UNIVERSITI MALAYSIA KELANTAN

Optimization Of Chicken Eggshell as Pore Forming Agent in Porous Ceramic Using Response Surface Methodology (RSM) Statistical Design

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DECLARATION

I declare that this thesis entitled "Optimization Of Chicken Eggshell as Pore Forming Agent in Porous Ceramic Using Response Surface Methodology (RSM) Statistical Design" is the results of my own research except for the cites in the references.

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Optimization Of Chicken Eggshell as Pore Forming Agent in Porous Ceramic Using Response Surface Methodology (RSM) Statistical Design

ABSTRACT

Ceramics that are porous have a high porosity percentage, which can range from 20% to 95%. In many different applications, such filtering systems, thermal insulators, and biomedical, porous ceramic is essential. Pore-forming agent (PFA) and ceramic powder make up a porous ceramic composition. Researchers looked into the viability of using food waste (coffee grounds and banana peels) as PFA to make porous porcelain. The food waste's high moisture content makes the waste preparation process challenging. It was discovered that chicken eggshells had a lower moisture content than other food waste, which will make the waste preparation process practical. The objective of this study to produce and characterize porous ceramic that incorporated chicken eggshell as a pore-forming agent (PFA). Using Response Surface Methodology (RSM) statistical approaches to optimize the weight percentage of added chicken eggshell and the sintering temperature to the physical and mechanical properties of the porous ceramic. Porous network ceramic structures are generally light and possess low mass, low density, and poor heat conductivity. Porous ceramic is created by combining pore-forming agents (PFA) with ceramic powder. A water absorption test, apparent porosity, bulk density, and crystalline phase (XRD characterization) were used to determine the physical properties. The mechanical properties were determined by compressive strength. The results showed that increasing the weight % of chicken eggshell (CES) in the porous ceramic reduces compressive strength and increases porosity. The purpose of this study is to determine the optimal optimization for porous ceramics by analyzing data from Minitab 16 using Response Surface Methodology (RSM) Statistical Design.

Keywords: Chicken Eggshell (CES), Pore-Forming Agent (PFA), Porous Ceramic

Pengoptimuman Kulit Telur Ayam sebagai Agen Pembentuk Liang dalam Seramik Berliang Menggunakan Reka Bentuk Statistik Metodologi Permukaan Tindak Balas (RSM)

ABSTRAK

Seramik yang berliang mempunyai peratusan keliangan yang tinggi, iaitu antara 20% hingga 95%. Dalam banyak aplikasi yang berbeza, sistem penapisan, penebat haba dan bioperubatan, seramik berliang adalah penting. Agen pembentuk liang (PFA) dan serbuk seramik membentuk komposisi seramik berliang. Penyelidik melihat ke dalam daya maju menggunakan sisa makanan (ampas kopi dan kulit pisang) sebagai PFA untuk membuat porselin berliang. Kandungan lembapan sisa makanan yang tinggi menjadikan proses penyediaan bahan buangan mencabar. Telah didapati bahawa kulit telur ayam mempunyai kandungan lembapan yang lebih rendah daripada sisa makanan lain, yang akan menjadikan proses penyediaan sisa praktikal. Objektif kajian ini untuk menghasilkan dan mencirikan seramik berliang yang menggabungkan kulit telur ayam sebagai agen pembentuk liang (PFA). Menggunakan pendekatan statistik Response Surface Methodology (RSM) untuk mengoptimumkan peratusan berat kulit telur ayam yang ditambah dan suhu pensinteran kepada sifat fizikal dan mekanikal seramik berliang. Struktur seramik rangkaian berliang biasanya ringan dan mempunyai jisim rendah, ketumpatan rendah, dan kekonduksian haba yang lemah. Seramik berliang dicipta dengan menggabungkan agen pembentuk liang (PFA) dengan serbuk seramik. Ujian penyerapan air, keliangan ketara, ketumpatan pukal, dan fasa kristal (pencirian XRD) digunakan untuk menentukan sifat fizikal. Sifat mekanikal ditentukan oleh kekuatan mampatan. Keputusan menunjukkan bahawa peningkatan % berat kulit telur ayam (CES) dalam seramik berliang mengurangkan kekuatan mampatan dan meningkatkan keliangan. Tujuan kajian ini adalah untuk menentukan pengoptimuman optimum bagi seramik berliang dengan menganalisis data daripada Minitab 16 menggunakan Reka Bentuk Statistik Metodologi Permukaan Respons (RSM).

Kata kunci: Kulit Telur Ayam (CES), Agen Pembentuk Liang (PFA), Seramik Berliang

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LIST OF ABBREVIATIONS

PFA	Pore Forming Agent
CES	Chicken Eggshell
KC	Kaolin Clay
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
CCD	Central Composite Design
RSM	Response Surface Methodology
ANOVA	Analysis of Variance

LIST OF SYMBOLS

℃	Degree Celsius
%	Percentage
2θ	Diffraction Angle
MPa	Compress Strength
g/cm^3	Bulk Density
ΔL/Lo	Linear Shrinkage

CHAPTER 1

INTRODUCTION

1.1 Background Study

High porosity, which is defined as the existence of tiny cavities or spaces inside the material, is a characteristic of clay-based materials. These pores can be divided into two basic categories: primary and secondary porosity, and they are typically in the nanometer to micrometre range (farah Anjum, 2022) (Muhammad Yasin Naz, 2022). Clay-based porous ceramic have the same properties as ceramic product but much lighter because of the air inside the pores of clay-based porous ceramic. Ceramics classified as porous have a high percentage of porosity between 20 and 95% (Atiqah, 2023). For several reasons, the porosity of clay-based materials is crucial. For instance, it may have an impact on the material's tensile strength, permeability, and water absorption capacity. Understanding the porosity of clay-based materials is essential for forecasting their behavior in the contexts of geology and engineering, such as when building foundations or cleaning up contaminated soil.

Organic waste or inorganic waste can be used to create pores inside the porous ceramic. Clay-based porous ceramic uses a pore forming agent (PFA) to form a pore. Ceramics and other porous materials can be produced using organic waste as a pore-forming agent. Organic material releases gases like carbon dioxide and water vapour as it burns or decomposes. When organic waste is added to clay and then heated to high temperatures, gases produced by the firing process can form voids or pores in the material, giving it a more porous structure.

According to a preliminary study conducted by (Fatin Mohamati, 2023), eggshells (CES) can be employed as a PFA for the creation of porous ceramics. CES are a PFA for thermal insulators due to their tiny and consistent particle size distribution, which ranges from 1.5 µm to 63 µm. The CES has a higher ignition point between 600 and 850 °C (Nadia et al., 2022), which requires high heat (firing temperature) to ignite the CES and gradually form pores during firing and is then capable of lowering both the embodied energy and carbon footprint for the porous thermal insulator.

However, the ceramic porosity, thermal conductivity, and water absorption improved when the material was utilized as a PFA in the clay product, leading to a decrease in the compressive strength of the clay product. It has been determined that the percentage of porosity and compressive strength should be balanced, which is correlated with the quantity of the pore-forming agent applied to the clay prior to burning (Salah Nasr, 2020). The porous ceramic will be evaluated using Response Surface Methodology's statistical experimental design known as Central Composite Design (CCD). This study will utilize RSM statistical design to optimize the wt.% of CES and the sintering temperature for the production of porous ceramic.

1.2 Problem Statement

Food waste, like banana peels and coffee grounds, has been effectively used as PFA for clay-based porous ceramics even though its moisture content ranges from 7.53 to 42.31%. The waste's high moisture content will make processing it more challenging, particularly when producing PFA powder through grinding. Preliminary study significantly successful in making porous ceramic using CES (Fatin Mohamati, 2023). On the other hand, the compressive strength of ceramic is reduced when the percentage of CES increases. This is because porous ceramic's decrease compressive strength is a result of the fact that its density decreased as the number of pores. However, Fatin.Mohamati (2023) has found that CES is suitable as PFA due to low moisture content. Utilizing RSM statistical design, this study aims to determine the optimal optimization of the weight percentage (wt.%) of CES added and the sintering temperature that is required to make porous ceramic with the best composition, particularly the compressive strength.

1.3 Objective

The objective of this research is:

1. To produce and charaterize the porous ceramic incorporated with chicken eggshell as pore-forming agent (PFA).
2. To optimize the wt.% of chicken eggshell added and sintering temperature to physical and mechanical properties of porous ceramic using Response Surface Methodology (RSM) statistical approaches.

1.4 Output

Using Response Surface Methodology (RSM) will assist in producing porous ceramic incorporated with CES that has balanced properties such as strength and porosity. Highest porosity but at the same time have highest possible compressive strength.

1.5 Scope of Study

The goal of the study is to discover the suitable percentage optimization addition of the CES as a pore-forming agent corporate data using Response Surface Methodology (RSM). This study will be carried out at the University Malaysia, Kelantan. The research will prepare the necessary materials like chicken eggshells (CES) and kaolin clay (clay-based porous ceramic). Students will prepare the CES using a variety of techniques, and the supervisor will provide the clay. Using statistical experiment design and Response Surface Methodology (RSM), this experiment aims to optimise the best percentage of formulation of the porous ceramic. The porous ceramic is characterised by its ability to absorb water, bulk density, apparent porosity, compressive test, and microstructural examination. X-Ray Diffraction (XRD), and Fourier Transfer Infrared Spectroscopy (FTIR) will be used to carry out this characterization. MINITAB software will be used to analyse the data that has been collected.

1.6 Significant of Study

The largest daily waste in Malaysia is CES. Recycling CES from the food production process into organic PFA can help to reduce pollution and protect the environment. By employing this technique, porous ceramics can be enhanced, and our industry can expand into a global market. The introduction of CES into the production of porous ceramics is the major goal. In addition, it is anticipated that the incorporation of CES into porous porcelain may improve its qualities. In this investigation, statistical design is used to optimise the incorporation of CES into porous ceramic. Therefore, the goal of this investigation is to find the CES with the best and optimum porosity, weight percentage, great compressive strength, and value that will affect the final porous ceramic product. Additionally, it may make the best use of the leftover chicken eggshells to create a material with the right combination of porosity and compressive strength.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of porous ceramic

Ceramics that have more than 15% porosity are typically referred to as porous ceramics. Macropores (pores with diameters ranging from a few tens of nanometers to microns), mesopores (usually pores between 2 nm and 50 nm in diameter), and micropores (often pores smaller than 2 nm in diameter) are the three types of pore diameters that make up porous ceramic materials (Yu Chen, 2021) (Nannan Wang, 2021). Several various techniques have been described in the literature for producing porous ceramics, including the organic final approach, the freeze casting method and the PFA method. The distribution of raw materials and the size of the raw material particles typically determine the manufacturing process, the kind of binder used, the amount of binder used, and the sintering of porous ceramic bodies. Both the final porosity and pore connectivity of the porous ceramic are significantly influenced by those parameters. According to some theories, increasing processing conditions like heat, sintering temperature, and time will decrease the porosity (He Li, 2020) (Yongsheng liu, 2020). In general, raw ceramic powder should have particles that are 2 to 5 times larger than pores to ensure that the pores that are created are of the right size.

2.1.1 Properties of Porous Ceramic

It is important to remember that porous ceramics' physical and mechanical is related. The first important physical characteristic of porous ceramics is shrinking. Ceramic manufacturers can identify the appropriate mould or die size for a certain burned ware size by evaluating the shrinkage of the sample after drying and sintering. In addition to shrinkage, apparent porosity, bulk density, and water absorption are important physical characteristics. In order to evaluate the mechanical strength of porous ceramics, compressive strength is crucial.

2.1.2 Pore-forming Agent

The type of raw material used and the firing temperatures, both of which have an impact on the finished product, are the major elements in the creation of ceramic. To create clay products with the right physical and mechanical qualities, additives are routinely utilised in ceramic production. Choosing an addition depends on the qualities that are needed. The best materials are lightweight ceramics with excellent compressive strength and little water absorption. Create porosity in the clay body by adding pore-forming materials, which are either organic or inorganic pore-forming agents, as one method of increasing such ceramic capacity. A pyrolytic material known as a PFA burns away during firing (Zivcova et al., 2018). To produce porous ceramics with a regulated microstructure, specific pore forming agents can be utilised, such as wheat flakes, starch, polymethyl methacrylate (PMMA), rice husk ash, poppy seed, and sawdust (Atiqah, 2023).

2.2 Inorganic PFA

Inorganic PFA refers to fugitive material that is not carbon-based and is utilised in the manufacturing of porous ceramic. industial waste is used to produce inorganic PFA, which is then used to make porous ceramic products. the developed an inorganic porous ceramic using fly ash as an inorganic PFA sintered with lead-zinc mine tailing.the study about the using of drinking water treatment sludge as inorganic PFA in the production of brick (Tantawy & Mohamed, 2017). Besides that, there is a study about the usage of inorganic PFA in brick manufacturing from paper mill sludge (Goel & Kalamdhad, 2017) (Ali Yaras, 2020).

2.2.1 Physical And Mechanical Properties of Porous Ceramic with Inorganic PFA

It is significant to know the physical and mechanical properties of porous ceramic. The porous ceramic's first important physical characteristic is shrinkkage. Ceramic manufacturers can choose the optimal mould or acceptable size for a certain fired ware size by studying the shrinkage of clays or bodies after drying and sintering. Aside from shrinkage, important physical characteristics include water absorption, apparent porosity, and bulk density. A porous ceramic's compressive strength must be considered while evaluating its mechanical strength.

2.3 Organic PFA

Most of the study is about focusing on utilizing the organic PFA on the food waste in produce the porous ceramic. The research about the use of banana peels as a PFA and effects of sintering on porous ceramic has been investigated (Mouiya et al., 2019). Since activities including food production, unethical food waste management, and transportation all contribute to environmental problems, this study will focus on food waste (Schanes et al., 2018). According to a recent study, several organic PFAs made from food waste are used in the manufacturing of porous porcelain. (Alzukaimi & Jabrah et al., 2019), have study about incorporated coffee ground waste as PFA to produce porous alumina ceramic.

2.3.1 Physical And Mechanical Properties of Porous Ceramic with Organic PFA

Organic PFAs are often derived from carbon-based compounds. Organic PFAs are generally less expensive and easier to separate, collect, and transport than inorganic PFAs (Munoz et al., 2019). Organic PFA also exhibits unique mechanical and physical characteristics. The study of organic PFAs indicates that the PFAs can be used to create porous ceramic with outstanding physical and mechanical properties. The viability of processing the food waste as PFA will, however, be marginally impacted by the high moisture content of these organic PFAs.

2.3.2 Moisture Content

The moisture content of food waste will influence the suitability of the food waste for the PFA application. The waste contains a significant amount of moisture, making preparation difficult, especially when the waste is ground into PFA powder form. PFA powder may agglomerate together and is difficult to produce with the proper particle size. It is clear from this organic part that organic PFA designed to treat food waste offers a number of advantages over inorganic PFA in terms of combustibility, toxicity, and cost-effectiveness. However, preparation for food waste is less practical due to high moisture content except for CES waste which have a much lower moisture content. Therefore, it is important to investigate the utilization of CES waste as PFA porous ceramic.

2.4 Chicken Eggshell

2.4.1 Properties of Chicken Eggshell

CES refers to the outer shell that protects the yolk and the white. When eggs are cracked for consumption or after an incubated egg hatches, chicken eggshell is obtained (Yusuff A.S., 2018). Eggshell is a solid waste, with production of several tons per day. Eggshells are mostly sent to the landfill with a high management cost. It is economical to transform the eggshell waste to create new values from these waste materials (Arabhosseini A., Faridi H et al.,2018). In this research, the CES will go through several process to determind the physical properties such as water absorption test, apparent porosity, bulk density, linear shrinkage, and crystalline phase (XRD characterization) while for the mechanical properties determined using compressive strength. All of the procedure needs to be done to get the suitable percentage of chicken eggshell waste as PFA for porous ceramic and the sintering temperature on the porous ceramic physical and mechanical properties. The moisture of CES has been declared as a lower moisture content due to others research. Previous research has investigated and claimed that the CES is low moisture and can be use as PFA.

2.4.2 Utilization of Chicken Eggshell (CES) as Pore Forming Agent (PFA)

Calcium carbonate, from which calcium or calcium oxide can be separated for a variety of uses, is the primary component of CES. The literature on the many uses of CES that has recently been published is reviewed in this current endeavor.(Ohji and Fukushima 2012) Catalysts for the generation of biofuel, the building industry, wastewater purification, the industrial sector, the food industry, the medical field, and agricultural applications are among the examined application possibilities. Aside from transesterification reactions, the specific areas of application include dentistry, therapeutics, bone formation, drug delivery, organic fertiliser, hydroxyapatite production, asphalt binder, cement additives and replacement in concrete, adsorbent of metals and dyes, and poultry feeds. Before being used, the CES is put through pretreatment and other modification procedures for the majority of the applications that have been found. According to earlier research, Fatin Mohamati (2023) has demonstrated that it is possible to successfully use CES as PFA in the fabrication of porous ceramics. Water absorption, apparent porosity, bulk density, and compressive strength were among the physical and mechanical characteristics that statistical analysis revealed to be very significant and demonstrated for the clay-based ceramic integrated with CES. CES can be used for porous ceramic, according to this study. The compressive strength is reduced, and the porosity is increased when the weight percentage of CES in porous ceramic is increased. The sintering temperature of 950℃ and a CES content of 20% were found to be appropriate for clay-based ceramics.

2.4.3 Summary

From the previous study Fatin Mohamati (2023), the fugitive substance, which serves as the PFA, and ceramic powder make up the formulation for making porous ceramic. Because of its exceptional physical and mechanical qualities, kaolin is preferred as the ceramic powder for creating porous ceramics. Regarding combustibility, toxicity, and cost-effectiveness, organic PFA, which concentrates on food waste, offers a number of advantages over inorganic PFA. However, aside from the CES, the preparation procedure is less practical due to the organic PFA's moisture content. The crystalline compound from the powdered form of the CES and its functional group demonstrated the material's applicability as a PFA for porous ceramic.

CHAPTER 3

MATERIALS AND METHODS

3.1 Research Flowchart

Research Flowchart of this project.

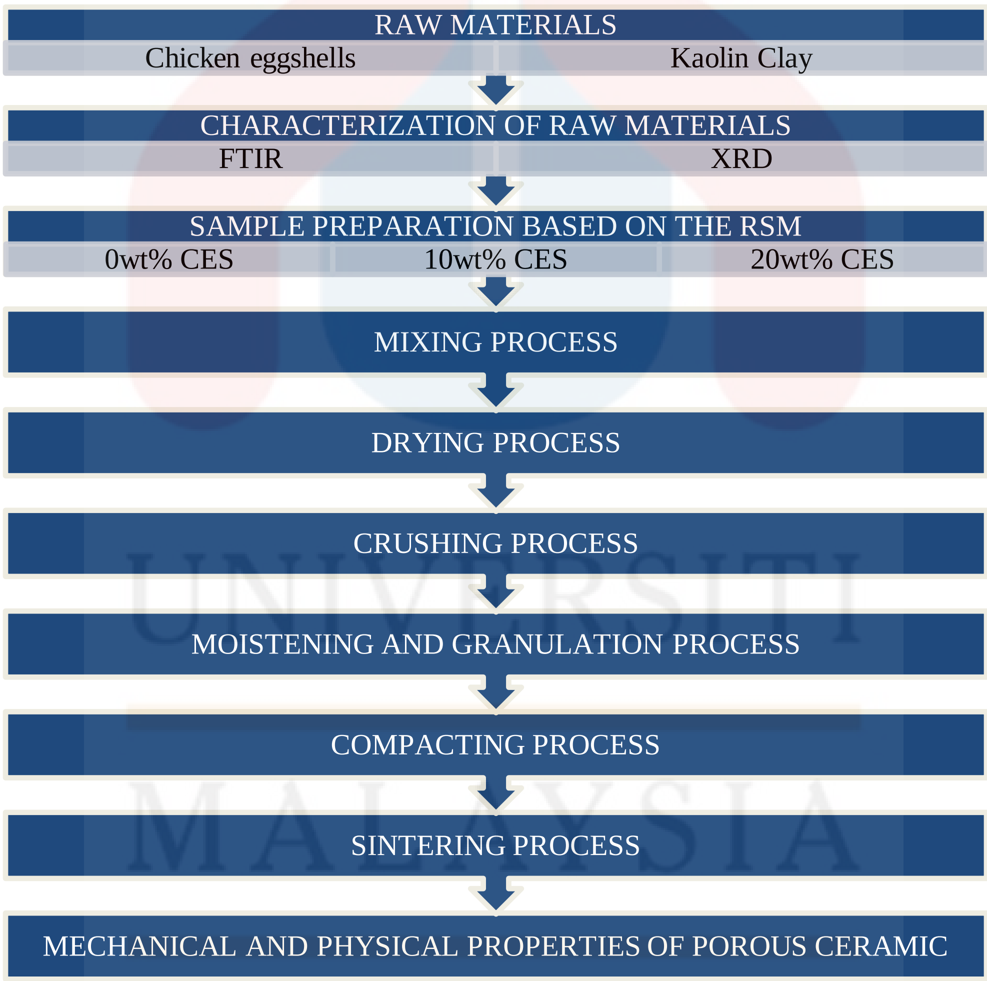


Figure 3.1: Research Flowchart

3.2 Materials

3.2.1 Chicken Eggshell

For 24 hours, the CES is dried at 100℃ degrees Celsius in an oven. Comparing the mass before and after the test allowed researchers to calculate the moisture content. In a high-speed processor and grinder, the dry CES was chopped and processed for 10 minutes. Materials that (will be) were sieved to an average particle size of 53 µm or less will be used to make and test porous ceramic produced from clay and powdered CES. The CES powder was subjected to an FTIR analysis using a particular machine. the crystalline phase of CES powder was described by XRD using Bruker Model D2 Phaser and CuKα radiantion (λ=1.5418 Å) in the range of 2θ=10° - 90°.

3.2.2 Kaolin Clay

The clay for the project is supplied by a local mill, Kaolin (Malaysia) Sdn. Bhd. This clay has a lot of kaolinite, a mineral that makes it malleable and white. This ceramic is very suitable for thermal, chemical, and structural stability. As a result, a porous ceramic is being created using kaolin clay and CES. The clay was characterized using XRD analysis instead of FTIR, the same approach that was used to characterize raw materials in the lab.

3.3 Fabrication of Porous Ceramic

3.3.1 Response Surface Methodology (RSM) Statistical Design

CCD was used to optimize the porous ceramic, establish a correlation between independent variables (factors) and dependent variables (response), and develop the model equation summarizing the experiment design. Several sources emphasized the need of optimizing the addition of coconut husk to porous ceramics in order to improve properties like RSM and CCD (Ayan et al., 2020). This Response Surface Methodology (RSM) statistical design combines coupled parameters to achieve higher optimisation output, which anticipates better adjustments that increase the weight percentage of coconut husk added.

3.3.2 Experimental Design Matrix

Central Composite Design (CCD) was used to improve the weight percentage of CES and the sintering temperature. A single factor experiment was used to determine the central values of the two factor conditions. In order to anticipate the ideal circumstances for preparation and the interaction between the preparation conditions, the RSM approach is used. In this study, a 2-factor (wt.% CES and sintering temperature 3-levels (-1, 0 and 1) CCD with a small number of experimental runs is used to assess and optimize the addition of CES into porous ceramic and achieve the best composition to produce the best porosity and strength. Also, coding was used to denote the level or range of each evaluated factors on scale where for the wt.% CES, '-1' for 0%, '0' for 10% and '1' for 20%. While for the sintering temperature, '-1' for 850 °C, '0' for 900 °C and '1' for 950 °C.

Table 3.1: Factors and their Respective number of levels investigated in Central Composite Design (CCD)

Factors	Notation	Unit	Levels (in coded)		
			-1	0	1
Wt.% CES	A	Wt.%	0	10	20
Sintering Temperature	B	℃	850	900	950

3.4 Preparation of Porous Ceramic Incorporated with Chicken Eggshell (CES)

Both materials were characterized using XRD, SEM, and FTIR. The sample was based on the Response Surface Methodology (RSM) before the mixing process. The fine CES was mixed with kaolin clay inside the cake mixer for 1 hour. The mixed materials were dry in electric oven for 100℃ for 24 hours. The mixed materials were put inside the mortar for the next step to make the powder. The powder combination was then undergo granulation. Using a water sprayer, distilled water was sprayed over the powder mixture to moisten it. The powder mixture has a moisture content of between 5 and 6 weight percent (10 to 12g) per 200g of powder mixture. After that, the wet powder was divided into granules using a 250 nm test sieve with a mesh size of 60. Using a 30 MPa uniaxial hydraulic press, the powder was compacted in a mould made of hardened tool steel. The compacted body were heated at 850℃, 900℃, and 950℃, respectively.

Porous ceramic samples was tested for water absorption, bulk density, linear shrinkage, and apparent porosity as part of the physical characteristics inspection procedure. Utilizing response surface methodology (RSM), the sample already examined.

3.5 Characterization of Porous Ceramic

All samples were characterised for water absorption, effective porosity, and bulk density, and the data were analysed using CCD by RSM. A specific sample was subjected to compressive strength characterisation, microstructural examination, and XRD based on the results of water absorption, apparent porosity, and bullk density. This test was used to measure the mechanical properties of porous ceramic samples.

3.5.1 Water absorption, Apparent Porosity, and Bulk Density

MS ISO 10545-3: 2001 was used to characterise water absorption in this study. This characterisation will assess the sample's water absorption, apparent porosity, and bulk density. The vacuum method is used in this study to extract the dry mass of the sample before weighing it to determine water absorption. This process was extracting air from a sample-filled chamber. After that, the samples were immersed in water for at least 30 minutes at room temperature. After that, the sample is withdrawn from the bath, patted dry, and weighed again to measure the saturated mass of cold water in the sample. Finally, while immersed in water, the sample is measured. The water displacement method is used to calculate the volume, V, of each sample. Following that, the samples are evaluated before and after immersion in water to determine water absorption (Equation 3.1), apparent porosity (Equation 3.2), and bulk density (Equation 3.3) using the equations below.

Water absorption (WA):

$$Wa = \frac{m_2 - m_1}{m_1} \; x \, 100 \qquad \text{equation 3.1}$$

Where:

m1: mass of dried porous ceramic

m2: mass of wet porous ceramic

Apparent porosity (AP):

$$P = \frac{m_2 - m_1}{m_2 - m_3} \; x \, 100 \qquad \text{equation 3.2}$$

Where:

m1: mass of dried porous ceramic

m2: mass of wet porous ceramic (in air)

m3: mass of wet porous ceramic (in water)

Bulk density (BD):

$$B = \frac{m_1}{m_{2 - m_3}} \qquad \text{equation 3.3}$$

Where:

m_1: mass of dried porous ceramic

m2: mass of wet porous ceramic (in air)

m3: mass of wet porous ceramic (in water)

3.5.2 Compressive Strength

A compressive strength is a critical attribute. In order to achieve this characterization, this test adheres to ASTM C109. You can use this approach to determine the compressive strength of hydraulic cement and other mortars. The tests can be used to determine conformity with regulations. This test investigates the effect of adding coconut husk on the strength and capacity of porous ceramics to withstand compressive stress. The compressive strength was determined using the Testometric Materials Testing Machine. The finished product was compressed with a compression machine, and the strength was estimated using Equation 3.4.

Compressive strength (CS):

$$\boldsymbol{MPa = \frac{F}{A}} \qquad \text{equation 3.4}$$

Where,

F: compressive strength (N/mm2)

A: surface area (mm2)

3.5.3 XRD Analysis

To investigate the structure and phases present in the porous ceramic, measurements were taken in the 10°- 90° at 2 range using Bragg-Brentano geometry.

Table 3.2: Experimental Design Matrix for Central Composite Design

(not include value for all response)

factor			Response			
Run Order	**Wt.% CES**	**Sintering Temperature**	**Water absorption (%)**	**Apparent Porosity (%)**	**Bulk Density (g/cm^3)**	**Compressive Strength (MPa)**
1	-1	-1				
2	0	0				
3	0	0				
4	-1	1				
5	-1	-1				
6	-1	-1				
7	0	0				
8	0	0				
9	0	1				
10	0	0				
11	1	0				
12	1	0				
13	0	0				
14	-1	1				
15	0	-1				
16	0	0				
17	1	-1				
18	1	1				
19	0	0				
20	0	0				
21	0	1				
22	-1	0				
23	-1	0				
24	0	-1				
25	1	1				
26	0	0				

*For factor 'A' (wt.% of CES), '-1', '0', and '1' represent 0 wt.%, 10 wt.%, and 20 wt.%.

*For factor 'B' (sintering temperature), '-1', '0', and '1' represent 850℃,900℃, and 950℃.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Stage 1: Characterization of Raw Materials

4.1.1 FTIR analysis of CES and Kaolin Clay

Functional group was present to determine the CES and the kaolin clay, FTIR pattern analysis was crucial. Figure 4.1(a) shows the FTIR pattern of the CES and kaolin clay powder.

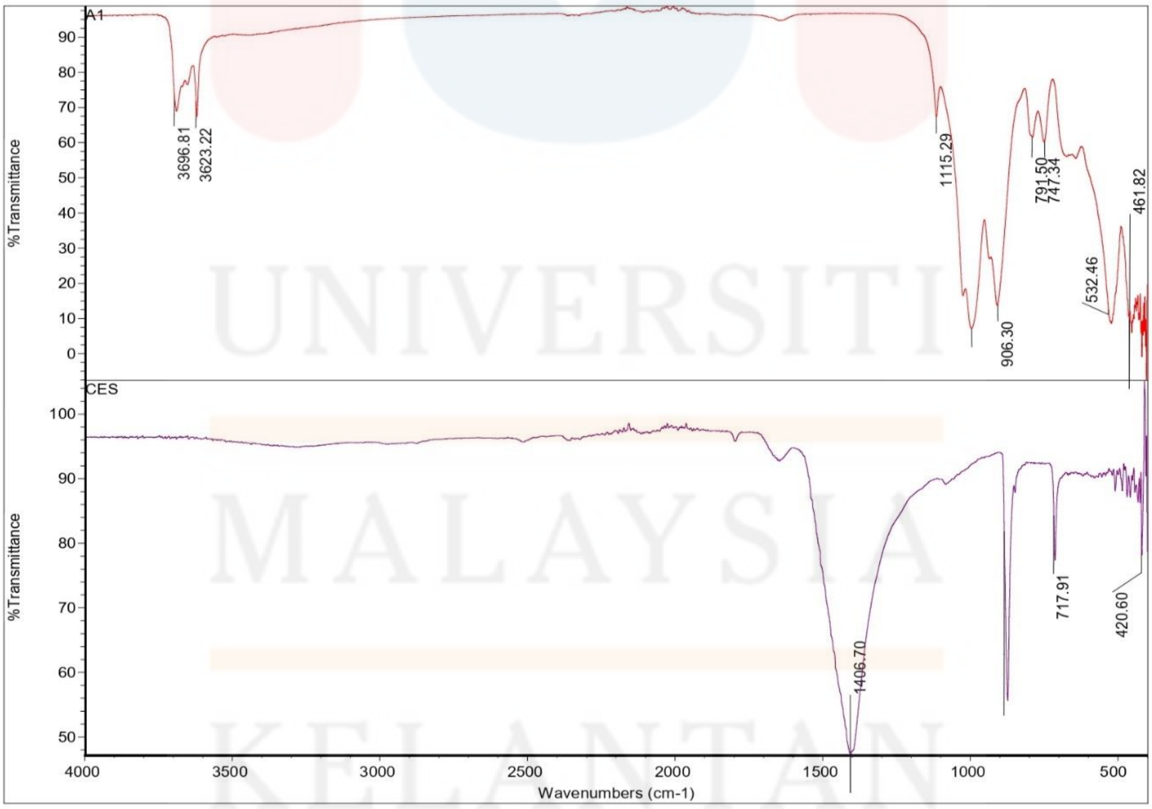


Figure 4.1(a): FTIR pattern of CES and Kaolin Clay powder.

According to figure 4.1, inorganic carbonate compounds' infrared absorption patterns are easily distinguished from one another. The strongest distinctive absorption was recorded at 1406.70cm cm^{-1}, 717.91 cm^{-1}, and 420.60 cm^{-1}. The two lower frequency bands are really sharp. The sample's hydration status influences all the band locations. The strong carbon hydrogen stretching absorption at 2900-1 has been blanked. The peak values of 1406cm^{-1}, 717.91cm^{-1}, and 420.60cm^{-1} are attributed to calcium carbonate. According to the findings, the FTIR pattern's peak and the one indicated in the literature review are similar. This indicates the comparability of the CES used in each of these investigations.

Table 4.1: The functional group and bond of CES.

Frequency(cm^{-1})	Bond	Functional Group
1406.70	C-C stretch (in-ring)	aromatics
872.69	C-H	aromatics
717.91	C-H	aromatics
420.60	C-Br	Alkyl Halides

Ftir of CES spectra have a broad peak around 1450 cm^{-1}, which is characteristic of C-C stretching vibrations. The peak at around 1400 cm^{-1} is attributed to C-C stretching vibrations in aromatic rings. This peak is most intense in the spectrum with the highest CaCO3 content. The peaks around 900-800 cm^{-1} and 750-700 cm^{-1} are assigned to C-H stretching vibrations. The spectrum of CaCO3 shows significant peaks overall, indicating that the presence of CaCO3 has a significant impact on the infrared spectrum of the CES.

According to figure 4.1, shows how inorganic hydrous aluminosilicate compounds' infrared absorption patterns are easily distinguished from one another. The strongest distinctive absorption was recorded at 1000 cm^{-1}, 906.30 cm^{-1}, and 532.46 cm^{-1}. The sample's hydration status influences all the band locations. The strong carbon hydrogen stretching absorption at 2900-1 has been blanked. The peak values of 1115.29cm^{-1}, 906.30cm^{-1}, and 532.46cm^{-1} are attributed to kaolinite, $Al_2Si_2O_5(OH)_4$.

Table 4.2: The functional group and bond of kaolin clay.

Frequency (cm $^{-1}$)	Bond	Functional Group
3696.81	O-H stretch	Alcohols
3623.22	O-H stretch	Alcohols
1115.29	N-O asymmetric stretch	Nitro compounds
791.50	C-Cl stretch	Alkyl halides
747.34	C-Cl stretch	Alkyl halides
532.46	C-Br Stretch	Alkyl halides
461.82	C-Br	Alkyl halides

Ftir of Kaolin clay spectra have a broad peak around 3600 cm^{-1}, which is characteristic of O-H stretching vibrations. The peak at around 1115.29 cm^{-1} is attributed to N-O asymmetric stretching vibrations in nitro compounds. This peak is most intense in the spectrum with the highest Kaolinite, $Al_2Si_2O_5(OH)_4$ content. The peaks around 800-700 cm^{-1} are C-Cl stretch vibration and 600-400 cm^{-1} are assigned to C-Br stretching vibrations. The spectrum of $Al_2Si_2O_5(OH)_4$ shows significant peaks overall, indicating that the presence of $Al_2Si_2O_5(OH)_4$ has a significant impact on the infrared spectrum of the CES.

4.1.2 XRD analysis of CES powder and kaolin clay

Determining the predominant crystalline phase in the CES powder at the high sintering temperature requires the use of XRD analysis. Because it will react with clay and affect the mechanical and physical properties of the porous ceramic, this phase is crucial. Figure 4.1(b) displays the results of the XRD investigation of the CES powder.

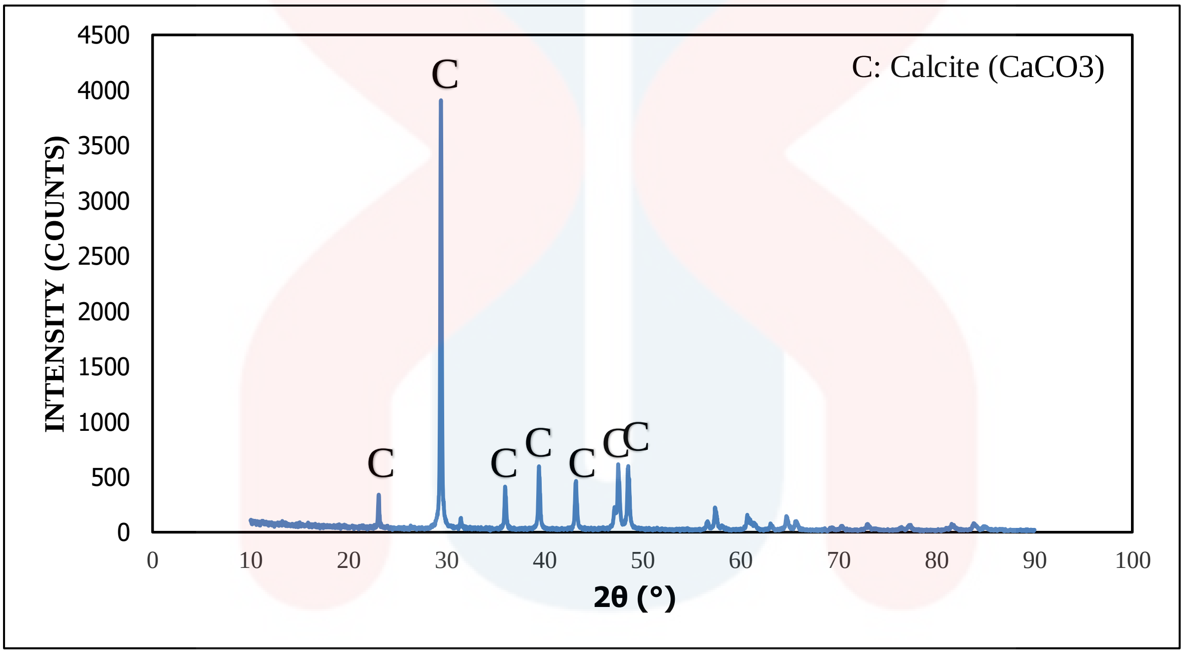


Figure 4.1(b): XRD analysis of CES

The most prevalent component of the CES powder is calcite, or CaCO3, as Figure 4.2(b) (COD 9016706) illustrates. Less common crystalline phases are massive kalsilite, diopside, sanidine, pyroxene-ideal, and quartz high. Through XRD examination, the crystalline phases discovered in the CES powder matched those discovered in the previous literature review. The phosphorus content of the CES powder contributed to the formation of whitlockite. On the other hand, the CaO may react with other oxides, such as MgO and SiO2, to produce diopside. Wang, C., and others, 2021).

In crystalline compounds, a number of oxides, such as SiO_2, Al_2O_3, Fe_2O_3, CaO, Na_2O, and MgO, can serve as fluxing agents. This may have an impact on the densification of sintered porous ceramic and help lower the sintering temperature (Sarmeen Akhtar et al., 2017). Nevertheless, the tiny amount of sample utilised in the XRD analysis may have resulted in a large amount of background noise, which might account for the presence of other crystalline phases that differ from the study stated in the literature review. This complicates the process of determining which crystalline phase the CES powder comprises. But XRD study was also done on raw kaolin to ascertain the material's principal crystalline shape.

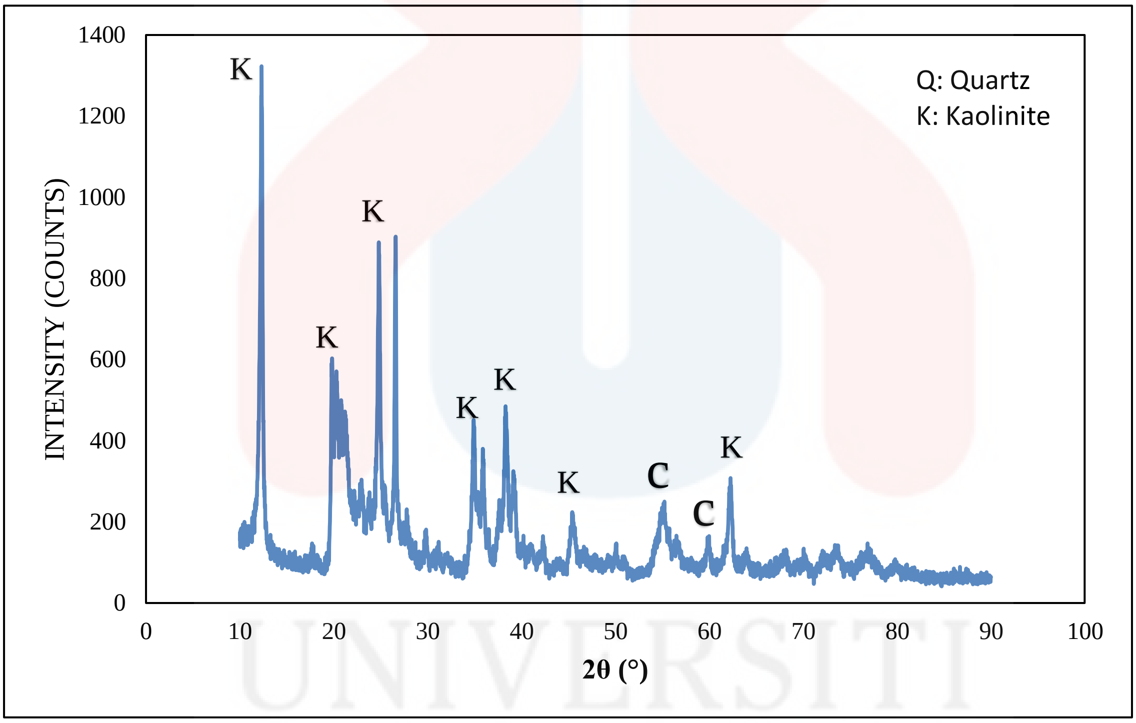


Figure 4.1(c): XRD analysis of kaolin clay

As shown in Figure 4.1(c), there were only two different stages in the raw kaolin clay: quartz and kaolinite. None of these phases existed in any other quantity. Kaolin has properties such as high refractoriness and low plasticity that make it perfect for usage in porous ceramics due to its unique mineralogical constitution and chemical composition. Kaolin is useful for a variety of uses due to its diverse properties. Compared to other ceramic materials like alumina, kaolin may be sintered at much lower temperatures due to its crystalline phase and the ensuing thermal characteristics (Hubadillah et al., 2018). The peak identified in this XRD analysis and the one mentioned in the literature review (Akbari et al., 2021) are comparatively similar, according to the observation.

4.2 Response Surface Methodology (RSM)

The impact of various operating factors and their interactions on the anticipated responses or properties of water absorption, apparent porosity, bulk density, and compressive strength of porous ceramic with the CES were statistically analysed using response surface methodology with two independent variables or operating factors. 'A' and 'B', or weight percentage (Wt.%) and sintering temperature, were the independent variables. Three levels made up the weight percentage of CES (Factor A), and the code values "-1," "0," and "1" were used to indicate each level. Concurrently, there were three levels for the sintering temperatures (Factor B), with the codes "-1," "0," and "1" designating each level. Table 4.2 shows the factors and it respective number of levels investigated in the response surface methodology statistical design. In term of responses (final properties of the porous ceramic incorporated with CES), the characteristics evaluated were water absorption, apparent porosity, bulk density, and compressive strength.

4.2.1 Experimental Design Matrix

Table 4.3 shows experimental design matrix and experimental response obtained from the total of 26 experimental runs in random order with two applications. As mentioned earlier, coding was used to be denote the level or range of each evaluated factor on a common scale of '-1', '0' and '1' for factor A and '-1', '0' and '1' for factor B. additionally, statistical analysis including model adequacy checking, analysis of variance (ANOVA), main effects and interaction plot, and contour plot were performed for each response.

Table 4.3: Experimental design matrix for central composite design

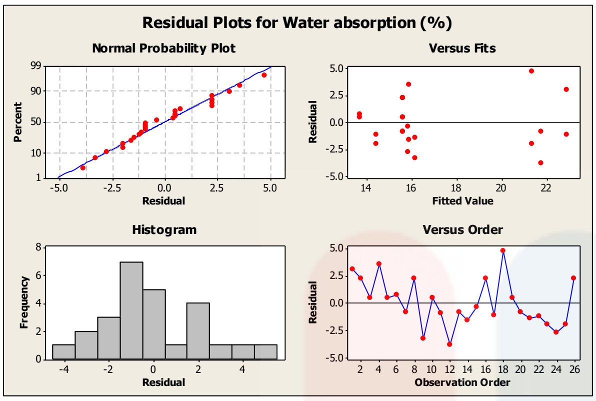
factor			**Response**			
Run Order	**Wt.% CES**	**Sintering Temperature**	**Water absorption (%)**	**Apparent Porosity (%)**	**Bulk Density (g/cm³)**	**Compressive Strength (MPa)**
1	-1	-1	25.98	40.07	1.54	75.54
2	0	0	17.89	33.08	1.85	124.78
3	0	0	16.11	26.17	1.62	113.31
4	-1	1	19.47	30.06	1.74	182.96
5	-1	-1	14.14	29.18	2.06	319.80
6	-1	-1	14.44	29.98	2.08	313.80
7	0	0	14.73	25.89	1.76	154.47
8	0	0	17.89	33.08	1.85	124.78
9	0	1	12.85	21.38	1.66	133.70
10	0	0	16.11	26.17	1.62	113.31
11	1	0	20.85	28.96	1.39	113.74
12	1	0	17.89	19.56	1.52	103.94
13	0	0	14.73	25.89	1.76	154.46
14	-1	1	14.32	23.06	1.77	182.96
15	0	-1	15.45	26.03	1.69	200.33
16	0	0	17.89	33.08	1.85	124.78
17	1	-1	21.79	47.00	1.76	76.88
18	1	1	26.10	39.37	1.51	65.78
19	0	0	16.11	26.17	1.62	113.31
20	0	0	14.73	25.89	1.76	154.46
21	0	1	14.71	23.53	1.60	136.34
22	-1	0	13.25	25.91	1.93	187.45
23	-1	0	12.46	30.14	1.90	189.29
24	0	-1	13.07	23.50	1.80	215.58
25	1	1	19.36	29.95	1.55	79.50
26	0	0	17.89	33.08	1.85	124.78

*For factor ‘A’ (wt.% of CES), ‘-1’, ‘0’, and ‘1’ represent 0 wt.%, 10 wt.%, and 20 wt.%.

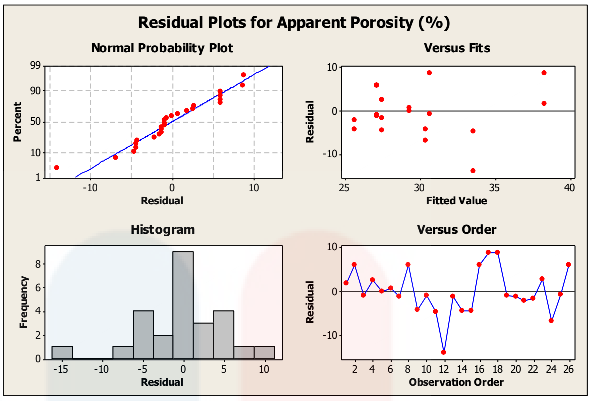
*For factor ‘B’ (sintering temperature), ‘-1’, ‘0’, and ‘1’ represent 850℃,900℃, and 950℃.

4.2.2 Model Adequacy Checking

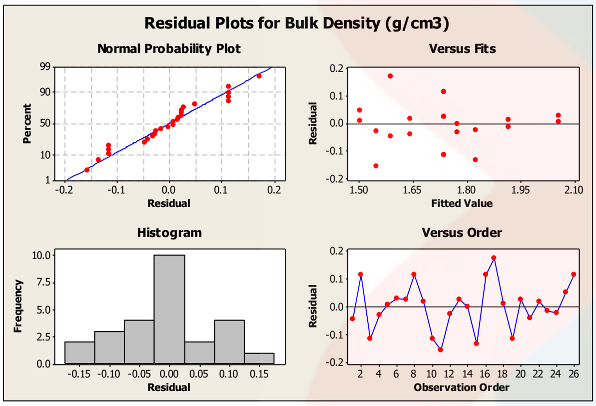
Before beginning the statistical analysis, a model sufficiency check was performed to validate a number of residual-related assumptions. In statistics, residuals are commonly described as the difference between actual and projected response values (aghahosseini et al. 2013). In this situation, the actual response value was obtained by experimentation (as indicated in table 4.1), and the anticipated response value was derived from a regression analysis equation. The sufficiency check governs three residuals' assumptions: (i) the residuals' independence assumption, (ii) the residuals' constant variant assumption, and (iii) the residuals' independence assumption. If these assumptions are correct, the regression model developed using equations should be able to adequately reflect the genuine experimental data (Tezcan et al., 2015). Several statistical residual plots, including the normal probability plot of residuals, the histogram of frequency vs. residual, the plot of residuals vs. fitted or predicted values, and the plot of residuals in time sequence or observation order, could be used to demonstrate that the three assumptions were correct. Figures 4.2(a) to 4.2(d) show the residual plots of porous ceramic with the CES for water absorption, apparent porosity, bulk density, and compressive strength.



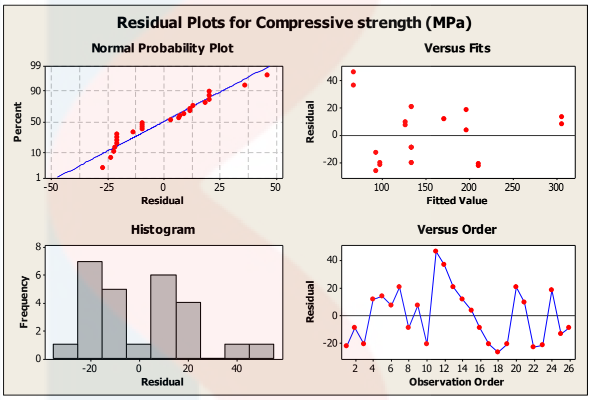
(a)



(c)



(b)



(d)

Figure 4.2: residual plot for (a) water absorption, (b) apparent porosity, (c) bulk density, (d) compressive strength that has 4 factor which is (i) Normal probability plot, (ii) Histogram of frequency versus residual, (iii) residual versus fit, (iv) Residual versus observation order of data

Except for bulk density, the majority of the residual points were distributed along a straight line as shown in the normal probability plots. This demonstrated that the data for water absorption, apparent porosity, bulk density, and compressive strength are normally distributed, meeting the first condition for determining the model's correctness (Saadat and Karimi-jashni, 2011). Furthermore, the histogram plot revealed that, except for bulk density, the histogram bar was nearly symmetrical. This provides more proof that the data on water absorption, apparent porosity, and compressive strength are distributed normally.

All histograms follow the same pattern as the standard histogram. The histogram has a dumbbell pattern, indicating that any data is normally distributed. This also supports normal distribution statistics for water absorption, apparent porosity, bulk density, and compressive strength (Prakash Talat et al., 2008). The residuals vs fitted value plots showed that the values for water absorption, apparent porosity, bulk density, and compressive strength were assigned randomly to assure consistent residual variance data. The residual against the observation order indicates that, regardless of observation order, the residual point is complete. This also implies that the residuals were independent of each other, in accordance with the third assumption.

4.2.3 Analysis of Variance (ANOVA)

Most statistical experiment analyses used analysis of variance (ANOVA) to determine the significant impact of an operating factor on the characteristics or reactions of a certain developed product or application. By observing the probability value, or more commonly known as the "p-value," following the analysis, an ANOVA could be used in this instance to determine the significant impact of the weight percentage of CES and the sintering temperature on the responses, such as water absorption, apparent porosity, bulk density, and compressive strength. The data and experimental design were analysed using the statistical tool MINITAB 16. Normally, the p-value is used to confirm the model's statistical significance. If the null hypothesis is true, the p-value probability value for a given statistical model is likely to be greater than or equal to the actual results observed (for example, the absolute mean difference between the two comparative classes of the sample). Thus, a p-value less than 0.05 is required. The p-value decreases as the evidence for rejecting the null hypothesis becomes stronger. The statistical relevance of the quadratic model was then proved (Prakash, Talat, et al. 2008).

Table 4.4(a) to 4.4(d) shows the ANOVA water absorption, apparent porosity, bulk density, and compressive strength for porous ceramics which are added with CES.

Table 4.4(a): ANOVA for water absorption of porous ceramic added with CES.

Source	P-value
Wt.% CES	0.000
Sintering Temperature	0.821
Wt.% CES* Sintering Temperature	0.288

The analysis showed that the p-value for the wt.% of CES linear factor for water absorption is less than 0.05, whereas the sintering temperature and wt.% CES*sintering temperature are higher. The p-value indicates that it is wt.% CES statistically significant at less than 0.05. It provides clear evidence against the null hypothesis; as the expectation is smaller than 0.05, the null hypothesis is correct. A lower p-value is commonly taken to indicate a stronger association between two variables. However, statistical relevance is unlikely to be relevant when it demonstrates that the difference in wt.% CES added has an influence on water absorption. A p-value lower than 0.05 is generally regarded as statistically significant. The water absorption weight percentage of the CES linear component has a p-value < 0.05. P-values for the sintering temperature and the interaction term between the weight percentage of CES and the sintering temperature exceed 0.05. According to the statistical investigation, these factors may have no statistically significant impact on water absorption. According to this statistical investigation, the weight % of CES may influence water absorption. Practical significance is not always inferred by statistical significance, and the study's context and field of interest should be considered when interpreting the findings.

Table 4.4(b): ANOVA for apparent porosity of porous ceramic added with CES.

Source	P-value
Wt.% CES	0.078
Sintering Temperature	0.165
Wt.% CES* Sintering Temperature	0.475

The investigation revealed that the p-value for the linear factor of apparent porosity and wt.% CES*sintering temperature is more than 0.05. The p-value indicates that the data is not significant. A lower p-value indicates a stronger link between the two variables. However, statistical relevance is unlikely to be relevant when it demonstrates that the difference in wt.% CES added has an effect on perceived porosity. A p-value greater than 0.05 is frequently regarded as statistically insignificant. The apparent porosity linear and interaction component has a p-value greater than 0.05. P-values for the weight percentage of CES, sintering temperature, and the weight percentage of CES interaction term are all more than 0.05. According to the statistical investigation, these elements may have no statistically significant impact on apparent porosity. According to statistical studies, the weight % of CES may affect perceived porosity. Practical significance is not always inferred by statistical significance, and the study's context and field of interest should be considered when interpreting the findings.

Table 4.4(c): ANOVA for bulk density of porous ceramic added with CES.

Source	P-value
Wt.% CES	0.000
Sintering Temperature	0.003
Wt.% CES* Sintering Temperature	0.158

The analysis showed that the wt.% CES*sintering temperature has a p-value more than 0.05, whereas the p-value for the bulk density linear factors involving wt.% of CES and Sintering Temperature is less than 0.05. The p-value indicates that the sintering temperature and weight percentage of CES are statistically significant at less than 0.05. A lower p-value is thought to suggest a stronger link between the two variables. Statistical significance, however, is unlikely to hold true in cases where evidence indicates that variations in sintering temperature and weight percentage of CES applied affect water absorption. A p-value of less than 0.05 is considered statistically significant. The linear component of the bulk density weight percentage of CES and sintering temperature has a p-value of less than 0.05. P-values for the interaction term between the weight percentage of CES and the sintering temperature are higher than 0.05. Based on the statistical analysis, this implies that these factors may not have a statistically significant effect on bulk density. This statistical study suggests that the bulk density may be affected by the interaction term between the sintering temperature and the weight percentage of CES. Statistical significance does not always imply practical relevance, and the context of the study and the specific subject of study should be considered when interpreting the results.

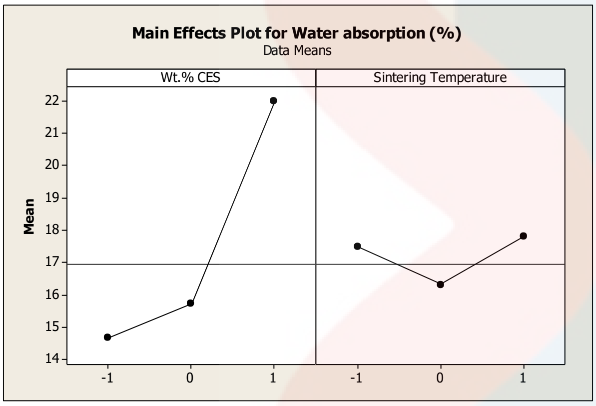
Table 4.4(d): ANOVA for compressive strength of porous ceramic added with CES.

Source	P-value
Wt.% CES	0.000
Sintering Temperature	0.000
Wt.% CES* Sintering Temperature	0.001

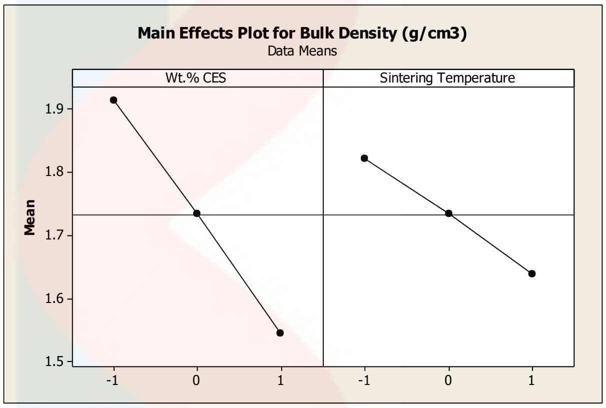
From the analysis, it was found that the p-value for wt.% of CES linear factor for compressive strength is below < 0.05 respectively. The p-value shows that it is statistically significant less than 0.05. The p-values being less than 0.05 indicate that the wt.% of CES, Sintering Temperature, and their interaction are statistically significant in relation to compressive strength This suggests that there is an effect of wt.% of CES, Sintering Temperature, and their interaction on compressive strength. the difference in wt.% of CES added has an effect not only on compressive strength but also on water absorption, apparent porosity, and bulk density. A p-value of 0.000 indicates that it is extremely unlikely that the observed result the effect of the sintering temperature, the weight percentage of CES, or their combination on compressive strength could have happened by accident. P-values higher than 0.05 indicate a lack of statistical significance in the bulk density, apparent porosity, and water absorption data. Stated differently, there is insufficient evidence in the data you have gathered to refute the null hypothesis, which holds that the independent variables have no significant impact on these properties.

4.2.4 Main Effect and Interaction Plots

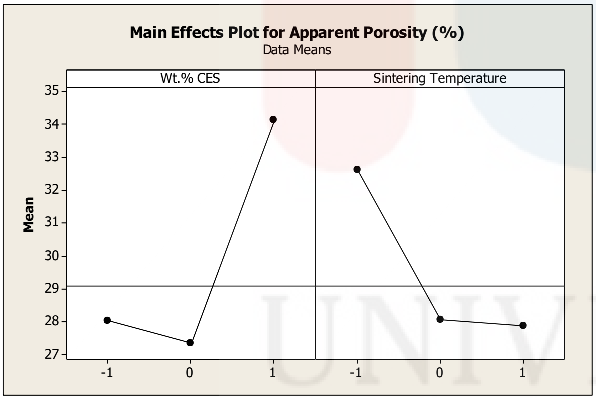
Figure 4.3(a) to figure 4.3(d) shows the main effects plot of water absorption, apparent porosity, bulk density, and compressive strength.



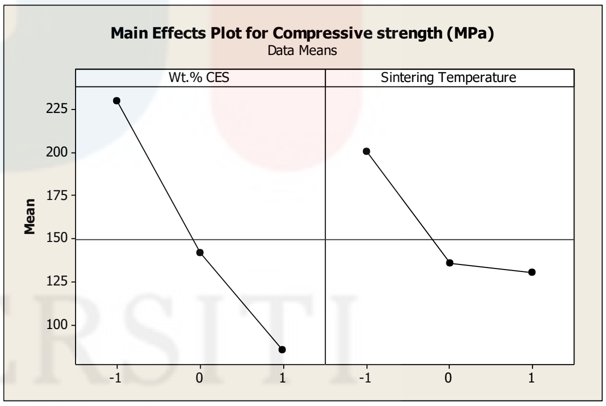
(a)



(c)



(b)



(d)

Figure 4.3: Main effect for (a) water absorption, (b) apparent porosity, (c) bulk density, (d) compressive strength

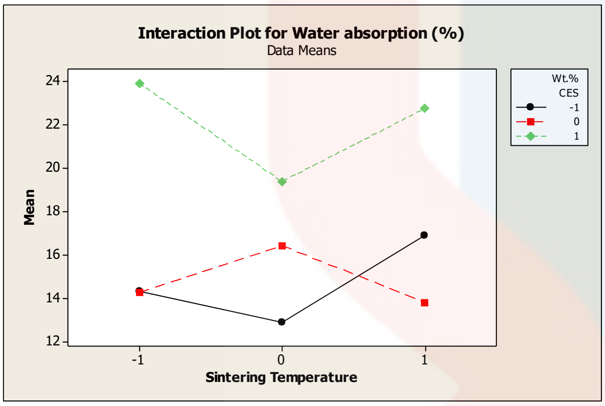
From the observation, as sintering temperature, and weight percentage of CES increase, so does the average water absorption of porous ceramic. This is probably because samples with higher weight percentages of CES have more PFA, which has a greater capacity to absorb water. Higher sintering temperatures can also produce a material that is more porous and has a greater capacity to absorb water. The plot's error bars reflect the data's variability. The fact that the error bars are so narrow suggests that the data is generally consistent. The main effects graphic demonstrates that water absorption, sintering temperature, and weight percentage CES are positively correlated. This implies that the water absorption of porous ceramic can be regulated by these factors. The figure just shows the primary impacts of sintering temperature and weight percentage of CES.

Figure 4.3(b) shows that the weight percentage of CES increases, the apparent porosity may first decrease and then increase once more. This might be because the compactness of the ceramic structure is affected by the presence of CES. It could reduce perceived porosity at lower percentages by filling up pores or gaps (Tian, Zhang et al. 2023). On the other hand, additional concentration may cause specific structural alterations that result in higher porosity. In ceramics, it is usual to see a decrease in apparent porosity as the sintering temperature increases. The impact of sintering temperature and the weight percentage of CES on apparent porosity may interplay. Apparent porosity may be optimised by a particular combination of CES concentration and sintering temperature, although the relationship may not be linear.

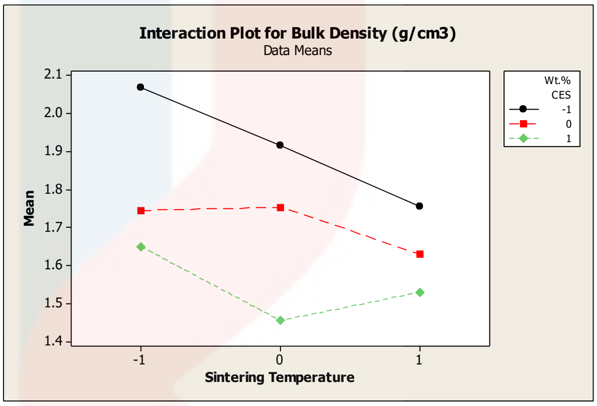
Figure 4.3(c) shows main effects plots of the pattern that decrease as wt.% CES composition rises implies that reduced bulk density is linked to increased wt.% CES. This could be because of unique characteristics brought forth by CES that influence how ceramic particles are packed, resulting in a less dense structure. The pattern that shows a decrease with decreasing sintering temperature suggests that bulk density decreases with decreasing temperature. This might be as a result of less effective particle structure and decreased densification of the porous caused by higher sintering temperatures. The increase of the temperature should make the material denser due to the energy is more to break down the bond between particles and allows the particles to bond together more tightly. However, since the wt.% of CES is increase as a PFA that lead to the decreased of the bulk density.

Figure 4.3(d) shows that the as the sintering temperature is increase, the compressive strength is decreasing. For the wt.% CES, the added CES as PFA can affected the properties of the compressive strength since the porous inside the sample could lead to the decrease of the compressive strength. For sintering temperatures, as the temperature getting higher, the compressive strength should be increase due to the sintering process that could increase the strength. however, the line for sintering temperature for the main effects plot show the decrease line which is because the added CES that lead to decreased. This shows that the significant of compressive strength is relation between sintering temperature and added CES to control the compressive strength.

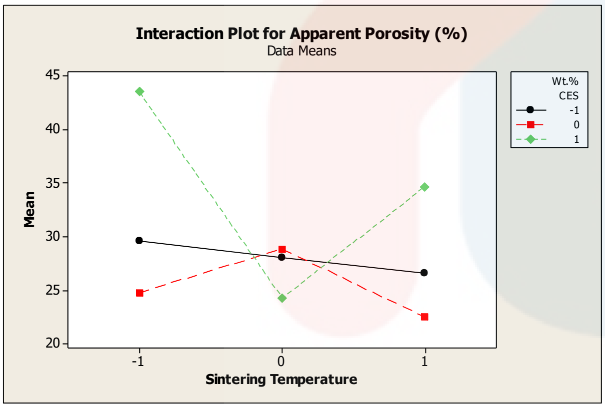
Figure 4.4(a) to figure 4.4(d) shows the interaction plot for water absorption, apparent porosity, bulk density, and compressive strength.



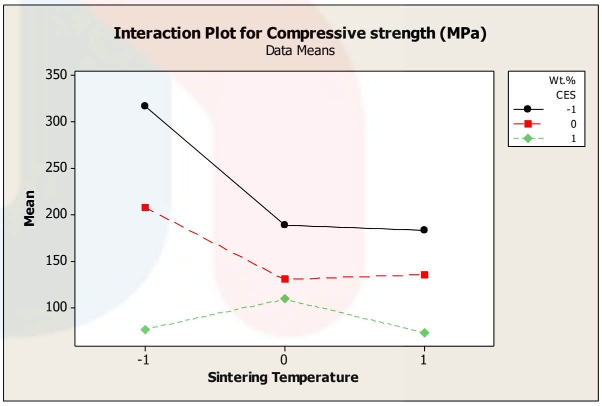
(a)



(c)



(b)



(d)

Figure 4.4 shows interaction plot for(a) water absorption, (b) apparent porosity, (c) bulk density, (d) compressive strength

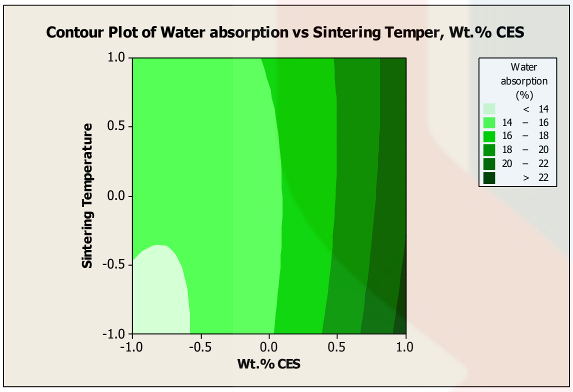
From the observation, the interaction plots at various sintering temperatures give impact of the weight percentage of chicken eggshells on water absorption may vary. there is a negative correlation between wt.% CES and water absorption. This means that as the wt.% CES increases, the water absorption decreases. This is likely due to the fact that higher sintering temperatures create a more dense material, which can absorb less water the consistent decrease in apparent porosity with increasing sintering temperature at 0% of CES (no CES added) may be attributed to the general behaviour of ceramics. Increased temperature usually leads to improved densification and packing of particles, which decreases porosity. The first rise in apparent porosity with higher sintering temperatures at 10% of CES could be ascribed to various sources, including material structural changes or improved sintering. Nonetheless, additional factors like material deterioration or changed CES interactions could result in a further drop in apparent porosity as the temperature rises.

Figure 4.4(c) shows interaction plots of the pattern that decrease as wt.% CES composition rises implies that reduced bulk density is linked to increased wt.% CES. This could be because of unique characteristics brought forth by CES that influence how ceramic particles are packed, resulting in a less dense structure. The pattern that shows a decrease with decreasing sintering temperature suggests that bulk density decreases with decreasing temperature. This might be as a result of less effective particle structure and decreased densification of the porous caused by higher sintering temperatures. The increase of the temperature should make the material denser due to the energy is more to break down the bond between particles and allows the particles to bond together more tightly. However, since the wt.% of CES is increase as a PFA that lead to the decreased of the bulk density.

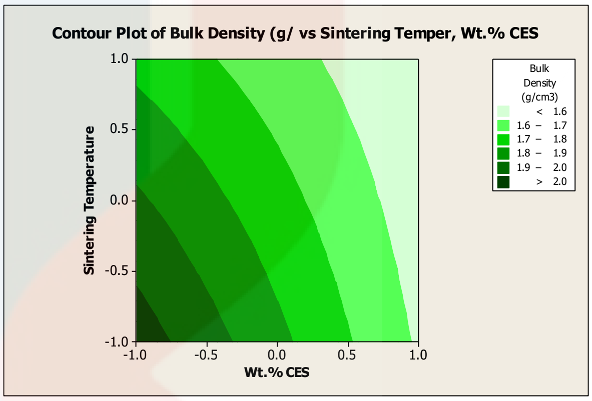
Figure 4.4(d) shows that main effects and interaction plot of compressive strength. figure shows that the decreased line as the temperature and wt.% of CES is increased. For the wt.% CES, the added CES as PFA can affected the properties of the compressive strength since the porous inside the sample could lead to the decrease of the compressive strength. For sintering temperatures, as the temperature getting higher, the compressive strength should be increase due to the sintering process that could ceramic increase the strength. however, the line for sintering temperature for the main effects plot and interaction plot show the decrease line which is because the added CES that lead to decreased. This shows that the significant of compressive strength is relation between sintering temperature and added CES to control the compressive strength.

4.2.5 Contour Plots

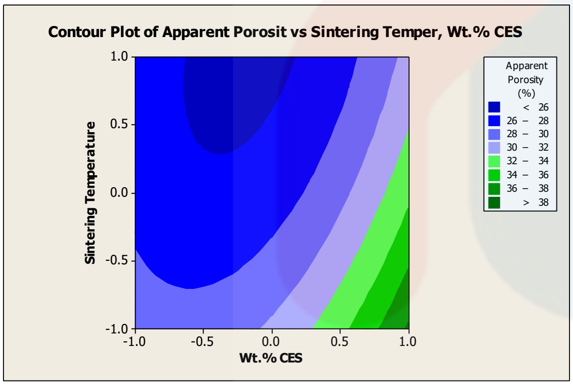
Figure 4.5(a) to figure 4.5(d) shows the contour plot of water absorption, apparent porosity, bulk density, and compressive strength.



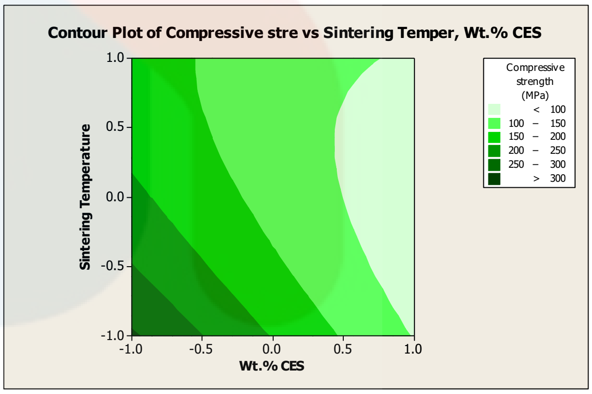
(a)



(c)



(b)



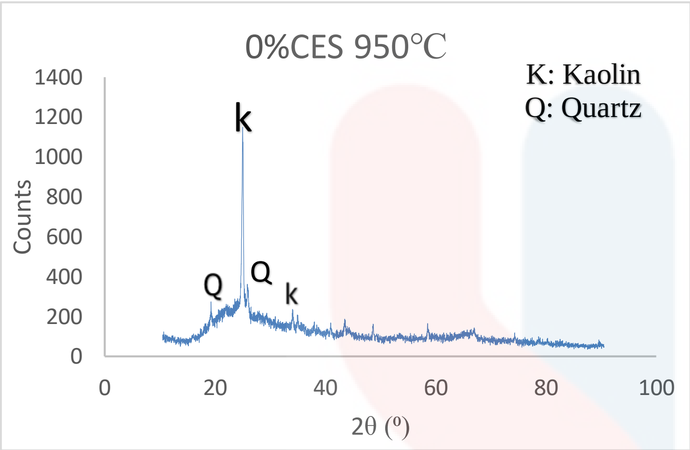
(d)

Figure 4.5 shows contour plots for (a) contour plot of water absorption, (b) contour plot of apparent porosity, (c) contour plot of bulk density, and (d) contour plot of compressive strength.

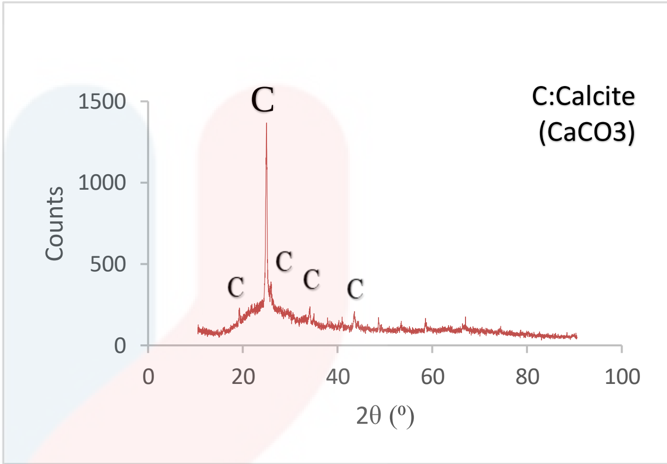
Figure 4.5(a) shows the contour plot of water absorption. From the observations, the most high-water absorption is the dark colour at '1', '-1' which is 850°C and 20% of CES. The contour plots of water absorption indicate a negative relationship between water absorption and sintering temperature. This indicates that the water absorption reduces as the sintering temperature rises. This is probably due to the material becoming less absorbent as a result of becoming denser at higher sintering temperature. Figure 4.5 (b) shows contour plot of apparent porosity. The highest porosity is the dark green on the bottom right coded '1', '-1' which is 850°C and 20% of CES. As the temperature rises, the porosity normally decreases. This means that higher temperatures produce denser materials with less void space. For bulk density, figure 4.5(c) shows that the highest bulk density is on the bottom left with dark green colour at code '-1', '-1' which is 850°C and 0% of CES. There appears to be a negative correlation between sintering temperature and bulk density. As the temperature rises, the bulk density typically falls. This means that low temperatures produce denser materials with less void space. However, the addition of CES as PFA may lower the density at high temperatures. According to the other study, higher temperatures result in denser materials. Figure 4.5(d) shows contour plots of compressive strength. From the observations, the highest compressive strength is '-1', '-1' which is 850°C and 0% of CES added. The figure shows that the compressive strength is the highest at the 850°C and 0% of CES. Due to added CES as PFA reduce the compressive strength of these porous ceramic. However, the figure shows the negative relationship between the sintering temperature and the wt.% at 0% CES. from others research shows that the higher the temperature. The higher the compressive strength. Overall, all the results is still relevant to be consider as the normal statistic.

4.3 XRD analysis of CES mixture incorporates with Kaolin clay.

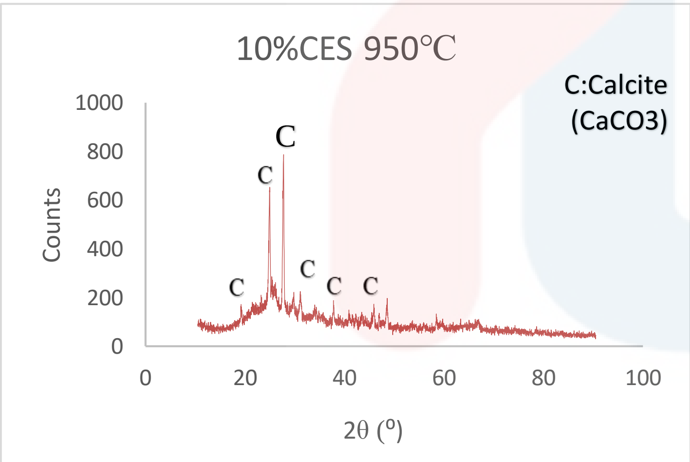
XRD analysis is used to identify the main crystalline phases in porous ceramics containing chicken eggshells by various compositions and sintering temperatures. In this analysis, compositions of 10 wt.% CES with 850°C, 10 wt.% of CES with 950°C, 20 wt.% of CES with 950°C, and 0 wt.% of CES with 850°C were used. The goal was to determine the crystalline phase under various compositions and sintering temperatures. Figures 4.6(a) to 4.6(d) depict peaks of porous ceramic combined with chicken eggshells, respectively.



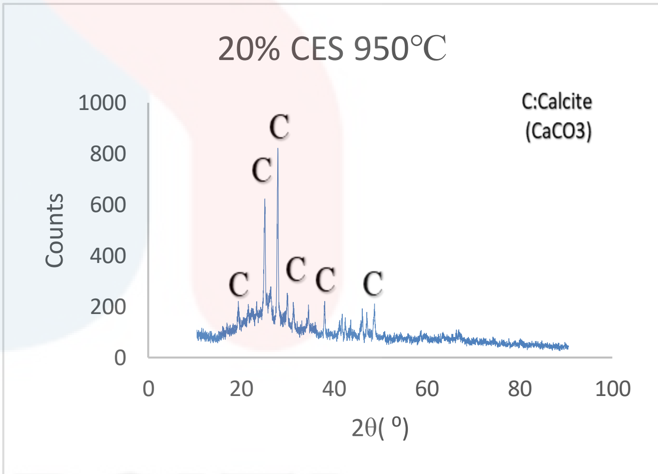
(a)



(b)



(c)



(d)

Figure 4.6(a) to 4.6(d) shows analysis XRD of CES incorporated with kaolin clay: (a) 0 wt.% of CES at 950℃, (b) 10 wt.% of CES at 850℃, (c) 10 wt.% of CES at 950℃, and (d) 20 wt.% of CES at 950℃

Figure 4.6(a) shows exhibited just two distinguishable phases: kaolinite and quartz. Neither of these phases was present in any other amount. Kaolin has features such as high refractoriness and low plasticity for porous ceramics due to its chemical composition and the mineralogical properties it possesses. These properties allow kaolin to be used in these applications. When compared to other ceramic materials like alumina, kaolin's crystalline phase and subsequent thermal characteristics allow for significantly lower temperature during the sintering process. From observation, the peak present in this XRD analysis is quite comparable with the XRD analysis that is mentioned in the literature review.

Figure 4.6(b) to 4.6(d) shows that CES incorporates with kaolin clay make different peak for different composition and temperature. Figure 4.6(b) shows the highest peak counts which mean the intensity will impact the physical and mechanical properties of the porous ceramic. This determines the primary crystalline phase present in CES powder at some sintering temperature and composition. The less common crystalline distinct phase are diopside, sanidine, phyroxene-ideal, quartz high and megakalsilite. The crystalline phases identified by XRD analysis of CES powder were the same as those identified by a previous literature survey. The presence of phosphorus in the CES powder contributed to the formation of whitlockite in the powder. On the other hand, the formation of diopside may come from the reaction between the CaO with other oxides such as MgO and SiO. The presence of several oxides in the crystalline compound such as SiO2O3, Fe2O3, CaO, Na2O and MgO may act as a fluxing agent, which is played a role in lowering the sintering temperature and influence the densification of sintered porous ceramic.

However, the quantity of sample utilized in the XRD analysis that may have generated a lot of background noise may be the reason of the existence of other crystalline phase that are different from the research indicated in the literature review. This lead to the difficulty of distinguishing the crystalline phase that is present in the CES powder. However, XRD analysis was also performed on unprocessed kaolin to identify the predominant crystalline from the materials.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

At the end of this project, possibility of reusing and utilizing CES as PFA in porous ceramic production was shown to be tremendous. The Response Surface Methodology (RSM) Statistical Approaches significantly can be used to optimize CES added and sintering temperature for the development of sustainable clay-based ceramic incorporated with CES. Several conclusions were made based on the result presented and discussed in Chapter 4:

a) The physical and mechanical properties were highly significant and proven for the clay-based ceramic incorporated with CES which the result of statistical analysis including water absorption, apparent porosity, bulk density, and compressive strength.

b) CES is suitable for porous ceramic based on this study. The increase of weight percentage of CES in the porous ceramic decreases the compressive strength and increases the porosity. The suitability of clay-based ceramic incorporated with CES were attained at 10wt.% of CES and sintering temperature of 850°C.

5.2 Recommendations

Further study on the thermal conductivity test could be carried out to evaluate its thermal insulating properties.

a) The compressive strength of porous ceramic can be improved, body formulations can be added by including flux material (feldspar as example) or filler (silica as example).

b) The further addition weight percentage of CES beyond 20% can be carried out to check the trend of porosity and compressive.

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