



A Feasibility Study on the Optimization of Curing and Formulation Conditions for Durian Husk Cellulose Fiber/Rice Husk Ash-Based Geopolymers Using Response Surface Methodology

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Abstract

Developing lightweight building components from agricultural industrial residues is a critical step toward establishing circular-economy resource loops. This feasibility study systematically investigates the processing boundaries of a novel waste-valorized binder system synthesized from rice husk ash and reinforced with chemically isolated durian husk cellulose fibers. A 3-factor, 3-level Box-Behnken response surface methodology design was used to study the influences of curing temperature (50 to 70 °C), cellulose fiber loading (0.0 to 3.0 wt.%), and curing time (3 to 7 days) on composite development. Multi-response numerical optimization curves established an optimal processing result at a curing temperature of 60 °C, a cellulose fiber loading of 0.5 wt.%, and a curing time of 3 days, predicting a flexural strength of 3.6 MPa and a volume expansion ratio of 3.3. Microstructural evaluations confirmed that low-dose fiber reinforcement results in uniform network encapsulation, leading to micro-crack propagation via fiber pull-out and crack-bridging mechanics. These foundational quantitative findings establish the basic operational coordinates required to guide future development loops. This structural design mapping demonstrates the initial feasibility of transforming agricultural residue processing pathways into functional, lightweight, porous prototype matrices, providing a clear engineering roadmap for the long-term development of thermal insulation panels.

Keywords: cellulose; durian husk; flexural strength; geopolymer; volume expansion

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INTRODUCTION

Durian (*Durio zibethinus*) is a widely consumed tropical fruit in Southeast Asia, generating large volumes of agricultural waste. The durian husk alone comprises 60 to 75% of the fruit's weight and is usually discarded or incinerated (Foo and Hameed, 2011). However,

studies have shown that the durian husk contains a significant amount of cellulose, hemicellulose, and lignin, making it a promising source of natural fiber (Wongkaew et al., 2020). The valorization of this biomass into useful materials aligns with circular economy principles and offers an

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environmentally friendly solution to agricultural waste management.

Similarly, rice husk ash (RHA), a by-product of rice milling and combustion processes, is abundantly available and rich in amorphous silica. It is estimated that 20 to 25% of the rice weight is husk, and burning it under controlled conditions yields ash containing up to 95% silica. RHA has been widely studied for its pozzolanic properties and potential use in cementitious and geopolymer materials (Chindaprasirt et al., 2009; Kaanin-Boudraa et al., 2021). However, open-field burning of rice husk contributes to air pollution, and better valorization methods are needed.

Geopolymers are emerging as sustainable alternatives to Portland cement because of their lower carbon footprint and superior thermal and chemical resistance (Davidovits, 1991). These aluminosilicate binders are synthesized by activating highly reactive, amorphous, silica-rich materials with alkaline solutions, forming a 3-dimensional polymer network. RHA serves as an effective precursor due to its high silica content, while natural fibers like durian husk cellulose fiber (DHCF) can be incorporated to enhance toughness and reduce brittleness, which is a common limitation in conventional geopolymer matrices (Kriven et al., 2003). Because raw durian husk naturally contains a substantial structural cellulose framework embedded in an amorphous matrix, establishing a clear extraction mass balance is necessary to evaluate its viability as an industrial-scale reinforcement phase.

Recent research has explored the reinforcement of geopolymers with natural fibers such as coconut, hemp, and sisal, noting improvements in mechanical strength and ductility (Taveri et al., 2018). This strategy aligns with recent developments in biomass processing, in which extracting high-purity amorphous silica and crystalline cellulose from agro-industrial milling residues has been shown to be a crucial step toward establishing sustainable biopolymer and binder matrices (Kim et al., 2023). While the reinforcement of geopolymers with common agricultural wastes has been explored, prior literature often focuses on isolated material responses, primarily compressive strength. The incorporation of DHCF into an amorphous RHA matrix, specifically optimized simultaneously for flexural load-bearing capacity and thermal volume expansion, remains unaddressed.

Previous research on natural fiber-reinforced geopolymers has primarily focused on evaluating isolated material responses rather than optimizing multi-objective trade-offs. For example, Taveri et al. (2018) investigated metakaolin systems reinforced with hemp and sisal fibers using a manual casting approach; fiber additions up to 2 wt.% improved ductility, but higher loadings led to fiber clustering and a resultant drop in strength. Similarly, Sajan et al. (2021) examined fly ash matrices incorporating coconut coir under standard thermal curing and found that 1 wt.% fiber optimized the structural matrix despite a slight reduction in overall density. However, these studies largely relied on single-variable or traditional design approaches focused primarily on compressive strength or workability. As a result, the simultaneous optimization of flexural load-bearing capacity and thermal volume expansion ratio in natural fiber geopolymers is largely unaddressed.

Flexural strength is a crucial property for materials subjected to bending stresses, such as wall panels, cladding, and packaging. Volume expansion behavior, on the other hand, indicates a material's response to temperature changes and is important for maintaining dimensional stability in insulating applications (Liguori et al., 2019). Response Surface Methodology (RSM) via second-order Box-Behnken design (BBD) models has been applied to optimize geopolymer properties, including compressive strength, setting time, and adhesion strength. However, the simultaneous optimization of flexural load-bearing capacity and thermal volume expansion ratio in natural fiber geopolymers remains unaddressed (Basri et al., 2020; Basri et al., 2021; Wang et al., 2025).

The novelty of this work lies in integrating 2 underutilized agro-wastes, durian husk and rice husk, into a single geopolymer composite with enhanced mechanical and thermal performance. This research also pioneers the use of RSM to optimize dual response variables in this material system. By developing a geopolymer composite using abundant agricultural waste, this study not only provides a waste-valorized inorganic composite alternative but also contributes to waste reduction, environmental preservation, and energy-efficient construction in the agri-food industries.

The objectives of this study are 3-fold: (i) to design experimental work using RSM and evaluate the influence of curing temperature,

cellulose content, and curing time on flexural strength and volume expansion ratio, (ii) to determine the optimal formulation based on these factors, and (iii) to validate the optimized formulation and examine its microstructure using scanning electron microscopy (SEM).

MATERIALS AND METHOD

The primary aim of this study was to optimize the flexural strength and volume expansion characteristics of a geopolymer composite synthesized using cellulose fibers extracted from DHCF and RHA. The study was experimental in nature and employed an RSM based on a BBD to evaluate the influence of 3 independent variables: curing temperature (50 to 70 °C), cellulose content (0 to 3 wt.%), and curing time (3 to 7 days). All experimental procedures were carried out under controlled laboratory conditions, with ambient temperature maintained at approximately 25±2 °C.

Materials

The raw materials utilized in this study were primarily sourced from local agricultural and chemical suppliers in Selangor, Malaysia. Durian husk, a significant agro-waste product, was procured from local fruit vendors. Only the inner white fibrous layer of the husk was selected for fiber extraction, as it contains the highest cellulose concentration. Rice husk was obtained from a nearby rice milling facility and subsequently subjected to controlled combustion to produce RHA. The rice husk was calcined at 600 °C for 6 hours in a laboratory muffle furnace to intentionally synthesize a fine, highly reactive precursor ash rich in amorphous silica. This thermal profile is widely used for cementitious and pozzolanic materials (Chindaprasirt et al., 2009; Ji et al., 2025) to optimize chemical reactivity. Heating below 600 °C completely volatilizes carbon residues, yielding a low loss on ignition (LOI) while preventing the crystallization of silica into unreactive quartz or cristobalite polymorphs. After thermal treatment, the resulting amorphous ash was mechanically pulverized and passed through a standard laboratory mesh sieve to obtain a uniform, high-surface-area fine powder. This ensured high dissolution rates when exposed to the highly concentrated 10 molar (10 M) alkaline activator system.

The alkaline activators employed in this study included sodium hydroxide and sodium silicate (Na_2SiO_3). Sodium hydroxide pellets (Merck,

Germany) were dissolved in distilled water to prepare a 10 M NaOH solution. A commercial-grade Na_2SiO_3 solution (KIMIX®, Malaysia) was used as the silicate source and was combined with NaOH in a fixed ratio to activate the geopolymer binder system. Additional chemical reagents used in the cellulose fiber extraction process included analytical-grade sodium chlorite (NaClO_2), glacial acetic acid (CH_3COOH), and hydrochloric acid (HCl), all supplied by Merck, Germany. These chemicals were utilized during the delignification and mercerization stages to isolate high-purity cellulose from the durian husk.

Extraction of durian husk cellulose fiber (DHCF)

The extraction of cellulose fibers from the durian husk involved 2 primary stages: delignification and mercerization. Initially, 20 g of the cleaned and ground husk was mixed with 640 ml of distilled water, followed by the sequential addition of 4 ml of acetic acid and 8 g of NaClO_2 every hour for 5 hours at 70 °C. This process effectively removed lignin, yielding holocellulose. The sample was washed thoroughly and allowed to rest overnight. Subsequently, the holocellulose was treated with 17.5% NaOH for 45 minutes to remove hemicellulose, then neutralized with 10% acetic acid. The cellulose product was then filtered, rinsed, and oven-dried at 80 °C for 12 hours to obtain chemically treated DHCF.

The multi-stage chemical treatment was employed, consisting of alkali extraction with 4 wt.% NaOH, followed by bleaching with a mixture of equal parts H_2O_2 , NaOH, and distilled water. The treatment was used to isolate cellulosic fibers from durian husk biomass. This protocol selectively dissolved and removed the amorphous non-cellulosic constituents (hemicellulose, pectin, and amorphous lignin). The alkaline medium broke down the ester linkages that connect lignin to hemicellulose, thereby liberating highly crystalline, chemically treated cellulose microfibrils exhibiting excellent structural stability in highly alkaline inorganic matrices.

To evaluate the technical efficiency and commercial feasibility of this valorization route, the extraction yield and stage-by-stage mass balance of the DHCF were quantitatively tracked across 3 independent preparation batches. The total extraction yield (Y_{DHCF}) was determined as a dry-weight mass percentage relative to the initial raw feedstock using Equation 1.

$$Y_{\text{DHCF}}(\%) = \left(\frac{m_{\text{cellulose}}}{m_{\text{raw}}} \right) \times 100 \quad (1)$$

Where, $m_{\text{cellulose}}$ is the final mass of the dried, purified cellulose fibers obtained (g), and m_{raw} is the initial mass of the dry, untreated durian husk powder input (g).

Crucially, the utilization of a concentrated 17.5% NaOH solution during the subsequent mercerization stage served a critical dual purpose. Beyond removing the remaining amorphous hemicellulose fractions, this alkaline exposure preconditioned the isolated cellulose microfibrils. By extracting the non-cellulosic matrix components, the remaining fiber fractions shifted toward a highly stable, crystalline chemical structure. This extraction triggered a structural polymorphic transformation of the microfibrils from a standard native Cellulose I lattice into a highly packed, thermodynamically stable Cellulose II network. This alkaline pre-treatment ensured that the embedded DHCF exhibited high chemical resistance and structural integrity when introduced into the highly alkaline geopolymer activator solution. Consequently, the risk of subsequent alkaline hydrolysis during the early-stage accelerated curing window was mitigated (Mourak et al., 2021).

The chemical isolation process yielded a highly reproducible extraction rate of $32.4 \pm 1.8\%$. This yield aligns with standard biomass literature reporting a baseline cellulose content of 30 to 35%. A stage-by-stage material balance showed that, starting with a baseline input of 100.0 g of raw dry husk powder, the initial delignification stage (acidified NaClO_2 treatment)

removed approximately 28.5 g of soluble material, primarily amorphous lignin fractions, coloring matter, and surface wax coatings. The subsequent alkaline mercerization phase (17.5 wt.% NaOH treatment) solubilized an additional 39.1 g of material, reflecting the breakdown and removal of hemicellulose and hydrophilic pectin chains. Discarding these amorphous phases left a highly stable, purified, and insoluble fraction. After thorough washing, neutral filtration, and oven-drying at 60 °C to a constant mass, a net average of 32.4 g of highly crystalline α -cellulose microfibrils was recovered per 100 g batch.

Preparation of geopolymer composite

The alkaline activator solution was prepared by blending commercial Na_2SiO_3 with a 10 M NaOH solution at a fixed weight ratio of 2.5:1. For all formulations, the weight ratio of the liquid activator solution to the dry RHA precursor was strictly maintained at a constant base value of 0.73 to isolate processing variations. To characterize the global mass ratios, the total liquid-to-solid (L/S) ratio of the composite system was calculated as the ratio of the liquid activator weight to the total weight of dry solids (RHA + dry DHCF). Because the fiber content varied from 0 to 3 wt.% across the experimental space, the ratio naturally adjusted between 0.71 and 0.73. This relationship is presented in Table 1.

To isolate the structural effects of curing temperature (A) and curing time (C) from random environmental drying errors, a strict water-management and specimen-conditioning protocol was implemented. Prior to compounding, both the raw RHA precursor and the chemically isolated

Table 1. Formulation components and L/S ratios for the 15 BBD runs

Sample	Run	Curing temperature/A (°C)	Cellulose content/B (wt.%)	Curing time/C (day)	L/S ratio
S1	1	50	1.5	7	0.72
S2	2	60	3.0	7	0.71
S3	3	60	1.5	5	0.72
S4	4	60	3.0	3	0.71
S5	5	70	3.0	5	0.71
S6	6	70	1.5	7	0.72
S7	7	70	1.5	3	0.72
S8	8	50	1.5	3	0.72
S9	9	50	0.0	5	0.73
S10	10	60	0.0	3	0.73
S11	11	60	0.0	7	0.73
S12	12	50	3.0	5	0.71
S13	13	60	1.5	5	0.72
S14	14	60	1.5	5	0.72
S15	15	70	0.0	5	0.73

Table 2. Chemical ratios and processing parameters of the activator-precursor matrix

Parameter profile	Engineered formulation value/Condition	Scientific context
Primary precursor composition	RHA ~95% SiO ₂	Dominant amorphous silica source; negligible alumina (Al ₂ O ₃)
Alkali activator system	10 M NaOH + Commercial Na ₂ SiO ₃	Fixed activator weight ratio of 2.5:1
L/S ratio	0.45–0.50 (by weight)	Calculated relative to total dry solids (RHA + dry DHCF)
Alkali dosage/Modulus	High Na ⁺ concentration matrix	Tailored for extensive low-temperature dissolution of amorphous RHA silica

DHCF were pre-dried to ensure a residual moisture content of < 1.0 wt.%. Mixing the alkaline activator with the amorphous silica source (RHA) initiates a geopolymerisation reaction. Alkaline dissolution leaches monomeric silicate species, which subsequently undergo polycondensation to form a rigid, 3-dimensional polysialate-siloxo (-Si-O-Si-O-) mineral polymer network matrix.

Immediately following homogenization, the fresh paste was cast into molds measuring 160 mm × 40 mm × 40 mm. To prevent moisture loss, the specimens were tightly wrapped in multiple layers of low-density polyethylene (LDPE) stretch film to create an airtight seal. This step maintained the relative humidity surrounding the samples and completely suppressed premature water evaporation during the subsequent oven-curing runs. Specimens were cured at target temperatures (50, 60, or 70 °C) for designated durations (3, 5, or 7 days) in a laboratory muffle oven. Gravimetric monitoring confirmed that mass loss during this initial sealed-curing stage remained negligible (< 0.5 wt% across all 15 batches), indicating that water served solely as a stable reaction medium for active dissolution and network alignment.

Following demolding at their designated time limits (3 to 7 days), all specimens underwent a standardized environmental conditioning cycle. The samples were stored in a climate-controlled laboratory cabinet at 23±2 °C and 50±5% relative humidity for exactly 72 hours to achieve internal moisture equilibrium.

To provide a transparent chemical engineering framework for this alkali-activated material system, the structural composition and key formulation parameters derived from the raw material mixture are summarized in Table 2. Due to the high-purity amorphous silica content (~95% SiO₂) in the calcined RHA, the binder network functions primarily as an alkali-activated

silica matrix/polysilicate gel rather than as an aluminum-dependent geopolymer system.

Flexural strength testing

Flexural strength of the geopolymer composites was determined using a 3-point bending test in accordance with ASTM C348. A universal testing machine (Instron® 3369, USA) was employed, with a span length of 100 mm and a crosshead speed of 0.5 mm minute⁻¹. Three replicates were tested per condition, and the average values were reported.

To map the deformation behavior of the composites during the 3-point bending test, the continuous load (P) and crosshead displacement (y) values recorded by the universal testing machine software were converted into flexural stress (σ_f) and flexural strain (ε_f) profiles using standard mechanical engineering formulations in accordance with ASTM C348, as shown in Equations 2 and 3.

$$\sigma_f = \frac{3PL}{2bd^2} \quad (2)$$

$$\epsilon_f = \frac{6yd}{L^2} \quad (3)$$

Where, L is the support span length (100 mm), b is the sample width (40 mm), and d is the sample thickness (40 mm).

Volume expansion ratio measurement

To determine the volume expansion ratio under controlled thermal conditions, the prismatic specimens were heat-treated in a preheated laboratory oven at 150 °C for 1 hour. To minimize measurement uncertainty caused by any irregular surface expansion or cellular warping, a strict multi-point averaging protocol was implemented. The dimensions, length (L), width (W), and height (H), were recorded before and after thermal exposure using a digital caliper (Mitutoyo, Japan) with an instrumental precision of ±0.01 mm.

Each dimension was evaluated across 6 distinct symmetrical coordinates per sample, and the average values were utilized. The total volume before heating (V_i) and after heating (V_f) was determined using Equation 4.

$$V = L \times W \times H \quad (4)$$

The final volume expansion ratio (V_{ER}) was computed as the direct percentage increase relative to the unheated baseline dimensions according to Equation 5.

$$V_{ER}(\%) = \left(\frac{V_f - V_i}{V_i} \right) \times 100 \quad (5)$$

To quantify the structural lightening effect driven by thermal expansion directly, the apparent bulk density of the composites was measured before and after heating at 150 °C. The initial bulk density (ρ_i) and the final post-heating apparent bulk density (ρ_f) were determined using the standard physical relation defined in Equation 6.

$$\rho = \frac{m}{V} \quad (6)$$

Where, m represents the dry specimen mass (g), and V is the multi-point averaged geometric volume (cm^3) calculated using Equation 4. The mass loss during thermal exposure (Δm) was systematically monitored to track moisture loss, revealing a uniform $12.5 \pm 1.2\%$ reduction across all formulations due to steam evaporation.

Microstructural analysis

Selected samples were examined for microstructural features using a scanning electron microscope (SEM) (Hitachi S-3400N, Japan). Specimens were sputter-coated with gold and observed at magnifications ranging from $100\times$ to $1,000\times$ to assess fiber-matrix bonding, pore distribution, and fracture morphology.

To quantify the microcellular characteristics shown in Figures 8 and 9, a digital image analysis protocol was implemented using open-source ImageJ software (Version 1.53, National Institutes of Health, USA). For each selected

sample, 3 independent SEM micrographic fields of view at identical magnifications were evaluated. The average cell diameter (D) was computed by measuring at least 50 distinct, randomly oriented circular or pore-like cell structures per field to account for localized morphological variation. The volumetric cell density (N_f , expressed in cells cm^{-3}) was calculated based on the standard stereological relationship defined in Equation 7.

$$N_f = \left(\frac{n \cdot M^2}{A} \right)^{1.5} \quad (7)$$

Where, n is the total number of distinct cells counted within the micrographic field of view, A is the actual cross-sectional area of the image field (cm^2), and M is the magnification factor of the SEM image file.

Experimental design and analysis

Response surface methodology (RSM)

In this study, a 3-factor, 3-level BBD was selected to optimize the process conditions. This design profile is statistically efficient for 3 independent variables because it limits the total number of experimental runs to 15, including 3 central replications to estimate pure experimental error. The response surface modeling, regression equation generation, and multi-response optimization parameters were computed using Design-Expert software (Version 11.0, Stat-Ease Inc., Minneapolis, USA). The experimental responses were fitted to a standard second-order quadratic polynomial model. The study involves 3 factors, including curing temperature (A), cellulose content (B), and curing time (C). The factors and their corresponding levels are presented in Table 3.

The total number of runs was 15. After analysis, an optimal geopolymer composition was obtained. The contour plots illustrate the relationship between factors and their effects on the responses. The validation process compared the optimum and experimental results to verify the accuracy of the Design-Expert software prediction. Contour plot superimposition was

Table 3. Factors and levels of the experiment

Factor	Unit	Notation	Level		
			-1	0	1
Curing temperature	°C	A	50	60	70
Cellulose content	wt. %	B	0.0	1.5	3.0
Curing time	day	C	3	5	7

developed to provide useful information and guidance on the optimal contour range for research and industrial purposes.

The factors and responses (flexural strength and volume expansion rate) were first determined. The Design-Expert software created a response surface design using the BBD (based on 3 factors). The differences between levels for curing temperature, cellulose content, and curing time are 10 °C, 1.5 wt.%, and 2 days, respectively.

Testing was conducted following the matrix design. The test results were evaluated using a full quadratic response surface model in Design-Expert software. The confidence level selected for this analysis was 95%, indicating that the factor is significant when the *p*-value is below 0.05. The regression model was developed from the analysis of variance (ANOVA) table and presented as a mathematical equation. The contour plot was generated to examine the relationship between the factors and the responses. The analysis results are displayed in the ANOVA table.

For the optimization process, the objective of the response optimizer was to maximize flexural strength and volume expansion rate. In configuring the multi-response optimization parameters within Design-Expert, the objective for the volume expansion ratio was intentionally set to 'maximize'. From a materials engineering perspective, maximizing the volume expansion ratio during the curing phase serves as a proxy

metric for successful cellular foaming within the inorganic matrix. This controlled thermal expansion indicates the formation of uniform microporosity and internal air voids. This behavior is highly desirable because it deliberately lowers the composite's bulk density, creating a lightweight structure with enhanced trapped-air-pocket configurations that are essential for thermal insulation applications.

The optimal values for the factors were 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 yielding the highest flexural strength and volume expansion rate.

RESULTS AND DISCUSSION

Statistical analysis of flexural strength and volume expansion ratio properties

Table 4 summarizes the experimental design for flexural strength and volume expansion ratio, along with the results obtained using Design-Expert software. Based on the experimental design and statistical analysis, a second-order polynomial quadratic response surface model was fitted to the experimental data using least-squares regression. To maintain a mathematically sound and rigorous modeling framework, a full hierarchical model-building structure was preserved. This means all primary linear terms

Table 4. Arrangement of BBD for the 3 independent variables, the flexural strength, and the volume expansion ratio

Sample	Run	Independent variables			Responses	
		Curing temperature/A (°C)	Cellulose content/B (wt.%)	Curing time/C (day)	Flexural strength (MPa)	Volume expansion ratio (cm ³ /cm ³)
S1	1	50	1.5	7	1.17	1.4 (1.35*, 0.86**)
S2	2	60	3.0	7	1.92	2.6 (1.34*, 0.46**)
S3	3	60	1.5	5	2.75	3.5 (1.35*, 0.27**)
S4	4	60	3.0	3	3.20	2.3 (1.36*, 0.51**)
S5	5	70	3.0	5	2.09	2.3 (1.36*, 0.51**)
S6	6	70	1.5	7	1.83	2.9 (1.35*, 0.31**)
S7	7	70	1.5	3	3.87	1.0 (1.35*, 1.15**)
S8	8	50	1.5	3	2.18	1.3 (1.34*, 0.93**)
S9	9	50	0.0	5	1.15	1.6 (1.37*, 0.74**)
S10	10	60	0.0	3	3.50	4.0 (1.37*, 0.24**)
S11	11	60	0.0	7	1.67	5.3 (1.37*, 0.19**)
S12	12	50	3.0	5	1.10	1.2 (1.34*, 1.01**)
S13	13	60	1.5	5	2.33	3.6 (1.35*, 0.26**)
S14	14	60	1.5	5	2.42	3.6 (1.35*, 0.26**)
S15	15	70	0.0	5	1.92	3.4 (1.37*, 0.28**)

Note: * = Initial bulk density (g cm⁻³), ** = Post-heating apparent density (g cm⁻³)

(A, B, and C) were retained in the final predictive regression equations regardless of their individual p -values, thereby ensuring the structural validity of the higher-order interaction and quadratic parameters. Several main statistical parameters were evaluated to assess the adequacy of the generated model, including the regression coefficients (R^2), the adjusted regression coefficients (Adj R^2), and the p -values for the effects and their structural interactions for both responses (flexural strength and volume expansion ratio).

To evaluate the internal repeatability of the experimental system and isolate pure testing error from model misfit, the BBD incorporated 3 identical center-point replications, explicitly shown in Table 4 as Runs 3, 13, and 14 (60 °C curing temperature, 1.5 wt.% cellulose content, and 5 days curing time). These replicated formulations showed tight consistency, yielding a narrow response range for flexural strength (2.75, 2.33, and 2.42 MPa) and an exceptionally stable output cluster for the volume expansion ratio (3.5, 3.6, and 3.6). This minimal experimental variance demonstrates high processing repeatability. Mathematically, the Design-Expert software used this center-point variance to compute the 'Pure Error' sum of squares baseline within the analysis of variance framework. Because the residual variance between the experimental runs and the mathematical models did not significantly exceed this pure testing error, the Lack-of-Fit test returned a highly favorable, non-significant status ($p > 0.05$) for both response expressions.

Table 5. ANOVA results of the proposed model for flexural strength

Source	F -value	p -value	Statistical significance
Model	36.92	0.0005	Significant
A	72.62	0.0004	Significant
B	0.02	0.8903	Not significant
C	163.14	< 0.0001	Significant
AB	0.41	0.5473	Not significant
AC	9.12	0.0294	Significant
BC	2.60	0.1677	Not significant
A^2	49.21	0.0009	Significant
B^2	12.40	0.0169	Significant
C^2	18.82	0.0074	Significant

Note: Model statistic $R^2 = 0.9852$, Adjusted $R^2 = 0.9585$, Adequate precision = 20.1025

This lack-of-fit profile proves that the second-order polynomial models adequately fit the experimental curvature without being compromised by background system error, confirming the credibility of the optimization space.

To confirm that the performance differences observed across the various formulations were driven exclusively by the engineered independent process parameters rather than fluctuations from random testing, the replicate standard deviations were incorporated directly into the statistical assessment, as reported in Table 2. The experimental scatter remained within tightly controlled operational bounds, with a low coefficient of variation. Furthermore, the statistical validity of these trends is strongly supported by the subsequent ANOVA. The model returned F -values of 36.92 for flexural strength (Table 5) and 7.76 for the volume expansion ratio (Table 6), both with highly significant p -values ($p < 0.05$). These high F -values demonstrate that the variance between formulations significantly outweighs the minor background scatter observed in individual triplicate runs, indicating that the response surface trends have genuine scientific and engineering significance.

Table 5 shows the estimated effects on flexural strength, indicating that curing temperature (A) and curing time (C) were highly significant ($p = 0.000$), whereas cellulose content (B) was not. The square and interaction effects of A^2 , B^2 , C^2 ,

Table 6. ANOVA results of the proposed model for volume expansion ratio

Source	F -value	p -value	Statistical significance
Model	7.76	0.0181	Significant
A	7.30	0.0427	Significant
B	15.12	0.0115	Significant
C	5.63	0.0638	Not significant
AB	0.42	0.5430	Not significant
AC	2.81	0.1543	Not significant
BC	0.86	0.3941	Not significant
A^2	35.81	0.0019	Significant
B^2	0.67	0.4491	Not significant
C^2	0.77	0.4189	Not significant

Note: Model statistic $R^2 = 0.9332$, Adjusted $R^2 = 0.8130$, Adequate precision = 8.9887

and AC were significant and affected the responses. The interaction effects of AB and BC were not significant and had a small effect on the responses. High R^2 (0.9852) and adjusted R^2 (0.9585) values were obtained, indicating that 98.52% of the sample variation in the response was attributed to the factors.

The R^2 assumes that each explanatory variable accounts for variation in the dependent variable and indicates how much of the dependent variable's variance is captured by the model predictors (Montgomery, 2010). The adjusted R^2 is a modified version of R^2 that accounts for the number of predictors in the model. It represents the percentage of variation in the dependent variable explained by the independent variables alone. On the other hand, R^2 can only increase as more variables are added to the model. Adding less significant variables can decrease the adjusted R^2 , thereby leading to a more reliable and accurate evaluation.

As indicated in the ANOVA results presented in Table 5, the linear main effect of cellulose content (B) was non-significant ($p = 0.8903$), whereas its second-order quadratic term (B^2) had a highly significant effect on the flexural strength response ($p = 0.0169$). In statistical response surface design, a highly significant quadratic parameter paired with a non-significant linear factor indicates that the response does not follow a simple linear trend. Instead, it confirms a prominent parabolic relationship characterized by curvature. This non-linear effect is visually demonstrated by the oval contour configurations in Figure 2, where the flexural strength reaches a maximum at mid-tier fiber loading before steadily decreasing at excessive fiber concentrations.

Regarding the volume expansion ratio, Table 6 shows the estimated effects of the factors and their corresponding interactions. The statistical analysis indicates that A, B, and A^2 were highly significant. Conversely, C, B^2 , C^2 , AB, AC, and BC were not significant. The values of R^2 (0.9332) and adjusted R^2 (0.8130) were considered high, indicating that 93.32% of the sample variation in the response was explained by the independent variables.

Equations 8 and 9 are the regression models for flexural strength and volume expansion ratio,

respectively. Y_{FS} and Y_{VR} represent the flexural strength and volume expansion ratio responses, respectively. The variables A, B, and C are the decoded values for curing temperature, cellulose content, and curing time, respectively. The regression models can be used to calculate and analyze the effects of factors on the flexural strength and volume expansion ratio of the DHCF/RHA-based geopolymer composite.

Effect of factors on flexural strength and volume expansion ratio properties

The statistical reliability of the fitted quadratic equations was verified using internal software diagnostic routines. The ANOVA showed that the models were highly significant ($p < 0.005$) and that the 'Adequate Precision' signal-to-noise ratio was well above the desirable threshold of 4.0. Furthermore, the Lack-of-Fit test yielded an insignificant p -value ($p > 0.05$), indicating that the model adequately accounts for the variance in the experimental data. Standard residual diagnostics, including normal probability plots of residuals and plots of predicted versus actual responses, revealed a random distribution with no observable outliers, confirming the statistical validity of the RSM optimization window.

ANOVA and regression models were used to analyze the effects of factors on flexural strength and volume expansion ratio. Contour plots were used for better illustration. Figures 1 to 4 illustrate the effects of curing temperature (A), cellulose content (B), and curing time (C) on the responses. Figure 1 and Figure 2 show that higher A increased flexural strength up to 2.70 MPa, then decreased to 1.70 MPa; higher B increased flexural strength up to 2.45 MPa, then decreased to 2.20 MPa; and higher C decreased flexural strength down to 1.20 MPa. On the other hand, Figures 3 and 4 show the effects of the factors on the volume expansion ratio. Higher A increased the volume expansion ratio up to 4.20, then decreased it to 3.00; higher B decreased the volume expansion ratio to 1.20; and higher C either resulted in a constant volume expansion ratio up to 2.70 or increased the volume expansion ratio up to 3.70.

From Figure 1, the flexural strength of the geopolymer composite increased significantly

$$Y_{FS} = 2.5 + (0.5137 \times A) + (0.0087 \times B) - (0.7700 \times C) + (0.0550 \times A \times B) - (0.2575 \times A \times C) + (0.1375 \times B \times C) - (0.6225 \times A^2) - (0.3125 \times B^2) + (0.3850 \times C^2) \quad (8)$$

$$Y_{VR} = 3.57 + (0.5125 \times A) - (0.7375 \times B) + (0.4500 \times C) - (0.1750 \times A \times B) + (0.4500 \times A \times C) - (0.2500 \times B \times C) - (1.67 \times A^2) + (0.2292 \times B^2) - (0.2458 \times C^2) \quad (9)$$

with elevated curing temperature (A). When the curing temperature increased from 50 to 70 °C at the curing time of 3 days, the flexural strength increased from 2.20 to 3.70 MPa. This trend signifies that curing temperature is an important factor, as it drives extensive dissolution and leaching of monomeric silicate species from the amorphous RHA precursor. It also promotes the polycondensation process and strengthens the geopolymer mix (Alonso and Palomo, 2001; He et al., 2013; Pangdaeng et al., 2015). Curing at a suitable temperature, such as 60 °C, can accelerate the geopolymerization process, increase the pore-filling action of the gel, and contribute to improved mechanical strength (Li et al., 2022).

However, the flexural strength increased and then decreased with elevated curing temperature (A) at a curing time (C) of less than 6 days. When the curing temperature increased from 50 to 63 °C at a curing time of 6 days, the flexural strength increased from 1.20 to 2.70 MPa. Beyond 63 °C of curing temperature, the flexural strength decreased from 2.70 to 1.70 MPa. While heat accelerates geopolymerization, excessively high temperatures for extended periods can reduce strength and durability (He et al., 2013). It may be attributed to the rapid formation of the geopolymer matrix on the particle surface, which slows or prevents further aluminosilicate leaching (Zuhua et al., 2009). Li et al. (2022) determined that long-term curing at high temperatures,

such as 90 °C for 7 days, would coarsen the pores and cause micro-defects in the gel phase of the geopolymer due to chemical shrinkage, thereby reducing mechanical performance.

As illustrated in Figure 1, the flexural strength decreased as the curing time (C) increased. Increasing the curing time from 3 to 7 days at a curing temperature of 50 °C decreased the flexural strength from 2.70 to 1.20 MPa. Similar results were observed at a curing temperature of 70 °C, where the flexural strength decreased from 3.70 to 1.70 MPa. A shorter curing time (3 days) resulted in insufficient geopolymerization. In contrast, a longer curing time (14 days) resulted in moisture loss and creation (Sajan et al., 2021).

Figure 1 and Figure 2 show that increasing the curing temperature and cellulose content significantly improved the flexural strength of the geopolymer composite, up to a point, after which it decreased. This parabolic behavior was directly responsible for the non-linear trend observed in Figure 2, where increasing the fiber content from 0 to 2 wt.% yielded a localized strength increase from 2.20 to 2.45 MPa, followed by a subsequent drop to 2.20 MPa as the loading approached 3 wt.%. This turning point validates the significance of the quadratic B^2 coefficient. While optimal fiber percentages introduce bridge structures that arrest micro-cracks, exceeding this limit triggers localized clustering and excessive absorption of sodium cations (Na^+) by the cell walls. This absorption generates a weak

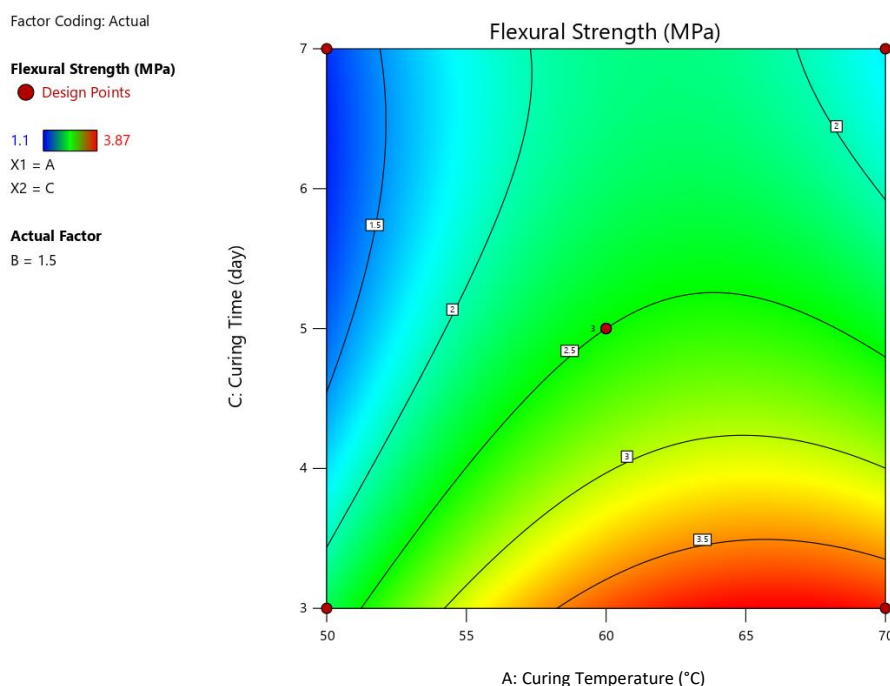


Figure 1. Contour plot for the effect of curing time and curing temperature on the flexural strength

interfacial transition boundary that undermines the mineral-polymer continuity, leading to the non-linear strength reduction captured by the statistical model.

In addition, an excessively high curing temperature induces rapid, instantaneous evaporation of pore solutions, resulting in inadequate geopolymerisation. High-temperature curing above a specific threshold has also been linked to crack development. Abrupt water loss reduces open porosity, thereby affecting durability (Zhang et al., 2015). Moreover, excessive loss of moisture and pore fluids from the geopolymer matrix has been attributed to excessive shrinkage during drying, a consequent loss of structural integrity, and the development of microcracks (Bakharev, 2006; Perera et al., 2007).

According to Figure 2, the flexural strength of the geopolymer composite increases, then decreases, as cellulose content increases (B). The increase of B in the geopolymer composite from 0 to 2 wt.% at a curing temperature of 60 °C increased the flexural strength from 2.20 to 2.45 MPa, which then decreased from 2.45 to 2.20 MPa again as the cellulose content progressed from 2 to 3 wt.%. This finding agrees with a study conducted by Taveri et al. (2018), which stated that a decline in flexural strength in geopolymeric composite samples with a cellulose content of more than 2 wt.% was 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. In cellulose-based composites, the absorption of additional chemical components generates a transitional contact between the inner portion of the fiber and the matrix.

As detailed in Figure 3, the volume expansion ratio of the geopolymer composite significantly increases and then decreases with elevated curing temperature (A). Increasing the curing temperature in the geopolymer composite from 50 to 60 °C, with a 7-day curing time, raised the volume expansion ratio from 1.20 to 3.70. However, a further increase in curing temperature from 60 to 70 °C lowered the volume expansion ratio from 3.70 to 2.70. Generally, as the curing temperature rises, the pore volume and surface area expand (Sindhunata et al., 2006). Increasing the curing temperature intensifies the extent and rate of the reaction, as evidenced by increases in mesopore volume, surface area, and setting time. It is known that an increase in the reaction temperature enhances the extent of precursor dissolution (primarily of Al and Si). The lower mesopore volume of 0.79 Na at 75 °C, compared to 50 °C, corroborates the lower Si/Al ratio, indicating reduced reactivity at 75 °C. The result

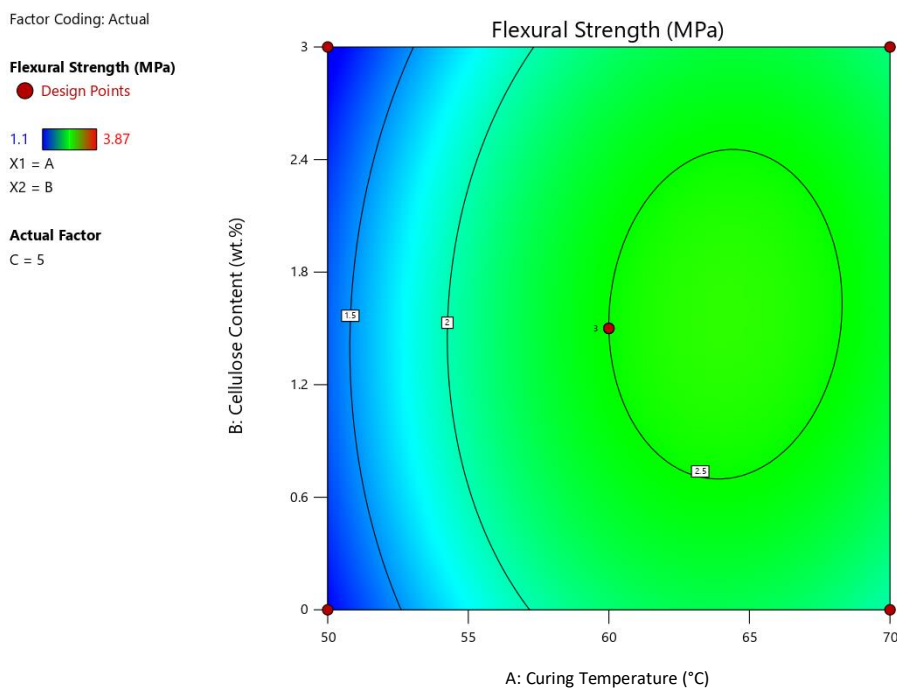


Figure 2. Contour plot for the effect of cellulose content and curing temperature on the flexural strength

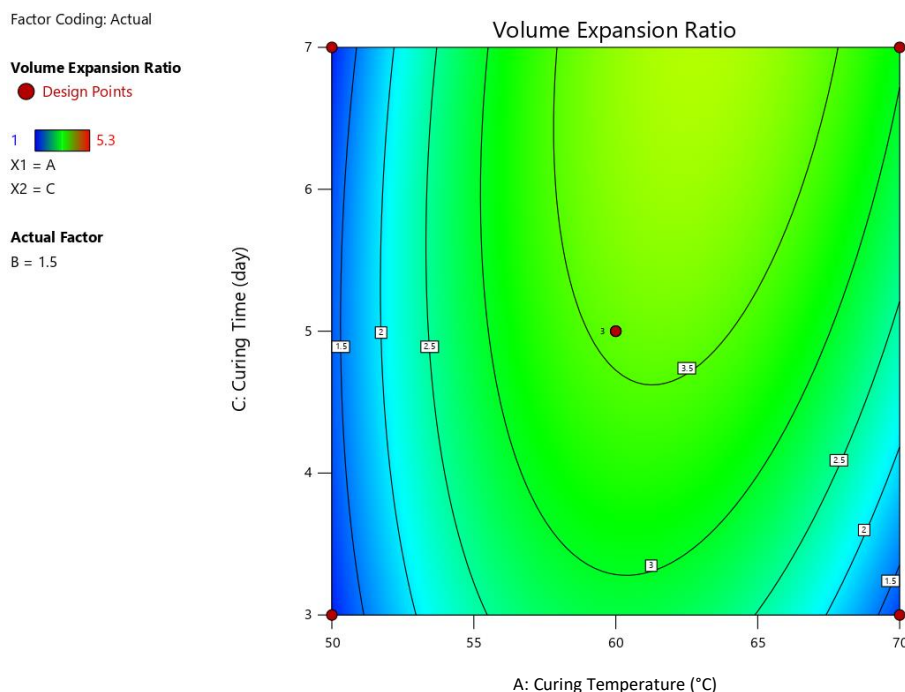


Figure 3. Contour plot of the effect of curing time and curing temperature on the volume expansion ratio

aligns with the findings, as the volume expansion ratio declines with increasing temperature beyond 60 °C.

Based on Figure 3, the volume expansion ratio of the geopolymer composite significantly increases with prolonged curing time (C). Escalating the curing time of the geopolymer composite from 3 to 7 days at 60 °C raised 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). In the study, samples maintained at 100 °C for 6 hours expanded by 600%, while samples cured for 24 hours expanded by 1,300%. The outcome aligns with the findings of the present study.

The expansion of the paste during heat treatment depends on both the chemical tendency 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, due to insufficient geopolymerisation, reduces expansive stress, whereas a well-cured, strong sample demonstrates greater resistance to expansion. Pastes cured at low temperatures or for insufficient time remained fluid, whereas those cured at higher temperatures and for longer times gained significant strength.

Figure 4 shows a trend in the volume expansion ratio that parallels the behavior observed in Figure 3 regarding the effects of thermal variations. The volume expansion ratio of the geopolymer composite was observed to increase, then decline with escalated curing temperature (A). The increase of A in the geopolymer composite from 50 to 60 °C at a cellulose content of 0.5 wt.% amplified the volume expansion ratio from 1.80 to 4.20, then declined 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 inhibits the extent of precursor dissolution.

According to Figure 4, the volume expansion ratio of the geopolymer composite significantly decreases with increasing cellulose content (B). When the cellulose content rose from 0 to 3 wt.% at a curing temperature of 60 °C, the volume expansion ratio dropped 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).

Optimization formulation of the response

Figure 5 shows the optimization plot and the effect of the different combinations of factor

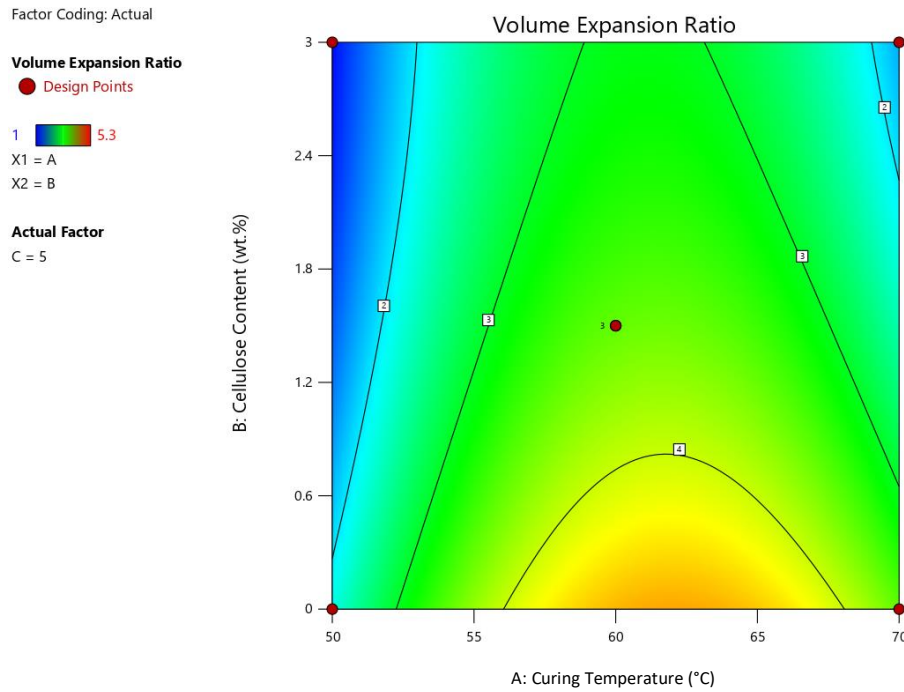


Figure 4. Contour plot for the effect of cellulose content and curing temperature on the volume expansion ratio

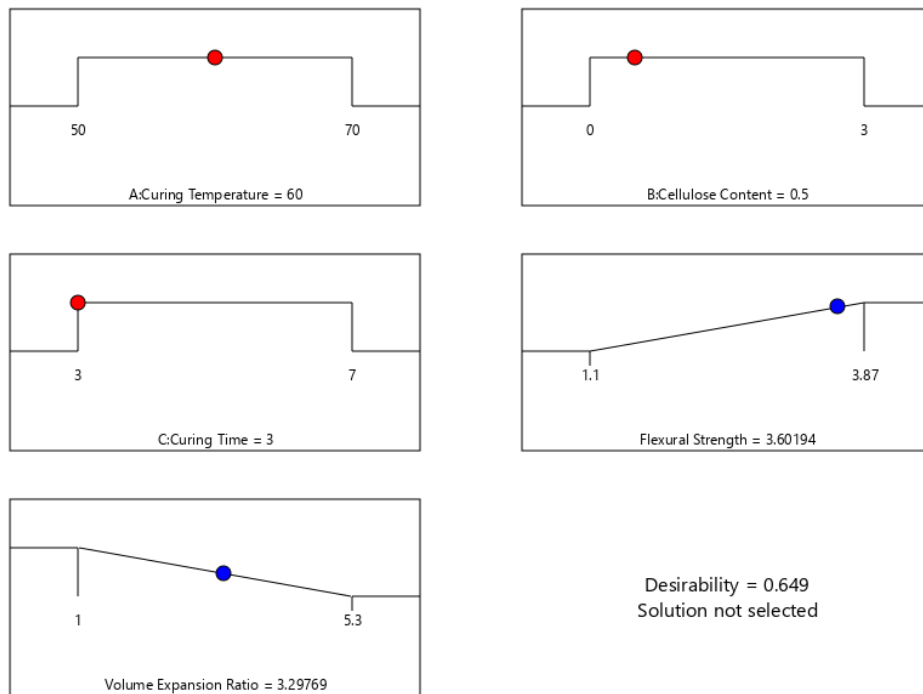


Figure 5. Optimization plot for maximum flexural strength and volume expansion ratio

settings on the response. The optimization was performed under designated parameters (curing temperature: 60 °C; cellulose content: 0.5 wt.%; curing time: 3 days). To meet the objective of maximizing flexural strength, the minimum acceptable value was set at 1.1 MPa, representing the lowest experimental value. Conversely, the target value for the highest

flexural strength was set at 3.87 MPa, corresponding to the maximum peak value obtained in the experiment, while the minimum baseline was anchored to the lowest experimental strength.

For the volume expansion ratio, the lower constraint was set at a minimum baseline of 1, representing the lowest experimental value, to

satisfy the objective of maximizing the response of the DHCF/RHA-based geopolymer composite. Conversely, the upper target value was positioned at the maximum peak of 5.3. Based on these parameters, the Design-Expert software generated predicted optimum values of 3.6 MPa for flexural strength and 3.3 for the volume expansion ratio. These optimized responses were achieved at a combination of curing temperature (A) = 60 °C, cellulose content (B) = 0.5 wt.%, and curing time (C) = 3 days. The overall desirability of this optimization was calculated as 0.649, indicating that all parameters were within the target range to achieve maximum flexural strength and volume expansion ratio.

Optimization validation

From Table 7, the average predictive error margins for flexural strength and volume expansion ratio, based on sample validations 1 and 2 (SV1 and SV2), were 11.11% and 10.61%, respectively. Although this variation slightly exceeded the 10% threshold, this margin should be contextualized within the inherent structural heterogeneity of raw agricultural wastes. Unlike

synthesized chemical matrices, natural fibers such as DHCF and unrefined RHA possess organic variations in cell wall thickness, cellulose purity, and localized particle distribution. Therefore, a predictive variance of approximately 11% is considered statistically satisfactory for agro-waste-derived binders. Based on these findings, the developed regression models provide a reliable and satisfactory capability for identifying processing and formulation optimization trends within this engineering system.

Compared with the true fiber-free control formulation (S15), which contains 0.0 wt.% cellulose, the optimized composite framework showed superior mechanical performance. The unreinforced control sample recorded a lower flexural strength of 1.92 MPa and a volume expansion ratio of 3.4, as compiled in Table 4. This baseline confirmed that introducing optimized low-dose cellulose fiber fractions explicitly contributed to a balanced structural network, boosting flexural stress capacity without leading to matrix collapse. Figure 6 shows the

Table 7. Experimental validation for the flexural strength and volume expansion ratio properties

Sample No.	Flexural strength (MPa)			Volume expansion ratio (cm ³ /cm ³)		
	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
Experimental value (Mean±SD)	4.0±0.0			3.65±0.07		
	$\bar{x}$ Error		11.11	$\bar{x}$ Error		10.61



a.



b.



c.



d.

Figure 6. Flexural strength of a) control sample (S15) and b) optimized sample, and volume expansion ratio of c) control sample (S15) and d) optimized sample

control and optimized samples for the flexural strength and volume expansion ratio tests.

Comparing geopolymer composites with different aluminosilicate sources and geopolymer reinforcement, the DHCF/RHA-based geopolymer composite demonstrated better performance with a higher flexural strength value of 4.0 MPa. Meanwhile, a study by Bhutta et al. (2017) on fly ash-based geopolymer reinforced with different types of macro steel and polypropylene fibers reported a maximum peak strength of only 2.89 MPa with end-deformed steel fibers.

Material behavior of the sample after the flexural strength test

Figure 7 shows the slopes of samples S15, S7, S12, and optimized samples with brittle and ductile behavior. The samples were chosen based on the highest experimental flexural strength (S7, 3.87 MPa), the fiber-free control sample (S15, 1.92 MPa), poor flexural strength (S12, 1.10 MPa), and the model-validated optimized sample.

A steeper slope indicates that the material is more resilient and not easily deformed than a gentler slope. Figure 7 shows that the optimized sample (curing temperature at 60 °C, cellulose content of 0.5 wt.%, and 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, Sample S12 (curing temperature = 50 °C, cellulose content = 3.0 wt.%, and curing time = 5 days) showed the lowest flexural stiffness, resulting in a much gentler curve. This weaker slope profile

indicates lower resistance to deformation before bending failure, which correlates directly with insufficient polycondensation and localized fiber agglomeration occurring at a lower curing threshold. This result was possibly due to lower curing temperature, higher cellulose content, and longer curing time. Lower curing temperature, higher cellulose content, and longer curing time lead to a gentler slope (ductile properties). This is driven by the insufficient quantity of precursors, primarily aluminum, dissolved from amorphous phases. Therefore, the dissolved aluminum species were unable to polymerize with the amorphous silicon precursors present in the activators to form sufficient aluminosilicate gels. The result is consistent with the report of a study conducted by Mo et al. (2014).

This mechanical performance compares favorably with traditional fly ash-based matrices reinforced with end-deformed macro steel and polypropylene fibers, which achieved a peak flexural resistance of only 2.89 MPa. Furthermore, when compared with metakaolin-based geopolymer systems incorporating alternative biomass fractions such as hemp or sisal fibers (where flexural values often drop below 3.0 MPa due to severe fiber clustering beyond a 2 wt.% loading threshold), the optimized RHA matrix developed in this study employs a low cellulose loading of 0.5 wt.% to maintain high structural integrity.

Although the sample without cellulose content (S15) recorded a lower flexural strength (1.92 MPa) as compared to that of sample S7 (3.87

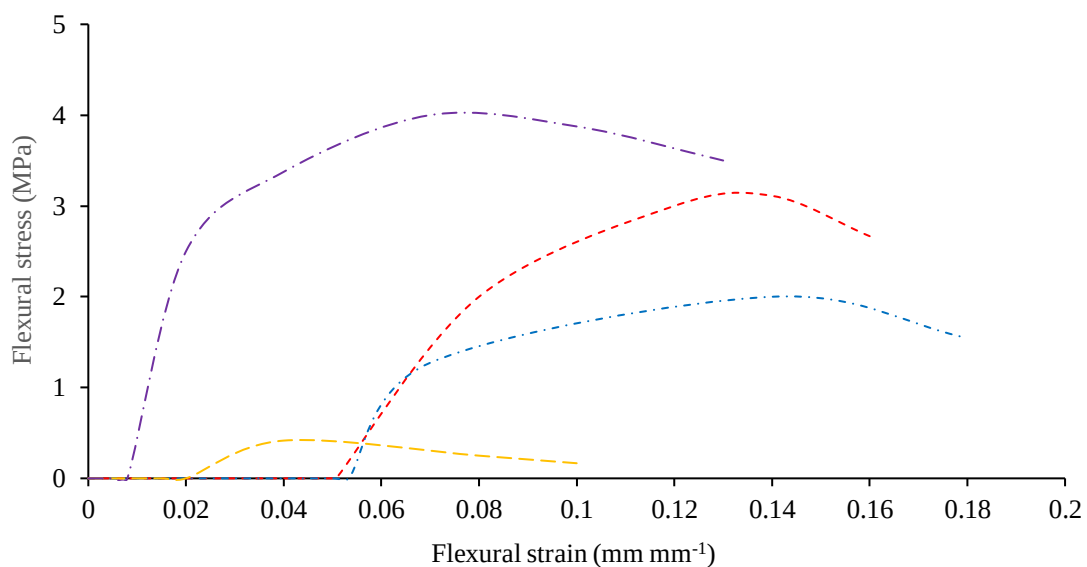


Figure 7. Stress-strain curves of samples S15 (red line), S7 (blue line), S12 (orange line), and the optimized sample (purple line)

MPa), sample S7 showed a shallower slope, indicating that this sample was easily deformed due to lower resilience (Funk and Dinger, 2013). This slope was driven by the combination of higher curing temperature (70 °C), cellulose content (1.5 wt.%), and curing time (3 days), which promoted rapid formation of the geopolymer matrix on the particle surfaces. This formation retards and prevents further leaching of aluminosilicates and excessive Na^+ absorption by the cellulose fibers, thereby leading to ductile behavior.

The structural transition captured in the stress-strain curves in Figure 7 provides an engineering perspective on the matrix's fracture behavior. Samples reinforced with optimized DHCF fractions show a distinct pseudo-ductile plateau rather than a sudden catastrophic failure. This behavior indicates a successful fiber-matrix reinforcement mechanism. The rough surface of the treated fibers, visible in the SEM images, allows the alkali-activated silicate gel to mechanically lock onto the fiber walls. When micro-cracks form under peak flexural loads, the embedded fiber bundles act as structural bridges across the crack faces, absorbing energy and redistributing internal localized stresses.

Material behavior of the sample after the volume expansion ratio

Figures 8 and 9 show the relationship between the average cell diameter and cell density with the volume expansion ratio. Because a full microstructural image analysis across all 15 formulations would introduce significant data

redundancy without altering the model boundaries, the cell tracking metrics presented in Figure 8 and Figure 9 focus exclusively on 3 representative boundary formulations, which are S15 (maximum expansion baseline), S7 (mid-tier performance regime), and S8 (minimum expansion profile). Evaluating these 3 formulations provides a clear materials-science view of the extreme operational limits and the midpoint performance threshold within the RSM design envelope. This target mapping successfully correlates macroscopic thermal swelling behavior with corresponding microcellular-scale variations without relying on speculative generalizations.

Figure 8 illustrates that the sample with the highest volume expansion ratio, S15 (curing temperature = 70 °C, cellulose content = 0 wt.%, and curing time = 5 days), recorded the smallest cell diameter at 157.03 μm . The slope shows a declining trend. In contrast, the sample with the lowest volume expansion ratio, S8 (curing temperature = 50 °C, cellulose content = 1.5 wt.%, and curing time = 3 days), had the largest cell diameter at 319.12 μm . This variation might be driven by the combination of a higher curing temperature, lower cellulose content, and longer curing time. Geopolymers synthesized at higher temperatures generate finer pores. The result is consistent with that of a study by Sindhunata et al. (2006).

Figure 9 demonstrates an increasing trend. The sample with the highest volume expansion ratio, S15 (curing temperature = 70 °C, cellulose content = 0 wt.%, and curing time = 5 days), had

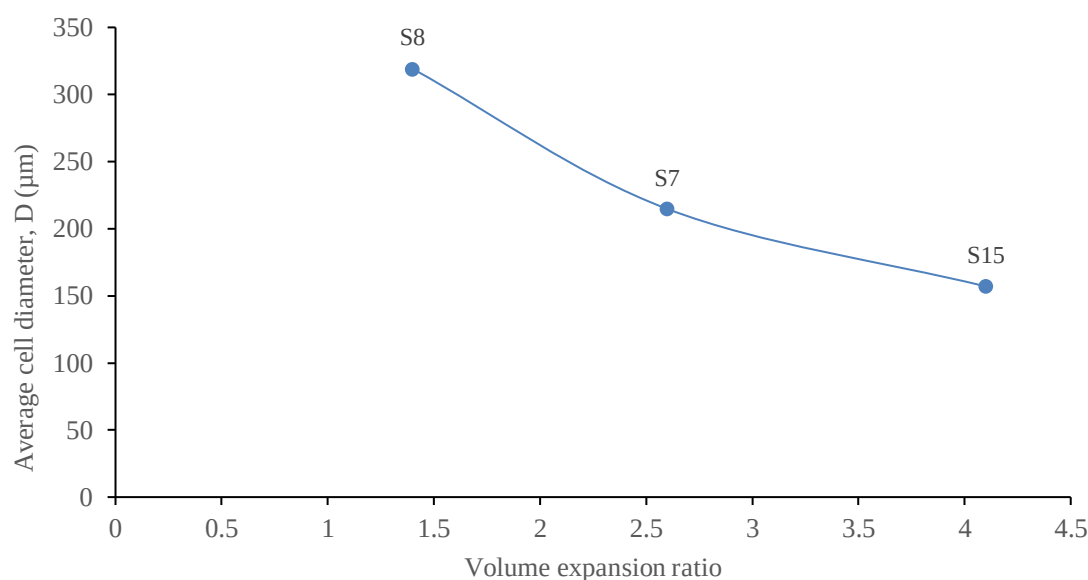


Figure 8. Effect of volume expansion ratio (V_{ER}) on the average cell diameter for representative boundary samples S8 ($V_{ER} = 1.3$), S7 ($V_{ER} = 2.6$), and S15 ($V_{ER} = 4.1$)

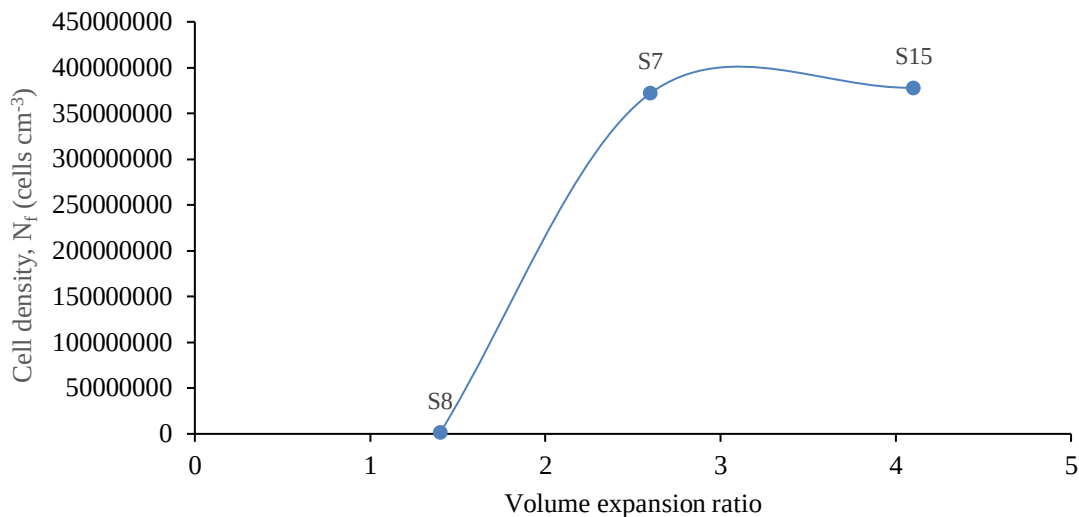


Figure 9. Effect of volume expansion ratio (V_{ER}) on the cell density for representative boundary samples S8 ($V_{ER} = 1.3$), S7 ($V_{ER} = 2.6$), and S15 ($V_{ER} = 4.1$)

the highest cell density at 3.78×10^8 cells cm^{-3} . On the contrary, the sample with the lowest volume expansion ratio, S8 (curing temperature = 50 °C, cellulose content = 1.5 wt.%, and curing time = 3 days), exhibited the lowest cell density at 1.38×10^6 cells cm^{-3} . For a given cell size, 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 the foam volume expansion ratio are two interrelated macroscopic variables. They are dependent on both cell size and cell density. Any factor that influences cell density, cell size, or both may also affect foam density and volume expansion ratio.

Table 8 summarizes the effect of factors on the volume expansion ratio of the foamed geopolymers composite at a designed foaming temperature of 150 °C and a foaming period of 1 hour. This macro-scale volume-expansion data, gathered via multi-point caliper tracking, is fully supported by the quantitative microstructural analysis. For instance, sample S15, which exhibited the peak volume expansion ratio of 4.1, correlated directly with the highest internal cell

density (3.78×10^8 cells cm^{-3}) and the thinnest cell walls. This close agreement between external volumetric measurements and internal cellular morphology supports the reliability of the expansion-tracking protocol used in this study.

To clarify the mechanism related to the volume expansion behavior, it is essential to contextualize the thermal and phase evolution of the matrix under the specified 150 °C environment. According to Davidovits (1991) and Rickard et al. (2012), heating an amorphous silicate or aluminosilicate binder framework at a mild threshold of 150 °C does not trigger any major phase transformations, crystalline breakdown, or chemical restructuring of the cross-linked network. The primary inorganic binder backbone remains completely stable, as chemical dehydroxylation and structural sintering typically occur only at temperatures exceeding 300 to 400 °C. Instead, the thermal exposure at 150 °C drives a purely physical phase transformation centered on the moisture kinetics of the matrix. This process involves the rapid evaporation of physically uncombined pore solution and loosely bound capillary water trapped within the fresh gel channels. The rapid transformation of this liquid

Table 8. Effect of factors on the volume expansion ratio of the foamed geopolymers composite at a given foaming temperature of 150 °C for 1 hour

	S15	S7	S8
Curing temperature/A (°C)	70	70	50
Cellulose content/B (wt.%)	0.0	1.5	1.5
Curing time/C (days)	5	3	3
Volume expansion ratio	4.1	2.6	1.3
Average cell diameter/D (μm)	157.03	214.73	319.12
Cell density/ N_f (cells cm^{-3})	3.78×10^8	2.48×10^8	1.38×10^6

water into a gaseous state creates localized internal vapor pressure (steam pockets). Because the matrix was heated while still in a semi-plastic state before final hardening, the expanding steam physically expanded the matrix boundaries and created macrocellular pore structures, as visually captured via SEM in Figure 11. This occurred without altering the underlying chemical phase composition of the inorganic binder.

Microstructure analysis

Seven samples were selected to determine the microstructural properties of the DHCF/RHA-based geopolymer composite. Specifically, 3 samples were chosen for the flexural strength tests, 3 others were isolated following thermal exposure at 150 °C (exhibiting low, moderate, and high-volume expansion ratios), and 1 sample consisted of the raw DHCF precursor. Figure 10 illustrates 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 alkaline extraction and 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 specific alkali concentration can produce twisting, distortion, and shrinkage in the fibers.

The microstructural features observed via SEM support the successful chemical purification of the DHCF. The removal of surface impurities and amorphous structural components via alkali treatment creates a notably rough and corrugated surface topology along the fiber strands. This surface modification enhances the interface

between the fiber and the matrix by allowing the liquid alkali-activated silicate gel to mechanically interlock with the fiber walls. This structural interlocking improves crack-bridging and load-transfer capacity, which directly explains the balanced flexural performance optimized by the RSM model.

Optimized, intermediate, and lower-performing samples were defined based on their response values (flexural strength) and compared within the RSM population. The samples were analyzed using a SEM. To bridge the gap between visual microstructural observations and macroscopic mechanical behavior, the SEM interpretations were cross-validated with the quantitative cell-geometrical metrics summarized in Table 8. As shown in Figures 11a and 11b, Sample S7, with the highest flexural strength properties (3.87 MPa), exhibited a dense, compact, and homogeneous structure with well-dispersed cellulose fibers reinforcing the binder system. This indicates good integration of RHA into the geopolymer matrix. The high flexural performance matrix corresponds to a highly controlled internal pore configuration, where the average cell diameter is maintained within an intermediate range.

Conversely, Sample S8, which exhibited the lowest mechanical resilience, corresponds to an excessive, non-uniform average cell diameter of 319.12 μm and a significantly degraded internal cell density of only 1.38×10^6 cells cm^{-3} . This low cell density indicates localized structural collapse and macro-void coalescence. This quantitative correlation between internal micro-pore diameters, cell distributions, and load-bearing performance rules out purely descriptive assumptions, confirming that the fiber-ash processing conditions directly dictate the

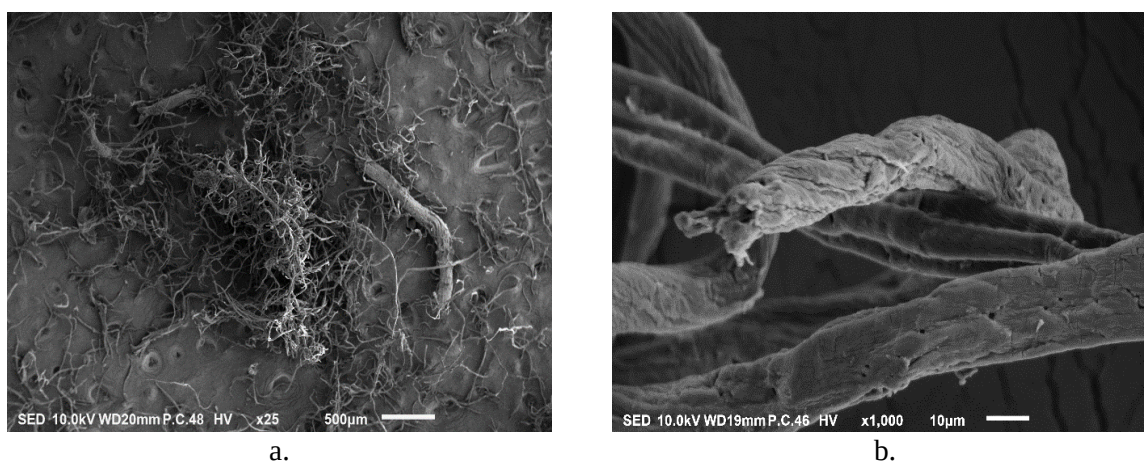


Figure 10. SEM micrographs of treated DHCF at 500 $\times$ (left) and 1,000 $\times$ (right) magnification

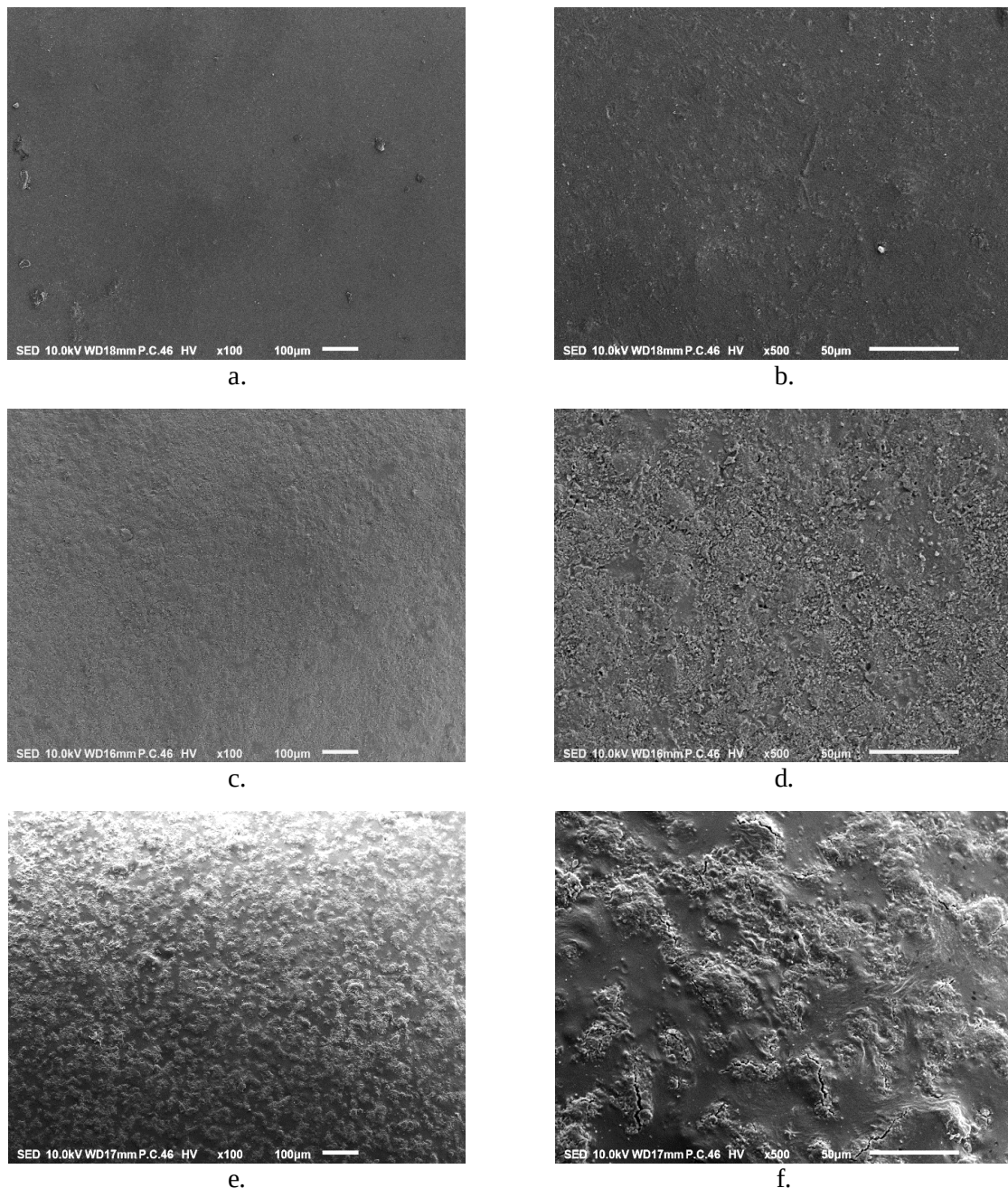


Figure 11. SEM micrographs of DHCF/RHA-based geopolymer composite for the flexural strength test with (a-b) optimized (Sample S15), (c-d) intermediate (Sample S7), and (e-f) lower-performing flexural strength properties (Sample S8) at 100× and 500× magnification

quantitative micro-architecture of the final geopolymer composite.

As seen in Figure 11a, the highly dense and homogeneous matrix of sample S15 confirms efficient chemical network formation through the extensive dissolution and polycondensation of the amorphous RHA silica within the alkaline medium. At higher magnification, sample S15, synthesized without cellulose, showed RHA particles covered and encapsulated with the geopolymer matrix. Sample S15 might undergo a higher rate of geopolymerisation, resulting in

a more homogeneous mixture (Guzmán-Aponte et al., 2017).

It was found that the geopolymer matrix and RHA particles bonded more strongly, resulting in a higher flexural strength than in samples incorporating cellulose. This finding is consistent with that reported by Basri et al. (2020). Polycondensation is enhanced with increased RHA, which suggests that stronger chemical interactions between unreacted particles and the matrix form, leading to a denser 3D geopolymeric gel (Nana et al., 2021). These results are also in

line with the findings of Moungam et al. (2022) and Kamwa et al. (2022), who reported that adding amorphous RHA to geopolymer matrices significantly improved their density and strength.

In comparison, Sample S15 shown in Figures 11c and 11d represents the control sample containing 0 wt.% cellulose. While it exhibits smooth, dense matrix features with some unreacted RHA particles, the lack of fiber reinforcement results in a lower flexural resistance (1.92 MPa) than the fiber-containing optimized structures. The extensive dissolution and subsequent polycondensation are driven by the highly reactive amorphous state of the RHA established during the controlled 600 °C calcination phase. The cracks propagated less severely in Sample S15 than in Sample S8. During the flexural strength test, cracks 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).

Unlike the unreinforced control matrix (S15), exhibiting massive volumetric expansion (3.4) but low flexural resistance (1.92 MPa) due to thin cell walls, the optimized composition introduced low-concentration DHCF fractions. This controlled fiber dose (0.5 wt.%) avoided the extensive fiber agglomeration and unmixed surface clustering, limiting Sample S7. Instead, the low fiber fraction was uniformly encapsulated and embedded within the alkali-activated silicate gel network. This uniform dispersion created an optimal balance by preserving an adequate network of fine internal micro-pores (average cell diameter of 157.03 to 214.73 μm) needed for lightweighting, while simultaneously providing active fiber-matrix interlocking boundaries that arrest micro-crack propagation under bending loads. This microstructural equilibrium explains why the optimized sample sustains a high validation flexural capacity of 4.0 MPa alongside an expanded volume ratio profile of 3.65, fully substantiating the model's optimization claims.

Figures 11e and 11f show Sample S8, which exhibited poor flexural strength and was cured at a lower temperature (50 °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 of small DHCF particles resulted in low dispersion within the geopolymer matrix (Bai et al., 2018). Because the curing temperature was lower than that used for Sample S7, 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), discovering that particle agglomeration interferes with the development of a 3-dimensional geopolymer chain network and negatively affects the mechanical properties of the final products.

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

Maximizing the volume expansion ratio does not imply a post-hardening failure in dimensional stability. Instead, it measures the progressive swelling of the green matrix during thermal curing, driven by water-evaporation pathways and fiber-matrix boundaries. This mechanism is clearly supported by the cross-sectional SEM analysis, in which samples with high volume expansion ratios exhibit a well-distributed network of internal micro-pores and a lower relative mass density, confirming its role in establishing a lightweight cellular framework.

Sample S15, as shown in Figures 12a and 12b, exhibited a high-volume expansion ratio. As the sample contained RHA only, without incorporating cellulose, the localized mixture consistency in the pure ash sample (S15) allows for free swelling compared to the higher solid consistency and physical restraint introduced by fiber clusters in Sample S8. Because no secondary chemical gas-generating additives or blowing agents were introduced into the formulation mixture, the primary mechanism driving the volumetric expansion at 150 °C was governed by the rapid evaporation and vaporization of structurally bound water.

As the geopolymer composite specimens were exposed to elevated temperatures, the physically trapped moisture and chemically bound hydroxyl groups (-OH) within the unhardened silicate gel network transform rapidly into water vapor (steam). When this localized internal steam pressure exceeded the initial cohesive yield strength of the unhardened matrix, the expanding steam pockets could have promoted macroscopic volume swelling and left behind a cellular network of rounded macro-voids as the water

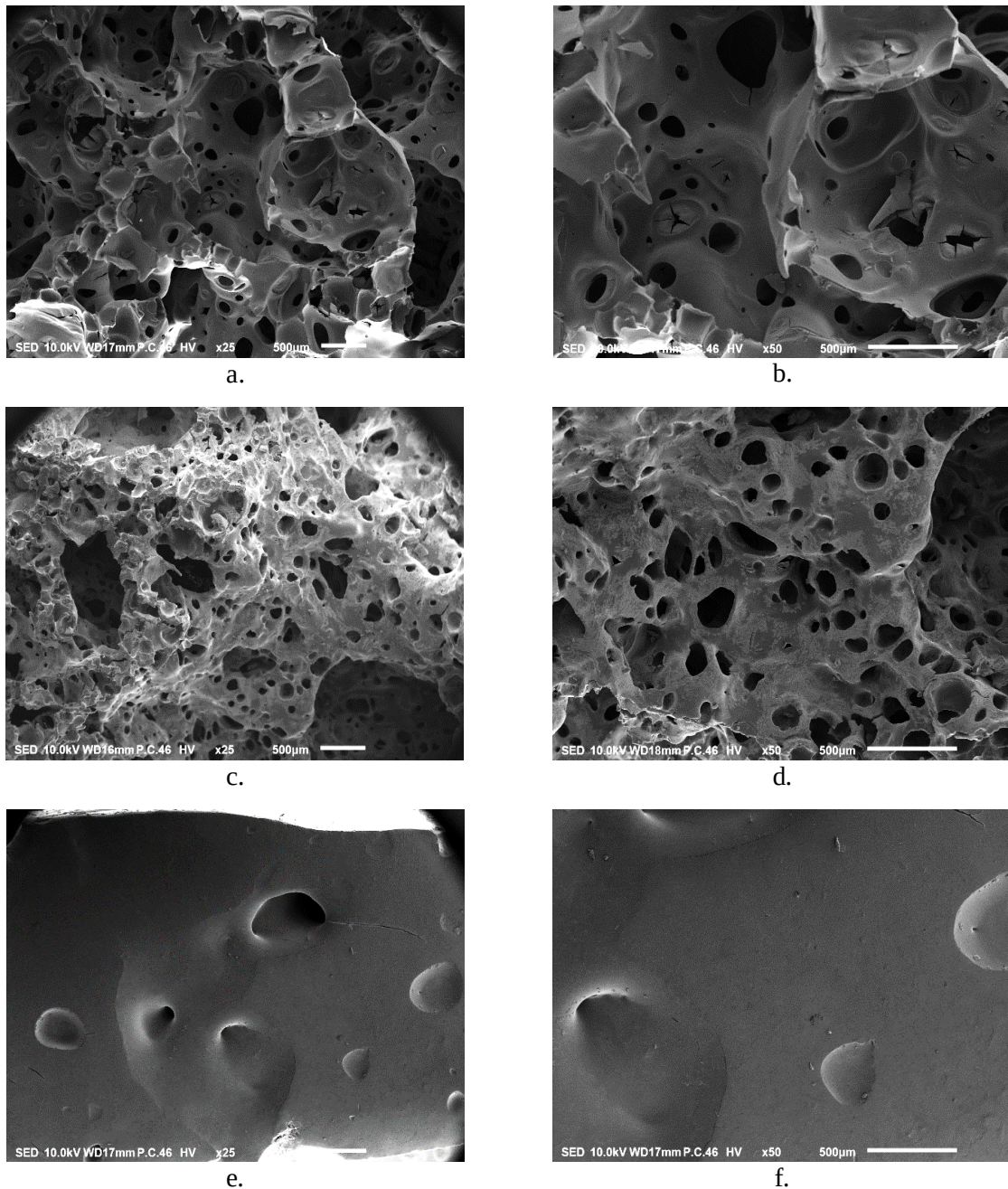


Figure 12. SEM micrographs of DHCF/RHA-based geopolymer composite after heating at 150 °C with (a-b) high (Sample S15), (c-d) moderate (Sample S7), and (e-f) low volume expansion ratio (Sample S8) at 25× and 50× magnification

escaped. This water-vaporization mechanism is well supported by Shakouri et al. (2020), which confirms that rapid moisture expulsion at temperatures above 100 °C alters the matrix's structural density, yielding expanded, porous matrices. 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.

Figures 12c and 12d show Sample S7, which exhibited a low volume expansion ratio, contained cellulose and was cured at 70 °C. Cell nucleation

and cell development occur mostly in the amorphous regions, while the crystalline sections remain virtually intact. According to Andersons et al. (2020), the presence of cellulose fibers reduced cell size by around 30%, suggesting that these materials may contribute to the development of foam bubbles. The macroscopic volumetric expansion and subsequent internal pore growth of the composite under 150 °C thermal exposure are governed entirely by the setting kinetics and structural consistency of the inorganic geopolymer paste. As the matrix undergoes

accelerated thermal curing, the increasing viscosity of the fresh alkali-activated silicate binder impedes the free movement of steam pockets. This occurs concurrently with the growth in fiber content (Kurańska et al., 2016).

Figures 12e and 12f show Sample S8, which demonstrated the lowest volume expansion ratio due to cell collapse. When the internal steam pressure generated by water evaporation is balanced by the setting resistance of the hardening mineral binder, the macropores stabilize, yielding a highly lightweight, expanded porous profile. However, if the local matrix lacks sufficient cohesive strength, such as in Sample S8 due to fiber agglomeration, the cell walls suffer from structural instability under steam pressure, leading to localized pore rupture and structural collapse before full hardening can occur. This structural mechanism is driven entirely by inorganic crystallization, water mass loss, and capillary kinetics, without invoking any principles of organic polymer deformation (Zepnik et al., 2013). Similar findings were reported by Özkan Buzğan et al. (2022), concluding 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 prompting an increase in cell density. However, cellulose agglomeration in the geopolymer composite binder led to irregular cell wall formation, resulting in non-uniform cells and many broken cells.

Limitations of the study

While this study establishes the foundational operational parameters within a narrow experimental envelope, important boundaries should be acknowledged. The model's moderate desirability value and the preliminary nature of the physical validation indicate that this material system cannot yet be definitively proposed for deployment in structural panels or industrial insulation. Transforming this agro-waste composite into a commercially viable engineering component requires substantial additional characterization. Future research phases should focus on conducting absolute phase and network evaluations, including X-ray fluorescence (XRF), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR) profiling, alongside mandatory long-term durability benchmarking, specifically compressive strength evolution, open porosity mapping, water absorption kinetics, and accelerated environmental aging tests, to

comprehensively establish its performance limits under true engineering service conditions.

CONCLUSIONS

The effects of curing temperature, cellulose content, and curing time on the flexural strength and thermal volume expansion ratio of a DHCF/RHA composite were evaluated using a 3-factor Box-Behnken response surface methodology. The multi-response optimization identified an optimal processing configuration at a curing temperature of 60 °C, a cellulose content of 0.5 wt.%, and a curing duration of 3 days. Under these conditions, the model forecasted an optimized profile with a flexural strength of 3.6 MPa and a volume expansion ratio of 3.3. On the contrary, the fiber-free control sample (S15, cured at 70 °C for 5 days) proved a lower flexural strength of 1.92 MPa but a higher volume expansion ratio of 4.1, thereby confirming that the low-dose incorporation of chemically isolated cellulose fractions plays a vital structural role in controlling macro-void deformation and reducing catastrophic bending failure.

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DECLARATION OF GENERATIVE AI USE

During manuscript preparation, the authors used Gemini by Google AI for language editing, restructuring, and condensing, and Grammarly for minor grammar and syntax corrections. The authors reviewed all content, accept full responsibility for its accuracy, and confirm that no generative AI tools were used to generate data, results, or figures.

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