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Dam Xuan Thang
thangdx@haui.edu.vn
Hanoi University of Industry
Ngo Thuy Van
Hanoi University of Industry
Pham Thi Thu Giang
Hanoi University of Industry
Nguyen Ngoc Linh
Thanh Do University

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Optimized oxidative-alkaline treatment of AP1 ramie fibers for enhanced cellulose purity and tensile strength

Dam Xuan Thang[1*], Ngo Thuy Van[1], Pham Thi Thu Giang[1], Nguyen Ngoc Linh[2]

[1]Faculty of Chemical Technology, Hanoi University of Industry

[2] Faculty of Pharmacy, Thanh Do University

Corresponding author: Dam Xuan Thang Email: thangdx@haui.edu.vn

Abstract

In this study, an optimized oxidative–alkaline treatment was developed to improve the structural and mechanical performance of AP1 ramie fibers. A 7% calcium hydroxide [$Ca(OH)_2$] solution was employed in combination with oxidizing agents—hydrogen peroxide (H_2O_2), calcium hypochlorite ($Ca(OCl)_2$), and sodium hypochlorite (NaClO)—to enhance delignification and hemicellulose removal. The treated fibers were characterized using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), tensile testing, colorimetric analysis, and thermogravimetric analysis (TGA) to evaluate their physicochemical transformations. Among the tested conditions, treatment with 4% H_2O_2 and 7% $Ca(OH)_2$ for 60 minutes yielded the highest performance enhancements, with tensile strength increasing by approximately 1.5 times—from 687.26 MPa (untreated) to 1061.60 MPa—and cellulose purity reaching 93%. Optimization of processing parameters using the Box–Behnken design and second-order regression modeling confirmed strong statistical significance ($R^2 > 0.99$), and model validation showed deviations below 5% between predicted and experimental values. These findings demonstrate the effectiveness of oxidative–alkaline processing for producing high-performance ramie fibers, and the enhanced mechanical and structural properties of treated AP1 ramie fibers suggest their strong potential for use in sustainable textile production and high-strength bio-composite applications.

Keywords: AP1 ramie fibers**,** alkaline - oxidative treatment**,** Box - Behnken Design (BBD)**,** Response Surface Methodology (RSM)**,** tensile strength**,** bio-composites.

1. Introduction

In recent years, natural fibers have received considerable attention as reinforcements in polymeric biocomposites due to their renewability, biodegradability, low cost, and environmental friendliness [1–3]. Among these, ramie fiber is particularly notable for its exceptionally high cellulose content (≈68–76 %), low lignin content, and superior tensile properties—often surpassing flax and jute—making it comparable to C-glass fiber in modulus and strength [1–4]. However, raw ramie fibers still contain considerable amounts of hemicellulose, lignin, pectin, and surface waxes, which impede effective interfacial bonding with hydrophobic polymer matrices [2, 4, 5].

To address these limitations, alkaline treatments such as $Ca(OH)_2$ or NaOH are widely employed to swell fibers, disrupt hydrogen bonding, remove amorphous fractions, and increase surface roughness and cellulose exposure [6–8]. When combined with oxidative agents like hydrogen peroxide (H_2O_2), reactive oxygen species (e.g., hydroxyl radicals, perhydroxyl anions) further promote delignification and hemicellulose cleavage, significantly improving cellulose purity and fiber morphology [9–12]. Notably, in kraft pulp bleaching, $Ca(OH)_2$ has been shown to replace NaOH with lower chemical demand and effective peroxide activation [13].

Several studies demonstrate that such alkaline–oxidative treatments enhance fiber crystallinity, thermal stability, and tensile performance. For instance, degummed ramie treated in alkali–peroxide systems shows significant cellulose exposure and improved mechanical

behavior [1, 7, 9]. Moreover, analogous treatments on bamboo and bagasse yield increases in Crystallinity Index, removal of lignin/hemicellulose, and improved fiber thermal/chemical characteristics [11, 12, 14].

To assess these modifications, characterization techniques such as Scanning Electron Microscopy (SEM), Fourier-transform Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA) are indispensable. SEM reveals surface ultrastructure changes (e.g., fibrillation), FTIR confirms reductions in non-cellulosic functional bands (C=O, C–O–C, aromatic), and TGA highlights increased thermal resistance and char residue, indicating higher cellulose content [11, 14–16].

While such qualitative evidence is robust, optimization of treatment parameters—including alkali and oxidant concentrations, temperature, and reaction time—is critical to balancing chemical efficacy and fiber integrity. This is exemplified in studies such as Mike et al. [10], where RSM was employed to optimize NaOH pretreatment of shea butter bark fiber, achieving nearly 98% cellulose content with minimal experimental runs. Response Surface Methodology (RSM), especially Box–Behnken Design (BBD), offers an efficient multivariable optimization tool, reducing experimental runs and enabling predictive modeling of tensile strength, yield, and crystallinity [6, 10, 13, 15, 17]. Specifically, optimization of ramie fabric treatments by BBD has identified ideal alkali concentration (~6 %), moderate temperature (≈39 °C), and short treatment durations (~30 min) to maximize breaking load (~518 N) and elongation (~23 %) [9, 18].

Despite this progress, research specifically targeting AP1 ramie fiber—the predominant cultivar in Vietnam—under alkaline–oxidative low-temperature conditions remains limited. Most existing protocols rely on extended heating (90–100 °C) and long durations (3–4 h), which pose sustainability and scalability challenges [9, 16].

Therefore, the present study aims to develop and optimize a low-temperature oxidative–alkaline treatment for AP1 ramie fibers using $Ca(OH)_2$ and H_2O_2, aiming to reduce processing time and chemical use while maximizing cellulose purity and tensile performance. The effects of concentration and duration on morphology, chemical composition, thermal stability, and mechanical strength were evaluated using SEM, FTIR, and TGA, with optimal processing conditions determined via BBD–RSM. This sustainable methodology aligns with circular economy paradigms in green composite material development [1, 2, 6, 9, 14, 16, 19].

2. Experiments

2.1. Materials

Raw AP1 ramie fibers (*Boehmeria nivea*) were collected from the Bo Bun sub-area, Nong Truong town, Moc Chau district, Son La province, Vietnam. The main chemicals used in the oxidative–alkaline treatments included analytical-grade calcium hydroxide ($Ca(OH)_2$, Vietnam), hydrogen peroxide (H_2O_2, 30% w/v, Vietnam), calcium hypochlorite ($Ca(OCl)_2$, 99.95% purity, China), and sodium hypochlorite solution (NaClO, 12% w/v, Vietnam). Other reagents such as sulfuric acid (H_2SO_4, 98%, Vietnam) were used for chemical composition analysis. All reagents were of analytical grade and used without further purification.

2.2. AP1 ramie fibers treatment method

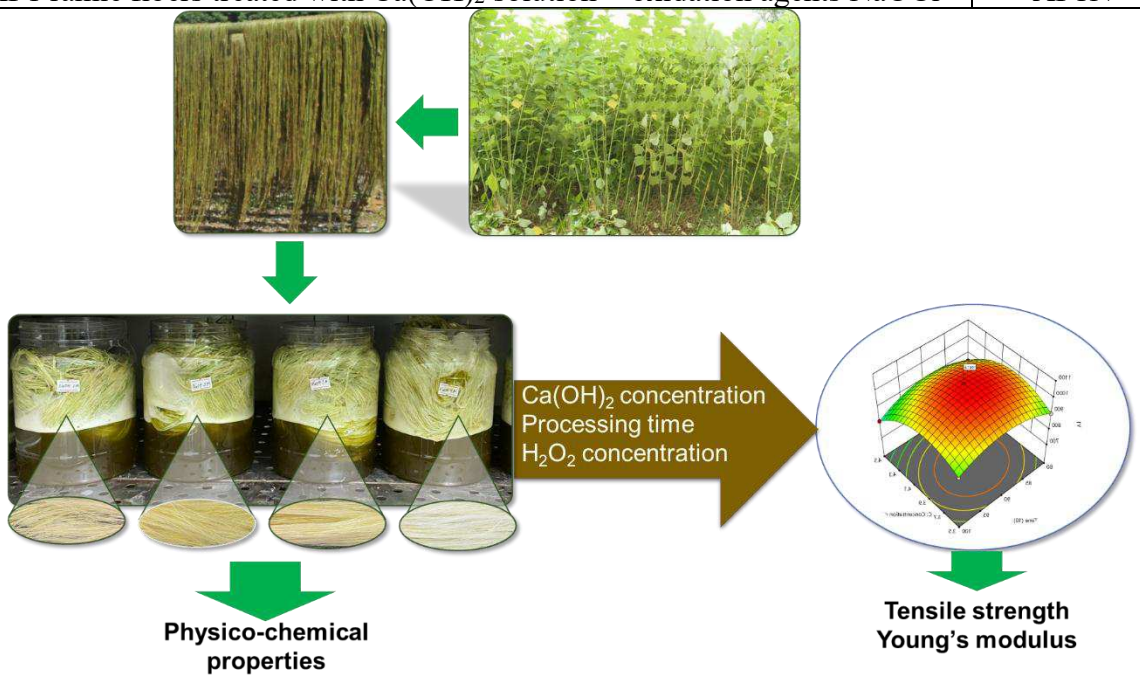
Prior to chemical treatment, the AP1 ramie fibers were combed, manually separated, and oven-dried at 70–80 °C for 2 hours to remove residual moisture. The dried fibers were then immersed in a 7% (w/v) calcium hydroxide ($Ca(OH)_2$) solution combined with various oxidizing agents: hydrogen peroxide (H_2O_2, 50%), calcium hypochlorite ($Ca(OCl)_2$, 99.95%), and sodium hypochlorite (NaOCl, 12%).

After treatment under varying conditions, fibers were thoroughly washed with distilled water until neutral pH (pH = 7) and dried again at 80 °C to constant weight.

The experimental samples were denoted as shown in Table 1, and the overall processing workflow is depicted in Figure 1.

Table 1. The symbols of all samples

Treatment methods	The symbols
AP1 ramie fibers didn't treat	AP1
AP1 ramie fibers treated with $Ca(OH)_2$ solution	AP10
AP1 ramie fibers treated with $Ca(OH)_2$ solution + oxidation agents H_2O_2	AP1H
AP1 ramie fibers treated with $Ca(OH)_2$ solution + oxidation agents $CaOCl_2$	AP1C
AP1 ramie fibers treated with $Ca(OH)_2$ solution + oxidation agents NaOCl	AP1N



Figue 1. Illustrative diagram of the experimental

2.3. Concentration of components in the AP1 ramie samples

2.3.1. Determination of hemicellulose concentration [19,20]

In the first step, 1 – 2 grams of dried AP1 ramie fibers were weighed into 150 ml of boiled water and stored at 100°C for 2 hours. Then, the AP1 ramie samples were filtered, washed, and dried at 105°C to constant weight, and recorded the value was m_1. Next to the step, the dried samples were put into the condenser with 150 ml H_2SO_4 1M, and boiled at 100°C for 1 hour. The fibers were also filtered and washed with distilled water 2 – 3 times, then dried at 105°C to constant weight, and the next value was m_2. The hemicellulose concentration was determined by the formula (1):

$$\% \text{ hemicellulose} = \frac{m_1 - m_2}{m_0} * 100\% \qquad (1)$$

2.3.2. Determination of cellulose concentration [19,20]

The value m_2 which was determined in hemicellulose concentration continued to be soaked in 10 ml H_2SO_4 (72%) solution for 4 hours. Then, the samples were taken into a 150 ml solution of H_2SO_4 1M and refluxed at 100°C for 2 hours, Then the samples were filtered, washed, and dried at 105°C in the oven. The recorded value was denoted as m_3. The cellulose concentration was determined by the formula (2):

$$\% \text{ cellulose} = \frac{m_2 - m_3}{m_0} * 100\% \qquad (2)$$

2.3.3. Determination of lignin concentration [19,20]

The value m_3 was continued using in this section, the weight of sample m_3 was calcined at 600°C for 4-6 hours, then weighed the obtained ash with mass m_4. The lignin concentration was determined using the formula (3):

$$\% \text{ lignin} = \frac{m_3 - m_4}{m_0} * 100\% \quad (3)$$

2.4. Experimental Design for Optimization

To systematically optimize the oxidative–alkaline treatment of AP1 ramie fibers, the Box–Behnken design (BBD), a widely recognized subclass of Response Surface Methodology (RSM), was employed. RSM is a statistical and mathematical tool that models complex multivariable systems using a reduced number of experiments, allowing for the analysis of individual and interactive effects among variables [11]. In this study, BBD was specifically selected for its capacity to efficiently fit second-order (quadratic) models, avoid extreme experimental combinations, and provide reliable predictions of optimal conditions without requiring exhaustive full factorial designs [21, 25].

Three independent variables were considered key to influencing the structural and chemical transformation of ramie fibers during treatment and were therefore selected as optimization factors: calcium hydroxide concentration (A, %, w/v), treatment time (B, minutes), and hydrogen peroxide concentration (C, %, v/v). These variables have been reported to significantly impact the removal of lignin and hemicellulose, the degree of cellulose purification, and the crystallinity index, which are directly correlated with the thermal and mechanical performance of natural fibers [3, 22, 23].

Table 2 summarizes the selected variables and their coded levels within the BBD scheme. Each factor was examined at three levels: low (–1), medium (0), and high (+1), leading to a total of 15 randomized experimental combinations, including five central replicates to estimate pure error.

Table 2. Independent variables and their coded levels used in the Box–Behnken experimental design

Independent Variables	Symbol	Coded Levels		
		Low(-1)	Medium(0)	High(+1)
Calcium hydroxide concentration (%)	A	6.5	7.0	7.5
Treatment time (minutes)	B	80	90	100
Hydrogen peroxide concentration (%)	C	3.5	4.0	4.5

Previous studies have demonstrated the successful application of BBD–RSM in optimizing chemical treatment conditions for various lignocellulosic substrates such as banana pseudostem [14], cassava bagasse [22], and wheat husk [23], leading to enhancements in tensile performance, cellulose recovery, and surface modification. Recent investigations on ramie and its composites have further emphasized that oxidant concentration and reaction time are critical determinants of fiber–matrix adhesion, structural degradation, and overall composite performance [3, 4].

Furthermore, methodological reviews of RSM have highlighted its utility in minimizing chemical usage, reducing energy consumption, and enhancing process scalability—features that align with the principles of green processing and circular economy in sustainable fiber valorization [11, 25].

The experimental matrix was generated using Design Expert v23.1.0. Regression modeling, analysis of variance (ANOVA), and three-dimensional response surface plots were conducted to evaluate the statistical significance of each factor, construct predictive equations, and determine optimal treatment conditions for maximizing fiber purity and mechanical performance.

2.5. Characterization of samples AP1 ramie

The Fourier-transform infrared (FTIR) spectra of the AP1 ramie fibers were recorded using a Nicolet iS10 spectrometer (Thermo Scientific, USA) in the range of 4000–400 cm^{-1}, with a resolution of 8 cm^{-1} and 16 scans per sample. The samples were prepared in pellet form by mixing with KBr.

The surface morphology of the treated and untreated fibers was examined using field emission scanning electron microscopy (FESEM) on a JSM–6510LV microscope (JEOL, Japan) at magnifications ranging from 5× to 300,000×.

Thermal stability of the fibers was evaluated by thermogravimetric analysis (TGA) using a TG209F1 instrument (NETZSCH, Germany). Measurements were conducted from 20°C to 600°C, at a heating rate of 10°C/min, under nitrogen atmosphere (flow rate: 10 cm^3/min).

The mechanical properties, including tensile strength (MPa), elongation at break (%), and Young's modulus (MPa), were measured using a Zwick Z2.5 tensile tester according to ASTM DIN 53503, with a crosshead speed of 5 mm/min.

3. Results and discussions

3.1. Investigating the influence of method treatment

3.1.1. Weight loss, cellulose, lignin concentration, and muscle properties of AP1 ramie fibers

The impact of oxidative–alkaline treatments on the chemical composition and mechanical behavior of AP1 ramie fibers is summarized in Table 3. All fiber samples were immersed in a 7% $Ca(OH)_2$ solution at ambient temperature for 1 hour, with or without additional oxidizing agents (H_2O_2, $Ca(OCl)_2$, NaOCl).

Table 3. Weight reduction, cellulose and lignin content, and mechanical properties of AP1 ramie fibers after alkaline and oxidative treatments.

Samples	Change in mass (%)	Cellulose concentration (%)	Lignin concentration (%)	Tensile (MPa)	Elongation at break (%)	Young modular, EY (GPa)
AP1	0.00	64.96	9.57	687.26 ± 4.19	1.21 ± 0.02	43.21 ± 1.10
AP10	10.78	76.08	5.78	706.15 ± 6.43	1.26 ± 0.01	52.12 ± 0.88
AP1H	26.53	96.03	1.38	1061.6 ± 5.95	1.36 + 0.03	89.35 ± 1.60
AP1C	30.07	95.12	1.54	830.10 ± 4.23	1.31 ± 0.01	80.12 ± 1.22
AP1N	31.83	93.33	1.47	784.60 ± 6.09	1.29 ± 0.03	78.58 ± 0.98

The untreated AP1 fibers had a cellulose content of 64.96% and a lignin content of 9.57%, with a tensile strength of 687.26 MPa. Following treatment with $Ca(OH)_2$ alone (AP10), modest improvements were observed in both cellulose content and tensile strength, attributed to the removal of amorphous hemicellulose and partial delignification via alkali-induced swelling and disruption of hydrogen bonding. This is consistent with prior studies indicating that alkaline treatment enhances the accessibility of cellulose fibrils by removing non-cellulosic components and increasing fibril alignment [25].

A markedly greater enhancement was observed with alkaline hydrogen peroxide (AP1H) treatment, which increased the cellulose content to 96.03% and reduced lignin to 1.38%. The tensile strength reached 1061.60 MPa, and Young's modulus rose to 89.35 GPa—highest among all samples. These improvements result from the generation of reactive oxygen species (ROS), including hydroxyl radicals (•OH) and perhydroxyl anions (HOO^-), via the alkaline decomposition of H_2O_2. These ROS selectively cleave lignin ether bonds and oxidize aromatic structures, leading to solubilization of low-molecular-weight fragments and facilitating delignification and cellulose purification [9, 26].

The underlying chemistry of lignin oxidation by alkaline hydrogen peroxide involves oxidative cleavage of conjugated carbonyl structures and β–aryl ether bonds, as detailed by

Gellerstedt and Agnemo [12]. These reactions disrupt the lignin macromolecule, enhancing the accessibility and crystallinity of the cellulose matrix.

Treatment with hypochlorite-based oxidants (AP1C and AP1N) also significantly reduced lignin (1.54% and 1.47%, respectively) and increased cellulose content, though the mechanical properties were inferior to those of AP1H. This could be due to the non-specific and more aggressive oxidative nature of hypochlorite ions (ClO^-), which may lead to oxidative cleavage of β-1,4-glycosidic bonds in cellulose chains, resulting in structural degradation [25]. Overall, the oxidative treatments can be ranked by efficiency as follows: H_2O_2 > $Ca(OCl)_2$ > NaOCl > $Ca(OH)_2$ > untreated. Hydrogen peroxide was found to be the most effective and fiber-preserving oxidant, enabling selective delignification while enhancing the mechanical performance of AP1 fibers for high-strength biocomposite applications.

3.1.2. FTIR spectra of AP1 ramie samples before and after being treated by different oxidants

Fourier-transform infrared (FTIR) spectroscopy was conducted to investigate the structural changes in AP1 ramie fibers subjected to different oxidative–alkaline treatments. Figure 2 presents the FTIR spectra of untreated fibers (AP1), alkali-treated fibers (AP10), and those treated with combined systems including hydrogen peroxide (AP1H), calcium hypochlorite (AP1C), and sodium hypochlorite (AP1N), all in 7% $Ca(OH)_2$ medium.

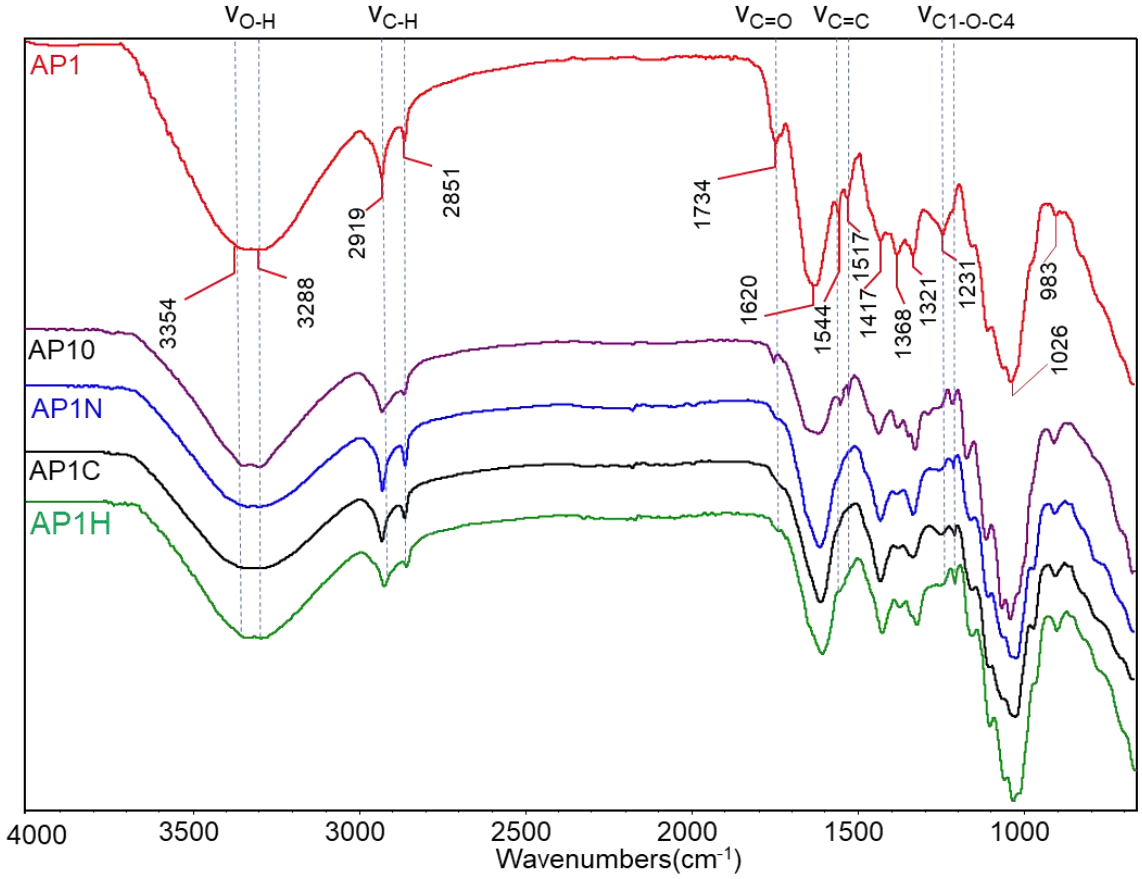


Figure 2. FTIR spectra of AP1 ramie fiber samples AP1, AP1H, AP1C, AP1N, AP10

A broad band between 3000–3500 cm^{-1}, assigned to O–H stretching vibrations of hydroxyl groups in cellulose and hemicellulose, showed a pronounced decrease after treatment, especially in the AP1H sample. This reduction reflects the disruption of hydrogen bonding networks and partial removal of hemicellulose and lignin [18, 25, 12, 32].

The C–H stretching vibrations at 2853 and 2940 cm^{-1}, characteristic of aliphatic CH_2 groups in cellulose, remained largely unchanged across all samples, indicating that the main cellulose backbone was structurally preserved [7, 18, 12, 28].

The most significant shift occurred at 1734 cm^{-1}, corresponding to the C=O stretching of hemicellulose and pectin ester bonds. This peak nearly disappeared in AP1H and decreased markedly in AP1C and AP1N, confirming effective hemicellulose removal—most prominently via alkaline hydrogen peroxide treatment [25, 26, 28, 29, 30, 32].

Lignin-associated aromatic skeletal vibrations at 1544 and 1517 cm^{-1} diminished substantially or vanished in the oxidant-treated samples, particularly in AP1H, indicating advanced delignification. These changes are attributable to the action of reactive oxygen species (ROS), such as hydroxyl radicals (•OH) and perhydroxyl anions (HOO^-), which selectively cleave aryl ether linkages and oxidize aromatic rings in lignin [10, 26, 30, 32].

The peak at 1231 cm^{-1}, corresponding to C–O stretching in aryl–alkyl ethers of lignin, also decreased significantly after oxidative treatments, supporting the above interpretation [18, 25, 26, 30, 32].

In contrast, peaks at 1423 cm^{-1} (CH_2 bending) and 1202 cm^{-1} (C–O–C stretching of glycosidic linkages) became more intense after treatment, especially in AP1H. This reflects enhanced exposure of crystalline cellulose domains and increased microfibrillar alignment, consistent with increased crystallinity [10, 18, 28].

Overall, the spectral data clearly indicate that alkaline hydrogen peroxide treatment (AP1H) is most effective in selectively removing amorphous non-cellulosic components while preserving and enhancing cellulose structure. The oxidation mechanism involves ROS-mediated cleavage of β–aryl ether and carbonyl-conjugated structures in lignin, as thoroughly described by Gierer [10].

3.1.3. SEM images of AP1 ramie samples

The surface morphology and microstructural transformations of AP1 ramie fibers subjected to different chemical treatments were examined using scanning electron microscopy (SEM), as illustrated in Figure 3.

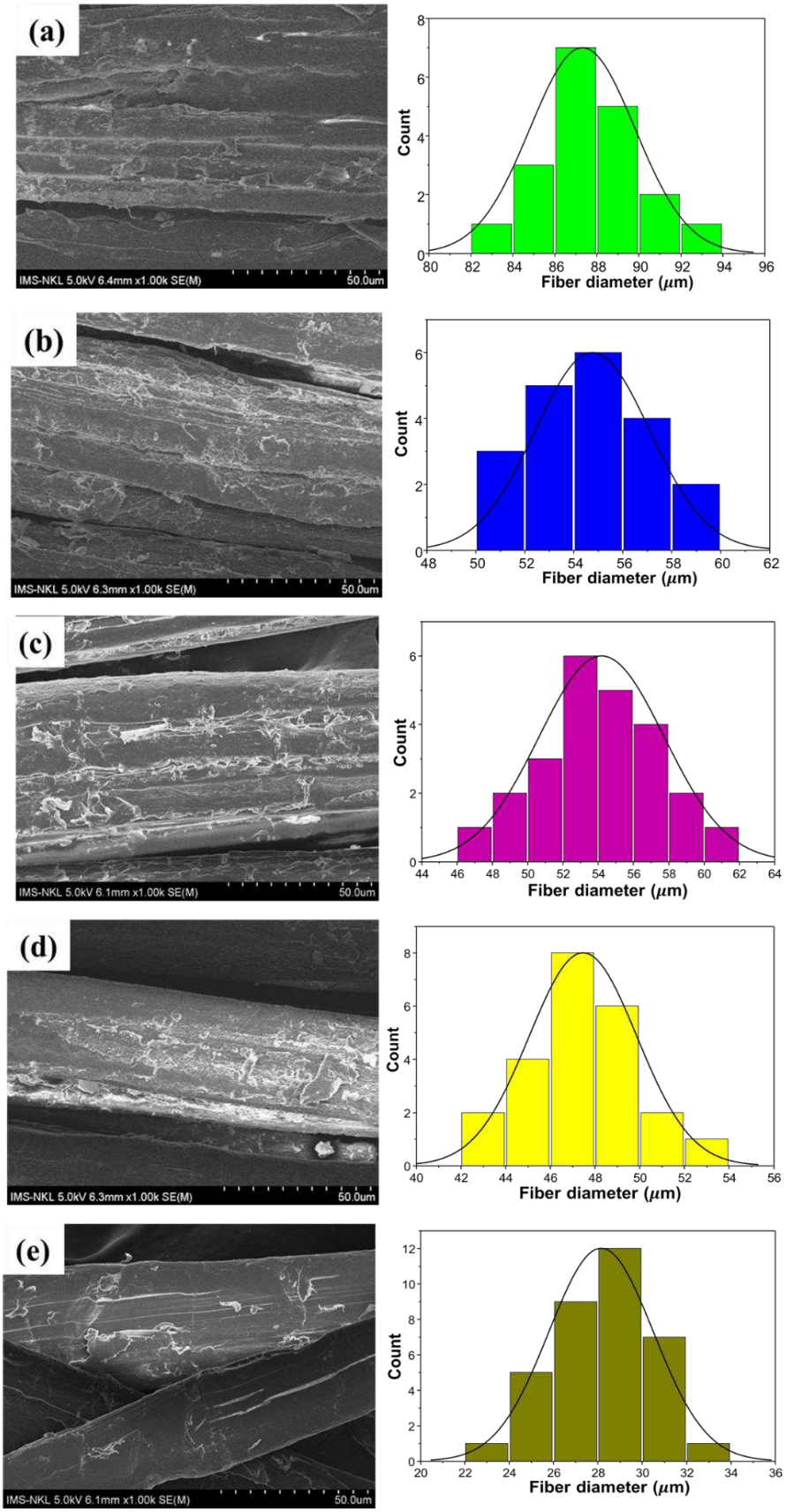


Figure 3. SEM images of AP1 ramie were treated with different oxidants AP1(a), AP10 (b), AP1N (c), AP1C (d), AP1H (e)

The untreated fibers (AP1, Figure 3a) exhibited densely packed bundles with irregular, coarse surfaces covered by waxy, lignin-rich, and hemicellulose-derived residues. These impurities hinder interfacial bonding and stress transfer in composite matrices. Similar observations were reported in raw kenaf and date palm fibers, where residual surface deposits contributed to poor mechanical performance in composites [34, 35].

After treatment with 7% $Ca(OH)_2$ alone (AP10, Figure 3b), minor surface smoothing and partial defibrillation occurred, but most non-cellulosic components remained. This outcome aligns with Modibbo et al [25], who showed that mercerization or mild alkaline treatments without oxidative agents fail to sufficiently disrupt the lignin–hemicellulose complex. The incomplete surface refinement confirms that alkaline treatment alone is inadequate for high-purity fiber preparation.

In the samples treated with NaOCl (AP1N, Figure 3c) and $Ca(OCl)_2$ (AP1C, Figure 3d), more noticeable fiber separation and removal of surface coatings were observed. However, residual irregularities and fibril agglomerations were still present. According to Liu et al [26], oxidants with lower redox potential or broader reactivity spectra (e.g., hypochlorites) are less effective at selective lignin cleavage and may require longer reaction times or elevated temperatures to achieve substantial purification. Moreover, hypochlorites can cause partial oxidative degradation, which affects the uniformity of surface cleaning.

The most significant morphological transformation occurred in the AP1H sample (Figure 3e), treated with hydrogen peroxide in alkaline medium. The surface was smooth, clean, and free of impurities, with clear microfibrillar separation and a more aligned structure. This can be attributed to the formation of hydroxyl radicals (•OH) under alkaline H_2O_2 conditions, which selectively cleave β-O-4 ether and aromatic linkages in lignin while leaving cellulose relatively untouched [12, 28]. Rafidison et al [29] also observed similarly refined and clean surfaces in fibers treated by oxidative–alkaline systems, demonstrating superior delignification and hemicellulose removal.

SEM-based fiber diameter analysis (Figure 3, right column) confirmed a progressive decrease in mean fiber diameter from 64 μm (AP1) to 37 μm (AP1H), along with a narrower and more symmetric diameter distribution in AP1H. This implies uniform defibrillation and better exposure of microfibrils. Such morphological improvement is crucial for enhancing fiber–matrix adhesion in composite applications. Edeerozey et al [34] reported that NaOH-treated kenaf fibers with cleaner surfaces and narrower diameter distributions led to better tensile performance. Similarly, Taha et al [36] emphasized that surface uniformity and fibril separation are strong indicators of optimized fiber–resin interaction.

Further validation comes from Zhang et al [37], who investigated copper ammonia-treated ramie fabrics and observed a reduction in fiber diameter from 35.8 μm to 24.6 μm after controlled treatment. Their SEM images revealed that reduced diameter was associated with less prickle and improved softness. These findings are in agreement with the present AP1H results, where smaller and more regular fibers suggest superior process selectivity and structural refinement.

What is particularly noteworthy is that the optimized AP1H process achieved these effects under mild conditions—room temperature and only 1 hour of treatment. This is in contrast to traditional bleaching or alkali treatments that often require temperatures above 90 °C for extended durations (3–4 hours) to induce similar morphological changes [12]. The environmental and energy-saving advantages of the H_2O_2–$Ca(OH)_2$ system further highlight its viability for sustainable composite production.

In summary, SEM analysis reveals that among the studied treatments, the AP1H protocol yields the most structurally refined ramie fibers. The high level of surface purity, microfibrillar separation, and diameter reduction observed in AP1H can be attributed to the selective delignification and hemicellulose removal facilitated by hydroxyl radicals. This

morphological improvement provides a strong foundation for the mechanical enhancement of AP1 fibers when used in advanced biocomposite materials.

3.1.4. The effect of the treatment method on the properties of ramie AP1 fibers

The thermal behavior of untreated (AP1) and alkaline hydrogen peroxide-treated (AP1H) AP1 ramie fibers was investigated using thermogravimetric analysis (TGA) and derivative thermogravimetry (dTG), as illustrated in Figure 4 and summarized in Table 4. Both samples exhibited a typical three-step thermal decomposition profile, characteristic of lignocellulosic fibers, consistent with previous observations on ramie, hemp, and banana fibers [1, 38].

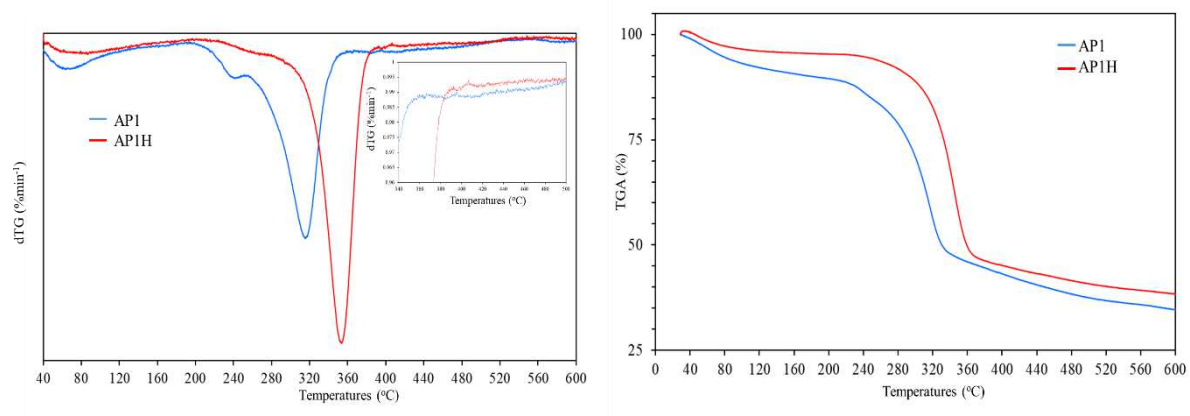


Figure 4. dTG and TGA diagrams of AP1 and AP1H ramie fibers

Table 4. Thermal degradation characteristics of AP1 and AP1H ramie fibers.

Temperature of decomposition stages (°C)	Maximum temperature (°C)		Mass reduction (%)		Remaining mass at 600°C (%)	
	AP1	AP1H	AP1	AP1H	AP1	AP1H
30 – 120	64	76	7.6	4.1	34.6	38.3
200 – 300	241	279	13.9	5.4		
300 – 380	315	354	39.8	46.4		
380 - 600	383	399	55.7	54.8		

In the first decomposition stage (30–120 °C), the observed mass loss corresponds to the evaporation of physically adsorbed moisture. Notably, the untreated AP1 fibers exhibited a higher moisture loss (7.6%) than AP1H (4.1%), suggesting that the oxidative–alkaline treatment significantly reduced hygroscopicity. This effect is attributed to the effective removal of hydrophilic hemicellulose and pectin, in agreement with the findings of Liu et al. on urea–H_2O_2-treated lignocellulosic materials and Widodo's study on peroxide-treated banana fibers [26, 38].

The second decomposition stage (200–300 °C) is primarily associated with the thermal degradation of hemicellulose and low-molecular-weight pectic substances. The AP1 fibers underwent a 13.9% weight loss, compared to only 5.4% in AP1H, clearly confirming the selective removal of these thermally labile fractions via hydrogen peroxide oxidation in alkaline medium. Similar reductions in hemicellulose-derived degradation have been reported in studies on peroxide-treated *Helicteres isora* and ramie fibers [39].

The most significant difference appeared in the third decomposition stage (300–600 °C), corresponding to cellulose and residual lignin degradation. The AP1H sample exhibited a marked shift in maximum degradation temperature to 354 °C, compared to 315 °C in AP1, indicating enhanced thermal stability due to increased cellulose purity and partial condensation of oxidized lignin fragments [9]. Moreover, the residual mass at 600 °C increased from 34.6% (AP1) to 38.3% (AP1H), suggesting the formation of a more thermally resilient carbonaceous framework—an observation consistent with Teli and Terega's findings on treated *Ensete* and kenaf fibers [40].

The dTG curves revealed a sharper and right-shifted degradation peak for AP1H, indicative of delayed decomposition onset and increased structural order. These features are hallmarks of cellulose-rich materials with reduced amorphous content, aligning with Widodo's thermal study of banana fibers post peroxide–alkali treatment [38].

The improved thermal behavior of AP1H can be attributed to the generation of reactive oxygen species (ROS), notably hydroxyl radicals (•OH) and superoxide anions (O_2^-), during the alkaline decomposition of H_2O_2. These ROS preferentially cleave β–aryl ether and conjugated carbonyl structures in lignin and hemicellulose while preserving the integrity of the cellulose backbone [9]. Consequently, the oxidative–alkaline treatment not only purifies the fiber composition but also enhances its thermal resilience.

Importantly, these enhancements were achieved under mild conditions—room temperature and a reaction time of just one hour—contrasting sharply with traditional thermal or bleaching methods that often require elevated temperatures (90–100 °C) and extended durations (3–4 hours) [38, 40]. The high thermal resistance and lower moisture uptake of AP1H fibers underscore the efficacy of this sustainable approach for applications in bio-based composites requiring thermal stability and environmental durability.

3.2. Effect of concentration of H_2O_2

Following the identification of hydrogen peroxide (H_2O_2) as the most effective oxidizing agent, a series of experiments was conducted to investigate the influence of varying H_2O_2 concentrations (1–5 wt.% based on fiber weight) on the treatment efficiency of AP1 ramie fibers. The corresponding samples were denoted as AP1H(1%), AP1H(2%), AP1H(3%), AP1H(4%), and AP1H(5%), as summarized in Table 5.

Table 5. Sample codes for AP1 ramie fibers treated with different H_2O_2 concentrations

Sample Names	Symbols
AP1 ramie fibers treated with 1% H_2O_2	AP1H(1%)
AP1 ramie fibers treated with 2% H_2O_2	AP1H(2%)
AP1 ramie fibers treated with 3% H_2O_2	AP1H(3%)
AP1 ramie fibers treated with 4% H_2O_2	AP1H(4%)
AP1 ramie fibers treated with 5% H_2O_2	AP1H(5%)

Figure 5 illustrates the effect of increasing H_2O_2 concentration on weight loss and cellulose content. Both parameters increased with H_2O_2 concentration: cellulose content improved from 80.5% at 1% to 94.7% at 5%, reflecting progressive removal of hemicellulose and lignin by oxidative delignification. Similar behavior has been observed in alkali–peroxide-treated ramie and bast fibers, confirming the effectiveness of H_2O_2 for fiber purification and compositional refinement [2, 33, 42].

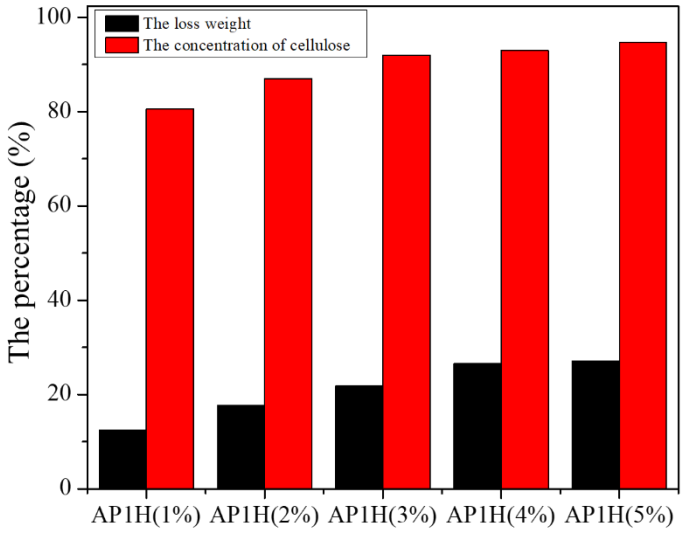


Figure 5. The loss of weight, the concentration of cellulose in different samples

Tensile strength trends (Figure 6) showed a gradual increase up to 4% H_2O_2 (1054.4 MPa), followed by a slight drop at 5% (998.2 MPa). This decline is attributed to over-oxidation, wherein excessive levels of reactive species (•OH, $O_2^{\bullet -}$) begin to degrade the cellulose backbone. Hydroxyl radicals are known to cleave β-1,4-glycosidic bonds in cellulose when generated in excess, leading to polymer chain scission and reduced mechanical integrity [9, 34].

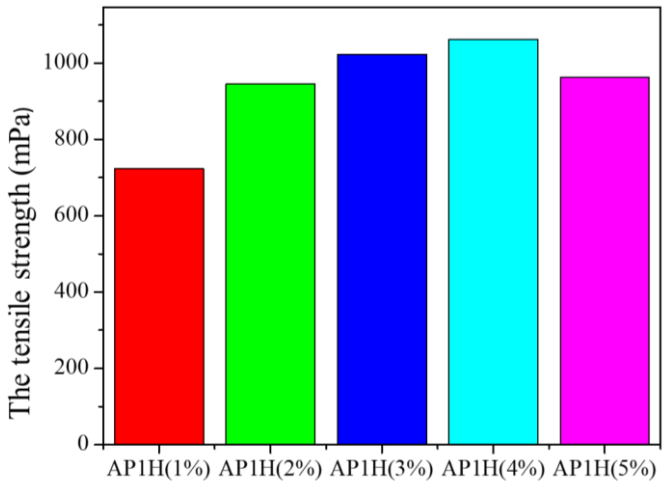


Figure 6. The tensile strength of different samples

FTIR spectra (Figure 7) further substantiated these trends. The peak at ~1734 cm^{-1} (C=O stretching in hemicellulose/pectin) progressively weakened and nearly disappeared at higher H_2O_2 levels, while C–O–C bands (1032–1060 cm^{-1}) became more intense, indicating selective removal of non-cellulosic components and improved cellulose crystallinity. This spectral evolution is consistent with earlier findings on peroxide-treated jute and kenaf fibers [32, 33].

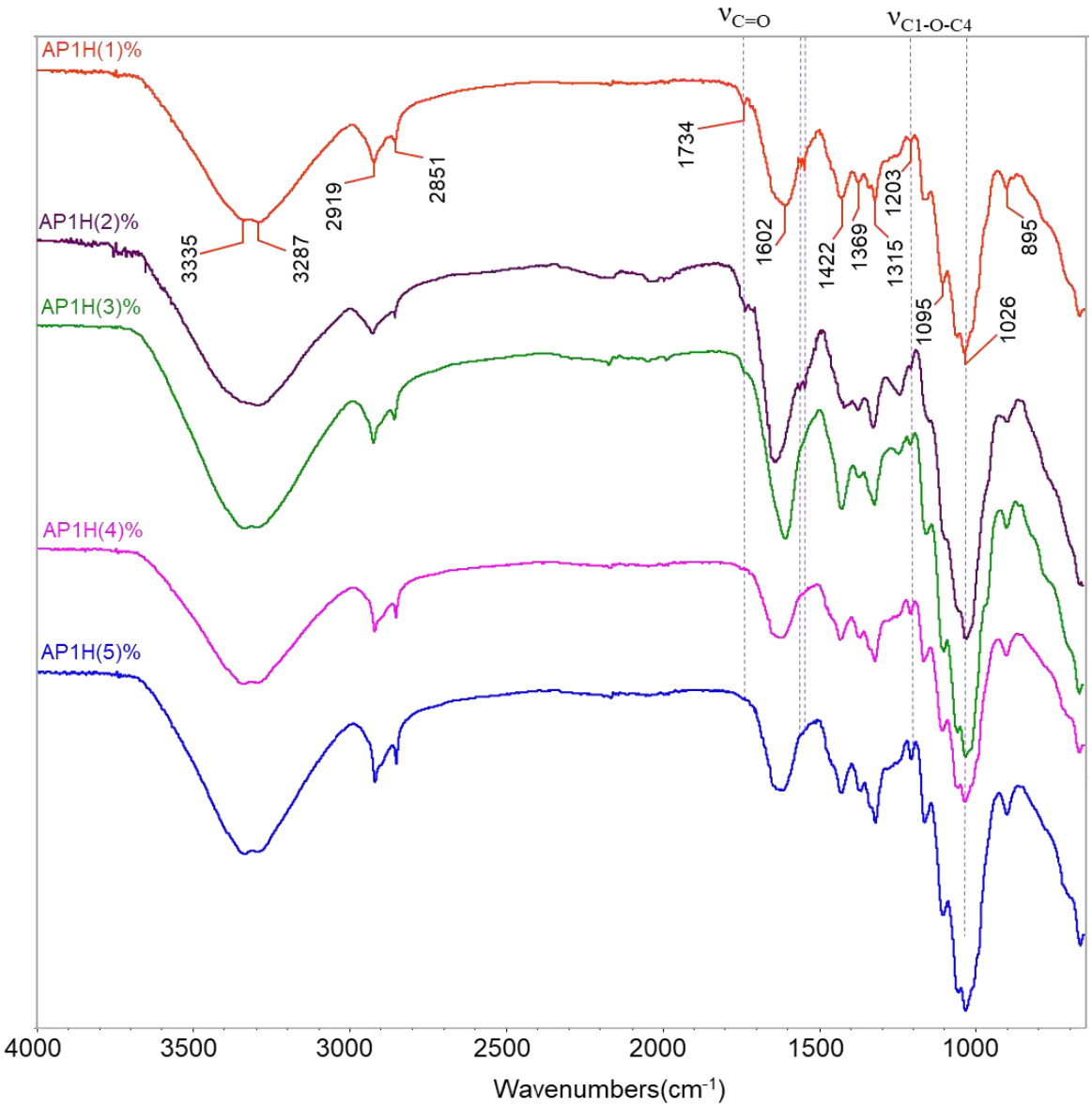


Figure 7. FTIR spectra of AP1 ramie fibers after treating with H_2O_2 in different concentrations (from 1% to 5%)

Quantitative analysis of FTIR band conversion (Figure 8) revealed enhanced reduction of lignin/hemicellulose-related peaks up to 4% H_2O_2, followed by a plateau or slight decline at 5%, suggesting possible oxidative damage to cellulose. Gellerstedt and Agnemo reported similar cellulose chain degradation under high peroxide loadings, particularly in amorphous domains [12].

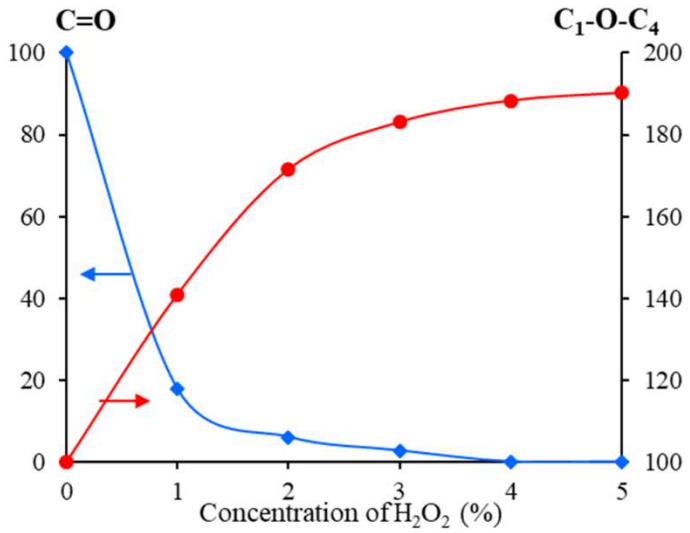


Figure 8. Effect of H_2O_2 concentration on the conversion efficiency of C=O, C1-O-C4 functional groups in AP1 ramie fibers during processing

Quantitative changes in FTIR absorbance (Figure 8) reinforce this trend. Decreases in lignin/hemicellulose bands, together with a rise in cellulose-associated bands, point to enhanced cellulose content up to 4% H_2O_2. However, at 5%, spectral intensity plateaus or even declines slightly, implying potential cellulose chain degradation. This correlates with tensile strength data and prior observations by Gellerstedt & Agnemo [12], who noted that excessive H_2O_2 leads to unwanted oxidation of cellulose amorphous regions.

The reaction mechanism underlying these observations is illustrated in Figure 9. Under alkaline conditions, H_2O_2 decomposes into hydroxyl radicals (•OH) and superoxide anions ($O_2^{\bullet -}$), which target β–O–4 linkages in lignin and ester groups in hemicellulose. However, excessive radicals can also attack amorphous cellulose, causing depolymerization [9, 26, 34].

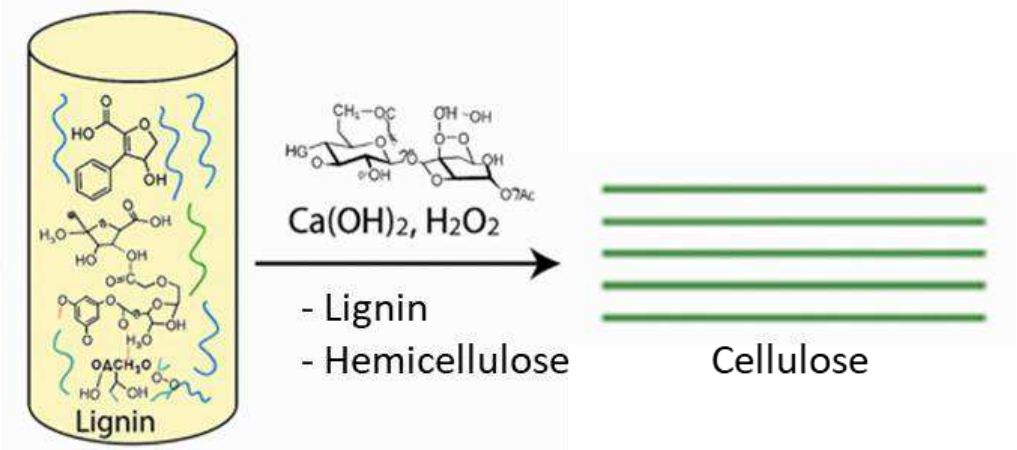


Figure 9. Schematic representation of the delignification and hemicellulose removal process in AP1 ramie fibers treated with a $Ca(OH)_2$ and H_2O_2 system.

Colorimetric data (Figure 10) supported this mechanism. ΔE values, indicating fiber whitening, increased with H_2O_2 concentration up to 4%, then plateaued. This mirrors chromophore removal trends observed in ramie fibers subjected to oxidative systems like Fenton or H_2O_2–iron catalysis [2, 42]. SEM images (not shown) revealed that at 5%, fibers displayed signs of surface erosion and microvoids, confirming over-oxidation effects.

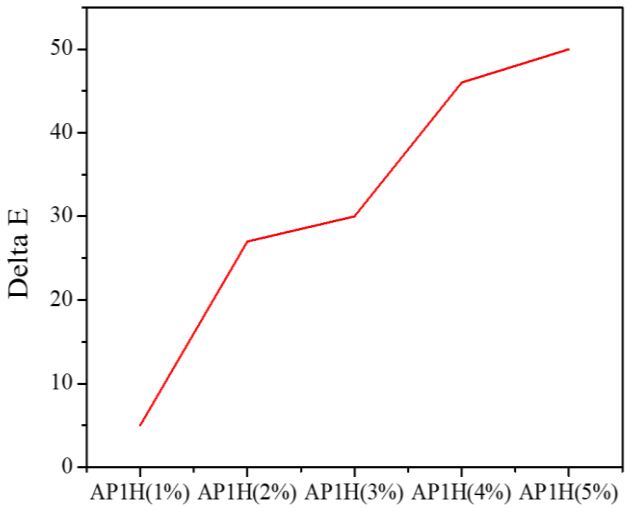


Figure 10. Effect of H_2O_2 concentration on the color change of AP1 ramie fibers

In summary, 4% H_2O_2 in combination with 7% $Ca(OH)_2$ appears to be the optimal condition for balancing fiber purification and structural integrity. At this level, hemicellulose and lignin are effectively removed, while cellulose remains largely intact. Above 4%, marginal gains in purity are offset by loss of mechanical properties and increased degradation, confirming prior findings on peroxide-based degumming optimization [2, 42].

3.3. The effect of time on the properties of ramie AP1 fibers under the best treatment conditions ($Ca(OH)_2$ alkaline solution adding H_2O_2 oxidation with concentration 4%)

The influence of treatment duration on the physicochemical and mechanical properties of AP1 ramie fibers was systematically investigated under the optimized alkaline–oxidative conditions, namely 7% calcium hydroxide ($Ca(OH)_2$) and 4% hydrogen peroxide (H_2O_2). The corresponding results are illustrated in Figure 11.

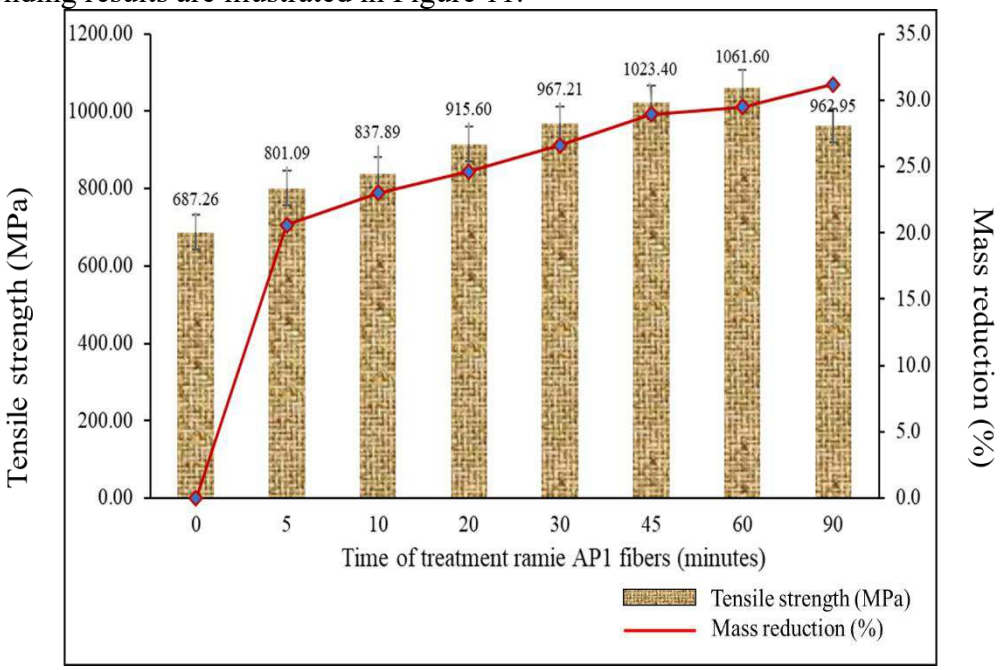


Figure 11. The effect of processing time of AP1 ramie fibers on weight loss and tensile strength

As shown, fiber weight loss increased rapidly during the initial 5 minutes of treatment, reaching approximately 21.4%, and gradually rose to 32.1% at 90 minutes. This trend indicates a time-dependent dissolution of amorphous constituents such as pectin, hemicellulose, waxes,

and loosely bound lignin. The alkaline environment facilitates fiber swelling and enhances peroxide penetration, thereby accelerating delignification and hemicellulose breakdown [9, 10, 12].

Similar behavior has been reported in other lignocellulosic systems, where increasing the treatment time improves compositional purification but simultaneously elevates the risk of fiber degradation [2, 39]. In particular, Acharya et al [39] observed that prolonged exposure to oxidative conditions caused partial damage to the fibril structure, leading to lower mechanical stability in some natural fiber systems.

Simultaneously, the tensile strength improved markedly from 687.3 MPa (untreated) to a maximum of 1061.6 MPa after 60 minutes, corresponding to the effective removal of non-cellulosic fractions and exposure of highly ordered cellulose regions. These structural refinements—confirmed by prior SEM and FTIR analyses—promote microfibrillar alignment and facilitate stress transfer at the fiber–matrix interface [2, 38, 42]. This trend is consistent with peroxide-treated ramie and other bast fibers, where tensile performance enhancement is associated with increased crystallinity and surface purification [26, 33].

However, extending the treatment to 90 minutes resulted in a slight decline in tensile strength (to 962.9 MPa), which is likely attributed to excessive oxidative exposure. Under prolonged alkaline–peroxide conditions, hydroxyl (•OH) and superoxide ($O_2^{\bullet -}$) radicals generated from H_2O_2 decomposition may begin to cleave β-1,4-glycosidic bonds within amorphous cellulose regions, leading to chain scission, microvoid formation, and structural weakening [9, 10, 18]. Gierer [10] and Zhou et al [41] also reported that over-treatment may disrupt hydrogen bonding and introduce defects in the cellulose network.

These findings highlight that a treatment duration of 60 minutes under the current system offers an optimal balance between component removal and structural retention. Extending beyond this duration yields diminishing returns in compositional purity and compromises mechanical integrity. Therefore, time optimization is essential for maximizing the performance of AP1 fibers in high-strength composite applications.

3.4. Optimizing the processing of AP1 ramie fibers.

3.4.1. The experimental results of optimizing the processing conditions of AP1 ramie fibers

To determine the optimal processing conditions for AP1 ramie fibers, a three-factor, three-level Box–Behnken Design (BBD) was implemented using Response Surface Methodology (RSM) in Design Expert version 23.1.0. The independent variables were calcium hydroxide concentration (A, %, w/v), treatment time (B, minutes), and hydrogen peroxide concentration (C, %, v/v), while the two selected response variables were tensile strength (Y_1, MPa) and Young's modulus (Y_2, GPa). This approach, as widely employed in fiber treatment optimization, enables efficient exploration of multivariable interactions with minimal experimental runs [11, 20].

The experimental matrix and measured responses are presented in Table 6. The highest tensile strength values—1061.6 and 1062.15 MPa—were observed at the center points (A = 7.0%, B = 90 min, C = 4.0%), confirming that moderate alkali and oxidant concentrations combined with sufficient processing time result in optimal fiber reinforcement. This trend aligns with the findings of Aly et al [5] and Cai et al [8], who reported enhanced mechanical properties in flax and ramie fibers under similarly balanced alkali treatments.

Table 6. Experimental matrix and results of the objective function values.

Run	$Ca(OH)_2$ concentration (% - A)	Time (minute - B)	H_2O_2 concentration (% - (C)	Y_1	Y_2
1	7.0	80	3.5	903.71 ± 6.27	77.02 ± 1.52
2	6.5	90	4.5	974.34 ± 4.33	76.83 ± 1.10
3	7.5	90	3.5	894.75 ± 3.87	75.42 ± 0.82

4	7.0	100	4.5	810.74 ± 4.06	72.61 ± 1.89
5	7.0	90	4.0	1061.6 ± 5.95	89.35 ± 1.60
6	7.5	90	4.5	713.01 ± 4.48	66.93 ± 1.11
7	6.5	80	4.0	1024.2 ± 3.00	67.35 ± 0.84
8	7.0	90	4.0	1059.42 ± 6.26	89.75 ± 0.72
9	7.0	100	3.5	911.94 ± 3.12	84.83 ± 1.52
10	7.0	80	4.5	853.67 ± 3.81	83.12 ± 1.16
11	7.5	80	4.0	816.85 ± 3.15	73.77 ± 1.47
12	6.5	100	4.0	965.97 ± 3.71	79.15 ± 1.73
13	7.5	100	4.0	844.85 ± 4.68	60.75 ± 2.11
14	6.5	90	3.5	953.17 ± 4.57	73.72 ± 1.75
15	7.0	90	4	1062.15 ± 3.75	90.14 ± 1.73

The statistical significance of the quadratic regression models for Y_1 and Y_2 was confirmed through analysis of variance (ANOVA), as shown in Table 7. The model for tensile strength yielded an F-value of 3141.19 ($p < 0.0001$), and that for Young's modulus exhibited an F-value of 118.95 ($p < 0.0001$). Among the main factors, calcium hydroxide concentration (A) and hydrogen peroxide concentration (C) showed the strongest influence on both responses ($p < 0.0001$), while treatment time (B) significantly affected tensile strength ($p = 0.0002$) but not modulus ($p = 0.2192$), indicating that stiffness is less sensitive to kinetic effects. These findings are consistent with prior reports by Cai et al [8] and Bekele et al [42].

Table 7. ANOVA analysis for the reactions.

Source	Objective function			
	Y_1		Y_2	
	F – value	P – value	F – value	P – value
Model	3141.19	< 0.0001	118.95	< 0.0001
A	9707.62	< 0.0001	52.25	0.0008
B	97.40	0.0002	1.97	0.2192
C	2246.20	< 0.0001	16.97	0.0092
AB	343.57	< 0.0001	158.08	< 0.0001
AC	1902.41	< 0.0001	34.53	0.0020
BC	120.94	0.0001	86.12	0.0002
A^2	3076.53	< 0.0001	594.16	< 0.0001
B^2	4471.41	< 0.0001	152.86	< 0.0001
C^2	8271.75	< 0.0001	43.32	0.0012
R^2	0.9998		0.9978	
R^2 correction	0.9995		0.9937	
Adeq Precision	183.2175		50.3296	

Interaction terms AB, AC, and BC also showed strong significance ($p < 0.0001$), underscoring the importance of parameter synergy. In particular, the interaction between $Ca(OH)_2$ and H_2O_2 concentrations (AC) exhibited a profound effect, which echoes observations by Widodo [38], who emphasized the role of oxidative–alkaline balance in enhancing fiber structure. Furthermore, the significant quadratic effects (A^2, B^2, C^2) support the non-linear nature of fiber behavior under treatment, highlighting the necessity for optimized process windows [11, 24].

The models demonstrated high predictive power, with R^2 values of 0.9998 for Y_1 and 0.9978 for Y_2, and adjusted R^2 values of 0.9995 and 0.9937, respectively. The high adequate precision values (183.22 for Y_1, 50.33 for Y_2) confirm excellent signal-to-noise ratios and modeling robustness.

Notably, these results align well with other natural fiber optimization studies. For instance, Bekele et al [42] showed that banana fibers achieved peak tensile performance at central composite variable settings using RSM. Similarly, Nguyen Thi Thuy Van [14] observed enhanced mechanical and chemical properties in banana fibers at optimized peroxide levels. Zhou et al [41] demonstrated that moderate oxidant dosages during Fenton-based degumming led to improved structural preservation and performance in ramie fibers.

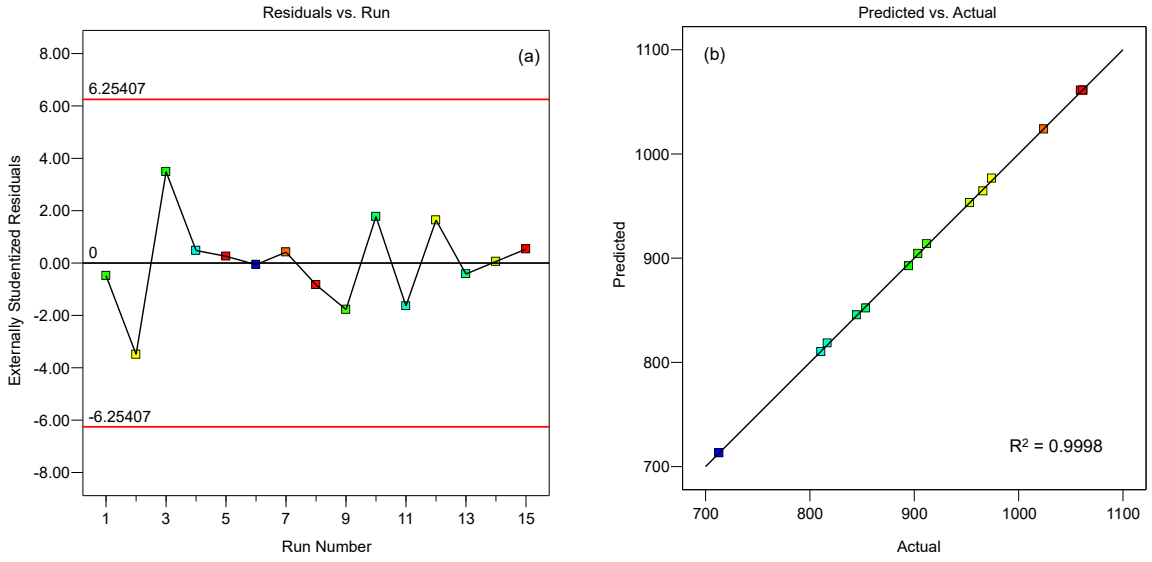
In summary, the quadratic regression models developed in this study are statistically significant, robust, and consistent with previous work on lignocellulosic fiber optimization. The identified optimal condition—7.0% $Ca(OH)_2$, 4.0% H_2O_2, and 90 minutes—yields superior tensile strength and modulus, providing a reliable foundation for upscaling AP1 fiber treatment in green composite applications.

3.4.2. Analyzing the significance of the model.

The statistical performance of the second-order regression model, constructed using the Box–Behnken design (BBD), was evaluated based on the analysis of variance (ANOVA) results presented in Table 6. The model exhibited exceptional predictive strength, with F-values of 3141.19 for tensile strength (Y_1) and 118.95 for Young's modulus (Y_2), both associated with p-values below 0.0001, confirming the overall significance of the model terms [5].

The coefficients of determination (R^2) were 0.9998 for Y_1 and 0.9978 for Y_2, indicating that the models explained over 99% of the variation in the responses. The adjusted R^2 values (0.9995 for Y_1 and 0.9937 for Y_2) further corroborated the robustness of the models by accounting for the number of predictors. In addition, the adequate precision ratios—183.22 for tensile strength and 50.33 for modulus—greatly exceeded the minimum threshold value of 4, highlighting excellent signal-to-noise ratios within the design space. These findings are consistent with earlier optimization studies on flax [5], ramie, and composite fibers [38], which emphasize the reliability of RSM-derived models for predicting fiber behavior.

The parity plots in Figure 12 demonstrate a strong correlation between experimental and predicted values, with data points tightly clustered along the 45° diagonal. The residuals were randomly distributed within ±6.25 units, suggesting a lack of systematic error and confirming the accuracy and lack of bias in the model.



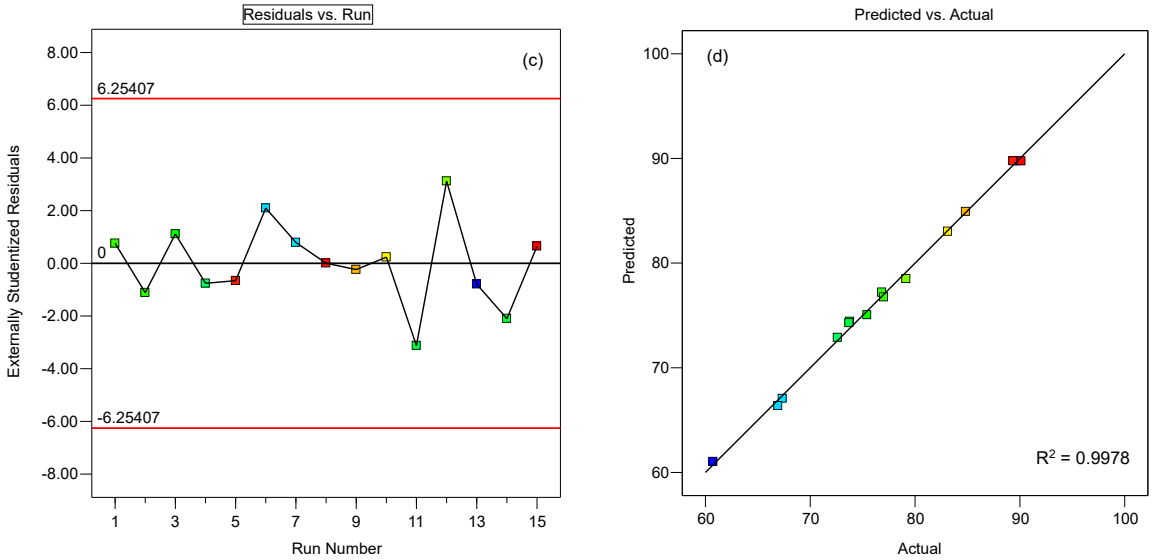


Figure 12. Experimental and predicted graphs, random distribution of Y_1, Y_2

After removing statistically insignificant variables ($p > 0.05$), the final second-order regression equations were derived as follows:

$$Y_1 = 1061.6 - 81.03A - 8.12B - 38.98C + 21.56AB - 50.73AC - 12.79BC - 67.14A^2 - 80.95B^2 - 110.10C^2 \quad (1)$$

$$Y_2 = 89.75 - 2.52A - 0.49B - 1.44C - 6.20AB - 2.9AC - 4.58BC - 12.83A^2 - 6.66B^2 - 3.69C^2 \quad (2)$$

The regression coefficients revealed that all linear terms (A, B and C) exerted negative effects on the responses, indicating that excessive levels of individual variables can reduce tensile strength and stiffness. This reflects the concept of an optimum in response surface methodology, beyond which further increases in process parameters lead to deterioration in fiber structure and properties [5, 41].

All interaction terms (AB, AC, and BC) were found to be statistically significant ($p < 0.001$), underscoring the importance of synergistic effects among process variables. Notably, the AC interaction (alkali–oxidant) had the strongest influence on tensile strength, supporting observations from Widodo [38], who emphasized the critical role of oxidative–alkaline balance in fiber refinement. For Young's modulus, the interaction term AB and the quadratic effect A^2 were particularly influential, suggesting that fiber stiffness is more sensitive to the interplay between alkali strength and reaction time, which concurs with the findings of Bekele et al [42].

These results are consistent with previous studies employing RSM for fiber optimization. Aly et al [5] observed a decline in tensile strength of flax fibers when alkali concentrations exceeded the optimal level. Similarly, Nguyen Thi Thuy Van [14] reported partial cellulose degradation at peroxide concentrations above 4% in banana fibers. Zhou et al. [41] found that oxidative degumming of ramie using Fenton reagents achieved superior mechanical properties at moderate oxidant levels, highlighting the importance of dosage control.

In conclusion, the BBD-based regression models developed in this study are statistically robust, highly predictive, and scientifically sound. They effectively capture the non-linear and interactive effects of treatment variables and provide a reliable foundation for optimizing AP1 ramie fiber processing in sustainable high-performance composite applications.

3.4.3. Analyzing the influence of technological parameters on the objective function Y1

The interactive effects of technological parameters — namely $Ca(OH)_2$ concentration (A), treatment time (B), and H_2O_2 concentration (C) — on the mechanical properties of AP1 ramie fibers were thoroughly examined using three-dimensional response surface plots and

their corresponding contour diagrams (Figure 13 for Y_1, Figure 14 for Y_2). These visualizations offer valuable insight into the nonlinear behavior and synergistic interactions between processing variables.

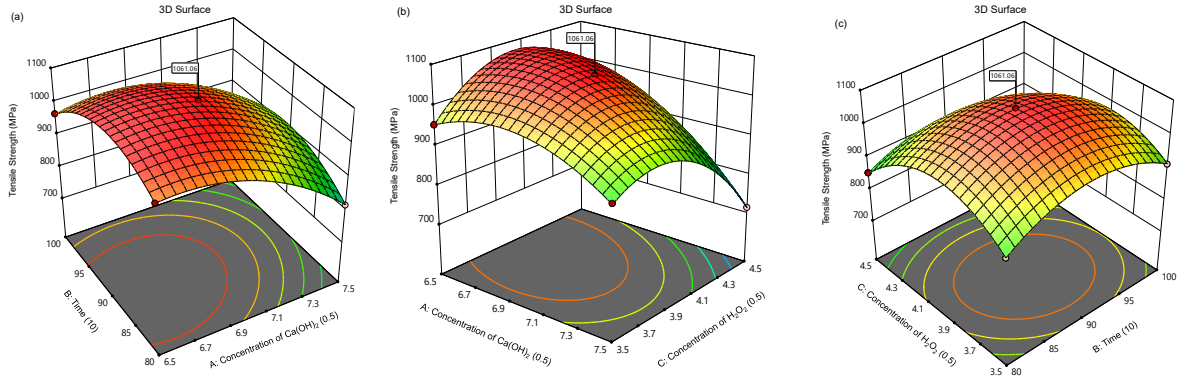


Figure 13. The response surface of technological factor pairs on the target function Y_1

As shown in Figure 13a, increasing both $Ca(OH)_2$ concentration and treatment time initially enhances tensile strength, likely due to the progressive removal of amorphous matrix components — primarily hemicellulose and surface lignin — which in turn exposes and aligns crystalline cellulose microfibrils. However, beyond a threshold (A > 7.0% and B > 90 minutes), tensile strength begins to decline, suggesting partial degradation of cellulose chains under extended alkaline–oxidative exposure. This trend is consistent with previous studies reporting mechanical deterioration caused by β-1,4-glycosidic bond cleavage when alkali or processing time is excessive [5, 8, 41].

Figure 13b illustrates the interaction between $Ca(OH)_2$ and H_2O_2 concentrations. An optimal region is evident at intermediate levels of both reagents, while higher peroxide concentrations (> 4%) reduce tensile strength due to intensified depolymerization of cellulose. This behavior aligns with the observations of Widodo [38] and Zhou et al [41], who emphasized that oxidative damage beyond a critical oxidant threshold leads to chain scission and fiber embrittlement.

In Figure 13c, the interaction between treatment time and H_2O_2 concentration further highlights the sensitivity of the fiber system to over-oxidation. Prolonged exposure at high peroxide levels promotes excessive delignification and degradation of structural polysaccharides, leading to a measurable reduction in tensile strength beyond 90 minutes. This is supported by Bekele et al [42], who reported that over-processing during peroxide bleaching caused microstructural damage and mechanical weakening in natural fibers.

The optimal region — centered around A = 7.0%, B = 90 minutes, and C = 4.0% — corresponds to a peak tensile strength of approximately 1062 MPa. This validates the predictive accuracy of the response surface model and is in close agreement with prior optimization studies on banana and ramie fibers. Nguyen Thi Thuy Van [14] demonstrated that peroxide concentrations above 4% adversely affected cellulose integrity in banana fibers, while Zhou et al [41] confirmed that moderate oxidant dosages in Fenton-based degumming of ramie yielded superior mechanical properties compared to more aggressive treatments.

Turning to Young's modulus (Y_2), the corresponding plots (Figure 14) indicate that stiffness is more sensitive to chemical concentrations than to treatment time. Figure 14a reveals a pronounced nonlinear relationship between $Ca(OH)_2$ concentration and modulus, with a maximum at ~7.0%, followed by a decline attributed to microvoid formation and cellulose matrix disruption [8, 38]. Figures 14b and 14c show that H_2O_2-related interactions (with time and alkali) exhibit parabolic trends: moderate oxidative levels enhance fiber stiffness by exposing crystalline domains, while excessive conditions lead to modulus reduction due to over-fragmentation, as also noted by Yaro et al [43].

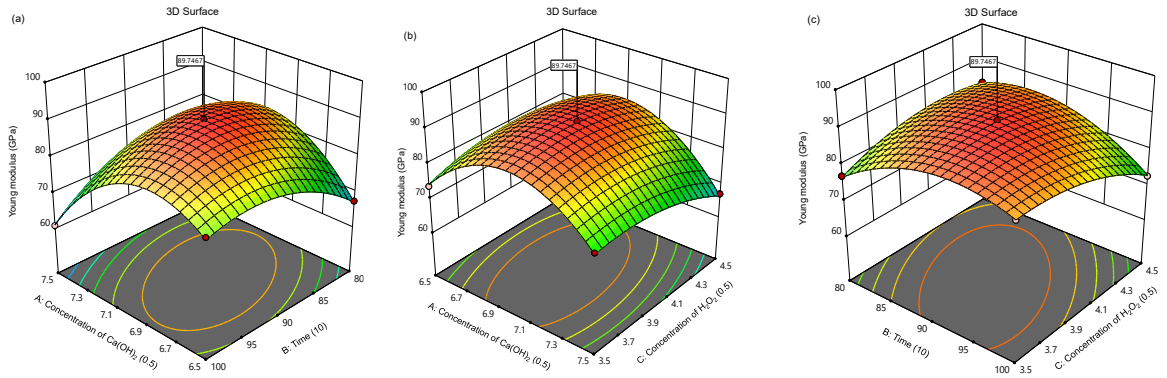


Figure 14. The response surface of technological factor pairs on the target function Y_2

Statistical analysis in Table 6 reinforces these empirical findings. All interaction terms (AB, AC, BC) were statistically significant ($p < 0.001$), confirming that fiber mechanics are governed by nonlinear, synergistic effects among chemical concentration and treatment duration.

In conclusion, the response surface and contour analyses clearly demonstrate that moderate levels of $Ca(OH)_2$ and H_2O_2, combined with a treatment time of 90 minutes, provide the most favorable conditions for optimizing tensile strength and stiffness in AP1 ramie fibers. The consistency between model predictions, experimental data, and prior literature [5, 8, 14, 38, 41–43] affirms the validity and applicability of the optimization strategy for sustainable bio-composite applications.

3.4.4. Optimization of AP1 ramie fiber processing conditions and validation testing

The optimal processing conditions for AP1 ramie fibers were identified using the desirability function approach integrated in Design Expert version 23.1.0. This multi-objective optimization technique aimed to simultaneously maximize tensile strength (Y_1) and Young's modulus (Y_2). Among 100 generated solutions, the second-ranked solution achieved the highest global desirability value (1.000), corresponding to the processing condition of 7.0% $Ca(OH)_2$ concentration, 90 minutes of treatment time, and 4.0% H_2O_2 concentration (Figure 15).

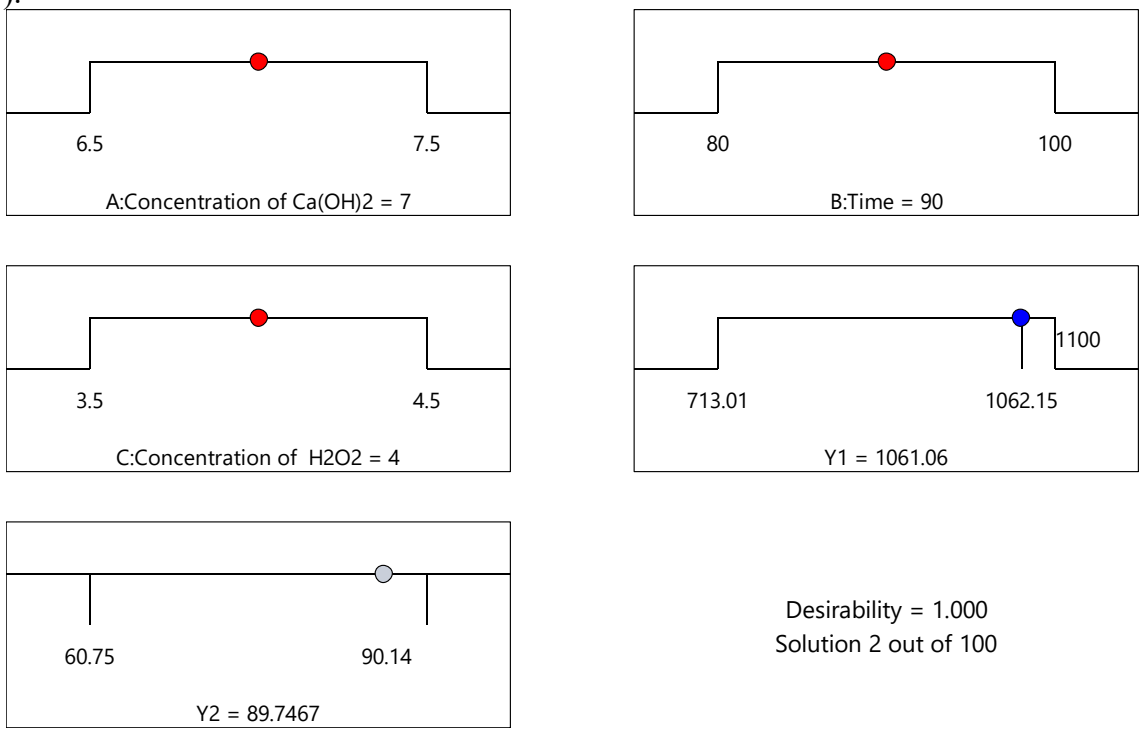


Figure 15. Optimal conditions and predicted values of the target functions Y_1, Y_2

Under these optimized conditions, the model predicted tensile strength and Young's modulus values of 1061.6 MPa and 89.35 MPa, respectively. These values reflect a favorable balance between efficient delignification and preservation of cellulose microstructure. The performance improvement is attributed to synergistic effects between alkali-induced fiber swelling, oxidative cleavage of hemicellulose and lignin linkages, and enhanced microfibrillar alignment—mechanisms previously documented in optimized fiber systems [41, 42].

To validate the model predictions, triplicate confirmation experiments were performed under the identified optimal conditions. The measured tensile strength and modulus were 1083.5 ± 4.13 MPa and 91.42 ± 1.77 MPa, respectively. These results deviated by only 2.06% and 2.32% from the predicted values (Table 8), confirming the high predictive accuracy and reliability of the developed RSM-based model.

Table 8. Confirmation test results with optimized AP1 ramie fibers processing parameters

$Ca(OH)_2$ concen-tration (%)	Time (min)	H_2O_2 concen-tration (%)	Tensile strength (%)		Young's modulus (MPa)		Error (%)	
7	90	4	Predicted	Actual	Predicted	Actual	Tensile strength	Young's modulus (MPa)
			1061.6	1083.5 ± 4.13	89.35	91.42 ± 1.77	2.06	2.32

These findings are in strong agreement with prior studies. Nguyen Thi Thuy Van [10] observed that banana fibers treated under moderate peroxide concentrations (~4%) exhibited enhanced tensile performance without compromising cellulose integrity. Similarly, Zhou et al. [35] reported that Fenton-based oxidative degumming of ramie achieved optimal mechanical outcomes when oxidant levels were maintained within a controlled range. Bekele et al [42] further demonstrated that excessive oxidative exposure during peroxide bleaching led to fibril disruption and loss of mechanical strength.

Furthermore, the use of the desirability function has been widely validated in natural fiber processing as an effective tool for solving multi-objective optimization problems. Aly et al [5] employed this technique to successfully optimize chemical treatments in flax fibers, achieving a close match between model predictions and experimental results. Similarly, Yaro et al [43] applied desirability analysis to composite optimization, confirming its robustness for balancing multiple performance criteria.

In summary, the integration of Box–Behnken design, response surface modeling, and desirability function analysis produced a statistically robust and practically scalable optimization framework. The validated optimal condition (7.0% $Ca(OH)_2$, 90 minutes, 4.0% H_2O_2) yielded AP1 ramie fibers with outstanding mechanical properties, making them highly suitable for sustainable, high-performance biocomposite applications.

With their outstanding mechanical performance (tensile strength exceeding 1080 MPa and Young's modulus of approximately 91 MPa) and thermally stable microstructure after optimal treatment, AP1 ramie fibers demonstrate significant potential as reinforcing agents in a wide range of bio-based polymer matrices. In poly(lactic acid) (PLA) systems, AP1 fibers can effectively enhance stiffness, tensile strength, and biodegradability, making them well-suited for sustainable packaging and resorbable biomedical materials. Similarly, in poly(hydroxyalkanoate) (PHA)-based composites, their incorporation improves mechanical resilience and structural integrity under challenging environmental conditions. Notably, recent findings by Thang et al reported the successful reinforcement of bio-based polyamide 11 (PA11) with $Ca(OH)_2$-treated and epoxy-functionalized AP1 fibers, resulting in a 2.5–4.0-fold increase in both tensile strength and modulus compared to neat PA11 [44]. This improvement

was attributed to the formation of covalent bonds between surface epoxy groups and the amide backbone of PA11, which enhanced interfacial adhesion and stress transfer efficiency. Beyond thermoplastics, AP1 fibers can also be integrated into epoxy resin systems derived from vegetable oils—such as epoxidized soybean oil (ESO) or black seed oil—to fabricate high-performance, eco-friendly coatings and composites for applications in construction and aerospace. These results highlight the versatile applicability of AP1 fibers in next-generation bio-composites, reinforcing their role as a key enabler in the transition towards sustainable, high-strength, and degradable polymeric materials.

4. Conclusions

In this study, an environmentally friendly oxidative–alkaline treatment process for AP1 ramie fibers was successfully developed and systematically optimized. The findings demonstrated that alkaline treatment using 7% $Ca(OH)_2$ alone resulted in limited delignification and insufficient mechanical enhancement. In contrast, the incorporation of hydrogen peroxide (H_2O_2) as an oxidant significantly improved treatment efficiency by enabling deeper penetration and more selective degradation of non-cellulosic components. Notably, the optimized process was conducted under mild conditions (room temperature, short duration), avoiding the need for elevated temperature typically required in conventional degumming protocols.

The optimal treatment condition—7.0% $Ca(OH)_2$ and 4.0% H_2O_2 for 60 minutes—was identified using response surface methodology (RSM), and experimentally validated with <3% deviation between predicted and measured responses. Under these conditions, the treated AP1 fibers exhibited a cellulose content of 93%, tensile strength of 1061 MPa (a 54.4% increase compared to untreated fibers), and a reduced average fiber diameter of 37 μm. These improvements are attributed to effective delignification, microfibrillar exposure, and enhanced crystallinity, which were corroborated by FTIR, SEM, and TGA analyses presented in previous sections.

The regression models developed using RSM and desirability function analysis exhibited strong predictive accuracy, confirmed by high R^2 values (>0.99) and satisfactory adequacy precision. The validated models provide a reliable statistical tool for optimizing oxidative treatment of lignocellulosic fibers, and the findings align well with prior studies on flax, banana, and ramie systems, while extending applicability to the underexplored AP1 ramie variety.

Importantly, this study provides a robust and scalable framework for processing natural fibers under low-energy conditions, thereby minimizing chemical consumption, energy input, and secondary pollution. The optimized AP1 fibers are well-suited for integration into high-performance, biodegradable composites, with potential applications in sustainable packaging, automotive interiors, and eco-friendly construction materials.

Overall, the proposed oxidative–alkaline method offers a compelling alternative to conventional harsh chemical treatments, combining operational simplicity, environmental compatibility, and superior material quality. This work contributes meaningfully to the advancement of green manufacturing systems, aligning with circular economy strategies and long-term carbon-neutral goals.

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Data availability The data used to support the findings of this study are included within the article. Declaration of competing interest.

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