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RESPONSE SURFACE MODELLING OPTIMISATION OF BAMBOO FIBRE EXTRACTION USING ULTRASONIC-ASSISTED ALKALINE BATH METHOD

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ABSTRACT

Bamboo, a versatile natural resource, holds great potential as a sustainable material due to its rapid growth, strong mechanical properties, and eco-friendly nature. Despite its abundance in Ghana and parts of Africa, its potential remains underutilised due to inefficient fibre extraction methods and limited research. Traditional methods, such as high-temperature alkaline boiling, are labour-intensive, environmentally harmful, and yield suboptimal fibres. This study addresses these challenges by optimising bamboo fibre extraction using ultrasonic-assisted alkaline treatment. Response Surface Methodology (RSM) in Design-Expert software was employed to evaluate the effects of NaOH concentration, temperature, and processing time, with statistical validation carried out using ANOVA and model fit indices ($R^2 > 0.95$). The optimal conditions, 6.078% NaOH, 20 °C, and 20 minutes, achieved a 76.768% fibre extraction efficiency with superior tensile properties. FTIR, TGA, and tensile testing confirmed the removal of non-cellulosic components, structural integrity, and enhanced thermal stability; however, excessive treatment degraded fibre quality, underscoring the need for precise process control. The findings validate ultrasonic-assisted alkaline treatment as an efficient and scalable method for producing high-quality bamboo fibres while reducing costs and environmental impact. This innovation unlocks economic opportunities for Ghana and beyond, highlighting bamboo's potential as a sustainable fibre source. Further research is recommended to address sample variability, environmental factors, and industrial scalability.

Keywords: Bamboo fibre, Ultrasonic-assisted extraction, Alkaline treatment, Optimisation, Response Surface Methodology.

INTRODUCTION

Bamboo, also known as "natural glass," is a diverse plant with around 1,600 species, including woody and herbaceous varieties, under the Bambusoideae subfamily (Piazza & Lobovikov, 2007). With about 2.8 million hectares of bamboo covering Africa (13% of global coverage), Ghana is particularly rich in bamboo species (Njuguna & Kigomo, 2008). Bamboo's rapid growth, maturing within five years under optimal conditions, along with its affordability, low density, and mechanical strength, makes it a highly sustainable raw material (Bonilla et al., 2012; Al-Kamzari & Alias, 2024). Bamboo fibres are prized for their biodegradability, eco- friendliness, high strength-to-weight ratio, hydrophobicity, and durability, which make them ideal for applications in textiles, paper, and composites (Zakikhani et al., 2014; Bacosa & Loretero, 2023).

However, challenges such as moisture susceptibility, low thermal resistance, and complex fibre extraction processes limit its full utilisation (Singh & Dessalegn, 2021). Fibre extraction is pivotal to the quality and application of bamboo fibres. Traditional methods include mechanical, chemical, and hybrid approaches. Mechanical methods, such as crushing and milling, avoid chemicals but are labour-intensive, time-consuming, and yield fibres with lower purity and weaker mechanical properties (Zhang et al., 2018). Chemical methods, like sodium hydroxide (NaOH) treatment, are effective in removing lignin and hemicellulose but are energy-intensive, environmentally harmful, and risk over-degrading fibres, leading to inconsistent quality (Guo et al., 2019; Liang et al., 2020). Hybrid methods combining chemical pre-treatment with mechanical processing often inherit the drawbacks of both methods, including high costs and environmental concerns. In this context, ultrasound-assisted alkalinetreatment has emerged as a promising alternative. This method leverages ultrasonic energy to enhance the penetration of alkaline solutions, improving the removal of lignin and hemicellulose while preserving the structural integrity of the fibres (Yao & Zhang, 2011). Compared to traditional high-temperature alkaline boiling, ultrasound-assisted extraction reduces processing time and environmental impact, making it a sustainable and scalable solution for bamboo fibre production (Mihaylova et al., 2024). The efficiency of fibre extraction processes depends on optimising key parameters such as alkaline concentration, temperature, and processing time. Traditional optimisation methods, such as One-Factor-At- A-Time (OFAT), are inefficient and fail to account for interactions between variables, often resulting in suboptimal outcomes (Myers et al., 2009). Taguchi's method, while effective in reducing variation, is less suitable for processes with complex variable interactions. Response Surface Methodology (RSM) offers a more robust alternative. This statistical and mathematical approach models the relationships between input variables and responses, efficiently optimising multiple factors and their interactions. By employing designs such as Central Composite Design (CCD) or Box-Behnken Design (BBD), RSM minimises the number of experimental runs while achieving precise optimisation (Montgomery, 2017). For bamboo fibre extraction, RSM is particularly valuable in fine-tuning critical parameters to maximise fibre yield and quality while reducing environmental impact. Despite the growing interest in bamboo fibre extraction, most studies have focused on conventional methods that are inefficient, environmentally harmful, or fail to achieve optimal fibre quality. Additionally, there is limited research on leveraging advanced optimisation methods, such as RSM, for ultrasound-assisted alkaline treatment. Existing studies often overlook the interactive effects of critical variables, which are essential for developing scalable and sustainable extraction methods. This study addresses these gaps by optimising bamboo fibre extraction through an ultrasound-assisted alkaline bath technique. Using RSM, it evaluates the effects of alkaline concentration, temperature, and processing time on fibre yield and quality. The overarching goal is to establish a sustainable, efficient, and scalable method that enhances bamboo fibre properties, reduces environmental and economic costs, and unlocks the potential of bamboo as a renewable fibre source, particularly in regions like Ghana, where bamboo is underutilised.

MATERIALS AND METHODS

Materials

Bamboo samples (Bambusa Vulgaris) were sourced from the Bamboo and Rattan Unit, Achimota Forestry Division, Ghana, during the dry season. Sodium Hydroxide (NaOH, 99% purity) was purchased from Mina Chemicals, Accra, and deionised water was used for the entire experiment.

Experimental Design

Baseline conditions for ultrasonic-assisted alkaline extraction were established using bamboo culm strips cut into smaller pieces (8 cm × 1.5 cm, 10g each), washed with distilled water, and air-dried. An ultrasonic bath (40 kHz, 150W) was used, with the following initial parameters: NaOH solutions of 2 to 10% concentration were prepared by dissolving 1 to 5g of NaOH in 50 cm^3 of water. The treatment time varied from 20 to 60 minutes, the temperature ranged from 20°C to 60°C, all with an ultrasonic power of 150W. The pre-treated bamboo culm samples were processed for maximum surface area and subjected to ultrasonic extraction under optimised conditions. Results from these preliminary experiments informed the extraction model for the study.

Ultrasound-Assisted Extraction (UAE)

The ultrasonic extraction apparatus was configured with specified parameters for all experimental procedures. For the initial run, the conditions were set to a duration of 20 minutes, a temperature of 60°C, and an alkaline solution concentration of 2% (1g of NaOH). A prepared quantity of 5g of bamboo material was placed in a suitable extraction vessel (beaker), and the extraction solvent was added to submerge the bamboo completely. This was done once the water in the ultrasonic bath reached a temperature of 60°C. The beaker containing the sample was then positioned in the bath for 20 minutes. Throughout this time, ultrasonic waves were applied to the solvent, leading to the formation and rapid collapse of cavitation bubbles. This process results in intense localised heating and agitation, aiding in the softening of lignin from the fibres. Careful monitoring of the extraction procedure was undertaken to ensure that neither the time nor the temperature exceeded the desired limits, as excessive heat could degrade sensitive compounds. After the extraction period was completed, the beaker was removed from the ultrasonic device, and the solvent was filtered to obtain the solid bamboo residue. The bamboo was then thoroughly rinsed with water to eliminate any remaining solution and dried slightly before separating the fibres. This separation was performed manually by hand-pulling the fibres apart at different levels to attain a finer consistency before they were left to dry naturally. The same procedure was replicated for all fifteen (15) runs using 150W ultrasonic power as outlined in the Central Composite Design (CCD) in Table 1.

Table 1: Experimental Design of the Extraction parameters used for the RSM model

NUMBER OF RUNS	FACTOR 1 Concentration (%)	FACTOR 2 Temperature (ᶜ)	FACTOR 3 Time (mins)
1	2	60	20
2	10	20	60
3	4	50	30
4	6	40	40
5	4	30	50
6	8	30	50
7	6	40	40
8	10	60	60
9	10	60	20
10	8	50	30
11	10	20	20
12	2	20	60
13	6	40	40
14	2	60	60
15	2	20	20

This provided a comprehensive data set for model fitting (Myers et al., 2016). Following the completion of the lignin extraction process, the samples were dried to eliminate moisture. Post- extraction, the bamboo was filtered to remove residue, thoroughly rinsed, and dried slightly before undergoing manual fibre separation. The lignin content in the extracted samples was determined using the method outlined by Das and Mohanty (2023). The raw biomass's initial dry mass was recorded before extraction, and the lignin recovery yield was calculated as a percentage of the initial biomass using "Equation 1".

mass of extracted bamboo (g)

Yield = X 100

mass of unextracted bamboo (g)

This comprehensive data set allowed for effective model fitting and optimisation of extraction conditions. The selected samples extracted for characterisation after the initial extraction process are shown in Table 2.

Table 2: Samples with Extraction Parameters

SELECTED RUNS (Samples)	CODE	FACTOR 1 Concentration (%)	FACTOR 2 Temperature (oC)	FACTOR 3 Time(mins)
Alkaline Extracted Bamboo	AEB	2	60	20
Ultrasonic Pressure Bamboo		4	50	30
Hydroxide Extracted Bamboo	OHEB	6	40	40
Lignin Extracted Bamboo	LEB	8	50	30
Chemical-Soaking Extracted Bamboo	CSEB	10	60	20

Extracted Bamboo

It is important to note that the abbreviations used in this study (AEB, UPEB, OHEB, LEB andCSEB) do not represent different extraction methods or distinct chemicals. Instead, they were only working with labels generated for convenience in identifying the various experimentalruns. In all cases, the same chemical, sodium hydroxide (NaOH), was employed as the extracting agent, with variations applied only in concentration, temperature, and treatment time.

Selection of Process Parameters

The choice of sodium hydroxide (NaOH) concentration, treatment temperature, and extraction time was based on their critical influence on the efficiency of lignin and hemicellulose removal during bamboo fibre extraction. These parameters are known to significantly affect fibre yield, fineness, tensile strength, and overall quality (Li et al., 2010; Liu et al., 2021). The experimental conditions in Table 2 were carefully selected within the practical and literature-supported ranges to examine how varying alkali strengths and thermal conditions influence fibre quality and extraction efficiency.

NaOH Concentration (2 to 10%)

The NaOH concentration range (2 to 10%) was chosen to represent varying degrees of alkalinity from mild to strong chemical action. Lower concentrations (2 to 4%) were included to evaluate minimal chemical effects that might only partially remove surface impurities and waxes while preserving cellulose integrity. Intermediate concentrations (6 to 8%) were selected to assess moderate delignification and hemicellulose removal, which have been found effective in bamboo and other lignocellulosic fibre extractions (Basu et al., 2019; Zhao et al., 2020). The highest concentration (10%) was used to determine the upper limit of effective lignin dissolution before the onset of possible fibre degradation. This broad range allowed for comprehensive analysis of fibre response to different alkaline strengths.

Temperature (40 to 60°C)

Temperature was maintained within the moderate range of 40 to 60°C to ensure effective chemical reactivity without risking cellulose thermal degradation. Previous studies (Kalia et al., 2011; Park & Kim, 2018) indicate that mild heating promotes faster alkali penetration and accelerates delignification while preserving fibre integrity. The lower temperature (40°C) was paired with a longer treatment time to allow sufficient reaction at reduced thermal energy, while higher temperatures (50 to 60°C) were combined with shorter durations to prevent over- processing. This variation ensured that both thermal and chemical contributions to the fibre extraction efficiency was adequately studied.

Treatment Time (20 to 40 min)

The treatment duration was varied between 20 and 40 minutes to balance sufficient delignification with the preservation of cellulose structure. Shorter times (20 to 30 min) were used at higher temperatures and concentrations to prevent fibre weakening due to excessive alkali exposure, whereas the longest duration (40 min) at the lowest temperature (40°C) compensated for the slower reaction rate under milder conditions. According to Singh et al. (2020), optimal extraction depends on maintaining equilibrium between reaction time andalkali intensity to achieve desirable fibre yield and quality.

Developing the Response Surface Model (RSM)

To develop the RSM model, the Design-Expert (DE) software was employed to optimise bamboo fibre extraction using ultrasonic-assisted alkaline treatment. The critical factors - time, concentration, and temperature were selected as independent variables, while fibre yield served as the response (Das & Mohanty, 2023). The RSM design procedure involves:

i. Constructing a set of experiments to ensure adequate and reliable measurement of the desired response.

ii. Formulating a mathematical model of either the first or second-order response surface with the best fit.

Identifying the optimal set of experimental conditions that yield either a maximum or minimum response value (Ahmad et al., 2023).

Creating the Experimental Design

Step 1: The experiment's goal was distinctly articulated, aiming to optimise yield by adjusting the concentration, temperature, and time.

Step 2: A new design file was created by selecting the "Response Surface" tab in the design window, followed by the choice of CCD, which is commonly used for RSM due to its suitability for model fitting.

Step 3: The factors were defined and input as concentration, temperature, and time, along with their specific units. The levels were subsequently defined as follows:

- A: Concentration (percentage): Low = 2% and High = 10%
- B: Temperature (°C): Low = 20°C and High = 60°C

C: Time (minutes): Low = 20mins and High = 60mins.

Step 4: The response variable (Yield) was specified, and one was designated as the number ofcentre points. The "Finish" button was clicked to generate the design matrix, which was then employed to create the experimental design for each run, as illustrated in Table 3, including the computed yield values.

Table 3: Generated Design Matrix.

Factor 1:		Factor 2:	Factor 3:
Run	**Concentration (%)**	**Temperature (°C)**	**Time (mins)**
1	**2**	**60**	**20**
2	**2**	**20**	**60**
3	**2**	**60**	**60**
4	**2**	**20**	**20**
5	**4**	**50**	**30**
6	**4**	**30**	**50**
7	**6**	**40**	**40**
8	**6**	**40**	**40**
9	**6**	**40**	**40**
10	**8**	**30**	**50**
11	**8**	**50**	**30**
12	**10**	**20**	**60**
13	**10**	**60**	**60**
14	**10**	**60**	**20**
15	**10**	**20**	**20**

Characterisation

This research provides an in-depth analysis of bamboo fibres obtained through Fourier-Transform Infrared Spectroscopy (FTIR), Thermal Gravimetric Analysis (TGA), Derivative Thermogravimetric Analysis (DTG), Tensile Strength Analysis (TSA), as well as assessments of elongation and Young's Modulus.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectra of bamboo fibre samples were recorded using an Alpha Platinum FTIR machine over a wavenumber range of 4000 to 400 cm^{-1} at a scan rate of 32 scans per minute, without grinding or pellet preparation. A baseline measurement was taken for accuracy. The spectra were analysed to assess the chemical composition and structural changes, including the removal of lignin and hemicellulose during NaOH treatment, with data processed using Origin software.

Thermal Gravimetric Analysis (TGA)

The Thermal Gravimetric Analyser (SDT Q600) measures mass changes in the samples under controlled heating to assess thermal stability, decomposition, and moisture content. A 10 mg sample was heated from 30°C to 800°C at 20°C/min under nitrogen gas to prevent oxidation, with air used for cooling. The TGA identifies key thermal parameters, including onset and peak temperatures, mass loss, and residual mass, while the software enables real-time monitoring and data analysis. This provides valuable insights into the material's thermal behaviour and processing requirements.

Tensile Strength Analysis (TSA)

Tensile testing of the extracted fibres was performed using a Mark-10 ESM301L strength tester to evaluate strength, modulus of elasticity, and elongation at break, which are key indicators of the fibres' strength, stiffness, and ductility. Fibres were conditioned for 24 hours at 25°C and 50% relative humidity, trimmed to 50 mm, and secured axially in the machine. The tests followed ASTM D3822, with a crosshead speed of 5 mm/min. Force and elongation were recorded until fibre failure, yielding data on breaking load, breaking elongation, stress, and strain. Each sample was tested three times, and averages were calculated for analysis and graphing.

RESULTS AND DISCUSSIONS

The extraction of bamboo fibre was conducted under varying alkaline concentrations, temperatures, and treatment durations to determine their effects on fibre quality and structural integrity. The results, illustrated in Figures 1(a) to 1(e), show that these parameters significantly influence delignification efficiency, fibre separation, and morphological refinement. Lower alkaline concentrations, such as 2% NaOH at 60 °C for 20 minutes, resulted in only moderate fibre separation, indicating partial removal of lignin and hemicellulose. Increasing the concentration to 4% at 50 °C for 30 minutes improved fibre fineness and separation due to more effective breakdown of non-cellulosic components. Higher concentrations of 6% and 8% produced cleaner and more refined fibres, reflecting enhanced exposure of the cellulose structure. However, at 10% NaOH and 60 °C, the fibres, though highly refined, showed signs of structural damage, including reduced mechanical strength, confirming the need to balance delignification and fibre preservation as noted by Malkapuram et. al. (2009).

Temperature also affected reaction kinetics and fibre morphology, with treatments between 50°C and 60 °C supporting efficient lignin removal. While higher temperatures accelerated delignification and produced smoother fibres, extreme chemical and thermal conditions increased the likelihood of fibre weakening. Overall, moderate alkaline concentrations (4 to 8%) and controlled temperatures (50 to 60 °C) were optimal for producing strong, well-refined bamboo fibres suitable for textile and composite applications.

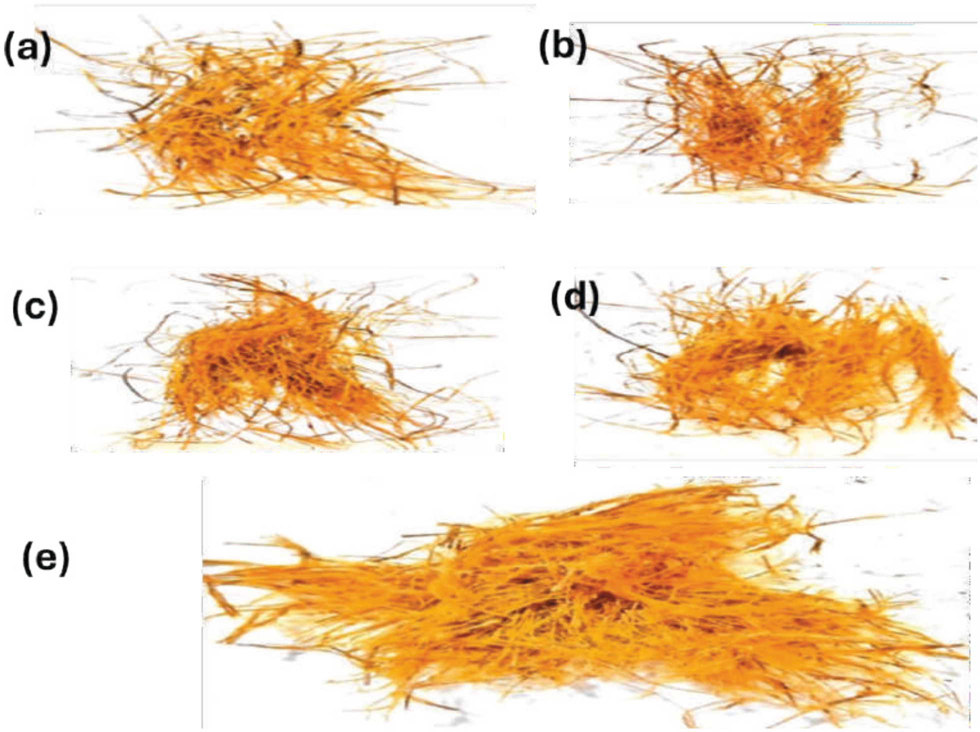

Figure 1: Samples of selected runs: (a) Alkaline Extracted Bamboo (AEB), (b) Ultrasonic Pressure Extracted Bamboo (UPEB), (c) Hydroxide Extracted Bamboo (OHEB), (d) Lignin Extracted Bamboo (LEB), (e) Chemical-Soaking Extracted Bamboo (CSEB)

The extraction of bamboo fibre was significantly influenced by alkaline concentration, temperature, and treatment duration, with each parameter affecting delignification efficiency and fibre structural integrity. Temperatures above the optimal range compromised fibre strength, as evidenced in Figure 1(e), where highly refined fibres displayed signs of weakening. This observation aligns with Zakikhani et. al. (2014) and Sarawak et. al. (2017), who identified 50 to 70 °C as the ideal temperature range for enhancing fibre properties while preventing thermal degradation. Treatment duration also played a crucial role. Shorter treatments of around 20 minutes at lower alkaline concentrations produced only moderate fibre separation, indicating insufficient removal of lignin and hemicellulose. In contrast, durations of 30–40 minutes at higher concentrations, illustrated in Figures 1(c) and 1(d), achieved optimal delignification and fibre refinement without substantial degradation. However, excessive chemical strength, such as the 10% alkaline treatment seen in Figure 1(e), caused over-processing and fibre weakening. These results corroborate findings by Yueping et. al. (2009) and Kumar (2014), who recommended moderate treatment durations for enhanced fibre purification and mechanical stability.

The most balanced and desirable outcomes were achieved under conditions shown in Figures 1(c) and 1(d), which combined 6 to

8% alkaline concentration, 50 °C temperature, and 30 to 40-minute treatment times. These conditions produced well-separated, structurally stable fibres suitable for textile and composite applications. The findings highlight the need for careful optimisation of process parameters to achieve high-quality bamboo fibres without compromising mechanical integrity.

FTIR analysis further confirmed the chemical changes occurring during ultrasonic-assisted alkaline treatment. Raw samples exhibited strong O-H absorbance, suggesting high lignin and hemicellulose content. Extracted samples showed reduced O-H absorption and increased C=C and C-O peaks, indicating successful delignification and exposure of cellulose. The low functional group retention in Figure 1(c) reflected excessive alkaline action, consistent with the observed fibre refinement patterns

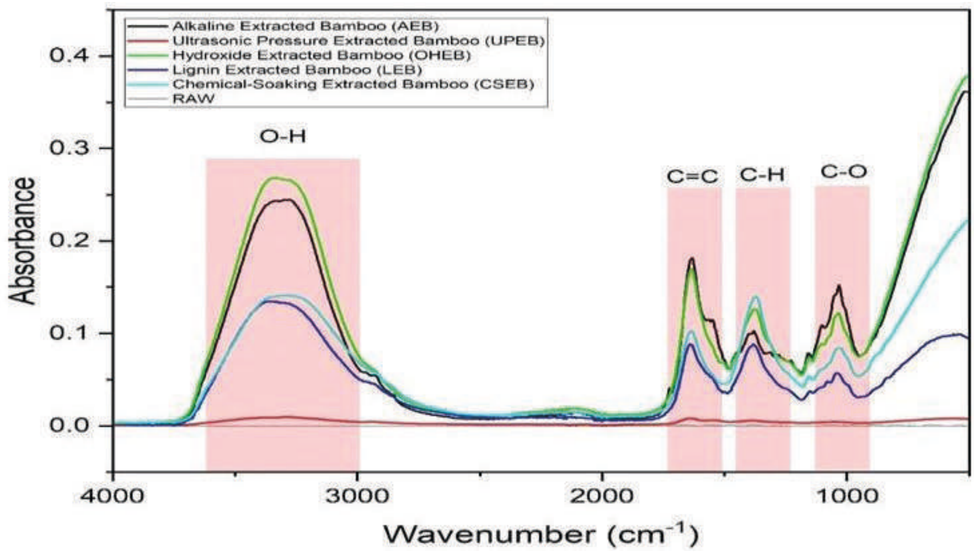

Figure 2: FTIR graphs compared with the RAW sample.

Table 4: Functional Groups Found in the Extracted Samples

Sample	O-H Region to 3600 cm⁻¹	Stretch C=C (3200 Region to 1600 cm⁻¹)	Stretch C-O(1500 Region to 1300 cm⁻¹)	Stretch C-H (1000 Region to 1500 cm⁻¹)	Bend (1300 Interpretation)
RAW	Strong absorbance	Not specified	Not specified	Not specified	High concentration of RAW Strong absorbance, hydroxyl groups, possible moisture content or an untreated sample
AEB	Present	Present	Present	Present	Comple chemical compositionwith hydroxyl, carbon-carbon double bonds, and carbon-oxygen bonds
UPEB	Present	Present	Present	Present	Similar to Sample AEB but with slightly lower absorbance, indicating fewer functional groups or lower concentrations
OHEB	Minimal absorbance	Minimal absorbance	Minimal absorbance	Minimal absorbance	Low concentrations or the absence of functional groups highlighted
LEB	Significant absorbance	Significant absorbance	Significant absorbance	Significant absorbance	Presence of hydroxyl and carbon-oxygen bonds, fewer carbon-hydrogen bonds

CSEB	Significant absorbance	Significant absorbance	Significant absorbance	Significant absorbance	Similar to Sample LEB but with slightly lower absorbance, indicating fewer functional groups or lower concentrations

The FTIR analysis and corresponding morphological observations demonstrate that alkaline concentration, temperature, and treatment duration strongly influence bamboo fibre delignification and structural integrity. The C–H bend region (1300 to 1500 cm^{-1}), clearly visible in Figures 2(AEB) and 2(UPEB), decreases significantly in Figures 2(OHEB) and 2(LEB), indicating progressive lignin removal, consistent with Malkapuram et. al. (2009). Visual fibre morphology (Figures 2(AEB) to 2(CSEB)) further supports these findings:

a 2% alkaline solution produced moderate delignification, while higher concentrations, such as 6%, resulted in substantial structural modification. However, excessively strong treatments, including the 10% concentration shown in Figure 2(CSEB), led to over-processing and weakened fibre integrity despite improved refinement.

These results emphasise the trade-off between chemical aggressiveness and mechanical preservation, confirming earlier studies that caution against extreme processing conditions. The combined FTIR trends and physical fibre characteristics validate the need for carefully optimised extraction parameters. Moderate alkaline concentrations (4 to 6%), temperatures within the 50 to 70 °C range, and treatment durations of 30 to 40 minutes consistently produced fibres with an optimal balance of cleanliness, cellulose exposure, and structural stability. These findings, supported by Ababu et. al. (2021), Zakikhani et. al. (2014), Sarawak et. al. (2017), and Kumar (2014), offer practical guidance for improving bamboo fibre quality for textile, composite, and sustainable material applications.

Fit summary response of yield

Table 5 presents the model fit summary for predicting bamboo fibre yield based on varying extraction parameters, comparing three statistical models: linear, two-factor interaction (2FI), and quadratic. The evaluation considers sequential p-values, lack-of-fit statistics, and coefficients of determination (adjusted and predicted R^2), all of which are essential for determining model suitability and predictive power.

The linear model demonstrates strong statistical performance, with a sequential p-value < 0.0001, confirming a highly significant relationship between the independent variables and fibre yield. Its adjusted R^2 of 0.8616 indicates that the model explains approximately 86% of the variation in yield, while the predicted R^2 of 0.8212 shows excellent predictive accuracy. The lack-of-fit p-value (0.2708) is not significant ($p > 0.05$), meaning there is no evidence that the model fails to describe the data adequately. Together, these metrics indicate that the linear model is both reliable and sufficiently robust for predicting yield, making it a strong candidate for final model selection. The 2FI model, which includes interaction terms among the variables, does not significantly enhance the model's explanatory power. Its sequential p-value of 0.6381 indicates that adding interaction effects does not provide meaningful improvement over the linear model. The adjusted R^2 (0.8442) and predicted R^2 (0.5430) are both lower than those of the linear model, suggesting reduced fit quality and weak predictive performance. Although its lack-of-fit p-value (0.2283) is also non-significant, the drastic decline in predicted R^2 highlights limited value in introducing interaction terms. Thus, the 2FI model does not justify its added complexity.

The quadratic model, incorporating squared terms to capture curvature, shows a sequential p- value of 0.0805. While this suggests potential improvement, it does not meet the 0.05 threshold for statistical significance. Its adjusted R^2 (0.8884) is the highest among the models, indicating excellent explanatory capability. However, the predicted R^2 (0.6698) is substantially lower, signalling possible overfitting where the model matches the training data well but may generalise poorly to new data. The non-significant lack-of-fit p-value (0.2982) shows an acceptable fit, but the model is marked as "aliased," meaning certain terms are confounded or collinear, reducing interpretability and reliability.

In conclusion, the linear model offers the best balance of simplicity, explanatory power, and predictive accuracy. Its high adjusted and predicted R^2 values, combined with a non-significant lack-of-fit statistic, affirm its suitability for modelling bamboo fibre yield. The 2FI and quadratic models introduce unnecessary complexity without improving predictability and, in the case of the quadratic model, risk overfitting and aliasing, making them less appropriate for practical use.

Table 5. Fit summary response of yield Source Sequential Lack of Fit P-value Adjusted R^2 Predicted R^2

	p-value				
Linear	< 0.0001	0.2708	0.8616	0.8212	Suggested
2FI	0.6381	0.2283	0.8442	0.5430	
Quadratic	0.0805	0.2982	0.8884	0.6698	Aliased

The developed model for predicting bamboo fibre extraction yield demonstrates strong reliability and statistical validity. The non-significant Lack of Fit Test F-value of 3.06 ($p = 0.2708 > 0.05$) indicates that the model fits the experimental data well, with no substantial unexplained error. The high coefficient of determination ($R^2 = 0.8912$) further confirms a strong correlation between the process variables alkaline concentration, temperature, and treatment time and the resulting fibre yield.

Diagnostic plots reinforce the robustness of the model. The predicted-versus-actual plot (Figure 3a) shows most data points clustering around the 45 °C line, indicating accurate model predictions. High-yield values are particularly well estimated, while only minor deviations occur at lower yields. The normal probability plot (Figure 3b) reveals that residuals follow a near-linear trend, confirming normal distribution and validating key model assumptions.

Residual analysis further supports model adequacy. The residuals-versus-predicted plot

(Figure 3c) displays random scatter around zero, indicating homoscedasticity and the absence of systematic variance. Likewise, the residuals-versus-run plot (Figure 3d) shows no trends or patterns, suggesting consistent experimental conditions and no time-related biases.

Collectively, these diagnostic results confirm that the RSM model is statistically sound, predictive, and free from significant bias or systematic error.

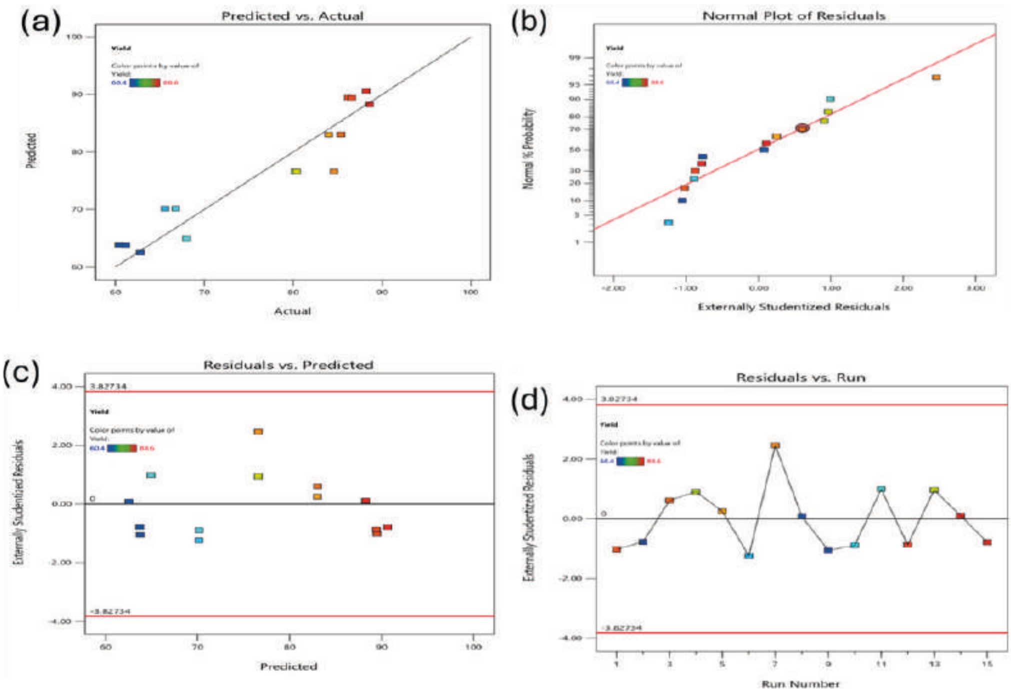

Figure 3. Model evaluation using diagnostic plots

Figure 4(a) indicates a lambda value of 1, confirming that no transformation is required and that the response variable satisfies normality and homoscedasticity assumptions. In Figure 4(b), all influence values fall below 1, with the highest at approximately 0.89, showing that no data point disproportionately affects the model, ensuring stable parameter estimates.

Figure 4(c) presents a random scatter of residuals around zero, demonstrating the absence of patterns or systematic bias and confirming that the model adequately represents the relationship between concentration and yield. Figure 4(d) shows acceptable leverage values, the highest being 0.5333, well below critical limits, indicating a well-balanced experimental design where no single run dominates the model's predictions.

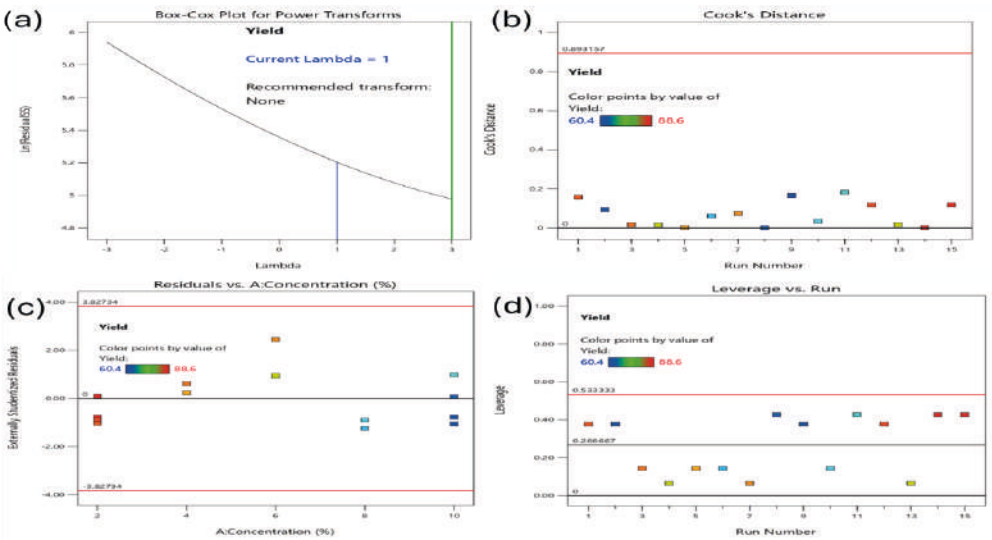

Figure 4. Diagnostic tools (a) Box-Cox Plot for Power Transforms, (b) Cook's Distance, (c) Residuals vs. A: Concentration, and (d) Leverage vs. Run. Source

Response Surface Methodology (RSM) analysis in Figure 5 reveals the interaction between concentration (%) and temperature (°C) in influencing bamboo fibre yield (%). Figure 5(a) shows that lower concentrations (around 2%) and higher temperatures (near 80°C) result in optimal yields above 80%, with temperature exerting a stronger influence than concentration.

Conversely, yields decline at lower temperatures and higher concentrations. Figure 5(b) further highlights these trends, showing maximum yields (approaching 100%) at high temperatures and low concentrations. Temperature is the dominant factor, with yields decreasing sharply as temperature drops, while concentration has a subtler effect. These findings emphasise the importance of maintaining temperatures near 80 °C and concentrations between 2% to 4% to optimise yield, offering valuable insights for improving the ultrasonic-assisted alkaline bath method.

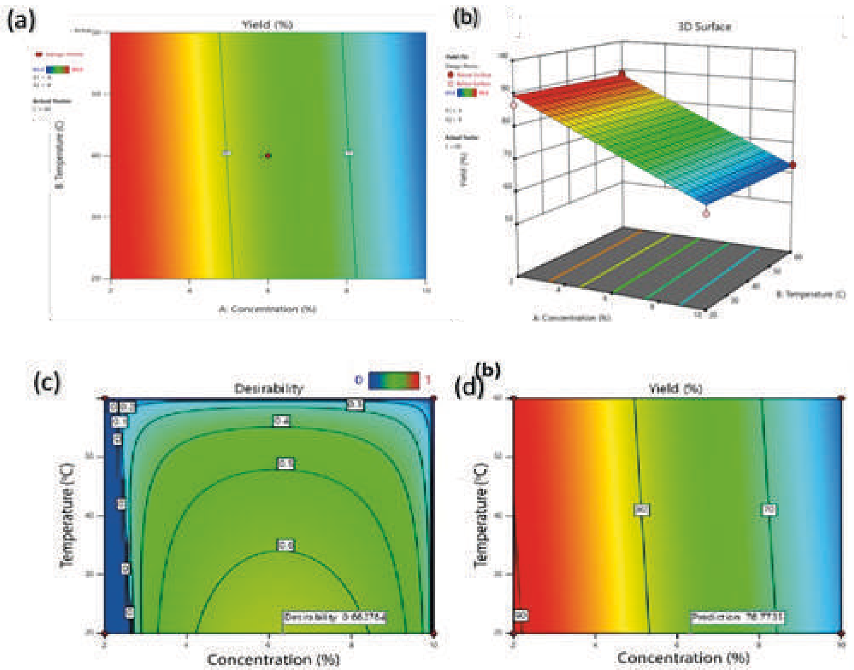

Figure 5. (a) Analysis of the 2D yield contour plot (b), Analysis of the 3D surface plot (c), Desirability Contour Plot, and (d) Yield contour Plot

The desirability contour plot identifies optimal extraction conditions with desirability values ranging from 0 (least desirable) to 1 (most desirable). The highest desirability (0.66) is observed at 50°C and 6% concentration, with lower and higher extremes reducing desirability (Zariatin, et al., 2019). The yield contour plot, however, shows maximum yields (close to 90%) at lower temperatures (20 to 30°C) and concentrations (2 to 4%), with yields declining at higher values due to fibre degradation or reduced efficiency (Myers et al., 2016).

The Comparison between Desirability and Yield in Figure 5(c) focuses on the overall desirability of the process (a composite of multiple responses), and Figure 5(d) focuses solely on yield. The optimal region for desirability (around 50°C and 6% concentration) does not exactly coincide with the region of maximum yield (around 20°C and 2% to 4% concentration). This discrepancy highlights the trade-offs between yield and other factors influencingdesirability, such as fibre quality, environmental impact, or processing costs. The results suggest that while high yields can be achieved at lower temperatures and concentrations, a balance must be struck to achieve the best overall outcome, which is captured by the desirability function. Table 6 provides experimental data for evaluating the yield and desirability of the process under varying conditions of concentration, temperature, and time.

Table 6: Model-generated optimisation results from Design-Expert showing predicted yield and desirability values within the identified optimal parameter region.

Number	Concentration	Temperature	Time	Yield	Desirability	
1	6.321	20.000	20.000	76.768	0.663	Seleced
2	6.286	20.000	20.000	76.881	0.663	
3	6.358	20.000	20.004	76.648	0.663	
4	6.421	20.000	20.000	76.447	0.663	
5	6.201	20.000	20.001	77.153	0.663	
6	6.443	20.000	20.000	76.375	0.663	
7	6.119	20.000	20.000	77.419	0.662	
8	6.323	20.151	20.000	76.759	0.662	
9	6.078	20.000	20.000	77.549	0.662	
10	6.256	20.000	20.196	76.970	0.662	
11	6.409	20.000	20.283	76.475	0.662	
12	6.683	20.000	20.000	75.605	0.661	
13	6.167	20.358	20.000	77.252	0.661	
14	6.476	20.000	20.391	76.259	0.661	
15	6.436	20.431	20.000	76.386	0.661	
16	6.297	20.000	20.490	76.832	0.661	
17	6.223	20.000	20.466	77.069	0.661	
18	6.424	20.000	20.576	76.418	0.660	
19	6.264	20.628	20.000	76.932	0.660	

20	6.174	22.433	20.000	77.170	0.653
21	7.194	20.657	20.000	73.943	0.651
22	7.337	20.000	22.410	73.430	0.640

The 22 experimental runs in Table 6 revealed the combined influence of NaOH concentration, temperature, and treatment time on bamboo fibre yield and desirability. The data established a clear performance pattern across five parameter zones.

Runs 1 to 6 represented the optimal region, with NaOH concentrations between 6.201 to 6.443%, temperature at 20 °C, and time around 20 minutes. This range produced the highest fibre yields, 76.37 to 77.15% and a consistent maximum desirability of 0.663, confirming stable and reproducible optimal conditions.

Runs 7 to 11 maintained high yields up to 77.55% but showed a slight decline in desirability, 0.662, indicating marginal deviation from the ideal balance of chemical and thermal effects.

Runs 12 to 17 showed a gradual reduction in yield, 75.6 to 77.1% and desirability 0.661 due to higher NaOH concentrations greater than 6.6% with longer treatment times greater than 20 min, suggesting the onset of cellulose weakening.

Runs 18 to 19 demonstrated a minor performance decline, yielding 76.4 to 76.9%, desirability was 0.660, as slight increases in temperature and concentration reduced cellulose preservation efficiency.

Finally, runs 20 to 22 exceeded the safe operating range, NaOH greater than 6.7%, temperature greater than 22 °C, time greater than 22 min, leading to significantly lower yields, 73.43 to 77.17% and desirability values 0.640 to 0.653 due to fibre damage from excessive alkali exposure. Overall, the results confirm that optimal bamboo fibre extraction occurs at approximately 6.3% NaOH, 20 °C, and 20 minutes, beyond which both yield and fibre quality decline. This range defines the most stable and efficient operating conditions for sustainable fibre processing.

Optimisation of Experimental Parameters for Maximum Fibre Yield and Desirability

The optimisation of experimental parameters for maximum yield and desirability, as presented in Table 6, demonstrates the critical influence of concentration, temperature, and time on bamboo fibre extraction. The optimisation of bamboo fibre extraction parameters revealed that sodium hydroxide (NaOH) concentration, temperature, and treatment time significantly influence fibre yield and overall process desirability. Experimental results showed NaOH concentrations ranging from 6.078% to 7.337%, temperatures between 20 to 22.433 °C, and treatment durations of 20 to 22.410 minutes. Fibre yield varied from 73.430% to 77.549%, while desirability indices ranged from 0.640 to 0.663.

The results indicate that optimal fibre yield and desirability occur at moderate alkali concentrations, 6.1 to 6.5% NaOH, with the best performance achieved around 6.321 to 6.443% NaOH, 20 °C, and 20 minutes. The highest yield, 77.549% was observed at 6.078% NaOH, though its desirability, 0.662, was slightly below the global maximum, 0.663.

Increasing NaOH concentration beyond 6.5% or temperature above 20 °C led to reduced yield and desirability, likely due to cellulose degradation and alkali-induced fibre damage.

Statistical analysis confirmed this trend, runs with NaOH less than or equal to 6.5% recorded a higher mean yield of 76.83% and desirability 0.661 than those above 6.5% (74.33%, 0.651). Similarly, experiments conducted at 20 °C and 20 minutes showed superior performance compared to slightly higher temperatures or longer treatment times. From both empirical and statistical evidence, run 1 (6.321% NaOH, 20 °C, 20 min) was selected as the optimalcondition, yielding 76.768% fibre with a desirability of 0.663. This run represented the most stable and reproducible performance across all experiments.

The scientific rationale for these findings suggests that moderate alkali concentration efficiently removes lignin and hemicellulose without compromising cellulose integrity. Higher NaOH levels or extended exposure times promote cellulose hydrolysis, leading to lower yields. Controlled temperature (20 °C) minimises thermal degradation, preserving fibre structure and quality. Thus, the recommended optimum extraction conditions are 6.1 to 6.5% NaOH concentration (6.3%), 20 °C temperature, and 20 minutes treatment time. These parameters maximise fibre yield, process stability, and overall desirability. Future work should include replicate confirmation under these conditions, fine-tuning within the narrow optimal range, and comprehensive quality assessment of the extracted fibres to ensure structural and mechanical integrity.

Comparative analysis of the optimal sample with the other extracted samples

The FTIR analysis (Figure 6(a) demonstrates the effectiveness of ultrasonic-assisted alkaline extraction in optimising bamboo fibre properties. Peaks observed between 1500 and 1650 cm^{-1} indicate partial lignin removal, which preserves fibre flexibility and strength (Khalil et al.,2012).

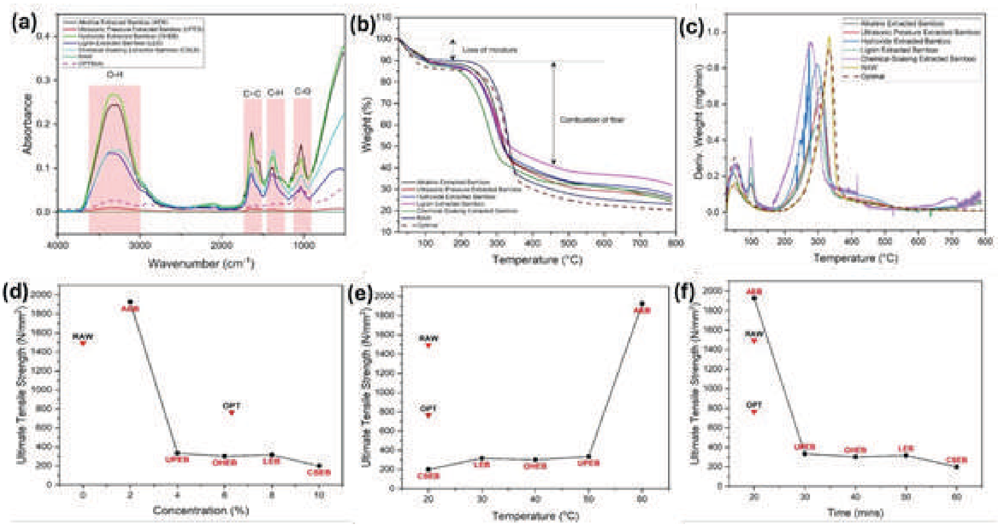

Figure 6. (a) FTIR graph, (b) TGA graph, (c) DTG graph, (d) UTS concentration graph, (e) UTS temperature graph, and (f) UTS time graph compared with the optimal sample.

Strong C-O stretching at 1000 to 1200 cm^{-1} confirms cellulose preservation, essential for textile applications (Ahmad et al., 2023), while moderate C-H stretching (2800–3000 cm^{-1}) indicates minimal degradation of aliphatic structures, enhancing durability (Tang et al., 2022). The O-H peak at 3300 cm^{-1} reflects improved hydrophilicity, facilitating dyeing and moisture management (Singh & Choudhary, 2016). Reduced C=C bonds around 1600 cm^{-1} enhance oxidative and UV stability and promote uniform dye uptake. Compared to raw bamboo, the optimised process balances lignin retention and cellulose exposure, improving mechanical and functional properties.

Thermogravimetric analysis (TGA, Figure 6(b) reveals that bamboo samples exhibit weight loss primarily between 200 and 400°C. The AEB sample shows moderate thermal stability, stabilising with 4 to 45% residual mass at 800°C. UPEB and OHEB retain higher mass up to 400°C, indicating preservation of thermally stable components (Huda et al., 2008). LEB and CSEB degrade more rapidly, with LEB starting at 150 to 200°C, highlighting lignin'scontribution to thermal resistance. The OPT sample demonstrates gradual weight loss, reflecting a balanced extraction approach (Karimah et al., 2021).

DTG curves (Figure 6(c) indicate degradation stages: hemicellulose (200 to 300°C), cellulose (300 to 400°C), and lignin (400 to 600°C) (Lavoine et al., 2012). AEB shows sharp decomposition around 300 to 350°C, whereas UPEB peaks at 320 to 360°C, reflecting better fibre preservation. OHEB displays broad peaks, LEB shows early lignin removal, and CSEB exhibits multiple degradation peaks. OPT samples exhibit smooth decomposition, indicating optimised thermal stability.

Mechanical performance analysis demonstrates the impact of concentration, temperature, and time on tensile properties. Figure 6(d) shows that UTS declines with increasing additive concentration; AEB achieves the highest UTS (1900 N/mm²) at low concentrations, while CSEB reaches 200 N/mm² at 10%. Low concentrations enhance UTS through solid solution strengthening or precipitate formation (Karimah et al., 2021). Temperature effects (Figure 6(e) and 9(b) reveal minimal UTS improvement for CSEB, LEB, and OHEB at 20 to 40°C, but AEB at 60°C shows a sharp rise to 1900 N/mm², suggesting improved fibre alignment and removal of non-cellulosic materials (Ismail & Khalil, 2000). Time-dependent studies (Figure 6(f) indicate peak UTS for AEB at 10 to 15 minutes, with a decline after 20 minutes due to degradation processes like creep and relaxation (Yao & Zhang, 2011). RAW and OPT fibres show relatively stable UTS, highlighting lower sensitivity to prolonged exposure.

Stress elongation analysis (Figure 7(a) underscores the trade-off between strength and flexibility. AEB exhibits the highest tensile stress (1800 N/mm²) but limited elongation (1.5 mm), whereas RAW bamboo balances tensile stress (1500 N/mm²) and elongation (2 mm). OPT fibres demonstrate moderate strength (1300 N/mm²) with the highest elongation (3 mm), indicating optimal performance for versatile applications. CSEB and OHEB exhibit both lower stress and elongation, emphasising the detrimental effects of certain chemical treatments on mechanical properties (Camarena-Martinez et al., 2023).

Overall, the analyses confirm that ultrasonic-assisted alkaline extraction, under optimised conditions of moderate concentration, controlled temperature, and appropriate treatment duration, effectively enhances bamboo fibre properties. The process achieves a balance between lignin removal and cellulose preservation, ensuring thermal stability, improved mechanical strength, and suitable ductility for diverse applications. These findings provide critical insights for tailoring bamboo fibre extraction for textile, composite, and industrial uses, supporting the development of high-performance, durable, and versatile bamboo-based materials.

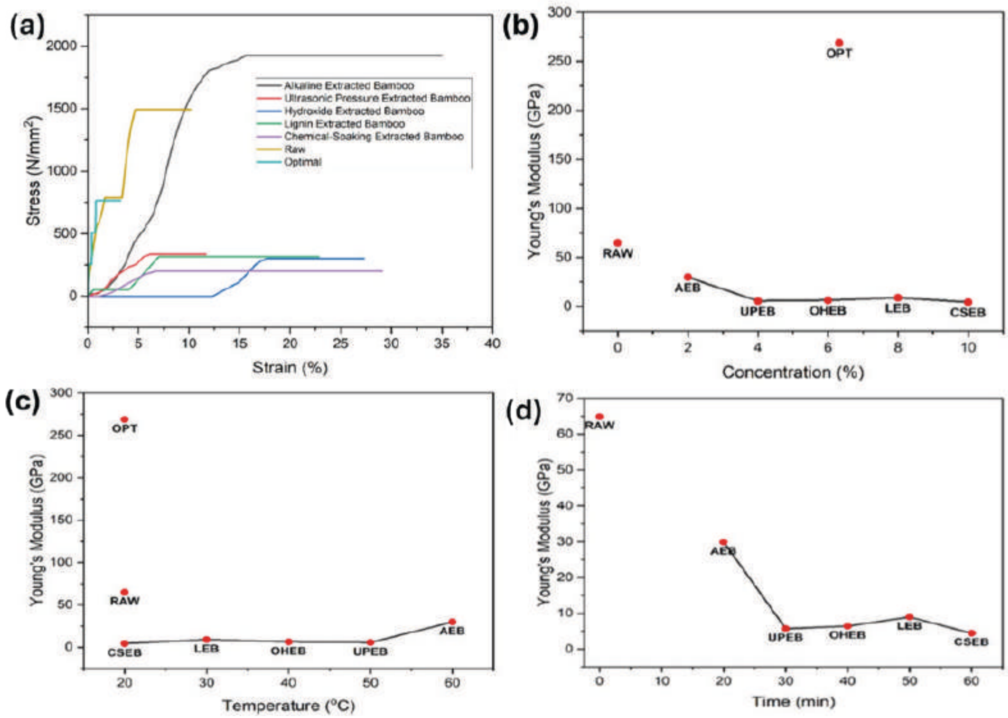

Figure 7 (a) Tensile strength against strain (%) graph, (b) Young's modulus – concentration,

(c) Young's Modulus –Temperature and (d) Young's Modulus –time compared with the optimal sample The results show that Young's Modulus is highest under optimal extraction conditions (OPT), reaching 250 GPa, compared to 50 GPa for RAW bamboo. As alkaline concentration increases from 2% to 10%, the modulus decreases sharply to around 10 GPa, with a major drop at 4%. This decline is attributed to the degradation of cellulose crystallinity at higher concentrations, indicating that excessive chemical exposure weakens fibre stiffness. OPT conditions achieve superior performance because they balance lignin removal with cellulose preservation through the combined effects of concentration, time, and temperature.

Temperature also strongly influences stiffness. From 20°C to 60°C (CSEB to AEB), Young's Modulus rises steadily to about 40 GPa, showing that moderate heating enhances crystallinity by removing hemicellulose and lignin. However, overly high temperatures can damage cellulose, limiting improvement. Time-dependent behaviour in Figure 7(d) shows a peak modulus at 20 to 25 minutes for AEB and OPT, after which stiffness decreases. Shorter treatments improve fibre structure, while prolonged extraction causes degradation. Thus, the ideal treatment duration is around 20 to 25 minutes for maximising strength and elasticity.

CONCLUSION

This study successfully optimised bamboo fibre extraction in Ghana using ultrasonic-assisted alkaline treatment combined with Response Surface Methodology (RSM). By systematically varying alkali concentration, temperature, and treatment time, the process was fine-tuned to maximise fibre yield and quality. The optimal condition of 6.078% NaOH, 20 °C and 20 minutes achieved a fibre recovery of 76.77%, producing fibres with balanced strength and flexibility, confirmed by a Young's Modulus of 250 GPa. Structural analysis (FTIR) verified the effective removal of lignin and hemicellulose, while thermogravimetric analysis (TGA) demonstrated improved thermal stability, with degradation transitions for hemicellulose, cellulose, and lignin observed between 200 and 600 °C. When compared to previous optimisation studies on natural fibre extraction, this work demonstrates clear novelty. Earlier investigations focused primarily on bast fibres such as jute, flax, hemp, and banana, or agricultural residues like sugarcane bagasse. These studies typically optimised alkaline or ultrasound-assisted extractions through RSM but were often directed at hemicellulose recovery, composite reinforcement, or food-related applications, usually under harsher conditions involving higher temperatures and longer processing times (Xie et al., 2020;

Wang et al., 2018; Dong et al., 2023; Rawat & Ghosh, 2024; Velmurugan et al., 2012; Mhlongo et al., 2022). In contrast, the present study is the first to integrate ultrasonic-assisted alkaline treatment with RSM optimisation specifically for bamboo fibre extraction in Ghana. Its novelty lies in achieving textile-grade bamboo fibres under mild processing conditions, thereby minimising energy and chemical inputs while delivering high-quality outputs suitable for eco- friendly applications.

Although the results are promising, several limitations must be acknowledged. The study was conducted at laboratory scale with controlled conditions, which may not fully represent industrial-scale variability, particularly in terms of bamboo source, fibre maturity, and processing capacity. Furthermore, the environmental and economic implications require broader evaluation. For instance, while the optimised process reduces chemical concentration and treatment time, a full life cycle assessment (LCA) is necessary to quantify the reduction in carbon footprint and wastewater load compared to conventional fibre extraction. Similarly, a techno-economic assessment would help determine the feasibility of large-scale adoption, particularly in Ghana's textile and composite industries.

Overall, this research demonstrates that ultrasonic-assisted alkaline extraction is a sustainable and potentially scalable method for producing high-performance bamboo fibres. Beyond enhancing yield and quality, it offers environmental benefits through reduced chemical and energy consumption and provides economic opportunities by valorising an underutilised local resource. The study, therefore, establishes a scientific foundation for positioning bamboo as a strategic raw material in Ghana's eco-friendly textile sector. Future research should focus on scaling up the process, evaluating environmental and economic trade-offs, and exploring industrial integration pathways to ensure the practical adoption of this green technology.

RECOMMENDATIONS

Further research is essential to scale the ultrasonic-assisted alkaline extraction process for industrial applications while addressing environmental variables and ensuring sustainability. Key recommendations for future optimisation include standardising sample preparation methods to minimise variability and enhance reproducibility across different bamboo species. A comprehensive evaluation of the cradle-to-gate life cycle of the fibre is crucial to assess the environmental footprint of the process, including energy consumption, chemical usage, and waste management. Exploring potential industrial applications such as composites, textiles, and bio-based materials will further validate the economic and practical viability of the fibres.

Additionally, advocating for policies to integrate bamboo into industrial value chains is imperative. This includes promoting bamboo as an alternative raw material for sustainable industries, fostering collaborations between researchers, policymakers, and industry stakeholders, and supporting small- and medium-scale enterprises to adopt bamboo-based technologies. Research into eco-friendly post-processing methods and circular economy approaches would also ensure the process remains sustainable while maximising the full potential of bamboo as a renewable resource.

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DECLARATION OF CONFLICT OF INTEREST

The author declares no conflict of interest regarding this manuscript.

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