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**UTILIZATION OF RICE HUSK ASH (RHA)
INTO CERAMIC BRICK**

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A thesis submitted in fulfilment of the requirement for the degree
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DECLARATION

I am Aneis Maasyirah Binti Hamzah with matric number F15A0014, hereby declare that the work presented herein as original work done by me and has not been published or submitted elsewhere for the requirement of a degree programme. Any literature date or work done by other and cited within this thesis has given due acknowledgement and listed in the reference section.

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LIST OF ABBRIVIATION

RHA	=	Rice husk ash
SEM	=	Scanning electron microscope
XRD	=	X-ray diffraction
MOR	=	Modulus of rupture
wt. %	=	Weight percentage
EDS	=	Enaergy dispersive spectroscopy

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Abstract

Silica from rice husk ash (RHA) and clay were progressively involved in the production of whiteware material especially ceramic. The increases in the amount of paddy residue generated from time to time has lead to the introduction of rice husk ash uses in the manufacturing of ceramic brick as the alternative approach towards sustainable industry. The influence of the addition of RHA weight percentage as the silica precursor for the body formulation (0 wt. %, 20 wt. % and 40 wt. %) and also range in firing temperatures (900°C, 950°C and 1000°C) were used as the parameter to study their effect on the firing shrinkage, volumetric shrinkage, water absorption, apparent porosity and bulk density. It has been found that the replacement of quartz by RHA reduced the water absorption as well as the apparent porosity especially at maturing temperature of 950°C to 1000°C with 40 wt. % of RHA. The utilization of RHA as the ceramic brick give the value of percentage water absorption at 0.71%, apparent porosity at 0.86%, firing shrinkage at 8% to 10% and volumetric shrinkage at 0.8%.The finding would be helpful to improve the properties of ceramics as well as reducing the energy consumption during firing process. Apart from developing the green technology in production of ceramic, it also helps in decrease the amount of waste generated from the agricultural waste.

Key words: Rice Husk Ash, Ceramic, Brick

Abstrak

Penggunaan silika daripada abu serkam padi berserta tanah liat dilihat semakin mendapat tempat dikalangan pengkaji terutamanya dalam penghasilan seramik. Peningkatan jumlah sisa padi yang dihasilkan dari masa ke semasa telah membawa kepada pengenalan penggunaan abu serkam padi dalam pembuatan bata seramik sebagai pendekatan alternatif ke arah industri yang mapan. Kesan terhadap formulasi bahan (0% berat, 20% berat dan 40% berat) serta suhu pembakaran yang berbeza (900 °C, 950 °C dan 1000 °C) menjadi parameter utama dalam mengkaji kesannya terhadap peratusan pengecutan pembakaran, peratusan pengecutan isipadu, peratusan penyerapan air, peratusan liang nyata serta peratusan ketumpatan seramik yang terhasil. Didapati bahawa penggantian kuarza oleh RHA dapat mengurangkan penyerapan air serta pengurangan liang-liang yang ketara terutamanya pada suhu matang iaitu 950°C serta 1000°C dengan 40 wt. % RHA. Penggunaan RHA dalam menghasilkan bata seramik telah memberi nilai peratusan penyerapan air pada 0.71%, penyingkiran liang-liang pada 0.86%, peratusan pengecutan pembakaran pada 8% hingga 10% serta pengecutan isipadu pada 0.80%. Hasil kajian ini dapat membantu dalam usaha menambah baik kualiti seramik serta mengurangkan penggunaan tenaga semasa proses pembakaran disamping membangunkan teknologi hijau dalam industri penghasilan seramik sekaligus membantu dalam mengurangkan jumlah sisa buangan yang dijana daripada sisa pertanian.

Kata kunci: Abu Sekam Padi, Seramik, Bata

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Economic development of South East Asia including Malaysia is influenced by their agricultural sector which includes cultivation of parboiled rice. Due to this, rice has become the second largest produced cereal in the world as it gains significant attention as a source of foreign exchange earnings and also contributes to government revenue (Andreola *et al.*, 2012). Along the production of rice paddy, by-product known as rice husk is generated as a result of milling process. Rice husk is a thin layer coated surrounding the paddy grain in whereby it contains approximately 75 wt. % of organic volatile content. The rest 25 wt. % of the weight is able to be converted into ash during firing process. The product formed upon incineration process is known as rice husk ash (RHA) and it contain approximately 10 wt. % of unburned carbon (M.I. Martin *et al.*, 2012)

The presence of highly silica in rice husk ash gives additional advantage to be used in wide range of application in manufacturing processes. Currently, rice husk ash

has been used as a value added material in synthesizing of new material as well as low cost substitute material that modified the properties of existing products such as cement and concrete (Kumar *et al.*, 2012). Due to its previous performance, this study is purpose to utilize rice husk ash into ceramic brick. According to Zhang (1996), he stated about the correct replacement level of rice husk ash has gained the possibility to produce high strength brick due to high silica mineral content in rice husk ash. The addition of rice husk ash (RHA) helps to refine the structure of pore as well as its density that contribute to increase the strength of fired brick.

Generally, there are several types of solid waste been used in production of ceramics including rice straw, olive core, palm fibre and saw dust. However, RHA is seen to have great potential due to the ability of RHA to produce low porosity structure which helps to increase the ceramic body strength (Zulkefli *et al.*, 2017). The quality of brick is affected by raw material composition and firing temperature (Gorhan *et al.*, 2012). Mineral content in the raw rice husk ash especially silicon dioxide is responsible to give strenght to final ceramic. Firing provide energy for the bonding of brick particles through a process called as sintering which can be achieved under certain level of heat (Gorhan *et al.*, 2012). Therefore, this study aims to evaluate possibility of using Malaysia's RHA as raw material to fabricate ceramic brick.

1.2 Problem Statement

More paddy been produced to carter for people food industry, human population increase, thus rice husk which is the waste generated will increase as well. Considering the high amount of RHA to be disposed every year, we need to find a way to properly manage the dust. One of the possible ways is utilizing into new product. Rice husk that

has been burned with high temperature will result in formation of ash called rice husk ash (RHA). RHA had been widely used in an industrial application such as production of construction, structural clay products as well as refractory for steel industry. Its properties of high melting point and low thermal conductivity had caused this RHA constituent as an excellent insulator in the steel industry. In other application such as construction industry, RHA had been used in the production of low-cost concrete and cement which replace the depended on Portland cement. Addition of RHA into mixtures of cement had led to the formation of calcium silicate hydrate gel which responsible to increase the strength of cement and thus help to prevent cracking (Andreola *et al.*, 2012).

The potential ability of RHA and readily abundance of source has caused the implementation of this raw material as silica precursor in the production of ceramics. Previous research stated about the uses of ceramic in construction application (tiles, cement and concrete) as well as the advance application such ceramic filter for nanofiltration process in bioreactor (Shin, 2002) and also material in production of electronic appliance. Furthermore, there is also trend on previous research about the utilization of RHA into a ceramic brick. There are many factors which influence the properties of ceramic brick. However, to the best of our knowledge, factor such as body formulation and firing temperature highly affected the properties of ceramic brick.

Correct formulation of RHA give effect especially to the porosity and water absorption of the ceramic brick. The formulation is depends on it purpose of use. RHA can produce high porosity ceramic brick as well as low porosity depending on the percentage of rice husk used where it suitability is mainly depends on the chemical constituent. Since RHA is able to be utilized into ceramic brick for wide domestic and industrial purpose, the control factors are important to be considered in order to enhance the quality of the ceramic brick at the same time replace the dependent of other

supplementary material such as silica fume and fly ash as well as reduce energy consumption which is high in cost. Therefore, body formulation and its firing temperature are the main parameters subjected to be evaluated in this study.

1.3 Objectives of Study

The objectives of this study are:

- i) To evaluate suitability of rice husk ash (RHA) as ceramic brick.
- ii) To study the effects of body formulation and firing temperature to properties of ceramic brick.

1.4 Scopes of Study

This research will cover the study on the utilization of rice husk as into ceramic brick. The experiment conducted in University Malaysia Kelantan, Jeli Campus. The rice husk ash was supplied by Syarikat Beras Bernas Sdn. Bhd and the Clay was supplied from Kaolin (Malaysia) Sdn. Bhd. The rice husk ash (RHA) is characterized in term of crystalline phase present using XRD. This study covers about the effect of body formulation and firing temperature. For the body formulation, the ceramics is added with 0 wt. % RHA, 20 wt. % RHA and 49 wt. % RHA. For the firing temperature, the temperatures were set at 900°C, 950°C and 1000°C. The ceramic properties which are the firing shrinkage, porosity, water absorption and bulk density is set to be the benchmark of the parameters study and been compared with the Malaysian standard industrial commercial tiles since this comparison is compulsory in order to justify the suitability of tiles produced with the industrial requirements.

CHAPTER 2

LITERATURE REVIEW

2.1 Paddy and Its Rice Husk Waste

Paddy cultivation had leads to contribute in increasing of the global rice production since the past few decades. Generation of abundance of paddy as well as its residue leads to the alternative approach to reduce the cost of dumping at the same time contribute to the green technology approaches that not only focus on the industrial development but at the same time concern about the environment. Figure 2.1 shows the total amount of rice produced between the years 2009 until 2017 covering of global country. This followed with the total amount of rice husk generated as the result of milling process of rice which shown in Figure 2.2.

From the statistic, it can see that the amount of rice produced as well as rice husk generated is increasing from year to year. According to the statistic provided by Malaysian Ministry of Agriculture, about 450,00 metric tonnes of rice husk are produced in Malaysia each year. Starting from 130.9 million tons in 2009, the amount of rice husk produced tends to increase until 152.8 million tons in 2017. During the

milling process of paddy, about 78 wt. % of paddy turns into rice and bran while the rest of 22 wt. % of the paddy weight is left as rice husk.

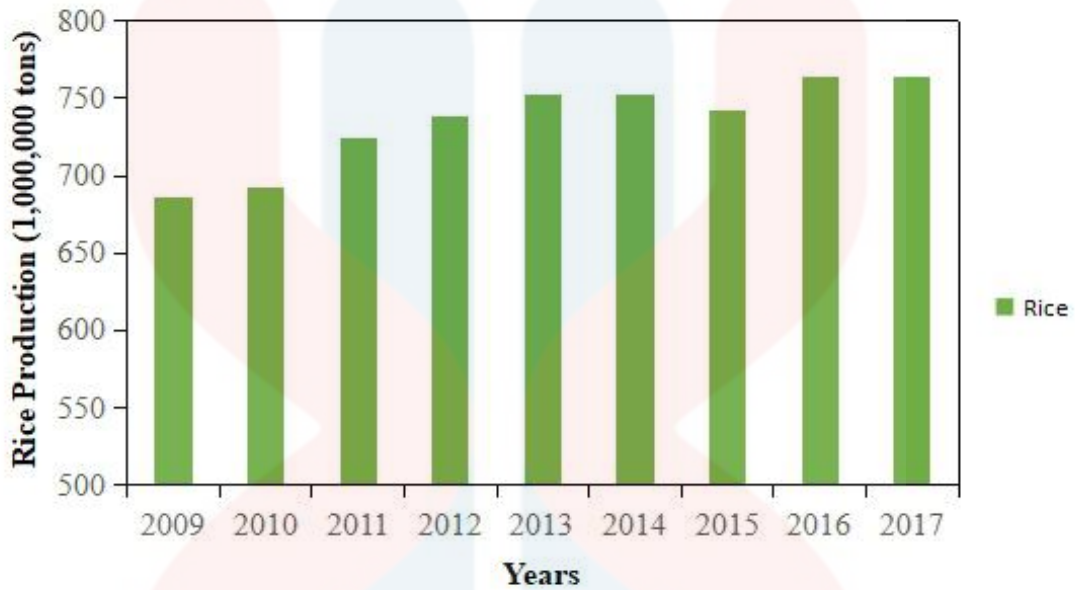


Figure 2.1: Global rice production generated from 2009-2017

Source: Food & Agriculture Organisation



Figure 2.2: Global rice husk generated from 2009-2017

Source: (Food & Agriculture Organisation)

This waste is massively produced especially in rice-producing countries including India, Bangladesh, Vietnam, Myanmar, Thailand and as well as Malaysia. This is due to the increase in world rice demand since rice is a staple food consumed by a large population of human all around the world. This classification of rice as main carbohydrate source leads in increasing paddy crops production to fulfill not only the local demand but also for the exportation purpose. Since hundred million tons of rice husks are produced annually, this then create the disposal problem In simple, this activity resulted in generating an abundance of rice husk that later can be used for other industrial purposes that will be further discussed in subsection 2.2.

2.2 Rice Husk Ash (RHA)

One of the alternative approaches in reducing the amount of rice husk accumulation is by converting this rice husk into ash that is believed to have a potential in enhancing the industrial sources. This ash produce during the incineration process contains around 80 wt. % to 90 wt. % of amorphous silica which has a wide range of applications in the industry (Ramchandra *et al.*, 2016). Due to these, several approaches have been made by the industry to return the waste into a beneficial material. Since the disposal of this ash leads to serious environmental issue and human health problem, an alternative is required to recycle it into a new product.

The current industrial research of rice husk (RH) application in industry is shown in Figure 2.3. With the advances of technology, many process developments have been proposed to recycle rice husk into new products.

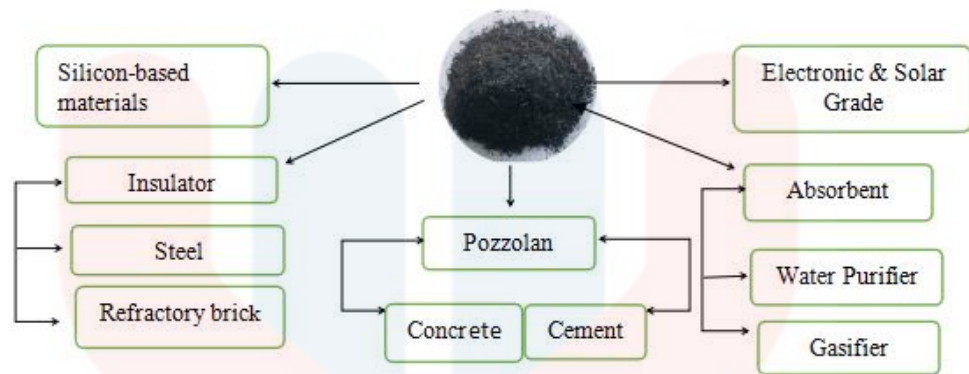


Figure 2.3: Schematic of application of rice husk ash in industry

According to Figure 2.3, the most application of rice husk can be utilized into the electronic and solar grade, adsorbent in water purification of wastewater, as silica gels to control the air quality and also in design and fabrication of construction material such as brick and insulator. By considering the main constituent of rice husk, its content of amorphous silica that formed as the result of the incineration process of rice husk at high temperature. It is expected to become a potential resource of a low-cost precursor in the production of silica-based material which provides additional value for commercial application.

Considering the importance of rice husk, a study is made to evaluate the potential and suitability of rice husk to be used in ceramic. There are various factors which influenced the ash properties including temperature, the rate of heating and the burning technique. Silica in the ash had been undergoing structural transformation depending on the combustion environment especially temperature and retention time (Kumar *et al.*, 2012).

2.2.1 Physical Properties

When dealing with material, it is important to recognize the characteristic of that particular material since this information can be used as the parameters to be studied.

Table 2.1 represents the general physical properties of RHA in term of colour, particle size, thermal conductivity and apparent porosity.

Table 2.1: General Physical Properties of Rice Husk Ash

Properties	Reference 1	Reference 2	Reference 3
	Mauro,2004	F. Andreola,2011	S.M. Tahir, 2017
Color	Black	Black & White	Black
Particle Size	fine	Fine	Fine
Conductivity	Low	Low	Low
Apparent Porosity	High	low	Low

According to Table 2.1, generally, there are two types of RHA including black and also white in colour. The formation of black ash is due calcinations of the potassium content in RH at high temperature and rate of heating. The tendency to form black ash increases with the increment of calcinations temperature and rate of heating applied. White ash is formed due to the RHA is treated with hydrochloric acid, (HCl) prior to calcinations. Treatment with HCl would oxidize carbon at low temperature (R. V. Krishnarao *et al.*, 2001). In term of thermal conductivity, RHA was found to be poor conductor while its porosity can be characterized quantitatively in which the increase of the porosity is depending on the amount of RHA been used.

2.2.2 Chemical Composition

Determination of chemical composition of the raw material is crucial since difference chemical composition will result in different properties. Table 2.2 shows the typical chemical properties of Rice Husk Ash reported by the various researchers.

Table 2.2: Chemical composition of rice husk ash

Oxide (wt.%)	Korotkova,2016	Matori,2009	Jenkin,1998
SiO₂	93.4	93.67	91.42
Al₂O₃	0.05	1.45	-
Fe₂O₃	0.06	0.47	-
CaO	0.31	1.30	3.21
MgO	0.35	0.57	0.01
K₂O	1.40	1.82	3.71
Na₂O	0.10	0.54	0.21
P₂O₅	0.80	-	-

The suitability of RHA as raw material for ceramic product is mainly dependent on its chemical composition. The chemical composition of rice husk with having the high amount of silicate has greatly influenced the ceramic properties. This is already approved by many research since by referring to table 2.2, silicate record the higher value of the chemical content in RHA followed by potassium oxide and alumina. In dense view, chemical composition of RHA will affect the strength of the ceramic product as well as its density and porosity.

2.2.3 Mineralogical Phase of Rice Husk Ash (RHA)

When RHA is utilized into ceramic, this RHA will pose several transformations on their natural phases which called as mineralogical phases of materials. This mineralogy of the RHA can be determined by using X-ray diffractometer (XRD). XRD is a device that used to evaluate the crystallography of RHA either this ceramic product having crystalline, amorphous or even mullite. Diagram 2.6 represents a sample of XRD pattern for RHA ceramic.

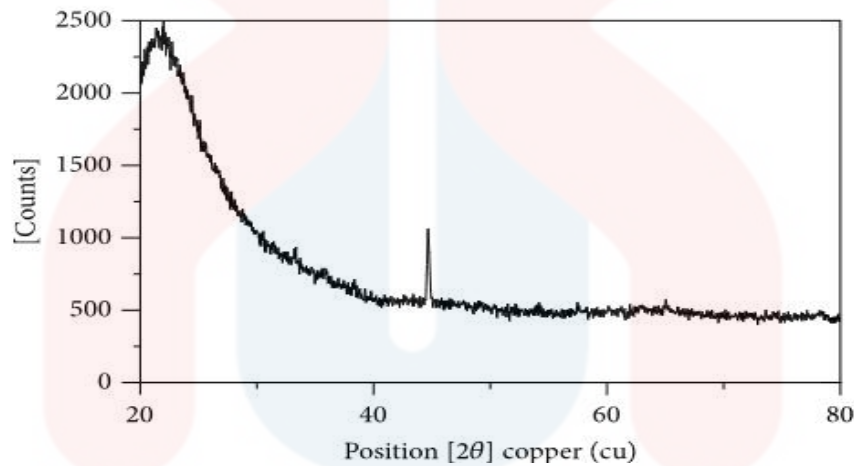


Figure 2.4: Typical XRD Pattern of RHA

Source: (S. M. Tahir,2017)

Figure 2.4 shows typical XRD pattern of RHA .From XRD pattern, it can be seen that RHA consists of major amorphous phase and lack of crystallinity. The present of amorphous phase is indicated by the presence of repeated hump peak while the crystalline phase can be determined by the sharp peak. This XRD pattern can be influenced by several factors such as the ration of rice husk ash and also the variation of sintering temperature. By relating to this research, the ceramic RHA is expected to have amorphous structure in which their characterization will be further discussed later.

2.3 Utilization of RHA into New Product

The application of greener technology in the industry has led to recycling idea of RHA highly potential to be utilized into other new product which provides the advantage to the industrial sector. Thus, the next subsection will review the utilization of RHA as an adsorbent and composite material.

2.3.1 Adsorbent

Rice husk ash is used as the adsorbent to treat the wastewater which contains heavy metal, dye and oil. This is due to the difference of polarity between the rice husk ash and the adsorbate leads to the attraction between these compounds resulting in the entrapment of the adsorbent on the surface of the rice husk ash (R. Lakshmi *et al.*, 2008). This process is influenced by several environmental conditions including concentration of adsorbate, temperature and also retention times of adsorption. Therefore, many researchers have deduced RHA could be regarded as the effective biomass adsorbent for waste water treatment.

2.3.2 Composite Material

Composite material is material that made by two or more constituent materials with having a significant difference of physical and chemical properties. Combination of rice husk ash as reinforcement filler of polypropylene matrix leads to formation of polymer composite (PMC). The utilization of rice husk ash is an alternative to replace the usage of silica as the reinforcement filler for production of polypropylene composite. The typical rice husk been used are the combination of white rice husk ash with black rice husk ash and amorphous rice husk ash at 10 wt.% to 40 wt.%. Transformation of

the crystalline rice husk ash to amorphous rice husk ash resulted in composites with improved tensile strength even without the coupling agent (Z Ismail *et al.*, 1995). This is usually applied in industry for the production of panel body and furniture.

2.4 Conventional Ceramic Product

2.4.1 An Overview

Ceramics can be classified as inorganic solid, non-metallic and heat resistant that is basically made up of the compound from a metallic and non-metallic constituent. Traditionally, ceramics are made of clay-based that have been formed by heating at high temperature. There are several types of ceramics which have their own properties but generally share some characteristics such as hard but brittle and also good insulator that can withstand with high temperature (Micheal, 1996). The most important is ceramics are resistant to corrosion which make this criterion as the additional value for its product.

A) Body Formulation

Ceramics compose of many elements. However, the most basic elements involved is alumina, quartz and zirconium. Ceramics contain ionic or covalently bond depending on their chemical composition. Most common ceramics are highly crystalline structure with the three-dimensional unit that formed from the repetitive of a unit cell of material. Traditional ceramic is produced from naturally occurring raw material such as clay and silicate-based compound. Due to this, traditional ceramics are often being referred as silicate ceramics.

Generally, in order to produce the traditional ceramic, it is highly dependent on the clay, silica and feldspar. According to Dinsdale, 1986 the main constituent of clay mineral is known as kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$). Clay plays an important role in the development of ceramic as it is known as the body former in ceramic. When clay interacts with water, its mixture will exhibit unusual rheology and flow under pressure (T. O. Mason *et al.*, 2016). This mechanism allows the clay mineral to be shaped into the desired shape. Other than that, silica which is the body filler of ceramic is added to provide strength of the product shape and also help in maintaining the shape of ceramic during firing process by reducing shrinkage.

Silica (SiO_2) which is the oxide that functions as filler to control the thermal expansion of ceramic during the firing process. Feldspar which is the flux, will melt during firing as it reacts with the other constituent to form a glassy phase which is responsible for the strength of fired ceramic. In addition, feldspar can be said to have the ability in reducing the temperature required (Ryan & Radford, 1987). The example of feldspar is orthoclase ($\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$) and anorthite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$). The general process in the manufacturing of ceramic involves beneficiation, mixing, forming, firing and finishing (Paul *et al.*, 2000).

B) Beneficiation and Mixing

Beneficiation is a process that involves pre-treatment including concentrating and sizing to particulate size. This process is followed by mixing process of batch. This batch is calculated and weighed according to its appropriate amount and can be also regarded as body formulation. Formulation of ceramic involves the selection of raw materials that fulfil the desired characteristics of the ceramic. The raw materials are

selected based on its workability and other requirements such as significant fusibility and chemical purity (Paul *et al.*, 2000).

C) Forming

Forming is a process to design the desired product shape. The most common technique is by powder compaction. Compaction process involves application of pressure to eliminate porosity and achieve the specific shape. The mixed raw material will undergo the forming process by placing the mixture into a mould and put under the pressing equipment. In order to apply pressure operation, the feed material required to have a lower moisture content to easier the holding and compressing process. Excess water contact will affect its stability since water is functioning to cling together but also act as lubricant which allowing the slide past towards another (Paul *et al.*, 2000).

D) Firing and Finishing

Firing process is performing to diffuse, bind and compact the ceramic body by undergo gradual heating from room temperature, through a hot zone and then back to the normal room temperature. The high temperature will drive out water and allow diffusion of particle, holding them in place and promoting the formation of dense ceramic body. Firing process also helps to obtain the bonding of particles for strength and reduction of porosity which will affect its performance. The normal temperature range is from 650 °C until 1150 °C. Unlike other material which will be ruptured during the introduction of high temperature, ceramic stability is increase with the increase in temperature applied until it reaches its maximum limit. Firing which provides heat

energy to the material system help in expanding the bonding between constituent particle until it forms a complete strong bonding and reducing the porosity between the particles thus increasing its stability. Lastly, the cooling process and product finishing to test the performance of the final product compatible with the specific standard.

2.4.2 Types of Conventional Ceramic

A) Porcelain Ceramic

Porcelain is the ceramic products that been fired at high temperature to achieved vitreous or glassy qualities such translucence and low porosity ceramic. These ceramics are generally consist of silica (SiO_2) and alumina (Al_2O_3). The natural ingredients of these components are quartz, feldspar, soapstone and clay.

Depending on the proportion of the silica and alumina as the material composition, two types of ceramics porcelain can be produce which is quartz porcelain and alumina porcelain. By comparing these two porcelains, alumina porcelain seem to have higher amount of more expensive alumina and processed at higher mechanical strength than silica porcelain (Dmitri, 2012).

B) Clay Floor Tile

Clay floor tile is made by special blue clay that enable the ceramics to have thin and smooth surface. The addition of ground glass and pottery ware may be involved in which it will promotes the provision of hard and impervious floor tile. This process involves the mixing of these material until it reaches the homogenous condition

followed with the compaction to obtain the green body. This green body later will be fired about 1300 °C and glazed to give a good appearance (Varghese, 2009).

C) Bioreactor

Usually, ceramic membrane has been used in bioreactor. Goldsmith R. L. (2004) has reported the performance of ceramic bioreactor for low-flow system in waste water industry. The ceramic membrane bioreactor is able to perform efficiently by controlling the specific environment required. According to Hang Sik Shin (2002), he stated that ceramic membrane bioreactor has the advantage in providing perfect retention of biomass and more reliable effluent compared by using the traditional process due to ceramic membrane have much better membrane permeation.

2.4.3 Utilization of Rice Husk Ash into Conventional Ceramics

Application of rice husk ash in ceramic industry has gained the attention since the past few years. The special characteristics of rice husk ash which believed to give high quality ceramics leads to increase of its application due to the industrial demand. Rice husk ash plays an important role as it act as filler for the ceramic body. This filler later will help to increase the mechanical strength of the ceramics been produced. The high amorphous silicates contain in the ash help to increase the densification between the particles in a ceramic during sintering process thus increase the body strength.

Availability of rice husk ash has become one of the advantages to ensure the continuous supply of the raw material. Ceramic from rice husk ash is expected to have resistance toward the moisture content in which these properties will reduce the

possibility of material slide which will affect the strength of the product. Lastly, proper formulation of rice hush ash composition will help to increase the resistance of decomposition since rice natural rice husk has the ability to resist fungal decomposition.

2.4.4 Types of Industrial Product Derived from Rice Husk Ash

A) Cement

Cement from rice husk ash perform better in increase the strength and durability of building materials. Rice husk ash act as reactive mineral additive which become attention recommendation in almost international building codes (Kumar *et al.*, 2012). Faiziev (2003) has report about the research on rice husk ash and found that rice husk ash is content highly reactive pozzolan which suitable to replace the use of silica fume of other mixture in manufacturing of low cost concrete block.

B) Power Plant Fuel

Direct combustion and gasification process of rice husk leads to generate heat energy that can be used in small industry such as low capacity boiler and also useful as an alternative fuel for household energy (Kumar *et al.*, 2012). This also supported by Shabbir (2009) where rice husk is also been implemented as fuel for brick kilns and furnace. The availability of rice husk and economic factors had gained the consideration to effectively use of these sources as fuel for power generation. However there is some technical factor also need to be considered in order to ensure the energy generated is at efficient level while emit environment pollution at minimum level.

C) Steel

Rice husk ash was used as turndish powder in the production of high quality of flat steel. This turndish powder act as excellent insulator which serve low thermal conductivity with high melting point. The advantage of this material is it prevent rapid cooling of steel and uniform solidification in casting process. Furthermore, the uses of rice husk ash as coated for molten metal record to give excellent insulator performance which approved by various research related to rice husk and rice husk ash (Kumar *et al.*, 2012).

2.5 Utilization of Rice Husk Ash Into Ceramic Brick

Brick is a small unit of building material which often made from fired clay with addition of binding agent such as cement and water. The uses of brick in construction is very well known since long time ago due to its general properties which is high strength, durable and light weight. Basically brick have high heat capacity which during heat transfer it allow heat to keep inside for long time. Research from previous study has state about the effect of rice husk ash in the production of brick where found that common problem occur due to the inconsistency of composition ratio and lack of knowledge for raw resource which affect the quality of brick (Sutas *et al.*, 2012). Proper composition of rice husk ash and the firing temperature affect the strength of brick and also increase temperature reduction where importance in reduce energy consumption during the production. Thus, this research is made to study the utilization of rice husk ash as ceramic brick with improvement on the properties compared with conventional ceramic brick.

2.6 Advantages

RHA is found to be superior to other supplementary material such as fly ash and silica fume (S. Luyi *et al.*, 2001). During the thermal treatment of rice husk, organic compounds will decompose and separately change to H₂O, CO, CO₂ and other volatile compound and the rest are carbon and SiO₂. The incineration process of rice husk lead to the production of porous composite which can be controlled by changing the mould pressure, raw material size and the particular temperature (Akihiro Nakata *et al.*, 2006). This shows that the rice husk ash is able to be used in the manufacturing of new product including the production of ceramic product.

According to analysis done by Andreola *et al.*, (2013), he found that the ceramic physical characteristic and composition of raw material is nearly similar to the chemical composition in the rice husk ash. With highly content of silica precursor inside the rice husk ash leads to the introduction of low cost precursor in ceramic production. With the addition of rice husk ash, it is proved to increase the mechanical strength of the product. The abundance of these biomass resource help to ensure the continuous supply of this raw material. Apart from preventing of cracking product, rice husk ash also improves the thermal expansion of the ceramic even under high temperature.

2.7 Limitations

The potential limitation associated with the utilization of rice husk ash as ceramic brick is due to its inconsistency in porosity. The porosity of ceramic can be reduced by densification during the sintering process. Densification leads to expansion of ceramic

element which result in entrapment of air been removed. This leads to reduce the porosity as well as increasing the body strength. However, in certain cases addition of rice husk ash may lead to inconsistency in porosity of ceramics. This may happen due to the presence of other constituent or impurities in RHA which act as additional flux in the ceramic.

During sintering, the additional flux will melt leading to glassy phase present in the brick. Eventually, the excessive glassy phase will seal-up the surface of ceramic brick. This flux also can be the filler of the ceramic body which cause the air particle inside the ceramic cannot be removed thus increase its porosity. In order to overcome this situation, perfect tuning of body formulation and also firing temperature since both factors are important due correct body formulation and firing temperature will leads to reduce the possibility of defect on the ceramic product thus preserve its quality.

CHAPTER 3

METHODOLOGY

3.1 Raw Materials

The major raw materials used in this project are rice husk ash and also China clay. The rice husk ash was collected from Syarikat Beras Bernas Sdn. Bhd while the clay is supplied by Kaolin (Malaysia) Sdn.Bhd. Rice husk ash was burned at temperature 700 Rice husk ash is expected to be the source of silica precursor and will be mixed with the clay according to the formulation. The optimum percentage if this raw material is formulated in order to produce an optimum quality of ceramic. The properties of rice husk ash ceramic produced will be compared with the commercial ceramic tile.

3.2 Apparatus and Instrument

In the preparation of cramic brick from rice husk ash and sample analysis for this project,

several apparatus and instruments were required. This following apparatus and instruments are lists as following:

- a) Heidolph Mixer
- b) Weight Balance
- c) Electric Oven
- d) Compressor
- e) X-Ray Diffractometer
- f) Scanning Electron Microscopy
- g) Vacuum pump
- h) Dessicator
- i) Density meter

3.3 Process Flow Diagram

This experiment involve the combination of clay and rice husk ash with wet mixing process followed by the correct ratio of raw material and firing temperature. The final product of ceramic brick then will be proceed with further charaterization test including firing shrinkage, water absorption, apparent porosity, bulk density and also phase analysis. The simple process flow is shown in the figure below.

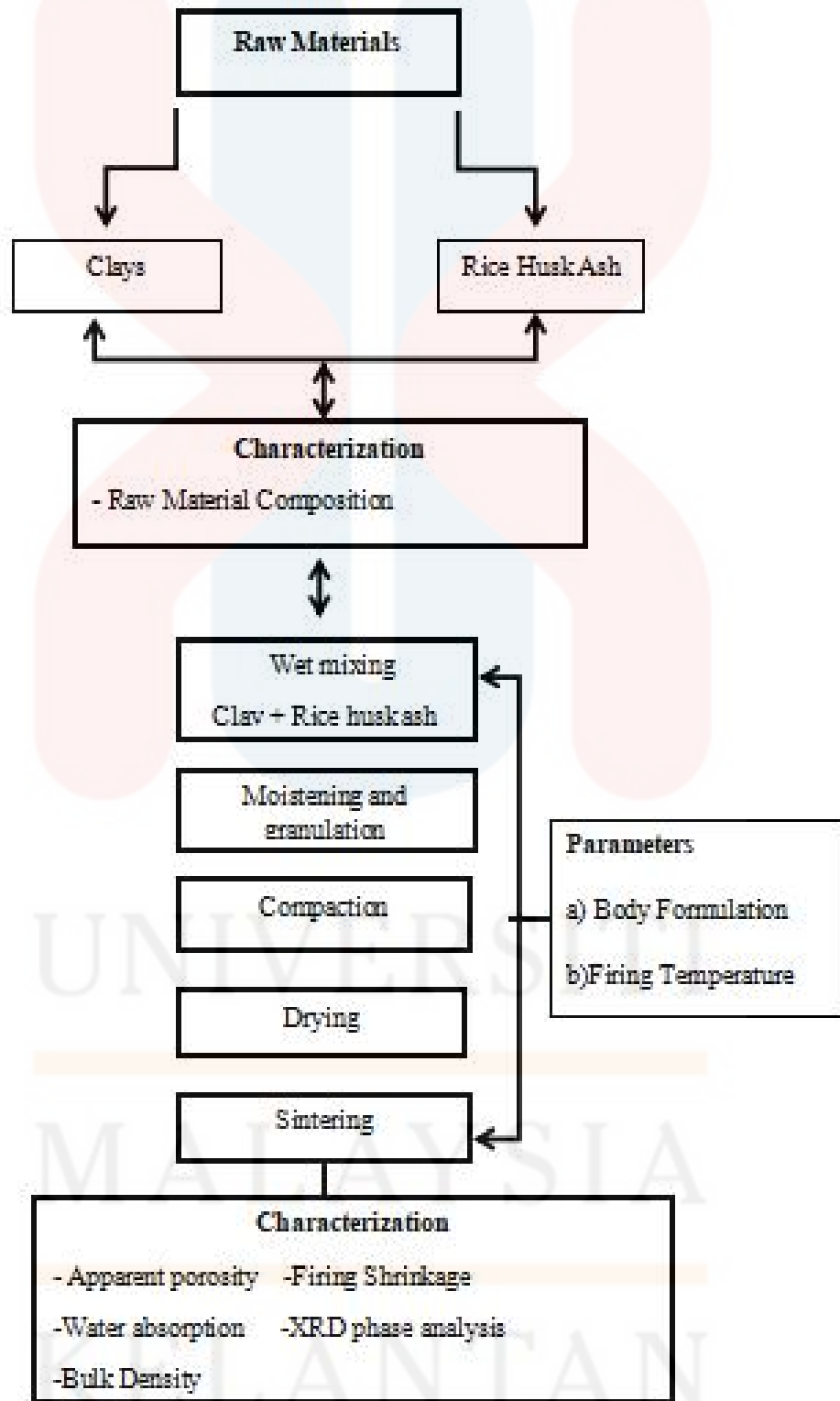


Figure 3.1 Process flow for utilization of RHA based ceramic brick

3.4 Preparation of Ceramic Body

Table 3.1: Body formulation of RHA and clay

RHA(wt. %)	CLAY(wt. %)
0	100
20	80
40	60

The raw material are firstly characterized in term of particles sizes and chemical composition. For the production of ceramic material, the specific amounts of raw material is prepared and mix inside a mixture for 30 minutes with addition of water to form a slurry solution. This slurry solution then been placed in a mould and let to dry inside an oven for overnight. After the drying process, the solid material was grinded by using mortar to obtain finely powder form. This followed with placing the powder inside a steel die for shaping purpose under pressing process. During the pressing process, the fine powder is compressed to bind all the powder and form a single compact material. This compact material is thermally heated with a different temperature at 900 °C, 950 °C and 1000 °C.

3.5 Characterization of Ceramic Brick Properties

Final product of ceramic brick is observe according to its property in term of firing shrinkage, water absorption, apparent porosity and bulk density. Method to measure these characteristic of ceramic brick is discussed in the following detail.

3.5. 1 Firing Shrinkage

Shrinkage is the condition of where the volume is change associated with water content in the particle. There are two type of shrinkage which is linear shrinkage and volumetric shrinkage. Linear shrinkage is defined as the one-dimensional decrease in particle mass, expressed as the percentage of the original dimension where the water content is produced from as given value to the shrinkage limit (ASTM D427). Linear shrinkage is calculated by using the following equation:

$$L_s = 1 - \frac{L_{avg}}{L_o} \times 100\%$$

Where,

L_s = linear shrinkage (%)

L_{avg} = Average length (mm)

L_o = original length (mm)

To calculate the volumetric shrinkage from a linear shrinkage test, uniform 3-dimensional shrinkage is assumed and the volumetric shrinkage is calculated as:

$$V_s = 1 - \frac{V_f}{V_o} \times 100\%$$

Where,

V_s = volumetric shrinkage (%)

V_f = final volume (mm)

$V_f = 0.5 L_f \pi r_f^2$

$$V_o = 0.5L_o\pi r^2$$

L_f = final length of fired body (mm)

L_o = original length of green body (mm)

r = final radius (mm)

3.5.2 Water Absorption, Apparent Porosity and Bulk Density

ISO 10545-3:1995(E) was used to determine water absorption, apparent porosity and bulk density. For water absorption, vacuum method is used instead of boiling method. The ceramic tile that has been weighed was placed vertically in the vacuum chamber (desiccators) at a pressure of 100 kPa for 30 minutes. After 30 minutes the mass of the suspended sample is determined by using Archimedes' method. The sample is immersed inside water at the same depth as it is used when the specimen is in place followed by drying with cotton cloth to remove all surface water. The saturated mass is then determined. In order to calculate this value, the following formulae are applied. In this calculation, 1 cm³ of water is assumed to be equivalent to 1 g of water.

$$E = \frac{M_2V - M_1}{M_1} \times 100\%$$

Where,

M_1 = mass of the dry sample

M_2 = mass of the wet sample

M_3 = mass of suspended sample

Apparent Porosity

The external volume, V expressed in cubic centimeters is calculated using the below equation:

$$V = \frac{M_2 - M_3}{\rho_w}$$

The volume of open pores, V_o and the volume of the impervious portion, V_i is expressed in the following equation:

$$V_i = \frac{M_2 - M_1}{\rho_w}$$

$$V_o = \frac{M_1 - M_3}{\rho_w}$$

The apparent porosity, P is expressed as a percentage in relation of the volume of the open pores of the sample to its exterior volume. The apparent porosity is calculated using this following equation:

$$P = \frac{M_1 - M_3}{M_2 - M_3} \times 100\%$$

Bulk Density

The bulk density, B , is expressed in gram per cubic centimeter of the sample is the quotient of its dry mass divided by the exterior volume including the pores. The bulk density is calculated using equation below:

$$B = \frac{M_1}{V}$$

3.5.3 Phase Analysis

Phase analysis is determined by using the X-ray Diffractometer. The scanning For the determination of ceramic body, XRD is used to evaluate the crystalline phases inside of the ceramic and also quantify the elements content inside it. Ceramics are made from the raw material such as clay and rice husk ash. These both raw material has a particular element composition as well as its arrangement. This will give each ceramic product have a different diffraction pattern which aslo can be called as the fingerprints of that material. The ratio of a specific element over others elements are depends on their specific application. So in this case, XRD are use to quantify of the elements.

Quantification of that particular element is performed by the identification of peaks for the element and the response of the signal to the element at different concentration. By plotting the concentration versus response, it will generates the curve called as calibration curve which later will be used to quantify the compound. For the crystalline phases, it can be determined by the interpretation of diffraction pattern that formed as a result for scattering of x-ray by the electron of atoms in solid crystal. The x-ray that been sent through the crystal will give a pattern of the atoms contained within the crystal are arranged. XRD is targeted to give results of quartz and mullite as both of this are generally recognized as the final crystal phases in the ceramic.

3.5.4 SEM and EDX Analysis

Scanning Electron Microscope (SEM) is used to investigate the microstructure and morphology of the ceramic material at an accelerated voltage 20kV. SEM help in provides physical properties of ceramic including the morphology, surface texture, material roughness and sometimes chemical composition of materials. In this study, SEM is use to observe the structure of ceramic specifically on the porosity of the ceramic. The limitation of other method such as by using optical microscope has restricted its application and thus SEM method is implemented. By using SEM the observation clear structure of ceramic can be reached such as either the structure is crystallite structure or amorphous.

Ceramic are assume to have crystalline structure in which it have relatively sharp and well defined pattern when been bombarded with x-ray while for amorphous it tends to have poorly defined pattern when expose to x-ray due to they are not arranged in a regular array. The physical structure such as porosity will support others studies such as the ceramic strength and also the water absorption. It is targeted to have less porosity thus will have low water absorption as these characteristics are important in producing ceramics for bioreactor application (Nanni, 2010).

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CHAPTER 4

RESULTS AND DISCUSSION

4.1. Characterization of Raw Material

The composition for raw material of ceramics is analyzed and shows that clay is mainly composed of aluminium oxide and silicon dioxide. The silicon dioxide is responsible to provide strength to the ceramics. For aluminium dioxide, it is responsible in changing the ceramic pigment after sintering process. The main composition of rice husk ash (RHA) is silicon dioxide. Figure 4.1 shows the two raw materials that have been used as the media make up of ceramics brick in this study while the major composition in weight percentage of both raw materials are shown in Table 4.1.

The raw material that used in this study are china clay and rice husk ash (RHA). This raw material was grinded to obtain fine powder texture. China clay has white in

colour due to the alumina content in its composition. Rice husk ash appears black in colour due to the presence of high silica content. The composition of the raw materials will further be discussed in the next part. China clay plays an important role as the binder and gives plasticity characteristic for the ceramic. When fired at high temperature (900°C to 1000°C) it will partially melt, resulting in tightening up the interaction between the bonds, thus causing the ceramic to have rock-like characteristics (A. Balasubramaniam, 2017).

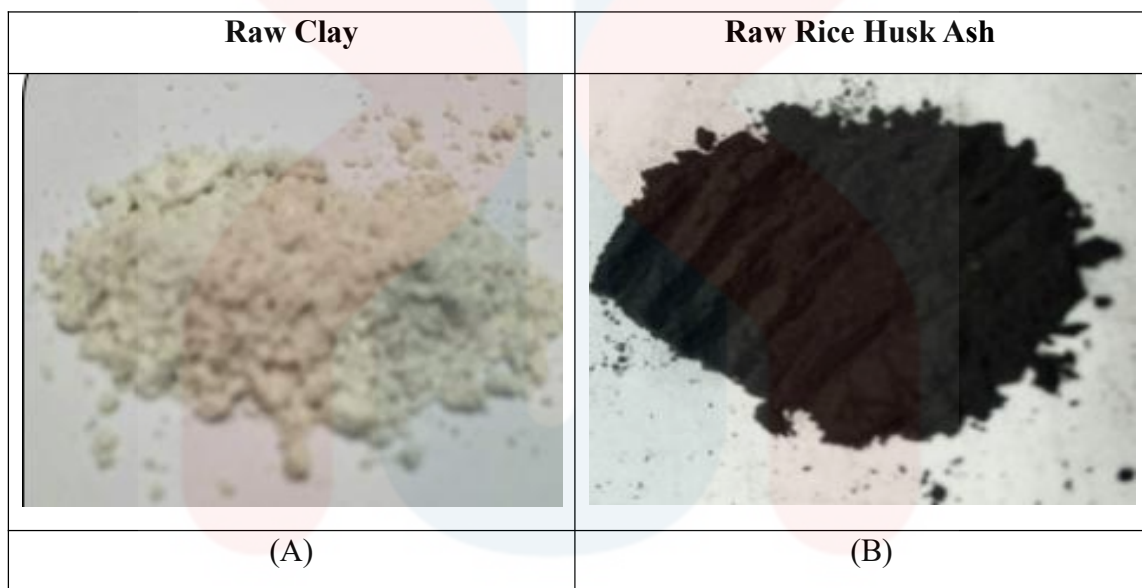


Figure 4.1: Raw materials of ceramics brick

RHA has the possibility to carry a function as filler and also flux. This depends on the compound that makes up the RHA itself. When it reacts as a filler, it will reduce the amount of binder needed in the production of ceramics. As it functions as a flux, this will help in reducing the melting point of the ceramic clay body. This thus causes the vitrification of the clay bodies to be increased since the conversion of the crystalline phases is reduced as well as its temperature needed.

Table 4.1 shows the quantitative data of compounds content in both raw material of clay and rice husk ash (RHA). As been mentioned in chapter 3, raw clay is supplied by Kaolin (Malaysia) Sdn. Bhd. and the raw rice husk ash from Syarikat Bernas. From this table, we can see that clay is highly content of silicon dioxide which is 57.63% followed by aluminium oxide which is 37.77%. Other compounds also found in clays including 1.82% of potassium oxide, 0.61% titanium oxide, 0.60% magnesium oxide and least amount of calcium dioxide which is 0.35%. Generally, silicon dioxide is functioning in give extra strength to the ceramics body while aluminium oxide is responsible in give the colour pigment of the final ceramic product.

Table 4.1: Chemical composition (weight %) of raw materials

Compound	Clay	Rice Husk Ash
SiO ₂	57.63	93.67
Al ₂ O ₃	37.77	1.45
Fe ₂ O ₃	-	0.47
CaO	0.35	1.30
MgO	0.60	0.57
K ₂ O	1.82	1.80
Na ₂ O	-	0.54
TiO	0.61	-
ZnO	-	0.1
CuO	-	0.1

Next, rice husk ash (RHA) shows to have silicon dioxide as the highest compound content in this raw material which is 93.67%. This is follow by 1.80% of potassium oxide, 1.45% aluminium dioxide, 1.30% calcium oxide, 0.54% sodium oxide, 0.47% of iron(III) oxide and small amount of 0.1% zinc oxide and copper oxide. Same

goes to clay, high silicon dioxide in RHA also help to provide strengthen to the ceramics body. High percentage of this compound in RHA compared to clay give a mean that RHA is having the ability to increase the ceramics product compared to using the clay alone as the raw material. Apart from giving strength, silicon dioxide also give the RHA to appears dark in colour. This is supported with the presence of iron(III) oxide, zinc and copper which is also black in colour.

Furthermore, in 2005, Bhowmick has reported about the mineralogical content of the Malaysian Kaolin. From the research, it stated that the kaolinite mineral obtained from Malaysian kaolin to have high amount of silica mineral which is 69.30 % and 24.3 % of alumina. However other researcher found that the kaolin constituents are compose of 56.29 % and 36.490 % of silica and alumina respectively (Aroke *et al.*, 2014). This data are more corresponding to the data obtained in table 4.1

4.1.1 X-Ray Diffraction (XRD) of Raw Materials

Figure 4.2 shows the XRD pattern of china clay. This graph is obtained by using the analysis software known as diffractive EVA. This raw material is observed at 2 theta counts range from 0 to 400 counts. The highest peak is at angle 13 degree with intensity of 378 counts.

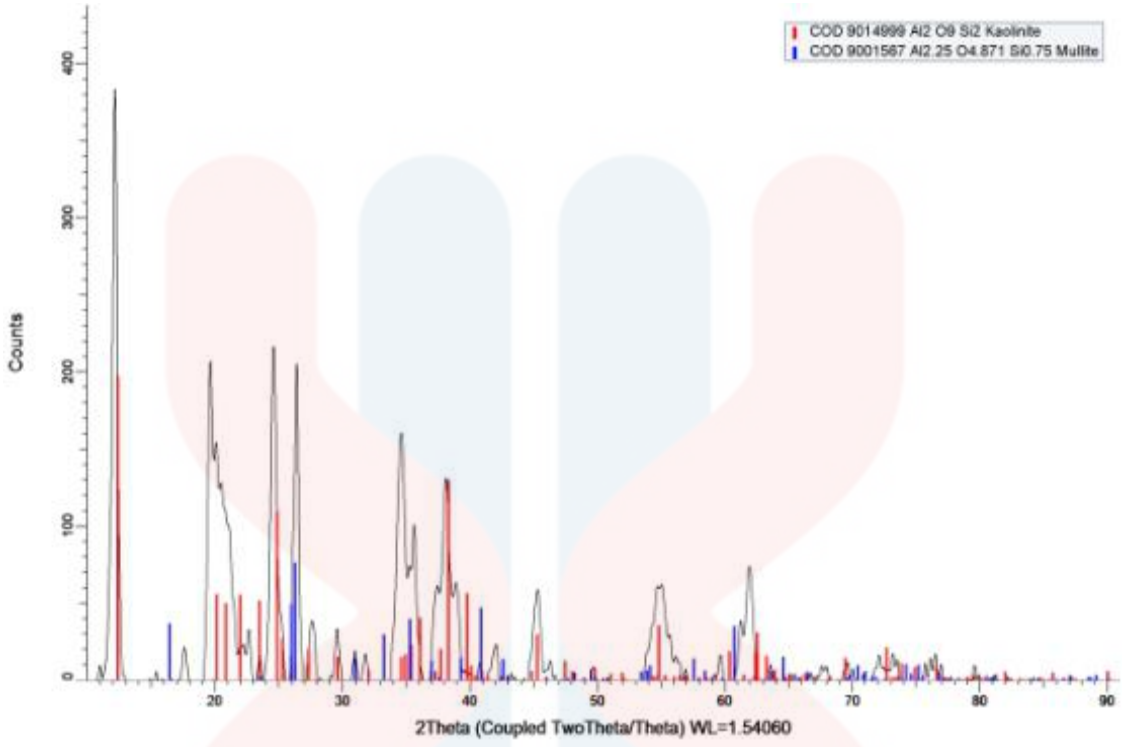


Figure 4.2 XRD pattern of china clay

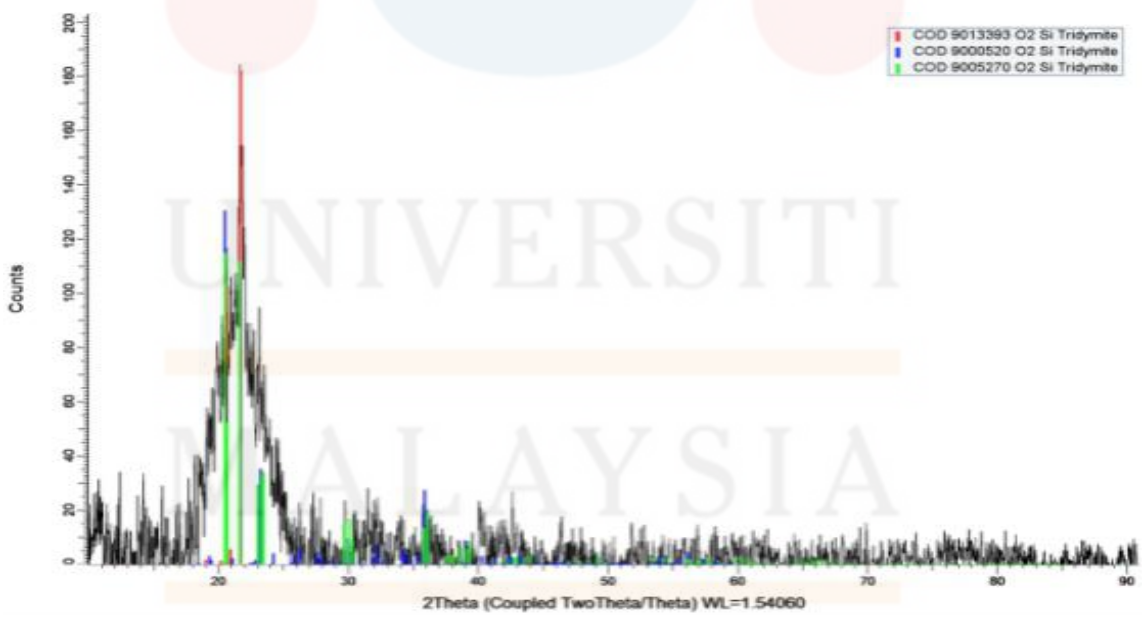


Figure 4.3 XRD pattern or rice husk ash (RHA)

According to Figure 4.2, it can say that china clay is having a crystalline structure. The crystallinity of china clay is 64.7%. Kaolinite mineral is detected in the china clay sample with reference COD 9014999. The natural crystal phase of china clay is quartz since it has high amount of silica and alumina content. This quartz will transform into other crystal phases depend on the temperature at of it being treated. In this study the raw clay indicate the small amount of mullite. Mullite is form as the process of combination of alumina and silica that fused together during sintering process.

Figure 4.3 shows the XRD pattern of raw RHA. RHA is amorphous at temperature below than 700°C. From this xrd patter, it shows that RHA content crystal structure known as tridymite. When the temperature is increased, it causes to the crystallization of the RHA from amorphous silica into cristobalite or trymidite depends on the purity of the raw material. The purity of silica is highly affected by chemical treatment compared to the thermal treatment (Jubair, 2011). Generally, the α -quartz is form at temperature less or equal to 573°C, β -quartz at 573°C to 870°C, cristobalite at 870°C to 1470°C and trymidite at 1470°C to 1710°C. (Jubair, 2011).

4.2 Physical Observation of Fired Brick

Physical feature of green body and fired ceramic brick is shown in the table 4.2 above. The difference of colour changes between the green body and final ceramic brick is discussed in the following section.

Table 4.2: Differences between green body with fired ceramic









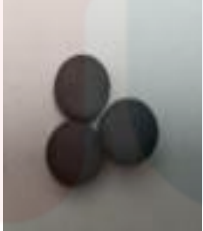



Weight Percentage of RHA (wt. %)	Before Firing	After firing at 900°C	After firing at 950°C	After firing at 1000°C
0				
20				
40				

Table 4.3: Colour change of ceramics (Willmann, 2003)

Ceramics	Additives	Colour
Alumina(Al_2O_3)	MgO	Ivory
	Cr_2O_3	Pink to ruby
	SiO_2	White
HA, hydroxyapatite	None	White
	Fe and Mn	Blue-wish
Zirconium(ZrO_2)	None	White
	Rare earth oxide	Yellow, Orange & Brown

Table 4.2 shows the physical comparison between the green body of the ceramics with after firing at temperature 900°C, 950°C and 1000°C. For all the three temperatures, the colour of 0 wt. % RHA before and after the sintering process is ivory white in colour. For 20 wt. % RHA, ceramics appear light grey but gradually turn into white after the sintering process. This change in colour also happen to the 40 wt. % RHA in which before the sintering process the samples appear dark grey colour however turns into white in colour after the sintering process at 24 hours. According to Willmann (2003), alumina turns white when it is sintered in reducing atmosphere or contains traces of silica (SiO₂). Since rice husk ash content of silica oxide, there is the possibility that silica is one of the factors that contribute to the change of colour after sintering process.

Table 4.3 below shows the changes of colour due to the presence of impurities or addition of mineral and oxide to the ceramics. From this table, it can say that ceramics that make up from oxide like silicon dioxide and magnesium oxide will appear white and ivory colour respectively. This was proven by the changes of colour in 100 wt. %c of clay from white to off-white or ivory colour. This happen due to the presence of magnesium oxide in clay is higher compared to RHA content, thus causing it to appear ivory like colour after sintering. For ceramic with addition of rice husk ash, it appears white in colour as the effect of silicon dioxide content make it to appear white after sintering. The changes of the ceramics colour differ from the green body is influenced by the transformation of crystalline structure as the effect of the temperature applied.

4.3 Firing Shrinkage

Shrinkage is the phenomena where the size of the final product is reduced from the original size due to certain causes. In this study, firing shrinkage is take into account in order to measure the reducing in size due to firing temperature that been applied. The removal of water and the additional addictive into the body formulation of ceramic will influence the percentage of shrinkage of the final product. For normal shrinkage, the volume will change linearly with the decrease of water content due to the surface tension forces exerted to the capillary moisture.

When discussing about the shrinkage effect, there are two type of shrinking that need to take into account. They are linear shrinkage and volumetric shrinkage. Linear shrinkage can be defined as the decrease in one-dimension in particle mass and expressed as the percentage of original dimension where the water content is reduced from a given value to the shrinkage limit (ASTM D427). Volumetric shrinkage is the decrease in volume of the sample particle where the water content is reduced from a given percentage to the shrinkage limit. The result from this two observation is discuss in the following part.

Table 4.4: Linear shrinkage and volumetric shrinkage for 4cm size

Temperature	Linear Shrinkage (%)			Volumetric Shrinkage		
	0 wt. % RHA	20 wt. % RHA	40 wt. % RHA	0 wt. % RHA	20 wt. % RHA	40 wt. % RHA
900	0	0.75	1.0	0	0.02	0.03
950	0.5	1.25	2.0	0.01	0.04	0.06
1000	2.0	2.25	2.25	0.06	0.07	0.07

Table 4.5: Linear shrinkage and volumetric shrinkage for 1cm size

Temperature	Linear Shrinkage (%)			Volumetric Shrinkage		
	0 wt. % RHA	20 wt. % RHA	40 wt. % RHA	0 wt. % RHA	20 wt. % RHA	40 wt. % RHA
900	5.3	8.0	8.0	0.79	0.80	0.81
950	7.0	8.0	8.0	0.80	0.81	0.81
1000	8.0	10.0	10.0	0.81	0.82	0.82

4.3.1 Linear Shrinkage

As been mentioned before, linear shrinkage is the one of the effect of firing temperature. When the temperature is increase, the percentage of the ceramic tends to shrink also increase due to more water is been removed from the fired body. At the same time the porosity is decrease. The addition of silica additive which is the RHA cause the percentage of linear shrinkage is increased too. This is because during the sintering process, energy will be provided to encourage the bonding between particle which called as densification in order to remove the porosity (MM.Halimawati, 2009). Furthermore, silica also act as the filler which filled up the fine pores presence in the ceramic body thus causing the body to become denser thus increase the body shrink.

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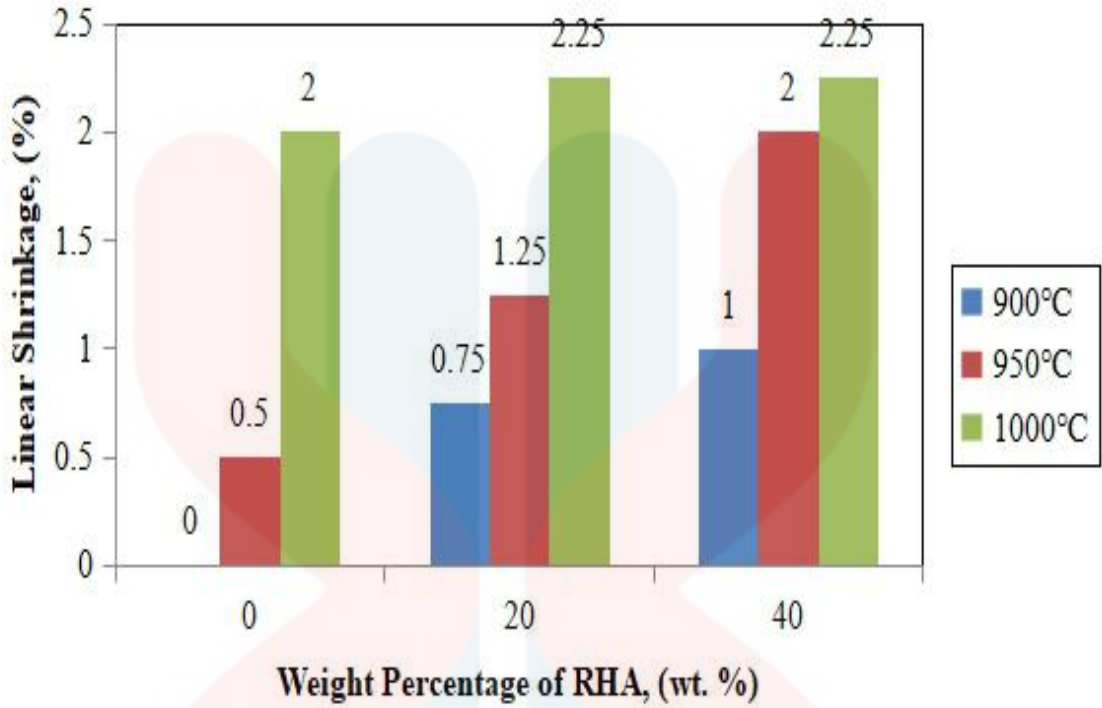


Figure 4.4: Graph linear shrinkage at 900°C, 950°C 1000 °C for 4 cm size

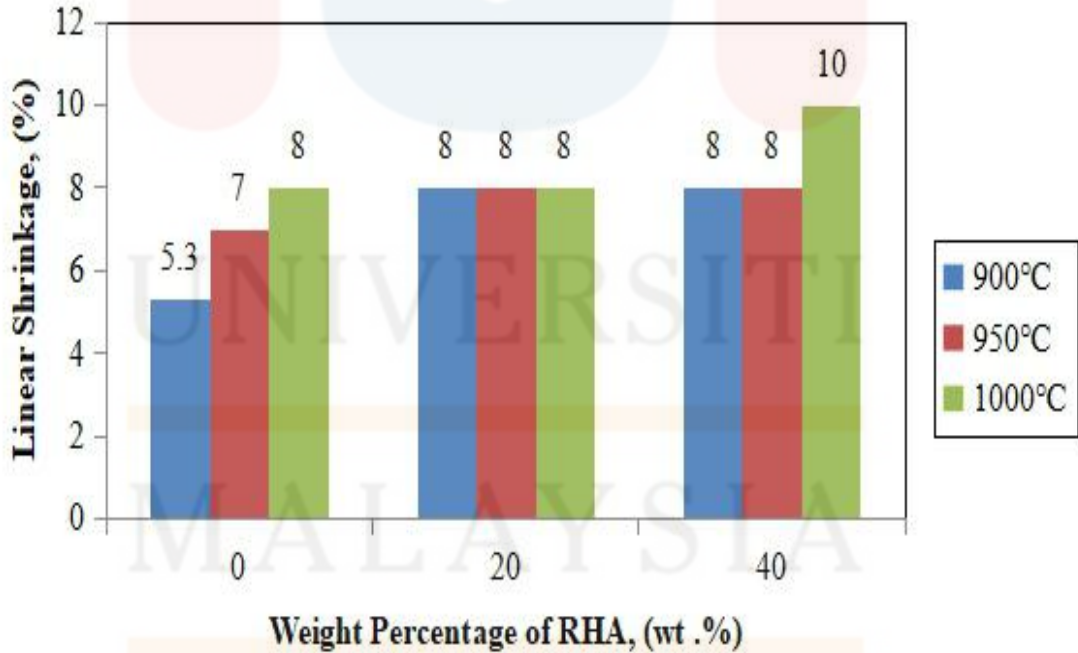


Figure 4.5: Graph linear shrinkage 900°C, 950°C 1000°C for 1 cm size

Figure 4.4 and Figure 4.5 shows the data regarding to the percentage of linear shrinkage between two difference size which is 1 cm and 4 cm size. From this graph, it can be explained that 4 cm sizes shrink about 3% compared to 10% of 1 cm size. This is because the effect of size and thickness of the green body. When temperature exerted on the small body even at 900°C, it will produce enough energy to hold the bond between them and thus caused the densification of the fired body which resulted in increase of the firing shrinkage.

This is explained from the graph where 900°C of 4cm have no shrinking effect while 1 cm ceramic size has about 5.3% of shrinkage. Furthermore, for 4 cm size it need 40 wt. % of RHA and minimum 950°C to reach about 2.25% of firing shrinkage. When looking to the 1 cm ceramic size, the percentage of firing shrinkage is constant from 0 wt. % at 900°C, all composition at 950°C and 0 wt. % and 20 wt. % RHA at 1000°C. However, the firing shrinkage increases to 10 % for 40 wt. % RHA at 1000°C. This may happen due to the conversion of crystal structure to other form such as from quartz to mullite, cristobalite or trydimite. This also happen due to inversion of one crystal form of silica such as from α to β (Cole, 1935). The increase in temperature at 1000°C help in improving the crystallinity and thus increase the densification and firing shrinkage.

4.3.2 Volumetric Shrinkage

Volumetric shrinkage is obtained by measuring the reducing in mass of the final ceramic from the green body. Figure 4.6 and Figure 4.7 shows the percentage of volumetric shrinkage of ceramic size 4 cm and 1 cm respectively.

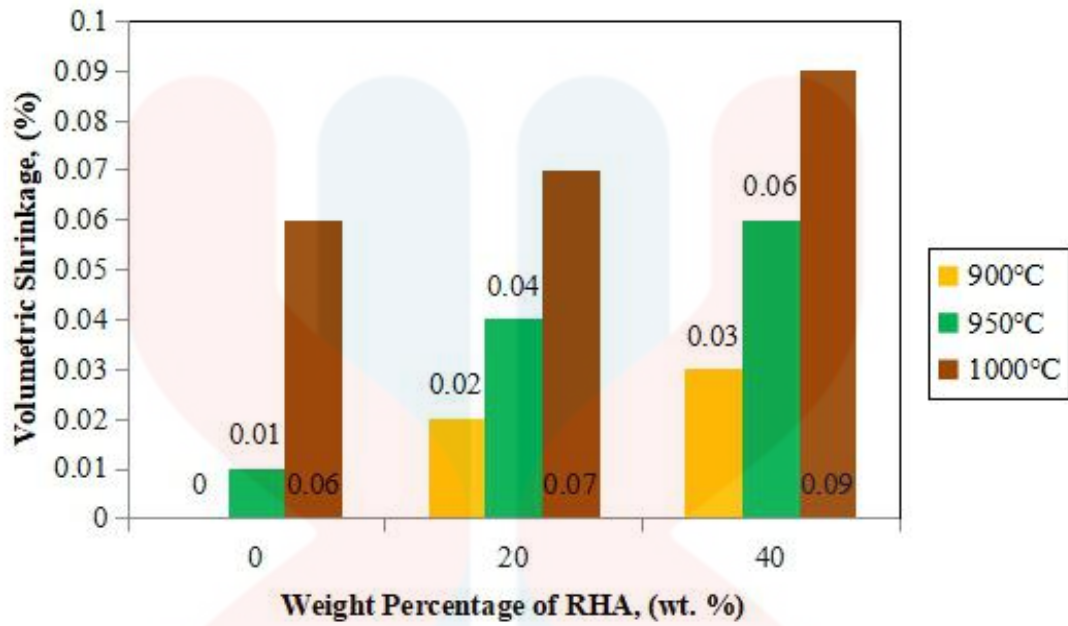


Figure 4.6: Graph volumetric shrinkage at 900°C, 950°C and 1000°C for 4 cm size

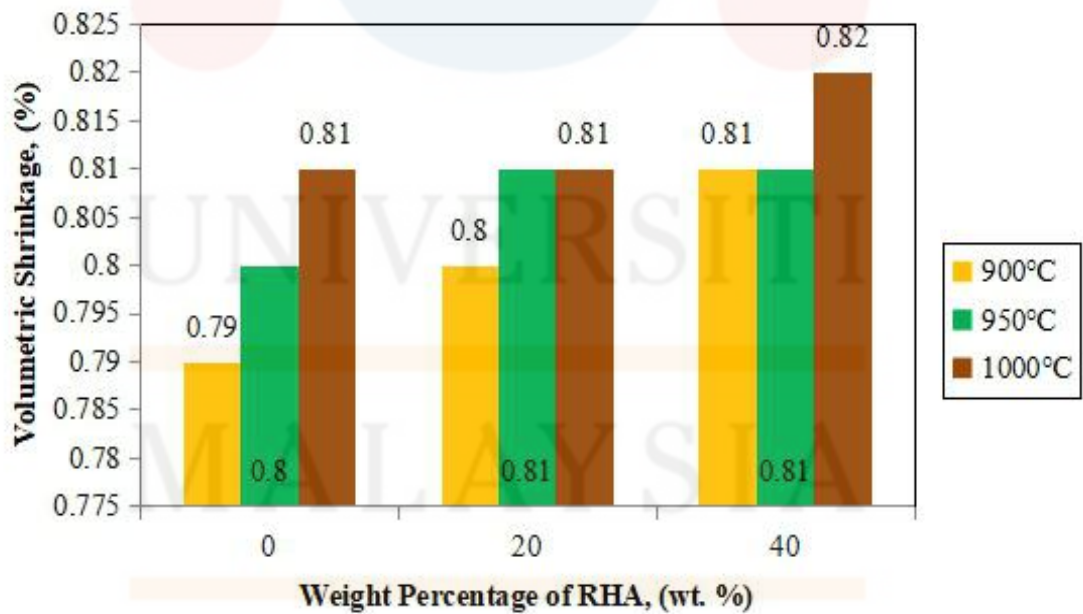


Figure 4.7: Graph volumetric shrinkage at 900°C, 950°C and 1000°C for 1 cm size

According to figure 4.5, ceramic at 900°C start to shrink when 20 wt. % of RHA is added and increase 0.01% when the silicate is added up to 40 wt.%. However, at 950°C the volume of ceramic start to shrink more early at 0 wt% of RHA with 0.02% and increase to 0.04 when 20 wt. % RHA is added and finally 0.06 when 40 wt.% RHA is added. At high temperature of 1000°C, the ceramics body is having high significant increases in volume reduction where the percentage of volume shrinkage are 0.06% up to 0.07% and finally 0.09%.

Differ with 4 cm diameter size, 1 cm size ceramic initiate volumetric shrinkage at 0 wt. % RHA when fired at 900°C. This reducing in volume is slightly increased when RHA is added. Ceramic at 1000°C is also obey this trend when it shows slightly increase in volumetric shrinkage especially at addition of 40 wt.% RHA . From this data, early conclusion can be made where the small size of ceramics tend to have high percentage of volumetric shrinkage compared to the big size due to certain factors including the moisture content in the ceramics as well as the gain size of the particle in the ceramics body.

4.3.3 Linear Shrinkage vs. Volumetric Shrinkage

When discussed about the shrinkage effect in ceramics, linear shrinkage and volumetric shrinkage are the common partner that take into consideration in order to measure the quality of the final product. The relation between linear shrinkage and volumetric shrinkage with its body formulation and the firing temperature is shown in the following section.

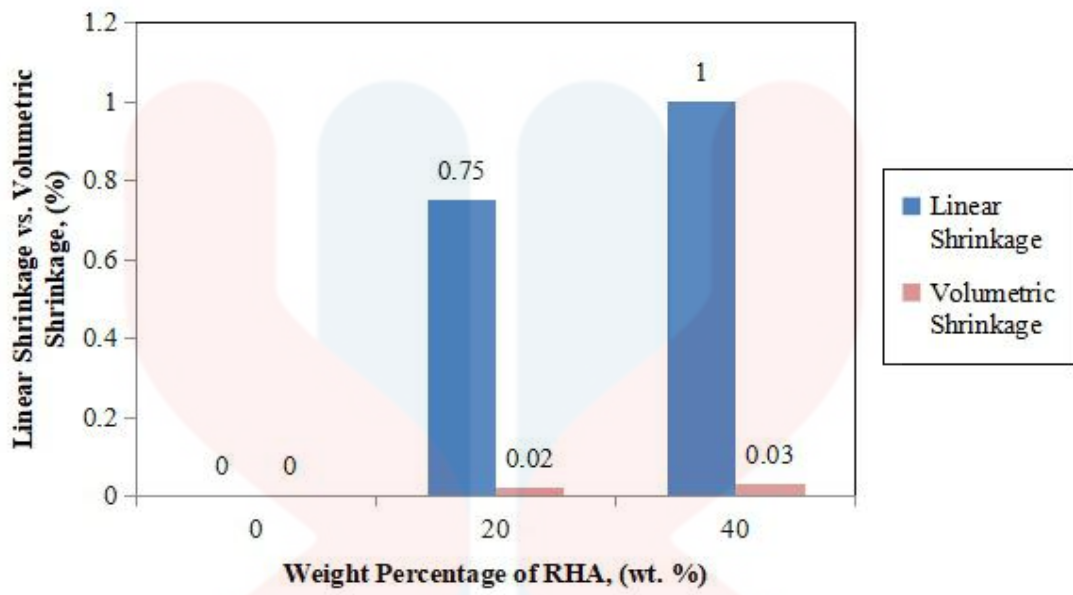


Figure 4.8: Graph linear shrinkage vs. volumetric shrinkage at 900°C for 4 cm size

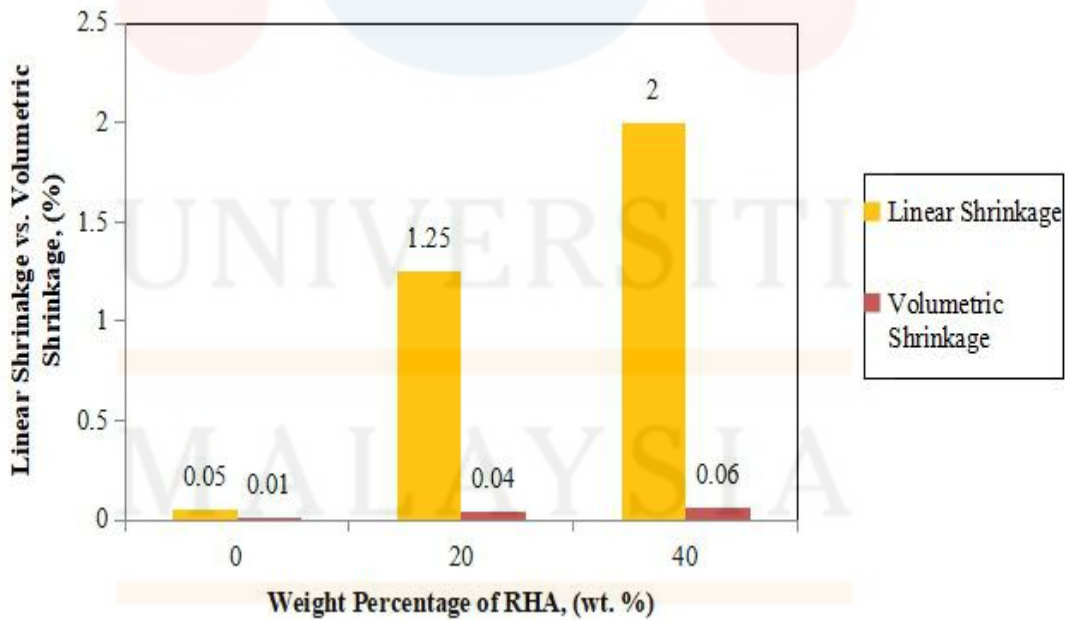


Figure 4.9: Graph linear shrinkage vs. volumetric shrinkage at 950°C for 4 cm size

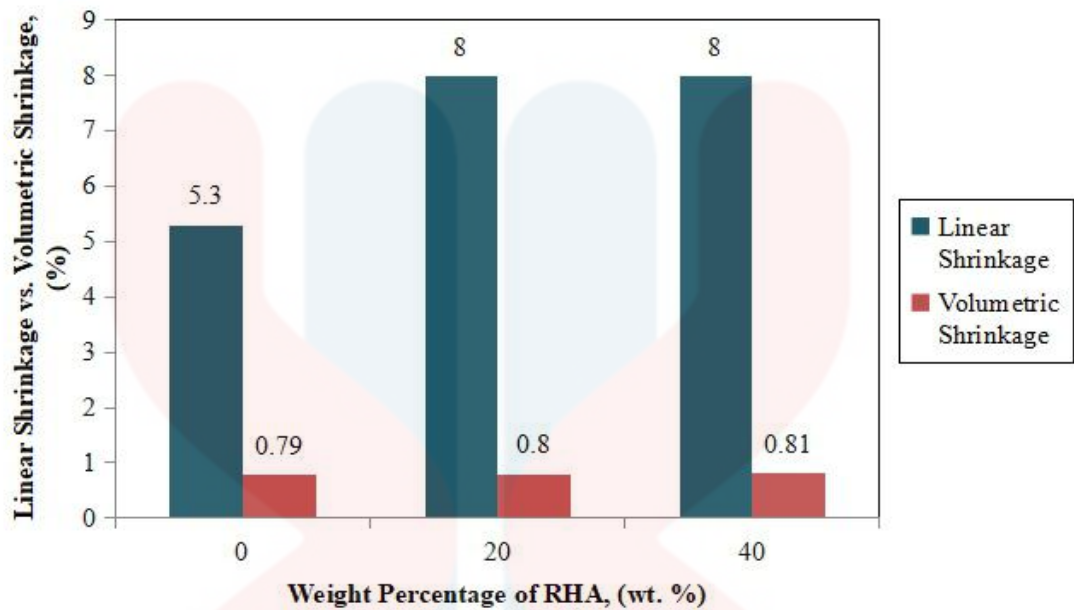


Figure 4.10: Graph linear shrinkage vs. volumetric shrinkage at 1000°C for 4cm size

Percentage of linear shrinkage is directly proportional to the volumetric shrinkage. This gives a mean that, when the volume of ceramic is reduced, the decreases in ceramic length from the original diameter also increase. The reduction in volume is much affected by the percentage of water been removed from the ceramic body after been fired at certain temperature. The highest percentage of liner shrinkage and volumetric shrink is dominated by 20 wt. % and 40 wt. % of RHA at 1000°C. The percentage of volumetric shrinkage versus linear shrinkage of ceramic size 1 cm is shows in the following data.

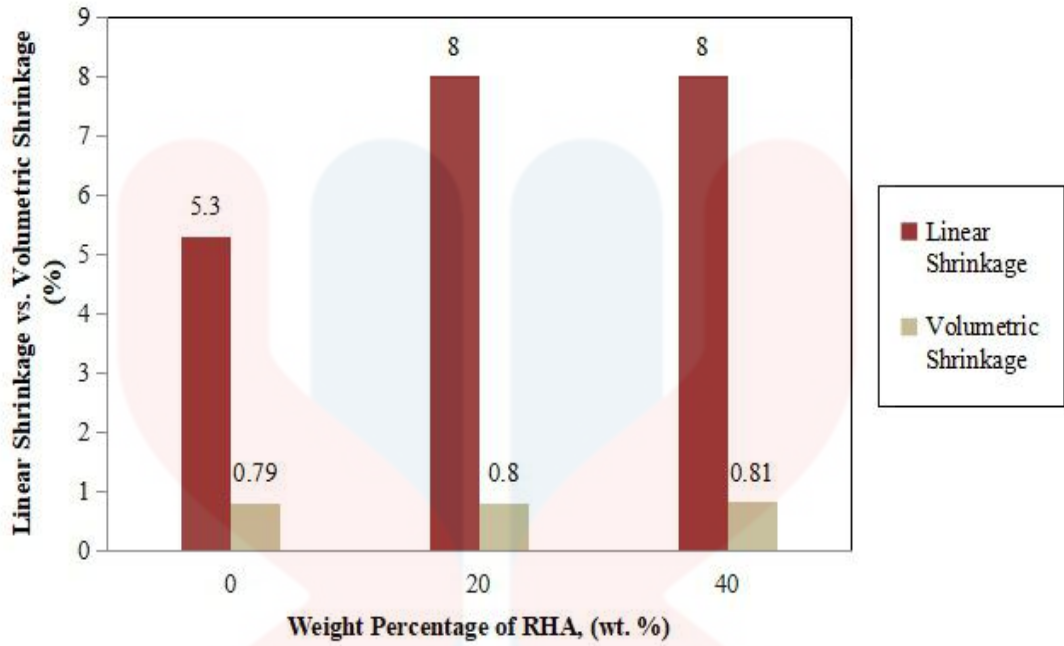


Figure 4.11: Graph linear shrinkage vs. volumetric shrinkage at 900°C for 1cm size

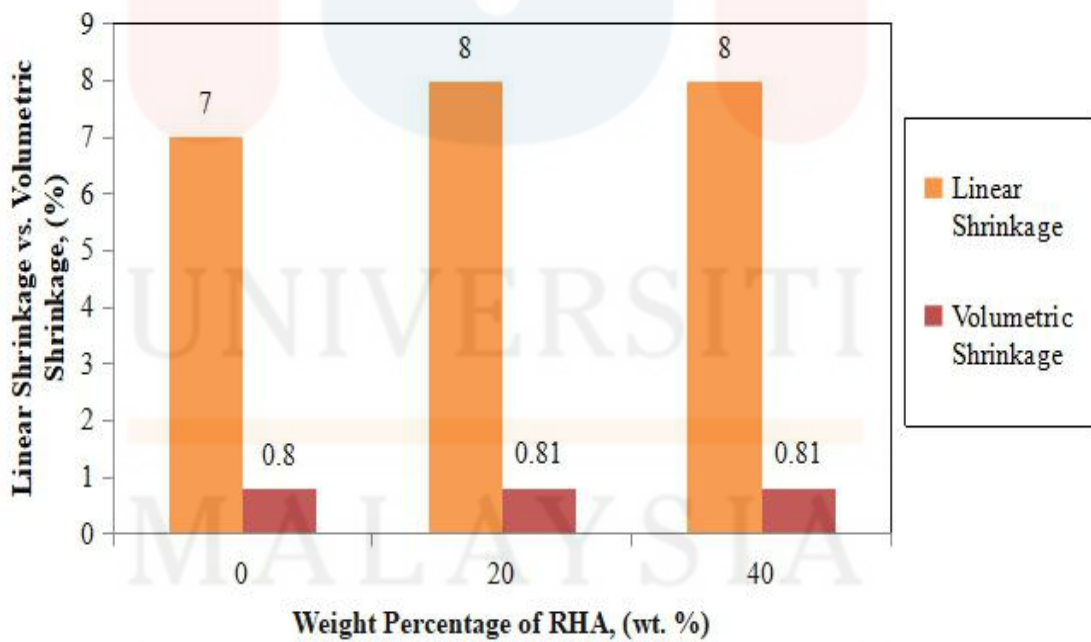


Figure 4.12: Graph linear shrinkage vs. volumetric shrinkage at 950°C for 1 cm size

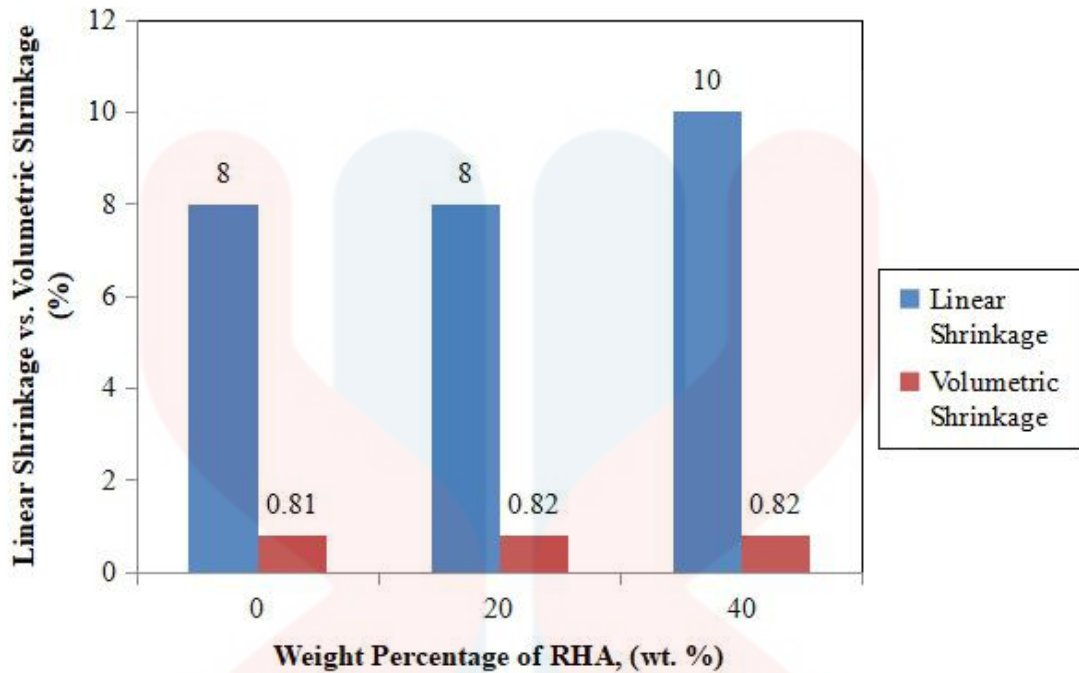


Figure 4.13: Graph linear shrinkage vs. volumetric shrinkage at 1000°C for 1cm size

From Figure 4.11, Figure 4.12 and Figure 4.13, it show the relationship between the effect of body formulation and firing temperature exerted on 1 cm of ceramic body to the percentage of linear shrinkage and volumetric shrinkage. The percentage of linear shrinkage is from the range 5% to 10% while for the volumetric shrink is 0.79% to 0.82%. There are many factors that affecting the shrinkage in ceramics. They include the flocculation in the moisture content where it responsible in the moisture expansion on the ceramic body.

In addition, drying process also plays an important role in affecting the shrinking in ceramics. Clay content which is the main ingredient in this study give significant impact on the ceramic shrinkage. Clay ceramic usually will have 14%-15% of firing shrinkage (Zamek, 2003). At low temperature 900°C, the silica content in the RHA does not affect significantly in the linear shrinkage. However, at temperature 1000°C, an increase of 20 wt. % and 40 wt. % of RHA content causes a notable increase

of the firing shrinkage which is 7% to 10%. This also contributes to the increase in volumetric shrinkage of the ceramic sample which later donating to the reduction in porosity ad the effect of densification process. The densification process is explain with the aid of diagram below.

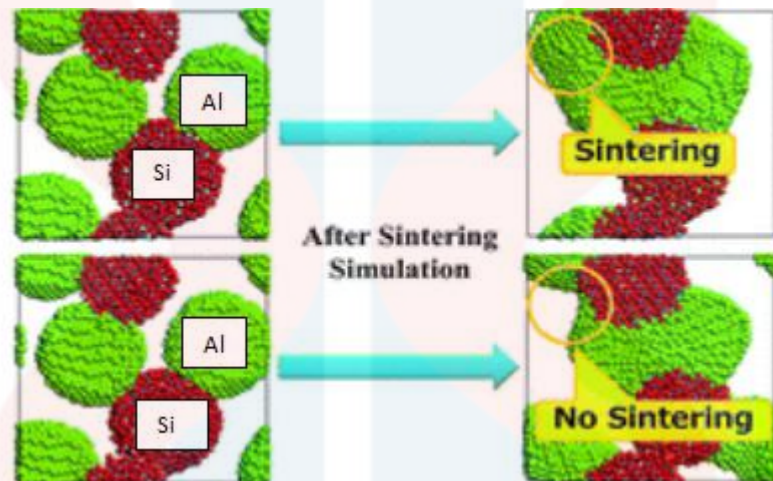


Figure 4.14: Effect of ceramic compound during sintering process

Source: J. Mater (2015)

Figure 4.14 shows the mechanism of sintering during firing process. Sintering is a process used to produce density-controlled material and component from metal or ceramic powder by applying the thermal energy. Sintering is influenced by a driving force which is the reduction of total interface energy. The reduction of total interface energy occurs via densification and grain growth. In the beginning of sintering process of ceramic, bridges form between the elemental crystallites. When the sample starts to shrink, it indicates that the residual porosity is now removed. This step is called as densification. Densification help to increase the body stability thus increase the strength of ceramic (Kang *et al.*, 2005).

4.4 Water Absorption, Apparent Porosity and Bulk Density

The effect on the addition of rice husk ash (RHA) into the clay mixture is shown in figure 4.4. The percentage of water absorption gives a result of negative growth trend in which the water absorption decreases with the increase of rice husk ash to clay ratio. This can relate with the porosity factor of the ceramic body. The increase of RHA portion has led to decrease its porosity. Furthermore, the addition of RHA at 20% by weight is more higher compared to RHA at 40% by weight but lower than the 0% by weight of RHA. This is supported by Mariana (2017) which stated that ceramic clay bodies or kaolinite ceramics commonly have 12% of water absorption performance due to the increase of porosity after being fired.

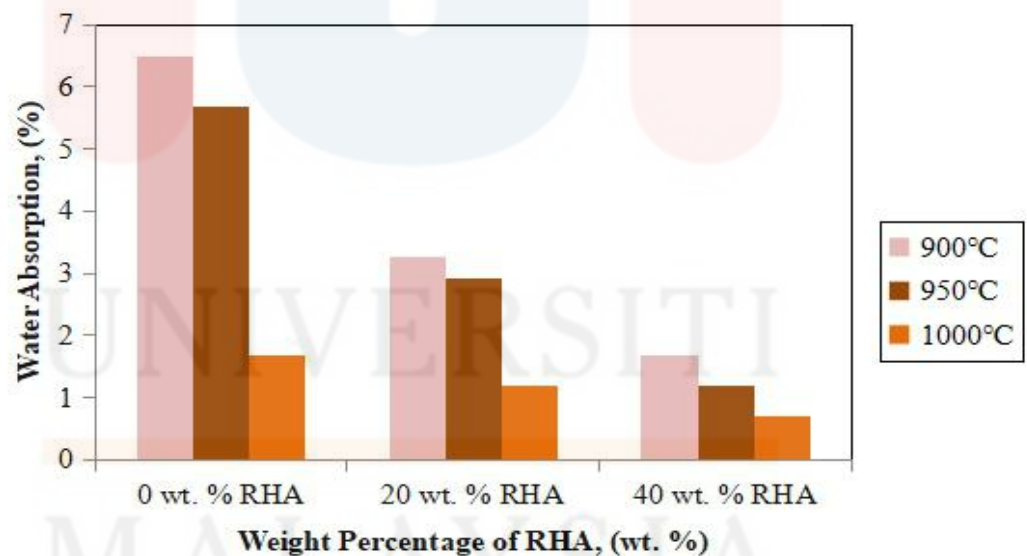


Figure 4.15: Graph of water absorption

Table 4.6: Data of water absorption

	0 wt. % RHA	20 wt. % RHA	40 wt. % RHA
900°C	6.48	3.26	1.69
950°C	5.68	2.92	1.18
1000°C	1.69	1.18	0.71

At firing temperature 900°C, the percentages of water absorption are 6.48%, 3.26% and 1.69% for 0 wt. % RHA, 20 wt. % RHA and 40 wt. % RHA respectively. For 950°C, the percentage of water absorption decrease to 5.68%, 2.92% and 1.18% for 0 wt. % RHA, 20 wt. % RHA and 40 wt. % RHA respectively. Firing temperature at 1000°C shows the lowest percentage of water absorption which is 1.69%, 1.18% and 0.71% for each of the RHA composition. This data shows positive trend on the reduction of water absorption as the portion of RHA been added is increased.

Hence a conclusion is made where the firing temperature at 1000°C with 40 wt. % of RHA is the best option to product ceramics with low percentage of water absorption. This is because lower water absorption is one of the important characteristic required in the production of high quality ceramics since improve the strength in ceramics body and thus increase it durability. The good ceramics quality also significantly affected by the porosity of its bodies which is discuss in the next part.

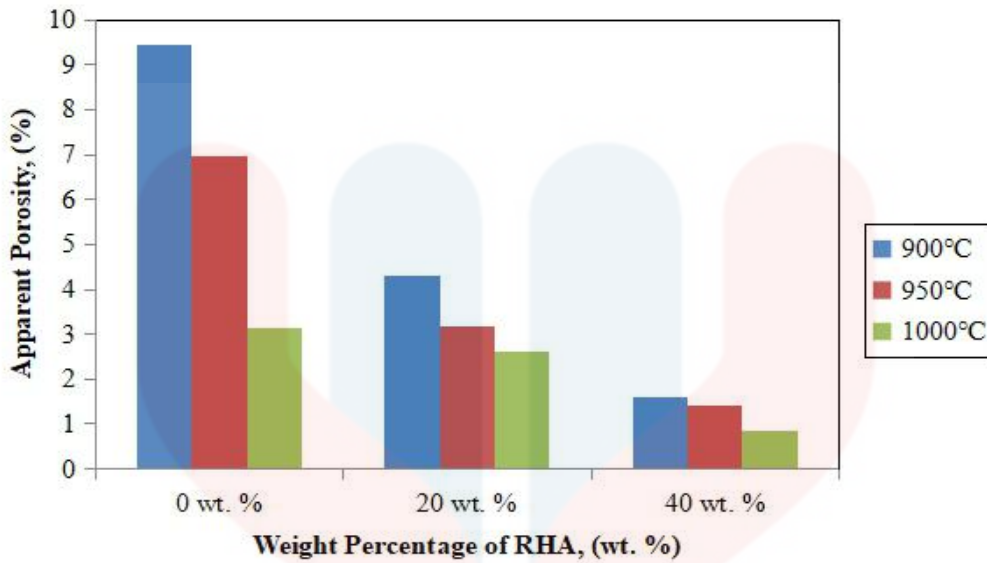


Figure 4.16: Graph of apparent porosity

Table 4.7: Data of apparent porosity

	0 wt. %	20 wt. %	40 wt. %
900°C	9.44	4.3	1.6
950°C	6.95	3.19	1.4
1000°C	3.14	2.62	0.86

The percentage of apparent porosity for sintered ceramics body is shown in the Table 4.7 above. According to the graph trend, it appears to have negative growth of the apparent porosity as the weight percentage of RHA is increased. At firing temperature 900°C, the apparent porosity of ceramics is 9.44%, 4.3% and 1.6%. For 950°C, the apparent porosity tends to decrease at 6.95%, 3.19% and 1.4%. . At firing temperature 1000°C, the apparent porosity continuously decrease and have 1.6%,1.4% and 0.86% for 0 wt. % RHA, 20 wt. % RHA and 40 wt. % RHA respectively.

The increase of temperature as well as the weight percentage of rice husk ash (RHA) has lead to decrease the apparent porosity of the ceramics body. RHA at 40 wt. % and 1000°C state to have the lowest value of apparent porosity. This may happen due to the pore space had been filled as the effect of densification process. Densification happens as a result of porosity removal thus makes the body dense. Silica content in RHA may react as the pores removing agent of the ceramics that increase it densification and reducing the apparent porosity when the firing temperature is increased. Therefore, it can say that the apparent porosity is decrease with the increase of firing temperature as well as the weight percentage of RHA. The bulk density which the other charactistic of ceramic is discuss in the Table 4.8.

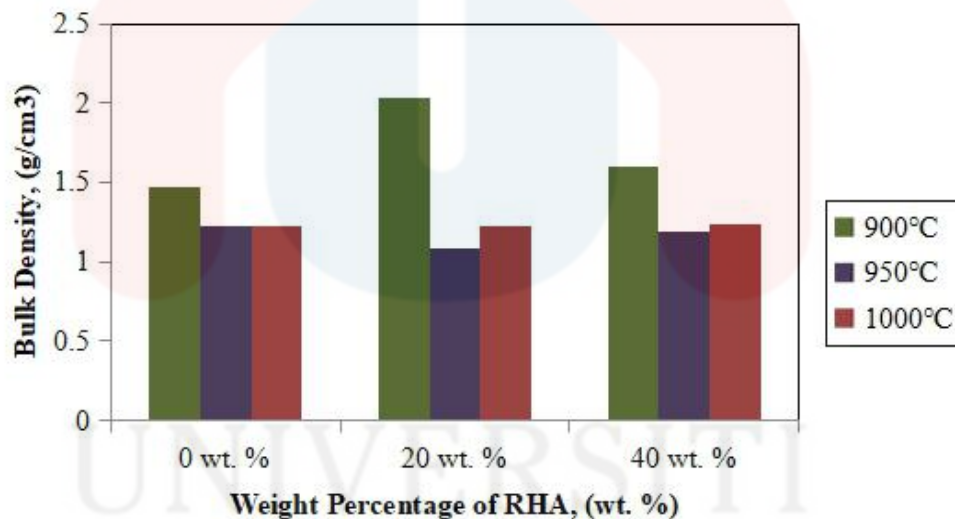


Figure 4.17: Graph of bulk density

Table 4.8 Data of bulk density

	0 wt. %	20 wt. %	40 wt. %
900°C	1.47	2.03	1.6
950°C	1.22	1.08	1.19
1000°C	1.22	1.23	1.24

At firing temperature 900°C, the bulk densities of the ceramics are 1.47%, 2.03% and 1.6% while firing temperature at 1000°C gives results of 1.22%, 1.23% and 1.24% for the bulk density percent. At firing temperature 950°C the bulk densities are 1.22%, 1.08% and 1.19% for 0 wt. % RHA, 20 wt. % RHA and 40 wt. % RHA respectively in which it is the lowest percentage of the bulk density. As the relation between the bulk density and the porosity are inversely proportional, the bulk density will decrease with the increase of the ceramics porosity (Andrew, 2013).

This is shown by comparing the 20 wt. % RHA and 40 wt. % RHA. Ceramics that composed of 20 wt. % of RHA shows to have low bulk density compared to 40 wt. % RHA for both 950°C and 1000°C of firing temperature. This can be related with the apparent porosity factor. When the apparent porosity is decrease, the densification of ceramic body is increase to make the body become dense thus increase it bulk density.

4.5 XRD Phase Analysis

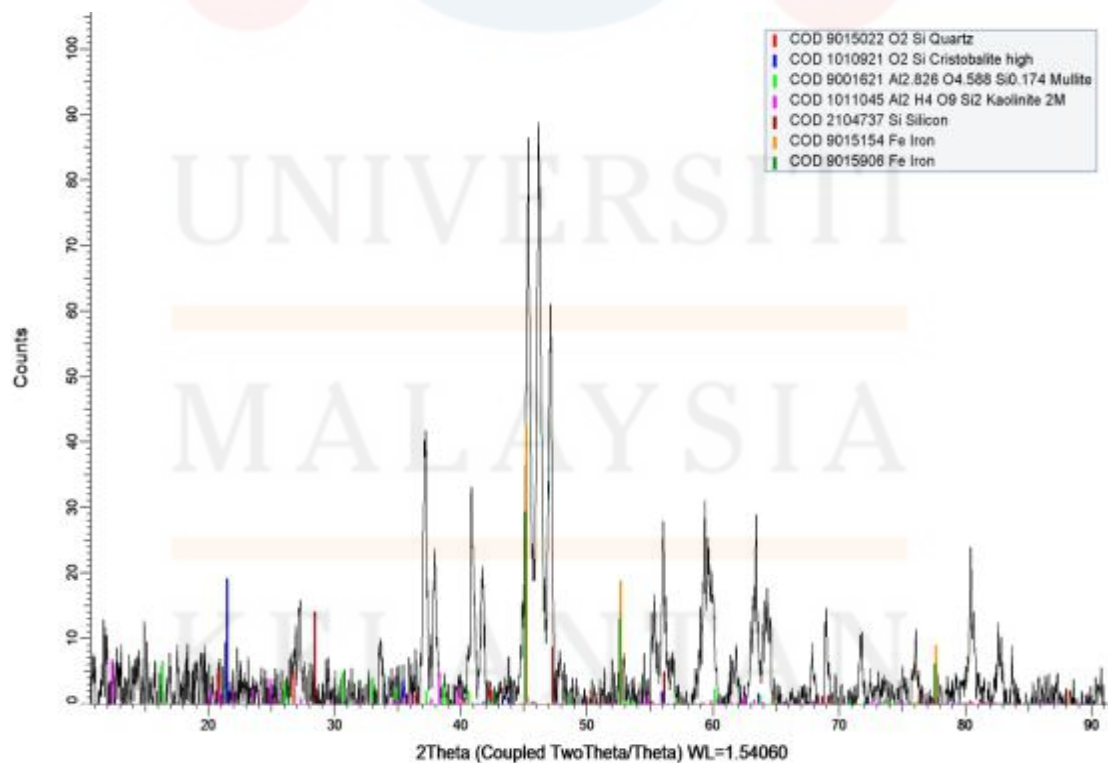


Figure 4.18: XRD pattern of 40 wt. % RHA at 900°C

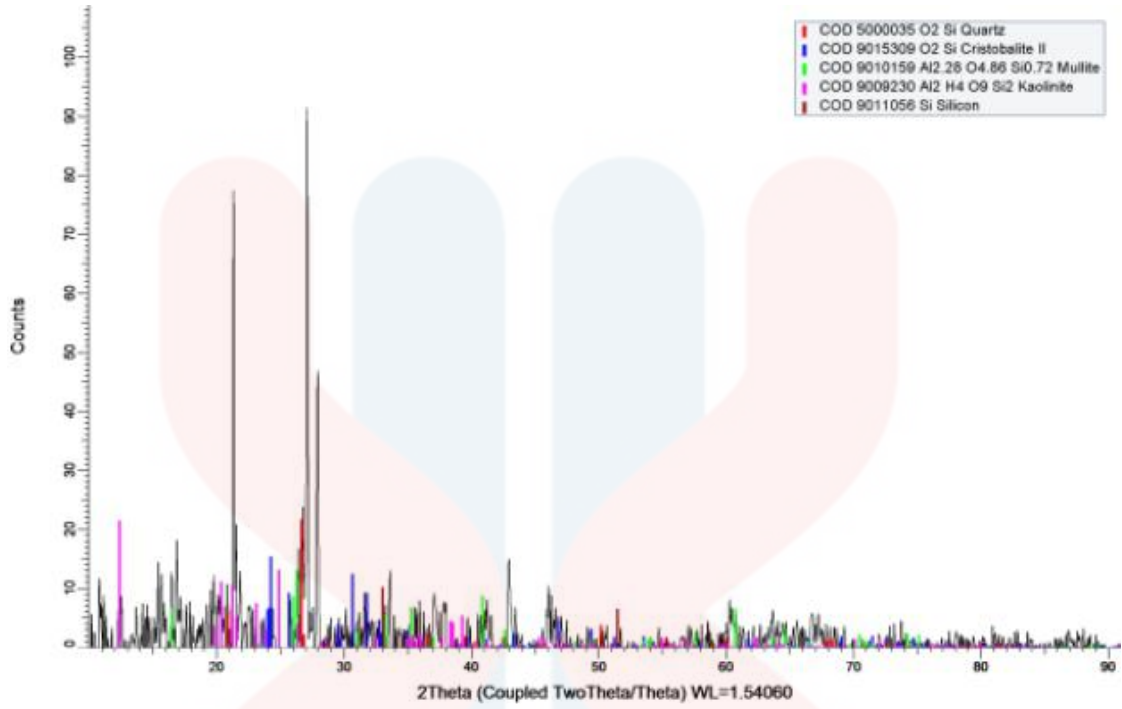


Figure 4.19: XRD pattern of 40 wt. % RHA at 950°C

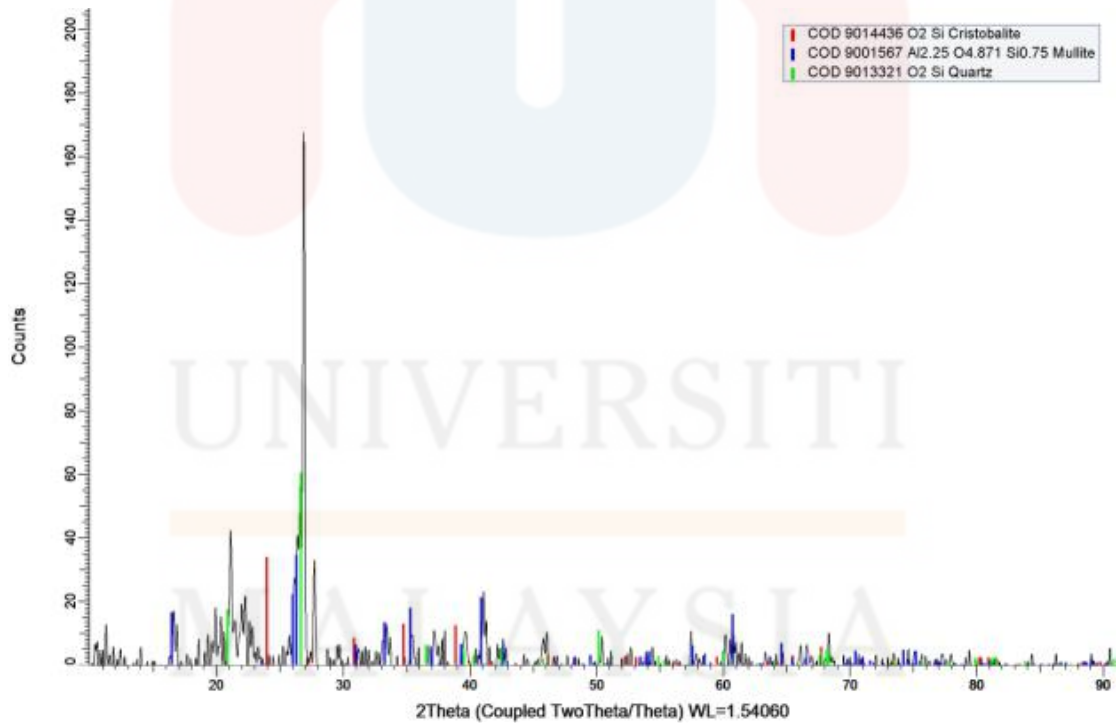


Figure 4.20: XRD pattern of 40 wt. % RHA at 1000°C

In this study, the presence of crystalline in silica which is the RHA ceramics sintered at temperature range 900 °C to 1000°C is examined by using x-ray diffraction (XRD) instrument. By using diffractive EVA software, this data is analysed at angle 2 theta from 20 degree to 90 degree and intensity at counts from 0 to 100. From the analysis, this ceramics body gives crystallinity value 69.6%. At this temperature, there are two major crystalline phases namely quartz and mullite with small intensity of cristobalite phases in all three temperatures.

All of these are the polymorph of silica in which they are having the same chemical formula but have different arrangement in their crystal structure. As the temperature increase, the intensity peak of quartz is decreases significantly while the peak high of mullite is remain constant. However, the peak intensity of cristobalite is increase with the increase in temperature. It confirmed that the reaction of sintering process can effectively transform the crystal structure of silica from quartz to mullite and cristobalite at temperature range of 900°C to 1000°C.

Silica is RHA is amorphous form at low temperature in between 700°C to 800°C (Cole, 1935). Crystalline tend to increase as the temperature increased as the effect of grain growth. At temperature 1000°C, the grain expansion in the in between the particle is partially developed. However, at temperature range above 1200°C, the grain can be clearly seen as the crystallinity increased. As been mentioned, sintering process will provide energy to encourage the particle bond together in order to form other crystalline phases. From this statement, it can be explained as well as the crystallinity increase, the firing shrinkage also increase while the water absorption will be decreased as the apparent porosity also decreased.

4.6 Morphology Analysis via Scanning Electron Microscope

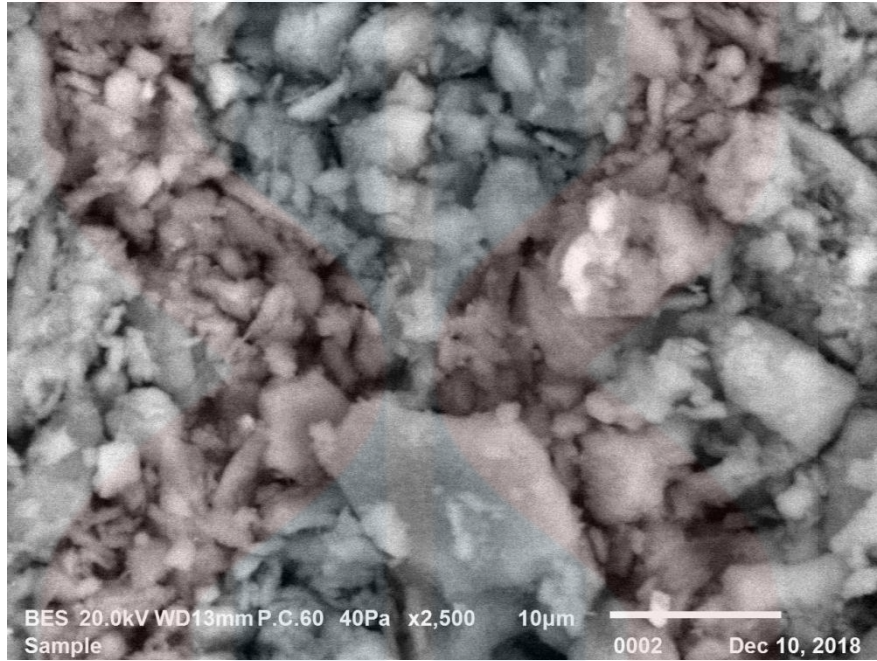


Figure 4.21: SEM for 40 wt. % RHA at 950 °C(x2500 magnification)

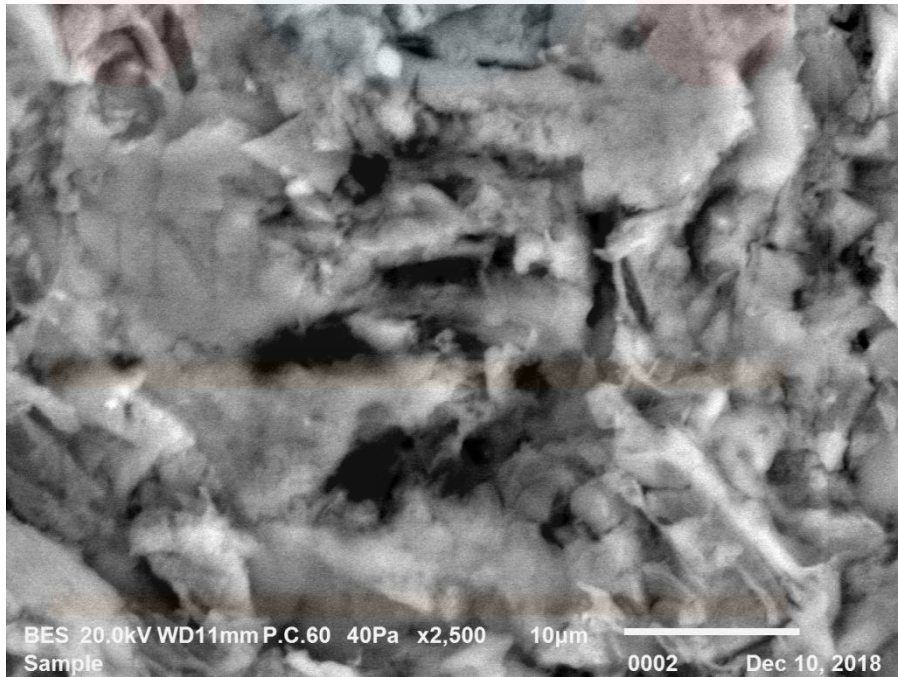


Figure 4.22: SEM for 0 wt. % RHA at 1000 °C(x2500 magnification)

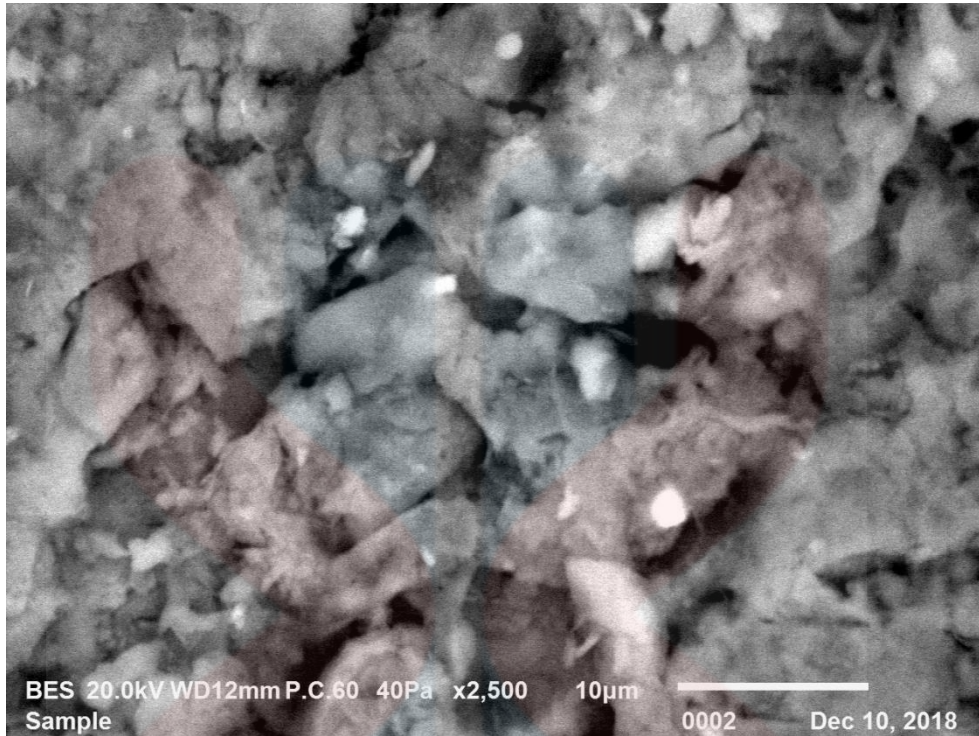


Figure 4.23: SEM for 20 wt. % RHA at 1000 °C(x2500 magnification)

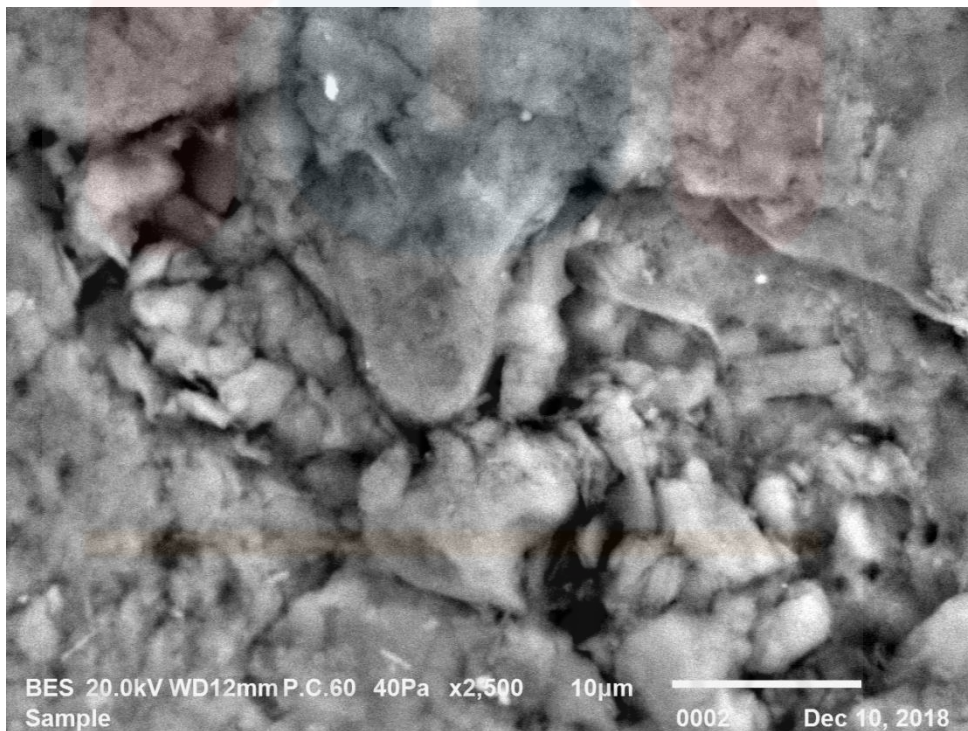


Figure 4.24: SEM for 40 wt. % RHA at 1000 °C(x2500 magnification)

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Figure 4.21 shows the morphology structure of ceramic brick from 40 wt. % RHA at 950 °C while Figure 4.22, Figure 4.23 and Figure 4.24 represent structure for ceramic brick at 1000 °C for 0 wt. %, 20 wt. % and 40 wt. % respectively. It is observed that the structure of ceramic brick is having irregular distributed pores with a relatively complex shape. This feature is related to the process of this material is getting its own characteristic structural arrangement. According to the spot area, the pores at 0 wt. % RHA is much clear compared to 20 wt. % and 40 wt. % RHA. The addition of RHA at specific temperature 1000 °C shows to give the ceramic brick low in porosity. This feature is important to be determined in which it help in fabrication of the quality support.

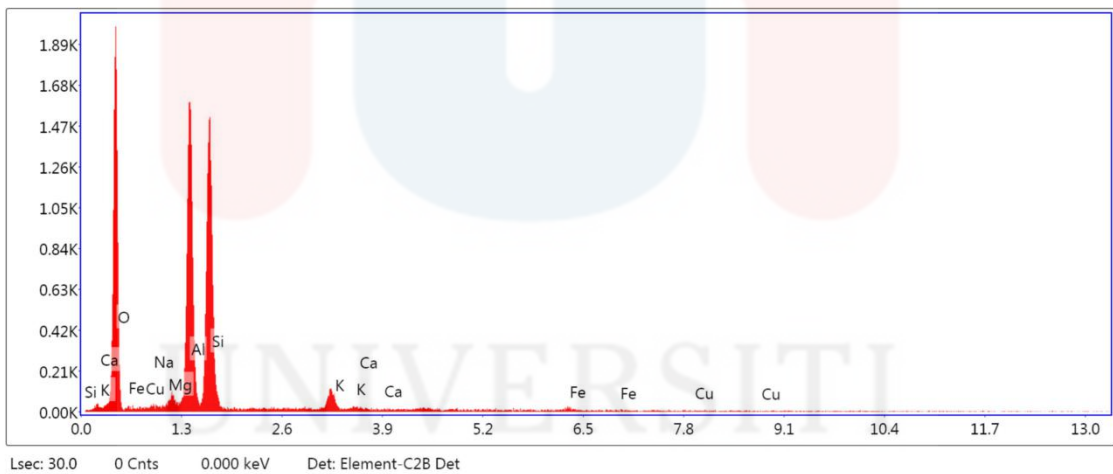


Figure 4.25: Graph energy dispersive spectroscopy (EDS) of 0 wt. % RHA

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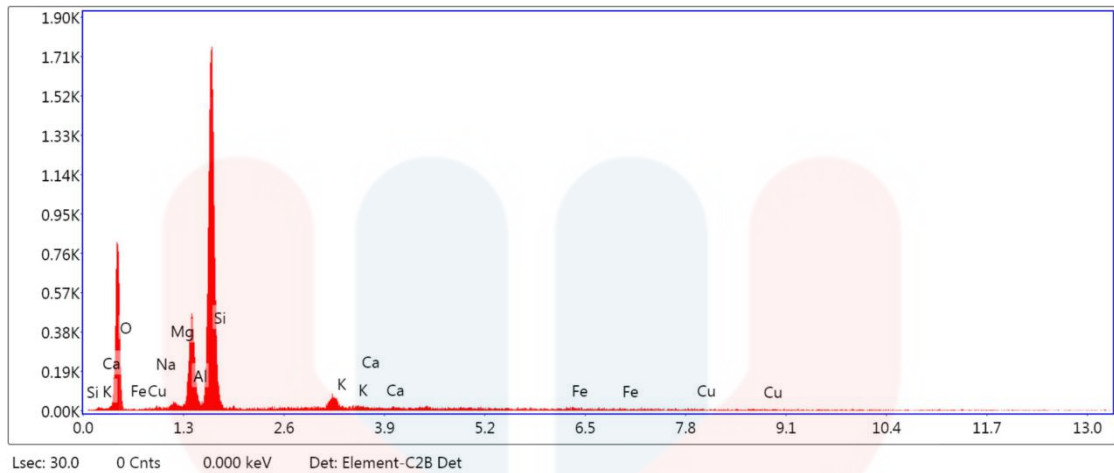


Figure 4.26: Graph energy dispersive spectroscopy (EDS) of 40 wt. % RHA

Figure 4.25 and Figure 4.26 shows the result of the composition of ceramic brick for 0 wt. % RHA and 40 wt. % RHA by energy dispersive spectroscopy. It can be seen that peaks relating to aluminium, oxygen and silicon demonstrating that the ceramic brick consist of aluminium oxide and silicon dioxide. However, the percentage of aluminium oxide is higher in 0 wt. % RHA while percentage of silicon dioxide in higher in 40 wt. % RHA. This is due to the nature of the raw material in which RHA is basically contain higly silica compared to clay. Since silica is proved to give strength to ceramic product, the combination of RHA with clay is recommended in order to produce better quality of ceramic brick.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this study, the utilization of rice husk ash (RHA) as the ceramic brick in bioreactor was successfully done. Based on the results obtained from the percentage of firing shrinkage, volumetric shrinkage, water absorption, apparent porosity, bulk density as well as the analysis from x-ray diffraction (XRD) result, it shows a positive finding in which indicate the suitability of RHA to be used in the production of ceramic brick specifically for bioreactor. From the overall data, the best result of percentage of water absorption is 0.71%, 0.81% of apparent porosity, 8% to 10% linear shrinkage and 0.82% of volumetric shrinkage at 1000°C of 40 wt. % RHA. From this finding, it can be conclude that RHA composition at 40 wt. % and 1000°C is the best choice for body formulation and firing temperature suitable for the utilization of ceramic brick for bioreactor.

Increase in temperature will increase the firing shrinkage as well as the volumetric shrinkage. This situation later cause to the decrease in water absorption of the ceramic bodies especially when the percentage of RHA been added is increase together with the decrease in its porosity. Reduction of porosity especially at temperature 1000°C is due to the effect of body dense as during densification process. Densification caused the particle to bond together as the result of energy provided from the firing temperature. The diffusion of particles causes the air space in the ceramics body being removed. The addition of RHA as silica precursor thus help to increase the percentage of pores removal as silica in RHA act as filler that filled up the air space between the particles grain in the ceramic body. At the same time, RHA also act as flux which reduce the melting point for the transformation of crystalline structure.

5.2 Recommendation

For the future study, I am suggesting for the research on the effect of body formulation and firing temperature on the ceramic strength which can be done by performing 3 point bending test to observe the modulus of ruptured (MOR) of the ceramic. Since strength affect the quality of ceramic, it is important to perform this investigation thus it will help to support either ceramic from rice husk ash (RHA) is suitable or not to be utilized as ceramic brick in bioreactor system. MOR test is unable to be done in this study due to the limitation in size of the ceramic sample.

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Appendex

(A) Figure



Figure 1: Raw clay



Figure 2: Raw RHA before sieve



Figure 3: Mixing of clay

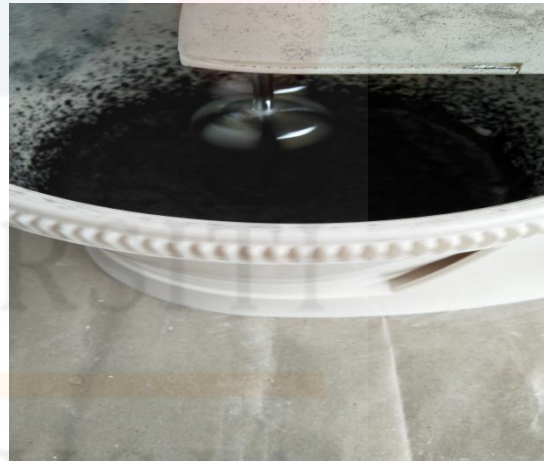


Figure 4: Mixing of clay and RHA

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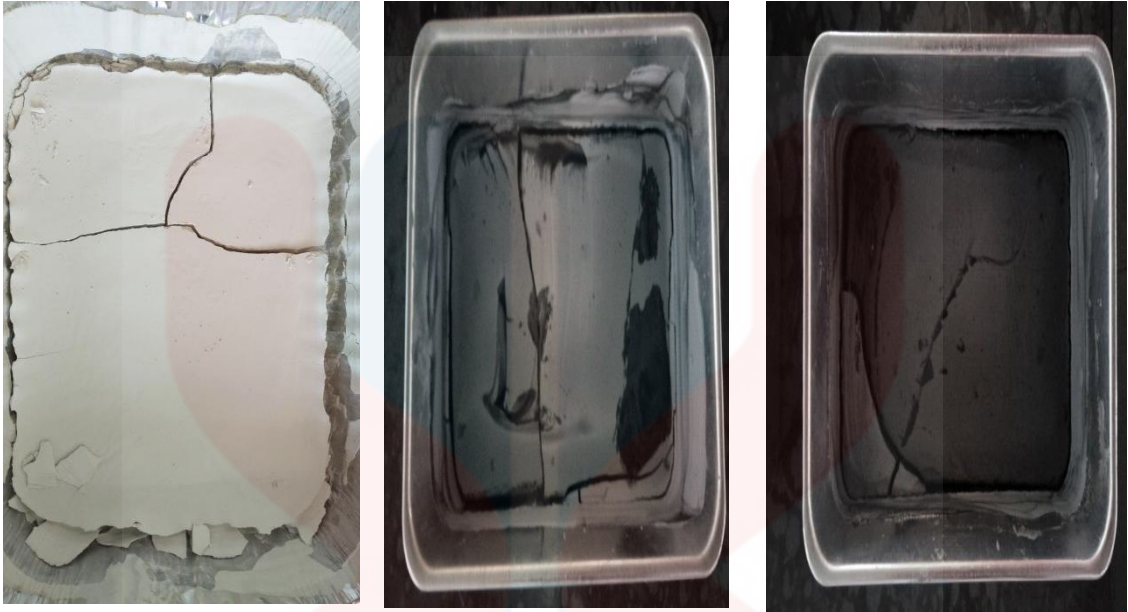


Figure 5: Drying process



Figure 6: Grinding

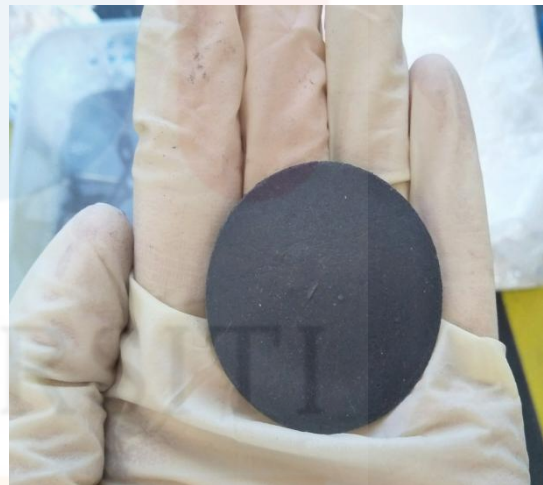


Figure 7: Compaction

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Figure 8: Firing process

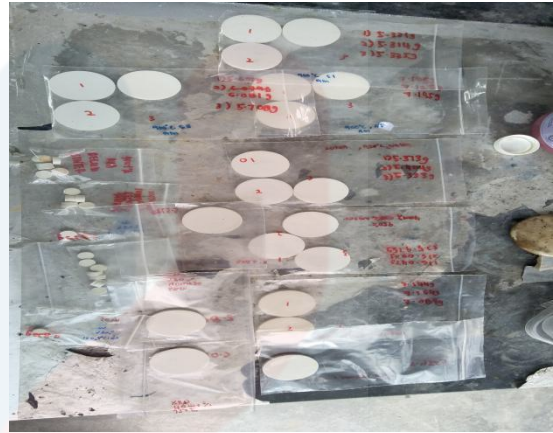


Figure 9: Fired ceramic brick



Figure 10: Water absorption

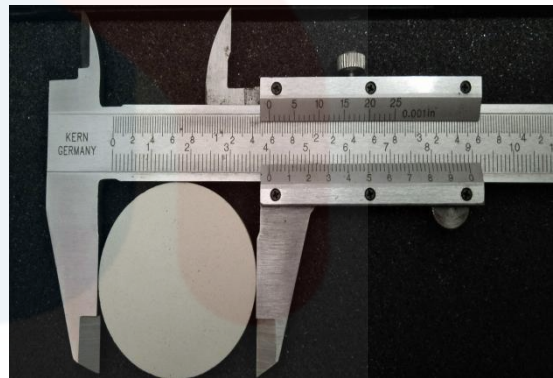


Figure 11: Fired ceramic brick

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(B) Table

Table 1: Percentage of firing shrinkage for 0 wt. % RHA at 900°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	4.0 x 4.0	0	0	0
4.0 x 4.0	4.0 x 4.0	0	0	
1.0 x 1.0	0.94 x 0.94	0.06	6	5.33
1.0 x 1.0	0.95 x 0.95	0.05	5	

Table 2: Percentage of firing shrinkage for 20 wt.% RHA at 900°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.97 x 3.97	0.02	0.75	0.75
4.0 x 4.0	3.97 x 3.97	0.02	0.75	
1.0 x 1.0	0.93 x 0.93	0.08	8	8.0
1.0 x 1.0	0.93 x 0.93	0.08	8	

Table 3: Percentage of firing shrinkage for 40 wt. % RHA at 900°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.96 x 3.96	0.04	1	1.0
4.0 x 4.0	3.96 x 3.96	0.04	1	
1.0 x 1.0	0.92 x 0.92	0.08	8	8.0
1.0 x 1.0	0.92 x 0.92	0.08	8	

Table 4: Percentage of firing shrinkage for 0 wt. % RHA at 950°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.98 x 3.98	0.02	0.5	0.5
4.0 x 4.0	3.98 x 3.98	0.02	0.5	
1.0 x 1.0	0.93 x 0.93	0.07	7	7.0
1.0 x 1.0	0.93 x 0.93	0.07	7	

Table 5: Percentage of firing shrinkage for 20 wt. % RHA at 950°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.95 x 3.95	0.05	1.25	1.25
4.0 x 4.0	3.95 x 3.95	0.05	1.25	
1.0 x 1.0	0.92 x 0.92	0.08	8	8.0
1.0 x 1.0	0.92 x 0.92	0.08	8	

Table 6: Percentage of firing shrinkage for 40 wt. % RHA at 950°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.92 x 3.92	0.08	2	2.0
4.0 x 4.0	3.92 x 3.92	0.08	2	
1.0 x 1.0	0.92 x 0.92	0.02	8	8.0
1.0 x 1.0	0.92 x 0.92	0.02	8	

Table 7: Percentage of firing shrinkage for 0 wt.% at 1000°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.92 x 3.92	0.08	2.0	2.0
4.0 x 4.0	3.92 x 3.92	0.08	2.0	
1.0 x 1.0	0.92 x 0.92	0.08	8	8.0
1.0 x 1.0	0.92 x 0.92	0.08	8	

Table 8: Percentage of firing shrinkage for 20 wt.% at 1000°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.91 x 3.91	0.09	2.25	2.25
4.0 x 4.0	3.91 x 3.91	0.09	2.25	
1.0 x 1.0	0.90 x 0.90	0.10	10	10.0
1.0 x 1.0	0.90 x 0.90	0.10	10	

Table 9: Percentage of firing shrinkage for 40 wt.% at 1000°C

Original diameter (cm)	Fired diameter (cm)	Diameter differences (cm)	Firing shrinkage (%)	Average firing shrinkage (%)
4.0 x 4.0	3.91 x 3.91	0.09	2.25	2.25
4.0 x 4.0	3.91 x 3.91	0.09	2.25	
1.0 x 1.0	0.90 x 0.90	0.10	10	10.0
1.0 x 1.0	0.90 x 0.90	0.10	10	

Table 10: Table for percentage of water absorption, apparent porosity and bulk density

Percentage of RHA	Firing Temperature	Suspended Dry Weight in Air, Md	Suspended Saturated Weight in Water, Msw	Suspended Saturated Weight in Air, Msa	Water Absorption (%)	Apparent porosity (%)	Bulk density	Average WA (%)	Average AP (%)	Average BD (%)
0 wt%	900	0.85	0.312	0.918	8.000	11.221	1.402	6.48	9.44	1.47
0 wt%	900	0.746	0.3	0.783	4.959	7.660	1.544			
20 wt%	900	0.718	0.382	0.735	2.367	0.160	2.033	3.26	4.30	2.03
20 wt%	900	0.746	0.41	0.777	4.155	8.447	2.032			
40 wt%	900	0.745	0.284	0.752	0.939	1.496	1.591	1.69	1.60	1.60
40 wt%	900	0.694	0.28	0.711	2.450	3.944	1.610			
0 wt%	950	0.82	0.21	0.875	6.707	8.270	1.233	5.68	6.95	1.22
0 wt%	950	0.817	0.18	0.855	4.651	5.629	1.210			
20 wt%	950	5.213	0.43	5.316	1.976	2.108	1.066	2.92	3.16	1.08
20 wt%	950	5.404	0.64	5.613	3.867	4.202	1.086			
40 wt%	950	6.006	0.989	6.038	0.533	0.633	1.189	1.18	1.40	1.19
40 wt%	950	6.03	1.053	6.14	1.824	2.162	1.185			
0 wt%	1000	0.992	0.248	1.02	2.823	3.627	1.284	2.82	3.14	1.22
0 wt%	1000	0.956	0.15	0.978	2.823	2.657	1.154			
20 wt%	1000	0.703	0.11	0.713	1.422	1.658	1.165	2.11	2.62	1.23
20 wt%	1000	0.717	0.18	0.737	2.789	3.590	1.287			
40 wt%	1000	0.768	0.12	0.775	0.911	1.068	1.172	0.71	0.86	1.24
40 wt%	1000	0.792	0.19	0.796	0.505	0.660	1.306			

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