

**OPTIMIZATION OF PLASTER OF PARIS AS PHASE CHANGE MATERIAL
CONSIDERING ITS THERMAL RESISTIVITY**

**BY
ADJARHO GODSTIME
ENG1905406**

**DEPARTMENT OF INDUSTRIAL ENGINEERING
FACULTY OF ENGINEERING,
UNIVERSITY OF BENIN,
BENIN CITY.**

SUPERVISED BY

ENGR. DR. B. O ERHUNMWUNSE

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CERTIFICATION

This is to certify that this project, **OPTIMIZATION OF PLASTER OF PARIS PROCESS PARAMETERS AS A PHASE CHANGE MATERIAL CONSIDERING ITS THERMAL RESISTIVITY** was carried out by **ADJARHO GODSTIME** with matriculation number **ENG1905406** in partial fulfilment of the award of Bachelor of Engineering (B.Eng) in the department of Industrial Engineering, University of Benin.

Engr. Dr. B. O. ERHUNMWUNSE
(Project Supervisor)

Date

Engr. Dr. E.M Etuk
(Project coordinator)

Date

Prof. R.O Edokpia
(Head of Department)

Date

DEDICATION

I appreciate almighty God along side both of my parents, my supervisor, and all my friends who have been with me through this process for their endless encouragement and patience.

ACKNOWLEDGEMENT

First of all, I would like to give my sincerest gratitude to my supervisor Dr. ERHUNMWUNSE, For his constant support and advising. He motivated and assisted me in completing this degree without his continuous help, this work would not have been achieved.

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ABSTRACT

Plaster of Paris (POP), known for its affordability and accessibility, exhibits potential as a phase change material (PCM) due to its capacity to store and release thermal energy during phase transitions. However, its efficacy hinges on various factors including particle size, mixing ratio, curing time, and temperature. This study delves into optimizing POP process parameters to enhance its suitability as a PCM, with a focus on thermal resistivity properties. Through systematic experimentation and analysis, we aim to pinpoint the ideal combination of process parameters to bolster thermal resistivity for applications in thermal energy management.

Our research commenced with the fabrication of POP molds, where we tailored diverse formulations by adjusting ratios of POP cement, fiber, and water to achieve specific attributes. Subsequent exposure to controlled heat allowed us to meticulously gauge thermal resistivity using precise thermotesting equipment. Analysis of these data enabled us to derive meaningful insights into material performance under varying conditions.

By leveraging response surface methodology and statistical analysis, our investigation pinpointed the optimal blend of water, fiber, and POP cement for maximizing thermal resistance in fiber-reinforced POP cast mixtures. The development of a predictive mathematical model facilitates accurate thermal performance forecasting across different process parameter configurations, facilitating informed decision-making in material selection and application.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND TO THE STUDY

Plaster of Paris, commonly known as POP, has a significant history in Nigeria, particularly in the construction and art sectors. The use of gypsum-based materials in construction dates back many decades. POP gained popularity due to its versatility, ease of use, and favorable properties for various applications.

- **Construction:** In the construction industry, POP is often used to produce decorative elements, moldings and surface finishes. It serves as a reliable material for creating complex designs on walls and ceilings.
- **Crafts:** POP is the preferred material of artists and craftsmen in Nigeria. Due to its malleability and quick setting properties, it allows for detailed sculptures, shapes and artistic creations.
- **Medical Applications:** In some cases, POP has been used in orthopedics to make plaster casts due to its ability to cure quickly and provide a rigid structure.

The introduction of POP in Nigeria reflects its adaptability in various fields and contributes to the functional and aesthetic aspects of construction and art.

Gypsum (POP) is used as a brittle material in various engineering applications, but its low deformability and susceptibility to cracking and instability have limited its use. However, the incorporation of natural fibers as reinforcement has shown promise for improving the mechanical properties of POPs. Kenaf bast fibers and sodium lauryl sulfate (SLS) modified with the Control System Tank Retting (CSTR) method were successfully used to reinforce the POP, resulting in higher tensile strength and stability. In addition, fiber-reinforced polymers (FRP) were

investigated as an alternative reinforcement for concrete structures and various products and configurations were analyzed and compared. Paper pulp has also been investigated as a reinforcement for gypsum, improving its flexural properties and reducing weight, making it suitable for lightweight construction applications. In addition, burlap and glass cloth reinforcements have been studied for fibrous gypsum, while high density gypsum and irregular glass mats have greater flexural strength and durability. Overall, these studies highlight the potential of various fiber reinforcements to improve the properties and performance of gypsum and cementitious materials.

Phase change materials (PCM) can be used for thermal energy storage with the aim of improving the energy efficiency of buildings. Recently, gypsum boards with incorporated paraffin-based PCM mixtures have also been brought onto the market. In the high-temperature environment created by a fire, paraffins, which have a relatively low boiling point, can evaporate and pass through the porous structure of the gypsum board into the fire zone, where they can ignite and thus negatively affect the fire resistance properties of the building. To assess the fire protection behavior of these building materials, an extensive experimental and computational analysis is carried out. The fire behavior and the most important thermophysical properties of PCM-treated gypsum boards are examined using a variety of standard tests and devices (scanning electron microscopy, thermogravimetric analysis, cone calorimeter). The results obtained are used to develop a special numerical model, which is implemented into a CFD code. The CFD simulations are validated using measurements obtained in a cone calorimeter. Additionally, the CFD code is used to simulate an ISO 9705 room exposed to fire conditions. This shows that the addition of PCM can negatively affect the fire safety of a plasterboard-clad building.

Drying processes play a fundamental role in various industries and contribute to food preservation, material production and product finishing. Although these processes are essential, challenges remain to achieve optimal efficiency, sustainability and profitability. As the industry evolves and technology advances, the need for a comprehensive understanding and improvement of drying methods becomes increasingly important.

Today, increasing energy consumption in the construction sector raises concerns related to energy inefficiency and excessive greenhouse gas (GHG) production. This means that the construction sector consumes around 40% of the primary energy produced annually in the European Union and is responsible for the production of almost 25% of greenhouse gases (GHG). Another important issue is related to the increase in fuel prices and the shift away from conventional energy sources, including the burning of fossil fuels, which are considered one of the main sources of excessive emissions. Although several mitigation strategies have been developed to improve energy performance, the results and observations obtained cannot be considered satisfactory with regard to the goals of sustainable development. Currently, maximum insulation is achieved through the use of traditional insulation materials such as polystyrene, mineral wool or polyurethane foam. Potential. Therefore, the thermal stability of buildings can only be improved through advanced energy renovation measures or a combination of several strategies. The recent development of innovative insulation materials such as vacuum insulated panels, gas-filled panels or aerogels represents a very efficient method for producing and characterizing new plasters with improved thermal energy storage perform.

Drying processes are of utmost importance in various industries and play a crucial role in the preservation, production and quality improvement of various materials. Several key aspects highlight the importance of drying processes:

- Food Industry: Preservation: Drying is often used to extend the shelf life of foods by reducing moisture content, preventing microbial growth and inhibiting enzymatic reactions.
- Quality Improvement: Controlled drying improves the quality, taste and nutritional value of certain foods.
- Textile and Garment Manufacturing: Product Finishing: Drying is an integral step in the textile industry for finishing fabrics and garments to ensure they meet desired specifications and standards.
- Energy efficiency: Optimized drying processes contribute to energy efficiency and cost-effectiveness in textile production.
- Pharmaceuticals: Stability and shelf life: Drying is essential for pharmaceuticals to ensure the stability and long shelf life of drugs and medicines.
- Controlled Conditions: Precise drying conditions are essential to maintain the effectiveness and safety of pharmaceutical formulations.
- Chemical Industry: Product Formulation: Drying is used in the chemical industry to formulate powders, granules and other dry forms of chemicals.
- Process efficiency: Efficient drying processes contribute to the overall efficiency of chemical production processes.
- Pulp and Paper Industry: Material Conditioning: Drying is used in paper production to condition the pulp and ensure the proper formation of paper sheets.
- Water Removal: Controlled drying helps to remove water from paper products, thereby affecting their final properties.

- **Biotechnology and Research: Sample Preservation:** Drying is used in biotechnology to preserve biological samples such as tissues and cultures for long-term storage.
- **Analysis Techniques:** Dried samples facilitate various analysis techniques and ensure accurate and reproducible results.
- **Material production and construction: Material processing:** Drying is an essential part of the production of building materials such as cement, ceramics and wood products.
- **Quality Control:** Proper drying guarantees the quality and durability of building materials.

1.2 STATEMENT OF PROBLEM

The optimization of Plaster of Paris (POP) as a phase change material (PCM) poses a wonderful undertaking because of the dearth of complete research addressing its thermal conductivity on this particular application. Despite the significant use of POP in production and scientific fields, its capacity as a PCM for green thermal power garage stays in large part untapped. The cutting-edge undertaking targets to cope with this hole via way of means of investigating and optimizing the manner parameters concerned in utilizing POP as a PCM, with a number one attention on improving its thermal conductivity. Addressing this hassle is vital for advancing the sector of phase change material, because the optimization of POP can provide an environmentally friendly, cost-effective, and effectively to be had answer for thermal power garage. The a success optimization of manner parameters on this undertaking might now no longer simplest make contributions to the medical expertise of POP as a PCM however additionally pave the manner for sensible packages in sustainable power structures and different rising technology reliant on green thermal management.

1.3 AIM AND OBJECTIVE

1.3.1 Aim

This research aims to optimize plaster of Paris process parameters as phase change material considering its thermal conductivity and efficiency when used as an insulation for a DC power dryer.

1.3.2 Objectives

1. Identify relevant process parameters of pop mixture
2. Produce pop mound using the selected range of process parameters
3. Subject the pop mound to heat and observe the temperature
4. Study the individual and combined interactions of the process parameter on the pop mound external temperature
5. Analyze the recorded temperature using response surface methodology

1.4 SCOPE OF THE STUDY

This study is limited to the optimization of plaster of Paris process parameters as phase change material considering its thermal resistivity and efficiency when used as an insulation material for a DC dryer.

1.4.1 RESEARCH METHOD

The following methods will be used to carry out the project;

- Procurement of materials (POP gypsum fibers, water and molds)
- Mixing of aggregates.
- Preparation of molds
- Application of separator (mixture of soap and peanut oil) to facilitate withdrawal of the melted sample from the mold

- Pouring
- Laboratory tests
- Calculations and analysis of results.
- Conclusion and recommendation

1.4.2 SIGNIFICANCE

The significance of the project titled "optimization of plaster of Paris (POP) process parameters as phase change material (PCM) for considering its thermal conductivity" is multifaceted, encompassing technological innovation, energy efficiency, and sustainability within the context of drying processes. The project aims to push the boundaries of conventional drying systems, setting the stage for advancements in industrial drying technology. The introduction of advanced thermal control through PCM in the drying process is expected to positively impact the quality of POP products.

1.5 LIMITATION

- Despite the potential of the project, certain limitations are inherent and may affect the completeness of the study:
- POP Industry Specificity: The study findings and recommendations may be more directly applicable to the Plaster of Paris (POP) industry. Extrapolating results to other industries may require additional research and validation.
- Climate Dependence: The performance of the proposed system can be influenced by external climatic conditions. Temperature and humidity fluctuations can affect the efficiency of the PCM and therefore the overall performance of the DC dryer.

- **Material Compatibility:** The effectiveness of PCM in the drying process depends on its compatibility with POP materials. Certain material interactions can limit the full potential of the thermal control mechanism.
- **Scaling Challenges:** Scaling the proposed system to industrial production levels may involve challenges that are not fully explored in this project. Issues related to larger manufacturing and implementation processes are outside the immediate scope.
- **Economic factors:** The economic analysis within the project is based on theoretical calculations and assumptions. Real economic factors such as market fluctuations and unexpected costs may affect the actual feasibility of the proposed system.
- **Test duration:** The project plan for experiments and evaluation may impose restrictions on the duration and variety of tests carried out. Long-term performance and durability aspects may require further investigation.
- **PCM Availability:** The availability and cost effectiveness of the chosen phase change material may influence the feasibility of widespread industry adoption.

CHAPTER TWO

LITERATURE REVIEW

The call Plaster of Paris (POP) had its origins from the reality that it became substantially mined from Montmartre in Paris district. But its use predates the commercial revolution, they were observed at the insides of pyramids. The want to immobilize the fracture to save you ache and deformity and all of the even as permitting mobilization has been the perennial trouble in orthopedics. Splints made from bamboo and timber sticks had been used within-side the historic instances however they could not be relied upon to keep the reduction. More substances had been attempted like wax, starch, cardboard however all led to failure. This became the time POP became starting to be utilized in production and with the aid of using sculptors, surgeons watching its houses stumble on the concept of the use of it in orthopedics. Patients with fractures within-side the lengthy bones of leg had been located in lengthy slender timber containers and the gaps packed with POP. This became but cumbersome and options had been sought. The concept of incorporating POP in bandages became stumble on with the aid of using surgeons, Antonius Mathijssen and Nikolai Ivanovich Pirogue within-side the 1850's.

Plaster of Paris is calcined gypsum (roasted gypsum), floor to a best powder through milling. When water is added, the extra soluble shape of calcium sulphate returns to the exceedingly insoluble shape, and warmth is produced $[2 (\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}) + 3\text{H}_2\text{O} \rightarrow 2 (\text{CaSO}_4 \cdot 2\text{H}_2\text{O}) + \text{Heat}]$. The putting of unmodified plaster begins off evolved approximately 10 min after blending and is entire in approximately forty five min; but, the forged isn't absolutely dry for seventy two h.⁷

The largest benefit of POP is the belongings that permits itself to moulding. This permits POP to be contoured in keeping with the limb that its miles carried out to. It is adequately inflexible and

permits immobilisation to permit recovery to take place. However it isn't without its headaches. Prolonged immobilisation in a POP makes the pores and skin beneath the forged vulnerable. The useless pores and skin isn't eliminated and results in scaling. Other headaches like ulceration, maceration, itching can result. Even burns may be as a result of the exothermic reaction. Staphylococcal contamination of the underlying pores and skin can bring about dermatitis. Cast syndrome, related to frame jacket casts, entails obstruction of the 1/3 part of the duodenum from duodenal constriction as a result of stretching of the advanced mesenteric vessels. The signs are nausea, vomiting, fever, and electrolyte imbalance. The maximum dreaded problem but is compartment syndrome and the ensuing sequelae Volkmann's Ischaemic contracture. The nerve palsies have additionally been related to bad plaster techniques. The different persistent problem is the "fracture disease" that consequences from extended immobilisation, main to osteoporotic bones and stiff joints. Complications while casts are utilized in fracture remedy are lack of reduction, malalignment, behind schedule and non-union. Localised hypertrichosis has additionally been pronounced as uncommon problem of inner fixation and POP application.⁸

Recently parameters like forged index and hole index had been evolved that are expecting forged failure. Nine cast index is the ratio among the inner diameter of the forged at the lateral view to the internal diameter of the forged on AP view. Gap index is the gap among plaster and pores and skin measured as a ratio to internal diameter of the plaster. Higher forged and hole index had been related to better failure rate. However maximum of the headaches may be averted through adhering to an excellent plaster technique. They encompass right padding specially of the bony prominences, fending off casts while the limb is swollen, retaining useful function of joints and stopping stress factors while moulding the forged. To save you compartment syndrome, care

need to be taken in youngsters and in sufferers with tender tissue injury (consisting of burns), more than one trauma, paralysis or paresis, head injury, or altered sensorium (because of medications, substance abuse, or psychosis). Evaluation of neurovascular fame and recording of abnormalities are essential.

The elements that reason thermal burns are a dip water temperature of >75F (24 C), use of extra than 8 layers, and use of a pillow (insufficient ventilation). Plaster residue within side the dip water did now no longer boom the exothermic reaction. Moisture at the outdoor of the forged reduced the temperature of the plaster.¹⁰ According to Halanski et al, the floor of the forged changed into 2.7 ± 1.9 °C cooler than the inner temperature. A dip water temperature of <24 °C did not result in a temperature high enough to cause burns, regardless of the number of layers. A dip water temperature of >50 °C, a twenty-four-ply forged thickness, use of a plastic pillow, over-wrapping of a curing plaster forged with synthetic, and use of a splint folded on itself had been related to temperatures inflicting burns.

2.1 Varieties of plaster of Paris

- Gypsum: This type of plaster is mainly composed of calcium sulfate hemihydrate, a white powder. Gypsum is made by heating gypsum to 120 to 180 °C. When the temperature exceeds 392 degrees Fahrenheit, anhydrite forms. The natural form of gypsum is the mineral bassanite. However, when dry gypsum powder is mixed with water, it rehydrates and turns back into gypsum.

Here are some common uses of plaster of paris:

- It is used to repair broken bones in medical facilities. Dentists use it to create dental models.
- It is used to make toys, jewelry, cheap jewelry, cosmetics, chalk, and statue molds.
- Acts as a fireproof material.
- Used to seal air spaces in laboratory equipment.

- Clay plaster: Clay plaster, a mixture of clay, sand, water and plant fibers, was widely used in ancient times and in utopian villages of the early 19th century. It was usually used for the construction of interior walls.
- Lime plaster: Lime plaster is a type of plaster made from a mixture of calcium hydroxide and sand. This gypsum begins to harden upon contact with carbon dioxide in the atmosphere, converting calcium hydroxide into calcium carbonate.
- Cement plaster: Cement plaster is a mixture of suitable gypsum, Portland cement, sand and water. This type of plaster was first introduced to the United States around 1909 and was known as “inflexible plaster.” It is primarily used in masonry to achieve a smooth surface in building construction. A layer of plaster is often applied to the cement plaster. Cement plaster is known for its strength, hardness, quick setting time and durability.
- Heat-resistant plaster: This type of plaster is often used for cladding walls and chimneys and as fireproofing on ceilings. Nowadays, heat-resistant plasters replace traditional gypsum plasters to withstand high temperatures.

Gypsum is often referred to as gypsum and has the chemical formula $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$.

Here are some notable properties of Gypsum

- It is usually white in color and comes in powder form.
- When mixed with water, it forms gypsum crystals and solidifies.
- The exothermic setting process can be catalyzed by sodium chloride. However, the process can be slowed or delayed by substances such as alum or borax.
- When heated to 473 K, gypsum forms anhydrous calcium sulfate, also known as burnt gypsum.

Additional Properties of Gypsum

Gypsum, another name for gypsum, is a dry white powder that can be easily worked with metal tools or even sandpaper. It can be made into different shapes according to your needs. However, it should be noted that this material is not very strong and may require external reinforcement when used in large quantities. It is often mixed with water to form a quick-setting paste. An American used gypsum plaster combined with fibers to create a ceiling panel with long-lasting strength (Miller, 1930).

Gypsum (POP) has been used as a brittle material in various engineering applications, but its low deformability and susceptibility to cracking and instability have limited its use. However, the incorporation of natural fibers as reinforcement has shown promise for improving the mechanical properties of POPs. Kenaf bast fibers modified by the Control System Tank Retting (CSTR) method and sodium lauryl sulfate (SLS) were successfully used to reinforce POP, resulting in higher tensile strength and stability. In addition, fiber-reinforced polymers (FRP) have been investigated as an alternative reinforcement for concrete structures, analyzing and comparing various products and configurations. Paper pulp has also been investigated as a reinforcement for gypsum, improving its flexural properties and reducing weight, making it suitable for lightweight

construction applications. In addition, burlap and glass cloth reinforcements for fiber plaster have been studied, with high density gypsum and irregular glass mats exhibiting increased flexural strength and durability. Overall, these studies highlight the potential of various fiber reinforcements to improve the properties and performance of gypsum and cementitious materials.

Benefits of Plaster of Paris (POP) as a Phase Change Material (PCM) for Drying:

While POP is traditionally known for its applications in construction and art, it also has beneficial properties when considered as a phase change material (PCM) for drying processes.

- **Heat Storage Capacity:** POP has a high heat storage capacity, which allows it to effectively store and release heat energy. This property is crucial in drying processes where controlled temperature is essential.
- **Fast Setting:** POP can set quickly when mixed with water. This feature is beneficial in PCM applications as it ensures a quick response to temperature changes during the drying cycle.
- **Compatibility with Various Substrates:** POP can be easily applied to various surfaces, making it suitable for use in various drying applications, including those involving different materials or products.
- **Cost Effectiveness:** POP is generally inexpensive and widely used. Its affordability makes it an attractive option for industries seeking cost-effective PCM solutions for drying processes.

- **Malleability:** The malleability of POP allows the creation of specific shapes and configurations. This can be beneficial when designing drying systems that require custom PCM configurations or vessels.
- **Non-toxic and safe:** POP is considered non-toxic and safe to use. This is particularly important in applications where the PCM may come into contact with food or other sensitive materials during the drying process.
- **Compatibility with sustainable practices:** Gypsum, the main component of POPs, is abundant and can be sustainably sourced. This is in line with growing trends in industries seeking environmentally friendly materials and processes.
- **Insulating Properties:** POP exhibits some insulating properties that can be beneficial in applications where maintaining a constant temperature is essential to the drying process.

Given these advantages, exploring the use of POPs as a phase change material in drying applications represents an interesting opportunity to exploit its inherent properties beyond its traditional uses in Nigeria.

2.1.1 PHASE CHANGE MATERIALS

A phase change material (PCM) is an ingredient that liquefies and solidifies at a certain temperature and has a high heat of fusion. It can store and release large amounts of energy. Heat is absorbed or released as the material changes from the solid to the liquid phase and vice versa; Therefore, PCMs can be referred to as latent heat storage (LHS).

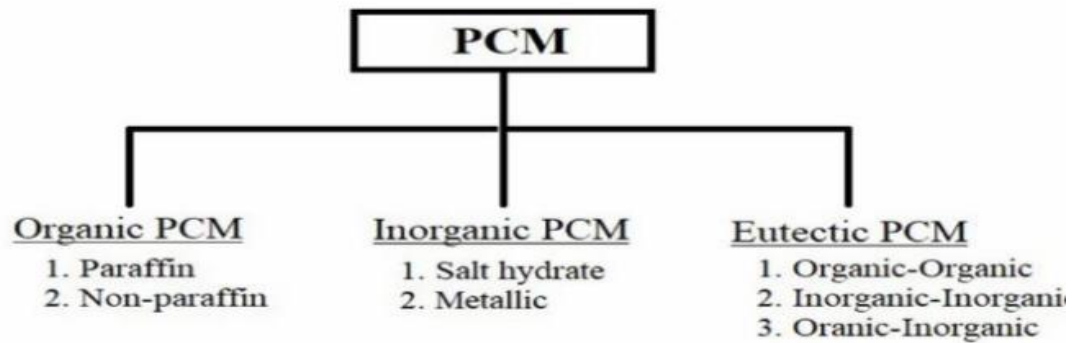


Figure 1.1: Classification of PCM

Major PCMs are classified as indicated above. The selection of PCM is done by meeting several criteria, such as: economic, thermal, kinetic, chemical, etc. When selecting PCM for any application, some important properties should be taken into account, such as:

- No corrosiveness.
- No deterioration after a long number of cycles, no leaks.
- Non-toxic and flammable.
- Melting temperature.
- High latent heat of fusion per unit volume.
- High specific heat.

2.2.6 Challenges and Future Directions

Despite its versatility, there are challenges such as brittleness and limited tensile strength. Future research directions could include exploring additives, new manufacturing techniques, and developing composite materials to address these limitations.

CHAPTER THREE

METHODOLOGY

This is the total approach selected to successfully carry out this study in a comprehensive and articulate way. This study is focused on the experimental study of a Plaster of Paris cast with the aim of getting an optimal mixture based on its thermal resistance, use scientific design of experiment and expert system to carry out statistical analysis and develop a mathematical model. The methodological steps employed to obtain and analyze the experimental data for this study are as follows;

- i. Design of experiment
- ii. Identification of input parameters range
- iii. Materials and equipment
- iv. Experimental data collection
- v. Experimental data analysis using response surface methodology (RSM)

3.1 Design of experiment.

This deals with the analysis and interpretation of controlled tests to evaluate the factors that controls the value of a parameter or a group of parameters. In this study, it will be used for the development of a scientific approach for optimal selection and combination of experimental input variables as it affects our targeted response using computerized software like MATLAB, JMP, SPSS, MINITAB and DESIGN EXPERT. In this study, design expert was selected because of its comparative testing ability, characterization, optimization and a robust parameter design. An experimental design is used to collect the data for proper polynomial approximations, there are various types of experimental designs e.g. Latin hyper cube design, Tagucchi design, D-

Optimal design, Factorial design and Central Composite designs. In this study, central composite design was used.

3.2 Identification of Input Parameters Range.

The key parameters used to be considered in this work are Plaster of Paris cement, water and fiber. The range of the process parameters used were obtained from relevant literatures as tabulated below.

Table 3.1: Process parameters and their levels

PARAMETERS	UNITS	MINIMUM VALUE	MAXIMUM VALUE
POP Cement	grams	26.60	33.40
Water	grams	13.30	16.70
Fiber	grams	6.70	8.30

3.3 Materials and equipment

The following materials and equipment would be used to effectively and successfully carry out this study: POP cement, water, fiber, wooden mold and a Vernier caliper and a (*indicate instrument for measuring thermal resistance*). The POP cast was made from a mixture of the stated process parameters on a wooden mold. The picture of the wooden mold for the POP cast is shown in Plate 3.1.

Plate 3.1: Wooden mold for POP casting

An easy to read thermal conductivity and resistivity meter (TLS-100 Thermtester) was used to measure the thermal resistance of the POP cast after being subjected to heat as shown in Plate 3.2.

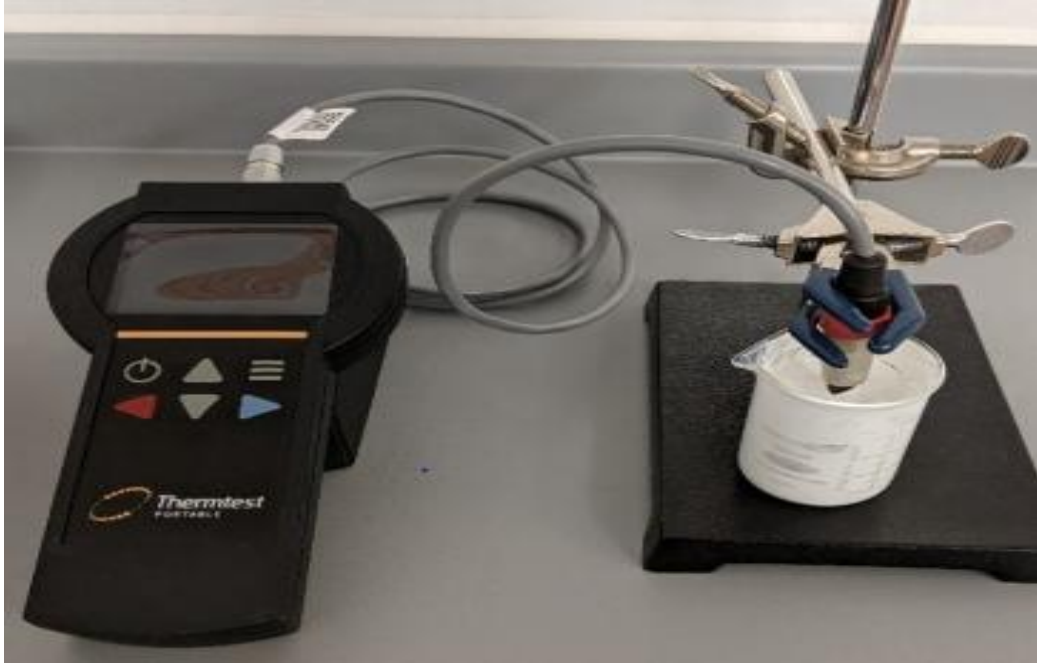


Plate 3.2: TLS-100 Thermtester <https://thermtest.com/tls-100>

3.4 Experimental Data Collection

The input parameters (POP cement, water and fiber) were used as factors for the design matrix, the response which is the output parameter was recorded from the POP cast samples make up the experimental matrix that was used as the study data. The central composite design (CCD) matrix was used to develop a statistical design of experiment (DOE). Design expert software version 13.05 was employed to carry out the experimental design. The CCD matrix generated an experimental design matrix having six (6) center points (n_o), six (6) axial points ($2n$) and eight (8) factorial points (2^n) which when imputed into Equation 3.1 resulted in twenty (20) experimental runs. The CCD matrix is presented in Table 3.2. The total number of experimental runs as generated by the CCD is given as: (Owolabi et al. 2018).

$$N = 2^n + n_o + 2n \quad \dots\dots(3.1)$$

where;

N: is the number of experimental runs based on CCD, 2^n : is the number of factorial points, n_0 : is the number of center points, $2n$: is the number of axial points and n : is the number of variables.

Table 3.1 shows the experimental matrix generated by the CCD.

Table 3.2 Central Composite Design (CCD) Experimental Matrix Factors

	Factor 1	Factor 2	Factor 3
Run	A:POP Cement	B:Water	C:Fiber
	Grams	Grams	Grams
1	28	14	7
2	28	14	8
3	30	13.3	7.5
4	30	15	7.5
5	32	16	7
6	28	16	8
7	30	15	7.5
8	30	15	7.5
9	30	15	8.3
10	32	14	8
11	30	15	7.5
12	30	15	7.5
13	32	16	8
14	30	15	7.5
15	28	16	7
16	33.4	15	7.5
17	26.6	15	7.5
18	32	14	7
19	30	15	6.7
20	30	16.7	7.5

The design factors for the CCD is showing the minimum, maximum, mean and standard deviation values for our input parameters is as presented in Table 3.2

Table 3.3 CCD Design Factors

Response	Name	Units	Minimum	Maximum	Mean	Std. Dev.
INPUT 1	POP Cement	Grams	26.60	33.40	30.00	1.70
INPUT 2	Water	Grams	13.30	16.70	15.00	0.8516
INPUT 3	Fiber	Grams	6.70	8.30	7.50	0.4155

Observations from Table 3.2 shows the factors for CCD matrix with their minimum, maximum, mean and standard deviation values. The minimum value of POP cement was observed to be 26.6grams with a maximum value of 33.4grams, mean value of 30.00 and a standard deviation of 1.70; the minimum value of water was observed to be 13.3grams with a maximum value of 16.7grams, mean value of 15.00 and a standard deviation of 0.8516 while the minimum value of fiber was observed to be 6.70grams with a maximum value of 8.30grams, mean value of 7.5 and a standard deviation of 0.4155.

3.5 Experimental Data Analysis using Response Surface Methodology

Response Surface Methodology (RSM) was used to analyze the data obtained from the experiment. RSM is used in industries to explore the relationship between a response variable and several input factors, to determine the optimal settings of the factors, and to optimize the process or product. RSM is extensively used in situations where there are many input factors that may influence one or more response variables, it is a combination of mathematical and statistical models for analyzing processes in which a target response is influenced by several variables and

the main objective is to optimize this response. It also has an important application in the design, development and formulation of new products as well as in the improvement of existing product designs. The basic components of Response Surface Methodology include experimental design, regression analysis and optimization algorithms which are used to investigate the empirical relationship. Response Surface Method (RSM) is used to develop empirical model, commonly called response surface, for the response of a process in terms of the relevant controllable factors. RSM determines the operating conditions that produce the optimum response. Response Surface Methodology allows you to specify and fit a model up to the second order. RSM fits a model and provides the ANOVA and the 'Lack of Fit' test separately when there is more than one response. Contour and Surface plots of each response for pairs of factors are also produced. The aim of the response surface is to help understand the topography of the surface plot using simple maximum or minimum, saddles and ridges 3D diagrams and to find the region with the optimum response using contour plots.

In this study, the final solution of the model optimization process was to determine the optimum value of each input variable namely: P.O.P cement (grams), water (grams) and fiber (grams) that will maximize the thermal resistance of a P.O.P cast.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 EXPERIMENTAL RESULT

MODEL OPTIMIZATION AND CONTROL USING RESPONSE SURFACE METHODOLOGY (RSM)

Response surface methodology as an experimental design method for optimization allows evaluation of the effect of multiple factors and their interactions in one or more response variables by relating the influence of responses to a number of input variables or factors. It is a variation of the simple linear regression, with the incorporation of the second order effects of non-linear relationships. It is a popular optimization technique to determine the best possible combinations of variables to determine a specific response to a phenomenon. RSM is particularly useful to understand the relationship between multiple predictor variables with one or more predictions by using sequence of experimental designs in determining an optimal response. In this study, the target of the RSM model was to maximize the thermal resistance (R) of the P.O.P mold.

The recorded experimental result is presented in Table 4.1.

Table 4.1 Experimental Results

	Factor 1	Factor 2	Factor 3	Response 1
Run	A:POP Cement	B:Water	C:Fiber	Thermal Resistance
	Grams	Grams	grams	^o C/W
1	28	14	7	0.3595
2	28	14	8	0.30026
3	30	13.3	7.5	0.3273
4	30	15	7.5	0.346
5	32	16	7	0.3197
6	28	16	8	0.2325
7	30	15	7.5	0.3571
8	30	15	7.5	0.2461
9	30	15	8.3	0.2156
10	32	14	8	0.1523
11	30	15	7.5	0.2861
12	30	15	7.5	0.2962
13	32	16	8	0.3206
14	30	15	7.5	0.2763

15	28	16	7	0.1524
16	33.4	15	7.5	0.1946
17	26.6	15	7.5	0.1974
18	32	14	7	0.3056
19	30	15	6.7	0.2764
20	30	16.7	7.5	0.2647

The final solution of the model optimization process was to determine the optimum value of each input variable namely: P.O.P cement (grams), water (grams) and fiber (grams) that will maximize the thermal resistance of a P.O.P mold. From the experimental result, a summary of the experimental result was developed as presented in Table 4.2.

Table 4.2 Summary of experimental result

Response	Name	Units	Observations	Minimum	Maximum	Mean	Std. Dev.
INPUT 1	POP Cement	Grams	20.00	26.60	33.40	30.00	1.70
INPUT 2	Water	Grams	20.00	13.30	16.70	15.00	0.8516
INPUT 3	Fiber	Grams	20.00	6.70	8.30	7.50	0.4155
RESPONSE	Thermal Resistance	$^{\circ}\text{C}/\text{W}$	20.00	0.1523	0.3595	0.2713	0.0634

Observations from Table 4.2 shows the summary of process and response values in their minimum and maximum values including their mean and standard deviation values. When fed into a typical response surface design, it revealed that the model is of the quadratic type and requires polynomial analysis order. The RSM design build information summary is presented as shown in Table 4.3.

Table 4.3 RSM Design Information

File Version	13.0.5.0		
Study Type	Response Surface	Subtype	Randomized
Design Type	Central Composite	Runs	20.00
Design Model	Quadratic	Blocks	No Blocks
Build Time (ms)	34.00		

To check for the suitability of the quadratic model on analyzing the experimental data, a sequential model sum of squares was calculated for the investigated response while considering a combination of different model sources. Table 4.4 shows the sequential model sum of square calculation for the thermal resistance response.

Table 4.4 Sequential Model Sum of Squares Calculations

Source	Sum Squares	of	Df	Mean Square	F-value	p-value	
Mean vs Total	1.47		1	1.47			
Linear vs Mean	0.0070		3	0.0023	0.5403	0.6615	
2FI vs Linear	0.0407		3	0.0136	6.12	0.0079	
Quadratic vs 2FI	0.0172		3	0.0057	4.93	0.0235	Suggested
Cubic vs Quadratic	0.0004		4	0.0001	0.0529	0.9934	Aliased
Residual	0.0112		6	0.0019			
Total	1.55		20	0.0774			

The highest order polynomial where the additional terms are significant and the model is not aliased was selected. Quadratic vs. 2FI (two factor interaction or two factorial) model sources was suggested because of its p-value of 0.0235 which is less than 0.05 was selected as best fit therefore validating the use of the quadratic polynomial in the analysis.

A lack of fit test was then carried out to test how well the quadratic model can explain the underlying variation associated with the response data, if the model has a significant lack of fit,

then it cannot be employed for predictions. Table 4.5 shows the lack of fit test for the thermal resistance response.

Table 4.5 Lack of Fit Tests result

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Linear	0.0604	11	0.0055	3.04	0.1151	
2FI	0.0197	8	0.0025	1.36	0.3813	
Quadratic	0.0026	5	0.0005	0.2835	0.9036	Suggested
Cubic	0.0022	1	0.0022	1.20	0.3234	Aliased
Pure Error	0.0090	5	0.0018			

The selected quadratic polynomial model source has an insignificant lack of fit with a p-value above 0.05 (i.e. 0.9036), therefore it was suggested for model analysis while the cubic polynomial was aliased to model analysis because of its significant lack of fit.

To assess the strength of the quadratic model, a one-way analysis of variance (ANOVA) table was generated for the thermal resistance response. This is needed to ascertain if the model is significant or not and also to evaluate the significant contributions of each process parameter including their combined and quadratic effects towards the response. Table 4.6 shows the ANOVA table for the thermal resistance response.

Table 4.6 ANOVA Table for Thermal Resistance

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	0.0649	9	0.0072	6.21	0.0043	Significant

A-POP Cement	0.0002	1	0.0002	0.1488	0.7078	
B-Water	0.0029	1	0.0029	2.47	0.1469	
C-Fiber	0.0040	1	0.0040	3.44	0.0934	
AB	0.0261	1	0.0261	22.52	0.0008	
AC	0.0038	1	0.0038	3.23	0.1023	
BC	0.0108	1	0.0108	9.28	0.0123	
A ²	0.0145	1	0.0145	12.49	0.0054	
B ²	0.0002	1	0.0002	0.1551	0.7020	
C ²	0.0027	1	0.0027	2.36	0.1554	
Residual	0.0116	10	0.0012			
Lack of Fit	0.0026	5	0.0005	0.2835	0.9036	not significant
Pure Error	0.0090	5	0.0018			
Cor Total	0.0765	19				

The Model F-value of 6.21 implies the model is significant. There is only a 0.43% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. In this case AB, BC, A² are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The Lack of Fit F-value of 0.28 implies the Lack of Fit is not significant relative to the pure error. There is a 90.36% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good because we desire we the model to fit.

To validate the adequacy of the quadratic model, a goodness of fit statistics was calculated for the response. Table 4.7 shows the goodness of fit statistics for the thermal resistance response.

Table 4.7 Goodness of Fit Statistics

Std. Dev.	0.0341	R²	0.8482
Mean	0.2713	Adjusted R²	0.7117
C.V. %	12.55	Predicted R²	0.5768
		Adeq Precision	8.9908

It is observed that the Predicted R² of 0.5768 is in reasonable agreement with the Adjusted R² of 0.7117; i.e. the difference is less than 0.2. Adequate Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The computed ratio of 8.9908 as observed indicates an adequate signal. This shows that the model can be used to adequately predict the targeted response.

For an optimal solution, the coefficient estimates statistics and their corresponding standard errors which is a measure of the difference between the experimental terms and the corresponding predicted terms are first considered and generated. Table 4.8 shows generated coefficient estimate statistics in terms of the coded factors for thermal resistance response.

Table 4.8 Coefficients Estimate Statistics for Thermal Resistance Response

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	0.3002	1	0.0139	0.2692	0.3311	
A-POP Cement	0.0035	1	0.0092	-0.0169	0.0240	1.0000
B-Water	-0.0144	1	0.0092	-0.0349	0.0060	1.0000
C-Fiber	-0.0174	1	0.0094	-0.0384	0.0035	1.0000
AB	0.0572	1	0.0120	0.0303	0.0840	1.0000
AC	-0.0217	1	0.0120	-0.0485	0.0052	1.0000
BC	0.0367	1	0.0120	0.0099	0.0635	1.0000
A ²	-0.0311	1	0.0088	-0.0508	-0.0115	1.02
B ²	0.0035	1	0.0088	-0.0162	0.0231	1.02
C ²	-0.0149	1	0.0097	-0.0365	0.0067	1.01

Inference: The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the variance inflation factors (VIF) are 1; VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable. The computed standard error measures the difference between the experimental terms and the corresponding predicted terms. Table 4.7 indicates a significantly correlated model with high VIFs values.

The optimal equation of the selected input variables (P.O.P cement, water and fiber) represented

by A, B and C, showing their individual, combined and quadratic interactions against the thermal resistance response was generated as presented in Equation 4.1

Thermal Resistivity

$$= + 7.39097 + 0.202527A - 1.52623B + 0.408141C + 0.028579AB - 0.021657AC + 0.073385BC - 0.007783A^2 + 0.003468B^2 - 0.059605C^2 \quad (4.1)$$

Where A = P.O.P cement, B = Water, C = Fiber.

This equation in terms of coded factors can be used to make predictions about the response for any given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

A statistical diagnostic was employed for each response to give actual insight on the model strength and the adequacy of the second order optimal polynomial equation. Table 4.8 shows the statistical diagnostic report for thermal resistance response.

Table 4:9 Statistical Diagnostic Report on Thermal Resistance Response

Run Order	Actual Value	Predicted Value	Residual	Leverage	Internally Studentized Residuals	Externally Studentized Residuals	Cook's Distance	Influence on Fitted Value DFFITS	Standard Order
1	0.3595	0.3581	0.0014	0.673	0.070	0.067	0.001	0.096	1
2	0.3003	0.2932	0.0071	0.673	0.364	0.347	0.027	0.498	5
3	0.3273	0.3347	-0.0074	0.617	-0.352	-0.336	0.020	-0.427	11
4	0.3460	0.3002	0.0458	0.166	1.473	1.580	0.043	0.705	17
5	0.3197	0.3063	0.0134	0.673	0.689	0.670	0.098	0.961	4
6	0.2325	0.2234	0.0091	0.673	0.468	0.449	0.045	0.644	7
7	0.3571	0.3002	0.0569	0.166	1.830	2.129	0.067	0.950	18
8	0.2461	0.3002	-0.0541	0.166	-1.738	-1.974	0.060	-0.881	20

9	0.2156	0.2341	-0.0185	0.577	-0.836	-0.822	0.095	-0.959	14
10	0.1523	0.1426	0.0097	0.673	0.496	0.477	0.051	0.684	6
11	0.2861	0.3002	-0.0141	0.166	-0.452	-0.434	0.004	-0.193	19
12	0.2962	0.3002	-0.0040	0.166	-0.128	-0.121	0.000	-0.054	16
13	0.3206	0.3015	0.0191	0.673	0.982	0.980	0.198	1.406	8
14	0.2763	0.3002	-0.0239	0.166	-0.767	-0.750	0.012	-0.335	15
15	0.1524	0.1416	0.0108	0.673	0.556	0.536	0.064	0.769	3
16	0.1946	0.2162	-0.0216	0.617	-1.025	-1.028	0.169	-1.304	10
17	0.1974	0.2042	-0.0068	0.617	-0.321	-0.306	0.017	-0.389	9
18	0.3056	0.2942	0.0114	0.673	0.585	0.564	0.070	0.809	2
19	0.2764	0.2899	-0.0135	0.577	-0.610	-0.590	0.051	-0.689	13
20	0.2647	0.2857	-0.0210	0.617	-0.994	-0.993	0.159	-1.260	12

Focus is made on the model maximizing the Adjusted R^2 and the Predicted R^2 . Lower residual values resulting to higher leverages as observed in Table 4:8 are indicators of a well fitted model.

A reliability plot of the predicted values against the actual values of each response was developed in order to establish the suitability of response surface methodology in using the quadratic model and also to assess the prediction accuracy. Figure 4.1 shows the reliability plot for the thermal resistance response.

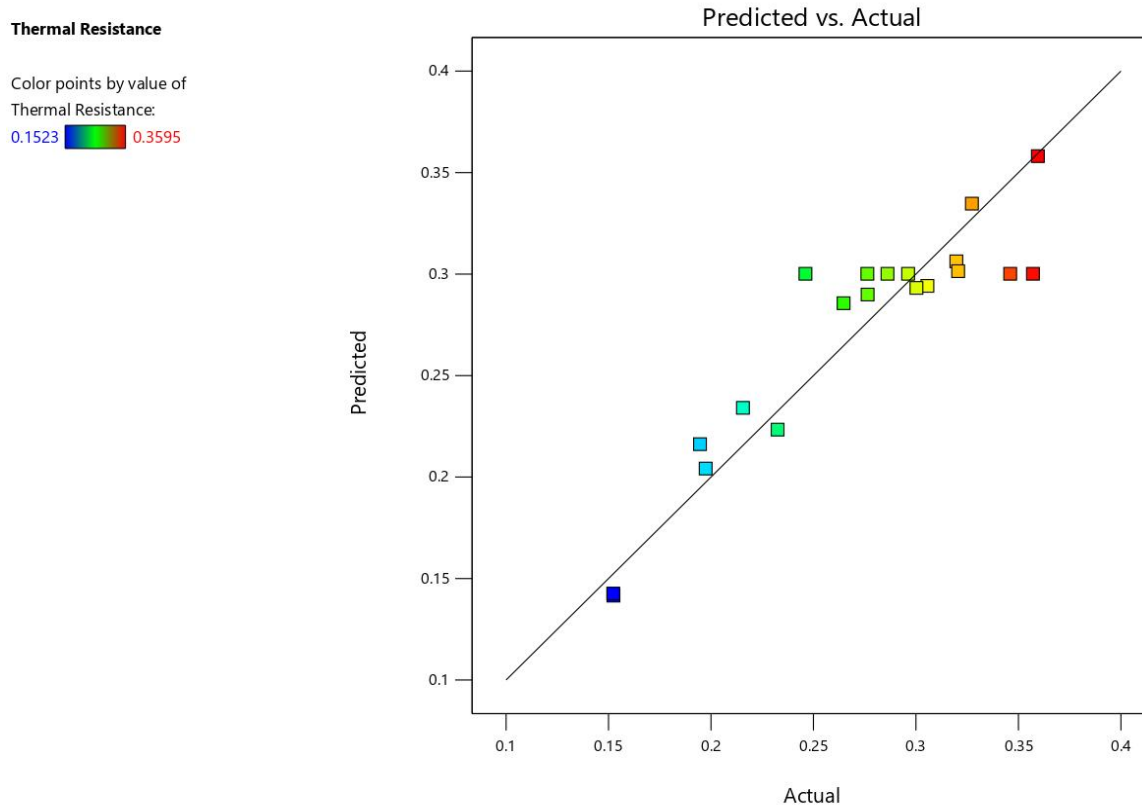


Fig 4.1: Reliability plot for Thermal Resistance

Observations from Figure 4.1 shows that the data points all cluster along the regression line of best fit therefore establishing the suitability of response surface methodology in using the quadratic model and also showing the prediction accuracy for the investigated response.

To satisfactorily accept a model, an appropriate statistical analysis output must be carried out. The normal probability plot of studentized residuals is the number of standard deviation of actual values based on the predicted values, and it is used to assess the normality of calculated residuals. It is the most significant assumption for checking the sufficiency of a statistical model. In this study, it was employed to ascertain if the residuals (observed – predicted) follows a normal distribution. Figure 4.2 shows the normality plot for the thermal resistance response.

Thermal Resistance

Color points by value of
Thermal Resistance:

0.1523  0.3595

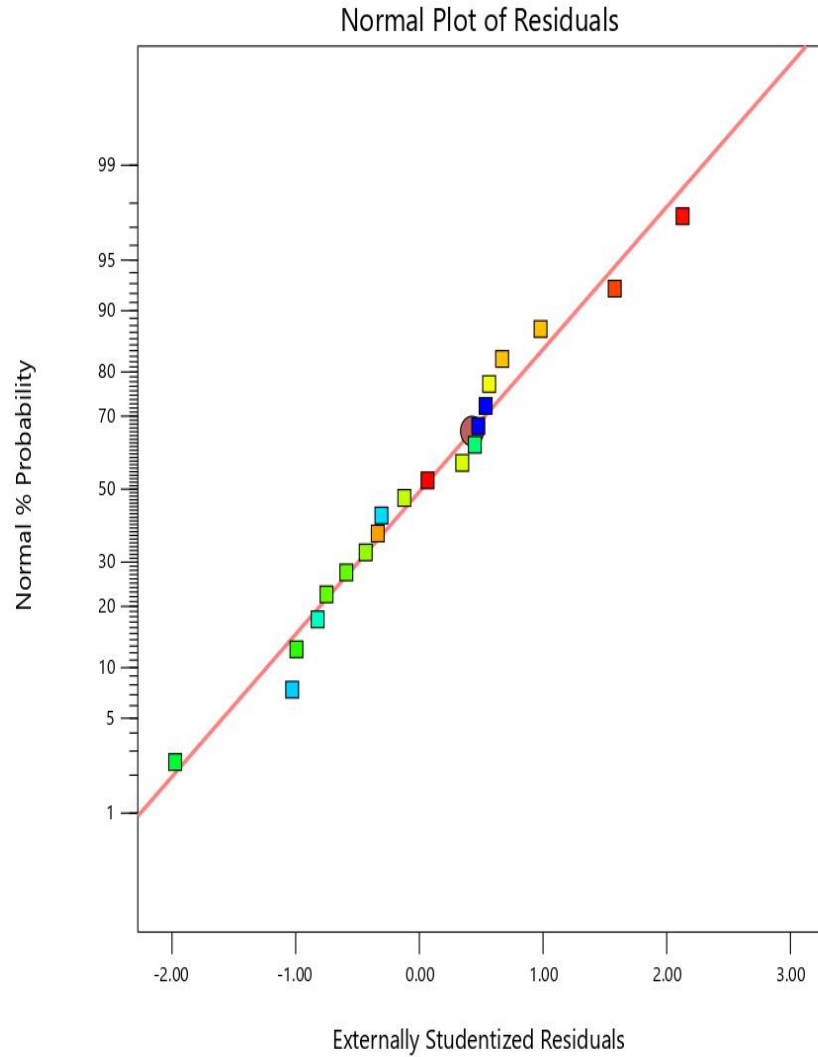


Fig 4.2: Normality plot for Thermal Resistance

The normal probability plot of studentized residuals was employed to assess the normality of the calculated residuals as observed in Figure 4.2. The points follow a linear trend despite the slight variations off the straight line. There is no defined curve or a “s-shape” aside the linear trend. This indicates that the computed residuals for the thermal resistance response are approximately normally distributed with no transformation of the response data required for better analysis. This implies that our developed model is satisfactory.

To check for drifts or lurking variables that may influence the experimental response, a plot of

residuals against the runs also known as a Run Order plot of residuals was produced. This is a special type of random scatter plot where residuals are plotted against the run order of the experimental data and it helps to identify the correlation between the sequential errors in the model. A regression line with a well fitted data will have a random scatter residual vs run plot but if it is a bad fit, then the plot will assume a certain pattern. Figure 4.3 shows the run order plot for thermal resistance response.

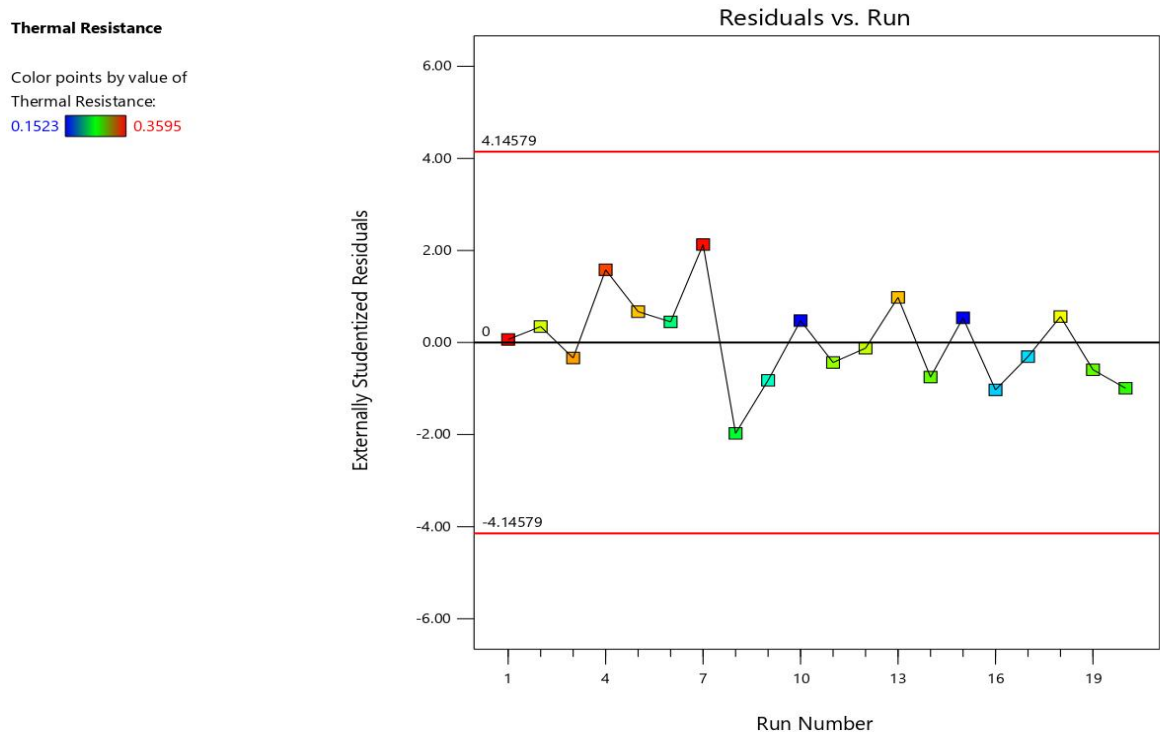


Fig 4.3: Run order plot for thermal resistance

The observed random scatter and absence of a pattern indicates that the stated response data is well fitted on the regression line. Observation from Figures 4.2 shows that the data is of good fit hence the random plot as observed. This is a useful technique for evaluating the goodness of fit for a model.

To check for the presence of possible outliers in the experimental data, a cook’s distance plot was

generated for individual responses. Cook's distance is a measure of how much the regression would change if the outlier is omitted from the analysis. A point that has a very high distance value in relation to other points may be an outlier and therefore should be investigated. The generated cook's distance plot to check for the possibility of an outlier in the thermal resistance experimental data and measure how much the regression would change if the outlier is omitted from the analysis is shown in Figure 4.4.

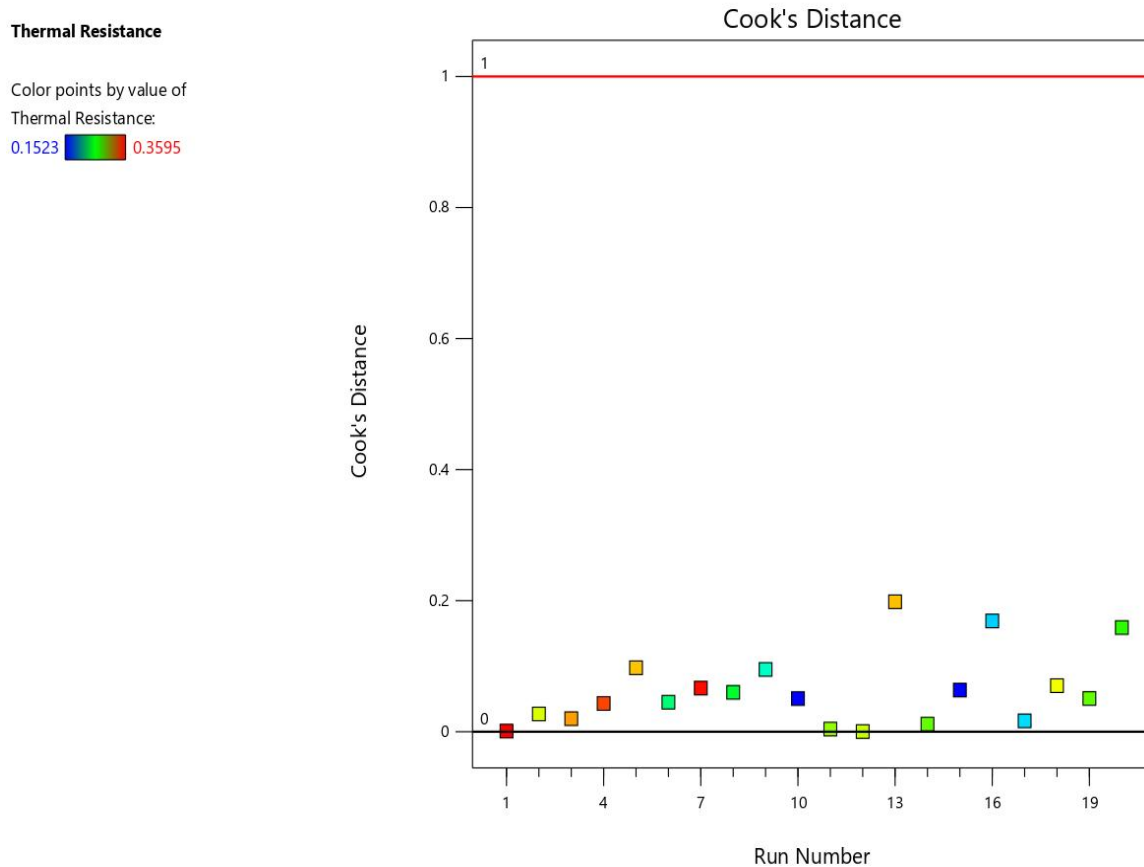


Fig 4.4: Cook distance plot for thermal resistance

The cook's distance plot has an upper bound of 1.00 and a lower bound of 0.00. Experimental values smaller than the lower bound or greater than the upper bounds are considered as outliers and must be properly investigated. Results of Figures 4.4 indicates that the calculated cook's

distance of the experimental data used for this analysis all fall within the lower and upper boundaries revealing the adequacy of the model.

A 3D surface plot and its contour plot was generated to visualize the interactions between the input and response variables, give a clear concept of the response surface and study the relationship between the combined input variables and the responses. 3D surface plot is a 3 dimensional surface plot which employs the use of color pallets to give a clear concept of the response surface, it provides a clearer picture of the interactions between the input and response variables. The 3D surface plot showing the relationship of water and POP on the investigated thermal response is shown in Figure 4.5 and Figure 4.6.

Factor Coding: Actual

3D Surface

Thermal Resistance (OC/W)

Design Points:

● Above Surface

○ Below Surface

0.1523  0.3595

X1 = A

X2 = B

Actual Factor

C = 7.5

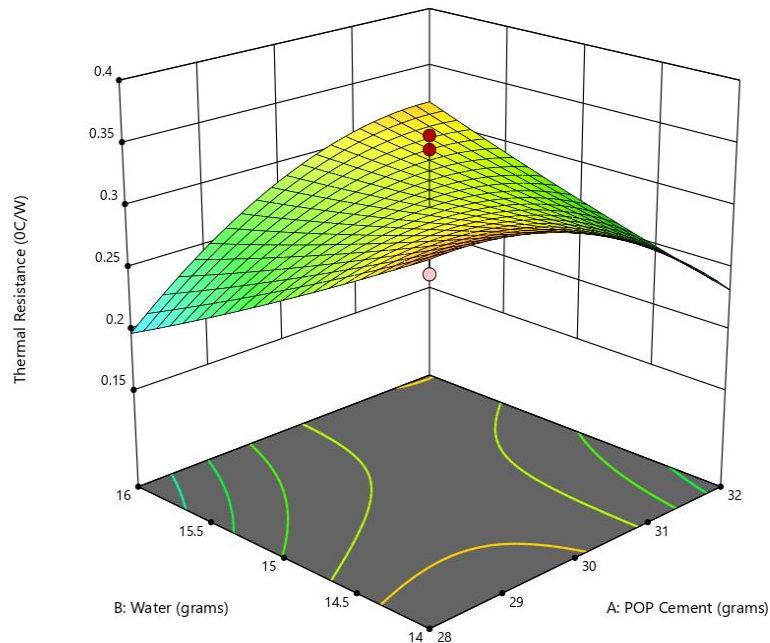


Fig 4.5: 3D surface plot of water and POP cement relationship with thermal resistance.

Factor Coding: Actual

Thermal Resistance (OC/W)

● Design Points

0.1523 0.3595

X1 = A

X2 = B

Actual Factor

C = 7.5

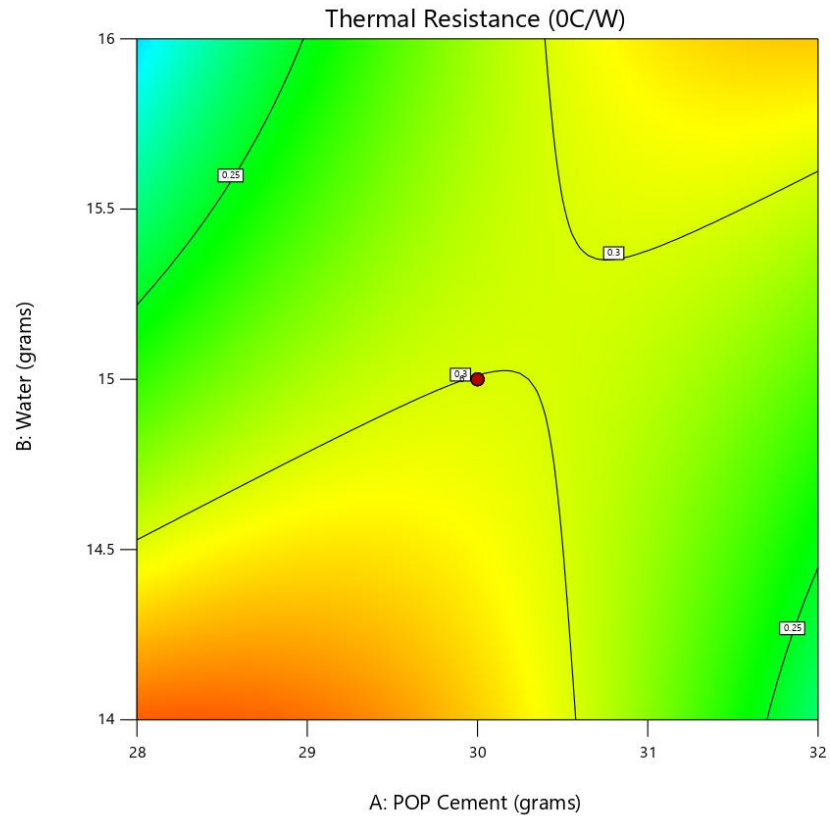


Fig 4.6: Contour plot of water and POP cement relationship with thermal resistance.

As the color pallets change from the blue to red zones, so does the response value increases accordingly.

Finally, numerical optimization was performed to ascertain the desirability of the overall model. In the numerical optimization phase, RSM was used to maximize the thermal resistance of the plaster of Paris cast mixture. The constraint set for the numerical optimization algorithm is presented in Table 4.11.

Table 4.10: Constraint setting for numerical optimization of thermal resistance response

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:POP Cement	is in range	28	32	1	1	3
B:Water	is in range	14	16	1	1	3
C:Fiber	is in range	7	8	1	1	3
Thermal Resistance	maximize	0.1523	0.3595	1	1	3

The constraint set to optimize the algorithm as shown in Table 4.11 displays the set goal of the RSM in optimizing the responses within their upper and lower limits with the input parameter set within range while the optimization goal of the targeted response was also stated. The numerical optimization produced thirteen (13) solutions and selected one as the optimal solution as presented in Table 4.12.

Table 4.11: Numerical Optimization Solutions

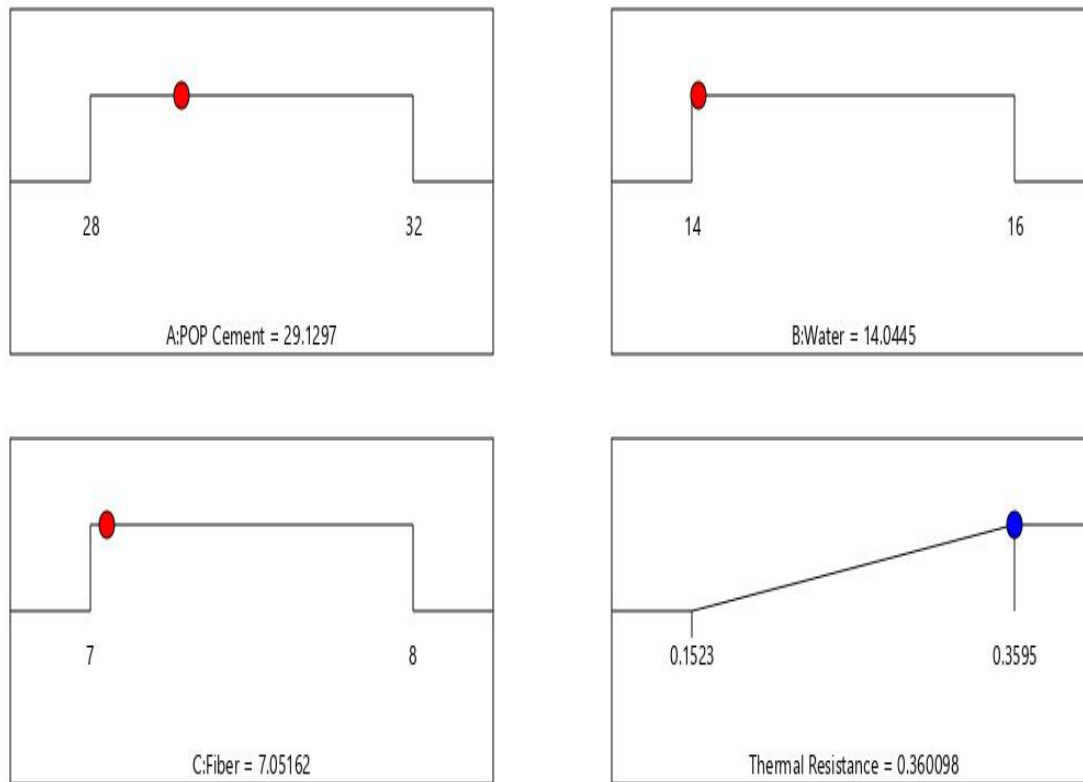
Number	POP Cement	Water	Fiber	Thermal Resistance	Desirability	
1	29.130	14.045	7.052	0.360	1.000	Selected
2	28.438	14.001	7.083	0.362	1.000	
3	28.880	14.008	7.025	0.364	1.000	

4	29.312	14.011	7.071	0.361	1.000	
5	29.000	14.019	7.099	0.361	1.000	
6	28.408	14.010	7.058	0.361	1.000	
7	28.225	14.003	7.045	0.360	1.000	
8	28.945	14.010	7.054	0.363	1.000	
9	28.649	14.021	7.005	0.363	1.000	
10	29.338	14.015	7.036	0.362	1.000	
11	29.173	14.034	7.052	0.361	1.000	
12	28.343	14.024	7.008	0.360	1.000	
13	29.028	14.029	7.083	0.360	1.000	

- Selected Optimal Solution by design expert with 100% desirability factor.

From the results of Table 4.12, it was observed that a 29.130 grams of POP cement, a 14.045gram of water and a 7.052grams of fiber will result in an optimal POP cast mixture having a thermal resistance of 0.360°C/W with a 100% desirability factor.

A graphical presentation of the optimal solution which is also known as the ramp solution showing the overall desirability of the optimal solution is presented in Figure 4.7.



Desirability = 1.000
 Solution 1 out of 79

Figure 4.7: Ramp Solution of Numerical Optimization.

Contour plots was used to visualize the model response and show the nature of the input variable based on the optimal solution. The contour plot of showing a combination of the thermal resistance response alongside the over desirability plot of the optimal solution is as shown in Figure 4.8.

Factor Coding: Actual

All Responses

0.000  1.000

X1 = A

X2 = B

Actual Factor

C = 7.05162

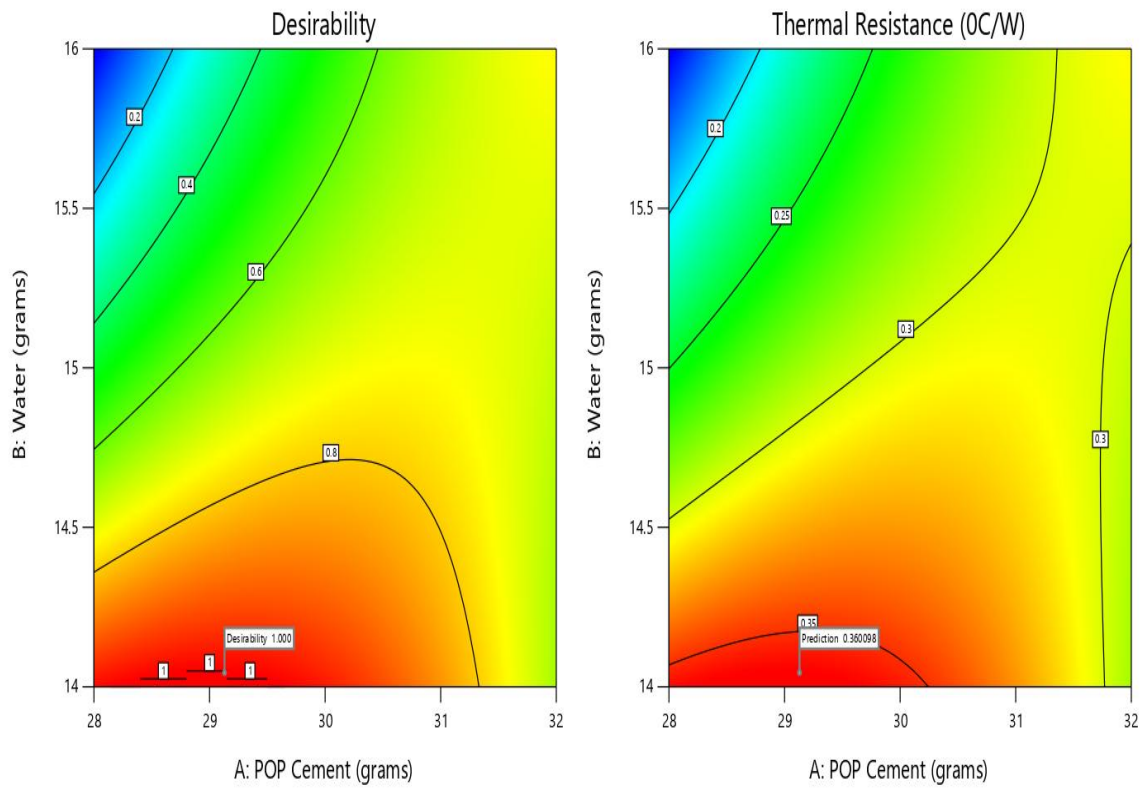


Figure 4.8: Optimization Contour Plot of Thermal Resistance Responses and Desirability. The contour plots as observed in Figures 4.8 displays the established optimal response values and the overall desirability with respect to the water and POP cement in a POP cast mixture with the fiber value kept at the optimal constant value. Contour plots was developed to show the models reliability response and show the effect of process parameters on the optimal desirability.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

In this study, we have successfully investigated the effects of water, fiber and POP cement as process parameters for on an optimal fiber POP cast mixture that would have a maximal thermal resistance by the application of response surface methodology in analyzing the experimental data through various statistical analytical methods, a mathematical model that can be used to carry out predictions of the investigated response at any given levels of the process parameters was also bdeveloped. A 3D surface and contour plot was also developed to visualize the effects of the stated process parameters on the investigated response with an optimal and desirability value obtained.

5.2 RECOMMENDATION

Based on the experimentation and data analysis carried out in this study, the following recommendations are made;

- i. Other expert systems such as adaptive neuro fuzzy inference system (ANFIS), genetic algorithm (GA) and artificial neural network (ANN) should applied to analyze our experimental data in other to further establish and/or compare optimal results obtained from this study.
- ii. The effects of POP cast process parameters on cast solidification rate, deterioration rate, brittleness and/or hardness alongside their effects on the thermal conductivity of POP and other PCM materials is also recommended for further studies.
- iii. Further investigations on the development of POP casts as PCM for lagging of thermal or heating systems should be explored.

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