



UNIVERSITI PUTRA MALAYSIA

***OPTIMIZATION OF FLEXURAL STRENGTH AND VOLUME EXPANSION
RATIO PROPERTIES OF DURIAN HUSK CELLULOSE FIBER/RICE
HUSK ASH-BASED GEO POLYMER COMPOSITE USING RESPONSE
SURFACE METHODOLOGY***

HANIS NABILAH BINTI MOHD YUSMIZAR

**Ip
FK 2022 10**

**OPTIMIZATION OF FLEXURAL STRENGTH AND VOLUME EXPANSION
RATIO PROPERTIES OF DURIAN HUSK CELLULOSE FIBER/RICE HUSK
ASH-BASED GEOPOLYMER COMPOSITE USING RESPONSE SURFACE
METHODOLOGY**

HANIS NABILAH BINTI MOHD YUSMIZAR

198329

**PROJECT REPORT SUBMITTED IN PARTIALLY FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF
BACHELOR OF ENGINEERING PROCESS AND FOOD WITH HONOURS**

DEPARTMENT OF PROCESS AND FOOD ENGINEERING

FACULTY OF ENGINEERING

UNIVERSITI PUTRA MALAYSIA

2022

ACKNOWLEDGEMENT

First and foremost, Alhamdulillah, the highest praise to the Almighty for blessing me with the time, health, and strength to complete my final year project.

I would also like to convey my deepest gratitude to my supervisor, Ts. Dr. Mohd Salahuddin Mohd Basri, for the consistent guidance, continual assistance, and wonderful support he has offered me during the final year project.

Next, I would like to thank both of my examiners, Assoc. Prof. Dr. Khairul Faezah Md. Yunos and Mdm. Zanariah Mohd Dom, for their objective assessment, constructive feedback and amazing encouragement regarding my senior thesis.

I'd also like to express my gratitude to my coordinator, Dr. Faiqa Shazaea Mohd Salleh, for coordinating the final year project with diligence, attention and sincerity for two semesters during EPF4949A and EFP4949B.

Then, I would like to extend my deepest gratitude to all the lecturers in the department of process and food engineering for their countless assistance and unwavering support throughout my four years of study.

Special thanks to the department's assistant engineer, Mdm. Siti Hajar Zakaria, who was of amazing assistance during my lab work.

I would also like to convey my gratitude to my family members, Mohd Yusmizar, Norsharini, Hakim Danish, Hana Malisa, and Harris Yassar, who have been incredibly supportive of me during the duration of my final year project.

Lastly, I'd want to thank my coursemates, particularly Sofia Aini, Nur Hanis, Nor Zafirah, Kiishhen Kobbin, and Olga Vallentine, for sharing the ups and downs with me during the preparation and completion of the senior thesis and bachelor's degree.

Also, thank you to everyone who contributed to my final year project in any way possible. I am extremely appreciative of everyone's efforts.

ABSTRACT

Durian husk accounts for 60 to 75% of the fruit's total weight, which becomes an excessive residue in the durian processing industry. However, durian husk is the most promising cellulosic resource owing to its high cellulose, hemicellulose, and lignin content. The objectives of the study are; (i) to design experimental works using response surface methodology and conduct experiments on flexural strength and volume expansion ratio, (ii) to determine the effect of factors on the properties of the durian husk cellulose fiber/rice husk ash-based geopolymer composite and (iii) to perform optimization and validation as well as to investigate and characterize the microstructure of the geopolymer composite. The durian husk cellulose fiber (DHCF) was extracted through delignification and mercerization. The geopolymer composite was prepared by mixing sodium silicate, sodium hydroxide, rice husk ash, and DHCF at a designated ratio. Three factors were studied, namely curing temperature (50, 60, and 70 °C), cellulose content (0, 1.5, and 3 wt.%), and curing time (3, 5, and 7 days). The responses studied are the flexural strength and volume expansion ratio. The results showed that an increase in the curing temperature up to 65 °C resulted in an increase in flexural strength and volume expansion ratio. An increase in cellulose content up to 1.5 wt.% resulted in increased flexural strength and decreased volume expansion ratio. Increased curing time lowers the flexural strength and improves the volume expansion ratio. The optimum flexural strength and volume expansion ratio could be achieved through the combination of curing temperature of 60 °C, cellulose content of 0.5 wt.%, and curing time of 3 days. This material can be utilized in the food and agricultural industry as an insulation material or sandwich panel.

ABSTRAK

Kulit durian menyumbang 60 hingga 75% daripada jumlah berat buah, yang menjadi sisa yang berlebihan dalam industri pemrosesan durian. Walau bagaimanapun, kulit durian adalah sumber selulosa yang paling menjanjikan kerana kandungan selulosa, hemiselulosa dan lignin yang tinggi. Objektif kajian adalah; (i) mereka bentuk kerja-kerja eksperimen menggunakan metodologi permukaan tindak balas dan menjalankan eksperimen tentang kekuatan lentur dan nisbah pengembang isipadu, (ii) menentukan kesan faktor ke atas sifat komposit geopolimer berasaskan serat selulosa /abu sekam padi dan (iii) melaksanakan pengoptimum dan pengesahan serta untuk menyiasat dan mencirikan struktur mikro komposit geopolimer. Serat selulosa kulit durian (DHCF) telah diekstrak melalui delignifikasi dan pemerseran. Komposit geopolimer disediakan dengan mencampurkan natrium silikat, natrium hidroksida, RHA, dan DHCF pada nisbah yang ditetapkan. Tiga faktor telah dikaji, iaitu suhu pengawetan (50, 60, dan 70 °C), kandungan selulosa (0, 1.5, dan 3 wt.%), dan masa pengawetan (3,5, dan 7 hari). Tindak balas yang dikaji ialah kekuatan lentur dan nisbah pengembang isipadu. Keputusan menunjukkan bahawa peningkatan dalam suhu pengawetan sehingga 65 °C mengakibatkan peningkatan dalam kekuatan lentur dan nisbah pengembang isipadu. Peningkatan kandungan selulosa sehingga 1.5 wt.% mengakibatkan peningkatan kekuatan lentur dan pengurangan nisbah pengembang isipadu. Peningkatan masa pengawetan merendahkan kekuatan lentur dan meningkatkan nisbah pengembang isipadu. Kekuatan lentur optimum dan nisbah pengembang isipadu boleh dicapai melalui gabungan suhu pengawetan 60 °C, kandungan selulosa 0.5 wt%, dan masa pengawetan selama 3 hari. Bahan ini boleh digunakan dalam industri makanan dan pertanian sebagai bahan penebat atau panel sandwic.

TABLE OF CONTENTS

CHAPTER 1	1
INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement.....	4
1.3 Research Objectives.....	6
1.4 Scope of the Study	6
CHAPTER 2.....	8
LITERATURE REVIEW	8
2.1 Geopolymer and Geopolymer Composite.....	8
2.1.1 Geopolymer	8
2.1.2 Geopolymer Composite.....	10
2.2 Rice Husk Ash (RHA)	12
2.2.1 Introduction to Rice Husk Ash	12
2.2.2 Rice Husk Ash-Based Geopolymer Composite.....	15
2.3 Cellulose Isolated from Durian Husk.....	16
2.3.1 Introduction to Durian Husk Cellulose Fiber (DHCF).....	16
2.3.2 Utilization DHCF in Geopolymer Composite	21
2.4 Properties of Fiber-Based Geopolymer Composite	22
2.4.1 Flexural Strength.....	22
2.4.2 Volume Expansion Ratio.....	23
2.5 Factor Affecting the Geopolymer Properties.....	24
2.5.1 Curing Temperature	24
2.5.2 Cellulose Content.....	25
2.5.3 Curing Time.....	27
2.6 Response Surface Methodology (RSM).....	28
2.6.1 Introduction to RSM	28
2.7 Potential Application of Geopolymer Composite in Food Industry	31
CHAPTER 3	35
METHODOLOGY.....	35
3.1 Overview.....	35
3.2 Materials.....	38
3.2.1 Rice Husk	38
3.2.2 Activated Alkaline Solution	39

3.2.3	Durian Husk.....	40
3.2.4	Cellulose Extraction Reagents	42
3.3	Experimental Design and Analysis.....	43
3.3.1	Response Surface Methodology (RSM).....	43
3.4	Sample Preparation	46
3.4.1	Durian Husk Cellulose Fiber (DHCF).....	46
3.4.2	Rice Husk Ash	47
3.4.3	Durian Husk Cellulose Fiber/Rice Husk Ash-Based Geopolymer Composite 49	
3.5	Experiment Procedure.....	51
3.5.1	Flexural Strength Test	51
3.5.2	Volume Expansion Ratio.....	52
3.6	Microstructure Analysis.....	53
3.6.1	Scanning Electron Microscope (SEM).....	53
CHAPTER 4		54
RESULTS AND DISCUSSION		54
4.1	Response Surface Methodology (RSM).....	54
4.1.1	Statistical Analysis of Flexural Strength and Volume Expansion Ratio Properties	54
4.1.2	Effect of Factors on Flexural Strength and Volume Expansion Ratio Properties	58
4.1.3	Optimization Formulation of The Response.....	66
4.1.4	Optimization Validation	68
4.1.5	Material Behaviour of Sample After Flexural Strength Test.....	70
4.1.6	Material Behaviour of Sample After Volume Expansion Ratio	72
4.1.7	Contour Plots Superimposition.....	74
4.2	Microstructure Analysis.....	77
CHAPTER 5		85
CONCLUSIONS AND RECOMMENDATION		85
5.1	Conclusion.....	85
5.2	Recommendation for Future Research.....	86
REFERENCES		87
APPENDICES		101
Appendix A1		101
Appendix B1		104
Appendix B2		105
Appendix C1		106

Appendix D1.....107

Appendix D2.....110

Appendix E1113

Appendix E2.....116



LIST OF TABLES

CHAPTER 2

Table 2. 1: Chemical composition of rice husk ash. (Antiohos et al., 2014; Chao-Lung et al., 2011; De Sensale, 2010; Giaccio et al., 2007; Madandoust et al., 2011; Park et al., 2016; Rößler et al., 2014; Zahedi et al., 2015).	15
Table 2. 2: The properties of durian rinds fiber. (Indran & Raj, 2015; Jabli et al., 2018; Lubis et al., 2020; Madurwar et al., 2013)	20
Table 2. 3: The mechanical properties of durian rind fibers. (Lubis et al., 2020).....	21

CHAPTER 3

Table 3. 1: Composition of rice husk. (Kumar et al., 2012).....	38
Table 3. 2: Factors and levels of the experiment.	43
Table 3. 3: Matrix design.	44
Table 3. 4: Physical properties of RHA. (Mohd Basri et al., 2021)	48
Table 3. 5: Chemical composition of rice husk ash. (Saravanan & Kumar, 2013)	49

CHAPTER 4

Table 4. 1: Estimated effects and coefficient for flexural strength.....	56
Table 4. 2: Estimated effects and coefficient for volume expansion ratio.....	57
Table 4. 3: Experimental validation for the flexural strength and volume expansion ratio properties.	68
Table 4. 4: Effect of factors on the volume expansion ratio of the foamed geopolymer composite at a given foaming temperature of 150 °C for 1 hour.	74
Table 4. 5: Sample number and performance.....	78

LIST OF FIGURES

CHAPTER 2

Figure 2. 1: Geopolymer frameworks. (Olawale, 2013)	10
Figure 2. 2: (a) Untreated durian rind, (b) holocellulose, and (c) cellulose (Tawakkal et al., 2012).....	18
Figure 2. 3: (a) Durian rind waste, (b) durian rind fiber and (c) cellulose fiber (Lubis et al., 2020).....	19
Figure 2. 4: Effect of both curing period and temperature curing on 28-day compressive strength of AAM with different molarities.(Safari et al., 2020)	28

CHAPTER 3

Figure 3. 1: Flowchart of the DHCF/RHA-based geopolymer composite preparation.	36
---	----

Figure 3. 2: Flowchart of the DHCF/RHA-based geopolymer composite testing procedure.....	37
Figure 3. 3: Rice husk. (The Star, 2022).....	38
Figure 3. 4: (a) Sodium hydroxide and (b) sodium silicate.....	39
Figure 3. 5: White inner and thorny outer husks of durian rind.	40
Figure 3. 6: Durian husk preparation process; (a) washing, (b) cutting, (c) drying, (d) grinding, (e) drying ground husk, and (f) place in an airtight container.....	41
Figure 3. 7: (a) Sodium chlorite and (b) acetic acid.	42
Figure 3. 8: Flowchart of DHCF preparation.	47
Figure 3. 9: Rice husk ash. (Deelert Intertrade Co., Ltd., n.d.)	48
Figure 3. 10: Flowchart of DHCF/RHA-based geopolymer composite preparation..	50
Figure 3. 11: Sketch of the three-point bending test.....	51
Figure 3. 12: Hitachi S-3400N Variable SEM	53

CHAPTER 4

Figure 4. 1: Contour plot for the effect of curing time and curing temperature on the flexural strength.	59
Figure 4. 2: Contour plot for the effect of cellulose content and curing temperature on the flexural strength.	61
Figure 4. 3: Contour plot for the effect of curing time and curing temperature on the volume expansion ratio.	63
Figure 4. 4: Contour plot for the effect of cellulose content and curing temperature on the volume expansion ratio.....	65
Figure 4. 5: Optimization plot for maximum flexural strength and volume expansion ratio.	67
Figure 4. 6: (a) control sample and (b) optimized sample for flexural strength, and (c) control sample and (d) optimized sample for volume expansion ratio.....	69
Figure 4. 7: Stress-strain curves of samples S5, S14, S13, and optimized sample. ...	71
Figure 4. 8: Effect of volume expansion ratio on the average cell diameter.....	72
Figure 4. 9: Effect of volume expansion ratio on the cell density.....	73
Figure 4. 10: Superimposed contour for responses affected by cellulose content and curing temperature.	75
Figure 4. 11: Superimposed contour for responses affected by curing time and curing temperature.....	76
Figure 4. 12: SEM micrographs of treated durian husk cellulose fiber at 500x (left) and 1000x (right) magnification.....	77
Figure 4. 13: SEM micrographs of DHCF/RHA-based geopolymer composite for the flexural strength test with (a-b) good (sample S5), (c-d) moderate (sample S14), and (e-f) poor flexural strength properties (sample S3) at 100x and 500x magnification.	79
Figure 4. 14: SEM micrographs of DHCF/RHA-based geopolymer composite after heated at 150°C with (a-b) high (sample S5), (c-d) moderate (sample S14), and (e-f) low volume expansion ratio (sample S3) at 25x and 50x magnification.....	82

APPENDIX B1

Figure B1. 1: 3-D view of three-point bending test set-up. (Basri et al., 2020).....	104
--	-----

CHAPTER 1

INTRODUCTION

1.1 Research Background

In Southeast Asia, durian (*Durio zibethinus* Murray) is the most popular fruit (Amid & Mirhosseini, 2012). Consumption of durian and its products has soared on the international trade market. The entire world harvest of durian is 1.4 mt, with Thailand, Malaysia, and Indonesia being the largest producers (Foo & Hameed, 2011). It has been aggressively extended and firmly established as a multifunctional food processing industry, but only one-third of durian is edible, while the seeds and shell become wastes that generate a substantial amount of biological waste (Amid & Mirhosseini, 2012). Commonly, durian wastes are burned or disposed of in landfills without consideration for the environment (Foo & Hameed, 2011). It was discovered that durian peel is a rich source of cellulose (Rachtanapun et al., 2012). Penjumras et al. (2014) reported that the cellulose content of durian peel is comparable to the cellulose content of wheat, barley, and bamboo cane fiber (Han et al., 2015).

Cellulose, the primary component of all plant matter, constitutes approximately one-third to two-thirds of all plant tissues and is continually replaced by photosynthesis (Mandal & Chakrabarty, 2011; Sun et al., 2004). It is the primary structural component that imparts strength and stability to plant cell walls (Dufresne, 2006), and it is structured into microfibril in the cell wall, interrupted by hemicellulose and surrounded by a lignin matrix. Depending on the type of plant, around 40 to 55 percent of plant materials consist of cellulose, 15 to 35 percent of lignin, and 25 to 40 percent of hemicellulose. Crystalline cellulose is associated with these chemicals in the natural

plant cell wall, making it difficult to extract pure cellulose (Rachtanapun et al., 2012). Chemically, cellulose is a linear natural polymer composed of anhydroglucose units joined at the one and four carbon atoms by glycosidic bond. This is supported by the presence of three hydroxyl groups (OH) with varying acidity or reactivity, with the secondary OH positioned at the C-2 and C-3 positions and the primary OH found at the C-6 position. It is also confirmed by the creation of strong intermolecular and intramolecular hydrogen bonds of numerous types (Mandal & Chakrabarty, 2011; Sun et al., 2004). In recent years, the interest in cellulose-based materials has increased due to the growing demand for renewable resources and environmental consciousness (Mohanty et al., 2005).

Global rice production in 2007 was predicted to be 649.7 Mt, with husk accounting for 20 to 25 percent of the total (Pocketbook, 2015). The agricultural industry generates a great deal of trash. The rice milling process creates a husk containing 22 percent of the paddy weight, with the remaining weights being obtained as rice, broken grains, and bran. This husk includes around 75 percent volatile organic content, and the remainder changed to ash during the burning process, known as rice husk ash (RHA). For every 1000 kg of paddy milled, roughly 220 kg (22%) of husk is produced, and when this husk is burned in the boilers, about 55 kg (25%) of RHA is produced (Nagrle et al., 2012).

By burning the rice husk under regulated conditions, more than 90 percent of pure silica may be extracted from the ash (Bakar et al., 2016). Amorphous silica and cementitious binder, when utilized in the appropriate conditions, produce pozzolanic activity, which possesses good thermal and mechanical properties. Substitution of RHA in cement and concrete increases corrosion resistance, strength, and mechanical properties due to its high silica concentration and amorphous nature. Simultaneously,

it lowers cement manufacturing costs while being environmentally favorable (Ramasamy, 2012).

Geopolymer or synthetic alkali aluminosilicate is produced by the reaction of aqueous alkali hydroxide and sodium aluminosilicate, also known as an inorganic polymer (Davidovits, 1991). These materials commonly replace cement and provide great long-term performance while lowering greenhouse gas emissions. Due to its better compressive strength, thermal conductivity, and fire resistance (Duxson et al., 2007), geopolymer should be employed as a composite material. Because of its high silica content, agricultural waste such as RHA is being investigated as an alternative for improving geopolymeric composite performance (Nuaklong et al., 2020).

Weather corrosion, fire, and chemical conditions may cause materials to deteriorate; thus, a suitable protective sandwich material is required to improve surface qualities and minimize high maintenance costs. As a result, geopolymer composite can be used as a core composite material in the agriculture and food industries. Due to its excellent resistance to corrosion, humidity, and pests, stainless steel is commonly used in the food and agriculture industries for equipment and storage needed to regulate internal temperature. If steel is exposed to the environment for an extended period, its composition will deteriorate (Krechkovska et al., 2018).

As a result, composite materials were frequently employed as insulation. While composite constructions utilized as insulation materials must have good thermal properties, their mechanical requirements for walls must not be jeopardized. The literature shows that intensive studies on the link between flexural and volume expansion ratio properties of geopolymer composite materials utilizing a statistical analysis approach are lacking. The goal of the flexural strength test was to use

statistical methods to investigate the impact of different material behaviors, such as brittle and ductile, on strength qualities and to find the best material formulation that can meet both flexural and volume expansion ratio properties. When storage or building structure is subjected to stresses, it will experience bending moments, which will cause fractures to form in the material and spread quickly. The goal of the three-point bending test was to see if the geopolymer material would be affected negatively by load-bending moments on such structures.

1.2 Problem Statement

As a result of the detrimental effects of higher surface temperatures on material qualities such as aging, corrosion, and degradation rate, these materials have restricted uses. Higher temperatures cause the material to expand and contract, which increases corrosion and cracking. High temperatures can lead to greater energy use and risk in some scenarios. For example, long-term exposure of storage tanks (silo) to hot weather may erode the equipment surface. It is critical to have an excellent protective composite material. A petrochemical tank must be regularly cooled with water spray to maintain the internal temperature; otherwise, excessive compound volatilization can trigger an explosion. On the other hand, this cooling approach wastes a lot of water, electricity, and tank maintenance costs.

In 2008, industry consumed 58.4 percent of total energy, construction 22.3 percent, and transportation 19.3 percent. Energy reduction limits in some industrial sectors could negatively impact the domestic output and national economy; thus, saving energy in the building sector is increasingly significant. Building on a larger scale needs a lot of energy. Surface coatings are the most effective approach to

reducing energy consumption in a building by limiting heat loss and gain (Al-Homoud, 2005). Over 40 percent of building cooling and heating overload is caused by heat loss and absorption by surface coating. Therefore, an excellent thermal insulation material may help limit energy usage. Low conductivity building and construction materials significantly reduce overheating and cooling while lowering energy consumption (Cha et al., 2012).

Durian is one of the largest commodities and is regarded as one of the largest by-products; its production increased from 65 tonnes in 2015 to 74 tonnes in 2017 and is expected to increase by 900,000 tonnes annually over the next two decades (Putri & Kurniyati, 2016). Durian husk is the most promising cellulosic resource due to its high cellulose and hemicellulose content, as well as its lignin content (E'zzati et al., 2018).

In place of glass fiber, natural cellulose fibers are increasingly used as reinforcing materials in thermoset and thermoplastic polymeric matrices due to their unique properties, such as renewability, low density, and high specific strength (Ochi, 2008). There are numerous cellulose-rich plants, including cotton, wood, bamboo, flax, hemp, sisal, and jute (Li et al., 2014). However, research on the utilization of cellulosic waste as filler has expanded quickly during the past decade. Although a number of natural fibers have been thoroughly studied, the utilization of durian husk as a natural source for the synthesis of cellulose has not yet been investigated.

In addition, rice husk has shown remarkable promise as a waste material in making geopolymer covering due to air pollution caused by burning rice husk in an open location. Malaysia has researched various aluminosilicate sources, including fly ash, palm oil fuel ash (POFA), kaolin, metakaolin, and dolomite. Fly ash performed well among geopolymer materials because of its high alumina and silica content and

environmentally friendly material. On the other hand, fly ash geopolymer has strong water resistance and low mechanical strength (Zain et al., 2017). RHA appears to be a more suitable replacement for other geopolymer materials. The chemical, mineralogical, and morphological content of these ashes vary depending on the processing settings. Because the ashes are predominantly made of silica, they are resistant to chemical etching (acid slag) and thermal shocks (temperature swings above 600 °C) and have low thermal conductivity qualities. These properties make the RHA a good candidate for a thermal insulator coating in the building industry since it promotes energy conservation while lowering costs (Gonçalves & Bergmann, 2007).

1.3 Research Objectives

1. To design experimental works using response surface methodology and conduct experiments on flexural strength and volume expansion ratio of durian husk cellulose fiber/rice husk ash-based geopolymer composite.
2. To determine the effect of curing temperature, cellulose fiber content, and curing time on the properties of the durian husk cellulose fiber/rice husk ash-based geopolymer composite and perform optimization and validation.
3. To investigate and characterize the microstructure of durian husk cellulose fiber/rice husk ash-based geopolymer composite.

1.4 Scope of the Study

The study focuses on optimizing the durian husk cellulose fiber/rice husk ash-based geopolymer composite by analyzing the flexural strength and volume expansion

ratio of the samples. Three factors were considered, namely, curing temperature, cellulose content, and curing time. Using the MINITAB software, the optimum factors for the durian husk cellulose fiber/rice husk ash -based geopolymer composite are determined through response surface methodology (RSM). It is a powerful tool for designing and optimizing experimental works and research. The optimized design is then validated through the experiment conducted.



CHAPTER 2

LITERATURE REVIEW

2.1 Geopolymer and Geopolymer Composite

2.1.1 Geopolymer

A synthetic alkali aluminosilicate substance, sometimes known as a "geopolymer," is produced when a solid aluminosilicate is reacted with a highly concentrated aqueous solution of alkali hydroxide or silicate (Davidovits, 1991), but more appropriately termed an 'inorganic polymer' (Van Wazer, 1970). Although the term "geopolymer" most commonly refers to the amorphous or crystalline products that result from the synthesis of alkali aluminosilicates with alkali hydroxide or alkali silicate solution, the term "low-temperature aluminosilicate glass" is also commonly used to refer to geopolymeric gels and composites (Rahier et al., 1996), 'alkali-activated cement' (Palomo & dela Fuente, 2003), 'geocement' (Krivenko, 1994), 'alkali-bonded ceramic' (Mallicoat et al., 2009), 'inorganic polymer concrete' (Sofi et al., 2007), and 'hydroceramic' (Bao et al., 2005).

Geopolymers are an intriguing class of aluminosilicate binders (Davidovits, 1991; Komnitsas & Zaharaki, 2007), commonly synthesized at a temperature below 100 °C (Davidovits, 2017); nonetheless, ambient temperatures have frequently been used to reduce the production cost of the binder (Deb et al., 2014; Sankar et al., 2018; Temuujin et al., 2009). Davidovits created the term 'geopolymer' in 1978 (Davidovits & Cordi, 1979) to refer to mineral polymers formed via geosynthetic, i.e., the chemical interaction of aluminosilicate precursors with alkali polysilicates, resulting in Al-O-Si links (Davidovits, 1994). However, acidic conditions such as phosphoric acid can also

be utilized for the chemical activation of Si and Al- rich precursors (Louati et al., 2016; Mathivet et al., 2019; Tchakouté & Rüscher, 2017).

Alumino-silicates are the primary raw material (precursor) for the manufacturing of geopolymers (Nikolov et al., 2017). Natural precursors consist of kaolin, zeolite, volcanic ash, and natural pozzolans; thermally treated precursors consist of metakaolin, fly ash, granulated blast furnace slag, calcined shales, and other industrial by-products. The alumino-silicate material must be activated using a second raw material, the so-called activator, which is often an alkaline solution. Typical activators include sodium and potassium hydroxide, sodium and potassium silicate (water glass) solutions, and sodium carbonate.

Mixing powdered aluminosilicates and liquid alkaline activators, often an aqueous alkali metal hydroxide or alkali silicate, yields geopolymer binders that are hardening pastes (water glass) (Koutnik et al., 2020). Aluminosilicates are partially dissolved by alkaline hydrolysis to the aluminate and silicate species, which are then converted into a three-dimensional polymer network by low temperature polycondensation. The principal effect of polymerization is the hardening of the geopolymer binder. (Davidovits, 1989, 2008; Duxson et al., 2007).

Davidovits has documented the structure and classification of geopolymers and discovered the empirical formula for its composition that can be stated as $M_n[-(SiO_2)_zAlO_2]_n \cdot H_2O$ (Davidovits, 1991; Guo et al., 2017; Rangan, 2014), where M is alkali cation such as Na^+ or K^+ , n is the degree of polymerization and z is the molar ratio of silicon to aluminium. Their microstructure comprises chains or networks of inorganic molecules that are covalently bonded (Davidovits, 2011). These molecules consist of one silicon or aluminium atom coupled to four oxygen atoms producing

tetrahedrons, which are joined in a three-dimensional network by a single oxygen atom, as depicted in Figure 2.1.

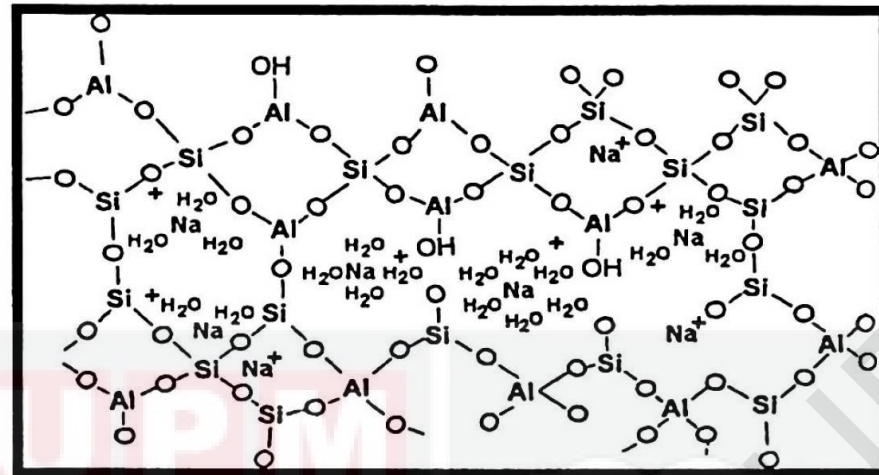


Figure 2. 1: Geopolymer frameworks. (Olawale, 2013)

2.1.2 Geopolymer Composite

Current geopolymer materials have shown substantial promise as heat-resistant materials for aircraft cabins, and they have been examined as a potential alternative to polymer composites (Lyon et al., 1997; Zhao et al., 2007). The geopolymer matrix has a lower mechanical strength than ceramics, limiting its use in structural applications. Since then, a wide range of geopolymer-based composites have been studied, including particulate (Barbosa & MacKenzie, 2003; Davidovits & Davidovics, 1991), continuous fiber (Rahman, 2019; Rahman, Jackson, et al., 2020), and short fiber (Korniejenko et al., 2021; Korniejenko et al., 2020) reinforced geopolymer composites. Since they can be manufactured using the same methods as traditional polymer composites, continuous fiber reinforced geopolymer composites have recently garnered significant interest. On the other hand, the high strength and modulus of the fibers make it possible to prevent catastrophic brittle failure in composites.

While fiber reinforced geopolymer composites of $\text{Si}/\text{Al} \leq 2$ still demonstrated poor mechanical properties due to the relatively weak geopolymer matrix, which is a huge hindrance to their wide application (Kriven et al., 2003).

The effect of alkali activator to binder (AAB) ratio and alkali activator molarity on the rice husk ash-based geopolymer was investigated by Kaur et al. (2018). After 28 days of testing, the maximum compressive strength of 39.95 N/mm^2 was achieved, according to the data. The AAB ratio and the molarity of the alkali activator solution were found to be proportional to compressive strength. As molarity grows, the microstructure becomes quite dense, owing to a high degree of geopolymerisation.

Yaseri et al. (2019) looked into making a sustainable geopolymer out of high-volume cement kiln dust (CKD) and RHA. An alkaline activator solution was prepared by combining RHA powder with a 10M sodium hydroxide solution to change the $\text{Na}_2\text{O}/\text{SiO}_2$ and $\text{H}_2\text{O}/\text{Na}_2\text{O}$ molar ratios (NaOH). The geopolymer was characterized using compressive strength testing, Fourier transforms infrared spectroscopy, mini-slump test, setting time, and x-ray diffraction. The findings show that increasing CKD up to the optimal level (45 percent) increased compressive strength linearly, but that compressive strength decreased beyond that level. The results also demonstrated that changing the SF/CKD ratio affects the setting time.

Mejía et al. (2016) explored industrial by-products as solid sources of silica in replacement of commercial sodium silicate (SS) to create geopolymeric binary binders. RHA spent diatomaceous earth (SDE), and commercial sodium silicate was utilized as silica sources to change the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio. The compressive strength was tested at 28, 180, and 360 days and mechanical and microstructural examinations. The findings show that RHA and SDE could be employed as

geopolymerisation silica sources. At 60 °C, these different silica sources slowed down the start and finish times of an FA/MK-based binary alkali-activated geopolymer system. Regardless of the silica source employed, the mechanical strength of the resulting geopolymer binders did not change considerably over time.

2.2 Rice Husk Ash (RHA)

2.2.1 Introduction to Rice Husk Ash

RHA is a plentiful agricultural by-product that has adverse effects when crop residues are burned (Sandhu & Siddique, 2017). Le et al. (2014) and Aprianti et al. (2015) in their work reported that, in 2013, approximately 742 million tonnes of rice were produced annually, which will generate approximately 160 million tonnes of RHA that will be thrown directly into the environment or burned in fields, causing severe environmental contamination. RH waste is dumped over a vast area, and during incineration, the burnt ash can spread over a large region due to the presence of air, which can lead to environmental contamination (Qin et al., 2018).

RHA is a comprehensive term for all types of ash resulting from burning rice husk (RH) (Zou & Yang, 2019). 17–20 percent of RHA is produced when RH is burned. RHA is a lightweight, bulky, and extremely porous substance with a density of around 180–200 kg/m³.

RHA can be utilized as an alternate cementitious binder and to regulate the SiO₂ concentration of the geopolymer owing to its high pozzolanic activity and strong heavy metal sorption property (Daifullah et al., 2003; Khalid et al., 1999; Krishnani et al., 2008). Furthermore, the previous study revealed that RHA combined with

flocculants such as aluminium or other chemical conditioning agents had a greater impact on the dewaterability and solidification of sludge (Qi et al., 2011).

There are two types of RHA, crystalline and amorphous, each of which has its applications (Prasara-A & Gheewala, 2017). Only crystalline silica remains after burning at temperatures exceeding 700 °C (Malhotra & Mehta, 2004), which can be utilized in the ceramic and steel sectors (Prasara-A & Gheewala, 2017). Amorphous silica is utilized as a supplementary cementitious material (SCM) in construction materials and as a filler in rubber or paint when RH is burned below 700 °C (He et al., 2017; Prasara-A & Gheewala, 2017).

Although amorphous silica can be produced through controlled burning of RHA at 500 to 700 °C, acquiring high purity materials is not always possible (Salas et al., 2009). Due to the temperature difference between the interior and exterior of the rice husk, two types of ash are produced: white RHA (WRHA) and black RHA (BRHA). WRHA and BRHA are the two forms of RHA, depending on whether the combustion was complete or incomplete (Ugheoke & Mamat, 2012). The controlled combustion of RH in the environment can produce WRHA containing >95% pure silica in a hydrated amorphous state with high porosity and reactivity (Vlaev et al., 2003). Controlled pyrolysis of RH in nitrogen or inert atmosphere yields BRHA with varying proportions of carbon and silica (Ghaly & Mansaray, 1999).

Burning time and the presence of oxygen influence the silica shape and surface area of RHA particles (Hwang & Chandra, 1996). Producing amorphous silica with a high surface area requires temperatures below 700 °C. Amorphous silica can also be produced by burning at temperatures below 500 °C with oxygen over an extended time (Muthadhi et al., 2007). Crystalline silica can be formed at temperatures above 800 °C. (Hwang & Chandra, 1996).

The raw rice husk will be heated in a furnace with a controlled temperature that begins at 10°C per minute and eventually escalates to 700°C per minute for approximately six hours before being cooled at room temperature (Thomas, 2018). The rice husk was treated with 1 N of hydrochloric acid for 24 hours, followed by thorough rinsing with water to neutralize the pH. The husk was then dried and reduced to ash in a muffle furnace at 600 °C for three hours.

On the other hand, rice husk is burned at a controlled temperature in a ferrocement furnace or sometimes in boilers to make RHA (Gautam et al., 2019). In a furnace, air ducts have two purposes: to deliver air to the husk during the combustion process and to serve as fire tunnels. Air ducts regulate the temperature of the disturbance. Attached to the air ducts that regulate the combustion temperature are electric fans. Additionally, air ducts minimize the carbon content of RHA. In the RHA combustion process, the boiler or furnace temperature is between 500 and 800 °C for 24 hours. After 24 hours, the temperature is roughly 52 °C. After 48 hours, the temperature of the ash is approximately 25 °C. At temperatures between 500 and 800°C, silica remains in an amorphous state.

The primary component of RHA is silica in amorphous and partially crystalline phases, with carbon as the principal contaminant and trace elements such as potassium and calcium also present (Chaudhary & Jollands, 2004; He et al., 2013) as depicted in Table 2.1. After burning at temperatures between 600°C–700°C for 2 hours, RHA will consist of 90%–95% SiO₂, 1%–3% K₂O, and about 5% of unburnt carbon (Singh, 2018). Assuming proper combustion, ash quality from various sources will not vary considerably.

Table 2. 1: Chemical composition of rice husk ash. (Antiohos et al., 2014; Chao-Lung et al., 2011; De Sensale, 2010; Giaccio et al., 2007; Madandoust et al., 2011; Park et al., 2016; Rößler et al., 2014; Zahedi et al., 2015).

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	MnO	Na ₂ O	K ₂ O	SO ₃	LOI
Value %	87.4	0.1	0.1	0.8	0.2	0	0	1.3	0.1	1.9
	93.2	0.8	0.6	1.1	0.6	0.2	0.7	3.4	0.4	8.5

*LOI is loss on ignition

2.2.2 Rice Husk Ash-Based Geopolymer Composite

RHA is an industrial waste or by-product of the burning of rice husks produced by a power plant. RHA is a highly porous particle with a small bulk weight and huge exterior surface area from burning rice husk. The silica and mineral forms of the RHA are significantly affected by the temperature and duration of combustion. Optimal burning conditions and time might create 90 to 95 percent silica content which is relatively an amorphous and crystal phase (Xu et al., 2012).

Due to reactive amorphous silica, it can be used as a pozzolanic material. RHA and fly ash (FA) are appropriate for geopolymer production because they contain high silica and alumina levels (Chindaprasirt et al., 2009). Several parameters influence geopolymer characteristics, including raw material reactivity, alkali activator quantity, and RHA composition. Fiber-reinforced lightweight geopolymer composites based on RHA and nano-alumina have been examined by Mohseni et al. (2019) for their mechanical and durability properties. Using scoria particles as lightweight aggregates (LWA) at replacement ratios ranging from 0 to 20 percent of the total aggregate volume is common. By volume, the binder is mixed with polypropylene fibers (PP)

that range from 0 to 1.0 percent by volume. PP fibers improved mechanical properties (particularly flexural strength) while LWA reduced compressive and flexural strength by a small amount.

Nana et al. (2021) studied the mechanical strength and microstructure of geopolymer composites based on metakaolin and volcanic ash, which were reinforced with reactive silica derived from RHA. In order to create the geopolymer composites, metakaolin was substituted for volcanic ash (VA), which ranged from 10–30 wt.% of VA and 0 to 20 wt.% of RHA. According to the findings, the mechanical and physical properties were affected mainly by the VA and RHA content. The optimal mechanical strengths (19.3 and 60.73 MPa for flexural and compressive strength, respectively) were obtained with the composite sample containing 20 wt.% of VA and 10 wt.% of RHA. The results showed that the mechanical strengths and the physical properties were primarily affected by the content of VA and RHA.

2.3 Cellulose Isolated from Durian Husk

2.3.1 Introduction to Durian Husk Cellulose Fiber (DHCF)

Durian rind is the most promising cellulosic resource due to its high cellulose content (60.54 %) and several components, namely hemicellulose (13.09 %) and lignin (15.45 %) (E'zzati et al., 2018). Cellulose, the most prevalent polysaccharide on Earth, is a linear β -1,4 glycosidic-linked polymer of glucose (Coseri, 2017). The β -conformation of cellulose positions functional groups such as -H and -OH alternately above and below the anhydroglucose unit plane (AGU). The total number of repeated structural units indicates the degree of polymerization (DP). Strong intramolecular or intermolecular hydrogen bonds exist between adjacent glucose units in the same chain

or different chain via the open hydroxyl groups present in glucose monomer units (Dufresne, 2012; Phanthong et al., 2018).

Alpha cellulose (α -cellulose) is a chemical substance used to manufacture nitrocellulose (Astika et al., 2017). This cellulose can be extracted from numerous plant sources. α -cellulose is long-chain cellulose insoluble in a 17.5 percent NaOH solution or a 600 - 1500 DP strong base solution. α -cellulose is utilized as a predictor and determinant of cellulose purity.

Cellulose from the durian rind was made in two processes (Tawakkal et al., 2012). The initial stage is the synthesis of holocellulose through chlorination or bleaching. The skin of the durian was ground using a grinder. After rinsing 20 g of samples with tap water to remove dust, they were soaked with 640 mL of distilled water in a 1000 mL beaker. Next, 4 mL of chloric acid, CH_3COOH , and 8 g of sodium chlorite, NaClO_2 were added to the beaker every hour for 5 hours, during which time the lignin was entirely separated from the fiber. The sample was then left overnight in the water bath. It was then washed and rinsed with tap water until the yellow hue (holocellulose is white) and chlorine dioxide odor was eliminated.

In the second step of this procedure, holocellulose is converted to cellulose by mercerization at room temperature. 80 mL of 17.5 percent NaOH was added to the holocellulose prepared in the previous step, and the mixture was thoroughly agitated with a glass rod. After that, 40 mL of NaOH solution was added to the mixture 3 times at 5-minute intervals. The combination was allowed to rest for 30 minutes, bringing the total time for NaOH treatment to 45 minutes. After adding 240 mL of distilled water and allowing it to stand for 1 hour, the mixture was filtered. Then, 800 mL of an 8.3 percent NaOH solution was added to the cellulose for 5 minutes before being washed with water. The alkaline cellulose was then neutralized by adding 120 mL of

acetic acid at a concentration of 10 percent for 5 minutes. The cellulose was then filtered, washed, and rinsed with distilled water until the cellulose residue was acid-free and then dried overnight at 80 °C in a vacuum oven.

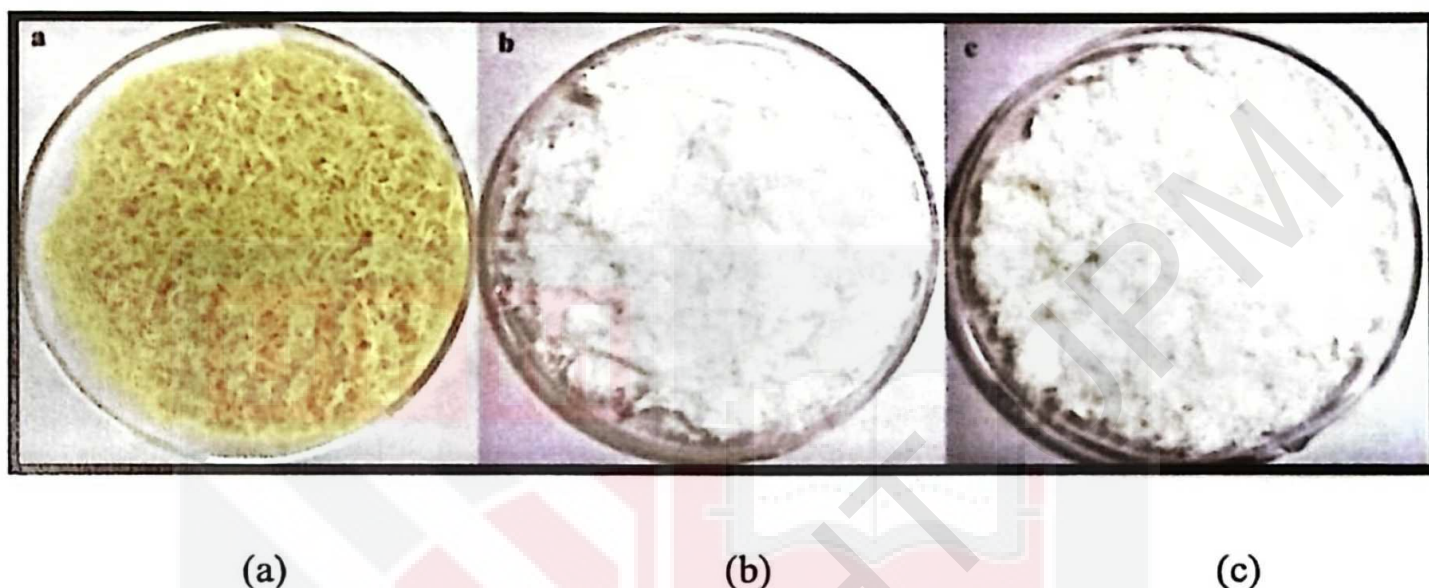


Figure 2. 2: (a) Untreated durian rind, (b) holocellulose, and (c) cellulose (Tawakkal et al., 2012)

On the other hand, 75 g of cleaned durian rind was steeped in 1000 mL of 3.5 percent HNO_3 and then autoclaved (Lubis et al., 2020). The resulting fiber was washed multiple times with distillate water until the pH was neutralized. The procedure was proceeded by dispersing the fiber in 750 mL of NaOH (2 wt.%) and refluxing it for 30 minutes at 70 – 80 °C. The fiber was then rinsed with distilled water until the pH was neutral, submerged in 500 mL of NaOCl (1.75 wt.%) at 70 - 80 °C for 30 minutes, and then washed until the pH was neutral. α -cellulose was produced by treating the fiber with 500 mL of NaOH (17.5 wt.%) and 500 mL of H_2O_2 (10 wt.%) at 70 - 80 °C for 30 minutes, followed by washing until the pH was neutral.

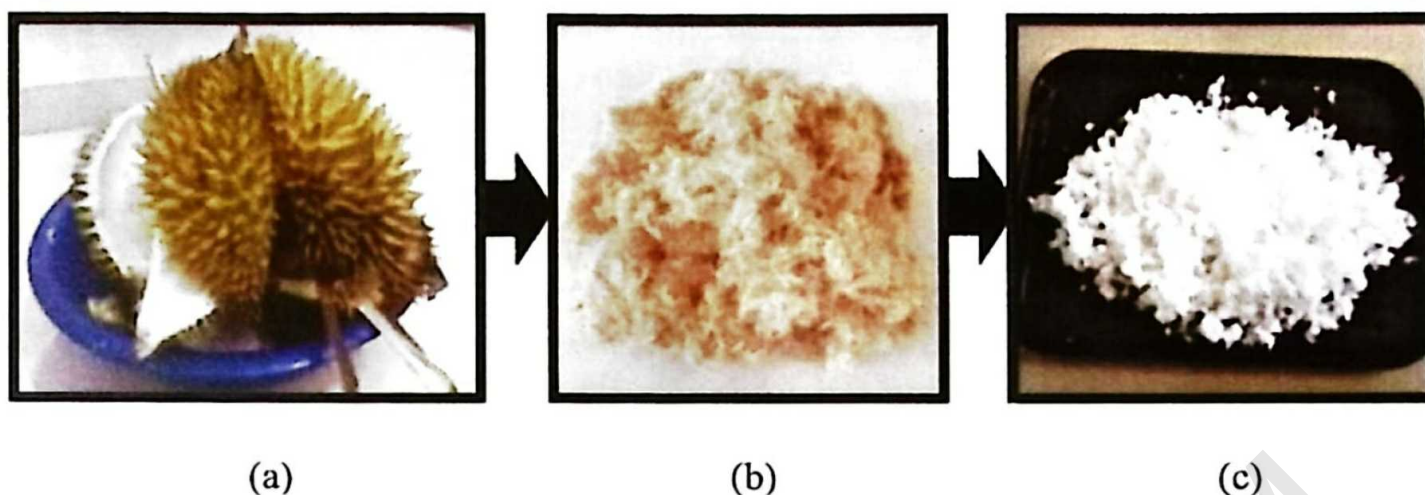


Figure 2. 3: (a) Durian rind waste, (b) durian rind fiber and (c) cellulose fiber (Lubis et al., 2020)

The chemical composition of natural fiber significantly impacts the fiber's mechanical property, water absorption behavior, fire resistance, resilience, and biodegradability (Indran & Raj, 2015; Penjumras et al., 2015). As seen in Table 2.2, the durian rind has a large amount of cellulose, approximately 57 percent. The high cellulose concentration significantly affected the mechanical characteristics of the fiber. Cellulose is a structural component that gives plant and fiber cell walls strength and stability. Cellulose possesses rigidity, high crystallinity, and is insoluble in an organic solvent. According to Table 2.2, the cellulose concentration of durian peels is comparable to that of jute, flax, hemp, kenaf, and banana fiber.

The durian peel's hemicellulose concentration can weaken fibers and make them simple to degrade. The concentration of hemicellulose in durian rinds is approximately 30 percent. The fiber's lignin content affects its structure, properties, morphology, flexibility, and hydrolysis. A fiber with a higher lignin concentration will be more flexible than others. A fiber with a low cellulose concentration will typically have a high lignin content (Reddy & Yang, 2005).

Table 2. 2: The properties of durian rinds fiber. (Indran & Raj, 2015; Jabli et al., 2018; Lubis et al., 2020; Madurwar et al., 2013)

Fiber	Chemical Properties				Physical Properties		
	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Moisture (%)	Elongation (%)	Tensile Strength (MPa)	Young's Modulus (GPa)
Durian rinds (present work)	57.4	22.0	13.6	3.3	-	-	-
Jute*	64.4	12.0	11.8	1.1	1.5	393.0	26.0
Flax*	64.1	16.7	2.0	3.9	1.8	773.0	27.0
Hemp*	68.0	15.0	10.0	-	2.7	500.0	70.0
Kenaf*	31.0	20.3	8.0	-	3.2	1500.0	53.0
Banana fiber*	31.27	15.0	15.1	9.7	2.0	690.0	-
					4.0	930.0	-

*Properties of other fiber.

The mechanical qualities of durian fiber are affected by the cell wall structure and chemical compositions, particularly the amounts of cellulose, hemicellulose, and lignin (Lubis et al., 2020). The mechanical quality of durian rinds fiber was determined using tensile strength, Young's modulus, and elongation at break, as shown in Table 2.3. Tensile strength, Young's modulus, and elongation at break all indicated the presence of cellulose. According to Table 2.3, the tensile strength and Young's modulus of durian rind are comparable to those of hemp and cotton fiber.

Table 2. 3: The mechanical properties of durian rind fibers. (Lubis et al., 2020)

Sample	Gauge length (mm)	Tensile strength (MPa)	Strain (mm/mm)	Young's modulus (MPa)
Durian fiber	10	60.495	0.0106	5707.075
	20	112.924	0.0202	5590.297
	30	169.431	0.0302	5610.301
	40	226.488	0.0400	5662.222
	50	298.791	0.0499	5987.797

2.3.2 Utilization DHCF in Geopolymer Composite

Khedari et al. (2001) studied the new lightweight composite construction materials with low thermal conductivity. This study's primary purpose is to examine the thermal conductivity, compressive strength, and bulk density of a new lightweight construction material made of cement, sand, and waste fibers from a young coconut (*Cocos nucifera*) and durian (*Durio zibethinus*). The analysis demonstrates that adding these fibers can reduce the thermal conductivity and weight of the sample. Thermal conductivity of 0.3506 W/m K, compressive strength of approximately 3.3 MPa (incubate in water for ten days), and bulk density of 1456 kg/m³ characterize the durian specimen. The optimal ratio of durian fiber to cement is 10 percent. The optimal fiber length is <2 mm, and the sand particle size is <0.71 mm. The thermal conductivity of durian fiber cement is 79 percent lower than that of standard mortar. As a result, it might be utilized to restrict heat transfer into buildings and save energy.

The thermal conductivity of a porous material is inversely related to the specimen's void volume. These voids result from the packing of fibers. Generally, it is more difficult to align and pack densely shorter fibers than longer ones (Morrissey et al., 1985). Thus, the short fiber length creates many voids for a given fiber content,

resulting in a specimen with a low heat conductivity. In addition, thermal conductivity is closely related to fiber composition.

Consequently, the thermal conductivity decreases with increasing fiber content (Krenchel, 1975; Swamy, 1975). Moreover, the higher the voids ratio, the lighter the specimen and the lower its heat conductivity. It can be inferred that a material's heat-insulating effectiveness is inversely proportional to its density (Salas et al., 1986).

2.4 Properties of Fiber-Based Geopolymer Composite

2.4.1 Flexural Strength

The impact of rice husk silica and silicon carbide whiskers on the mechanical characteristics and morphology of sodium geopolymer was studied by Rahman, Shah, et al. (2020). According to the findings, when combined, rice husk ashes and silicon carbide whiskers boosted flexural strength by 27 percent and 97 percent, respectively. The addition of silica and silicon carbide whiskers improved flexural strength, suggesting that silica particles are compatible with metakaolin-based geopolymers and aid consolidation.

The effects of reactive silica from rice husk ash on the mechanical strength and microstructure of metakaolin/volcanic ash-based geopolymer composites were investigated by Nana et al. (2021). The geopolymer composites were prepared by replacing metakaolin with volcanic ash, VA (10–30 wt.%), and RHA (0–20 wt.%). According to the findings, the VA and RHA content had the greatest impact on mechanical strengths and physical attributes. The composite sample contained 20% VA and 10% RHA and exhibited the best mechanical strengths (19.3 and 60.73 MPa for flexural and compressive strength, respectively). It is due to the additive, which

boosts the $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio and improves the compactness and microstructure of geopolymer matrices.

2.4.2 Volume Expansion Ratio

Lee et al. (2019) investigated the influence of the ratio of GGBS/FA and the ratio of $\text{SiO}_2/\text{Na}_2\text{O}$ on the behavior of compressive strength and volume expansion ratio in geopolymer mortar. Results showed that each sample had a volume expansion ratio of 0.4 % or less, and the expansion ratio of $\text{SiO}_2/\text{Na}_2\text{O} = 1.4$ and 1.5 was the lowest at only 0.1 percent or less.

The volume expansion of an aluminum-waste-based porous geopolymer was examined by Leiva et al. (2019) concerning the fly ash-paved percentage and the setting temperature. Faster setting and higher gas hydrogen emission were observed when Pavai concentration was raised to 50% and setting temperature increased from 40 to 70°C. As a result, the volume expansion of the material is improved.

Liguori et al. (2019) developed a multifunctional porous material that combines the microporosity of the zeolites with the mesoporosity of the geopolymer matrix with micro to macro-sized pores. In order to optimize the operating conditions, the influence of the foaming agent (silicon) concentration, relative humidity, and curing duration on zeolite crystallization in the sample was investigated. Silicon content significantly affects the volume increase, although the pace of the foaming process remains constant. For instance, adding silicon increased its volume by 200 or 100 percent, depending on whether the paste included 1 or 0.5 wt.% silicon. Regardless of silicon quantity, expansion begins approximately 30 minutes after silicon addition and is accomplished in less than 10 minutes.

2.5 Factor Affecting the Geopolymer Properties

2.5.1 Curing Temperature

Li et al. (2022) studied the effect of curing temperature on high-strength metakaolin-based geopolymer composite (HMGC) with quartz powder and steel fibers. The main purpose is to investigate the influence of curing temperature (from room temperature to 90 °C) on the compressive and flexural strength of the proposed HMGC. The results indicate that heat curing can greatly accelerate the geopolymerisation process and increase the HMGC's early strength. However, prolonged heat curing at high temperatures (such as 90 °C for 7 days) will impair the HMGC's mechanical strength. Chemical shrinkage can be attributed to the expansion of pores and micro-defects in the gel phase of the HMGC during prolonged curing at high temperatures.

Sajan et al. (2021) studied the combined effect of curing temperature, curing period, and alkaline concentration on the mechanical properties of fly ash-based geopolymer composite. The primary purpose is to undertake a complete analysis of the effects of three governing parameters on the mechanical characteristics of a geopolymer, including the uniaxial compressive strength, Poisson's ratio, and Young's modulus of elasticity. The test findings indicate that the geopolymer's mechanical strength increases as the curing temperature rises from 20 °C to 60 °C. The reduced geopolymerisation reaction rate resulting in a high percentage of unreacted fly ash microspheres is responsible for the poorer strength at a lower curing temperature. Nonetheless, when the temperature is increased above 60 °C, in this case (80 °C), the mechanical strength decreases at the 12 M and 14 M alkaline concentrations for the 3-day curing period due to the formation of calcium silicate hydrate, increases slightly

at the 7-day curing period due to the progression of geopolymerisation, and is nearly the same at the 14 days due to the completion of geopolymerisation.

Azevedo et al. (2019) studied the effect of curing temperature, activator solution composition, and particle size in Brazilian fly ash-based geopolymer production. This study aimed to examine the production of fly ash-based geopolymers utilizing fly ash originating from specific thermoelectric power plants and to investigate the effects of the research materials solution, curing temperature, and particle size of the fly ash on the properties of the inorganic polymers produced. The rise in temperature to 90 °C favored the reactions of the synthesis of the N-A-S-H gel (geopolymer), and the mechanical strength increased to values close to 90 MPa after 24 hours. It is because the curing temperature employed in the first 24 hours affects the geopolymerisation reaction and, consequently, the development of the N-A-S-H gel.

2.5.2 Cellulose Content

Ye et al. (2018) studied the effects of cellulose, hemicellulose, and lignin on the morphology and mechanical properties of metakaolin-based geopolymer. This research aimed to examine the effect of the chemical composition of natural fibers, mainly cellulose, hemicellulose, and lignin, on geopolymer characteristics. According to the results, a low concentration (5 wt. %) of lignin, cellulose, and hemicellulose improved the flexural and compressive strength of pure geopolymer. Higher levels of lignin and hemicellulose resulted in a porous morphology, lower density, and brittle fractures in geopolymer-based composites, which decreased their flexural and compressive strength. The addition of cellulose fibers to geopolymer-based

composites resulted in a denser structure, fewer pores, practically constant density, and a distinct ductile failure due to the bridging mechanism of the fibers inhibiting crack development.

Taveri et al. (2018) studied the mechanical performance of glass-based geopolymer matrix composites reinforced with cellulose fibers. The objective is to compare the effects of chemical (alkalinity and silica-to-alumina ratio) and physical (relative humidity and soaking time) parameters on the mechanical and microstructural properties of geopolymers, in addition to the effect of fiber content in terms of bending strength, fracture toughness (chevron notch test), and fracture micromechanics. The mechanical properties of geopolymer samples were compared with those of cellulose fiber–geopolymer matrix composites containing varying amounts of fiber (1 wt.%, 2 wt.%, and 3 wt.%). The composites displayed greater strength and fracture resilience, with the greatest benefit reported at 2% fiber concentration. The flexural strength of samples of geopolymeric composites containing more than 2 wt.% cellulose decreases due to excessive Na^+ absorption by the cellulose fibers.

Roopchund et al. (2022) conducted an experiment using cellulose nanocrystals to improve the mechanical properties of fly ash-based geopolymer construction materials. The objectives of this study were to determine the effect of cellulose nanocrystals on the mechanical properties of fly ash-based geopolymers, to create a database of the effects of cellulose nanocrystals concentration and activator concentration on the mechanical properties of the formed geopolymers, and to develop an empirical framework for the development of green building materials. Low concentrations of cellulose nanocrystals (less than 0.5 percent) improved the mechanical characteristics of geopolymers.

2.5.3 Curing Time

Pumice-based geopolymer paste has been studied by Safari et al. (2020) for the effect of curing time on the mechanical properties of this paste. Samples were cured at room temperature, 60, 80, and 100 °C for 24, 48, 72, and 120 hours with varied alkali concentrations (8, 10, 12, 14, 16, and 18 M). As shown in Figure 2.4, the strength of the geopolymer increases proportionally to the increase in curing time up to 72 hours, but after that point, the strength gradually declined as the curing time rose. After 72 hours of curing at 60 °C, most polymerizations were achieved. In addition, the findings indicate that high early strengths may be accomplished by curing the material at elevated temperatures for a shorter amount of time. The faster geopolymerisation reaction may do with this highly early strength development. Compared with lower curing temperatures and longer curing times; however, the benefits in compressive strength are not as significant. Other studies have also concluded that a longer curing period increases the polymerization process. These studies also discovered that the optimal compressive strength was achieved after curing for 96 hours at 60 °C (Hardjito & Rangan, 2005; Joseph & Mathew, 2012). Moreover, they mentioned that the increase in strength after curing for 48 hours does not constitute a substantial change.

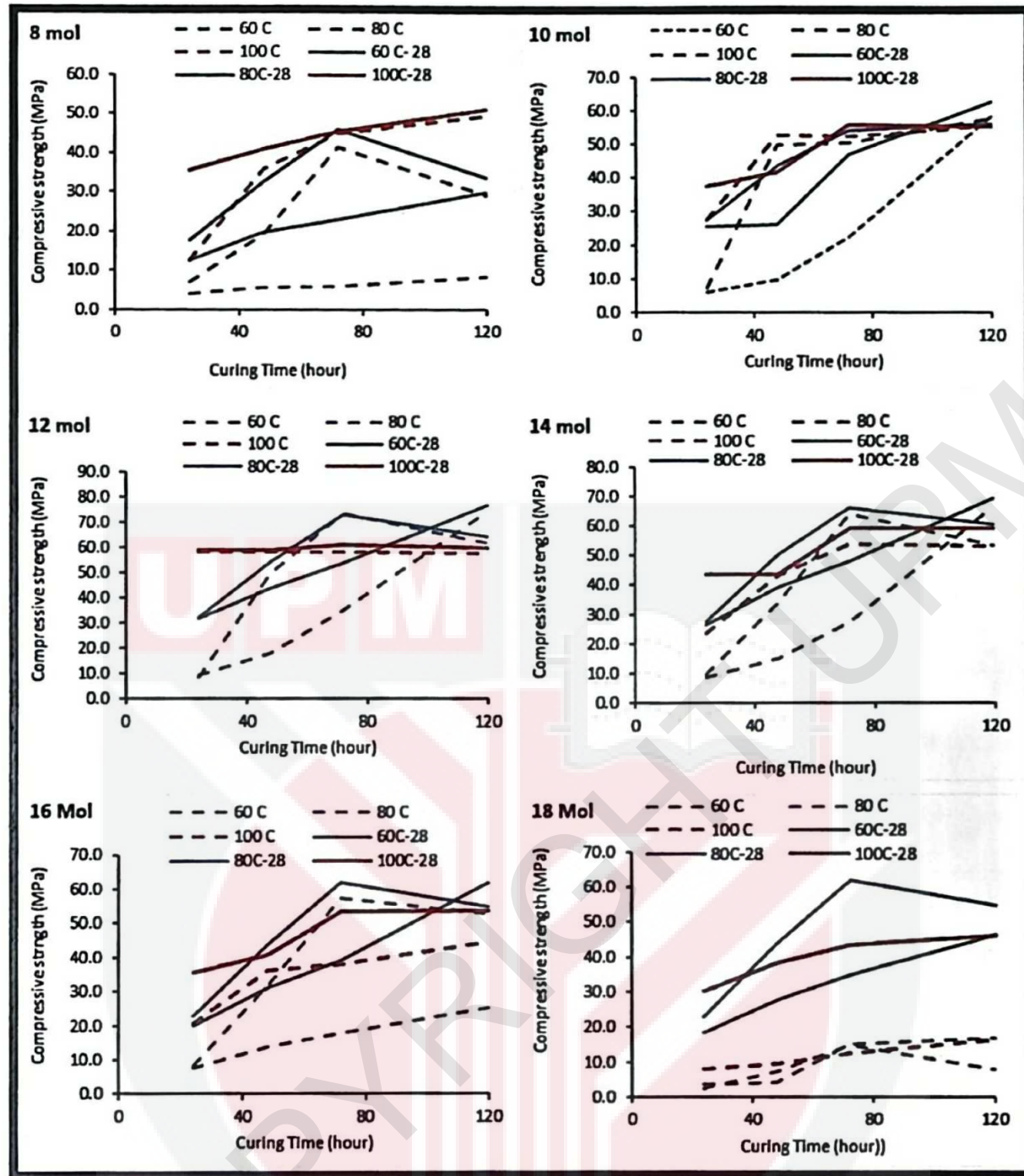


Figure 2. 4: Effect of both curing period and temperature curing on 28-day compressive strength of AAM with different molarities.(Safari et al., 2020)

2.6 Response Surface Methodology (RSM)

2.6.1 Introduction to RSM

According to Baş and Boyacı (2007), the most popular optimization strategy in recent years has been response surface methodology (RSM), which involves mathematical and statistical data to investigate various process parameters. It also

included an empirical modeling technique to assess the relationship between multiple process variables and responses and the various requirements required and analyze the impact of these process factors on the combined replies (Han et al., 2015).

The RSM approach minimizes the number of tests necessary to create useful data from a set of inputs, minimizing the cost of each experimental run. This kind of simulation is viewed as a quick and easy way to screen and illustrate the functions of each variable involved in the large experimental region (Basri et al., 2020).

Zahid et al. (2018) studied the statistical modeling and mix design optimization of fly ash-based engineered geopolymer composite using response surface methodology. The primary objective was to apply the RSM optimization technique to model the effects of principal variables (NaOH molarity, NaOH/Na₂SiO₃ and curing temperature) on the setting time, compressive strength, elastic modulus, first crack strength, flexural strength, flexural toughness, and ductility index of engineered geopolymer composites (EGC). A central composite design (CCD) method was used. The results indicated that all models are significant since the difference between predicted R² and adjusted R² was less than 0.2. In the present investigation, setting time, compressive strength, elastic modulus, first crack strength, flexural strength, flexural toughness, and ductility index were maximized, and all variables (NaOH molarity, curing temperature, and NaOH/Na₂SiO₃) were kept within range.

Demir and Derun (2019) studied the response surface methodology application to fly ash-based geopolymer synthesized by alkali fusion. The main objective of this paper is to examine the effects of various synthesis parameters on geopolymerisation. The curing temperature and Al₂O₃ impact were determined as synthesis parameters. A central composite design (CCD) method was used. The optimal Al₂O₃ addition and

curing temperatures were determined to be 70.76 percent and 36.75 °C, respectively, based on optimization and analytical results. The optimal geopolymer sample exhibited a 97 percent degree of geopolymerisation response.

Zhao et al. (2021) studied the optimization for preparing composite geopolymer using response surface methodology and its application in lead-zinc tailings solidification. One objective of this research was to explore the interrelation of variables (i.e., water glass module, alkaline content, and curing temperature) and compressive strength by using RSM, as well as to optimize synthesis process conditions to obtain higher strength of the matrix. The main objective of this study was to investigate the relationship between factors (water glass module, alkaline content, and curing temperature) and compressive strength using RSM, as well as to improve synthesis process parameters to increase the matrix's strength. A central composite design (CCD) method was used. Under optimal conditions, the maximum compressive strength was determined to be 91.13 MPa, as determined by the results. The optimum water glass module was 1.06, the optimum alkaline content was 13.81 percent, and the optimum curing temperature was 30 °C.

Mohd Basri et al. (2020) studied the optimization of adhesion strength and microstructure properties by using response surface methodology to enhance the rice husk ash-based geopolymer composite coating. The primary purpose of this research was to use statistical analysis to optimize the adhesion strength of a rice husk ash-based geopolymer composite coating (GCC) and to investigate its microstructure. A central composite design (CCD) method was used. Results showed that with an RHA/AA ratio of 0.25 and a curing temperature of 75 °C, the optimal adhesion strength is 4.75 MPa. The microstructure investigation demonstrated that a coating with a high adhesion strength had excellent interfacial adhesion with the substrate.

Chen et al. (2022) studied the modeling and optimization of fly ash-slag-based geopolymer using the response surface method and its application in soft soil stabilization. This study aims to optimize the process parameters of FA-slag-based geopolymer pastes and expand their application area in soft soil site ground improvement projects. Three-level Box Behnken was used. The optimal conditions were determined to be 9.988 percent alkali equivalent, 1.030 activator modulus, and 0.328 slag replacement ratio, as well as the worldwide desirability of the anticipated compressive strength values of 3 days (28.10 MPa) and 28 days (54.69 MPa).

Zhang et al. (2020) studied the preparation of spent fluid catalytic cracking catalyst (SFCC)-metakaolin-based geopolymer and its process optimization through the response surface method. This research aims to determine the impacts of 4 variables, including SFCC catalyst content, solid alkali activator to raw material ratio, H₂O to raw material ratio, and SFCC catalyst D50, on the compressive strength of SFCC catalyst-metakaolin based geopolymer using RSM. The Box-Behnken technique was utilized. According to the quadratic model, replacing metakaolin with an adequate amount of SFCC catalyst increases the compressive strength of geopolymers, whereas replacing metakaolin with an excessive amount of SFCC catalyst decreases the compressive strength of geopolymers. With a 20 percent replacement of SFCC catalysts, the greatest compressive strength of 41.22 MPa was reached, 21.6 percent greater than the geopolymer without SFCC catalyst.

2.7 Potential Application of Geopolymer Composite in Food Industry

There have been a number of applications of geopolymers as cements and concretes (Gourley & Johnson, 2005; Huiskes et al., 2016; Kong & Sanjayan, 2010;

Pan et al., 2009; Zhao & Sanjayan, 2011), heat and fire resistant materials (Ali et al., 2017; Barbosa & MacKenzie, 2003; Büchler, 1999; Cheng & Chiu, 2003; Davidovits, 1991; Davidovits, 1999; Galiano et al., 2017; Liefke, 1999; Pan & Sanjayan, 2010; Pan et al., 2012; Pistol et al., 2016; Shaikh & Hosan, 2016; Wang & Zhao, 2019; Zhang et al., 2014; Zhang et al., 2018), thermal insulator (Büchwald et al., 2004; Liguori et al., 2017; Natali Murri et al., 2017; Novais et al., 2016; Rickard & Van Riessen, 2014; Rickard et al., 2013; Roviello et al., 2015; Sakkas et al., 2013; Samal et al., 2015; Temuujin et al., 2011), thermal energy storage concretes (Colangelo et al., 2017), refractory material (Chindaprasirt & Rattanasak, 2018; Kamseu et al., 2015; Kamseu et al., 2010; Perera & Trautman, 2005), intumescent coating for steel fire protection (Carabba et al., 2019; Watolla et al., 2017), and precursors to ceramic formation (Bell et al., 2009a, 2009b). It also has applications in high-tech composites (Flowerday et al., 2006; Giancaspro et al., 2006; Lyon et al., 1997; Papakonstantinou & Balaguru, 2007; Ribero & Kriven, 2016) and medicine (Jämstorp et al., 2010).

One of the applications is thermal insulation. From grain storage silos to anaerobic digesters, insulation offers the food processing and agribusiness industries the same advantages as its numerous other applications, including enhanced process control, condensation and mould growth control, protection from temperature variance (from freeze protection to heat loss), and cost and energy savings. Moreover, considering insulation's role in enhancing the efficiency of anaerobic digestion, reducing the total amount of landfill gas in the air, and contributing to the formation of a renewable energy source, its green value and benefits rise tenfold.

Generally, thermal insulation materials with low thermal conductivity have been developed and utilized extensively in the construction industry (Al-Homoud, 2005; Jelle, 2011). However, the insulating materials' mechanical characteristics are

insufficient for use as structural members alone. As a result, the sandwich structure with insulation material as the core layer offers a viable solution to this issue. The sandwich structure has established a reputation in recent years as an effective building construction technique due to its high strength-to-weight ratio and acceptable levels of acoustic and thermal insulation (Cui et al., 2021). A typical sandwich construction consists of skin layers that are sufficiently rigid to handle compressive and tensile loads and a core layer that is sufficiently thick to withstand shear force. Typically, an adhesive is utilised to bind the skin layers and core layer together. Normal skin layers are composed of a range of substances, such as steel or aluminium, fiber-reinforced polymer, and cement-based composites. In contrast, typical core layer materials include polymeric foams, mineral wool, and balsa wood.

Besides that, Jämstorp et al. (2010) studied the mechanically strong geopolymers that offer new possibilities in treating chronic pain. The researchers argue that geopolymers, a family of materials derived from clay, may solve the problem of locating controlled-release materials with the requisite characteristics for safe and sustained oral delivery of highly strong opioids. They demonstrate that the opioid Fentanyl and the structurally related sedative Zolpidem may be encapsulated in metakaolin-based geopolymer pellets to create sustained-release dose forms with mechanical strengths comparable to human teeth. The results given in this study may provide new options for the future development of drug delivery systems for highly potent pharmaceuticals utilizing geopolymers and materials with variable pore structure and high strength.

In addition, Topal (2020) conducted a study on using geopolymer composites for nitrogen and phosphorus removal from poultry litter. With a growing global population, the demand for animal agriculture will increase significantly in the next

years to meet humankind's nutritional requirements. The poultry industry is a major branch of the meat industry. The improper disposal of chicken litter or its usage as manure can lead to the leaching of nitrates and phosphates, contaminating aquifers, a vital clean water source. Current waste management strategies are not sustainable in the long run since they rely heavily on disposal. On the other hand, geopolymers are non-crystalline aluminosilicate materials that offer various advantages, including a higher nutrient removal capability, reduced cost needs, and, most importantly, a high adaptability to various applications.

CHAPTER 3

METHODOLOGY

3.1 Overview

The fabrication of durian husk cellulose fiber/rice husk ash-based geopolymer composite used rice husk ash (RHA), alkaline activator (AA), and durian husk cellulose fiber (DHCF) as main materials. Factors that affect the properties of the geopolymer composite are the curing temperature, cellulose content, and curing time. The experiment is designed and optimized using response surface methodology (RSM). Prepared samples are subjected to the flexural strength test and volume expansion ratio. Studies on the microstructure analysis using a scanning electron microscope (SEM) are also conducted. The optimum formulation is then validated. Figures and 3.2 show the sample preparation and sample testing procedure.

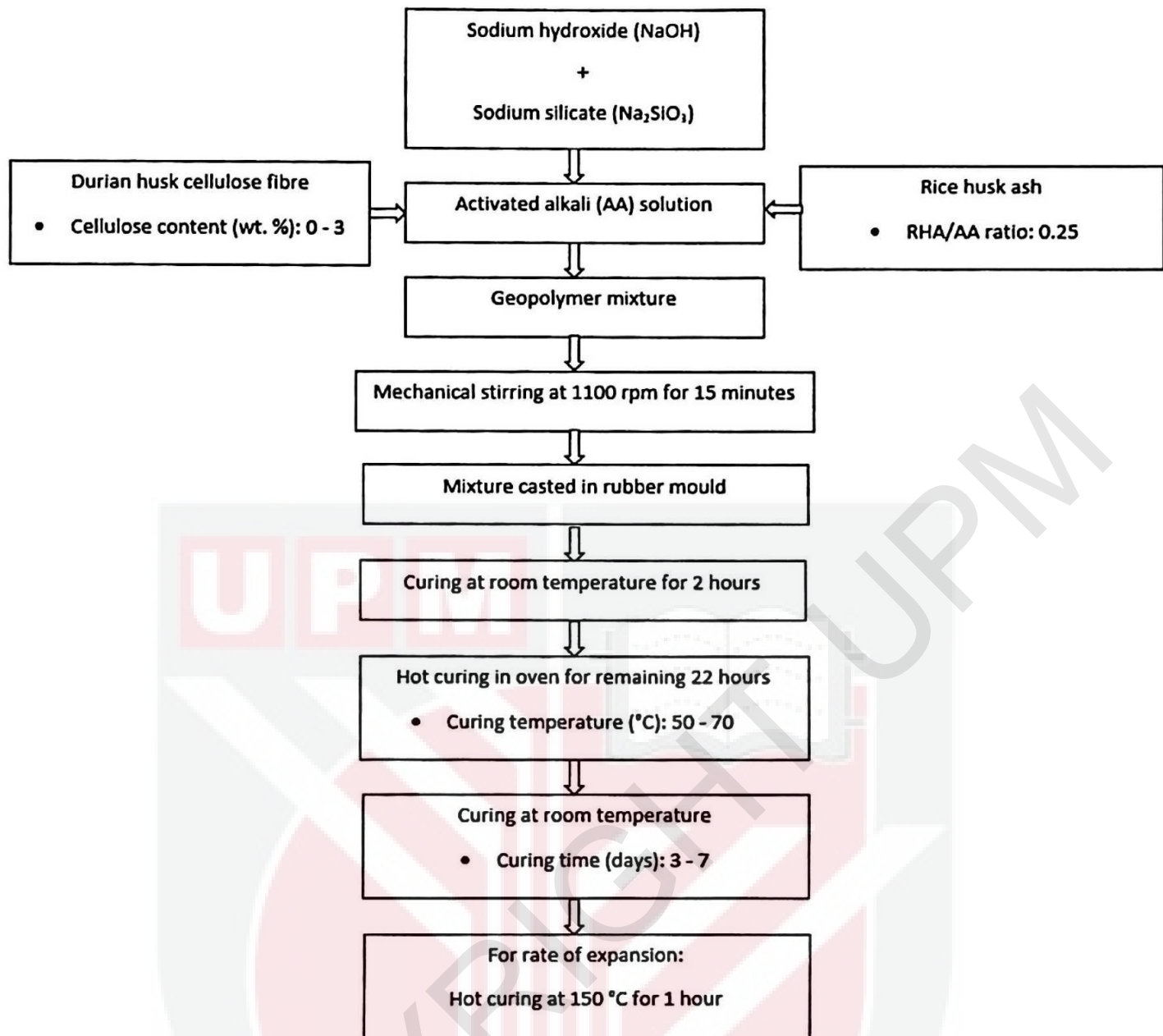


Figure 3. 1: Flowchart of the DHCF/RHA-based geopolymer composite preparation.

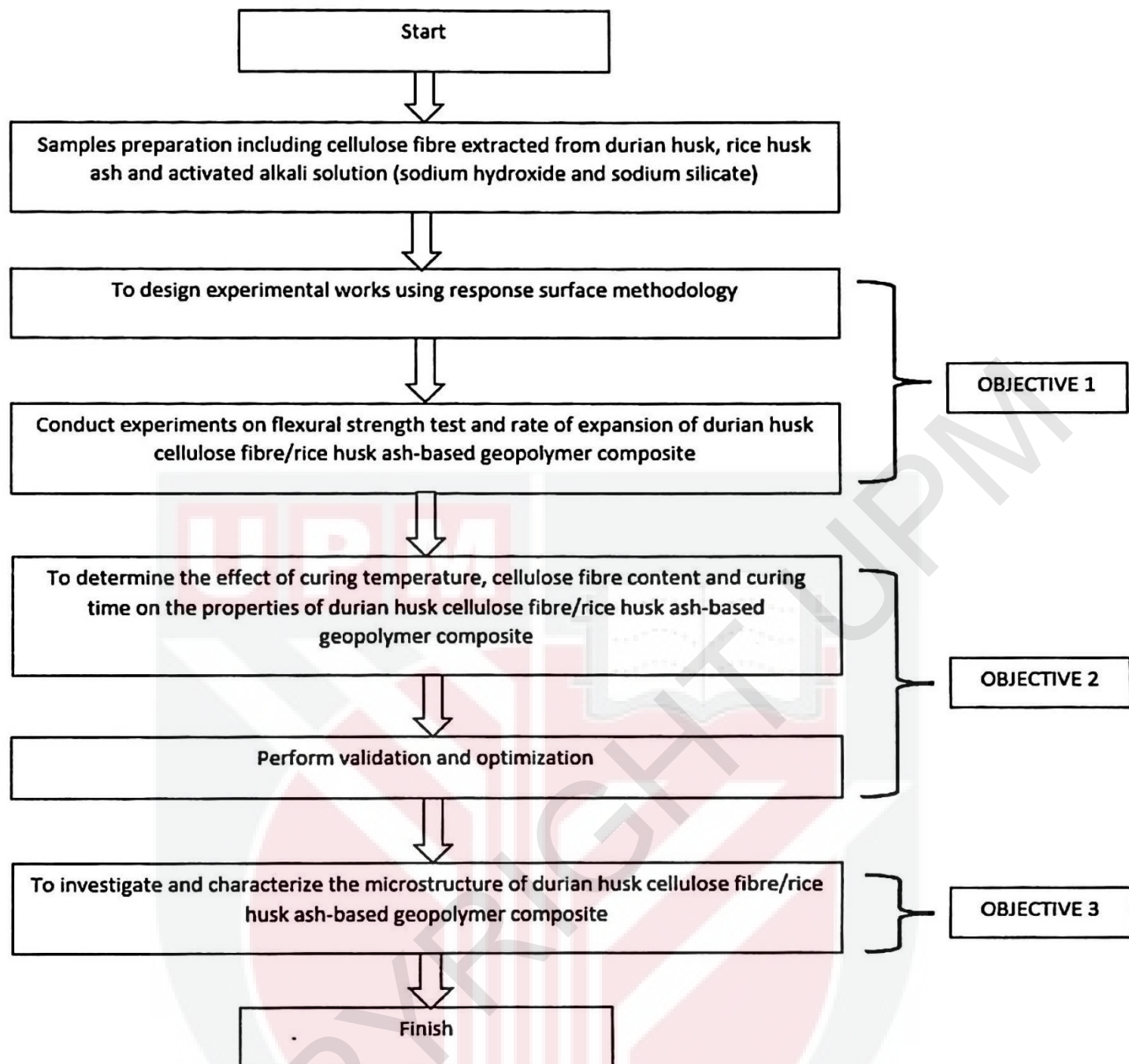


Figure 3. 2: Flowchart of the DHCF/RHA-based geopolymer composite testing procedure.

3.2 Materials

3.2.1 Rice Husk

Rice husk (RH) as depicted in Figure 3.3 was obtained from Maero Tech Sdn. Bhd. (Nilai, Negeri Sembilan). RH consists of 70 to 90 % of the organic matter, such as cellulose and lignin, and the remainder consists of minerals such as silica and alkalis (Karera et al., 1986).



Figure 3. 3: Rice husk. (The Star, 2022)

Table 3.1 demonstrates a typical study of rice hulls. The composition of each element in the table depends on rice variety, soil chemistry, meteorological circumstances, and the geographical location of the crop (Chakrabarti et al., 1985).

Table 3. 1: Composition of rice husk. (Kumar et al., 2012)

Property	Bulk density	Hardness	Ash (%)	C (%)	H ₂ (%)	O ₂ (%)	N ₂ (%)	S (%)	Moisture (%)
Range	96	5	22	35	4	31	0.20	0.04	8
	–	–	–		–	–	–	–	–
	160	9	29		5	37	0.32	0.08	9

3.2.2 Activated Alkaline Solution

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) obtained from Evergreen Engineering Resources (Semenyih, Selangor) were used as alkaline activators. The activated alkaline solution is an important component in the geopolymerisation process since the breakdown of aluminosilicate sources requires alkali hydroxide. In contrast, sodium silicate solution serves as a binder, alkali activator, and dispersant or plasticizer (Liew et al., 2011).

A mixture of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate solution is the most common alkaline solution used in geopolymerisation. A previous study has found that combining sodium hydroxide and sodium silicate solutions produces an efficient alkaline solution (Fernández-Jiménez et al., 2005; Hardjito et al., 2004; Palomo et al., 1999; Xu & Van Deventer, 2000). Figure 3.4 shows the activated alkaline solution used in the experiment.

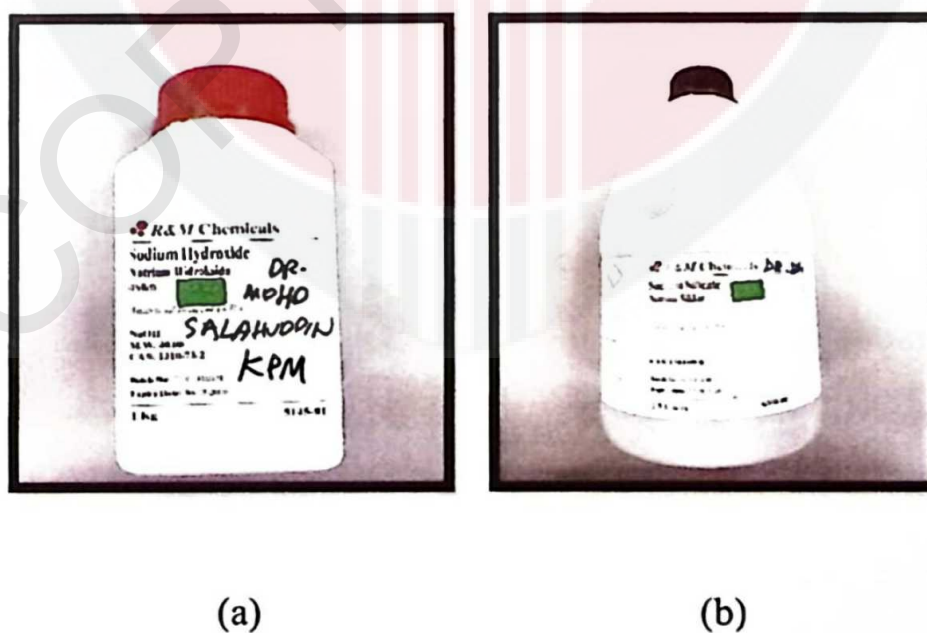


Figure 3. 4: (a) Sodium hydroxide and (b) sodium silicate.

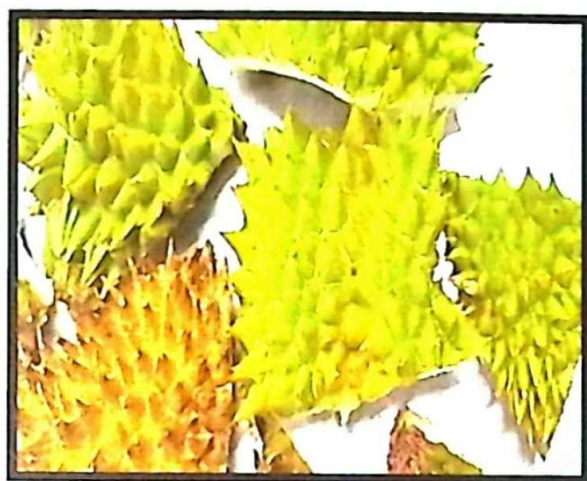
3.2.3 Durian Husk

In the experiment, durian husk is employed as a source of cellulose fiber bought from the local market. As seen in Figure 3.5, the white inner husk is removed from the entire durian husk, leaving the green thorny outer husk. Only the green thorny outer husk is used instead of the entire durian husk because the green thorny outer husk contains more crude fiber than the white inner husk.



Figure 3. 5: White inner and thorny outer husks of durian rind.

Based on Figure 3.6, the durian husk was cleaned to eliminate dust and grime before being sliced into smaller pieces. The material is then dried in an oven at 60 °C overnight and ground into shreds. Ground durian husk is dried in an oven to remove excess moisture and kept in an airtight container for use in cellulose extraction.



(a)



(b)



(c)



(d)



(e)



(f)

Figure 3. 6: Durian husk preparation process; (a) washing, (b) cutting, (c) drying, (d) grinding, (e) drying ground husk, and (f) place in an airtight container.

3.2.4 Cellulose Extraction Reagents

Figure 3.7 shows sodium chlorite (NaClO_2), and acetic acid (CH_3COOH)), which are acquired from Evergreen Engineering Resources (Semenyih, Selangor) for the extraction of durian husk cellulose fiber.

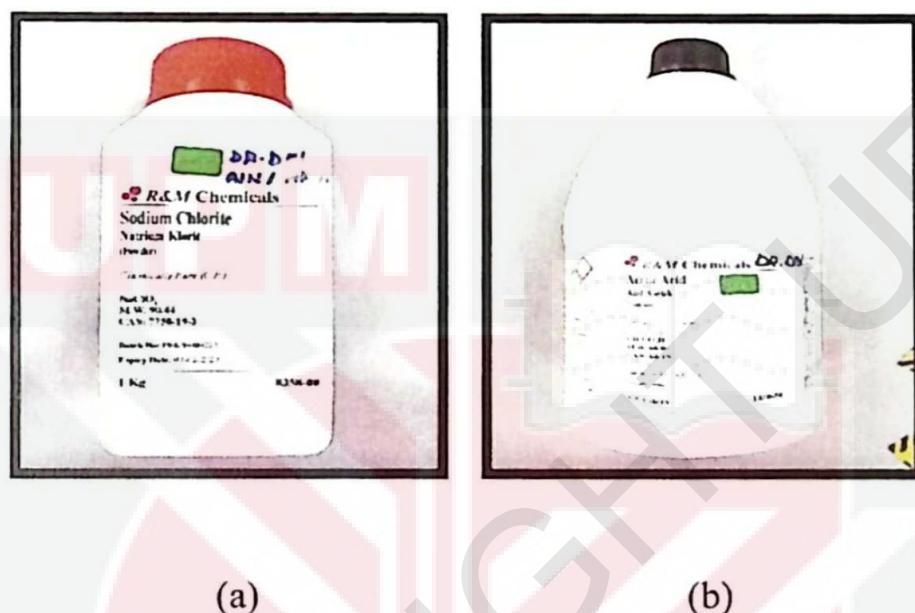


Figure 3. 7: (a) Sodium chlorite and (b) acetic acid.

Holocellulose was produced from durian husk fiber. Delignification necessitates the use of NaClO_2 and CH_3COOH . Mercerization, which transforms generated holocellulose into cellulose, requires NaOH solutions of 17.5 percent and 8.3 percent w/v and CH_3COOH solutions of 10 percent v/v. The appropriate amount of ingredients was dissolved in distilled water.

3.3 Experimental Design and Analysis

3.3.1 Response Surface Methodology (RSM)

The experimental design and analysis were conducted using MINITAB software (Minitab Statistical Software, Minitab, LLC, Pennsylvania State University). The study involves three factors which are curing temperature (V_1), cellulose content (V_2), and curing time (V_3). Table 3.2 shows the factors and levels being studied.

Table 3. 2: Factors and levels of the experiment.

Factors	Unit	Notation	Levels		
			-1	0	1
Curing Temperature	°C	V_1	50	60	70
Cellulose Content	wt.%	V_2	0	1.5	3
Curing Time	day	V_3	3	5	7

The total number of runs was 15 runs. After analysis, an optimum composition of geopolymer was obtained. The contour plots illustrate the relationship between factors and their effects on the responses. The validation process was performed by comparing the optimum and experimental results to verify the accuracy of the prediction made by MINITAB software. Contour plots superimposition were developed to provide useful information and guidance on the optimum range or contour for research and industrial purpose.

Factors and responses (flexural strength and volume expansion rate) were first determined. The MINITAB software created a response surface design using the Box-Behnken design (based on three factors). The difference between levels for curing temperature, cellulose content, and curing time is 10 °C, 1.5 wt.%, and two days, respectively. The software generates the summary and design table of the experiment

as depicted in Table 3.3, and the excel worksheet was created based on the design table in MINITAB.

Table 3. 3: Matrix design.

Sample	Coded Factor			Uncoded Factor		
	V ₁	V ₂	V ₃	V ₁	V ₂	V ₃
S1	0	0	0	60	1.5	5
S2	1	1	0	70	3.0	5
S3	-1	0	-1	50	1.5	3
S4	-1	1	0	50	3.0	5
S5	1	-1	0	70	0.0	5
S6	-1	-1	0	50	0.0	5
S7	0	0	0	60	1.5	5
S8	0	0	0	60	1.5	5
S9	0	-1	1	60	0.0	7
S10	0	1	-1	60	3.0	3
S11	0	1	1	60	3.0	7
S12	1	0	1	70	1.5	7
S13	0	-1	-1	60	0.0	3
S14	1	0	-1	70	1.5	3
S15	-1	0	1	50	1.5	7

Testing was conducted following the matrix design. The testing results were analyzed using MINITAB software under a full quadratic residual plot.

The confidence level selected in this analysis was 95%, indicating that the factor is significant when the P-value is below 0.050 and vice versa. The regression model was developed from the ANOVA table and presented in the form of a mathematical equation. The contour plot was generated to study the relationship between factors and responses. Results from the analysis are displayed in the ANOVA table.

For the optimization process, the objective of the response optimizer was to obtain the maximum flexural strength and volume expansion rate. The optimum value for the factors was obtained from the optimization plot. The superimposition plot overlapped the contour plot of flexural strength and volume expansion rate. The overlapped region indicated the geopolymer composition, which possesses maximum flexural strength and volume expansion rate.

3.4 Sample Preparation

3.4.1 Durian Husk Cellulose Fiber (DHCF)

From Figure 3.8, the durian husk cellulose fiber (DHCF) was prepared using two-step processes with some modifications, according to Penjumras et al. (2014). The first step of the process is the production of holocellulose through the delignification method, also known as the chlorination or bleaching process. The durian husk was ground in a grinder, followed by 20 g of the samples rinsed with tap water to eliminate dust before being soaked in 640 mL of distilled water in a 1000 mL beaker. Next, 4 mL of CH_3COOH and 8 g of NaClO_2 were added to the beaker hourly for 5 hours to separate lignin from fiber completely. The sample was then placed in the water bath and left overnight. Then, it was washed and rinsed with tap water until the yellow samples turned white, indicating the colour of the holocellulose and the chlorine dioxide odor was eliminated.

The second step is the conversion of holocellulose to cellulose through the mercerization method at room temperature. 80 mL of 17.5 percent NaOH was added to the holocellulose prepared in the previous step, and the mixture was thoroughly stirred with a glass rod. Then, another 40 mL of NaOH solution was added to the mixture 3 times at 5-minutes intervals for 45 minutes of NaOH treatment, followed by adding 240 mL of distilled water and allowing it to stand for 1 hour before filtering. Next, 800 mL of 8.3 percent NaOH solution was added to the cellulose for 5 minutes before rinsing with water. After that, 120 mL of 10 percent acetic acid was added to neutralize the alkaline cellulose for 5 minutes. Finally, the cellulose was filtered, washed, and rinsed with distilled water until it was acid-free and then dried overnight in a vacuum oven at 80°C.

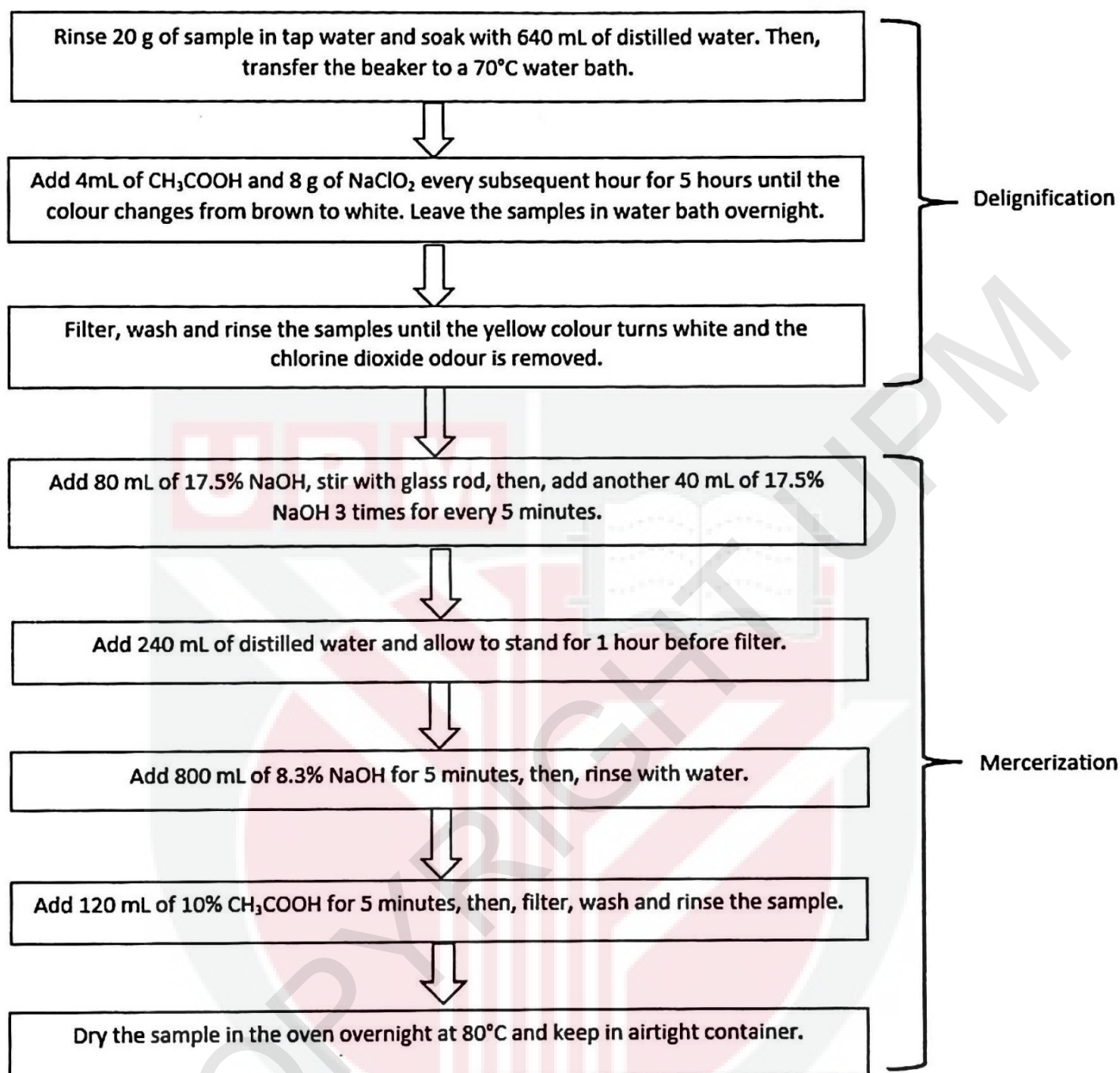


Figure 3. 8: Flowchart of DHCF preparation.

3.4.2 Rice Husk Ash

Rice husk ash (RHA) acquired from the supplier has been burned at a predetermined temperature and duration. The grinding machine (RT-N08) is used to reduce the particle size. Next, RHA is sieved using a control sieve shaker (Retsch AS200) to obtain RHA with a particle diameter of $<125 \mu\text{m}$, as depicted in Figure 3.9.

The diameter of the sieved particles is subsequently evaluated using particle size analyzers (Malvern Mastersizer 2000).



Figure 3. 9: Rice husk ash. (Deelert Intertrade Co., Ltd., n.d.)

Prior to grinding, RHA particle size ranges from 1 to 100 μm (Mohd Basri et al., 2021). As the first structure of the RH, the particles emerge as plates and thin shell-like structures with rectangular indents on the surface. In addition, the porous and cellular surfaces of RHA result from sponge-like particles. In order to enable pozzolanic activity, the RHA particles must be reduced to exceedingly tiny particle sizes. Table 3.4 depicts the physical parameters of the RHA ground surface.

Table 3. 4: Physical properties of RHA. (Mohd Basri et al., 2021)

Physical Properties	RHA
Particle Size	< 125 μm
Colour	Light grey
Structure	Powder form
Odour	Non

Rich in silica, burnt ash is utilized as a reinforcement in the production of composites. Rice husk has 10 to 25 percent more ash than other biomass fuels; its 85 to 87 percent silica content demonstrates its high porosity, light weight, and large exterior surface area (Rozainee et al., 2008). Burning conditions (temperature and duration), heating rate, crop incineration technique, and fertilizer used in rice cultivation on agricultural land all affect the ash quality (Mansaray & Ghaly, 1999). Table 3.5 shows a typical analysis of RHA.

Table 3. 5: Chemical composition of rice husk ash. (Saravanan & Kumar, 2013)

Constituent	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	N ₂ O	K ₂ O	LOI
%	94.04	0.25	0.14	0.62	0.44	0.02	2.49	3.52

*LOI is loss of ignition

3.4.3 Durian Husk Cellulose Fiber/Rice Husk Ash-Based Geopolymer Composite

Figure 3.10 illustrates the methods for sample preparation. First, an AA solution is made by combining sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH) in a set ratio of 3.5. The mixture is then manually stirred. The mixture is swirled while RHA and DHCF are added at a ratio of 0.25. Before being poured into a rubber mould, the liquid is mechanically agitated for 15 minutes at 1100 rpm.

The samples are then cured at ambient temperature for two hours before undergoing a 22-hour oven curing. The samples are then allowed to continue curing at room temperature. Following this, samples are prepared for flexural strength testing.

In order to determine the rate of expansion, samples must be re-cured in an oven at 150 °C for 1 hour, then left to cool for a while and tested for their properties.

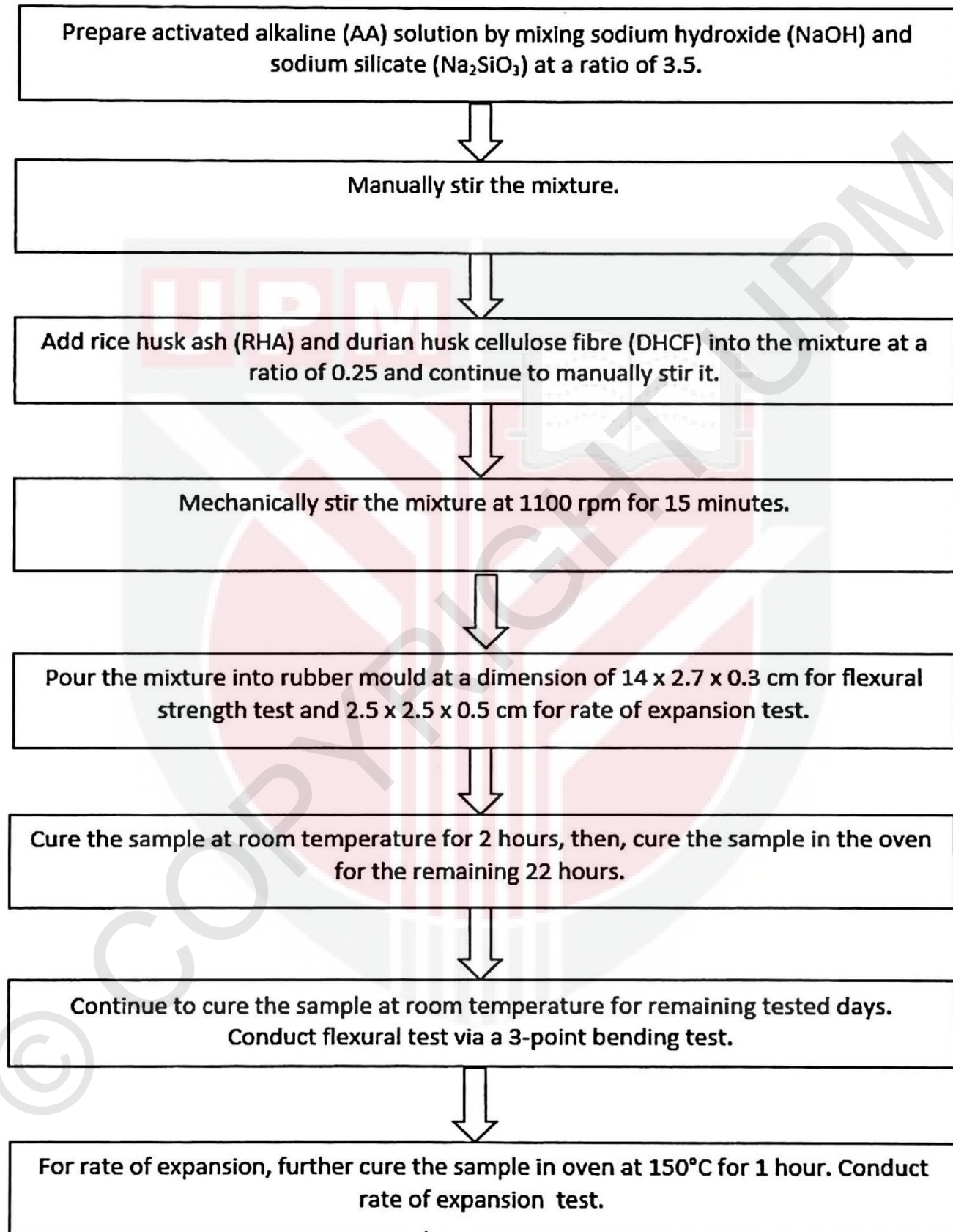


Figure 3. 10: Flowchart of DHCF/RHA-based geopolymer composite preparation.

3.5 Experiment Procedure

3.5.1 Flexural Strength Test

The flexural test, also known as the three-point bending test, was performed on geopolymer composite samples in accordance with ASTM standard D7264 (International, 2007). The sample dimensions for a flexural test setup were mm in length, mm in breadth, and mm in depth. The specimens were loaded vertically in the center on two basic supports till failure. The samples were loaded at a rate of mm/min at normal temperature and relative humidity. The sample was then put on two supports with an mm span length. These data were generated by calculating the flexural stress and strain of the sample using Instron software. Based on the equation, the results of the stress-strain curves are utilized to calculate values such as the flexural strength test.

$$FS = \frac{3PL}{2BH^2} \quad (1)$$

where FS is flexural strength or modulus of rupture (MPa), P is the breaking load (N), L is the span of the simple supports (mm), B is the width of the specimen (mm), and H is the thickness of the specimen (mm).

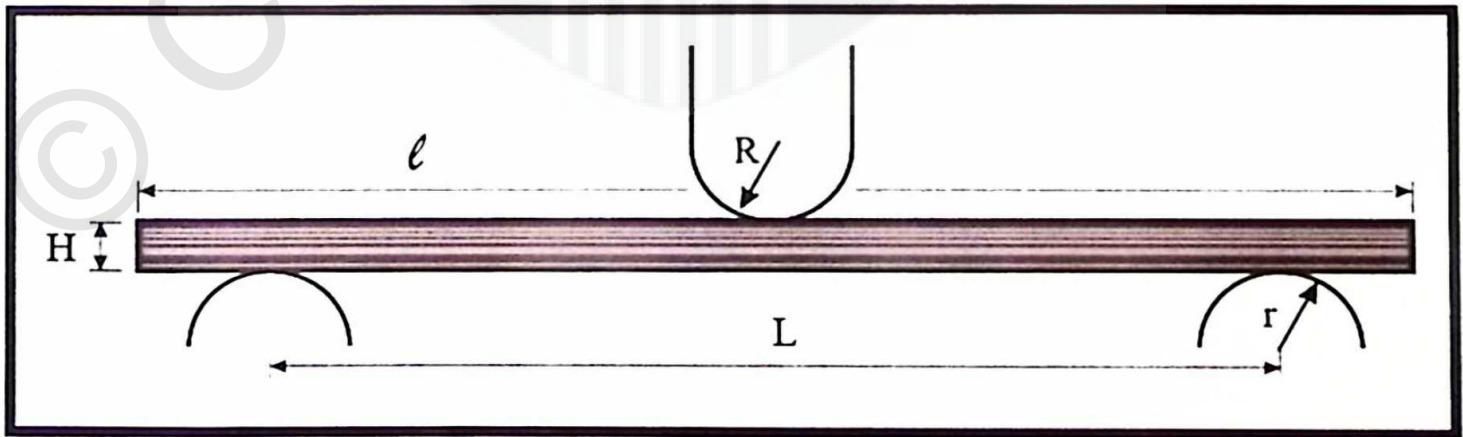


Figure 3. 11: Sketch of the three-point bending test.

3.5.2 Volume Expansion Ratio

The average cell diameter, cell density, and volume expansion ratio were determined based on the method by Xu et al. (2007). Image J was utilized to analyze scanning electron microscope (SEM) images to determine the average cell size. The average number of cell diameters on the micrograph, D , was estimated using the formula:

$$D = \frac{\sum d_i n_i}{\sum n_i} \quad (2)$$

where n_i was the number of cells with a perimeter-equivalent diameter of d_i .

The cell density N_0 , defined as the number of cells per unit volume of the original (unfoamed) polymer, was calculated from SEM micrographs. The technique described was as follows (Kumar & Suh, 1990). The cell density N_f , defined as the number of cells per unit volume of the foam, was computed by dividing the number of cells by the volume of the foam through the formula:

$$N_f = \left(\frac{nM^2}{A} \right)^{3/2} \quad (3)$$

where n is the number of cells in micrograph, A was the area of the micrograph (cm^2), and M was the magnification factor.

The volume expansion ratio of the foamed polypropylene (PP), R_v , was described as the ratio of the virgin PP bulk density (ρ_p) to that of the foamed PP (ρ_f):

$$R_v = \frac{\rho_p}{\rho_f} \quad (4)$$

3.6 Microstructure Analysis

3.6.1 Scanning Electron Microscope (SEM)

Employing SEM, the differences in sample microstructure were analyzed. As depicted in Figure 3.12, the Hitachi S-3400N variable SEM was utilized. Seven samples were analyzed, including three samples before the volume expansion ratio test, three samples after the test, and the cellulose sample. After mounting the samples using a conductive adhesive, gold-palladium powder was sputtered-coated onto them. For viewing, the stub containing the sample was placed in the SEM's sample chamber, and 25, 50, 100, 500, and 1000x micrographs of the sample surface were produced.



Figure 3. 12: Hitachi S-3400N Variable SEM

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Response Surface Methodology (RSM)

The objective of this study is; (i) to design experimental works using response surface methodology and conduct experiments on flexural strength and volume expansion ratio of durian husk cellulose fiber/rice husk ash-based geopolymer composite, (ii) to determine the effect of curing temperature, cellulose fiber content and curing time on the properties of the durian husk cellulose fiber/rice husk ash-based geopolymer composite and perform optimization and validation, and (iii) to investigate and characterize the microstructure of durian husk cellulose fiber/rice husk ash-based geopolymer composite. The complete design matrix and response values are given in Appendix B1, and data were analyzed using MINITAB software.

4.1.1 Statistical Analysis of Flexural Strength and Volume Expansion Ratio Properties

According to the experimental design and analysis, a linear regression model was fitted to the experimental data using the least square technique. Several main parameters were considered in evaluating the statistical results, namely the regression coefficients, the standardized error of coefficient, and the P of the effects of factors and their interaction for both responses, flexural strength, and volume expansion ratio. Complete statistical results for this study are shown in Appendix C1 and C2.

Table 4.1 shows the estimated effects for flexural strength, which indicates that curing temperature (V_1) and curing time (V_3) were highly significant with $P = 0.000$, while the cellulose content (V_2) was not significant. The square and interaction effects of $V_1^*V_1$, $V_2^*V_2$, $V_3^*V_3$, and $V_1^*V_3$ were significant and had an effect on the responses. The interaction effects of $V_1^*V_2$ and $V_2^*V_3$ was not significant and had small effect on the responses. High R^2 values (0.9852) and R^2 (adjusted) (0.9585) were obtained, which indicates that 98.52 percent of the sample variation in the response was attributed to the factors.

R^2 assumes that each explanatory variable explains the variation in the dependent variable and indicates how much the influence of the explanatory variables/cause the response variable (Glen, 2013). R^2 (adjusted) is a modified version of R^2 that has been adjusted for the number of predictors in the model. It is the percentage of variation explained by only the independent variables that affect the dependent variable. R^2 , on the other hand, can only rise. With the addition of less significant variables, adjusted R^2 can decrease, resulting in a more reliable and accurate evaluation.

Table 4. 1: Estimated effects and coefficient for flexural strength.

Term	Notation	Coefficient	Std. Error of Coefficient	P-value
Constant		2.5000	0.0984	0.000
Curing Temperature (°C)	V ₁	0.5138	0.0603	0.000
Cellulose Content (wt.%)	V ₂	0.0088	0.0603	0.890
Curing Time (days)	V ₃	-0.7700	0.0603	0.000
Curing Temperature (°C) *	V ₁ * V ₁	-0.6225	0.0887	0.001
Curing Temperature (°C)				
Cellulose Content (wt.%) *	V ₂ * V ₂	-0.3125	0.0887	0.017
Cellulose Content (wt.%)				
Curing Time (days) *	V ₃ * V ₃	0.3850	0.0887	0.007
Curing Time (days)				
Curing Temperature (°C) *	V ₁ * V ₂	0.0550	0.0853	0.547
Cellulose Content (wt.%)				
Curing Temperature (°C) *	V ₁ * V ₃	-0.2575	0.0853	0.029
Curing Time (days)				
Cellulose Content (wt.%) *	V ₂ * V ₃	0.1375	0.0853	0.168
Curing Time (days)				
R ² = 0.9852		R ² (adj) = 0.9585		

Some of the factors and their interactions were significant for the volume expansion ratio. The results in Table 4.2 showed that V₃, V₂*V₂, V₃*V₃, V₁*V₂ V₁*V₃, and V₂*V₃ were not significant. Factors and their interactions were significant for V₁, V₂, and V₁*V₁. Value for R² (0.9332) and R² (adjusted) (0.8130) were considered high, which indicated that 93.32 percent of the sample variation in the response was attributed to the independent variables.

Table 4. 2: Estimated effects and coefficient for volume expansion ratio.

Term	Notation	Coefficient	Std. Error of Coefficient	P-value
Constant		3.567	0.310	0.000
Curing Temperature (°C)	V ₁	0.513	0.190	0.043
Cellulose Content (wt.%)	V ₂	-0.738	0.190	0.012
Curing Time (days)	V ₃	0.450	0.190	0.064
Curing Temperature (°C) *	V ₁ * V ₁	-1.671	0.279	0.002
Curing Temperature (°C)				
Cellulose Content (wt.%) *	V ₂ * V ₂	0.229	0.279	0.449
Cellulose Content (wt.%)				
Curing Time (days) *	V ₃ * V ₃	-0.246	0.279	0.419
Curing Time (days)				
Curing Temperature (°C) *	V ₁ * V ₂	-0.175	0.268	0.543
Cellulose Content (wt.%)				
Curing Temperature (°C) *	V ₁ * V ₃	0.450	0.268	0.154
Curing Time (days)				
Cellulose Content (wt.%) *	V ₂ * V ₃	-0.250	0.268	0.394
Curing Time (days)				
R ² = 0.9332		R ² (adj) = 0.8130		

Equations 7 and 8 were the mathematical regression models for the flexural strength and volume expansion ratio.

$$y_{FS} = -22.17 + 0.857(V_1) - 0.027(V_2) - 0.644(V_3) - 0.006225(V_1)(V_1) - 0.1389(V_2)(V_2) + 0.0963(V_3)(V_3) + 0.00367(V_1)(V_2) - 0.01287(V_1)(V_3) + 0.0458(V_2)(V_3) \quad (5)$$

$$y_{VR} = -56.3 + 1.961(V_1) + 0.32(V_2) - 0.39(V_3) - 0.01671(V_1)(V_1) + 0.102(V_2)(V_2) - 0.0615(V_3)(V_3) - 0.0117(V_1)(V_2) + 0.0225(V_1)(V_3) - 0.0833(V_2)(V_3) \quad (6)$$

Y_{FS} and Y_{VR} represent the responses of flexural strength and volume expansion ratio, respectively. The V₁, V₂, and V₃ are the decoded values of the curing

temperature, cellulose content, and curing time, respectively. The regression models can be used to calculate and analyze the effect of factors on the flexural strength and volume expansion ratio performance of durian husk cellulose fiber/rice husk ash-based geopolymer composite.

4.1.2 Effect of Factors on Flexural Strength and Volume Expansion Ratio Properties

ANOVA and the regression models were used to analyze the effect of factors on the flexural strength and volume expansion ratio properties. Contour plots were used for better illustration. Figures 4.1, 4.2, 4.3, and 4.4 illustrate the effect of curing temperature (V_1), cellulose content (V_2), and curing time (V_3) on the responses. Figures 4.1 and 4.2 show that higher V_1 increased flexural strength up to 2.70 MPa then decreased to 1.70 MPa, higher V_2 increased flexural strength up to 2.45 MPa then decreased to 2.20 MPa, and higher V_3 decreased flexural strength down to 1.20 MPa. On the other hand, Figures 4.3 and 4.4 indicate the effect of the factors on the volume expansion ratio property. Higher V_1 increased volume expansion ratio up to 4.2 then decreased down to 3.0, higher V_2 decreased volume expansion ratio down to 1.2, and higher V_3 either resulted in constant volume expansion ratio up to 2.7 or increased volume expansion ratio up to 3.7.

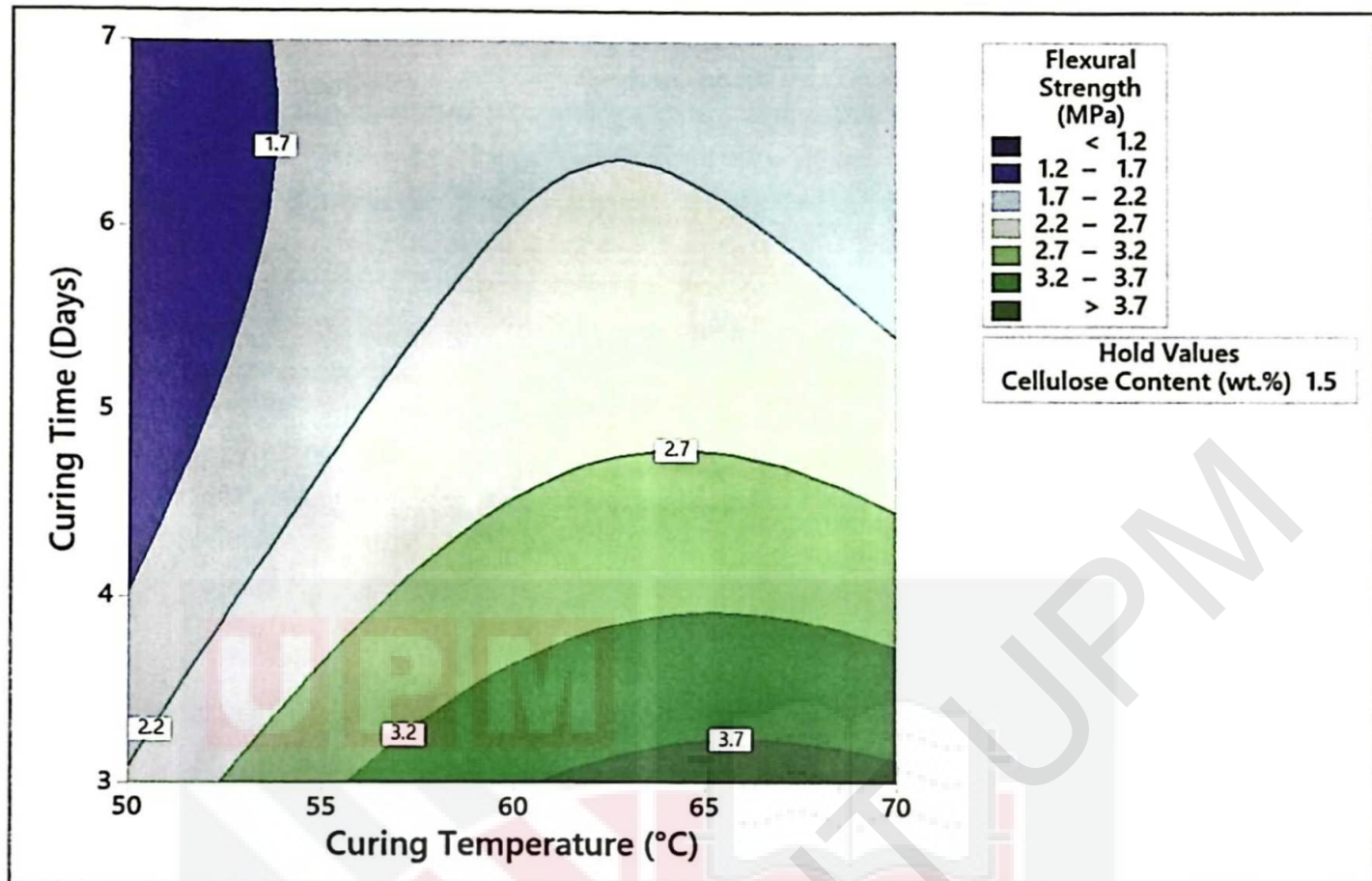


Figure 4. 1: Contour plot for the effect of curing time and curing temperature on the flexural strength.

From Figure 4.1, the flexural strength of the geopolymer composite significantly increases with increasing curing temperature (V_1). When the curing temperature increases from 50 to 70 °C at the curing time of three days, the flexural strength increases from 2.20 to 3.70 MPa. It shows that the curing temperature is one of the important factors as it is responsible for the dissolution of Si and Al species in precursors. It also promotes the polycondensation process and strengthens the geopolymer mix (Alonso & Palomo, 2001; He et al., 2013; Pangdaeng et al., 2015). Curing at a suitable temperature such as 60 °C can accelerate the geopolymerisation process and increase the pore-filling action of the gel, and contribute to an improved mechanical strength (Li et al., 2022).

However, the flexural strength is observed to increase, then decrease with increasing curing temperature (V_1) at a curing time (V_3) below six days. When the curing temperature increases from 50 to 63 °C at a curing time of six days, the flexural strength increases from 1.20 to 2.70 MPa. Beyond 63 °C of curing temperature, the flexural strength decreases from 2.70 to 1.70 MPa. While heat thermally accelerates the geopolymerisation processes, too much high temperature over an extended period can decrease strength and durability (He et al., 2013). It may be due to the rapid formation of the geopolymer matrix on the particle surface, which slows or prevents further leaching of aluminosilicates (Zuhua et al., 2009). Li et al. (2022) determined that long-term curing under high temperatures such as 90 °C and seven days would coarsen the pores and cause micro-defects in the gel phase of the geopolymer due to chemical shrinkage, thus, reducing the mechanical performance.

Based on Figure 4.1, the flexural strength decreases with increasing curing time (V_3). Increasing V_3 from three to seven days at a curing temperature of 50 °C decreases the flexural strength from 2.70 to 1.20 MPa. Similar results at a curing temperature of 70 °C decreases the flexural strength from 3.70 to 1.70 MPa. A shorter curing time (three days) led to insufficient geopolymerisation. In contrast, a longer curing time (14 days) resulted in moisture loss and microcracks creation (Sajan et al., 2021).

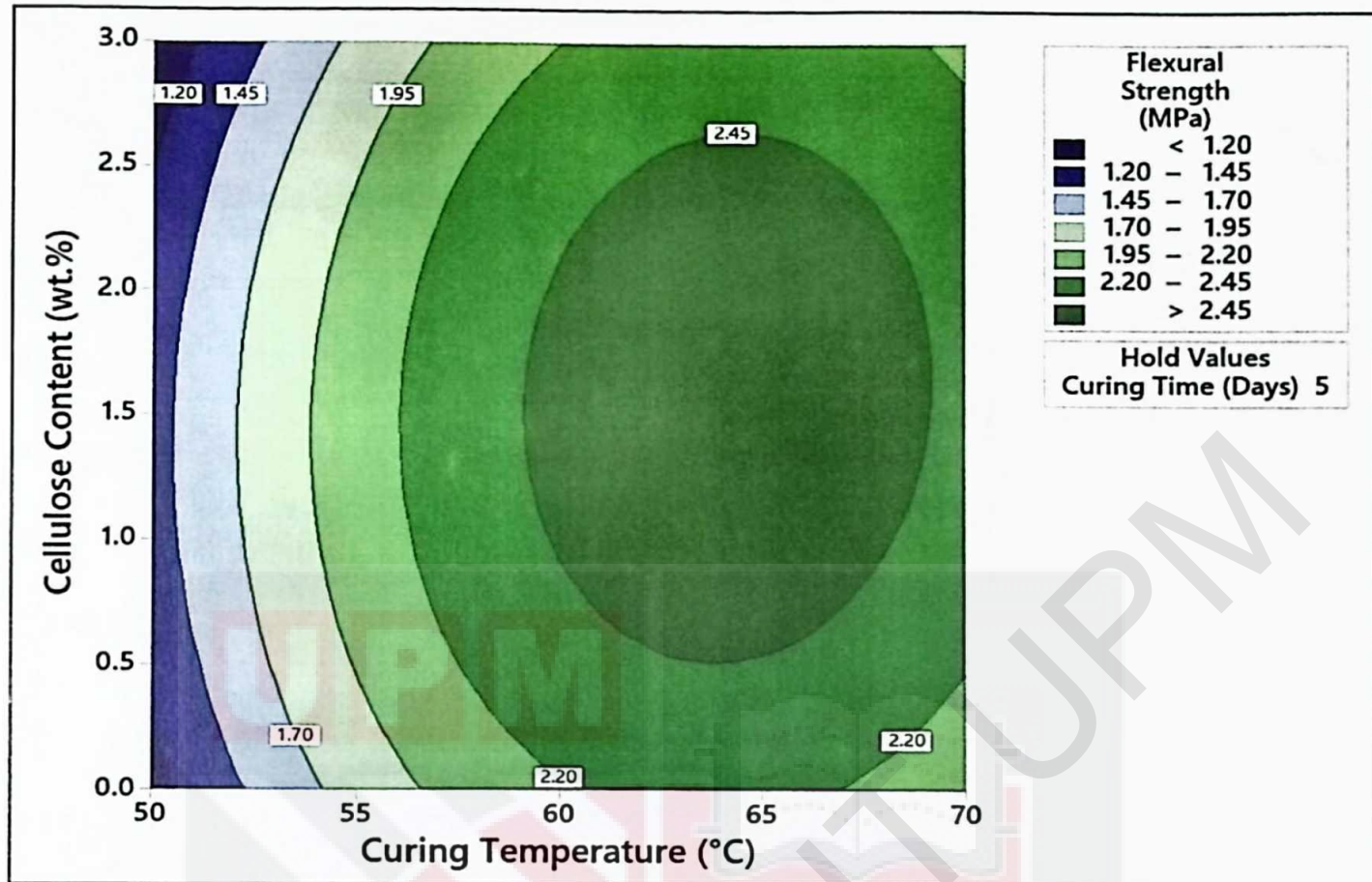


Figure 4. 2: Contour plot for the effect of cellulose content and curing temperature on the flexural strength.

Figures 4.1 and 4.2 show that the increase in curing temperature and cellulose content can significantly improve the flexural strength of the geopolymer composite up to a certain value before it lowers.

From Figure 4.2, the increase in curing temperature shows a similar trend in flexural strength to Figure 4.1. The flexural strength of the geopolymer composite is observed to increase, then decrease with increasing curing temperature (V_1). The increase of V_1 in geopolymer composite from 50 to 64 °C at cellulose content of 0.5 wt.% increases the flexural strength from 1.20 to 2.45 MPa. However, it decreases from 2.45 to 2.20 MPa at a temperature of 64 to 70 °C. Extremely high temperatures result in the development of excessive gels, which inhibit the dissolution of Si and Al, leaving them unreacted (Ahmari & Zhang, 2012). In addition, an excessively high

curing temperature induces instantaneous quick evaporation of pore solutions, which results in an inadequate geopolymerisation. High temperature curing above a particular threshold has also been linked to the development of cracks. Abrupt water loss reduces open porosity, which has a detrimental effect on its durability. (Mohsen & Mostafa, 2010; Zhang et al., 2015). Moreover, excessive loss of moisture and pore fluids from the geopolymer matrix has been attributed to excessive shrinkage during drying, consequent loss of structural integrity, and the development of microcracks (Bakharev, 2006; Perera et al., 2007).

According to Figure 4.2, the flexural strength of the geopolymer composite is observed to increase then decrease with increasing cellulose content (V_2). The increase of V_2 in geopolymer composite from 0 to 2 wt.% at a curing temperature of 60 °C increases the flexural strength from 2.20 to 2.45 MPa, then decreases from 2.45 to 2.20 MPa again at cellulose content of 2 to 3 wt.%. This finding agrees with a study conducted by Taveri et al. (2018), a decrease in flexural strength in geopolymeric composite samples with a cellulose content of more than 2 wt.% is caused by excessive Na^+ absorption by the cellulose fibers. Mercerization is a well-known process involving the absorption of alkali cations (particularly Na^+) by cellulose fibers. The interference of alkali cations between polymeric chains causes cellulose to swell, forming electrostatic layers of positively charged Na^+ ions and negatively charged polymeric chains (Wang, 2008). In cellulose-based composites, the absorption of additional chemical components generates a transitional contact between the inner portion of the fiber and the matrix.

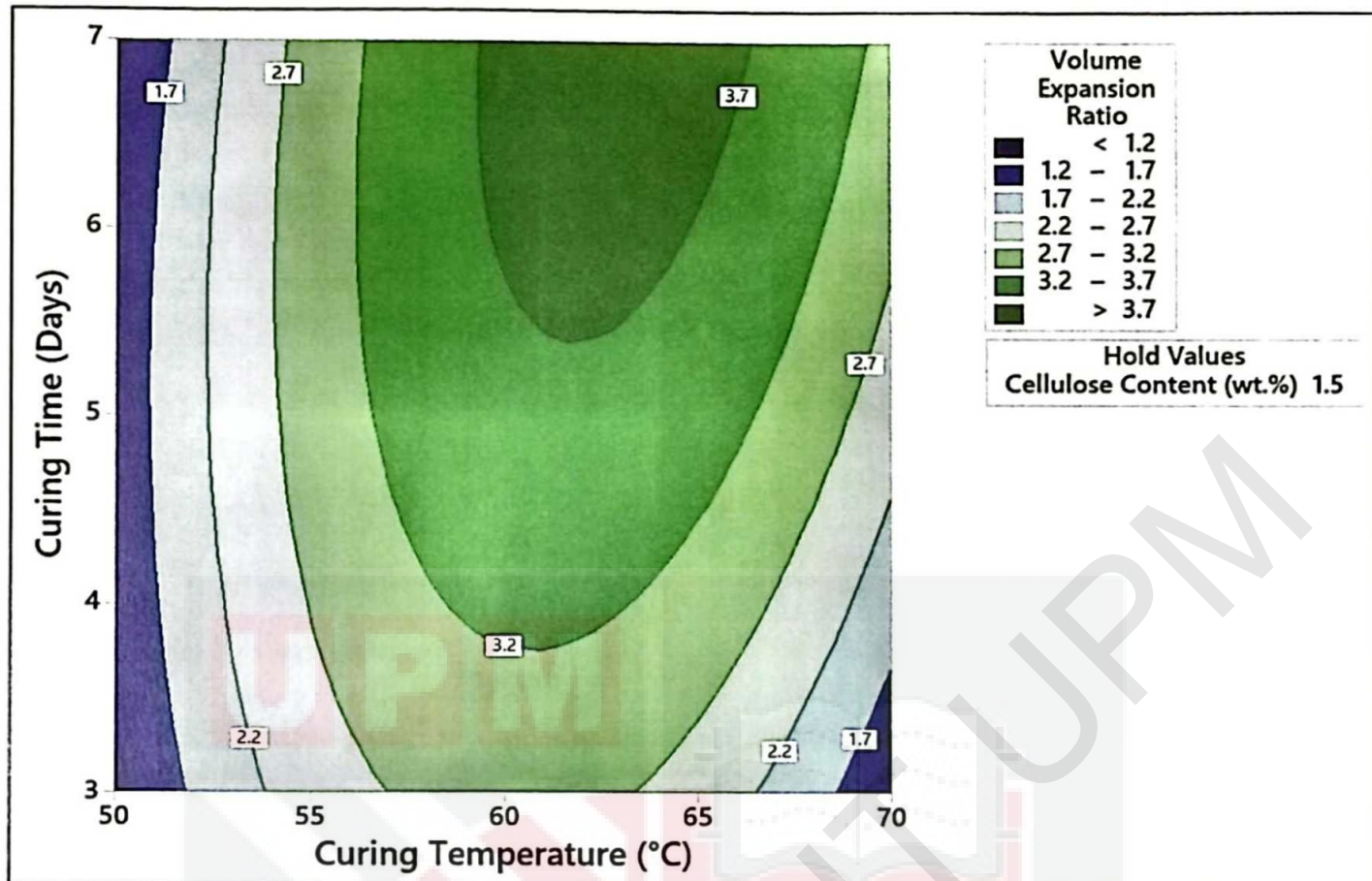


Figure 4. 3: Contour plot for the effect of curing time and curing temperature on the volume expansion ratio.

From Figure 4.3, the volume expansion ratio of the geopolymer composite significantly increases and then decreases with increasing curing temperature (V_1). The increase of V_1 in geopolymer composite from 50 to 60 °C at a curing time of seven days increases the volume expansion ratio from 1.20 to 3.70. However, further increase in curing temperature from 60 to 70 °C decreased the volume expansion ratio from 3.70 to 2.70. Generally, as the curing temperature rises, the pore volume and surface area increase (Sindhunata et al., 2006). Increasing the curing temperature enhances the extent and rate of the reaction, as evidenced by a rise in mesopore volume, surface area, and setting time. It is known that increasing the reaction temperature increases the extent of precursor dissolution (primarily Al and Si). However, when the temperature of geopolymer 0.79Na was elevated from 50 to 75 °C for 24 hours, the

Si/Al ratio reduced from 1.78 to 1.60, which appears to be atypical behavior. The lower mesopore volume of 0.79Na at 75 °C compared to 50 °C corroborates the lower Si/Al ratio, indicating that the reactivity is lower at 75 °C. The result aligns with the findings as the volume expansion ratio reduces as the temperatures increases beyond 60 °C.

Based on Figure 4.3, the volume expansion ratio of the geopolymer composite significantly increases with increasing curing time (V_3). The increase of V_3 in geopolymer composite from three to seven days at a curing temperature of 60 °C increases the volume expansion ratio from 2.20 to 3.70. The volume expansion during heat treatment is typically proportional to the curing temperature and time (Shakouri et al., 2020). From the study, samples fixed at 100 °C for six hours expanded by 600 percent while samples cured for 24 hours expanded by 1300 percent. The achieved outcome conforms to the researcher's results. The expansion of the paste during heat treatment is dependent on both the chemical desire to expand due to stress generated by the expansion mechanism and the paste's resistance to expansion, which is proportional to its strength. The gain in strength of the paste samples is highly correlated with curing temperature and time. Low strength, as a result of insufficient geopolymerisation, reduces the expansive stress, whereas a well-cured and strong sample demonstrates more resistance to expansion. Pastes cured at low temperatures or for an inadequate amount of time remained fluid, whereas those cured at higher temperatures and for longer gained significant strength.

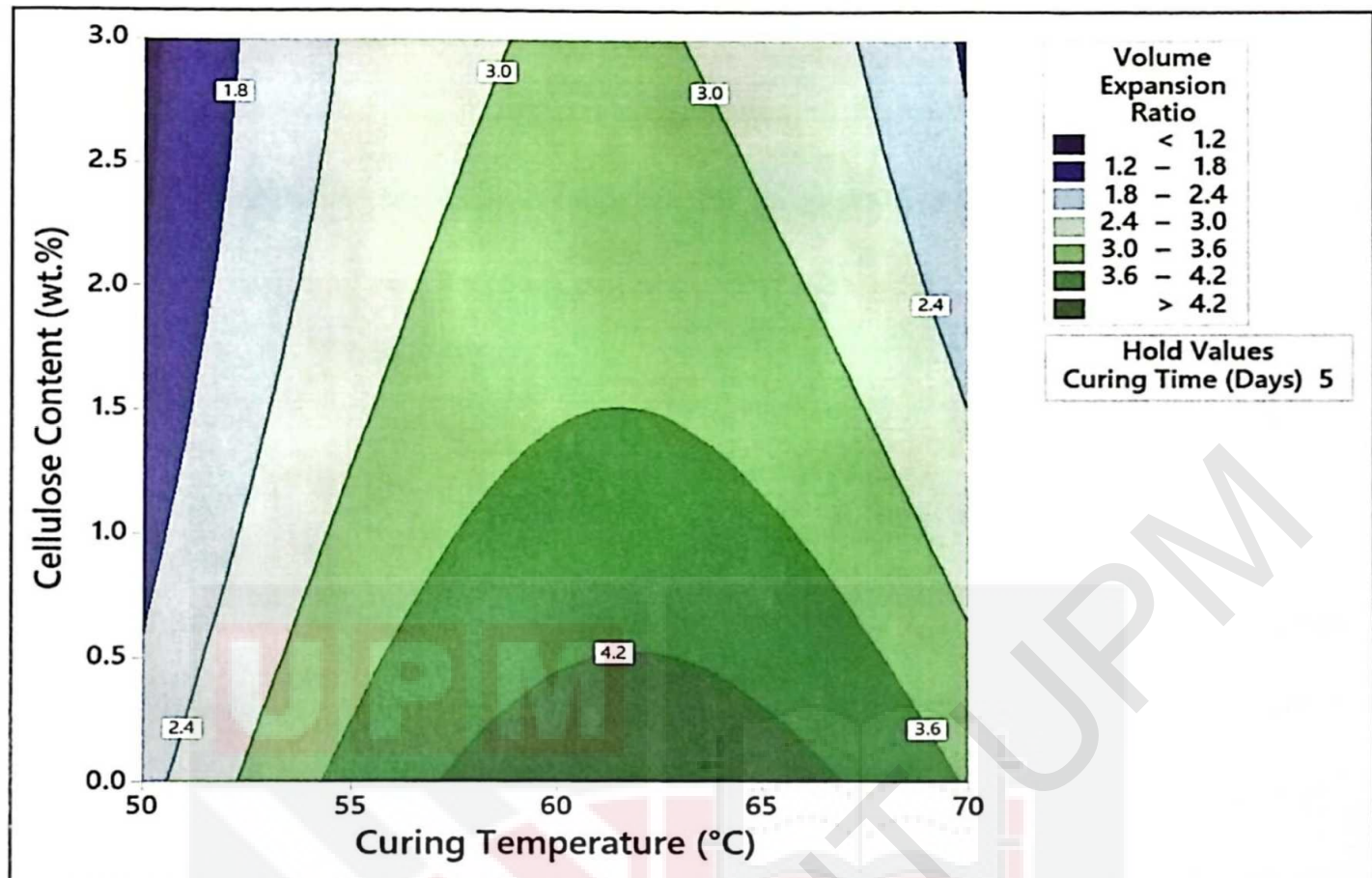


Figure 4. 4: Contour plot for the effect of cellulose content and curing temperature on the volume expansion ratio.

Figure 4.4 shows a similar pattern in the volume expansion ratio to that in Figure 4.3 as the curing temperature increases. The volume expansion ratio of the geopolymer composite is observed to increase, then decrease with increasing curing temperature (V_1). The increase of V_1 in geopolymer composite from 50 to 60 °C at cellulose content of 0.5 wt.% increases the volume expansion ratio from 1.80 to 4.20, then decreases from 4.20 to 3.00 beyond 60 °C. An increase in curing temperature enhances the extent and rate of the reaction. However, a further increase in the temperature would inhibit the extent of precursor dissolution.

According to Figure 4.4, the volume expansion ratio of the geopolymer composite significantly decreases with increasing cellulose content (V_2). The increase of V_2 in geopolymer composite from 0 to 3 wt.% at a curing temperature of 60 °C

decreases the volume expansion ratio from 4.20 to 3.00. The addition of cellulose fibers to geopolymer-based composites resulted in a denser structure, fewer pores, nearly constant density, and a discernible ductile failure due to the bridging mechanism of the fibers restricting crack propagation (Ye et al., 2018).

4.1.3 Optimization Formulation of The Response

Figure 4.5 shows the optimization plot and the effect of the different combinations of factor settings on the response. The optimization was performed under designated parameters (curing temperature: 60 °C; cellulose content: 0.5 wt.%; curing time: 3 days). Since the objective was to maximize the flexural strength, the lowest values, the minimum acceptable value, were set at 1.10 MPa (the lowest value). The target values, the goal to be achieved (the highest flexural strength), were set at 3.87 MPa (the highest value of result from the experiment). Based on Appendix B1, the minimum value was set as the lowest value of flexural strength, and the target value was close to the highest value of flexural strength.

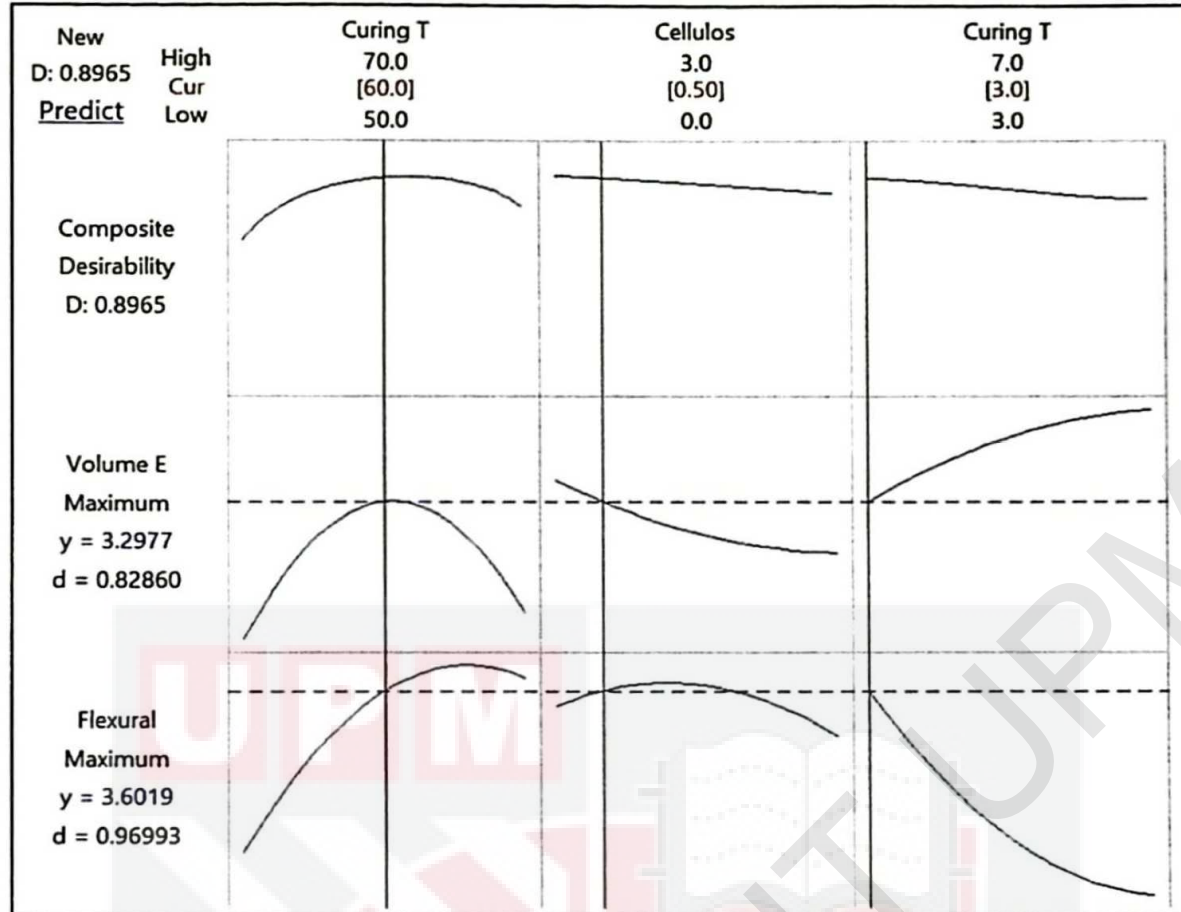


Figure 4. 5: Optimization plot for maximum flexural strength and volume expansion ratio.

For volume ratio expansion, lower values, which are the minimum acceptable, were set at 1.00 (the lowest value) as the objective was to maximize the volume expansion ratio of the DHCF/RHA-based geopolymer composite. The target value for volume expansion ratio properties was set at 5.30 (the highest value). The Minitab software generates the predicted optimum flexural strength and volume expansion ratio values of 3.2977 MPa and 3.6019, respectively, which can be achieved with the combination of curing temperature (V_1) = 60 °C, cellulose content (V_2) = 0.5 wt.% and curing time (V_3) = 3 days. The desirability of optimization was calculated as 0.8965, indicating that all parameters were within the target to obtain the maximum flexural strength and volume expansion ratio properties.

4.1.4 Optimization Validation

From Table 4.3, it was found that the average error for the flexural strength and volume expansion ratio was well below 15 percent at 11.11 percent and 10.61 percent, respectively. It was concluded that the developed regression model established using this method could accurately optimize the responses.

Table 4. 3: Experimental validation for the flexural strength and volume expansion ratio properties.

Sample no.	Flexural Strength (MPa)			Volume Expansion Ratio		
	Experimental value	Predicted value	Error (%)	Experimental value	Predicted value	Error (%)
SV1	4.0	3.6	11.11	3.7	3.3	12.12
SV2	4.0	3.6	11.11	3.6	3.3	9.09
	$\bar{x}$ Error		11.11	$\bar{x}$ Error		10.61

Compared with the control sample without cellulose content (S5), the control sample recorded lower flexural strength and higher volume expansion ratio at 3.20 MPa and 4.10, respectively. Figure 4.6 shows the control and optimized samples for the flexural strength and volume expansion ratio test.

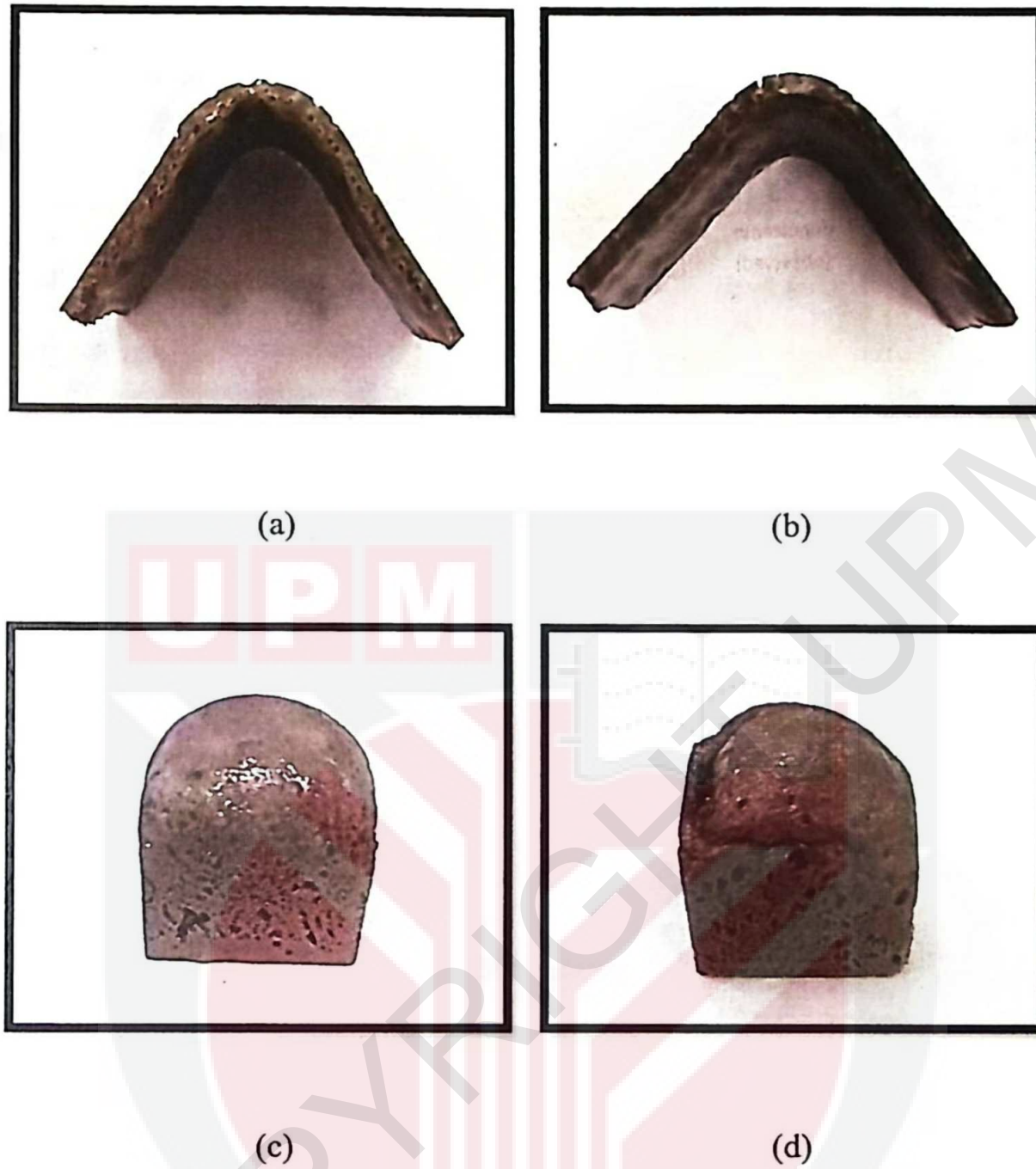


Figure 4. 6: (a) control sample and (b) optimized sample for flexural strength, and (c) control sample and (d) optimized sample for volume expansion ratio.

Comparing geopolymer composites with different aluminosilicate sources and geopolymer reinforcement, durian husk cellulose fiber/rice husk ash-based geopolymer composite showed better performance with higher flexural strength value of 4.00 MPa . Meanwhile, fly ash-based geopolymer reinforced with different types of macro steel and polypropylene fibers in a study by Bhutta et al. (2017) reported a maximum peak strength of only 2.89 MPa with end-deformed steel fibers.

4.1.5 Material Behaviour of Sample After Flexural Strength Test

Figure 4.7 shows slopes of samples S5, S14, S3, and optimized samples with brittle and ductile behavior. The samples were chosen based on best flexural strength (S5), which is also a sample without the addition of cellulose fiber, median flexural strength (S14), poor flexural strength (S3), and optimized sample (Optimized).

A steeper slope indicates that the material was more resilient and not easily deformed than a gentler slope. Figure 4.7 showed that the optimized sample (curing temperature at 60 °C, cellulose content of 0.5 wt.%, curing time at 3 days) had the steepest slope indicating it was the most resilient and brittle among the ductile samples tested in resisting deformation. In contrast, S3 (curing temperature = 50 °C, cellulose content = 1.5 wt.%, curing time = 7 days) was the extreme opposite. This result was probably due to lower curing temperature, higher cellulose content, and curing time. Lower curing temperature, higher cellulose content, and curing time lead to a gentler slope (ductile properties). It is due to the quantity of precursors, primarily aluminium, dissolved from amorphous phases being insufficient. Therefore, it was unable to polymerize with the amorphous silicon precursors present in the activators to form an adequate quantity of aluminosilicate gels. The result is consistent with a study conducted by Mo et al. (2014).

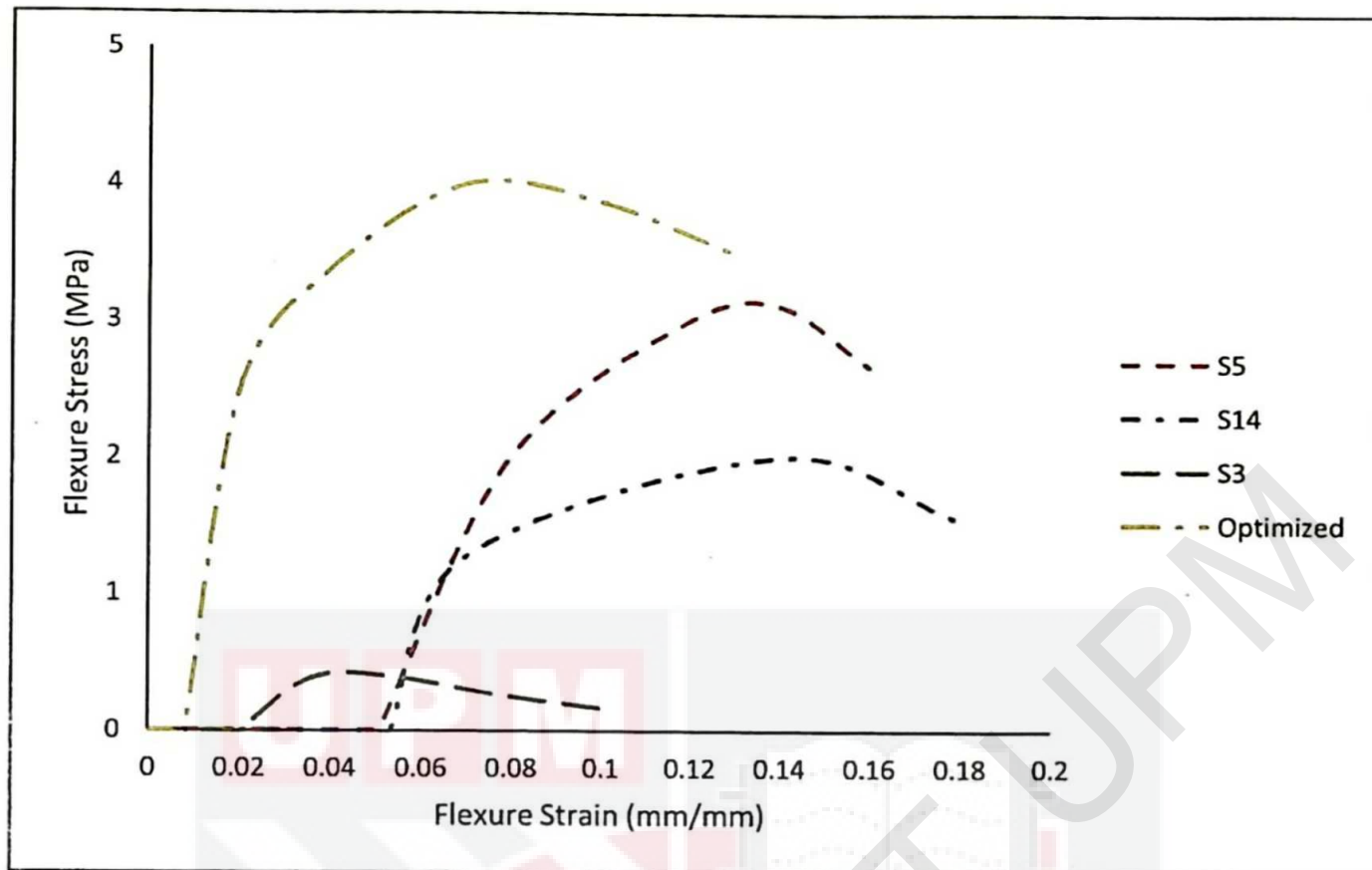


Figure 4. 7: Stress-strain curves of samples S5, S14, S13, and optimized sample.

From Appendix B1, although the sample without cellulose content (S5) recorded lower flexural strength (1.92 MPa) as compared to that of sample S14 (3.87 MPa), sample S14 showed a shallower slope which indicates that this sample was easily deformed due to lesser resilience (Funk & Dinger, 2013). This slope was due to the higher curing temperature (70 °C), cellulose content (1.5 wt.%), and curing time (7 days). It is because the surface of the particle experiences a rapid formation of a geopolymer matrix. This formation retards and prevents further leaching of aluminosilicates and the excessive Na^+ absorption by the cellulose fibers, which leads to ductile behavior.

4.1.6 Material Behaviour of Sample After Volume Expansion Ratio

Figures 4.8 and 4.9 show the relationship between the average cell diameter and cell density with the volume expansion ratio. The samples were chosen based on the volume expansion ratio performance. The sample with the best performance (S5) has the highest volume expansion ratio, and the sample with moderate performance (S14) has an intermediate volume expansion ratio. In contrast, the sample with poor performance (S3) has the lowest volume expansion ratio. Complete statistical results for this study are shown in Appendix D1 and D2.

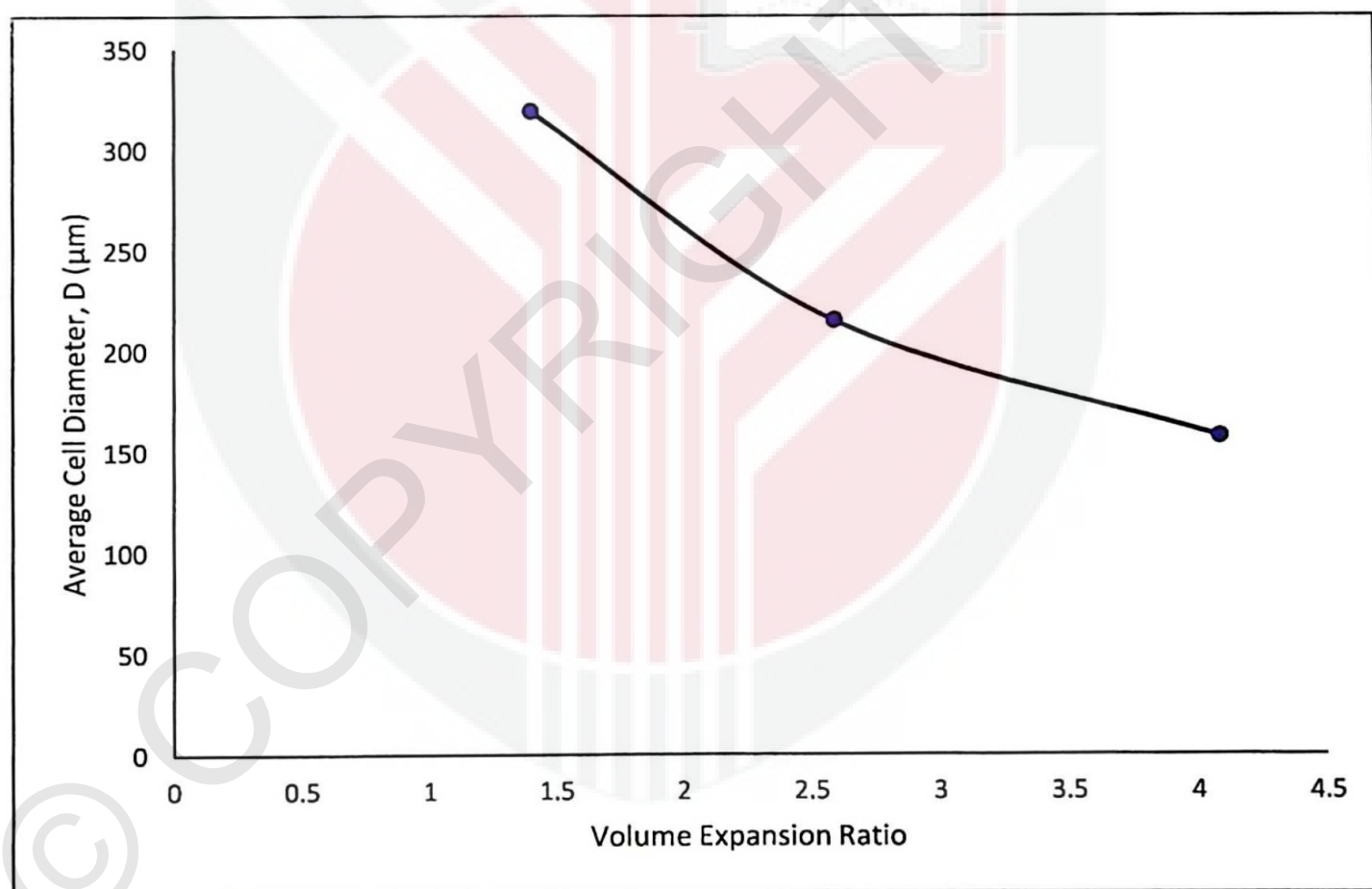


Figure 4. 8: Effect of volume expansion ratio on the average cell diameter.

Figure 4.8 shows that the sample with the highest volume expansion ratio, S5 (curing temperature = 60 °C, cellulose content = 0 wt.%, curing time = 3 days), is associated with the smallest cell diameter at 157.03 μm. The slope shows a reducing

trend. In contrast, sample with the lowest volume expansion ratio, S3 (curing temperature = 50 °C, cellulose content = 1.5 wt.%, curing time = 7 days) possess the largest cell diameter at 319.12 μm . It might be due to higher curing temperature, lower cellulose content, and curing time. Geopolymers synthesized at higher temperature generates finer pores. The result is consistent with a study conducted by Sindhunata et al. (2006).

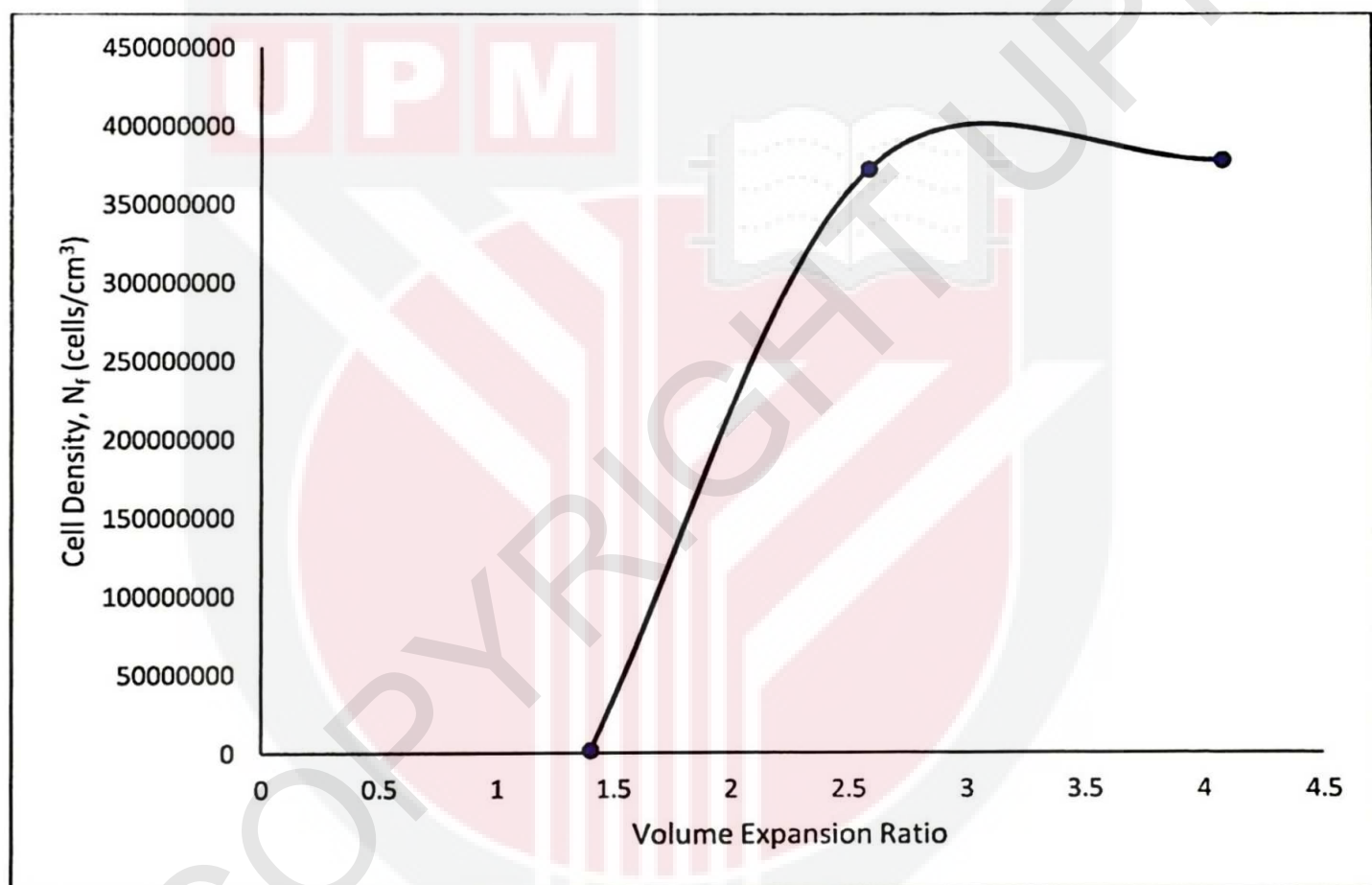


Figure 4. 9: Effect of volume expansion ratio on the cell density.

Figure 4.9 demonstrates a slope with an increasing trend. Sample with the highest volume expansion ratio, S5 (curing temperature = 60 °C, cellulose content = 0 wt.%, curing time = 3 days) is associated with the highest cell density at 3.78×10^8 cells/cm³. On the contrary, sample with the lowest volume expansion ratio, S3 (curing temperature = 50 °C, cellulose content = 1.5 wt.%, curing time = 7 days) possess the

lowest cell density at 1.38×10^6 cells/cm³. For a given cell size or cell size for a given cell density, an increase in cell density must lead to a decrease in foam density and an increase in the volume expansion ratio (Xu et al., 2007).

According to a study by Xu et al. (2007), foam density and foam volume expansion ratio are two macroscopic interrelated variables. They are dependent on both cell size and cell density. Any factor that influences cell density, cell size, or both parameters may influence the foam density and volume expansion ratio. Table 4.4 summarizes the effect of factors on the volume expansion ratio of the foamed geopolymer composite at a designed foaming temperature of 150 °C and a foaming period of 1 hour.

Table 4. 4: Effect of factors on the volume expansion ratio of the foamed geopolymer composite at a given foaming temperature of 150 °C for 1 hour.

	S5	S14	S3
Curing Temperature, V ₁ (°C)	60	70	50
Cellulose Content, V ₂ (wt.%)	0	1.5	1.5
Curing Time, V ₃ (days)	3	7	7
Volume Expansion Ratio	4.1	2.6	1.3
Average Cell Diameter, D (µm)	157.03	214.73	319.12
Cell Density, N _r (cells/cm ³)	3.78×10^8	2.48×10^8	1.38×10^6

4.1.7 Contour Plots Superimposition

A method used to plot the overlaid graphs for various response surfaces is the superimposition of the contour plots. This method is superior to the classical one-factor-at-a-time (OFAT) approach, lacking the interaction of selected variables and

cumbersome experimental runs (Jampala et al., 2017). The superimposed contour plots serve as a reference template that is easy to use in estimating the response for any given factor value within the available range. Figure 4.9 and Figure 4.10 shows a feasible representation of the optimum range for the processing condition and formulation of the geopolymer composite. The contour plot is superimposed from the flexural strength and volume expansion ratio in Figures 4.1, 4.2, 4.3, and 4.4, respectively. The light green and light blue region represent the contour region for flexural strength and volume expansion ratio, respectively. Based on the overlaid contour plots from Figure 4.9, the optimum range for the highest flexural strength and volume expansion ratio (represented by the white region) was found to be 59 to 67 °C of curing temperature and 0.5 to 1.5 wt.% of cellulose content.

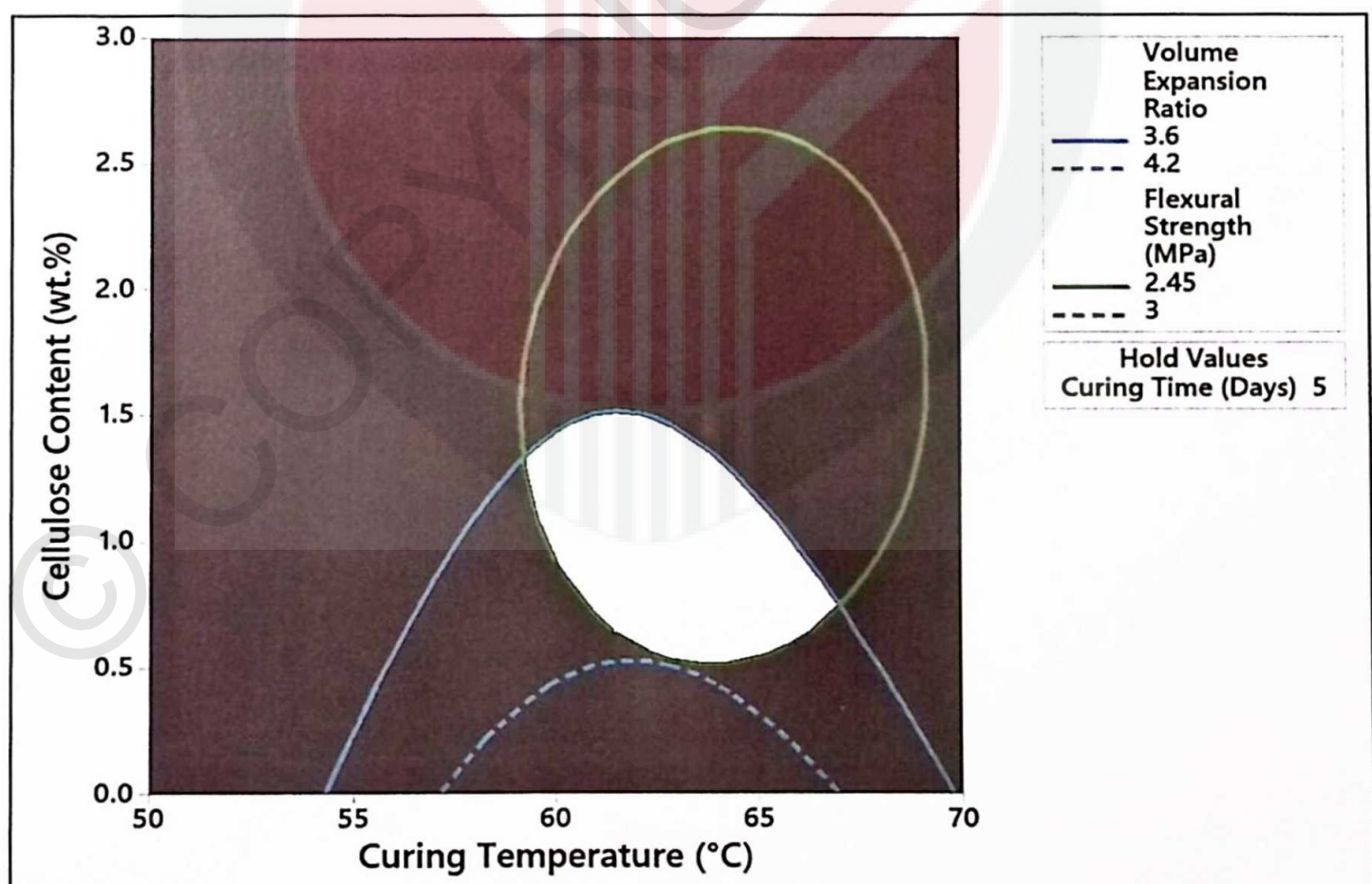


Figure 4. 10: Superimposed contour for responses affected by cellulose content and curing temperature.

From Figure 4.10, the optimum range for the highest flexural strength and volume expansion ratio (represented by the white region) were found to be 57 to 67 °C curing temperature and 4 to 5 days of curing time.

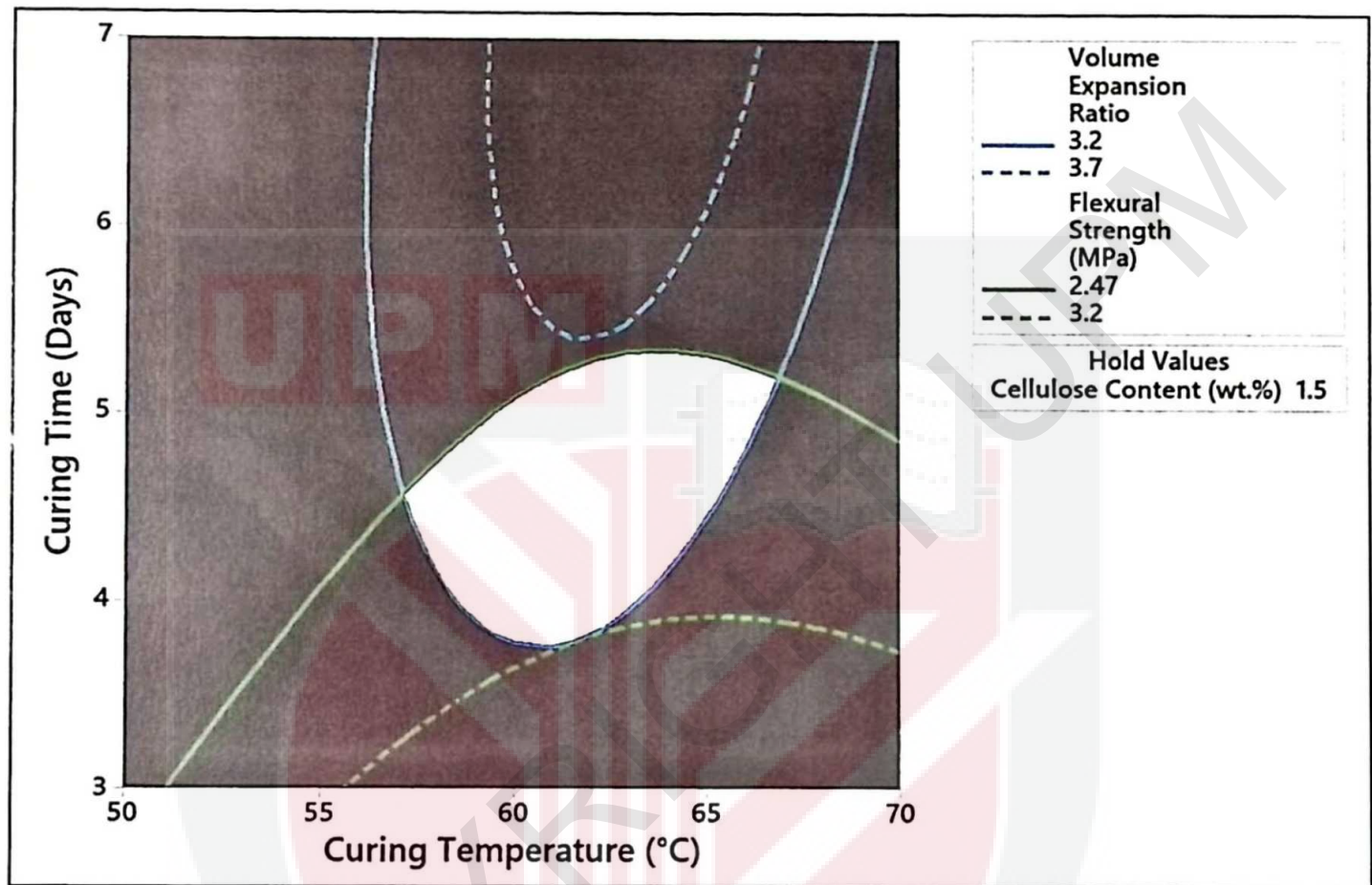


Figure 4. 11: Superimposed contour for responses affected by curing time and curing temperature.

4.2 Microstructure Analysis

Seven samples were selected to determine the microstructural properties of the DHCF/RHA-based geopolymer composite. Three samples were chosen for the flexural strength tests, three after exposure to 150°C, which exhibited low, moderate, and high volume expansion ratio, and one sample of DHCF. Figure 4.12 illustrate the microstructure of the treated DHCF with an average diameter of 11.50 μm . During alkali treatment, cellulose microfibrils were more likely to agglomerate because the cell wall had lost its matrix. According to Chen et al. (2017), changes in fiber length and diameter can be attributed to hemicellulose loss during the alkaline extraction process during the aggregation of cellulose microfibrils. The cellulose fiber was also observed to be distorted to a certain extent. According to Liu and Hu (2008), mercerization at a certain alkali concentration can produce twisting, distortion, and shrinking effects on the fibers.



Figure 4. 12: SEM micrographs of treated durian husk cellulose fiber at 500x (left) and 1000x (right) magnification.

Good, moderate, and poor samples were based on the value of the responses (flexural strength) and were compared within the population from the experimental design for RSM. Comparing with previous literature is not valid since the nature and method of the experiment were different. In addition, there is extremely limited literature on DHCF/RHA-based geopolymer composite. The samples were analyzed using a scanning electron microscope (SEM). Table 4.5 shows the sample number and their performance.

Table 4. 5: Sample number and performance.

Performance	Sample number	Curing temperature (°C)	Cellulose content (wt.%)	Curing time (days)
Good	S5	60	0	3
Moderate	S14	70	1.5	7
Poor	S3	50	1.5	7

Figure 4.13 illustrates the SEM micrographs of the DHCF/RHA-based geopolymer composite prior to the flexural strength test at 100x and 500x magnifications. As shown in Figure 4.13(a) and (b) at 100x magnification, sample S5, which possesses good flexural strength properties, shows smooth, dense, compact, and homogeneous structures with slight unreacted RHA particles. It indicates a good integration of RHA into the geopolymer matrix systems. At higher magnification, sample S5, without cellulose, showed RHA particles covered and encapsulated with the geopolymer matrix. Sample S5 may have undergone a higher rate of geopolymerisation, resulting in a more homogeneous mixture (Guzmán-Aponte et al., 2017).

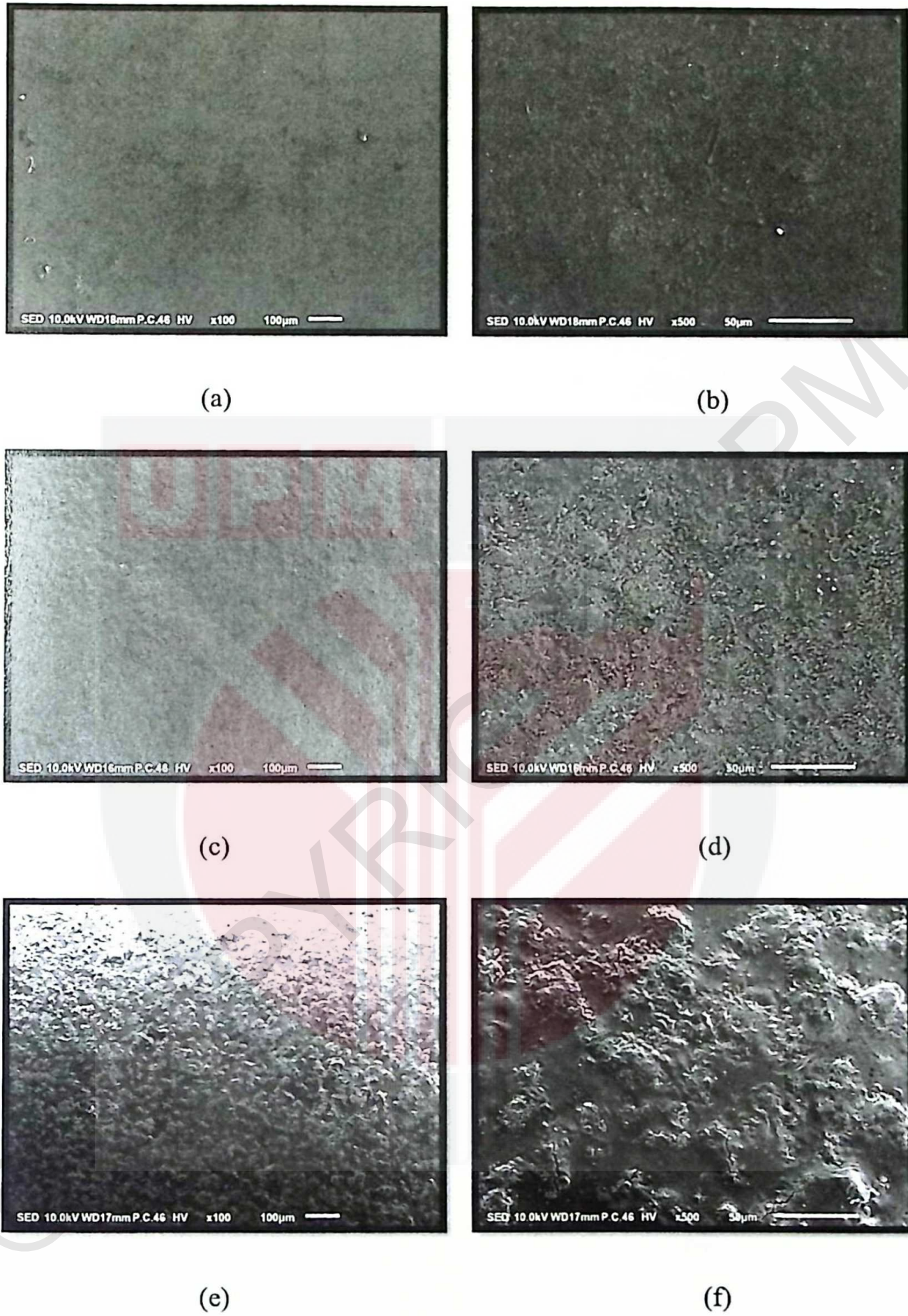


Figure 4. 13: SEM micrographs of DHCF/RHA-based geopolymer composite for the flexural strength test with (a-b) good (sample S5), (c-d) moderate (sample S14), and (e-f) poor flexural strength properties (sample S3) at 100x and 500x magnification.

It was found that the geopolymer matrix and RHA particles bonded more strongly, resulting in a higher flexural strength than samples that were incorporated with cellulose. This finding is consistent with that of Basri et al. (2020). Polycondensation is enhanced by increased RHA, which suggests that stronger chemical interactions between unreacted particles and matrix are formed, leading to a denser 3D geopolymeric gel (Nana et al., 2021). These results were in agreement with Beleuk à Mounkam et al. (2022) and Tome et al. (2022), who concluded that adding amorphous RHA to geopolymer matrices contributed significantly to their density and strength improvement.

In comparison to sample S5, sample S14 in Figures 4.13(c) and (d) showed the surface is covered with excess durian husk cellulose (DHCF), which is not able to be mixed with the geopolymer matrix. However, the cellulose is well distributed and very less agglomeration. Small voids and cracks formed on the sample surface, contributing to lower flexural strength than sample S5. However, the cracks propagated less severely as compared to sample S3. Upon flexural strength test, the cracks were nucleated from voids and propagated along the interface. Multiple wide cracks formed in the composite, thus lowering the overall flexural strength and modulus (Basri et al., 2020).

Figure 4.13(e) and (f) show sample S3, which exhibited poor flexural strength and was cured at a lower temperature (50°C) as compared to that sample S14 (70°C). According to Panesar et al. (2017), cellulose (nano)fibers are known to form agglomeration due to the high concentration of polarized hydroxyl groups on their surfaces. The agglomeration between small particles of DHCF resulted in low dispersion in the geopolymer matrix (Bai et al., 2018). Due to lower curing temperature

than sample S14, the cellulose agglomerated before the geopolymer binder hardened. At elevated temperatures, geopolymerisation processes develop a hard structure earlier in the process (Rovnaník, 2010). The finding was supported by Hassaan et al. (2015). They discovered that particle agglomeration interferes with geopolymer chain three-dimensional network development and negatively affects the mechanical properties of the final products.

It was observed that the agglomerated cellulose particles were formed by the unreacted or partially reacted cellulose particles, and cracks are observed, pointing to a weaker and more porous composite matrix. These cracks could be developed due to drying shrinkage during geopolymer curing (He et al., 2012; ul Haq et al., 2014). The cracks lessen the binding capacity and strength of the geopolymer (Hosseini et al., 2021).

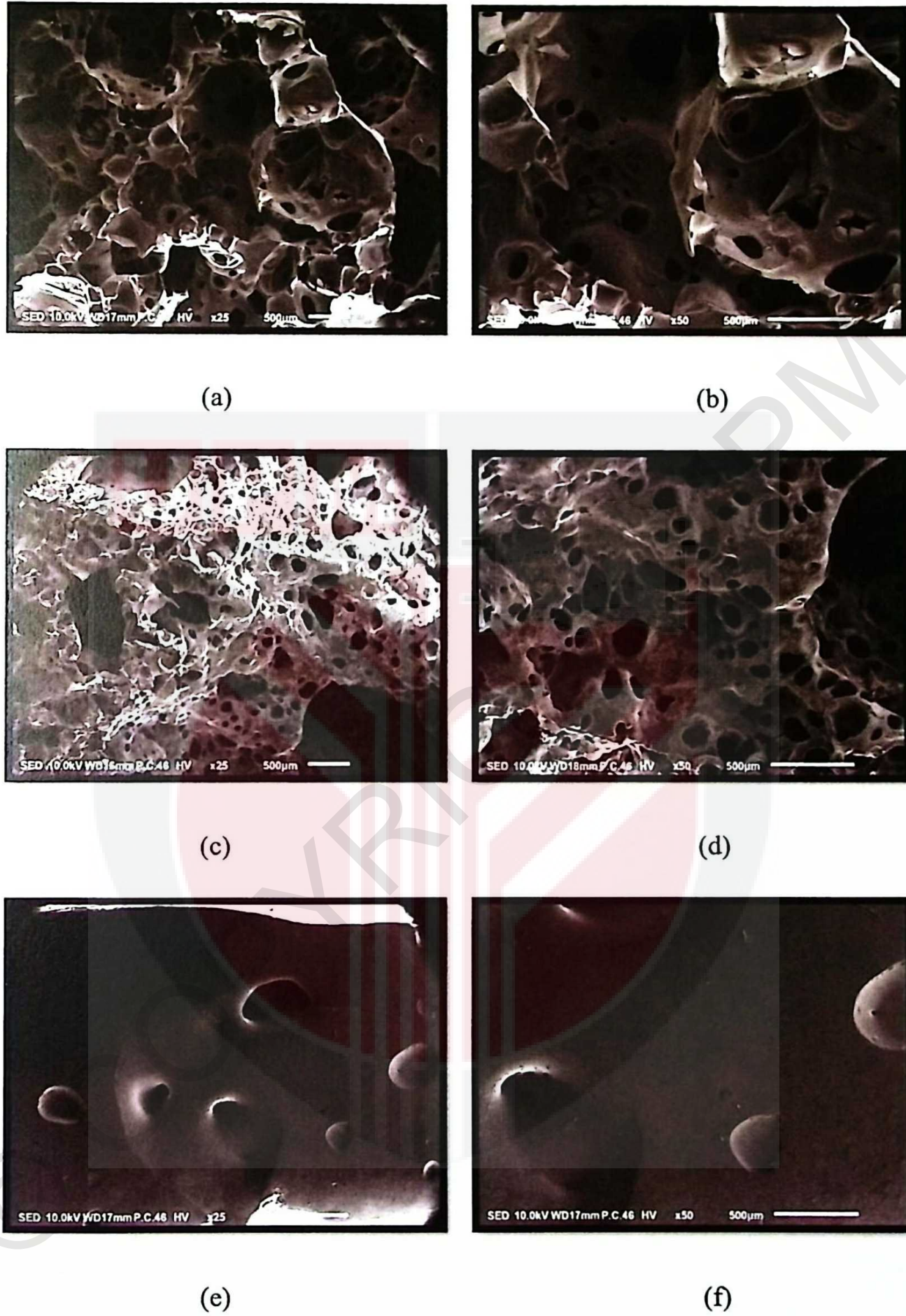


Figure 4. 14: SEM micrographs of DHCF/RHA-based geopolymer composite after heated at 150°C with (a-b) high (sample S5), (c-d) moderate (sample S14), and (e-f) low volume expansion ratio (sample S3) at 25x and 50x magnification.

Sample S5, as shown in Figures 4.14 (a) and (b), exhibited a high volume expansion ratio. As the sample contained only RHA without incorporating cellulose, it has a higher liquid-to-solid (S/L) ratio of 4.0 compared to sample S14 of 3.7. It is due to the generation of carbon dioxide (CO_2) gas produced within the geopolymer matrix. In addition, the latency period matched the plastic period, and the setting time coincided with the period in which gas was generated. As gas is generated, the hydrogen bubbles try to escape from the material. When the geopolymer is still fluid in the plastic period where the setting has not yet taken place, the gas bubbles generated can leave the geopolymer. However, while the geopolymer is setting and the material is hardening, the gas becomes trapped in the structure, expanding the geopolymeric composite matrix and increasing the expansion volume. Similar results were obtained by Xu et al. (2007). The crystalline phase of the geopolymer composite binder could be partially or entirely disrupted as it began to foam.

Consequently, it is expected that CO_2 gas will be diffused across the amorphous and crystalline phases. The geopolymer composite began to expand, and the cells became larger. Furthermore, without the addition of cellulose to the geopolymer matrix, the cell walls appeared to be thinner.

Figure 4.12 (c) and (d) show sample S14 which exhibited a low volume expansion ratio and contained cellulose, and was cured at 70°C . Nearly all the CO_2 gas will dissolve and diffuse through the amorphous phase when the foaming temperature approaches 150°C . Thus, cell nucleation and cell development occur mostly in the amorphous regions, while the crystalline sections remain virtually intact. Deformability and foamability (the ability of the polymer to foam) are also weak due to the high viscosity and low water content of the geopolymer composite binder.

Therefore, the cell size is likely to be small (Doroudiani et al., 1996). According to researchers, the presence of cellulose fibers reduced cell size by around 30 percent, suggesting that these materials may have helped to facilitate the development of foam bubbles (Andersons et al., 2020). Larger fiber content was associated with an apparent growing density of foams, most likely due to an increase in the viscosity of the polyol premix. It was observed concurrently with the growth in fiber content (Kurańska et al., 2016).

Figure (e) and (f) shows sample S3, which exhibited the lowest volume expansion ratio due to the cell collapse. The sample expanded at a similar rate as sample S14. However, the expansion was halted at a certain point, and the cell wall started to collapse. During the foaming process, the aggregation of cellulose and the melt stiffness can cause instabilities in the foam network, and foam collapse can occur owing to a lack of cell wall rigidity (Zepnik et al., 2013). Similar findings were reported by Özkan Buzğan et al. (2021). They concluded that an increase in filler ratios, in which the filler acted as a heterogeneous nucleation site during cell formation, caused the cell size of foams to decrease while simultaneously causing an increase in cell density. However, the cellulose agglomerated in the geopolymer composite binder led to irregular cell wall construction, which resulted in the formation of non-uniform cells and many broken cells.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 Conclusion

The influence of various factors on the flexural strength and volume expansion ratio of durian husk cellulose fiber/rice husk ash-based geopolymer composite, as well as the optimal composition, were determined using MINITAB software via response surface methodology (RSM). Results from RSM demonstrated an increase up to 65 °C and 1.5 wt.% in the flexural strength of the geopolymer composite due to curing temperature and cellulose content, respectively while decrease in flexural strength due to curing time (3 to 7 days). In comparison, the results also illustrated an increase in volume expansion ratio due to curing temperature up to 65 °C, a decrease due to cellulose content (0 to 3 wt.%), and an increase due to curing time (3 to 7 days).

The optimum flexural strength and volume expansion ratio values are 3.2977 MPa and 3.6019, respectively. The ideal value to obtain the optimum composition is through curing temperature = 60 °C, cellulose content = 0.5 wt.%, and curing time = 3 days. The control sample (S5), the sample with best performance in both flexural strength and volume expansion ratio, had a flexural strength of 1.92 MPa and a volume expansion ratio of 4.10, respectively. The DHCF/RHA-based geopolymer composite showed good flexural strength and volume expansion ratio properties when the curing temperature, cellulose content, and curing time were 60 °C, 0.5 wt.% and 3 days, respectively. The optimal factors improve the flexural strength and volume expansion ratio of the geopolymer composite.

5.2 Recommendation for Future Research

Several potential improvements to DHCF/RHA-based geopolymer composite were identified. The following are the suggestions proposed for future research:

- a) To use a different cellulose fiber source as reinforcement with the RHA-based geopolymer composite, enhancing the flexural strength and volume expansion ratio properties.
- b) To reduce the cellulose fiber content and minimize the absorption of additional chemical components from mercerization, thus, enhancing the mechanical properties.
- c) To conduct a thermal conductivity test on the foamed DHCF/RHA-based geopolymer composite.

REFERENCES

- Ahmari, S., & Zhang, L. (2012). Production of eco-friendly bricks from copper mine tailings through geopolymerization. *Construction and Building Materials*, 29, 323-331.
- Al-Homoud, M. S. (2005). Performance characteristics and practical applications of common building thermal insulation materials. *Building and environment*, 40(3), 353-366.
- Ali, A. M., Sanjayan, J., & Guerrieri, M. (2017). Performance of geopolymer high strength concrete wall panels and cylinders when exposed to a hydrocarbon fire. *Construction and Building Materials*, 137, 195-207.
- Alonso, S., & Palomo, A. (2001). Alkaline activation of metakaolin and calcium hydroxide mixtures: influence of temperature, activator concentration and solids ratio. *Materials Letters*, 47(1-2), 55-62.
- Amid, B. T., & Mirhosseini, H. (2012). Optimisation of aqueous extraction of gum from durian (*Durio zibethinus*) seed: A potential, low cost source of hydrocolloid. *Food Chemistry*, 132(3), 1258-1268. <https://doi.org/https://doi.org/10.1016/j.foodchem.2011.11.099>
- Andersons, J., Kirpluks, M., & Cabulis, U. (2020). Reinforcement efficiency of cellulose microfibers for the tensile stiffness and strength of rigid low-density polyurethane foams. *Materials*, 13(12), 2725.
- Antiohos, S., Papadakis, V., & Tsimas, S. (2014). Rice husk ash (RHA) effectiveness in cement and concrete as a function of reactive silica and fineness. *Cement and Concrete Research*, 61, 20-27.
- Aprianti, E., Shafigh, P., Bahri, S., & Farahani, J. N. (2015). Supplementary cementitious materials origin from agricultural wastes—A review. *Construction and Building Materials*, 74, 176-187.
- Astika, J., Chandrawati, C., Hendrajaya, I., & Soemarno, Y. O. (2017). The exploration of alpha cellulose in kapok fruit as raw material for rocket propellant production. *Agricultural Research & Technology: Open Access Journal*, 12(4).
- Azevedo, A. G. S., Strecker, K., Barros, L. A., Tonholo, L. F., & Lombardi, C. T. (2019). Effect of curing temperature, activator solution composition and particle size in Brazilian fly-ash based geopolymer production. *Materials Research*, 22.
- Bai, T., Song, Z.-G., Wu, Y.-G., Hu, X.-D., & Bai, H. (2018). Influence of steel slag on the mechanical properties and curing time of metakaolin geopolymer. *Ceramics international*, 44(13), 15706-15713.
- Bakar, R. A., Yahya, R., & Gan, S. N. (2016). Production of high purity amorphous silica from rice husk. *Procedia chemistry*, 19, 189-195.
- Bakharev, T. (2006). Thermal behaviour of geopolymers prepared using class F fly ash and elevated temperature curing. *Cement and Concrete Research*, 36(6), 1134-1147.
- Bao, Y., Grutzeck, M. W., & Jantzen, C. M. (2005). Preparation and properties of hydroceramic waste forms made with simulated Hanford low-activity waste. *Journal of the American Ceramic Society*, 88(12), 3287-3302.

- Barbosa, V. F., & MacKenzie, K. J. (2003). Thermal behaviour of inorganic geopolymers and composites derived from sodium polysialate. *Materials research bulletin*, 38(2), 319-331.
- Baş, D., & Boyacı, I. H. (2007). Modeling and optimization I: Usability of response surface methodology. *Journal of food engineering*, 78(3), 836-845.
- Basri, M. S. M., Mustapha, F., Mazlan, N., & Ishak, M. R. (2020). Optimization of rice husk ash-based geopolymers coating composite for enhancement in flexural properties and microstructure using response surface methodology. *Coatings*, 10(2), 165.
- Beleuk à MOUNGAM, L. M., Lemougna, P. N., Kaze, R. C., Mohamed, H., Deutou Nemaleu, J. G., Billong, N., Kamseu, E., Mvondo-Ze, A. D., & Tonle Kenfack, I. (2022). Synthesis of Volcanic Ash-based Porous Inorganic Polymers Using Biomass as Pore Inducing Agent: Phase Evolution and Descriptive Microstructure. *Silicon*, 14(6), 2595-2608.
- Bell, J. L., Driemeyer, P. E., & Kriven, W. M. (2009a). Formation of ceramics from metakaolin-based geopolymers. Part II: K-based geopolymer. *Journal of the American Ceramic Society*, 92(3), 607-615.
- Bell, J. L., Driemeyer, P. E., & Kriven, W. M. (2009b). Formation of ceramics from metakaolin-based geopolymers: Part I—Cs-based geopolymer. *Journal of the American Ceramic Society*, 92(1), 1-8.
- Bhutta, A., Borges, P. H., Zanotti, C., Farooq, M., & Banthia, N. (2017). Flexural behavior of geopolymer composites reinforced with steel and polypropylene macro fibers. *Cement and Concrete Composites*, 80, 31-40.
- Büchler, C. (1999). Fire safety in industrial buildings and nuclear power plants with airfilters made of geopolymer composite. Proceedings of the Second International Conference Geopolymere, Geopolymer Institute, Saint-Quentin, France,
- Buchwald, A., Hohmann, M., Kaps, C., Bettzieche, H., & Kühnert, J.-T. (2004). Stabilised foam clay material with high performance thermal insulation properties. CFI (Ceramic Forum International/Berichte der DKG),
- Carabba, L., Moricone, R., Scarponi, G. E., Tugnoli, A., & Bignozzi, M. C. (2019). Alkali activated lightweight mortars for passive fire protection: A preliminary study. *Construction and Building Materials*, 195, 75-84.
- Cha, J., Seo, J., & Kim, S. (2012). Building materials thermal conductivity measurement and correlation with heat flow meter, laser flash analysis and TCI. *Journal of Thermal Analysis and calorimetry*, 109(1), 295-300.
- Chakrabarti, M., Virkar, A., & Maiti, H. (1985). Formation of forsterite using rice husk ash for high frequency insulation. *Transactions of the Indian Ceramic Society*, 44(1), 1-9.
- Chao-Lung, H., Le Anh-Tuan, B., & Chun-Tsun, C. (2011). Effect of rice husk ash on the strength and durability characteristics of concrete. *Construction and Building Materials*, 25(9), 3768-3772.
- Chaudhary, D., & Jollands, M. (2004). Characterization of rice hull ash. *Journal of applied polymer science*, 93(1), 1-8.
- Chen, H., Yu, Y., Zhong, T., Wu, Y., Li, Y., Wu, Z., & Fei, B. (2017). Effect of alkali treatment on microstructure and mechanical properties of individual bamboo fibers. *Cellulose*, 24(1), 333-347.
- Chen, K., Wu, D., Zhang, Z., Pan, C., Shen, X., Xia, L., & Zang, J. (2022). Modeling and optimization of fly ash-slag-based geopolymer using response surface

- method and its application in soft soil stabilization. *Construction and Building Materials*, 315, 125723.
- Cheng, T.-W., & Chiu, J. (2003). Fire-resistant geopolymer produced by granulated blast furnace slag. *Minerals engineering*, 16(3), 205-210.
- Chindaprasirt, P., Jaturapitakkul, C., Chalee, W., & Rattanasak, U. (2009). Comparative study on the characteristics of fly ash and bottom ash geopolymers. *Waste Management*, 29(2), 539-543.
- Chindaprasirt, P., & Rattanasak, U. (2018). Fire-resistant geopolymer bricks synthesized from high-calcium fly ash with outdoor heat exposure. *Clean Technologies and Environmental Policy*, 20(5), 1097-1103.
- Colangelo, F., Cioffi, R., Roviello, G., Capasso, I., Caputo, D., Aprea, P., Liguori, B., & Ferone, C. (2017). Thermal cycling stability of fly ash based geopolymer mortars. *Composites part b: engineering*, 129, 11-17.
- Coseri, S. (2017). Cellulose: To depolymerize... or not to? *Biotechnology advances*, 35(2), 251-266.
- Cui, Y., Hao, H., Li, J., & Chen, W. (2021). Failure mechanism of geopolymer composite lightweight sandwich panel under flexural and edgewise compressive loads. *Construction and Building Materials*, 270, 121496.
- Daifullah, A., Girgis, B., & Gad, H. (2003). Utilization of agro-residues (rice husk) in small waste water treatment plans. *Materials Letters*, 57(11), 1723-1731.
- Davidovits, J. (1989). Geopolymers and geopolymeric materials. *Journal of thermal analysis*, 35(2), 429-441.
- Davidovits, J. (1991). Geopolymers: inorganic polymeric new materials. *Journal of Thermal Analysis and calorimetry*, 37(8), 1633-1656.
- Davidovits, J. (1994). Geopolymers: man-made rock geosynthesis and the resulting development of very early high strength cement. *Journal of Materials education*, 16, 91-91.
- Davidovits, J. (1999). Fire proof geopolymeric cements. *Proceeding of Geopolymer*, Davidovits, J. (2008). *Geopolymer chemistry and applications*. Geopolymer Institute.
- Davidovits, J. (2011). *Geopolymer Chemistry & Applications*, Institut Géopolymère (Geopolymer Institute), Saint-Quentin, France.
- Davidovits, J. (2017). Geopolymers: Ceramic-like inorganic polymers. *J. Ceram. Sci. Technol*, 8(3), 335-350.
- Davidovits, J., & Cordi, S. (1979). Synthesis of new high temperature geo-polymers for reinforced plastics/composites. *Spe Pactec*, 79, 151-154.
- Davidovits, J., & Davidovics, M. (1991). Geopolymer: ultra-high temperature tooling material for the manufacture of advanced composites. *How Concept Becomes Reality.*, 36, 1939-1949.
- De Sensale, G. R. (2010). Effect of rice-husk ash on durability of cementitious materials. *Cement and Concrete Composites*, 32(9), 718-725.
- Deb, P. S., Nath, P., & Sarker, P. K. (2014). The effects of ground granulated blast-furnace slag blending with fly ash and activator content on the workability and strength properties of geopolymer concrete cured at ambient temperature. *Materials & Design (1980-2015)*, 62, 32-39.
- Demir, F., & Derun, E. M. (2019). Response surface methodology application to fly ash based geopolymer synthesized by alkali fusion method. *Journal of Non-Crystalline Solids*, 524, 119649.
- Doroudiani, S., Park, C. B., & Kortschot, M. T. (1996). Effect of the crystallinity and morphology on the microcellular foam structure of semicrystalline polymers. *Polymer Engineering & Science*, 36(21), 2645-2662.

- Dufresne, A. (2006). Comparing the mechanical properties of high performances polymer nanocomposites from biological sources. *Journal of Nanoscience and Nanotechnology*, 6(2), 322-330.
- Dufresne, A. (2012). Potential of nanocellulose as a reinforcing phase for polymers. *J-For-Journal of Science & Technology for Forest Products and Processes*, 2(6), 6-16.
- Duxson, P., Fernández-Jiménez, A., Provis, J. L., Lukey, G. C., Palomo, A., & van Deventer, J. S. (2007). Geopolymer technology: the current state of the art. *Journal of materials science*, 42(9), 2917-2933.
- E'zzati, M. S. N., Anuar, H., & Salimah, A. S. M. (2018). Effect of coupling agent on durian skin fibre nanocomposite reinforced polypropylene. IOP Conference Series: Materials Science and Engineering,
- Fernández-Jiménez, A., Palomo, A., & Criado, M. (2005). Microstructure development of alkali-activated fly ash cement: a descriptive model. *Cement and Concrete Research*, 35(6), 1204-1209.
- Flowerday, A., Wright, P., Ledger, R., & Gibson, A. (2006). Investigation into the use of geopolymers for fire resistant marine composites. RINA, Royal Institution of Naval Architects International Conference-Advanced Marine Materials and Coatings,
- Foo, K., & Hameed, B. (2011). Transformation of durian biomass into a highly valuable end commodity: Trends and opportunities. *Biomass and Bioenergy*, 35(7), 2470-2478.
- Funk, J. E., & Dinger, D. R. (2013). *Predictive process control of crowded particulate suspensions: applied to ceramic manufacturing*. Springer Science & Business Media.
- Galiano, Y. L., Leiva, C., Arenas, C., Arroyo, F., Vilches, L., Pereira, C. F., & Villegas, R. (2017). Behaviour of fly ash-based geopolymer panels under fire. *Waste and Biomass Valorization*, 8(7), 2485-2494.
- Gautam, A., Batra, R., & Singh, N. (2019). A study on use of rice husk ash in concrete. *Engineering Heritage Journal (January 1, 2019)*, 01-04.
- Ghaly, A., & Mansaray, K. (1999). Comparative study on the thermal degradation of rice husks in various atmospheres. *Energy Sources*, 21(10), 867-881.
- Giaccio, G., de Sensale, G. R., & Zerbino, R. (2007). Failure mechanism of normal and high-strength concrete with rice-husk ash. *Cement and Concrete Composites*, 29(7), 566-574.
- Giancaspro, J., Balaguru, P., & Lyon, R. E. (2006). Use of inorganic polymer to improve the fire response of balsa sandwich structures. *Journal of materials in civil engineering*, 18(3), 390-397.
- Glen, S. (2013). Adjusted R2/Adjusted R-Squared: What is it used for. Retrieved from *Statistics How To: <https://www.statisticshowto.com/adjusted-r2>*.
- Gonçalves, M., & Bergmann, C. (2007). Thermal insulators made with rice husk ashes: Production and correlation between properties and microstructure. *Construction and Building Materials*, 21(12), 2059-2065.
- Gourley, J., & Johnson, G. (2005). Developments in geopolymer precast concrete. World congress geopolymer,
- Guo, B., Liu, B., Yang, J., & Zhang, S. (2017). The mechanisms of heavy metal immobilization by cementitious material treatments and thermal treatments: A review. *Journal of environmental management*, 193, 410-422.

- Guzmán-Aponte, L. A., Mejía de Gutiérrez, R., & Maury-Ramírez, A. (2017). Metakaolin-based geopolymer with added TiO₂ particles: physicochemical characteristics. *Coatings*, 7(12), 233.
- Han, H.-Z., Li, B.-X., Wu, H., & Shao, W. (2015). Multi-objective shape optimization of double pipe heat exchanger with inner corrugated tube using RSM method. *International Journal of Thermal Sciences*, 90, 173-186.
- Hardjito, D., & Rangan, B. V. (2005). Development and properties of low-calcium fly ash-based geopolymer concrete.
- Hardjito, D., Wallah, S. E., Sumajouw, D. M., & Rangan, B. V. (2004). On the development of fly ash-based geopolymer concrete. *Materials Journal*, 101(6), 467-472.
- Hassaan, M. M., Khater, H. M., El-Mahllawy, M. S., & El Nagar, A. M. (2015). Production of geopolymer composites enhanced by nano-kaolin material. *Journal of advanced ceramics*, 4(4), 245-252.
- He, J., Jie, Y., Zhang, J., Yu, Y., & Zhang, G. (2013). Synthesis and characterization of red mud and rice husk ash-based geopolymer composites. *Cement and Concrete Composites*, 37, 108-118.
- He, J., Zhang, J., Yu, Y., & Zhang, G. (2012). The strength and microstructure of two geopolymers derived from metakaolin and red mud-fly ash admixture: a comparative study. *Construction and Building Materials*, 30, 80-91.
- He, Z.-h., Li, L.-y., & Du, S.-g. (2017). Creep analysis of concrete containing rice husk ash. *Cement and Concrete Composites*, 80, 190-199.
- Hosseini, S., Brake, N. A., Nikookar, M., Günaydın-Şen, Ö., & Snyder, H. A. (2021). Mechanochemically activated bottom ash-fly ash geopolymer. *Cement and Concrete Composites*, 118, 103976.
- Huiskes, D., Keulen, A., Yu, Q., & Brouwers, H. (2016). Design and performance evaluation of ultra-lightweight geopolymer concrete. *Materials & Design*, 89, 516-526.
- Hwang, C. L., & Chandra, S. (1996). The use of rice husk ash in concrete. In *Waste materials used in concrete manufacturing* (pp. 184-234). Elsevier.
- Indran, S., & Raj, R. E. (2015). Characterization of new natural cellulosic fiber from *Cissus quadrangularis* stem. *Carbohydrate polymers*, 117, 392-399.
- International, A. (2007). *Standard test method for flexural properties of polymer matrix composite materials*. ASTM International.
- Jabli, M., Tka, N., Ramzi, K., & Saleh, T. A. (2018). Physicochemical characteristics and dyeing properties of lignin-cellulosic fibers derived from *Nerium oleander*. *Journal of Molecular Liquids*, 249, 1138-1144.
- Jampala, P., Tadikamalla, S., Preethi, M., Ramanujam, S., & Uppuluri, K. B. (2017). Concurrent production of cellulase and xylanase from *Trichoderma reesei* NCIM 1186: enhancement of production by desirability-based multi-objective method. *3 Biotech*, 7(1), 1-13.
- Jämstorp, E., Forsgren, J., Bredenberg, S., Engqvist, H., & Strømme, M. (2010). Mechanically strong geopolymers offer new possibilities in treatment of chronic pain. *Journal of Controlled Release*, 146(3), 370-377.
- Jelle, B. P. (2011). Traditional, state-of-the-art and future thermal building insulation materials and solutions—Properties, requirements and possibilities. *Energy and Buildings*, 43(10), 2549-2563.
- Joseph, B., & Mathew, G. (2012). Influence of aggregate content on the behavior of fly ash based geopolymer concrete. *Scientia Iranica*, 19(5), 1188-1194.

- Kamseu, E., Djangang, C., Veronesi, P., Fernanda, A., Melo, U. C., Sglavo, V. M., & Leonelli, C. (2015). Transformation of the geopolymer gels to crystalline bonds in cold-setting refractory concretes: Pore evolution, mechanical strength and microstructure. *Materials & Design*, 88, 336-344.
- Kamseu, E., Rizzuti, A., Leonelli, C., & Perera, D. (2010). Enhanced thermal stability in K₂O-metakaolin-based geopolymer concretes by Al₂O₃ and SiO₂ fillers addition. *Journal of materials science*, 45(7), 1715-1724.
- Karera, A., Nargis, S., & Patel, M. (1986). Silicon-based materials from rice husk. *Journal of scientific and industrial research*.
- Kaur, K., Singh, J., & Kaur, M. (2018). Compressive strength of rice husk ash based geopolymer: The effect of alkaline activator. *Construction and Building Materials*, 169, 188-192.
- Khalid, N., Rahman, A., Ahmad, S., Toheed, A., & Ahmed, J. (1999). Adsorption behavior of rice husk for the decontamination of chromium from industrial effluents. *Journal of radioanalytical and nuclear chemistry*, 240(3), 775-781.
- Khedari, J., Suttisonk, B., Pratinthong, N., & Hirunlabh, J. (2001). New lightweight composite construction materials with low thermal conductivity. *Cement and Concrete Composites*, 23(1), 65-70.
- Komnitsas, K., & Zaharaki, D. (2007). Geopolymerisation: A review and prospects for the minerals industry. *Minerals engineering*, 20(14), 1261-1277.
- Kong, D. L., & Sanjayan, J. G. (2010). Effect of elevated temperatures on geopolymer paste, mortar and concrete. *Cement and Concrete Research*, 40(2), 334-339.
- Korniejeko, K., Figiela, B., Miernik, K., Ziejewska, C., Marczyk, J., Hebda, M., Cheng, A., & Lin, W.-T. (2021). Mechanical and Fracture Properties of Long Fiber Reinforced Geopolymer Composites. *Materials*, 14(18), 5183.
- Korniejeko, K., Lin, W.-T., & Šimonová, H. (2020). Mechanical properties of short polymer fiber-reinforced geopolymer composites. *Journal of Composites Science*, 4(3), 128.
- Koutnik, P., Soukup, A., Bezucha, P., Šafář, J., & Kohout, J. (2020). Low viscosity metakaolinite based geopolymer binders. *Construction and Building Materials*, 230, 116978.
- Krechkovska, H., Student, O., Lesiuk, G., & Correia, J. (2018). Features of the microstructural and mechanical degradation of long term operated mild steel. *International Journal of Structural Integrity*.
- Krenchel, H. (1975). Fibre spacing and specific fibre surface. *Fibre reinforced cement and concrete*, 1975, 69-79.
- Krishnani, K. K., Meng, X., Christodoulatos, C., & Boddu, V. M. (2008). Biosorption mechanism of nine different heavy metals onto biomatrix from rice husk. *Journal of hazardous materials*, 153(3), 1222-1234.
- Kriven, W. M., Bell, J. L., & Gordon, M. (2003). Microstructure and microchemistry of fully-reacted geopolymers and geopolymer matrix composites. *Ceramic Transactions*, 153(1994), 227-250.
- Krivenko, P. V. (1994). Alkaline cements. Proceedings of the 1st International Conference on Alkaline Cements and Concretes, Kiev, Ukraine, 1994,
- Kumar, A., Mohanta, K., Kumar, D., & Parkash, O. (2012). Properties and industrial applications of rice husk: a review.
- Kumar, V., & Suh, N. P. (1990). A process for making microcellular thermoplastic parts. *Polymer Engineering & Science*, 30(20), 1323-1329.

- Kurańska, M., Prociak, A., Michałowski, S., Cabulis, U., & Kirpluks, M. (2016). Microcellulose as a natural filler in polyurethane foams based on the biopolyol from rapeseed oil. *Polimery*, 61.
- Le, H. T., Nguyen, S. T., & Ludwig, H.-M. (2014). A study on high performance fine-grained concrete containing rice husk ash. *International Journal of Concrete Structures and Materials*, 8(4), 301-307.
- Lee, W.-H., Wang, J.-H., Ding, Y.-C., & Cheng, T.-W. (2019). A study on the characteristics and microstructures of GGBS/FA based geopolymer paste and concrete. *Construction and Building Materials*, 211, 807-813.
- Leiva, C., Luna-Galiano, Y., Arenas, C., Alonso-Fariñas, B., & Fernández-Pereira, C. (2019). A porous geopolymer based on aluminum-waste with acoustic properties. *Waste Management*, 95, 504-512.
- Li, M., Wang, L.-j., Li, D., Cheng, Y.-L., & Adhikari, B. (2014). Preparation and characterization of cellulose nanofibers from de-pectinated sugar beet pulp. *Carbohydrate polymers*, 102, 136-143.
- Li, Q., Chen, S., Zhang, Y., Hu, Y., Wang, Q., Zhou, Q., Yan, Y., Liu, Y., & Yan, D. (2022). Effect of Curing Temperature on High-Strength Metakaolin-Based Geopolymer Composite (HMGC) with Quartz Powder and Steel Fibers. *Materials*, 15(11), 3958.
- Liefke, E. (1999). Industrial applications of foamed inorganic polymers. *Geopolymere*, 99, 189-200.
- Liew, Y., Kamarudin, H., Al Bakri, A. M., Binhussain, M., Luqman, M., Nizar, I. K., Ruzaidi, C., & Heah, C. (2011). Influence of solids-to-liquid and activator ratios on calcined kaolin cement powder. *Physics Procedia*, 22, 312-317.
- Liguori, B., Aprea, P., Roviello, G., & Ferone, C. (2019). Self-supporting zeolites by geopolymer gel conversion (GGC). *Microporous and Mesoporous Materials*, 286, 125-132.
- Liguori, B., Capasso, I., Romeo, V., D'Auria, M., Lavorgna, M., Caputo, D., Iannace, S., & Verdolotti, L. (2017). Hybrid geopolymeric foams with diatomite addition: Effect on chemico-physical properties. *Journal of Cellular Plastics*, 53(5), 525-536.
- Liu, Y., & Hu, H. (2008). X-ray diffraction study of bamboo fibers treated with NaOH. *Fibers and Polymers*, 9(6), 735-739.
- Louati, S., Baklouti, S., & Samet, B. (2016). Acid based geopolymerization kinetics: effect of clay particle size. *Applied Clay Science*, 132, 571-578.
- Lubis, R., Wirjosentono, B., & Septevani, A. A. (2020). Preparation, characterization and antimicrobial activity of grafted cellulose fiber from durian rind waste. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 604, 125311.
- Lyon, R. E., Balaguru, P., Foden, A., Sorathia, U., Davidovits, J., & Davidovics, M. (1997). Fire-resistant aluminosilicate composites. *Fire and materials*, 21(2), 67-73.
- Madandoust, R., Ranjbar, M. M., Moghadam, H. A., & Mousavi, S. Y. (2011). Mechanical properties and durability assessment of rice husk ash concrete. *Biosystems engineering*, 110(2), 144-152.
- Madurwar, M. V., Ralegaonkar, R. V., & Mandavgane, S. A. (2013). Application of agro-waste for sustainable construction materials: A review. *Construction and Building Materials*, 38, 872-878.
- Malhotra, V. M., & Mehta, P. K. (2004). *Pozzolan and cementitious materials*. Crc Press.

- Mallicoat, S., Sarin, P., & Kriven, W. (2009). Novel, alkali-bonded, ceramic filtration membranes. *Developments in Advanced Ceramics and Composites: A Collection of Papers Presented at the 29th International Conference on Advanced Ceramics and Composites*, January 23-28, 2005, Cocoa Beach, Florida, Ceramic Engineering and Science Proceedings,
- Mandal, A., & Chakrabarty, D. (2011). Isolation of nanocellulose from waste sugarcane bagasse (SCB) and its characterization. *Carbohydrate polymers*, 86(3), 1291-1299.
- Mansaray, K., & Ghaly, A. (1999). Thermal degradation of rice husks in an oxygen atmosphere. *Energy Sources*, 21(5), 453-466.
- Mathivet, V., Jouin, J., Gharzouni, A., Sobrados, I., Celerier, H., Rossignol, S., & Parlier, M. (2019). Acid-based geopolymers: understanding of the structural evolutions during consolidation and after thermal treatments. *Journal of Non-Crystalline Solids*, 512, 90-97.
- Mejía, J. M., de Gutiérrez, R. M., & Montes, C. (2016). Rice husk ash and spent diatomaceous earth as a source of silica to fabricate a geopolymeric binary binder. *Journal of cleaner production*, 118, 133-139.
- Mo, B.-h., Zhu, H., Cui, X.-m., He, Y., & Gong, S.-y. (2014). Effect of curing temperature on geopolymerization of metakaolin-based geopolymers. *Applied Clay Science*, 99, 144-148.
- Mohanty, A. K., Misra, M., & Drzal, L. T. (2005). *Natural fibers, biopolymers, and biocomposites*. CRC press.
- Mohd Basri, M. S., Mustapha, F., Mazlan, N., & Ishak, M. R. (2020). Optimization of adhesion strength and microstructure properties by using response surface methodology in enhancing the rice husk ash-based geopolymer composite coating. *Polymers*, 12(11), 2709.
- Mohd Basri, M. S., Yek, T. H., A Talib, R., Mohamed Amin Tawakkal, I. S., Kamarudin, S. H., Mazlan, N., Maidin, N. A., & Ab Rahman, M. H. (2021). Rice Husk Ash/Silicone Rubber-Based Binary Blended Geopolymer Coating Composite: Fire Retardant, Moisture Absorption, Optimize Composition, and Microstructural Analysis. *Polymers*, 13(6), 985.
- Mohsen, Q., & Mostafa, N. Y. (2010). Investigating the possibility of utilising low kaolinitic clays in production of geopolymer bricks. *Ceramics-Silikaty*, 54(2), 160-168.
- Mohseni, E., Kazemi, M. J., Koushkbaghi, M., Zehtab, B., & Behforouz, B. (2019). Evaluation of mechanical and durability properties of fiber-reinforced lightweight geopolymer composites based on rice husk ash and nano-alumina. *Construction and Building Materials*, 209, 532-540.
- Morrissey, F., Coutts, R., & Grossman, P. (1985). Bond between cellulose fibres and cement. *International Journal of Cement Composites and Lightweight Concrete*, 7(2), 73-80.
- Muthadhi, A., Anitha, R., & Kothandaraman, S. (2007). Rice husk ash-Properties and its uses: a review. *Journal of the Institution of Engineers. India. Civil Engineering Division*, 88(5), 50-56.
- Nagrle, S., Hajare, H., & Modak, P. R. (2012). Utilization of rice husk ash. *Carbon*, 2(6), 42.
- Nana, A., Epey, N., Rodrique, K. C., Deutou, J. G. N., Djobo, J. N. Y., Tomé, S., Alomayri, T. S., Ngouné, J., Kamseu, E., & Leonelli, C. (2021). Mechanical strength and microstructure of metakaolin/volcanic ash-based geopolymer

- composites reinforced with reactive silica from rice husk ash (RHA). *Materialia*, 16, 101083.
- Natali Murri, A., Medri, V., & Landi, E. (2017). Production and thermomechanical characterization of wool–geopolymer composites. *Journal of the American Ceramic Society*, 100(7), 2822-2831.
- Nikolov, A., Rostovsky, I., & Nugteren, H. (2017). Geopolymer materials based on natural zeolite. *Case Studies in Construction Materials*, 6, 198-205.
- Novais, R. M., Buruberri, L., Ascensão, G., Seabra, M., & Labrincha, J. (2016). Porous biomass fly ash-based geopolymers with tailored thermal conductivity. *Journal of cleaner production*, 119, 99-107.
- Nuaklong, P., Jongvivatsakul, P., Pothisiri, T., Sata, V., & Chindaprasirt, P. (2020). Influence of rice husk ash on mechanical properties and fire resistance of recycled aggregate high-calcium fly ash geopolymer concrete. *Journal of cleaner production*, 252, 119797.
- Ochi, S. (2008). Mechanical properties of kenaf fibers and kenaf/PLA composites. *Mechanics of materials*, 40(4-5), 446-452.
- Olawale, M. D. (2013). Syntheses, characterization and binding strength of geopolymers: A review.
- Özkan Buzğan, E., Kaya, G., Kertmen, M., & Türksöy, H. (2021). Properties of textile air conditioner waste dust-filled polyurethane rigid foam composites. *Plastics, Rubber and Composites*, 1-11.
- Palomo, A., & dela Fuente, J. L. (2003). Alkali-activated cementitious materials: Alternative matrices for the immobilisation of hazardous wastes: Part I. Stabilisation of boron. *Cement and Concrete Research*, 33(2), 281-288.
- Palomo, A., Grutzeck, M., & Blanco, M. (1999). Alkali-activated fly ashes: A cement for the future. *Cement and Concrete Research*, 29(8), 1323-1329.
- Pan, Z., & Sanjayan, J. G. (2010). Stress–strain behaviour and abrupt loss of stiffness of geopolymer at elevated temperatures. *Cement and Concrete Composites*, 32(9), 657-664.
- Pan, Z., Sanjayan, J. G., & Kong, D. L. (2012). Effect of aggregate size on spalling of geopolymer and Portland cement concretes subjected to elevated temperatures. *Construction and Building Materials*, 36, 365-372.
- Pan, Z., Sanjayan, J. G., & Rangan, B. V. (2009). An investigation of the mechanisms for strength gain or loss of geopolymer mortar after exposure to elevated temperature. *Journal of materials science*, 44(7), 1873-1880.
- Panesar, D., Leung, R., Sain, M., & Panthapulakkal, S. (2017). The effect of sodium hydroxide surface treatment on the tensile strength and elastic modulus of cellulose nanofiber. In *Sustainable and nonconventional construction materials using inorganic bonded fiber composites* (pp. 17-26). Elsevier.
- Pangdaeng, S., Sata, V., Aguiar, J., Pacheco-Torgal, F., & Chindaprasirt, P. (2015). Apatite formation on calcined kaolin–white Portland cement geopolymer. *Materials Science and Engineering: C*, 51, 1-6.
- Papakonstantinou, C. G., & Balaguru, P. N. (2007). Fatigue behavior of high temperature inorganic matrix composites. *Journal of materials in civil engineering*, 19(4), 321-328.
- Park, K.-B., Kwon, S.-J., & Wang, X.-Y. (2016). Analysis of the effects of rice husk ash on the hydration of cementitious materials. *Construction and Building Materials*, 105, 196-205.
- Penjumras, P., Rahman, R. A., Talib, R. A., & Abdan, K. (2015). Mechanical properties and water absorption behaviour of durian rind cellulose reinforced

- poly (lactic acid) biocomposites. *International Journal on Advanced Science, Engineering and Information Technology*, 5(5), 343-349.
- Penjumras, P., Rahman, R. B. A., Talib, R. A., & Abdan, K. (2014). Extraction and characterization of cellulose from durian rind. *Agriculture and Agricultural Science Procedia*, 2, 237-243.
- Perera, D., Uchida, O., Vance, E., & Finnie, K. (2007). Influence of curing schedule on the integrity of geopolymers. *Journal of materials science*, 42(9), 3099-3106.
- Perera, D. S., & Trautman, R. L. (2005). Geopolymers with the potential for use as refractory castables. *Advances in Technology of Materials and Materials Processing Journal(ATM)*, 7(2), 187-190.
- Phanthong, P., Reubroycharoen, P., Hao, X., Xu, G., Abudula, A., & Guan, G. (2018). Nanocellulose: Extraction and application. *Carbon Resources Conversion*, 1(1), 32-43.
- Pistol, K., Rickard, W. D., & Gluth, G. J. (2016). Mechanical properties of fly ash-based geopolymer concretes at high temperature. *Bautechnik*, 93(8), 521-530.
- Pocketbook, F. S. (2015). World food and agriculture. *FAO Rome Italy*.
- Prasara-A, J., & Gheewala, S. H. (2017). Sustainable utilization of rice husk ash from power plants: A review. *Journal of cleaner production*, 167, 1020-1028.
- Putri, D. A., & Kurniyati, Z. (2016). Effect of sodium chloroacetate towards the synthesis of CMC (carboxymethyl cellulose) from durian (*Durio zibethinus*) peel cellulose. *International Journal of Innovative Research in Advanced Engineering (IJIRAE)*, 3(3), 28-32.
- Qi, Y., Thapa, K. B., & Hoadley, A. F. (2011). Application of filtration aids for improving sludge dewatering properties—a review. *Chemical Engineering Journal*, 171(2), 373-384.
- Qin, L., Gao, X., & Chen, T. (2018). Recycling of raw rice husk to manufacture magnesium oxysulfate cement based lightweight building materials. *Journal of cleaner production*, 191, 220-232.
- Rachtanapun, P., Luangkamin, S., Tanprasert, K., & Suriyatem, R. (2012). Carboxymethyl cellulose film from durian rind. *LWT-Food Science and Technology*, 48(1), 52-58.
- Rahier, H., Van Mele, B., Biesemans, M., Wastiels, J., & Wu, X. (1996). Low-temperature synthesized aluminosilicate glasses. *Journal of materials science*, 31(1), 71-79.
- Rahman, A. S. (2019). Effects of nanofibers on properties of geopolymer composites. In *Nanotechnology in Eco-efficient Construction* (pp. 123-140). Elsevier.
- Rahman, A. S., Jackson, P., & Radford, D. W. (2020). Improved toughness and delamination resistance in continuous fiber reinforced geopolymer composites via incorporation of nano-fillers. *Cement and Concrete Composites*, 108, 103496.
- Rahman, A. S., Shah, C., & Gupta, N. (2020). Simultaneous effects of rice husk silica and silicon carbide whiskers on the mechanical properties and morphology of sodium geopolymer. *Journal of Composite Materials*, 54(29), 4611-4620.
- Ramasamy, V.-w. (2012). Compressive strength and durability properties of rice husk ash concrete. *KSCE Journal of Civil Engineering*, 16(1), 93-102.
- Rangan, B. V. (2014). Geopolymer concrete for environmental protection. *The Indian concrete journal*, 88(4), 41-59.
- Reddy, N., & Yang, Y. (2005). Biofibers from agricultural byproducts for industrial applications. *TRENDS in Biotechnology*, 23(1), 22-27.

- Ribero, D., & Kriven, W. M. (2016). Properties of geopolymer composites reinforced with basalt chopped strand mat or woven fabric. *Journal of the American Ceramic Society*, 99(4), 1192-1199.
- Rickard, W. D., & Van Riessen, A. (2014). Performance of solid and cellular structured fly ash geopolymers exposed to a simulated fire. *Cement and Concrete Composites*, 48, 75-82.
- Rickard, W. D., Vickers, L., & Van Riessen, A. (2013). Performance of fibre reinforced, low density metakaolin geopolymers under simulated fire conditions. *Applied Clay Science*, 73, 71-77.
- Roopchand, R., Andrew, J., & Sithole, B. (2022). Using cellulose nanocrystals to improve the mechanical properties of fly ash-based geopolymer construction materials. *Engineering Science and Technology, an International Journal*, 25, 100989.
- Rößler, C., Bui, D.-D., & Ludwig, H.-M. (2014). Rice husk ash as both pozzolanic admixture and internal curing agent in ultra-high performance concrete. *Cement and Concrete Composites*, 53, 270-278.
- Roviello, G., Ricciotti, L., Ferone, C., Colangelo, F., & Tarallo, O. (2015). Fire resistant melamine based organic-geopolymer hybrid composites. *Cement and Concrete Composites*, 59, 89-99.
- Rovnaník, P. (2010). Effect of curing temperature on the development of hard structure of metakaolin-based geopolymer. *Construction and Building Materials*, 24(7), 1176-1183.
- Rozainee, M., Ngo, S. P., Salema, A., Tan, K. G., Ariffin, M., & Zainura, Z. (2008). Effect of fluidising velocity on the combustion of rice husk in a bench-scale fluidised bed combustor for the production of amorphous rice husk ash. *Bioresource Technology*, 99(4), 703-713.
- Safari, Z., Kurda, R., Al-Hadad, B., Mahmood, F., & Tapan, M. (2020). Mechanical characteristics of pumice-based geopolymer paste. *Resources, Conservation and Recycling*, 162, 105055.
- Sajan, P., Jiang, T., Lau, C., Tan, G., & Ng, K. (2021). Combined effect of curing temperature, curing period and alkaline concentration on the mechanical properties of fly ash-based geopolymer. *Cleaner Materials*, 1, 100002.
- Sakkas, K., Nomikos, P., Sofianos, A., & Pantias, D. (2013). Slag based geopolymer for passive fire protection of tunnels. *Underground. The Way to the Future.—Informa UK Limited*, 343-349.
- Salas, A., Delvasto, S., de Gutierrez, R. M., & Lange, D. (2009). Comparison of two processes for treating rice husk ash for use in high performance concrete. *Cement and Concrete Research*, 39(9), 773-778.
- Salas, J., Alvarez, M., & Veras, J. (1986). Lightweight insulating concretes with rice husk. *International Journal of Cement Composites and Lightweight Concrete*, 8(3), 171-180.
- Samal, S., Phan Thanh, N., Petriková, I., & Marvalová, B. (2015). Improved mechanical properties of various fabric-reinforced geocomposite at elevated temperature. *Jom*, 67(7), 1478-1485.
- Sandhu, R. K., & Siddique, R. (2017). Influence of rice husk ash (RHA) on the properties of self-compacting concrete: A review. *Construction and Building Materials*, 153, 751-764.
- Sankar, K., Stynoski, P., Al-Chaar, G. K., & Kriven, W. M. (2018). Sodium silicate activated slag-fly ash binders: Part I—Processing, microstructure, and

- mechanical properties. *Journal of the American Ceramic Society*, 101(6), 2228-2244.
- Saravanan, S., & Kumar, M. S. (2013). Effect of mechanical properties on rice husk ash reinforced aluminum alloy (AlSi10Mg) matrix composites. *Procedia engineering*, 64, 1505-1513.
- Shaikh, F. U. A., & Hosan, A. (2016). Mechanical properties of steel fibre reinforced geopolymer concretes at elevated temperatures. *Construction and Building Materials*, 114, 15-28.
- Shakouri, S., Bayer, Ö., & Erdoğan, S. T. (2020). Development of silica fume-based geopolymer foams. *Construction and Building Materials*, 260, 120442.
- Sindhunata, Van Deventer, J., Lukey, G., & Xu, H. (2006). Effect of curing temperature and silicate concentration on fly-ash-based geopolymerization. *Industrial & Engineering Chemistry Research*, 45(10), 3559-3568.
- Singh, B. (2018). Rice husk ash. In *Waste and supplementary cementitious materials in concrete* (pp. 417-460). Elsevier.
- Sofi, M., Van Deventer, J., Mendis, P., & Lukey, G. (2007). Engineering properties of inorganic polymer concretes (IPCs). *Cement and Concrete Research*, 37(2), 251-257.
- Sun, J., Sun, X., Zhao, H., & Sun, R. (2004). Isolation and characterization of cellulose from sugarcane bagasse. *Polymer degradation and stability*, 84(2), 331-339.
- Swamy, R. (1975). Fibre reinforcement of cement and concrete. *Matériaux et Construction*, 8(3), 235-254.
- Taveri, G., Bernardo, E., & Dlouhy, I. (2018). Mechanical performance of glass-based geopolymer matrix composites reinforced with cellulose fibers. *Materials*, 11(12), 2395.
- Tawakkal, I. S. M., Talib, R. A., Abdan, K., & Ling, C. N. (2012). Mechanical and physical properties of kenaf-derived cellulose (KDC)-filled polylactic acid (PLA) composites. *BioResources*, 7(2), 1643-1655.
- Tchakouté, H. K., & Rüschler, C. H. (2017). Mechanical and microstructural properties of metakaolin-based geopolymer cements from sodium waterglass and phosphoric acid solution as hardeners: A comparative study. *Applied Clay Science*, 140, 81-87.
- Temuujin, J., Rickard, W., Lee, M., & van Riessen, A. (2011). Preparation and thermal properties of fire resistant metakaolin-based geopolymer-type coatings. *Journal of Non-Crystalline Solids*, 357(5), 1399-1404.
- Temuujin, J., Williams, R., & Van Riessen, A. v. (2009). Effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient temperature. *Journal of materials processing technology*, 209(12-13), 5276-5280.
- Thomas, B. S. (2018). Green concrete partially comprised of rice husk ash as a supplementary cementitious material—A comprehensive review. *Renewable and Sustainable Energy Reviews*, 82, 3913-3923.
- Tome, S., Nana, A., Kaze, C. R., Djobo, J. N. Y., Alomayri, T., Kamseu, E., Etoh, M.-A., Etame, J., & Kumar, S. (2022). Resistance of alkali-activated blended volcanic ash-MSWI-FA mortar in sulphuric acid and artificial seawater. *Silicon*, 14(6), 2687-2694.
- Topal, G. G. (2020). *A Study on the Use of Geopolymer Composites for Nitrogen and Phosphorus Removal from Poultry Litter* [Oklahoma State University].

- Ugheoke, I. B., & Mamat, O. (2012). A critical assessment and new research directions of rice husk silica processing methods and properties. *Maejo international journal of science and technology*, 6(3), 430.
- ul Haq, E., Padmanabhan, S. K., & Licciulli, A. (2014). Synthesis and characteristics of fly ash and bottom ash based geopolymers—A comparative study. *Ceramics international*, 40(2), 2965-2971.
- Van Wazer, J. R. (1970). Equilibria and kinetics in inorganic polymerizations. *Inorg. Macromol*, 1(8).
- Vlaev, L., Markovska, I., & Lyubchev, L. (2003). Non-isothermal kinetics of pyrolysis of rice husk. *Thermochimica acta*, 406(1-2), 1-7.
- Wang, Y. (2008). *Cellulose fiber dissolution in sodium hydroxide solution at low temperature: Dissolution kinetics and solubility improvement*. Georgia Institute of Technology.
- Wang, Y., & Zhao, J. (2019). Facile preparation of slag or fly ash geopolymer composite coatings with flame resistance. *Construction and Building Materials*, 203, 655-661.
- Watolla, M.-B., Gluth, G., Sturm, P., Rickard, W., Krüger, S., & Scharrel, B. (2017). Intumescent geopolymer-bound coatings for fire protection of steel. *Journal of ceramic science and technology*, 8(3 (Topical issue: Geopolymers)), 351-364.
- Xu, H., & Van Deventer, J. (2000). The geopolymerisation of aluminosilicate minerals. *International journal of mineral processing*, 59(3), 247-266.
- Xu, W., Lo, T. Y., & Memon, S. A. (2012). Microstructure and reactivity of rich husk ash. *Construction and Building Materials*, 29, 541-547.
- Xu, Z.-M., Jiang, X.-L., Liu, T., Hu, G.-H., Zhao, L., Zhu, Z.-N., & Yuan, W.-K. (2007). Foaming of polypropylene with supercritical carbon dioxide. *The Journal of Supercritical Fluids*, 41(2), 299-310.
- Yaseri, S., Verki, V. M., & Mahdikhani, M. (2019). Utilization of high volume cement kiln dust and rice husk ash in the production of sustainable geopolymer. *Journal of cleaner production*, 230, 592-602.
- Ye, H., Zhang, Y., Yu, Z., & Mu, J. (2018). Effects of cellulose, hemicellulose, and lignin on the morphology and mechanical properties of metakaolin-based geopolymer. *Construction and Building Materials*, 173, 10-16.
- Zahedi, M., Ramezani pour, A. A., & Ramezani pour, A. M. (2015). Evaluation of the mechanical properties and durability of cement mortars containing nanosilica and rice husk ash under chloride ion penetration. *Construction and Building Materials*, 78, 354-361.
- Zahid, M., Shafiq, N., Isa, M. H., & Gil, L. (2018). Statistical modeling and mix design optimization of fly ash based engineered geopolymer composite using response surface methodology. *Journal of cleaner production*, 194, 483-498.
- Zain, H., Abdullah, M. M. A. B., Hussin, K., Ariffin, N., & Bayuaji, R. (2017). Review on various types of geopolymer materials with the environmental impact assessment. MATEC Web of Conferences,
- Zepnik, S., Hildebrand, T., Kabasci, S., Radusch, H.-J., & Wodke, T. (2013). Cellulose acetate for thermoplastic foam extrusion. *Cellulose-Biomass Conversion; De Ven, TV, Kadla, J., Eds*, 17-44.
- Zhang, H. Y., Kodur, V., Qi, S. L., Cao, L., & Wu, B. (2014). Development of metakaolin-fly ash based geopolymers for fire resistance applications. *Construction and Building Materials*, 55, 38-45.

- Zhang, H. Y., Kodur, V., Qi, S. L., & Wu, B. (2015). Characterizing the bond strength of geopolymers at ambient and elevated temperatures. *Cement and Concrete Composites*, 58, 40-49.
- Zhang, H. Y., Kodur, V., Wu, B., Yan, J., & Yuan, Z. S. (2018). Effect of temperature on bond characteristics of geopolymer concrete. *Construction and Building Materials*, 163, 277-285.
- Zhang, R., Liu, T., Zhang, Y., Cai, Z., & Yuan, Y. (2020). Preparation of spent fluid catalytic cracking catalyst-metakaolin based geopolymer and its process optimization through response surface method. *Construction and Building Materials*, 264, 120727.
- Zhao, R., & Sanjayan, J. G. (2011). Geopolymer and Portland cement concretes in simulated fire. *Magazine of Concrete research*, 63(3), 163-173.
- Zhao, S., Xia, M., Yu, L., Huang, X., Jiao, B., & Li, D. (2021). Optimization for the preparation of composite geopolymer using response surface methodology and its application in lead-zinc tailings solidification. *Construction and Building Materials*, 266, 120969.
- Zhao, X., Qian, T., Mei, G., Kwan, C., Zane, R., Walsh, C., Paing, T., & Popovic, Z. (2007). Active health monitoring of an aircraft wing with an embedded piezoelectric sensor/actuator network: II. Wireless approaches. *Smart materials and structures*, 16(4), 1218.
- Zou, Y., & Yang, T. (2019). Chapter 9-Rice Husk, Rice Husk Ash and Their Applications. Rice Bran and Rice Bran Oil, Chemistry, Processing and Utilization. *Urbana, Illinois (USA): AOCS Press/Academic Press*, 207-246.
- Zuhua, Z., Xiao, Y., Huajun, Z., & Yue, C. (2009). Role of water in the synthesis of calcined kaolin-based geopolymer. *Applied Clay Science*, 43(2), 218-223.

APPENDICES

Appendix A1

The calculation on the concentration of related materials used in the lab work.

- 10M NaOH

Molecular weight of NaOH = 40 g/mol

$$10M NaOH = 10 \frac{mol}{L} \times 40 \frac{g}{mol}$$

$$NaOH = 400 g/L$$

Therefore, 400 g of NaOH is required for every 1000 mL of NaOH solution.

- 17.5% NaOH

$$17.5\% NaOH = \frac{x \text{ gram NaOH}}{100 \text{ mL NaOH} + \text{distilled water}}$$

$$NaOH = 17.5 g$$

Therefore, 17.5 g of NaOH is required for every 100 mL of NaOH solution.

- 8.3% NaOH

$$8.3\% NaOH = \frac{x \text{ gram NaOH}}{100 \text{ mL NaOH} + \text{distilled water}}$$

$$NaOH = 8.3 g$$

Therefore, 8.3 g of NaOH is required for every 100 mL of NaOH solution.

- 10% CH₃COOH

$$10\% \text{ CH}_3\text{COOH} = \frac{x \text{ mL CH}_3\text{COOH}}{100 \text{ mL CH}_3\text{COOH} + \text{distilled water}}$$

$$\text{NaOH} = 10 \text{ mL}$$

Therefore, 10 mL of CH₃COOH is required for every 100 mL solution.

- AA ratio = 3.5

$$3.5 = \frac{\text{Na}_2\text{SiO}_3}{\text{NaOH}}$$

$$\text{Mass of Na}_2\text{SiO}_3 = 14.32 \text{ g}$$

$$\text{Mass of NaOH} = \frac{\text{Na}_2\text{SiO}_3}{3.5} = \frac{14.32 \text{ g}}{3.5} = 4.09 \text{ g}$$

- RHA/AA ratio = 0.25

$$0.25 = \frac{\text{RHA}}{\text{AA}}$$

$$\begin{aligned} \text{Mass of AA solution} &= \text{Mass of Na}_2\text{SiO}_3 + \text{Mass of NaOH} \\ &= 14.32 + 4.09 = 18.41 \text{ g} \end{aligned}$$

$$\text{Mass of RHA} = 0.25 \times \text{AA} = 0.25 \times 18.41 \text{ g} = 4.60 \text{ g}$$

- Cellulose content = 3 wt.%

$$3\text{wt.}\% = \frac{\text{Mass of cellulose}}{\text{Total mass needed}}$$

Flexural Test

$$\text{Length} = 14 \text{ cm}$$

$$\text{Width} = 2.7 \text{ cm}$$

$$\text{Height} = 0.4 \text{ cm}$$

$$\text{Volume} = 15.12 \text{ cm}^3$$

$$\text{Density} = 1.3 \text{ g/cm}^3$$

$$\text{Mass needed for flexural test} = V \times \rho = 15.12 \times 1.3 = 19.66g$$

Rate of Expansion

$$\text{Length} = 2.5 \text{ cm}$$

$$\text{Width} = 2.5 \text{ cm}$$

$$\text{Height} = 0.5 \text{ cm}$$

$$\text{Volume} = 3.13 \text{ cm}^3$$

$$\text{Density} = 1.3 \text{ g/cm}^3$$

$$\text{Mass needed for rate of expansion} = V \times \rho = 3.13 \times 1.3 = 4.06g$$

$$\text{Total mass needed} = 19.66 + 4.06 = 23.72 \text{ g}$$

$$\text{Mass of cellulose} = \frac{3}{100} \times 23.72 = 0.71g$$

- Total materials mass = Mass of (AA solution + RHA + Cellulose)
= 18.41 + 4.60 + 0.71
= 23.72 g

Appendix B1

The calculation on the sample testing set-up applied in the lab work.

- Flexural strength testing set-up – distance between support, l

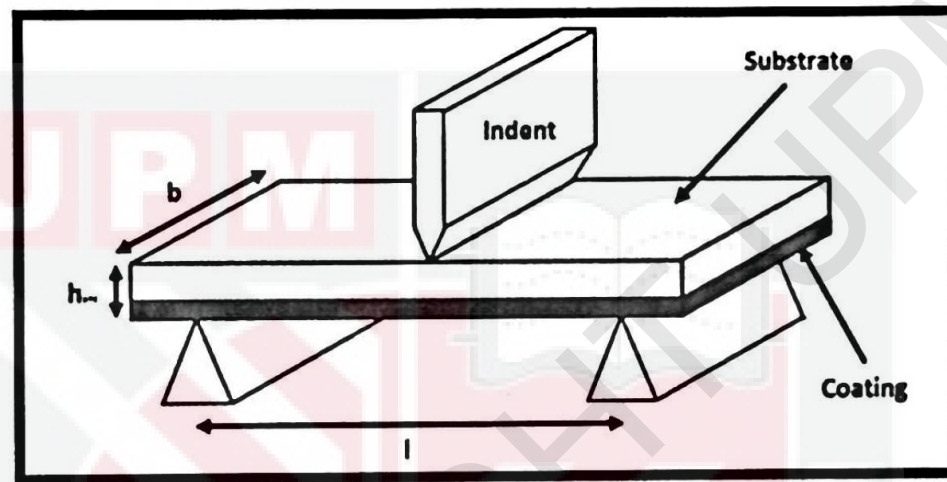


Figure B1. 1: 3-D view of three-point bending test set-up. (Basri et al., 2020)

$$\text{Distance between support, } l = (2r + 3t) \pm \frac{t}{2}$$

where r = radius of the indent = 0.4 cm

t = thickness of the specimen = 0.273 cm (on average of 51 samples)

$$\text{Distance between support, } l = [(2 \times 0.4) + (3 \times 0.273)] \pm \frac{0.273}{2} = 1.5 \text{ cm or } 1.8 \text{ cm}$$

Therefore, the distance between support, l is 1.5 cm.

Appendix B2

The calculation on the volume expansion ratio.

- Density of sample before

mass of sample, $m_i = 0.22 \text{ g}$

volume of sample, $V_i = 0.17 \text{ cm}^3$

density of sample, $\rho_i = \frac{m_i}{V_i} = \frac{0.22 \text{ g}}{0.17 \text{ cm}^3} = 1.27 \text{ g/cm}^3$

- Density of sample after

mass of sample, $m_f = 3.91 \text{ g}$

volume of sample, $V_f = 13.13 \text{ cm}^3$

density of sample, $\rho_f = \frac{m_f}{V_f} = \frac{3.91 \text{ g}}{13.13 \text{ cm}^3} = 0.30 \text{ g/cm}^3$

- Volume expansion ratio $= \frac{\rho_i}{\rho_f} = \frac{1.27 \text{ g/cm}^3}{0.30 \text{ g/cm}^3} = 4.25$

Appendix C1

Flexural Strength & Volume Expansion Ratio – RSM

Run Order	Sample	Coded Factor			Uncoded Factor			Flexural Strength (MPa)	Volume Expansion Ratio
		Curing	Cellulose	Curing	Curing	Cellulose	Curing		
		Temperature	Content	Time	Temperature	Content	Time		
		(V ₁)	(V ₂)	(V ₃)	(V ₁)	(V ₂)	(V ₃)		
1	S1	0	0	0	60	1.5	5	2.75	3.5
2	S2	1	1	0	70	3.0	5	2.09	2.3
3	S3	-1	0	1	50	1.5	3	2.18	1.3
4	S4	-1	1	0	50	3.0	5	1.10	1.2
5	S5	1	-1	0	70	0.0	5	1.92	3.4
6	S6	-1	-1	0	50	0.0	5	1.15	1.6
7	S7	0	0	0	60	1.5	5	2.33	3.6
8	S8	0	0	0	60	1.5	5	2.42	3.6
9	S9	0	-1	1	60	0.0	7	1.67	5.3
10	S10	0	1	-1	60	3.0	3	3.20	2.3
11	S11	0	1	1	60	3.0	7	1.92	2.6
12	S12	1	0	1	70	1.5	7	1.83	2.9
13	S13	0	-1	-1	60	0.0	3	3.50	4.0
14	S14	1	0	-1	70	1.5	3	3.87	1.0
15	S15	-1	0	1	50	1.5	7	1.17	1.4

Appendix D1

Flexural Strength – RSM

Response Surface Regression: Flexural Strength (MPa) ... g Time (Days)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	9.66116	1.07346	36.92	0.000
Linear	3	6.85533	2.28511	78.59	0.000
Curing Temperature (°C)	1	2.11151	2.11151	72.62	0.000
Cellulose Content (wt.%)	1	0.00061	0.00061	0.02	0.890
Curing Time (Days)	1	4.74320	4.74320	163.14	0.000
Square	3	2.45288	0.81763	28.12	0.001
Curing Temperature (°C)*Curing Temperature (°C)	1	1.43079	1.43079	49.21	0.001
Cellulose Content (wt.%)*Cellulose Content (wt.%)	1	0.36058	0.36058	12.40	0.017
Curing Time (Days)*Curing Time (Days)	1	0.54729	0.54729	18.82	0.007
2-Way Interaction	3	0.35295	0.11765	4.05	0.083
Curing Temperature (°C)*Cellulose Content (wt.%)	1	0.01210	0.01210	0.42	0.547
Curing Temperature (°C)*Curing Time (Days)	1	0.26522	0.26522	9.12	0.029
Cellulose Content (wt.%)*Curing Time (Days)	1	0.07562	0.07562	2.60	0.168
Error	5	0.14537	0.02907		
Lack-of-Fit	3	0.04757	0.01586	0.32	0.813
Pure Error	2	0.09780	0.04890		
Total	14	9.80653			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.170514	98.52%	95.85%	89.99%

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	2.5000	0.0984	25.39	0.000	
Curing Temperature (°C)	0.5138	0.0603	8.52	0.000	1.00
Cellulose Content (wt.%)	0.0088	0.0603	0.15	0.890	1.00
Curing Time (Days)	-0.7700	0.0603	-12.77	0.000	1.00
Curing Temperature (°C)*Curing Temperature (°C)	-0.6225	0.0887	-7.02	0.001	1.01
Cellulose Content (wt.%)*Cellulose Content (wt.%)	-0.3125	0.0887	-3.52	0.017	1.01
Curing Time (Days)*Curing Time (Days)	0.3850	0.0887	4.34	0.007	1.01
Curing Temperature (°C)*Cellulose Content (wt.%)	0.0550	0.0853	0.65	0.547	1.00
Curing Temperature (°C)*Curing Time (Days)	-0.2575	0.0853	-3.02	0.029	1.00
Cellulose Content (wt.%)*Curing Time (Days)	0.1375	0.0853	1.61	0.168	1.00

Regression Equation in Uncoded Units

Flexural Strength (MPa) = -22.17 + 0.857 Curing Temperature (°C)
 - 0.027 Cellulose Content (wt.)
 - 0.644 Curing Time (Days)
 - 0.006225 Curing Temperature (°C)*Curing Temperature (°C)
 - 0.1389 Cellulose Content (wt.)*Cellulose Content (wt.)
 + 0.0963 Curing Time (Days)*Curing Time (Days)
 + 0.00367 Curing Temperature (°C)*Cellulose Content (wt.)
 - 0.01287 Curing Temperature (°C)*Curing Time (Days)
 + 0.0458 Cellulose Content (wt.)*Curing Time (Days)

Fits and Diagnostics for All Observations

Obs	Flexural Strength (MPa)	Fit	Resid	Std Resid
1	2.750	2.500	0.250	1.80
2	2.090	2.142	-0.052	-0.62
3	2.180	2.261	-0.081	-0.95
4	1.100	1.005	0.095	1.11
5	1.920	2.015	-0.095	-1.11
6	1.150	1.097	0.053	0.62
7	2.330	2.500	-0.170	-1.22
8	2.420	2.500	-0.080	-0.57
9	1.670	1.656	0.014	0.16
10	3.200	3.214	-0.014	-0.16
11	1.920	1.949	-0.029	-0.34
12	1.830	1.749	0.081	0.95
13	3.500	3.471	0.029	0.34
14	3.870	3.804	0.066	0.78
15	1.170	1.236	-0.066	-0.78

Response Optimization: Volume Expansion Ratio, ... ral Strength (MPa)

Multiple Response Prediction

Variable	Setting			
Curing Temperature (°C)	62.1212			
Cellulose Content (wt.%)	0			
Curing Time (Days)	3			
Response	Fit	SE Fit	95% CI	95% PI
Volume Expansion Ratio	3.563	0.470	(2.355, 4.771)	(1.729, 5.396)
Flexural Strength (MPa)	3.595	0.149	(3.211, 3.979)	(3.012, 4.178)

Appendix D2

Volume Expansion Ratio – RSM

Response Surface Regression: Volume Expansion Ratio ... Time (Days)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	20.1142	2.2349	7.76	0.018
Linear	3	8.0725	2.6908	9.35	0.017
Curing Temperature (°C)	1	2.1012	2.1012	7.30	0.043
Cellulose Content (wt.%)	1	4.3512	4.3512	15.12	0.012
Curing Time (Days)	1	1.6200	1.6200	5.63	0.064
Square	3	10.8592	3.6197	12.58	0.009
Curing Temperature (°C)*Curing Temperature (°C)	1	10.3078	10.3078	35.81	0.002
Cellulose Content (wt.%)*Cellulose Content (wt.%)	1	0.1939	0.1939	0.67	0.449
Curing Time (Days)*Curing Time (Days)	1	0.2231	0.2231	0.78	0.419
2-Way Interaction	3	1.1825	0.3942	1.37	0.353
Curing Temperature (°C)*Cellulose Content (wt.%)	1	0.1225	0.1225	0.43	0.543
Curing Temperature (°C)*Curing Time (Days)	1	0.8100	0.8100	2.81	0.154
Cellulose Content (wt.%)*Curing Time (Days)	1	0.2500	0.2500	0.87	0.394
Error	5	1.4392	0.2878		
Lack-of-Fit	3	1.4325	0.4775	143.25	0.007
Pure Error	2	0.0067	0.0033		
Total	14	21.5533			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.536501	93.32%	81.30%	0.00%

Coded Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	3.567	0.310	11.51	0.000	
Curing Temperature (°C)	0.513	0.190	2.70	0.043	1.00
Cellulose Content (wt.%)	-0.738	0.190	-3.89	0.012	1.00
Curing Time (Days)	0.450	0.190	2.37	0.064	1.00
Curing Temperature (°C)*Curing Temperature (°C)	-1.671	0.279	-5.98	0.002	1.01
Cellulose Content (wt.%)*Cellulose Content (wt.%)	0.229	0.279	0.82	0.449	1.01
Curing Time (Days)*Curing Time (Days)	-0.246	0.279	-0.88	0.419	1.01
Curing Temperature (°C)*Cellulose Content (wt.%)	-0.175	0.268	-0.65	0.543	1.00
Curing Temperature (°C)*Curing Time (Days)	0.450	0.268	1.68	0.154	1.00
Cellulose Content (wt.%)*Curing Time (Days)	-0.250	0.268	-0.93	0.394	1.00

Regression Equation in Uncoded Units

Volume Expansion Ratio = -56.3 + 1.961 Curing Temperature (°C)
 + 0.32 Cellulose Content (wt.%) - 0.39 Curing Time (Days)
 - 0.01671 Curing Temperature (°C)*Curing Temperature (°C)
 + 0.102 Cellulose Content (wt.%)*Cellulose Content (wt.%)
 - 0.0615 Curing Time (Days)*Curing Time (Days)
 - 0.0117 Curing Temperature (°C)*Cellulose Content (wt.%)
 + 0.0225 Curing Temperature (°C)*Curing Time (Days)
 - 0.0833 Cellulose Content (wt.%)*Curing Time (Days)

Fits and Diagnostics for All Observations

Obs	Volume Expansion Ratio	Fit	Resid	Std Resid	
1	3.500	3.567	-0.067	-0.15	
2	2.300	1.725	0.575	2.14	R
3	1.300	1.137	0.163	0.61	
4	1.200	1.050	0.150	0.56	
5	3.400	3.550	-0.150	-0.56	
6	1.600	2.175	-0.575	-2.14	R
7	3.600	3.567	0.033	0.08	
8	3.600	3.567	0.033	0.08	
9	5.300	4.987	0.313	1.16	
10	2.300	2.612	-0.313	-1.16	
11	2.600	3.013	-0.413	-1.54	
12	2.900	3.062	-0.162	-0.61	
13	4.000	3.588	0.412	1.54	
14	1.000	1.263	-0.262	-0.98	
15	1.400	1.137	0.263	0.98	

R Large residual

Response Optimization: Volume Expansion Ratio, ... ral Strength (MPa)

Multiple Response Prediction

Variable	Setting
Curing Temperature (°C)	62.1212
Cellulose Content (wt.%)	0
Curing Time (Days)	3

Response	Fit	SE Fit	95% CI	95% PI
Volume Expansion Ratio	3.563	0.470	(2.355, 4.771)	(1.729, 5.396)
Flexural Strength (MPa)	3.595	0.149	(3.211, 3.979)	(3.012, 4.178)

Appendix E1

Average Cell Diameter – J Image

- S5 – High

	Label	Area	Mean	Min	Max	Angle	Length
1		1587.28	9.34	6.03	35.00	-89.42	394.44
2		1285.69	14.67	8.63	24.00	-79.95	319.65
3		492.06	6.54	4.17	40.00	-32.62	118.25
4		333.33	13.98	9.60	19.10	-95.71	80.08
5		793.64	8.16	4.00	31.00	20.28	195.38
6		841.26	11.36	7.14	18.00	49.64	209.15
7		666.66	8.28	5.57	13.60	-15.75	161.44
8		1174.59	12.57	6.82	27.00	-79.00	292.23
9		1190.46	52.61	9.04	245.00	-76.72	294.74
10		428.57	11.86	9.69	15.23	57.53	103.89
11		634.91	17.44	8.66	40.00	-75.26	156.55
12		841.26	7.83	6.23	9.72	14.57	205.83
13		1095.22	11.93	7.00	58.00	-90.00	270.92
14		603.17	9.97	6.06	60.00	-115.87	146.11
15		460.31	12.25	7.50	35.00	-94.09	111.84
16		761.89	10.77	8.30	15.37	19.98	186.53
17		619.04	13.51	5.63	52.00	-63.44	151.45
18		476.18	14.20	9.00	18.76	-22.17	116.15
19		587.29	6.29	3.67	10.00	-80.27	141.48
20		539.67	12.19	9.90	14.62	-77.66	130.51
21		650.78	12.03	7.53	57.00	-108.89	160.01
22		460.31	13.71	8.78	37.00	-70.91	109.62
23		492.06	14.94	10.71	52.00	-34.22	120.45
24		380.95	6.39	5.00	15.00	-46.74	93.01
25		1174.59	31.75	22.07	42.91	20.93	290.04
26		428.57	17.76	4.68	73.41	-32.47	103.89
27		555.55	8.31	6.00	13.00	-118.07	135.46
28		587.29	13.92	5.33	51.00	-70.56	143.65
29		650.78	36.44	29.11	62.01	-20.73	157.61
30		301.58	19.15	11.67	70.00	-40.60	73.46
31		1063.48	14.21	10.00	21.21	-96.91	264.87
32		777.77	4.33	3.44	7.00	-105.78	190.45
33		460.31	21.19	19.00	24.00	19.09	109.62
34		380.95	14.83	11.48	24.00	-92.49	91.72
35		428.57	14.62	8.80	17.81	-105.64	103.43
36		555.55	12.15	7.02	41.00	-42.61	135.34
37		396.82	9.83	5.00	42.00	-53.62	94.03
38		412.69	10.03	7.88	15.00	-80.91	100.87

39		428.57	12.64	9.37	23.00	-62.45	103.36
40		412.69	6.45	5.00	8.20	-53.13	99.60
41		507.93	6.49	4.20	17.00	-33.18	123.76
42		682.53	12.68	5.41	42.86	-34.44	169.08
43		523.80	21.66	8.03	71.20	-25.77	128.30
44		317.46	5.75	4.78	6.89	-32.01	75.17
45		793.64	7.96	3.00	13.00	-85.33	195.87
46		539.67	5.56	5.00	7.00	-118.89	131.96
47		428.57	20.73	16.15	44.00	-85.60	103.89
48		365.07	10.44	8.09	19.00	-84.81	88.01
49		904.75	10.62	6.61	22.00	-88.98	223.14
50		587.29	9.80	7.17	14.00	-80.54	145.41
51	Mean	641.26	13.44	8.18	34.70	-55.84	157.03
52	SD	284.34	8.28	4.73	35.71	44.39	71.47
53	Min	301.58	4.33	3.00	6.89	-118.89	73.46
54	Max	1587.28	52.61	29.11	245.00	57.53	394.44

- S14 – Medium

	Label	Area	Mean	Min	Max	Angle	Length
1		2040.15	28.47	23.10	93.00	-59.35	503.16
2		706.82	28.66	24.11	40.00	-51.55	174.01
3		1124.49	32.11	24.07	37.42	7.54	274.92
4		3036.13	24.62	18.48	58.65	-37.66	754.38
5		867.47	19.61	18.00	20.94	-19.80	212.99
6		433.73	29.82	26.77	33.65	38.66	102.66
7		594.38	33.39	27.51	43.29	-59.86	143.68
8		433.73	39.06	34.28	46.00	-46.55	104.90
9		674.70	37.33	21.20	67.84	34.08	164.52
10		449.80	26.86	25.00	32.00	-116.57	107.55
11		819.27	29.37	23.04	83.00	-67.56	199.48
12		771.08	57.57	41.43	96.00	-102.27	188.68
13		883.53	23.66	21.00	28.00	-71.57	215.47
14		273.09	28.35	25.00	33.63	-60.26	64.63
15		578.31	40.05	20.27	119.00	-103.24	139.99
16		690.76	44.21	29.27	83.32	-24.10	166.85
17		963.85	26.66	20.15	82.00	-27.00	238.40
18		706.82	96.23	22.00	245.00	95.32	173.09
19		899.60	34.91	23.94	65.00	-73.20	221.89
20		803.21	32.56	20.63	74.00	-32.38	194.59
21		963.85	29.88	20.09	63.09	-27.44	234.84
22		1140.56	41.63	23.00	76.00	-53.13	280.56
23		835.34	36.77	28.00	83.70	74.06	204.25
24		931.72	26.91	23.68	38.00	-35.66	226.91
25		883.53	50.44	32.00	72.00	-91.06	216.47
26		497.99	39.76	30.77	79.00	-46.33	121.90
27		658.63	25.24	22.00	33.00	-36.87	160.32

28		755.02	35.86	26.21	47.00	-107.65	185.06
29		915.66	35.36	25.43	44.97	-68.96	223.30
30		947.79	21.45	18.25	43.00	-81.03	231.29
31		1076.30	23.16	22.00	29.00	-14.88	265.41
32		401.61	25.02	18.14	29.60	-75.38	95.27
33		562.25	21.94	18.00	31.00	-95.04	136.80
34		497.99	39.26	24.04	164.00	-57.38	118.97
35		931.72	58.44	24.00	161.90	-92.01	228.60
36		1317.26	84.22	67.03	150.00	-60.60	326.62
37		771.08	69.00	44.18	199.00	-67.29	186.84
38		626.50	41.34	27.07	134.00	-52.52	151.51
39		546.18	52.49	43.59	87.00	-102.34	131.29
40		1429.71	24.83	18.00	128.76	52.87	351.91
41		835.34	35.23	19.00	241.75	28.07	204.41
42		771.08	30.81	20.38	171.25	28.22	186.49
43		787.15	26.23	18.57	143.45	-81.53	190.46
44		947.79	72.36	46.76	121.14	-93.95	233.02
45		706.82	54.55	22.00	169.00	-48.81	170.42
46		1156.62	69.48	52.14	168.60	73.61	284.09
47		690.76	43.06	24.07	87.00	-34.44	170.09
48		514.05	25.37	23.14	43.00	-63.44	125.47
49		1542.16	29.89	18.00	252.64	10.95	379.66
50		1493.97	70.80	37.22	255.00	-1.25	368.83
51	Mean	877.75	39.09	26.52	94.57	-39.97	214.74
52	SD	450.81	17.25	9.91	65.42	51.02	112.50
53	Min	273.09	19.61	18.00	20.94	-116.57	64.63
54	Max	3036.13	96.23	67.03	255.00	95.32	754.38

• S3 – Low

	Label	Area	Mean	Min	Max	Angle	Length
1		3278.21	26.63	0.00	51.14	15.15	805.33
2		606.47	5.93	1.13	26.00	-67.01	145.13
3		901.51	71.68	14.36	245.00	78.28	219.15
4		442.56	45.48	28.65	186.00	65.38	106.89
5	Mean	1307.18	37.43	11.03	127.03	22.95	319.12
6	SD	1327.67	27.97	13.43	105.45	65.87	327.47
7	Min	442.56	5.93	0.00	26.00	-67.01	106.89
8	Max	3278.21	71.68	28.65	245.00	78.28	805.33

Appendix E2

Cell Density (N_f) – J Image

- S5 – High

number of cells in the micrograph, $n = 163$ cells/cm

magnification factor, $M = 25x$

area of the micrograph, $A = 0.50996 \text{ cm} \times 0.38247 \text{ cm} = 0.1950 \text{ cm}^2$

$$N_f = \left(\frac{nM^2}{A} \right)^{\frac{3}{2}} = \left(\frac{163 \times 25^2}{0.1950} \right)^{\frac{3}{2}} = 3.78 \times 10^8 \text{ cells/cm}^3$$

- S14 – Medium

number of cells in the micrograph, $n = 125$ cells/cm

magnification factor, $M = 25x$

area of the micrograph, $A = 0.513026 \text{ cm} \times 0.384770 \text{ cm} = 0.1974 \text{ cm}^2$

$$N_f = \left(\frac{nM^2}{A} \right)^{\frac{3}{2}} = \left(\frac{125 \times 25^2}{0.1974} \right)^{\frac{3}{2}} = 2.48 \times 10^8 \text{ cells/cm}^3$$

- S3 – Low

number of cells in the micrograph, $n = 4$ cells/cm

magnification factor, $M = 25x$

area of the micrograph, $A = 0.518219 \text{ cm} \times 0.388664 \text{ cm} = 0.2014 \text{ cm}^2$

$$N_f = \left(\frac{nM^2}{A} \right)^{\frac{3}{2}} = \left(\frac{4 \times 25^2}{0.2014} \right)^{\frac{3}{2}} = 1.38 \times 10^6 \text{ cells/cm}^3$$