the over-firing may not be noticeable to the naked eye, some indications are that the bisque is hard to glaze, under-glazes appear darker or dimmer, or glaze colours seem washed out. The ware will also seem denser or smaller than normal, due to shrinkage.

2.4 Under firing

The complete opposite of over-firing is under-firing. Under-firing may not be evident until the ware has been glazed and used. Green ware or bisque can sound hollow or brittle. A glaze that performs poorly maybe caused from under fired green ware or even under fired glaze. Problems

that can occur are: blistering, crazing, pinholes, colorations and black specks in the ware or glaze surface. Fractured pieces can be directly associated with under fired bisque.

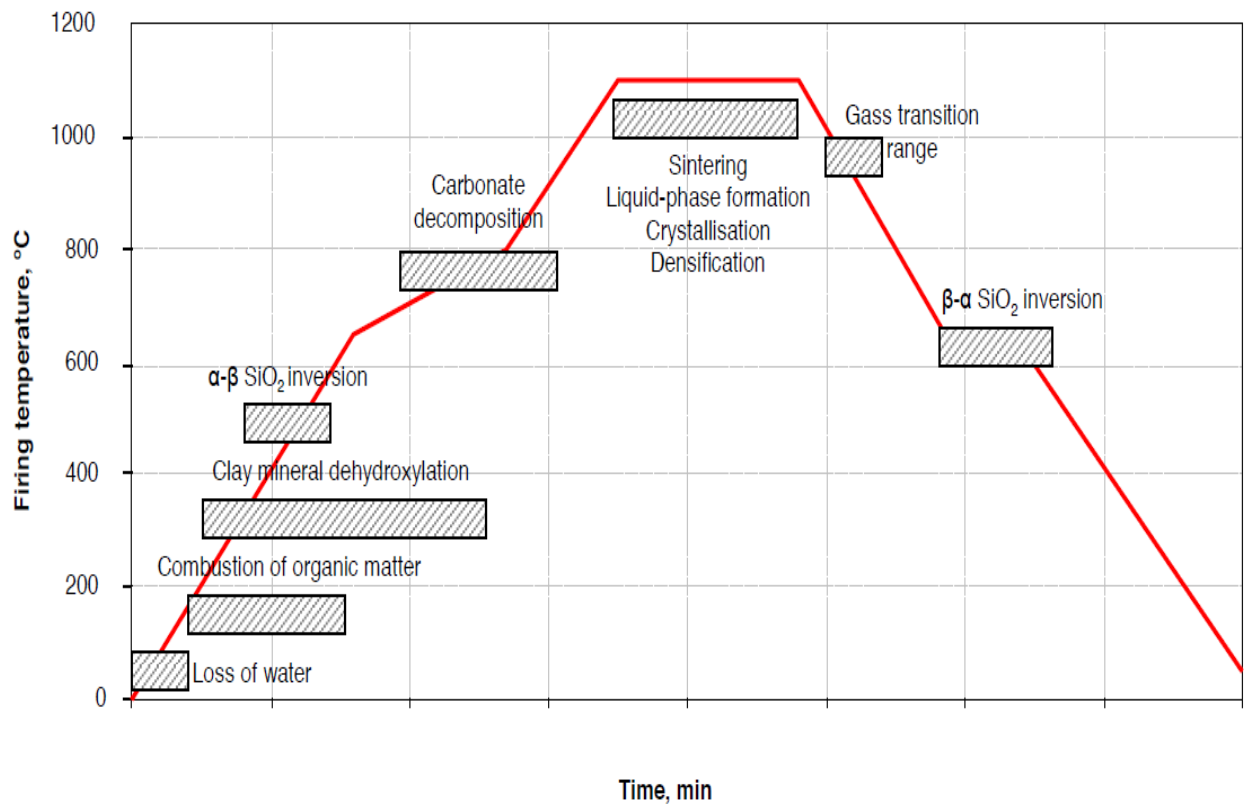


Figure 2.1: Typical firing curve with the physico-chemical reactions for traditional ceramic compositions (Lundin, S. T, 1964).

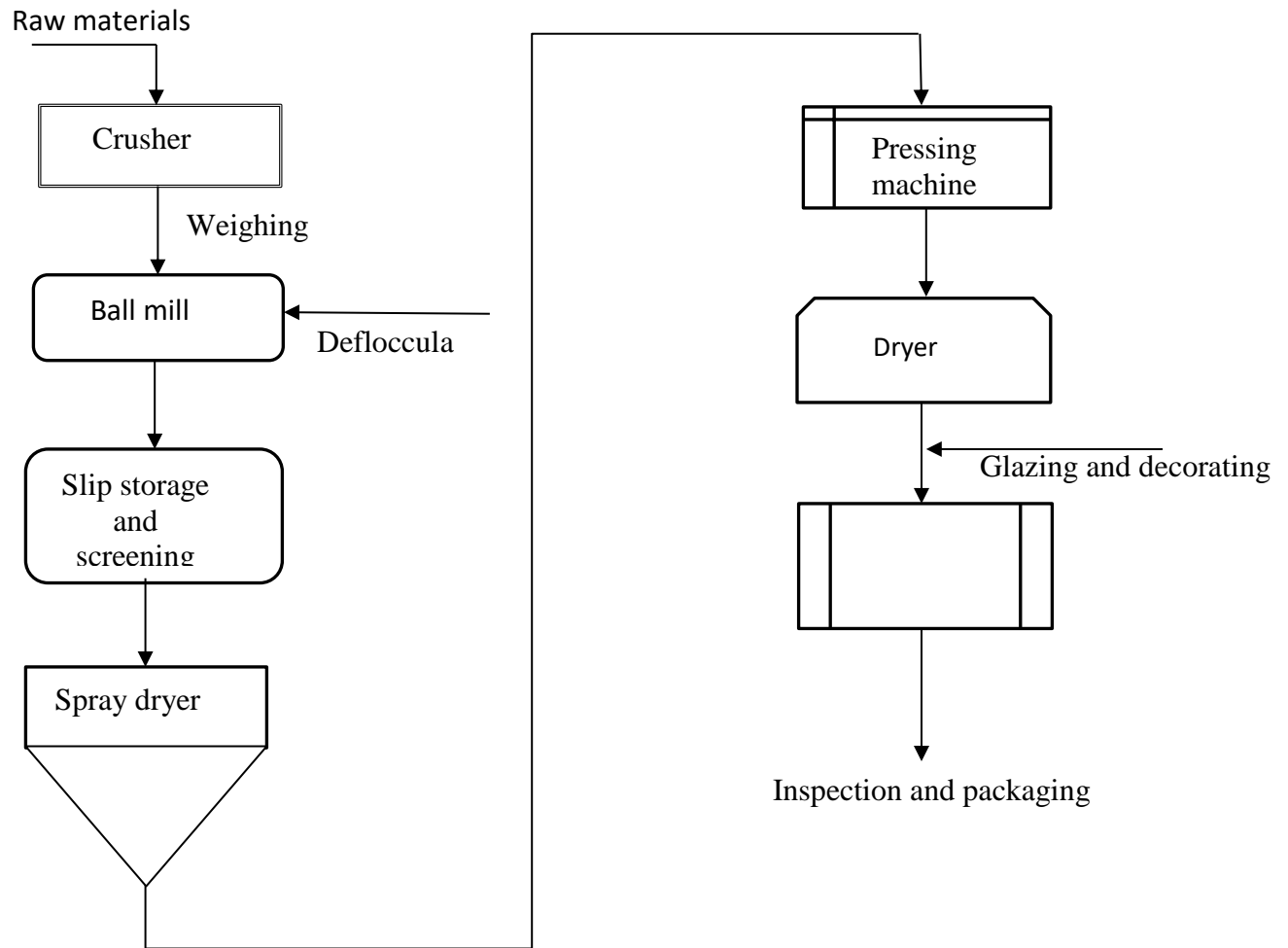


Figure 2.2: Main ceramic wall tiles production flow diagram (taken from TCSC).

3. MATERIALS AND METHODS

3.1 Sample collection and preparation for analysis

The Sugarcane bagasse ash has been collected from Wonji Shoa Sugar Factory, directly from boiler plant (after the sugarcane burned) and then two kilogram of the collected sample has been sieved by 250 μ m sieve size at Addis Ababa Institute of Technology (AAiT), School of Chemical and Bio- Engineering Laboratory for compositional analysis which has been performed at Ethiopian Geological Survey Laboratory (EGSL) and the result is shown in table 3.2. The crystal phases of SCBA and silica sand has been analysed using X-ray diffraction (XRD) at Adama Science and Technology University (ASTU), School of Mechanical, Chemical and Materials Science Engineering Laboratory.

Similarly, ceramic raw materials: clay, feldspar, quartz and kaolin have been collected from Tabor Ceramic Products Share Company. Clays (clay and kaolin) are used for plasticity and feldspar and quartz are used as fluxing and maintain dimensional stability during thermal treatments of the samples respectively. Water assists to wet milling, proper homogenization and keep fluidity and suspension property of mixtures with deflocculantes (STPP and Sodium silicate). Here the main advantages of deflocculantes are for suspension without affecting density of the slurry. The sugar cane bagasse ash replaced by quartz/silica sand (which is commonly called Arero silica sand by the factory) with 20%, 35% and 50%. Whereas all other ceramic wall tiles body compositions (Clay, Feldspar and Kaolin) including 1.2g and 1g amount deflocculants of silicate and STPP respectively are remained constant. Three hundred fifty grams (350g) of the ceramic wall tiles body composition and one hundred fifty litres (150L) /150g of water prepared for one batch cycle of the ball milling process to obtain the standard particle size (500 μ m) of the samples.

Table 3.1: The body compositions of wall tiles by replacing SCBA by 20% of silica sand

Raw Materials	Feldspar	Kaolin	Clay	Water	Silicate	STPP	Silica sand	SCBA	Total
Amount (g)	147	140	45.5	150	1.2	1.0	14.0	3.5	502.2

Table 3.2: The body compositions of wall tiles by replacing SCBA by 35% of silica sand

Raw Materials	Feldspar	Kaolin	Clay	Water	Silicate	STPP	Silica sand	SCBA	Total
Amount (g)	147	140	45.5	150	1.2	1.0	11.375	6.125	502.2

Table 3.3: The body compositions of wall tiles by replacing SCBA by 50% of silica sand

Raw Materials	Feldspar	Kaolin	Clay	Water	Silicate	STPP	Silica sand	SCBA	Total
Amount (g)	147	140	45.5	150	1.2	1.0	8.75	8.75	502.2

The total of 1004.4g of the samples has charged into two equal sizes pot mills each holding 502.2g and containing the grinding media (different size balls) and tightened, milled by predetermined speed (rpm) for 10 minutes for each batch cycle. The grinded sample has sieved by 500µm sieve size and put in tray dryer of 150°C temperature for about 4 hours, in order to remove the moisture from the samples. The dried sample was grinded by using mortar and pestle equipment and again sieved by the same sieve size (30mesh/500µm). Finally, the moisture content (7%) of the grinded sample has been adjusted by spraying water manually to the sample and made it ready for pressing machine (160g of the sample required for making one piece of the sample) at different pressing pressure (15, 17 and 19MPa.). As (M.F.Quered *et al.*, 2008) stated limiting the moisture content to the standard values (5-10%) significantly increases the mechanical strength of the pressed samples. The following recipe has been used for the above formulation of the ceramic wall tiles production which was taken from TCSC.

Table 3.4: Ceramic wall tile body compositions.

Body compositions	Amount (g)
Feldspar	147
Kaolin	140
Water	150
Clay	45.5
Silica sand	17.5
Silicate	1.20
STPP	1.00
Total	502.2

Source: Tabor Ceramic products share company

3.2 Experimental strategies

The intimate mixing of the raw materials is extremely important to the uniform completion of the solid-state reactions which take place during firing. Thus the response surface Design (Box Behnken Design (BBD), which is proposed for three-level designs for fitting response surface) in which a response of interest is influenced by several variables has been used. Adopting Box Behnken Design (BBD) method can sharply reduce the number of experimental sets without decreasing the accuracy of the optimization compared with traditional factorial design methods (M Manohar *et al* , 2014) analysis of variance (ANOVA) was used as one of the primary tools for statistical data analysis using Design-Expert software. The mixing of materials with partial replacements of quartz by SCBA (50%, 35%, and 20%) has been investigated with the body compositions with other raw materials remains constant for the production of better quality wall tiles.

Table 3.5: Experimental runs of 3 factors and 3 levels using BBD method

Standard	Run	Block	Factor 1 Comp. (%)	Factor 2 Press (MPa.)	Factor 3 Temp. (°C)
1	12	1	20	15	1100
2	11	1	50	15	1100
3	17	1	20	19	1100
4	10	1	50	19	1100
5	5	1	20	17	1000
6	9	1	50	17	1000
7	4	1	20	17	1200
8	14	1	50	17	1200
9	7	1	35	15	1000
10	13	1	35	17	1000
11	8	1	35	15	1200
12	1	1	35	19	1200
13	16	1	35	17	1100
14	6	1	35	17	1100
15	15	1	35	17	1100
16	3	1	35	17	1100
17	2	1	35	17	1100

The mixture of ash into the ceramic body has been homogenized in a ball mill (Speediness Grinding Machine), sieved and then dried using tray dryer. After drying the samples moisture content has been adjusted by manual spraying and the powder prepared for pressing at 15MPa, 17MPa and 19Mpa pressure using the pressing machine (CBZ Type Equipment for Manufacturing Tiles Sample). The pressed sample again dried in the same dryer type and simultaneously engobe and the glaze has been respectively applied (for glazed samples only), after the surface of the samples scrubbed with wet close. Then firing has been carried out in a furnace/kiln at 1000°C, 1100°C and 1200°C temperatures as shown in table 3.6 result from Design Expert software using Box Behnken design method.

The quality of the fired ceramic tiles has been determined on the basis of green and dry strength. Water absorption, crazing test (for glazed) and fired tensile strength/Modulus of rupture(MOR), has been carried out according to (Standards, 1985a). The mineralogical analysis of the specimen performed by X-ray diffraction (XRD) with the measuring condition of 40kV, 30mA and 2Theta angle of diffraction using copper (cu) as target element at scan speed of 4.0000 deg./min.

3.3 Materials and Equipments used for the experimental work

The following table shows the raw materials, equipment's and chemicals used during the experimental works.

Table 3.6: The raw materials, equipment's and chemical used during the laboratory works

Materials	Main significance
Raw materials	
Feldspar	To form the liquid phase and flux during firing
Kaolin and Clay	To improve the plasticity or cohesion during formation
Silica sand/quartz	To maintain dimensional stability of the material
SCBA	To lower the sintering temperature and improves tiles quality
Water	For wet milling and facilitate clay plasticity during forming
Chemicals	

Glaze	To give chemical resistance, glassy phase and aesthetic value for tiles
Engobe	To cover the ceramic body before glazing takes place
Silicate and STPP	Deflocculating agents
Equipments	
30 micro sieve	To get uniform particle sizes and granules
Electronic balance	For weighing the samples
Two small identical pot mills	Contain grinding media/balls and assist grinding
Speediness grinding machine	Grind the mixed compositions rotating at a specified speed(rpm)
Tray dryer	To remove the moisture content from the sample
Small plates	milled samples put in the dryer by this plates
Mortar and pestle	To commune the dried sample to standard particle size
Manual spray dryer	To spray water to achieve standard moisture content (7%)
Hydraulic pressing machine (laboratory)	To form the rectangular(10x10cm ²) piece /wall tiles
Mechanical strength (MOR) measuring Machines	To determine the mechanical strength of green, dried and fired wall tiles
Electric kilns	To give the sample stable crystalline phase through thermal treatment of samples/wall tiles
Water absorption equipment (vacuum method)	Determine the quantity of water a tile can absorb expressed as a percentage of the dry tile weight.
Autoclave	For thermal shock and crazing test analysis
Buller rings and Buller rings box	To estimate the firing temperature of the samples at specified location in the kiln.
Manual printers	To print the sample with the required surface design that improves the aesthetic values of the wall tiles.
Polyethylene plastics	To store and maintain the moisture content and assist for the aging of the prepared powder samples
Refractory plate	To place dried wall tiles on it and put into the kiln

3.4. Preparation of Ceramic Wall Tiles

In view of the fact that the study was based on the investigation for the partial incorporation of SCBA in wall tile body compositions, which had formulated according to the current recipe of the Tabor Ceramic Share Company (Table 3.4) and the experimental procedure is as follows.

Firstly, the raw materials weighed by using electronic balance (G&G[®], JJ600 Electronic scale) according to the percentage composition of silica sand/quartz replaced by SCBA (Table 3.1-3.3). Then the mixed and weighed compositions were charged into two equal-size pot mills containing the media/balls, figure 3.7 (b) and milled for 10 minutes with pre-set speed (rpm) by using milling machine (YJKS- Speediness grind machine) which is shown in figure 3.7 (a). The samples were prepared according to the recipes mentioned in tables 3.1 – 3.3 and milled by using a ball mill (wet type). The ball mill was used for the mixing of the formulated samples ensuring appropriate mixing and homogenous compositions. A uniform moisture distribution of the samples was achieved when the desired quantity of water was added and stored away in polyethylene bags. This mode of storing (at least for 24 hours) allowed a uniform distribution of moisture and assisted the samples to gain excellent particle interlock bonding during pressing. Particle size (sieved by 500 μ m) and moisture content (7%) of the powder is maintained constant (done manually). The grinded raw materials have sieved by using 500 μ m sieve size and dried in tray dryer (fig. 3.4) in order to remove the water added intended for milling, which is one of the drawbacks of the wet milling operation. The dried samples again grinded manually using mortar and pestle (fig.3.1) and the moisture content of the powder maintained prior to sieving, which enables to obtain controlled agglomerates (granules), separating larger size agglomerates which influences proper packing of particles during the pressing process.



Figure 3.1: Mortar and pestle used for grinding samples.

After the powder aging (at least 24 hours) maintained the mixed compositions were ready for making the wall tile specimens and pressed using a hydraulic press (CBZ Type Equipment for Manufacturing Ceramic Tile Sample). Forming of the tiles takes place by mechanically compressing the powder in the die/mould and is one of the most cost-efficient forming methods for making ceramic wares with regular geometry. The rectangular specimens/wall tiles of size $10 \times 10 \text{ cm}^2$ were made by pressing using hydraulic press, fig. 3.2 (a) at a pressing pressure of 15, 17 and 19MPa where the green wall tiles were obtained fig. 3.2 (b).

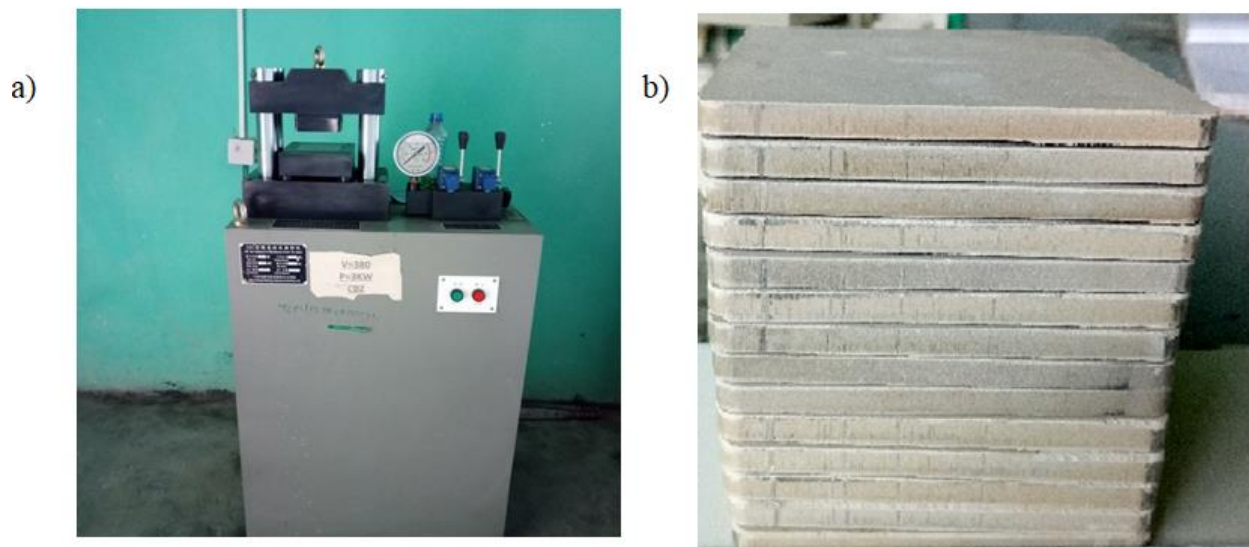


Figure 3.2: a) CBZ Type equipment for manufacturing Ceramic Tile Sample b) Pressured wall tiles.



Figure 3.3: Green and dry strength testing machines

After the green pressing the wall tiles were placed directly onto a 20x30cm² fired wall tiles and put into the tray dryer (figure 3.4) operated at constant temperature value usually 150°C. This process allowed air to circulate on all sides of the tiles and instantaneously the samples dried in order to achieve standard moisture content (< 1%).



Figure 3.4: Laboratory tray dryer

Finally, a set of dried wall tiles were directly placed onto the refractory plate and fired at 1000°C, 1100°C and 1200°C using an electric kiln (SX3 – 11-16 High speed Electrical Furnace) fig 3.5. The above procedure repeated to produce glazed wall tiles where engobe and glazes applied.



Figure 3.5: Electrical kiln firing at 1000, 1100 and 1200°C and the products for 2 hours

3.5 Characterization and Evaluation of the product

The total number of samples (wall tiles) prepared for different analyses were eighty-eight (88) among which 64 were for analysis of incorporated SCBA replacing quartz and 24 of the samples were prepared for controlling purposes using the factory's body compositions/without SCBA replacement. The prepared samples were: seventeen samples for unglazed, eleven samples for green strength analysis, ten samples for dry strength tests, seventeen samples for glazed analysis, four samples for printing test, five samples for XRD analysis and display/show. In addition to this the factory body compositions pressed for experimental controlling purpose were: six samples unglazed, six samples for green strength comparison, six for dry strength and six samples for glazed analysis.

3.5.1 Green and dry strength tests

The green and dry strength of the prepared wall tiles were analysed using strength testing machine, figure 3.3 (DPK-500 Electric blank Bending Instrument). Green strength, or handling strength, can be defined as the strength of a material processed to form its final ultimate tensile strength. The mechanical strength of the pressed wall tiles depends particularly on the bonding between particles and the absence of defects. Bonding strength and the number of inter-particle contacts depend directly on the moisture content, and the pressed density. Similarly, dry strength is the strength of wall tiles after drying at 150°C until the moisture content of the samples be less than 1%. This is important to withstand the load applied during printing/decoration process.

3.5.2 Modulus of rupture (MOR)

Modulus of rupture machines measures the flexural breaking load (bending strength) of samples/ceramic wall tiles by applying load to the tested piece. The range of bending strength machines available cover uses in tableware, sanitary ware, porcelain and ceramic tile manufacturing plants. The modulus of rupture/flexural strength of the ceramic wall tiles can be using the expression below. (taken from the factory manual).

$$\text{Modulus of rupture (MOR)} = \frac{1.5 \times F_{\max} \times L}{t^2 \times d} \times 10 \quad (3.1)$$

Where: F_{\max} = maximum load before rupture (kN), L= distance between the supports in (mm),

t = thickness of the specimen at the plane of failure in (mm) d = width of the specimen in (mm)

The breaking strength tends to increase with the increase of the material's modulus of rupture and of the thickness of the wall tiles. The decrease in (MOR) with an increase in ash content can be attributed to the presence of quartz particles, which is one of the main ash component. According to A.E. Souza *et al.*, (2011) quartz does not participate in the formation of new phases at sintering temperatures lower than 1000°C; however, at 1100°C and 1200°C, X-ray diffraction showed peaks corresponding to the formation of cristobalite. Silica (quartz, tridymite and cristobalite) has structural transitions of order-disorder, reversible, at various temperatures between 110°C and 1470°C. The fired wall tile's breaking strength (MOR) which is one of the quality parameters of the ceramic wall tiles has been measured using fired strength measuring machine (SKZ-10000 Ceramics brick crack modulus of rupture measuring instrument) as shown in fig 3.5 (a) both for glazed and unglazed samples fig. 3.6 (b).

3.5.3 Water absorption

The structural properties of the tiles described in terms of their porous structure. The most important of these properties is water absorption which constitutes a measure of porosity as well as a classification property that several other important properties depend on. On the other hand, water absorption is related to the microstructure of a sintered ceramic matrix and evaluates amount of the open pores of the fired specimen. Water absorption was then calculated by the equation:

$$\text{Water absorption(\%)} = \frac{W_{\text{wet}} - W_{\text{d}}}{W_{\text{wet}}} \times 100 \quad (3.2)$$

Where: W_{wet} – soaked weight (g) and W_{d} - dry weight (g)

High water absorption corresponds to a porous structure, while compact, vitrified structures feature shows low water absorption. A.E. Souza *et al.*, (2011), indicated that water absorption (WA) is an important property because, it is related to the effective realization of the sintering process by the liquid phase and observed that water absorption did not change much at temperatures below 1000°C, above this temperature, WA drops from 25% to values less than 8%, even becoming more increased at 1200°C. This indicates that the formation of the liquid phase, with a decrease in open porosity is greater at those temperatures. For this specific study, the Vacuum method has been used for water absorption analysis which is the contemporary method facilitating the water

absorption process as the vacuum has been created (made the pores free and ready the water to enter the body easily) within 18 minutes (vacuum time). The specimen was subjected to water absorption machine, figure 3.8 (a) (TXY-400 Ceramice Absorb Water Ration Determining Tester) for 40 minutes soaking time, the water on the surface of samples carefully removed with dry close. Then the soaked sample's mass (W_{wet}) weighed again and the percentage water absorption calculated using equation (3.2).

3.5.4 Shrinkage

Variation of dimensional stability among products or within the same product type is one defect that currently most affects ceramic wall tiles quality (James S. Reed, 2000). The thermo-mechanical properties of white ware bodies change greatly during the reconstructive and the displaced transformation of free silica due to change in volume. This change in dimension during drying and firing is called shrinkage which is one of the quality measurements of the ceramic wall tiles products (Prasad, 2001). As mentioned in many literatures, shrinkage is occurred as a result of the reduction in volume of particles during sintering process. Shrinkage properties were determined by initially measuring the original length of the green tiles along the centre and re-measuring the lengths after drying and firing of the tiles, using a calliper and the dry and fire shrinkage of the tiles calculated using the following equations (3.3) and (3.4) respectively.

$$S_d = \frac{L_g - L_d}{L_g} \times 100 \quad (3.3)$$

Where: S_d – dry linear shrinkage, L_g – green length of test specimen and L_d – Dry length of the test specimen

$$S_d = \frac{L_g - L_f}{L_g} \times 100 \quad (3.4)$$

Where: (S_f) and (L_f) – the fired linear shrinkage and length respectively.



Figure 3.6: Fired strength analysis a) MOR testing machine b) glazed and unglazed samples.

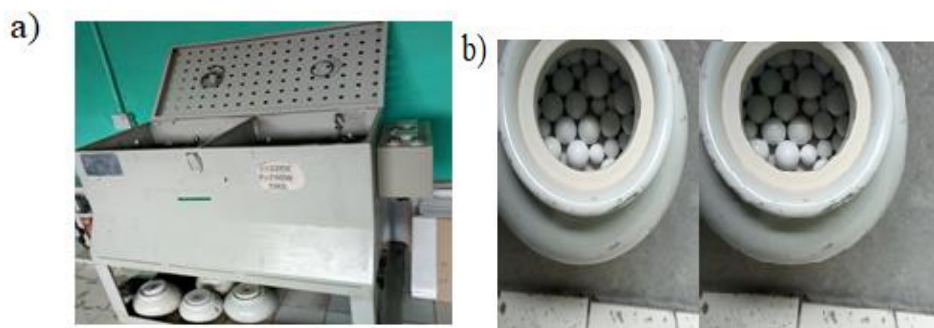


Figure 3.7: a) Speediness grinding machine, b) pot mills containing balls/medias

3.5.5 Crazeing test (CT)

Crazeing is defined as a hairline crack at the glazed tile surface (UNE, 1983). This defect arises as the result of tensile stress in a certain part of the glaze coating, which exceeds its mechanical strength, giving rise to a fracture at the surface. This test has been done by using autoclave equipment (CZ-1. OB Crack Resistance Measuring Instrument) as shown in figure 3.8 (b). The test was performed by putting only the glazed tiles specimen in the machine for two hours at a steam temperature of 160°C. The autoclave cooled slowly (at least four hours needed before opening, to prevent cracks due to sudden change of cooling) and the samples from the equipment taken out and crazeing test analysed by smearing the surface of the tiles using methylene blue and cleaned off immediately with water then fine lines observed, fig. 3.8 (c). There is no test value for crazeing. The sample either passes or fails and any tile claiming craze or crackle resistance should be able to pass the ASTM C424 (Michael F. and Byrne, 200).

Crazing is created due to the mismatch between the glaze and the ceramic body. The main factor that influences stress relations between a glaze and a ceramic body is the difference in coefficients of thermal expansion.

Table 3.7: Glaze and Engobe Compositions applied for this experimental work.

Materials	Compositions w/w%	
	Glaze	Engobe
A/t kaolin	-	4
B/Kaolin	10	16
Ball Clay	6	10
Feldspar	15	35
KS9602	6	-
KS9813	1	-
KT1304	32	10
TZ8003	8	-
TZ8007	15	22
Zirconium	7	3
Total	100	100

Source: Tabor Ceramic Products S.C



Figure 3.8: a) Water absorption machine b) Crazing Test Machine/Autoclave, c) crazed tiles

3.5.6 X-Ray diffraction (XRD) Analysis

Mineralogical composition or crystal phase analysis of the raw materials and products used for ceramics is important as minerals have a great influence on the technological behaviour and the final properties of the fired product. For this specific work the mineralogical analysis of the raw materials (Silica sand, SCBA) and the selected product (35% of ash replaced quartz and fired at 1100°C) has been analysed by using X-ray diffraction (X-Ray Diffractometers; XRD-7000S) using Cu-K α radiation with a voltage of 40 kV and current intensity of 30 mA at scanning speed of 4.000deg/min. From XRD results the crystalline (grain) size of the particles can be calculated by using Scherrer equation given below.

$$D = \frac{\kappa\lambda}{\beta \cos \theta} \quad (3.5)$$

Where: D = Crystallites size (nm)

$K = 0.9$ (Scherrer constant)

$\lambda = 0.15406\text{nm}$ (wave length of the x-ray sources)

β = FWHM (radians)

θ = peak position (radians)

4 RESULTS AND DISCUSSION

4.1 SCBA Chemical composition

Sugarcane bagasse ash had higher silicon dioxide (SiO_2) composition once the sugarcane bagasse burned at an average temperature of 700°C in boiler plant as confirmed from its chemical analysis (table 4.1). This composition provides the ash properties similar to fine sand (non-plastic material composed mainly of silica sand/quartz), which is used for ceramic wall tiles production promoting dimensional stability by decreasing the plasticity of the clayed raw materials (kaolin, other clays). The typical role of ceramic raw materials used for processing a ceramic wall tiles is considerably influenced by their mineralogical compositions, crystal phases as well as their physical and chemical properties. Thus the XRD analysis confirmed the presence of vital crystalline phase that are important in productions of ceramic wall tiles. Here in this investigation; the partial replacement of silica sand/quartz by SCBA appeared very comprehensive in production of ceramic wall tiles as observed both from physical tests (glossiness, planarity, shininess) and technological tests (Water absorption, shrinkage, MOR and LOI). This is due to the fact that higher amount of fluxing oxides (Na_2O , K_2O and MgO) in ash composition improved the quality of ceramic wall tiles by fusing at lower temperatures and dissolving materials like quartz which melt at higher temperatures. Similarly, optimum iron oxide (Fe_2O_3) composition facilitates the fluxing oxides in lowering the sintering temperature without affecting the tiles body-colour resulted during the sintering process. The crystal structure and the grain particle size of the ash improved the mechanical property (MOR) and porosity of ceramic wall tiles pressed even at lower pressing pressure (17MPa) compared to the conventionally pressed wall tiles at 19MPa by the company (Tabor Ceramic S.C).

Table 4.1: Analytical method of silicate analysis results for SCBA and silica sand (wt.%)

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI
SCBA	72.02	7.20	4.16	0.44	1.28	2.16	4.52	0.16	0.47	0.09	1.29	5.90
Silica sand	84.34	9.37	1.76	0.68	0.34	1.96	2.98	-	-	0.18	-	-

Source: SCBA analysed at EGSL and for Silica sand the data taken from Tabor Ceramic Factory.

4.2 Analysis the ceramic wall tiles

4.2.1 Green test

As observed from tables 4.2 and 4.3 the mechanical strength (MOR) of the samples with SCBA and the factory body composition are almost similar as calculated using equation (3.1). The pressing process has been closely controlled i.e. the powder uniformly filled and evenly distributed in the die. Hence the shape distortion during firing has been minimized and low firing shrinkage has been maintained. Obviously insignificant variation in dimensions (esp. thickness) has been observed in both compositions i.e. with or without SCBA as shown in tables 4.2 and 4.3. This variation is due to non-uniformity of die-wall friction and inaccuracy of the pressing operation (semi-manual) during pressing which causes compactness differences of the tiles.

Table 4.2: Wall tiles Green strength pressed at different pressures and 7% w/w M.C

Run	Composition (%)	Pressure (MPa)	Dimensions (mm)		Machine reading (kN)	MOR(kg/cm ²)
			Thickness	Length		
12	20	15	7.10	106.70	31.90	8.89
17	20	19	7.00	106.60	33.80	9.71
5	20	17	7.00	106.50	32.40	9.80
16			7.00	106.60	33.70	9.68
15	35	17	7.00	106.60	33.60	9.65
2			7.00	106.50	33.60	9.65
7	35	15	7.10	106.60	32.70	9.13
1	35	19	6.90	106.60	34.20	10.17
11	50	15	7.10	106.60	33.10	9.24
10	50	19	7.00	106.50	34.10	9.80
9	50	17	7.00	106.60	33.80	9.71

*Three runs 16, 15 & 2 correspond to 35 & 17 composition and pressure respectively. M.C = Moisture Content

This implies that the mechanical properties of green wall tiles slightly depend on the sample's composition but to some extent influenced by applied pressure which directly has an effect upon the thickness of the body. It rather depends on grinding and dispersion of raw materials during

milling process which increases the proportion of submicron particles that can significantly improve the particle packing during pressing. Hence, for a specified time the composition with SCBA grinded more because of its less hardness property of the ash compared to quartz. This directly affected the mechanical strength of the wall tiles.

From table 4.2 and 4.3 both compositions (with and without SCBA) the green strength of the wall tiles pressed at 17 and 18MPa are 9.72 and 9.42kg/cm² respectively, which is within the standard range of the company's and ISO standard specifications (5-12kg/cm²). From these results replacing SCBA has advantage of saving energy requirement for pressing the ceramic wall tiles i.e. modulus of rupture of the body containing SCBA pressured at 17MPa and the factory body composition pressured at 18MPa are virtually equal. This is due to crystal structure which improves interlocking of particles and finer grain size of the sample with SCBA obtained during milling process and appropriate granule formed.

Table 4.3: Ceramic wall Tiles Green strength test (Factory composition) at 7% w/w. M.C

Trials	Code	Dimensions (mm)		Machine reading(kN)	MOR(kg/cm ²)
		Thickness	Length		
1	C _{L1} P ₁₈	6.50	106.20	24.90	8.32
2	C _{L2} P ₁₈	6.70	106.10	35.00	11.02
3	C _{L3} P ₁₈	6.80	106.20	30.30	9.26
4	C _{L4} P ₁₈	6.90	106.40	32.70	9.99
5	C _{L5} P ₁₈	6.80	106.30	29.70	8.79
6	C _{L6} P ₁₈	7.00	106.20	31.60	9.11

M.C. - Moisture content, C_{L(1-6)} - factory body compositions, P₁₈- constant pressure applied at 18MPa.

4.2.2 Drying test

The pressed tiles were placed directly into tray dryer in order to remove the moisture content to the standard value (<1%) preceding the firing process to start and the modulus of rupture of the samples was calculated using equation (3.1). From table 4.3 the average value of the mechanical strength of the dried wall tiles containing SCBA compositions is 29.88kg/cm² which implies that the samples got enough drying strength for the next process as it met the drying quality standard specified by ISO (20-30kg/cm²). Better drying strength was obtained when compared with the factory body composition/without SCBA which is in average 26.86kg/cm². As

observed from tables 4.4 and 4.5 the average values of the mechanical strength of samples with SCBA (27.4kg/cm^2) pressed at 15MPa and the samples without SCBA (26.79kg/cm^2) pressed at 18MPa are nearly the same. This confirmed that the drying strength of the samples is slightly affected by the uniaxial applied pressure relatively more influenced by the composition, microstructure and grain size of the powder.

Table 4.4: Ceramic wall Tiles dry strength test result (with SCBA)

Run	Composition(%)	Pressure (MPa)	Dimensions (mm)		Machine reading(kN)	MOR (kg/cm^2)
			Thickness	Length		
12	20	15	7.20	106.20	92.70	25.57
17	20	19	6.90	106.30	107.90	31.98
5	20	17	7.00	106.10	117.40	33.87
16			7.10	106.20	105.70	29.62
3	35	17	7.00	106.10	85.60	24.69
7	35	15	6.90	106.10	87.90	26.10
1	35	19	7.00	106.20	109.20	31.48
11	50	15	7.00	106.20	105.90	30.53
10	50	19	6.90	106.20	108.40	32.16
9	50	17	6.90	106.20	110.40	32.81

*The average value of dry strength of the body containing SCBA is 29.88kg/cm^2 .

This is due to the improved microstructure and finer grain size of SCBA which has been contributed for uniform drying rate. The more uniform grain size within the body compositions the better will be the diffusion (water) from internal to external surface that assisted for drying rate and enhanced mechanical strength of the wall tiles.

Table 4.5: Ceramic wall Tiles dry strength test results (Factory composition)

Trials	Code	Dimensions (mm)		Machine reading(kN)	MOR(kg/cm ²)
		Thickness	Length		
1	C _{L1} P ₁₈	7.40	106.20	110.60	28.53
2	C _{L2} P ₁₈	7.30	106.10	98.40	26.16
3	C _{L3} P ₁₈	7.20	106.30	102.70	27.96
4	C _{L4} P ₁₈	7.20	106.10	97.50	26.18
5	C _{L3} P ₁₈	7.20	106.40	88.90	24.18
6	C _{L4} P ₁₈	7.10	106.30	99.10	27.74

*The average value of dry strength of the body without SCBA is 26.79kg/cm²

4.2.3 Firing Shrinkage

It is apparent that the manufacturing process starts with raw materials preparation and ends with packing. All stages are equally important to ensure the overall performance of the production process. However; the firing stage is the most sensitive process and the bottleneck of the tile production cycle. The linear drying and firing shrinkage of the samples with SCBA compositions was determined using equations (3.3&3.4) and summarized as in table 4.6. Here the average linear firing shrinkage is the shrinkage calculated from green to fire (total linear shrinkage) it is neither green to dry nor dry to fire. The average linear shrinkage of the samples with the ash compositions of 20, 35 and 50% are 6.01, 5.15 and 5.33% respectively. On the other hand, the average linear shrinkage of the ash compositions with 20 and 50% are relatively higher which did not meet the limited standard values specified by ISO (3-5%) this could be due to lower fluxing compounds (less liquid phases) in 20% formulation and a bit higher organic matters released in case of 50% formulation which directly related to shrinkage of samples. Here 35% ash replaced nearly met the standard value than other compositions.

From table 4.6 the average linear shrinkage values fired at temperatures of 1000°C, 1100°C, and 1200°C are 3.62%, 5.23% and 7.53% respectively, whereas the average linear shrinkage from table 4.7 are 2.21, 4.46 and 5.3% respectively at the same firing temperature. Here the wall tiles formulated from ash and fired at 1100°C only fit the stated standard values. From these values, it is clear that the linear shrinkage greatly depends upon the firing temperature.

As the tiles firing temperature increased the linear shrinkage has been increased too. When the percentage linear shrinkage of the wall tiles containing SCBA fired at 1100°C compared with the percentage linear shrinkage of the body composition of the factory/without SCBA fired at 1200°C, their values are virtually equal (5.23 and 5.3%). This indicates that the wall tiles replaced quartz fired at lower temperature value, provided the fact that more presence of the fluxing oxides (Na_2O and K_2O) especially of K_2O in the waste (SCBA) than in silica sand (table 4.1). As A.E. Souza, *et al.*, (2011) revealed the more presences of iron oxide (Fe_2O_3) modifies the sintering process because they produce a sharp decrease in the viscosity of the liquid phase. But much amount of iron oxide makes the tiles reddish in colour; nevertheless, in this work, the SCBA content of this oxide (4.4%) did show the improved colour of the produced wall tiles. Even better body-colour has been acquired compared to the conventionally produced wall tiles.

Table 4.6: Wall tiles production Laboratory data result (unglazed)

Run	Code	Green analysis (mm)		Dry analysis (mm)			Fir analysis (mm)			Shrinkage values (%)		
		Thickness.	Length	Thickness	Length	Wt.(g)	Thickness.	Length	Wt.(g)	G-D	D-F	G-F
12	C ₂₀ P ₁₅ T ₁₁	7.00	106.40	6.70	106.40	147.40	6.30	101.10	138.90	0.00	4.98	4.98
17	C ₂₀ P ₁₉ T ₁₁	7.00	106.50	7.00	106.50	147.50	6.20	100.80	139.10	0.00	5.35	5.35
16		7.00	106.50	7.00	106.50	147.60	6.50	100.30	145.60	0.00	5.82	5.82
6		7.10	106.40	6.90	106.40	147.80	6.30	100.80	139.70	0.00	5.26	5.26
15		7.20	106.50	7.00	106.20	148.20	6.50	101.90	140.20	0.28	4.05	4.32
3	C ₃₅ P ₁₇ T ₁₁	7.10	106.50	7.00	106.10	148.40	6.30	100.90	140.20	0.38	4.90	5.25
2		7.10	106.50	7.10	106.40	148.70	6.30	100.60	140.60	0.09	5.45	5.54
11	C ₅₀ P ₁₅ T ₁₁	7.20	106.40	7.10	106.10	146.60	6.30	100.60	138.50	0.28	5.18	5.45
10	C ₅₀ P ₁₉ T ₁₁	7.00	106.50	7.00	106.50	146.40	6.60	101.00	139.10	0.00	5.16	5.16
5	C ₂₀ P ₁₇ T ₁₀	7.00	106.60	6.90	106.40	147.20	6.31	102.50	138.30	0.19	3.67	3.85
9	C ₅₀ P ₁₇ T ₁₀	7.00	106.40	6.90	106.40	146.20	6.50	102.70	139.20	0.00	3.48	3.48
7	C ₃₅ P ₁₅ T ₁₀	7.00	106.50	7.00	106.40	147.10	6.50	102.90	138.50	0.09	3.29	3.76
13	C ₃₅ P ₁₉ T ₁₀	7.00	106.60	7.00	106.40	147.30	6.50	103.00	137.70	0.19	3.19	3.38
14	C ₅₀ P ₁₇ T ₁₂	7.00	106.50	7.00	106.40	147.20	6.30	98.80	138.50	0.09	7.14	7.23
4	C ₂₀ P ₁₇ T ₁₂	7.00	106.50	7.00	106.30	147.80	6.20	96.00	139.10	0.19	9.69	9.86
8	C ₃₅ P ₁₅ T ₁₂	7.00	106.50	6.90	106.50	148.30	6.40	89.00	140.10	0.00	7.04	7.04
1	C ₃₅ P ₁₉ T ₁₂	7.10	106.60	7.10	106.50	147.90	7.00	101.50	138.90	0.09	5.92	6.00

* From $C_xP_yT_z$ – the subscripts x , y , and z are replaced ash composition, pressing pressure and firing temperatures respectively. G-D, D-F, and G-F mean the green to dry, dry to fire and green to fire shrinkage values. Wt.- weight, the run numbers are trail numbers taken from design expert software. Run numbers like 16, 6, 15, 3, and 2 correspond to C₃₅P₁₇T₁₁ only

Table 4.7: Ceramic Wall tiles produced without SCBA (unglazed).

Run	Code	Green analysis(mm)		Dry analysis(mm)			Fire analysis (mm)			Shrinkage values (%)		
		Thickness	Length	Thickness	Length	Wt.(g)	Thickness	Length	Wt.(g)	G-D	D-F	G-F
5,9	CL1P18T10	7.10	106.40	6.90	106.40	143.40	6.80	104.20	137.80	0.00	2.07	2.07
7,13	CL2P18T10	7.00	106.50	7.00	106.30	142.40	6.50	104.00	134.20	0.19	2.16	2.35
12,17,16,6	CL3P18T11	7.10	106.40	7.10	106.40	147.80	6.70	105.60	138.70	0.00	4.60	4.60
15,3,2,11,10	CL4P18T11	7.01	106.50	7.10	106.30	148.90	6.60	101.90	140.80	0.19	4.14	4.32
14,4	CL5P18T12	7.20	106.70	7.20	106.50	148.90	6.80	101.10	140.70	0.19	5.07	5.25
8,1	CL6P18T12	7.20	106.70	7.20	106.50	149.20	6.80	101.00	140.80	0.19	5.16	5.35

* $C_{Lx}P_yT_z$ - Lx, y, and z are the factory composition, pressing pressure and fired temperature respectively done for controlling purposes.

4.2.4 Modulus of rupture analysis (Fired)

The modulus of rupture is the property of the material that constitutes the wall tiles tile and is generally inversely proportional to the level of water absorption. This analysis determines the mechanical properties of the ceramic wall tiles.

Table 4.8: Modulus of Rupture and Loss on ignition for SCBA containing unglazed samples

Run	Code	Dry	Fir analysis (mm)			Machine Load(kN)	MOR (kg/cm ²)	LOI (%)
			Wt. (g)	Thickness	Length			
12	C ₂₀ P ₁₅ T ₁₁	147.40	6.30	101.10	138.90	572	213.82	5.70
17	C ₂₀ P ₁₉ T ₁₁	147.50	6.20	100.80	139.10	546	211.37	5.60
16		147.60	6.50	100.30	145.60	692	244.95	6.09
6		147.80	6.30	100.80	139.70	811	304.07	5.50
15	C ₃₅ P ₁₇ T ₁₁	148.20	6.50	101.90	140.20	693	241.45	5.40
3		148.40	6.30	100.90	140.20	753	282.04	5.50
2		148.70	6.30	100.60	140.60	760	284.67	5.40
11	C ₅₀ P ₁₅ T ₁₁	146.60	6.30	100.60	138.50	742	278.75	5.50
10	C ₅₀ P ₁₉ T ₁₁	146.40	6.60	101.00	139.10	749	257.66	5.12
5	C ₂₀ P ₁₇ T ₁₀	147.20	6.31	102.50	138.30	607	223.81	6.05
9	C ₅₀ P ₁₇ T ₁₀	146.20	6.50	102.70	139.20	444	153.49	4.79
7	C ₃₅ P ₁₅ T ₁₀	147.10	6.50	102.90	138.50	454	156.6	5.85
13	C ₃₅ P ₁₉ T ₁₀	147.30	6.50	103.00	137.70	459	158.21	6.50
14	C ₅₀ P ₁₇ T ₁₂	147.20	6.30	98.80	138.50	771	294.92	5.90
4	C ₂₀ P ₁₇ T ₁₂	147.80	6.20	96.00	139.10	949	373.59	5.90
8	C ₃₅ P ₁₅ T ₁₂	148.30	6.40	89.00	140.10	938	385.96	5.70
1	C ₃₅ P ₁₉ T ₁₂	147.90	7.00	101.50	138.90	867	264.92	6.10

*Loss on Ignition (LOI) is the materials lost during drying to the firing process and calculated from the percentage of weight loss between the dried and fired samples.

The modulus of rupture of the wall tiles (unglazed) as a function of the SCBA composition and sintering temperature can be seen in table (4.8). The average values of the modulus of rupture for the body compositions containing SCBA by 20, 35 and 50% are 255.65, 270.78 and 246.21 kg/cm² respectively. Generally, from these values it is possible to conclude that all values obtained are

within the range of the factory (Tabor Ceramics) production standard (200-300kg/cm²) and replacing silica sand/quartz by SCBA with the above-specified compositions is advantageous in order to produce wall tiles having better mechanical strength compared to the factory body composition (without SCBA) as shown in table (4.9), which is averagely 200.75kg/cm² and less compared with that of the body compositions containing SCBA which is averagely 257.55kg/cm² (average values of MOR at 20, 35 and 50 ash compositions).

Also, it can be observed that the average values of the modulus of rupture for the body compositions containing SCBA and fired at 1000, 1100 and 1200°C are 173.03, 254.62 and 329.85kg/cm² respectively. These values indicated that mechanical strength (MOR) of the samples increased with sintering temperature. The temperature at which the modulus of rupture was maximal was 1200°C for these particular formulations. From the results, it can be seen that the modulus of rupture increased with increasing densification (less pores), but the best values which meet the factory standard has been obtained at 35% SCBA replacement and 1100°C sintering temperature which are 270.78kg/cm² and 254.62kg/cm² respectively.

Table 4.9: Modulus of rupture and loss on ignition for factory body composition (unglazed)

Run	Code	Dry Wt.(g)	Fir analysis (mm)			Machine Load(kN)	MOR (kg/cm ²)	LOI (%)
			Thickness	Length	Wt. (g)			
5,9	CL1P18T10	143.4	6.80	104.20	137.80	552	171.85	3.91
7,13	CL2P18T10	142.4	6.50	104.00	134.20	328	111.97	5.76
12,17 16,6	CL3P18T11	147.8	6.70	105.60	138.70	587	185.74	6.16
15,3,2 11,10	CL4P18T11	148.9	6.60	101.90	140.80	578	195.32	5.40
14,4	CL5P18T12	148.9	6.80	101.10	140.70	767	246.10	5.50
8,1	CL6P18T12	149.2	6.80	101.00	140.80	914	293.56	5.60

* $C_{Lx}P_y T_z$ - Lx, y, and z are the factory composition, pressing pressure and fired temperature respectively done for controlling purposes. The run numbers indicate that trials performed with SCBA containing compositions at the same temperature values for controlling purposes.

4.2.4 Water absorption

Water absorption is the most dominating quality parameter to evaluate the prepared wall tiles as it has a direct relationship with the sintering process of ceramic wall tiles, porosity and shrinkage.

As presented in table 4.10 the water absorption in relation with body composition and firing temperature has been tested. From this table, the average water absorption for 20, 35 and 50% SCBA incorporated in the body composition of wall tiles are 6.08, 6.95 and 7.17% respectively. From these body formulations, as the compositions of SCBA increases water absorption of the prepared wall tiles also increases, where almost all values are within the standard values (6-8%) set by Tabor ceramic factory.

Table 4.10: Water absorption for tiles fired at 1000°C, 1100°C and 1200°C (for unglazed body)

Run	Code	Soaking weight (g)		Water absorption (%)
		Before	After	
12	C ₂₀ P ₁₅ T ₁₁	138.9	149.5	7.10
17	C ₂₀ P ₁₉ T ₁₁	139.2	148.2	6.07
16		138.3	149.8	7.68
6		139.6	150.2	7.06
15	C ₃₅ P ₁₇ T ₁₁	140	150.7	7.10
3		140.3	150.5	6.78
2		140.4	150.3	6.59
11	C ₅₀ P ₁₅ T ₁₁	138.4	148.9	7.05
10	C ₅₀ P ₁₉ T ₁₁	78.4	85.1	7.87
5	C ₂₀ P ₁₇ T ₁₀	71.3	78.9	9.63
9	C ₅₀ P ₁₇ T ₁₀	57.5	65.1	11.67
7	C ₃₅ P ₁₅ T ₁₀	67.6	76.1	11.17
13	C ₃₅ P ₁₉ T ₁₀	59.1	67.2	12.05
14	C ₅₀ P ₁₇ T ₁₂	135.6	138.5	2.09
4	C ₂₀ P ₁₇ T ₁₂	137.0	139.1	1.51
8	C ₃₅ P ₁₅ T ₁₂	138.3	140.2	1.36
1	C ₃₅ P ₁₉ T ₁₂	139.0	141.4	1.69

*T₁₀, T₁₁ and T₁₂ Shows firing temperature at 1000°C, 1100°C, and 1200°C respectively.

The average water absorption values of the samples fired at each sintering temperature are significantly different. From table 4.10 the average water absorption of the samples fired at 1000°C, 1100°C, and 1200°C are 11.13, 7.03 and 1.66% respectively. Both the average water absorption values of samples fired at 1000°C and 1200°C did not meet the minimum and maximum standard values. This is due to under fired conditions for wall tiles fired at 1000°C and its sensitivity to high temperature at 1200°C firing temperature value as A.E. Souza *et al.*, (2011) stated. It's under fired properties (nonglossy and non-shine) are observed physically, no measure values for this properties.

Again from this table the average water absorption of the samples without SCBA fired at 1000°C, 1100°C, and 1200°C are 10.46, 8.79 and 0.42% respectively. Hence the average of water absorption of the sample's composition replaced quartz by 35% of SCBA and fired at 1100 °C meets the standard value than others. But still the value with SCBA composition better confirms the standard at 1100°C firing temperature. Generally, from both tables (4.10 &4.11), it is possible to conclude that firing temperature significantly affects the wall tiles water absorption.

Table 4.11: Water absorption without SCBA fired at 1000°C, 1100°C and 1200°C (unglazed)

Run	Code	Soaking weight (g)		Water absorption (%)
		Before	After	
5,9	CL1P18T10	76.7	84.3	9.02
7,13	CL2P18T10	71.8	81.5	11.90
12,17, 16,6	CL3P18T11	76.4	82.5	7.39
15,3,2, 11,10	CL4P18T11	140.8	155.9	10.19
14,4	CL5P18T12	140.8	141.5	0.49
8,1	CL6P18T12	140.4	140.9	0.35

C_L indicates the factory composition fired at 1000⁰C, 1100⁰ and 1200⁰C for the aim of experimental control with corresponding runs.

Regardless of wall tiles (glazed or unglazed) water absorption is the most important parameter to determine its the quality. Thus from the above value (8.79%) the wall tile fired at 1100°C by 35% SCBA replacement with silica sand has some better mechanical properties. Abrasion resistance

tends to increase as water absorption decreases, reflecting increase in the structural compactness likewise moisture expansion and porosity have a direct relationship with water absorption.

4.3.4 Loss on Ignition

Loss on ignition is the percentage loss of materials during drying to firing at different stages: a) removal of physically adsorbed water or free moisture removal from the kaolinite/clay particles, b) dehydration of hydroxides, releasing H₂O molecules from their structures; c) removal of chemical water bounded to the kaolinite structure; d) associated with the decomposition of carbonates (calcite and dolomite) or organic compounds with the release of CO₂. As shown from tables 4.8 and 3.9 the samples with and without SCBA presented an average loss on ignition (LOI) of 5.68 and 5.39% respectively. This indicates that SCBA has slightly more organic compounds compared to silica sand and high LOI value is indicative of unburned carbon, a compound that interferes with the hydration reaction and increases the demand for water (J. Torres Agredo et al , 2014). Generally, in almost all silica sand replaced by compositions of SCBA, the loss on ignition (LOI) of the wall tiles are within the limit standard (2.5-5%) with that of the factory standard.

4.3.5 Crazing test

The wall tiles have generally tested for their crazing resistances by autoclaving at 160°C steam temperature making stress on the tiles specimens for a certain period of time (2hours for this specific test). Crazing test performed for glazed wall tiles only and the data recorded (table 4.12 and 4.13) from the replicated experiments (17 samples with SCBA compositions and 6 samples from factory body composition) whereas, other properties (Shrinkage, water absorption, MOR and Loss on ignition) has been also analysed. The experiment revealed that the glazed fired tiles with 50% replacement shown some defects such as holes, bubbles and lack of glossy/ shiny appearance they had cracks and had no shiny appearance. Among the performed laboratory experiments the ceramic body composition of 35 % ash fired at 1100°C is relatively better both in physical structure and in quality wise pass the criteria.

4.3.8 X-ray diffraction analysis

As previously expressed, the mineralogical compositions of the samples were determined using X-ray diffraction analyses. The XRD patterns (Fig.4.1) shows a little difference between

quartz/silica sand and sugarcane bagasse ash in terms of their crystal structures, which suggests that the phase transformations have taken place to produce wall tiles. From these figures the XRD results revealed that the development of the crystalline peaks of SCBA, quartz/silica sand and the selected wall tiles product correspondingly.

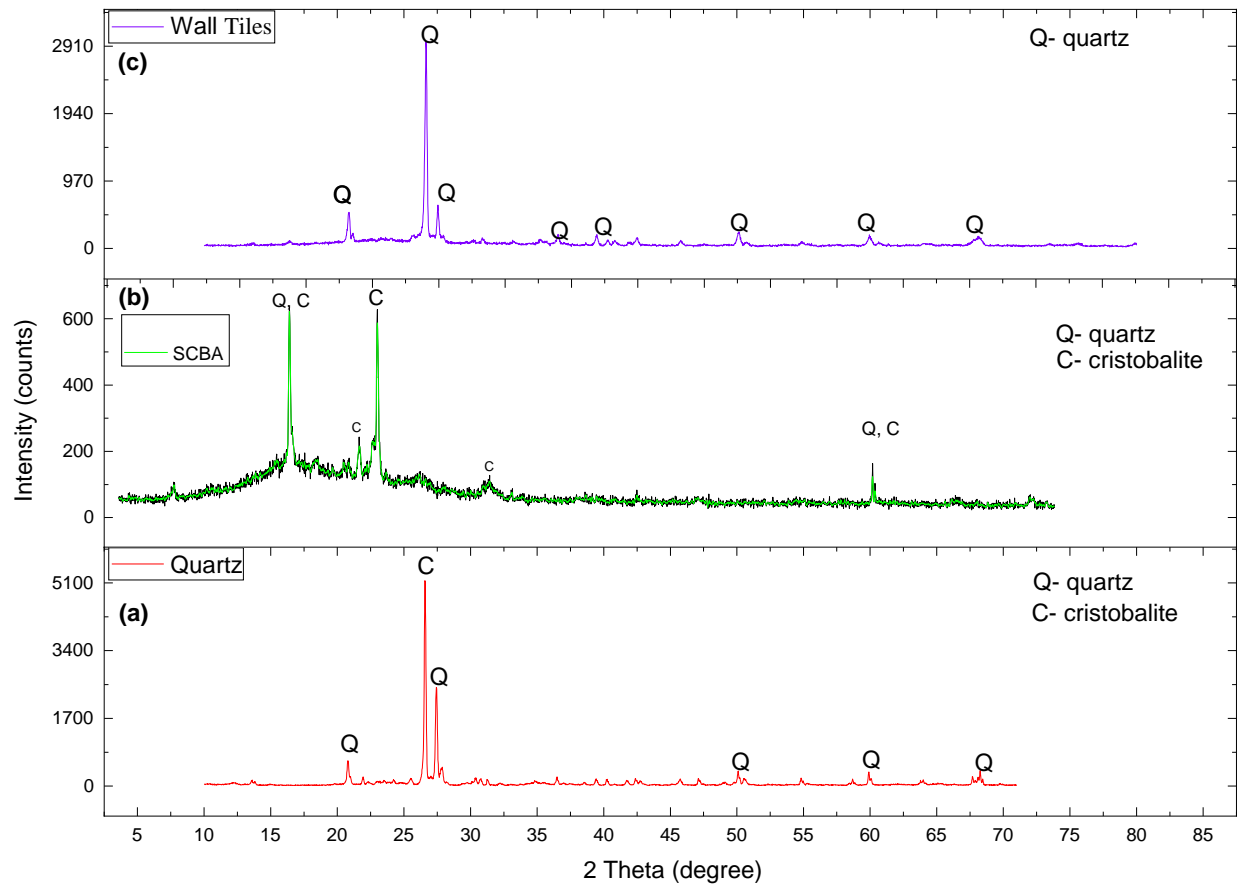


Figure 4.1: XRD analysis for Quartz, SCBA and Wall tiles (Fired at 1100°C)

As (E. Arif, M.W. and Clark, N. Lake, 2016) understood the XRD patterns show a broad hump that is distinctive of amorphous minerals. The shape, position and width of peak provide the information about its structure. Sharp and intense peaks shown in SCBA are may be due to the re-crystallisation i.e. from mullite phase to crystalline (cristobalite) phase with temperature and this implies that the crystallinity increases as temperature increases. However, from the peaks the degree of crystallinity of relatively pure quartz/silica sand is higher than that of SCBA since crystallinity depends on intensity of the peaks. This lower crystallinity minimizes the worsen circumstances occurred due to higher crystallinity that negatively affects the quality of the

products. From the selected wall tile (35% SCBA, fired at 1100°C and pressed at 17MPa), XRD analysis result indicated that the bagasse ash and quartz became highly crystalline structure as evident from sharp reflection peaks. The XRD pattern of the SCBA waste is shown in Fig. 4.1(b) and the main crystalline phases like quartz (SiO₂) and cristobalite (SiO₂) were identified with predominance of quartz as confirmed by chemical analysis (Table 4.1).

During sintering process cristobalite contribute to stable crystal phase in earthenware ceramic products that need to possess high porosity. From the nature of peaks and XRD result analysis by using ‘match software’ and Powder Diffraction Files (PDF) or ICDD, the wall tiles product contains both silica sand and SCBA compositions that are suitably contributed in strengthening of the mechanical property of the product. As shown from the figure 4.1(b) the first peak shows that the cristobalite (PDF#820512) crystal phase that contributes for strengthening of the wall tiles fig. 4.1(c). On the other hand, the alkali metal oxides that contribute during sintering process could not be identified by x-ray diffraction. This is due to the low concentration of these elements in the samples which do not form new crystalline phases, as they may be in the glassy phase and/or in solid solution within the crystalline phases to contribute in lowering sintering temperature as mentioned in chapter one. In other words, the average crystalline size calculated from the major peaks of SCBA and quartz/silica sand are 5.087nm and 5.487nm respectively. The smaller the crystal size (grain size) the higher the particle dissolutions and surface area of the particles during sintering process and resulted in enhancing the bonds between particles.

Table 4.12: Wall tiles production Laboratory data result (Glazed)

Run	Code	Green analysis(mm)		Dry analysis (mm)			Fire analysis (mm)			Shrinkage values (%)			Soaking wt. (g)		H ₂ O Abs. (%)	LOI (%)	Machine reading (kN)	MOR (kg/cm ²)
		Thick ness	Length	Thick ness	Length	Wt.(g)	Thick ness	Length	Wt.(g)	G-D	D-F	G-F	Before	After				
12	C ₂₀ P ₁₅ T ₁₁	6.80	106.0	6.60	106.0	147.2	6.50	101.0	146.1	0.00	4.72	4.72	67.3	74.5	9.66	0.75	594	208.79
17	C ₂₀ P ₁₉ T ₁₁	6.80	106.2	6.70	106.2	147.1	6.70	102.0	145.7	0.00	3.95	3.95	72.2	78.4	7.91	0.95	587	192.30
16		6.70	106.1	6.60	106.1	147.0	6.60	101.9	145.6	0.00	3.96	3.96	73.1	79.6	8.16	0.95	607	205.12
6		6.70	106.1	6.70	106.1	147.2	6.60	108.8	145.9	0.00	4.05	4.05	72.3	78.8	8.25	0.88	710	239.93
15	C ₃₅ P ₁₇ T ₁₁	6.70	106.1	6.70	106.1	147.7	6.70	108.8	145.8	0.00	4.05	4.05	75.1	82.1	8.04	1.29	807	264.89
3		7.00	106.5	7.00	106.5	147.6	6.50	100.3	145.6	0.00	5.82	5.82	73.9	80.1	7.74	1.36	803	284.24
2		6.90	106.4	6.90	106.4	147.5	6.60	100.7	146.2	0.00	5.38	5.38	72.9	80.4	9.33	0.88	801	273.91
11	C ₅₀ P ₁₅ T ₁₁	6.90	106.2	6.70	106.2	146.8	6.80	102.5	145.6	0.00	3.48	3.48	98.9	109	9.27	0.82	722	228.50
10	C ₅₀ P ₁₉ T ₁₁	7.00	106.5	7.00	106.5	146.4	6.60	98.7	145.0	000	7.32	7.32	75.4	85.1	11.39	0.90	799	278.76
5	C ₂₀ P ₁₇ T ₁₀	6.70	106.2	6.50	106.1	145.3	6.40	105.3	144.1	0.09	0.75	0.85	58.3	64.9	10.17	0.83	597	207.65
9	C ₅₀ P ₁₇ T ₁₀	6.70	106.1	6.50	106.1	147.0	6.50	105.1	146.0	0.00	0.94	0.94	58.4	64.7	9.74	0.14	487	164.51
7	C ₃₅ P ₁₅ T ₁₀	6.80	106.2	6.70	106.2	146.8	6.60	105.3	145.7	0.00	0.85	0.85	58.2	65.8	11.5	0.75	463	151.41
13	C ₃₅ P ₁₉ T ₁₀	6.70	106.1	6.60	106.1	147.0	6.60	105.3	145.6	0.00	0.85	0.85	57.3	67.1	14.61	0.95	507	165.96
14	C ₅₀ P ₁₇ T ₁₂	6.70	106.1	6.70	106.1	146.9	7.00	102.5	145.9	0.00	3.39	3.39	58.7	59.2	0.84	0.68	802	239.52
4	C ₂₀ P ₁₇ T ₁₂	6.80	106.2	6.80	106.1	147.1	6.30	100.2	146.1	0.09	5.56	5.65	59.8	61.1	2.13	0.68	908	342.47
8	C ₃₅ P ₁₅ T ₁₂	6.90	106.2	6.90	106.2	146.8	6.20	100.4	145.9	0.00	5.46	5.46	66.8	67.4	0.89	0.61	842	327.25
1	C ₃₅ P ₁₉ T ₁₂	6.90	106.3	6.90	106.1	149.2	6.70	100.7	148.2	0.19	5.09	5.27	69.7	72.8	4.26	0.67	847	281.06

Table 4.1: Data for Ceramic Wall tiles produced by the Factory body compositions (Glazed).

Run	Code	Green analysis(mm)		Dry analysis (mm)			Fire analysis (mm)			Shrinkage values (%)			Soaking wt. (g)		H ₂ O Abs. (%)	LOI (%)	Machin ^e reading (kN)	MOR (Kg/cm ²)
		Thick.	Length	Thick.	Length	Wt.(g)	Thick.	Length	Wt.(g)	G-D	D-F	G-F	Before	After				
5,9	CL1P18T11	7.10	106.2	7.00	106.2	147.8	6.90	105.4	148.4	0.00	0.75	0.75	77.3	85.2	9.27	1.01	602	179.9
7,13	CL2P18T11	7.20	106.4	7.00	106.3	147.7	6.80	103.5	146.3	0.09	2.63	2.73	76.4	81.1	5.79	1.19	563	176.46
12,7 16,6	CL3P18T10	7.20	106.4	7.20	106.4	149.0	6.90	101.5	147.0	0.00	4.33	4.33	67.9	75.1	4.59	1.3	698	210.31
15,21 1,10	CL4P18T10	7.10	106.2	7.00	106.2	147.9	7.00	101.6	146.8	0.00	4.33	4.33	74.0	81.8	9.34	0.74	580	174.74
14,4	CL5P18T12	7.10	106.2	7.10	106.2	148.2	6.90	103.5	147.0	0.00	2.54	2.54	69.8	70.2	0.57	0.81	827	251.74
8,1	CL6P18T12	7.10	106.4	7.00	106.3	147.7	6.90	102.6	146.9	0.09	3.48	3.57	60.7	69.8	1.94	1.22	864	265.31

4.4 Experimental analysis using design expert software

Box-Behnken design is used for designing the experiment since it has the maximum efficiency for an experiment involving three factors and three levels. The study was conducted on the replacement of quartz/silica sand by SCBA according to the selected experiment design method (BBD) listed in table 3.6. Using this design method, the following four main steps has been followed.

1) An experimental plan is provided based on the chosen factors and levels. 2) Statistically designed experiments were performed. 3) The coefficients in the mathematical model are estimated and the accuracy of the model is checked. 4) Response analysis is performed to predict the optimal conditions, and these predictions is virtually same with experimentally obtained result. The most dominant quality parameter; water absorption has been selected for analysis of variance (ANOVA) using design expert software as follows.

4.4.1 Response (Water Absorption)

From table 4.13 the model F-value of 99.83 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are not significant. In this case A, B, AB, B² are significant model terms. As shown in Table 4.13 the linear coefficients (*A and B*), a quadratic term coefficient (B²) and cross product coefficients (*AB*) were significant, with very small *p*-values (*p* < 0.05). The other term coefficients were not significant (*p* > 0.05). Therefore, A, AB and B² were important factors in the ceramic wall tiles production process. Specifically, from these model it is possible to deduce that the temperature linear and polynomial increments determine the water absorption capacity of the ceramic wall tiles, which needs to optimize in order to get the standard water absorption criteria.

Values greater than 0.1000 indicate the model terms are not significant. The F-value of 853.39 (table 4.13) implies that model parameter B (temperature) is the factor that highly influenced water absorption of the wall tiles. This confirms that in ceramic wall tile production as sintering temperature increases the liquid phases formed due to fluxing compounds minimizes the porosity of the tiles and its water absorption capacity also reduced. Hence, temperature should be optimised in order to produce the wall tiles that meet standard water absorption criteria.

Table 4.2: Analysis of variance table [Partial sum of squares]

Source	Sum of Squares	df	Mean squares	F Value	p-value Prob > F	
Model	188.73	9	20.97	99.83	0.0001<	Significant
A Composition	3.60	1	3.60	17.16	0.0043	
B Temperature	179.27	1	179.27	853.39	<0.0001	
C-Pressure	0.50	1	0.50	2.38	0.1668	
AB	0.53	1	0.53	2.54	0.1552	
AC	2.03	1	2.03	9.67	0.0171	
BC	0.076	1	0.07	0.36	0.5674	
A ²	0.013	1	0.013	0.063	0.8092	
B ²	2.44	1	2.44	11.61	0.0113	
C ²	0.35	1	0.35	1.65	0.2404	
Residual	1.47	7	0.21	-	-	
Lack of Fit	0.79	3	0.26	1.53	0.3357	Not significant
Pure Error	0.68	4	0.17	-	-	
Cor Total	190.20	16	-	-	-	
Std. Dev.		0.46		R-Squared	0.9923	
Mean		6.79		Adj R-Squared	0.9823	
C.V. %		6.75		Pred R-Squared	0.9282	
Press		13.66		Adeq Precision	30.752	

Source: design expert software ANOVA result.

The lack of fit test, measures the failure of the model to represent experimental data in the experimental domain at point which are not included in regression analysis. Lack of fit was found non-significant (p value 0.3357) and "F-value" of 1.53 implies that it is not significant relative to the pure error. There is a 33.57% chance that a "Lack of Fit F-value" this large could occur due to noise. Non-significant lack of fit is good that indicates we want the model to fit. Thus the above non-significant lack of fit suggests that the model equation was adequate to predict water absorption under any sets of combination of the variables (composition, temperature and pressure).

The coefficient of determination ($R^2 = 0.9923$), the adjusted coefficient of determination ($R^2_{Adj} = 0.9823$) and the coefficient of variation (C.V. = 6.75%) are shown above, where the values indicate that the accuracy and the general availability of the polynomial model are adequate, and the R^2_{Pred} of 0.9282 is in reasonable agreement with the R^2_{Adj} . In other words the calculated model was able to explain 99.23% of the results. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 30.752 indicates an adequate signal. This model can be used to navigate the design space.

4.4.2 Final Equation in Terms of Coded Factors

Using multiple regression analysis on the experimental data, the response variable (water absorption) and the test variables (composition, temperature and Pressure) were related by the following second-order polynomial equation.

$$\begin{aligned} \text{Water Abs.} = & +7.04 + 0.67A - 4.73B + 0.25C - 0.6AB + 0.71AC - 0.14BC - 0.056A^2 \\ & - 0.76B^2 + 0.29C^2 \end{aligned} \quad (4.1)$$

Where: A - Composition (%),
 B -Temperature (°C) and
 C - Pressure (MPa)

4.4.3 Surface plots

2-D and 3-D plots has been drawn for different combination of parameters which exhibit the trend of variation of response (water absorption) within the selected range of input parameters and also influence of each parameter over the other parameters. Few such typical plots are shown in figures below. The patterns of the plots are almost alike for counter and 3-D plots and hence below only the 3-D plot has been selected for discussions.

Design-Expert® Software

Water Abs.

● Design Points

X1 = A: Composition

Actual Factors

B: Temperature = 1100.00

C: Pressure = 17.00

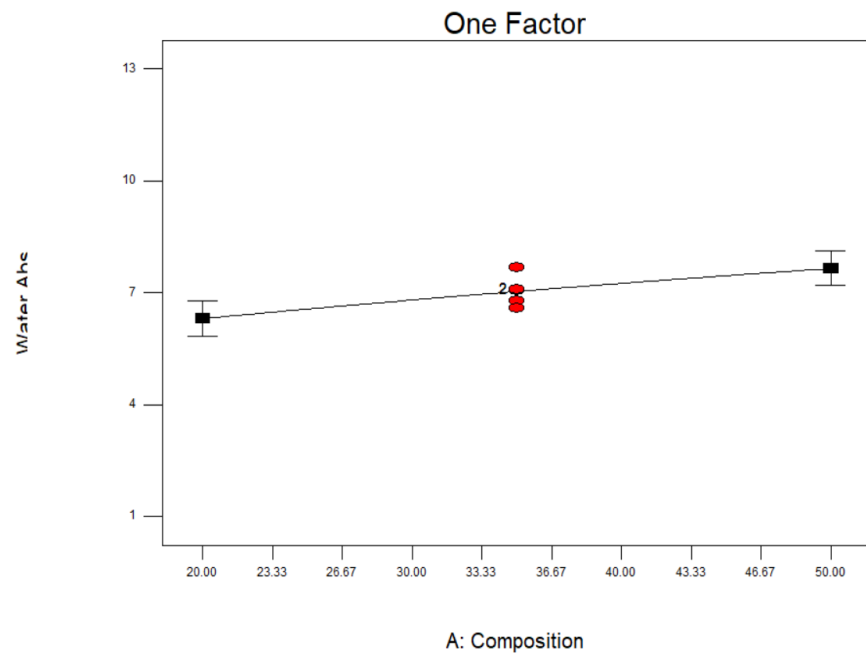


Figure 4.2: The effect of composition on water absorption

As observed from fig. 4.2 water absorption and composition have a direct relationship. On the other hand, as the composition increased water absorption has also slightly increased. Thus the increment of composition insignificantly affects water absorption capacity of the wall tiles.

Design-Expert® Software

Water Abs.

● Design Points

X1 = B: Temperature

Actual Factors

A: Composition = 35.00

C: Pressure = 17.00

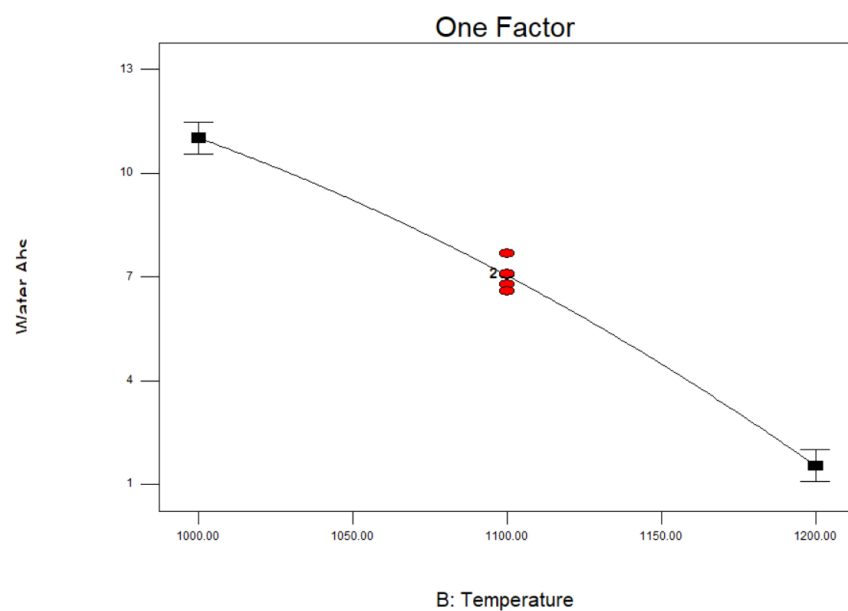


Figure 4.3: The effect of firing temperature on water absorption

Figure 4.3 indicates that as temperature increases water absorption drastically decreases which confirms that as firing temperature increases the porosity become narrower and the probability of the water to enter into the fired wall tiles becomes less, where it could be zero (water absorption for electrical insulators is 0%). From this graph (fig. 4.3) it is vivid that optimum firing temperature is highly important in order to obtain standard water absorption (6-8%) for wall tiles production. On the other hand pressing pressure has no significant effect on water absorption as shown on fig 4.4 below. However, the graph indicates that at higher and lower pressing pressure a bit water absorption increment has observed. This is may be due to non uniformity of preperd samples and/or semi-manual pressing operation process.

Figure 4.4 below indicates that water absorption of fired cramic wall tile do not affected by pressing pressure. Here eventhough pressing pressure could be one of the main factors, its influence after firning might be no significant, this is porosity which is directly related to water absorption capacity of fired wall tiles determined by the glassy phases/fluxes formed during sintering process. The pressing pressure directly influences the green and dry strength of the wall tiles. Hence the relationship between water absorption and pressing pressure observed below is the expected result from the practical point of view.

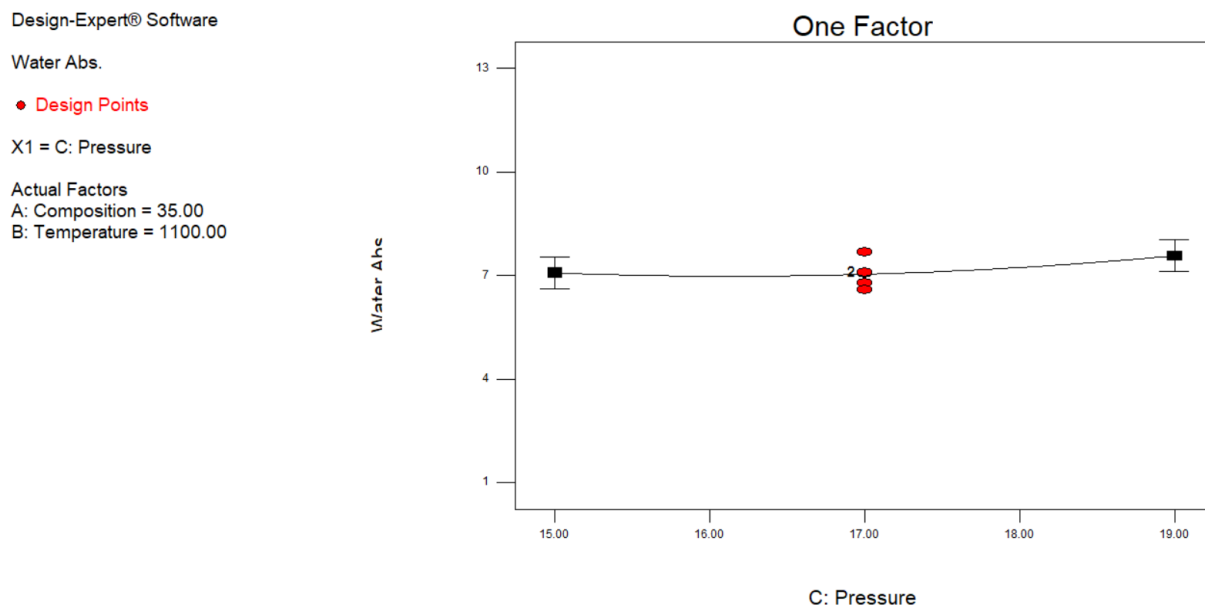


Figure 4.4: The effect of Temperature on water absorption

4.4.4 Interaction effects

The relationship between response and factors has been illustrated in 3-D response surfaces and 2-D plots generated by the model for wall tile produced.

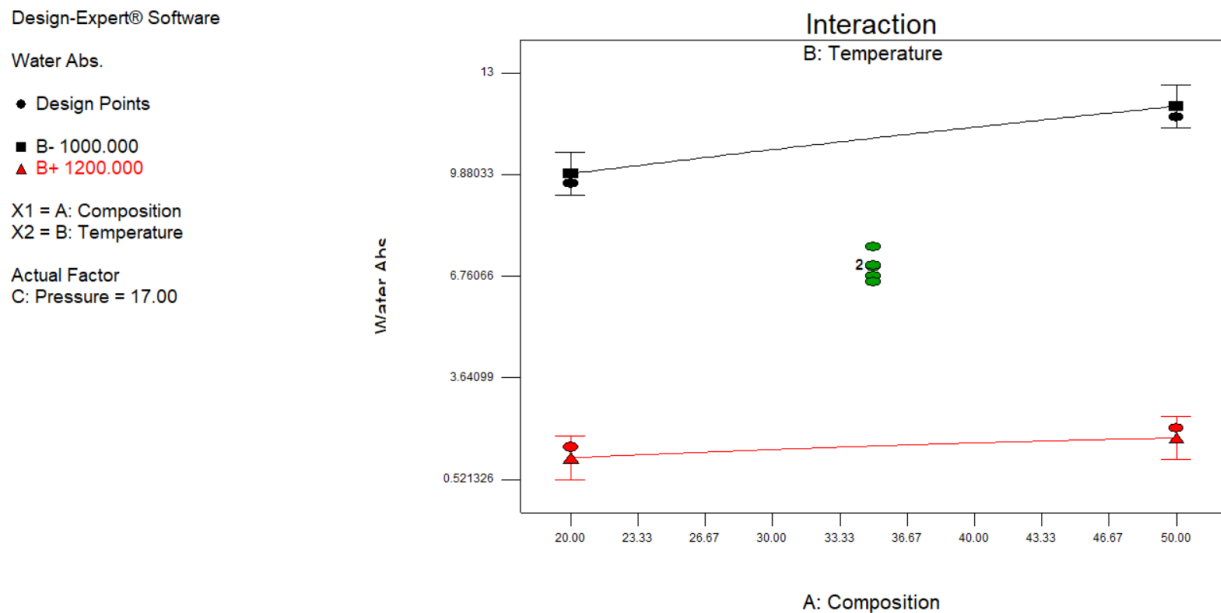


Figure 4.5: Interaction effects of Composition and Temperature on water absorption

From fig. 4.5 at low temperature (1000°C) value there is slight water absorption increment observed as the ash composition increased. This is due to the under fired wall tiles that resulted in higher porosity existence which is directly related to water absorption capacity. This implies that liquid phases did not create at this temperature and likewise organic matters within the sample disappeared but the pores have not been filled with liquid phases. Similarly, at higher temperature value (1200°C) water absorption insignificantly increased. Even though, water absorption drastically decreased as temperature increased it could be reversed if much amount of organic matters present within the samples that disappeared during firing result in pores formations and as well facilitate water absorption. Generally, as temperature increases water absorption decreases for all compositions.

Design-Expert® Software

Water Abs.

● Design Points

■ C- 15.000

▲ C+ 19.000

X1 = B: Temperature

X2 = C: Pressure

Actual Factor

A: Composition = 35.00

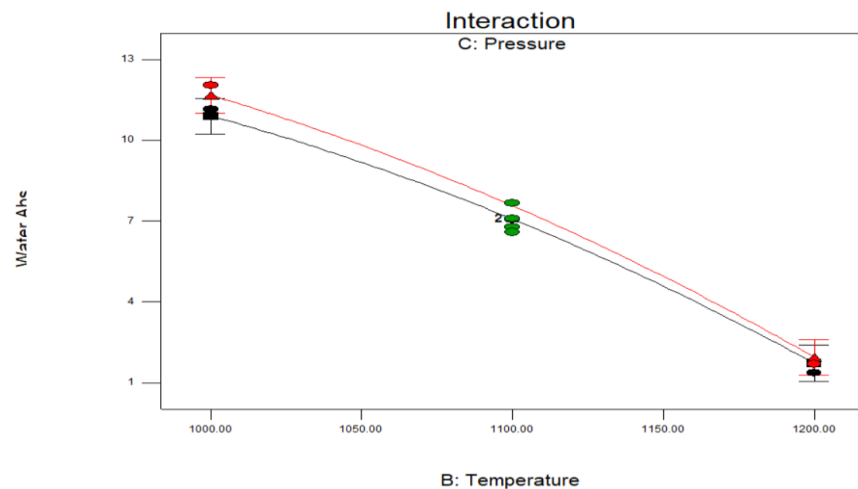


Figure 4.6: The effect of Temperature and pressure on water absorption

Figure 4.6 shows that temperature and pressure interactions has significant on water absorption. However at high (19MPa) and low (15MPa) pressure values there is no significant change on water absorption. Even at higher temperature the effect of pressure on water absorption become very much insignificant. This is due to the fact that temperature is more determinant on porosity of the wall tiles than pressure as the effect of fluxing compounds become visible at higher temperature.

Design-Expert® Software

Water Abs.

Color points by value of
Water Abs.:

■ 12.05

■ 1.36

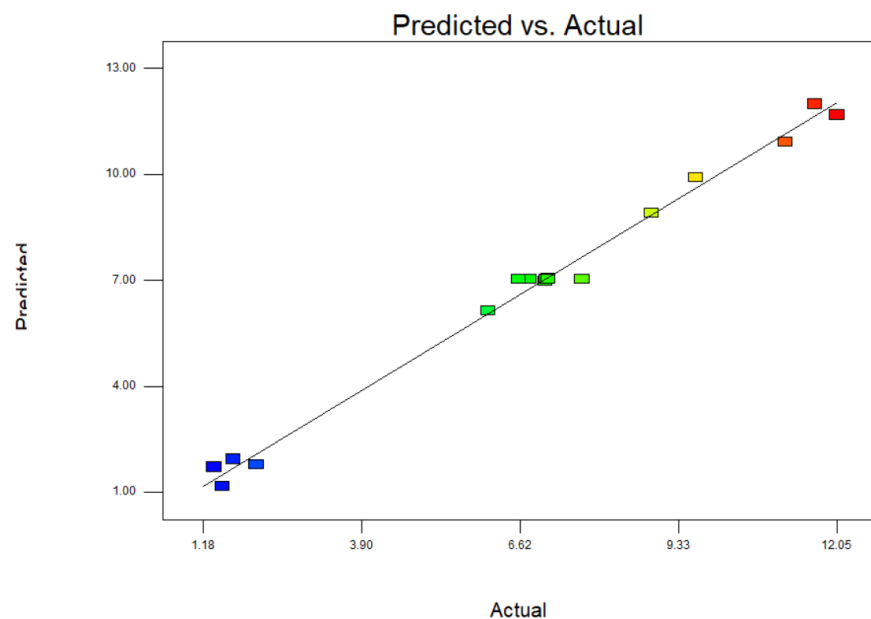


Figure 4.7: The predicted vs actual plot of water absorption

In the figure 4.8, ideal line is plotted taking the predicted value same as the measured value and considered as a reference/diagonal line. A measured value of the response is plotted and its closeness to the ideal line depicts the accuracy (fitness) of the model. The model developed for the response (water absorption) is considered accurate, where almost all the measured-values are aligning or closer with the ideal line. In this cases, predicted and the experimental values follow close match and the extent of deviation is minimal. In general the observed and predicted water absorption plotted in fig. 4.8 suggests a satisfactory correlation between experimental and predicted value, where the data points localized close to the diagonal line.

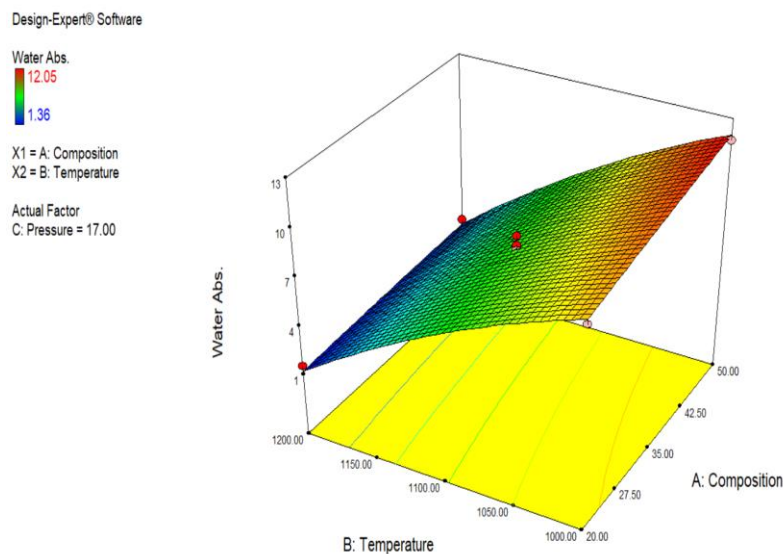


Figure 4.8: Interaction effects of Composition and Temperature on water absorption (3-D)

The 3D plot represents response surface of two independent variables where remaining variables are fixed at their respective zero level. Figures (4.8 -4.10) indicate that the main and their interaction effects (composition, temperature and pressure).

Fig.4.9 represents response surface plot of two independent variables firing temperature and composition of the wall tiles. It can be inferred that minimum water absorption has observed at higher firing temperature. However composition has no significant effect in water absorption. Actually temperature is considered as one of the critical parameter for the production of wall tiles product.

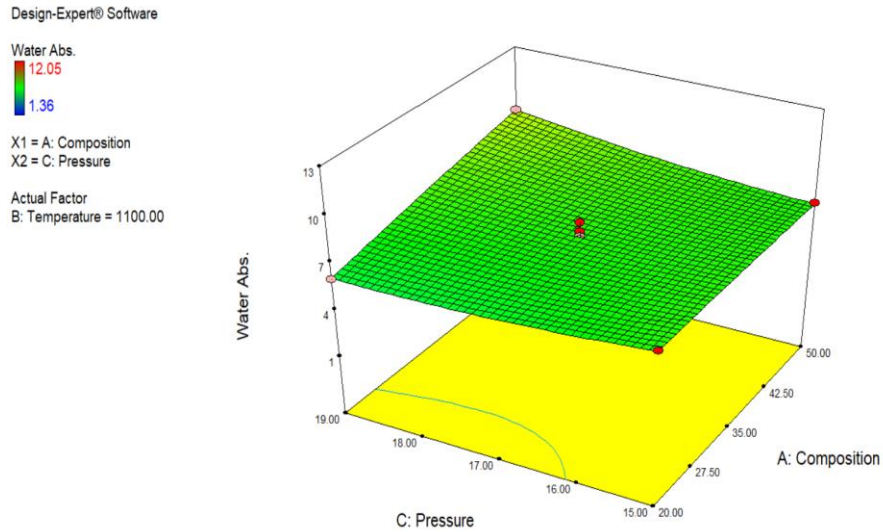


Figure 4.9: Interaction effects of Composition and Pressure on water absorption

Figure 4.10 it has been seen that water absorption was found there is no significant change with increase in pressure and composition. But as shown from the figure at higher ash composition water absorption influenced which could be resulted from higher porosity of the tiles occurred as a result of disappearance of organic matters (higher in SCBA) during firing.

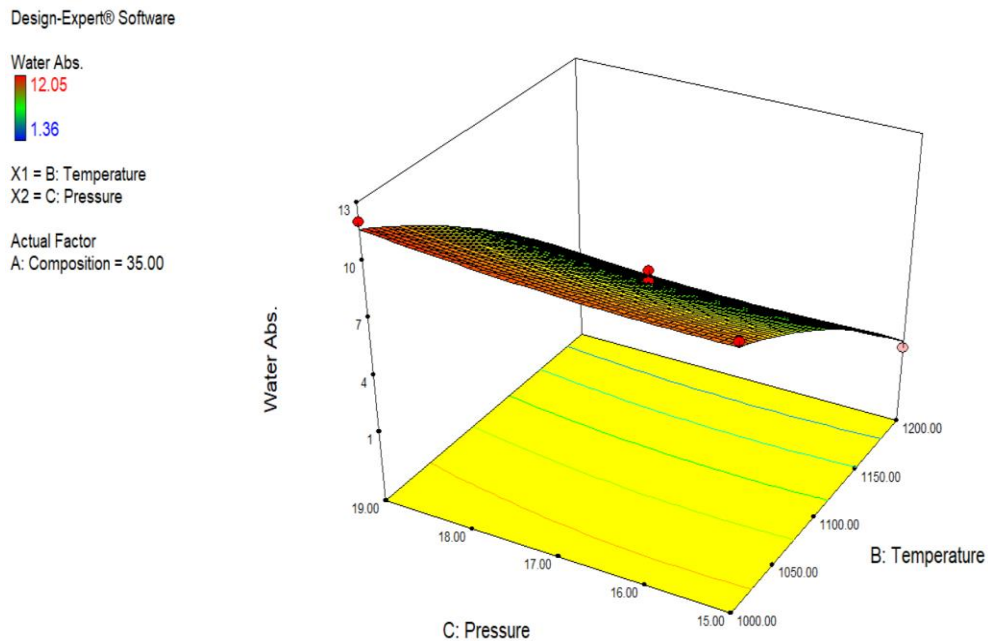


Figure 4.10: Interaction effects of pressure and Pressure on water absorption

Figure 4.11 again indicates that maximum water absorption resulted at minimum temperature value, in other words as temperature increases water absorption drastically decreases whereas pressure has no significant effect on water absorption capacity of the wall tiles. As observed from the graph both at low and high temperature values standard water absorption has not obtained that indicated temperature need to be optimized. From this graph optimum water absorption observed around the center values, at 1100°C temperature and 17MPa pressure.

5. CONCLUSION

This investigation confirmed valorisation of SCBA for the production of ceramic material (wall tiles) promoting appropriate utilization of this waste instead of discarding to the environment which causes adverse effect on human being as well as environments. The compositional and mineralogical content of the SCBA revealed and confirmed that the potential of the waste to be replaced the ceramic raw material (quartz) acquiring both environmental and economic benefits. The SCBA waste is chemically rich in silica particles (72.02%), quartz/ cristobalite (SiO_2) and also contains fluxing compounds like: Na_2O , K_2O , hematite and etc., which contributed to lower the sintering temperature during production of ceramic wall tiles. The compositional and mineralogical analysis of SCBA and silica sand were performed by using atomic absorption spectrometer and (XRD) respectively. The experiments were conducted replacing quartz by SCBA with 20, 35 and 50% and the effects of sintering temperature at 1000°C, 1100°C and 1200°C and pressing pressure at 15, 17 and 19MPa were investigated. Hence, 1100°C sintering temperature, replacement of SCBA by 35% and pressing pressure at 17MPa were selected as optimum values according to the experimental study and confirmed by statistical analysis (Appendix table A). The quality parameters for ceramic wall tiles (green and dry strengths, fired strength, water absorption, and crazing tests) has been analysed accordingly.

Generally, the following conclusion has drawn from the investigation carried out in this thesis work:

- i. The SCBA generated from the burning of sugar cane bagasse which is an agricultural waste can be utilized in basic mixture, used for the manufacture of ceramic wall tiles by adding it as a partial replacement with quartz. In other words, it is possible to obtain quality wall tiles product that fit the standards similar to those conventionally produced.
- ii. The laboratory data showed that the linear shrinkage and water absorption generally decreased with increased sintering temperature. Such result was expected because of the densification of the tile bodies and due to the more percentage presence of fluxing compounds within the waste.
- iii. This agro-industry waste (SCBA) can be used as a product with higher value added with respect to environmental and economic issues. It is therefore an attractive alternative and renewable source of ceramic raw material as realized in this investigation.

- iv. Beyond reducing the environmental impact of deposition of waste, it benefits the ceramic factories in lowering the cost of production of wall tiles by reducing the energy consumption of: quarrying from the earth surface, transportation and during grinding as quartz is the harder material in nature compared to other raw materials.
- v. It's possible to get good product quality at lower firing temperature due to the fact that SCBA contains higher fluxing compounds (K_2O , Na_2O and MgO) than quartz. Thus it is feasible to replace quartz by SCBA with 35%, firing at $1100^{\circ}C$ and pressing at 17MPa and get better quality parameters (water absorption, shrinkage, mechanical strength (MOR) and crazing.
- vi. According to this study for ceramic factory operating with capacity of 100 tonnes (Tabor ceramic for example) raw materials among which 5% is silica sand/quartz, it is possible to reduce about 1,750kg SCBA disposal per day for 35% of silica sand/quartz is replaced by SCBA.
- vii. Similarly, 35% incorporation of SCBA enhances technological utilizations of the waste (production of ceramic wall tiles) and improves natural resource consumptions, where resource limitations are common.
- viii. Since SCBA reduces the worsen conditions of cracking due to quartz transformations the company's loss would be significantly minimized.

6. RECOMMENDATION

- i. It is recommended that sugarcane bagasse should be explored as a basic raw material for ceramic wall tile production. This recommendation is due to the fact that the SCBA used in the production of wall tiles in this investigation have shown to be appropriate for ceramic wall tiles production that are thermally and mechanically stable and exhibit good crazing test at specified percentage of replacement (35%).
- ii. It is the noble option that sugar factories where, the ash wastes production is going on should set up in utilization of the waste for the production of ceramic materials rather than just dumping as waste and/or using as fertilizer which negatively affect the physico-chemical characteristics of the soil that has been proved by many researchers.
- iii. The reuse approach for ceramic wall tiles is environmentally attractive and can contribute to the provision of low cost raw material, conservation of natural resources and the management of a pollutant waste.
- iv. The utilization of sugarcane bagasse ash for advanced applications should be subjected to further studies because of its natural property.
- v. The differential Thermal Analysis (DTA) and gravimetric (TG) thermal analysis of the formulations should be studied further in order to identify the temperature differences between samples and determine the weight gain or loss of a condensed phase due to gas release as function of temperature for studying decomposition reactions.

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9. APPEDICES

Table A1: Optimum value selected from ANOVA

Constraints						
Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	importance
Composition	is in range	20	50	1	1	3
Temperature	is in range	1000	1200	1	1	3
Pressure	is in range	15	19	1	1	3
Solutions						
Number	Composition	Temperature	Pressure	Water Abs.	Desirability	
1	44.06	1106.02	16.79	7.05923	1.000	
2	32.65	1097.18	16.51	7.04923	1.000	
3	29.16	1093.12	16.60	7.09936	1.000	
4	44.68	1099.15	17.27	7.59526	1.000	
5	33.38	1110.56	15.89	6.46549	1.000	
6	42.42	1116.78	18.68	7.20034	1.000	
7	39.90	1107.90	17.70	7.06824	1.000	
8	42.56	1087.52	15.11	7.63193	1.000	
9	25.14	1110.45	15.24	6.52693	1.000	
10	25.54	1097.14	15.86	6.92769	1.000	
11	46.17	1120.79	17.36	6.58243	1.000	
12	23.09	1071.46	18.69	7.65076	1.000	
13	28.23	1087.22	16.33	7.34933	1.000	
14	20.48	1101.69	15.32	6.84039	1.000	
15	42.72	1113.52	18.14	7.12619	1.000	
16	32.26	1083.78	15.46	7.71492	1.000	
17	28.33	1071.65	16.52	7.98981	1.000	
18	46.32	1116.30	15.84	6.33259	1.000	
19	45.92	1117.05	18.30	7.23209	1.000	
20	43.81	1112.84	17.63	7.00223	1.000	
21	33.87	1086.53	15.72	7.59093	1.000	
22	38.41	1105.88	16.60	6.8372	1.000	
23	26.89	1075.95	16.28	7.78304	1.000	
24	27.81	1109.34	15.78	6.44484	1.000	
25	33.97	1099.18	18.41	7.31985	1.000	
26	20.13	1100.03	18.24	6.14782	1.000	
27	25.03	1083.54	15.98	7.46628	1.000	
28	<u>34.38</u>	<u>1115.66</u>	<u>17.10</u>	<u>6.26731</u>	<u>1.000</u>	<u>Selected</u>
29	34.83	1097.37	15.15	7.17684	1.000	
30	34.44	1110.05	15.45	6.54461	1.000	

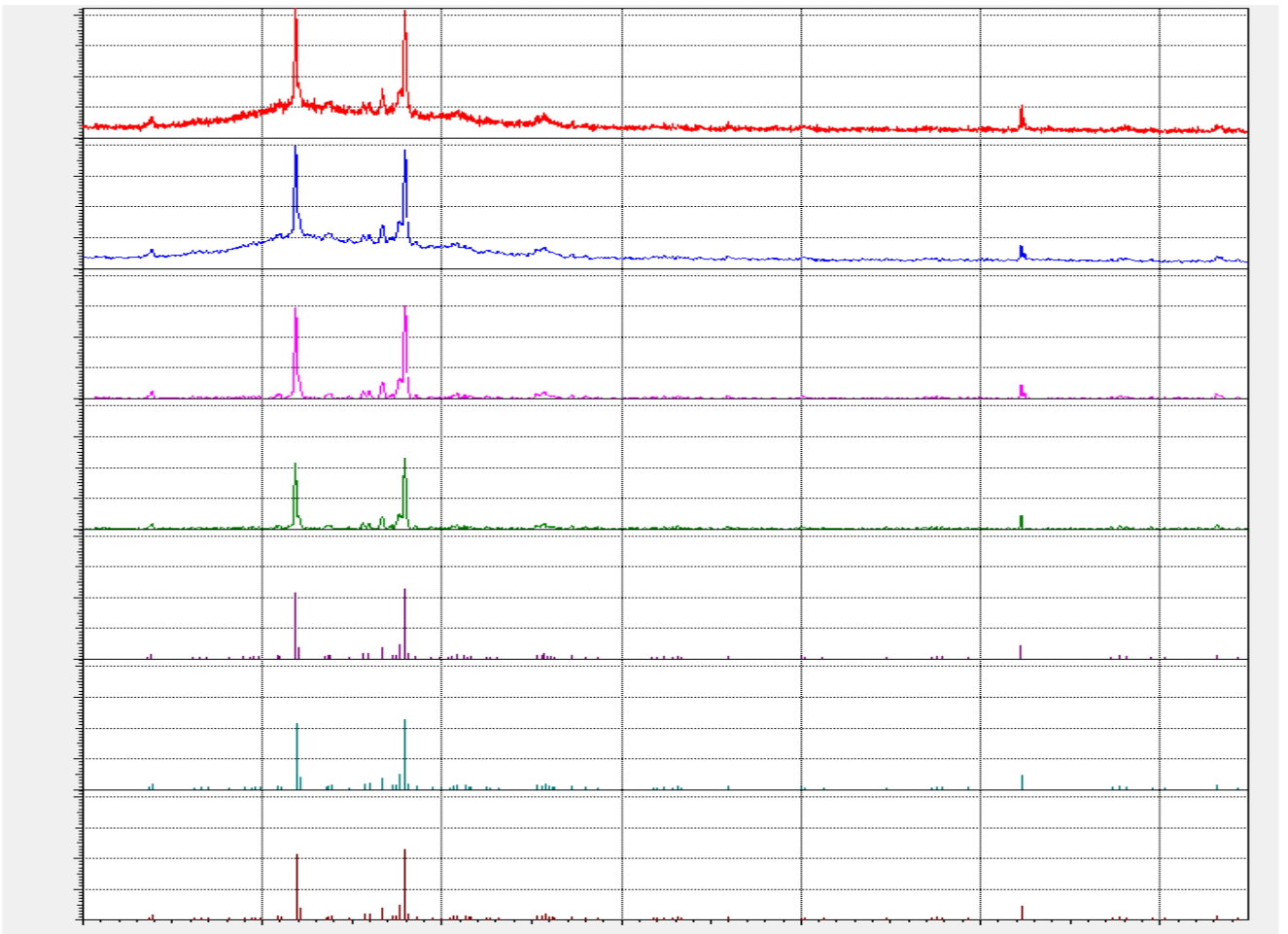


Figure A1: SCBA peacks analysed with standard data (defractometre data)

< Group: Standard Data: Quartz-ss >

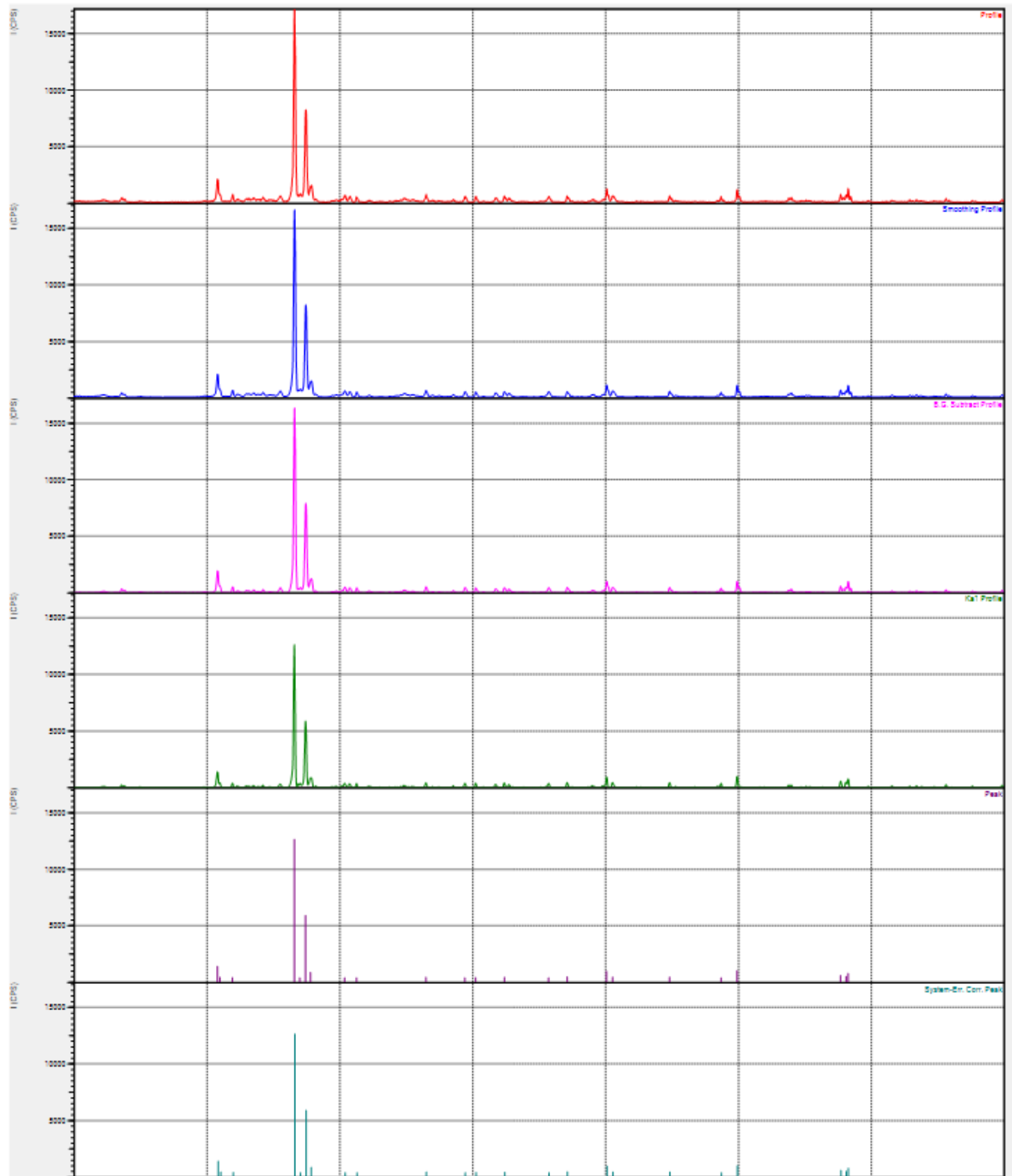


Figure A2: Quartz/silica sand peaks analysed with standard data (diffractometre data)