

RESEARCH ARTICLE

Preliminary evaluation of boric acid-treated cellulose derived oil palm empty fruit bunches

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Abstract - This study investigates the effect of boric acid (BA) treatment on the physicochemical properties of cellulose derived from oil palm empty fruit bunches (OPEFB), with the goal of enhancing thermal stability. The work follows a systematic one-factor-at-a-time (OFAT) approach to screen the effects of BA concentration, reaction time, and treatment temperature on the physico-chemical properties of the cellulose derived OPEFB. Characterization techniques including Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and thermogravimetric analysis (TGA) were employed. FTIR confirmed the reduction in free hydroxyl groups and the successful formation of borate ester, particularly at a 3% (w/v) BA concentration. Preserved crystallinity was observed under moderate treatment conditions, as shown by the XRD analysis. In addition, improved thermal stability was achieved, as demonstrated by the TGA analysis, particularly at 3% (w/v) boric acid (BA), 2 h, and 25°C. This study shows that under these conditions, moderate thermal stabilization can be achieved through chemical crosslinking without significant structural degradation. Furthermore, this study demonstrates that sustainable cellulose-based materials with enhanced performance can be developed using a boric acid modification approach.

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1. Introduction

Cellulose, a natural polysaccharide composed of β -1,4-linked glucose units, plays a fundamental role as a structural component in plant cell walls. Its linear molecular structure allows extensive hydrogen bonding, enabling functionalization through chemical reactions to produce materials with desired properties [1]. Despite its abundance, renewability, and biocompatibility, cellulose suffers from a notable limitation: low thermal stability [2]. At elevated temperatures, cellulose begins to thermally degrade at approximately 250 °C, losing structural integrity [3]. This property restricts its use in high-performance applications where thermal stability is crucial, particularly in construction and automotive materials [4]. However, chemical modification of cellulose can overcome this limitation by producing a structurally and thermally stable reinforcement material suitable for advanced composites. A promising but underutilised source of cellulose is the oil palm empty fruit bunches (OPEFBs) produced as byproducts of the palm oil industry. OPEFBs are rich in cellulose, comprising up to approximately 65% of the bulk component of OPEFB fibres, alongside hemicellulose and lignin [5]. While this abundance makes EFBs an attractive feedstock for value-added applications, their complex composition introduces challenges, particularly regarding thermal degradation and variability in fibre quality. These issues pose a significant barrier to their adoption in advanced material applications, especially those requiring dimensional and mechanical stability under thermal stress.

To address the thermal limitations of OPEFB-derived cellulose, chemical modification using boric acid (BA) has emerged as a viable solution. BA is a well-established flame retardant known for its selective interaction with hydroxyl groups on cellulose chains, forming borate esters [6]. This reaction enhances thermal stability by promoting crosslinking and forming a protective char layer during thermal decomposition [7]. The mechanism not only suppresses volatile-compound release but also improves moisture resistance and reduces flammability. Importantly, unlike stronger acids, BA offers a milder route to cellulose modification, reducing the risk of polymer degradation [7]. Previous studies indicate that BA-treated cellulose not only improves thermal stability but also retains or even enhances mechanical properties such as tensile strength and fire retardancy [7]. Furthermore, a few studies reported BA as a crosslinking agent in microfibrillated cellulose composites [8], reinforcement of (nano)-cellulose particle boards [9–10] and nanocellulose adhesive [10]. BA modification has emerged as a promising approach to address cellulose's inherent limitations, particularly its thermal instability, a significant challenge when using cellulose as a reinforcement material in high-performance composites [11]. Cellulose, especially when derived from sustainable sources such as empty fruit bunches of the oil palm industry, offers an environmentally friendly and cost-effective alternative to synthetic fibres. However, its susceptibility to thermal degradation, which typically occurs between 60 °C and 110 °C due to the loss of water in the samples, and results in rapid weight loss between 300 °C and 400 °C, restricts its application in processes that require elevated temperatures [3]. This limitation is further compounded by cellulose's hydrophilic nature, which leads to moisture absorption, plasticization, and further reduction in thermal and mechanical performance [12]. To overcome these drawbacks without compromising cellulose's mechanical integrity, BA modification is used as a targeted chemical strategy.

This research aims to investigate the effect of BA treatment on the physicochemical characteristics of cellulose extracted from OPEFB, with a specific focus on improving its thermal stability. To achieve this, cellulose was extracted using a multi-step pretreatment process involving hot water, alkaline treatment, bleaching, and deep eutectic solvent

(DES) treatment, followed by sonication as described in our previous study [13]. We then treated the extracted cellulose with varying concentrations of BA at different reaction times and temperatures using a one-factor-at-a-time (OFAT) approach. However, this study is intended as a preliminary evaluation of boric acid-treated cellulose derived from oil palm empty fruit bunches, consistent with the scope of this study. The OFAT approach was therefore deliberately adopted to establish a baseline study and identify suitable treatment conditions. Fourier transform infrared spectroscopy (FTIR) was used to examine chemical changes, particularly the formation of borate esters and the reduction in hydroxyl content. X-ray diffraction (XRD) provides insights into changes in crystallinity, helping to understand whether structural order is maintained after modification. Finally, thermogravimetric analysis (TGA) quantifies thermal stability by analysing degradation profiles and residual mass at elevated temperatures. The anticipated improvements in thermal stability through BA treatment could transform OPEFB cellulose into a more durable, flame-resistant, and versatile material. This modification could expand the industrial uses of OPEFB cellulose, particularly in sectors where materials must withstand heat and moisture without compromising structural integrity. Additionally, this study supports the sustainable utilisation of agricultural waste, offering a pathway to create value-added products from OPEFB while reducing reliance on non-renewable materials.

2. Materials and Methods

2.1 Materials

Oxalic acid dihydrate ($C_2H_2O_4$, $\geq 99\%$), choline chloride ($C_6H_{14}ClNO$, $\geq 98\%$), boric acid, sodium hydroxide pellets (NaOH, 98%), and hydrogen peroxide (H_2O_2 , 35%) were of analytical grade. All chemicals were purchased from Sigma-Aldrich, USA, except for the latter two, which were obtained from Hamburg (Germany) and System (Malaysia), respectively.

2.2 Chemical treatment of OPEFBs

OPEFBs were collected from oil palm plantations in Raub, Pahang, cleaned to remove dirt and debris, cut into smaller pieces, and ground into powder. The ground OPEFBs sample was chemically treated as described in previous studies [13]. The material was heated in hot water at 90 °C for 1 h to remove soluble impurities. After treatment, the OPEFBs were filtered and dried overnight. The dried EFBs were subjected to alkaline delignification with 4% (w/v) sodium hydroxide (NaOH). This treatment broke down lignin and other non-cellulosic materials. The mixture was filtered, and the residue was rinsed with distilled water three times for 15 min each, then filtered and dried overnight. The pretreated OPEFBs were bleached with 20% (v/v) hydrogen peroxide (H_2O_2) solution for 2 h. The samples were then filtered under vacuum, rinsed three times (15 min each), and dried overnight to enhance cellulose purity by removing residual lignin and coloured compounds. To remove hemicellulose and further purify the cellulose, the dried and bleached OPEFBs were treated with a deep eutectic solvent (DES) composed of choline chloride and oxalic acid for 90 min, using a 1:10 solid-to-liquid ratio. Post-treatment, the material was filtered under vacuum and dried overnight. After DES treatment, the OPEFBs underwent three additional bleaching cycles with 20% (v/v) hydrogen peroxide under the same conditions, each followed by overnight drying. The bleached cellulose was then sonicated in three consecutive runs with 40–45% amplitude at 80% intensity (280 W) in a cold-water bath using an ultrasonicator (Fisherbrand™ Model 705 Sonic Dismembrator). After sonication, the material was filtered under vacuum and dried overnight, yielding highly purified cellulose ready for chemical modification.

2.3 Cellulose Modification using boric acid (C-BA)

The one-factor-at-a-time (OFAT) approach was used to screen key process parameters, including BA concentration, reaction time, and temperature, as tabulated in Table 1. A mixture of OPEFB cellulose and BA was prepared in a 1:5 ratio in a centrifuge tube. The mixture was then placed in an incubator shaker to allow the reaction to proceed under controlled conditions.

Table 1. The parameters of one-factor-at-a-time (OFAT) approach		
Parameters	Values	Remarks
Boric acid concentration (BAC) (% w/v)	1	C-BA_c1%
	3	C-BA_c3%
	5	C-BA_c5%
Reaction time (h)	2	C-BA_t2h
	3	C-BA_t3h
	4	C-BA_t4h
Temperatures (°C)	25	C-BAs_25 °C
	40	C-BAs_40 °C
	55	C-BAs_55 °C

2.3.1 Screening of boric acid concentration (C-BAC)

Boric acid was added to the purified cellulose at varying concentrations (e.g., 1, 3, and 5% (w/v)) while keeping the reaction time constant at 2 h and the temperature at 25 °C. All samples, including one control sample of cellulose not treated with BA, were analysed using FTIR. The concentration that yields the most favourable cellulose properties was selected for further screening.

2.3.2 Screening of reaction time

Using the best BA concentration from the previous step, the reaction time was varied (2, 3 and 4 h) while maintaining a constant temperature of 25 °C. All samples, including one control sample of cellulose not treated with BA, were analysed using FTIR. The reaction time that yielded the most favourable cellulose properties was selected for further screening.

2.3.3 Screening of temperature

Finally, the temperature was varied (25 °C, 40 °C, and 55 °C) while keeping both the BA concentration and reaction time constant at their best values. All samples, including one control sample of cellulose not treated with BA, were analysed using FTIR. We selected the temperature that yielded the most favourable cellulose properties for further XRD and TGA analysis.

2.4 Characterisation and Analysis

To evaluate the effectiveness of the cellulose extraction and subsequent modification processes, we subjected the final samples to a series of analytical techniques as described in our previous study [13–14]. Fourier transform infrared spectroscopy (Perkin Elmer, Norwalk, USA) was used to identify functional groups in the samples and to confirm the chemical modifications introduced by BA treatment. The spectral regions were set between 400 and 4000 cm⁻¹. Thermogravimetric analysis (TGA SDTA 851e Mettler Toledo, USA) assessed the thermal stability of the modified cellulose by measuring weight loss over increasing temperatures. Meanwhile, X-ray diffraction (Bruker D8 Advance, Germany) was conducted to examine the crystallinity and structural organisation of the cellulose. Eq. (1) below was used to determine the crystallinity index of both samples:

$$\text{Crystallinity index} = \frac{I_{200} - I_{am}}{I_{200}} 100 \quad (1)$$

where I_{200} is the height of the 200 peaks, representing both crystalline and amorphous regions, while I_{am} is the lowest height between the 200 and 110 peaks, which represents the amorphous region [15].

3. Results and Discussion

3.1 Screening and Analysis of Boric Acid Modification

The FTIR spectrum of cellulose treated with BA is shown in Figure 1(a) for different BA concentrations, (b) for different temperatures, and (c) for different reaction times. The overall pattern of peak width and shape is similar across all samples. However, cellulose treated with boric acid (C-BA) showed a decrease in the peak intensities compared to the untreated cellulose (UC). Moreover, it seems that the disappearance of new bands is probably due to the comparatively lower concentration of BA in the formulation. The significant differences across all spectra are evident in the spectral band related to the free hydroxyl group of cellulose treated with BA. Cellulose treated with high BA concentration showed a relatively low spectral band compared to low BA concentration and untreated cellulose. However, the peaks observed at 1600, 1500, and 1450 cm⁻¹, corresponding to the benzene ring, overlapped with the B–O–B and B–O–C stretching vibration bands [16]. This result indicates better crosslinking between cellulose and BA, as reported in a previous study [6]. In contrast, changes in temperature or reaction time did not significantly affect the chemical structure of cellulose treated with BA. Only a slightly higher peak intensity of the hydroxyl group (-OH) was observed for cellulose treated with BA at 55 °C. The peaks in all samples treated at either longer (4 h) or shorter (2 h) reaction times showed similar intensities.

Figure 2 shows the diffraction patterns of untreated and BA-treated cellulose at various temperatures. Although the overall profiles remain similar for all samples, the treated cellulose exhibits lower diffraction intensities than the untreated control. Furthermore, as summarised in Table 2, the chemical modification slightly reduced overall crystallinity. Notably, the intensity of the sample treated with BA at 25 °C is almost like that of the untreated cellulose. The decrease in diffraction intensity can be attributed to BA-induced swelling of cellulose. BA may attack the amorphous regions of cellulose, damaging the crystalline structure. The intermolecular and intramolecular hydrogen bonds in the crystalline regions of cellulose are disrupted and partially replaced by B–O–C bonds during the treatment process [16]. This is supported by the lower intensity of hydroxyl group peaks observed in the FTIR spectra. The diffraction patterns for both untreated and BA-treated cellulose at various temperatures are presented in Figure 2. Although the overall profiles remain similar for all samples, the treated cellulose exhibits lower diffraction intensities than the untreated control. Furthermore, as summarised in Table 2, the chemical modification slightly reduced overall crystallinity.

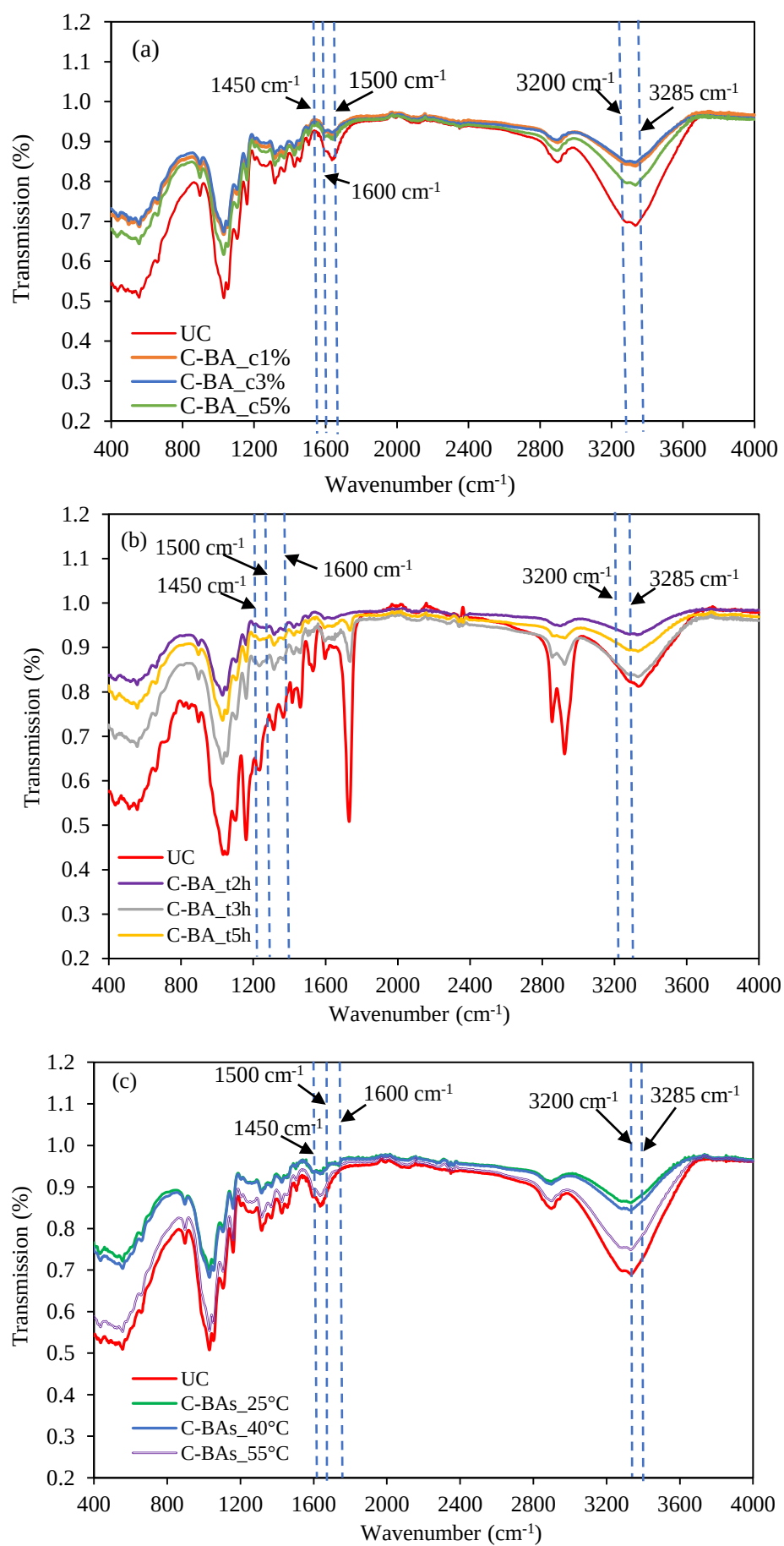


Figure 1. FTIR results for varying (a) concentration of boric acid, (b) reaction time and (c) temperatures

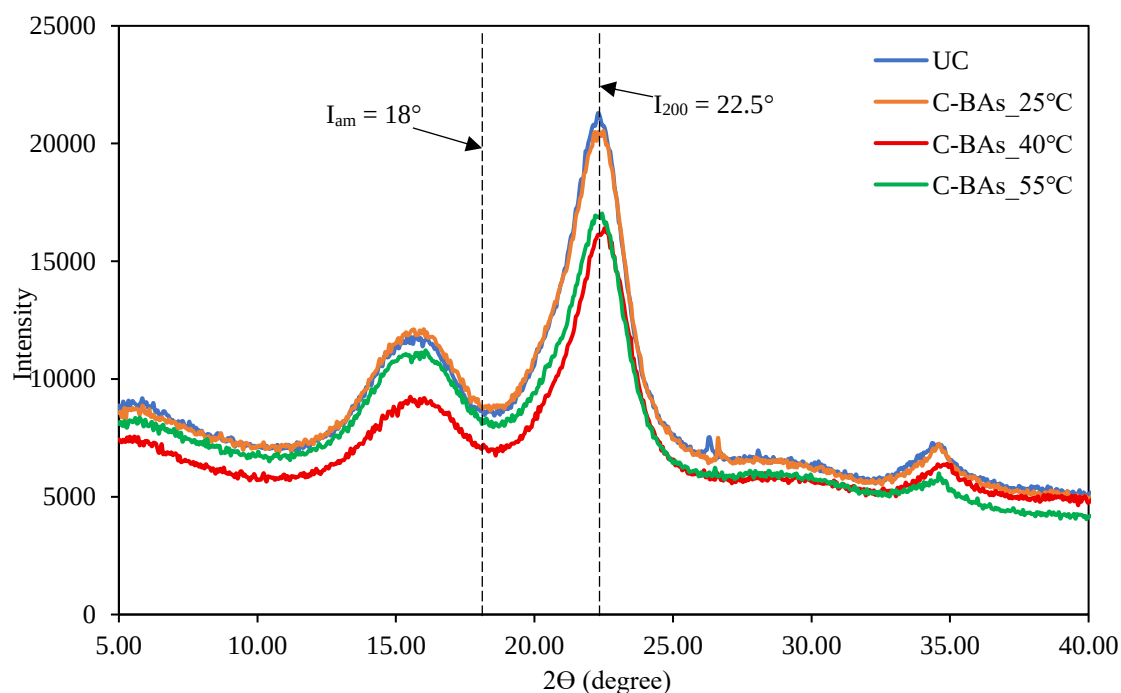


Figure 2. XRD Results of both untreated (UC) and boric acid-treated cellulose (C-BA) samples at different temperatures

Table 2. Degree of crystallinity of both untreated (UC) and boric acid-treated cellulose (C-BA) samples

Samples	Crystallinity index (%)	Crystallinity ratio (I_{200}/I_{am})
Untreated cellulose (UC)	58	2.38
C-BAs_25°C	57	2.30
C-BAs_40°C	56	2.28
C-BAs_55°C	51	2.03

As shown in Figure 3, all untreated and treated oil palm biomass cellulose samples exhibited single-step degradation, as indicated by a single peak in the derivative thermogravimetry (DTG) chromatograms. The results indicate that the degradation was mainly due to one primary component, with a degradation temperature range of 226–402 °C for the treated cellulose, while the untreated sample ranged from 243–401 °C. T_{max} values of BA-treated cellulose samples were lower than those of untreated cellulose samples (345.18 °C) (Table 3). This result showed that boric acid decreased the temperature at the maximum degradation rate, which might be due to changes in cellulose crystalline behaviour. Additionally, the free hydrogen ion, generated from the complexation of the boric acid and cellulose, catalyses the degradation of cellulose [16–17].

Table 3. Data obtained from TGA and DGA curves

Properties	Untreated cellulose (UC)	C-BAs at 25 °C	C-BAs at 40 °C	C-BAs at 55 °C
Residual (%)	20.1375	11.6299	11.8708	15.6609
Weight (mg)	0.74	0.407	0.4202	0.5685
T_{onset} (°C)	322.94	318.89	317.16	318.65
T_{peak} (°C)	345.18	341.45	339.05	340.6

TGA offered a macroscopic view of the thermal stability of both untreated and BA-treated cellulose samples. Untreated cellulose showed the highest thermal stability, as shown in Figure 4. In contrast, the boric acid-treated samples showed lower final residues: the 25 °C sample retained about 11.6%, the 40 °C sample about 12%, and the 55 °C sample about 15.7% (Table 3). The TGA curves consistently showed that the untreated sample maintained a higher mass throughout the temperature range. These results initially seem to contradict previous studies, which reported that the char residue increased when the thermal degradation rate of borated wood decreased [16]. The incorporation of borate groups likely facilitates alternative degradation pathways, increasing volatile compound production and reducing final residue. Thus, while BA treatment enhances certain aspects of thermal stability, the preservation of the native cellulose structure remains critical for maximising char yield and overall thermal stability [16].

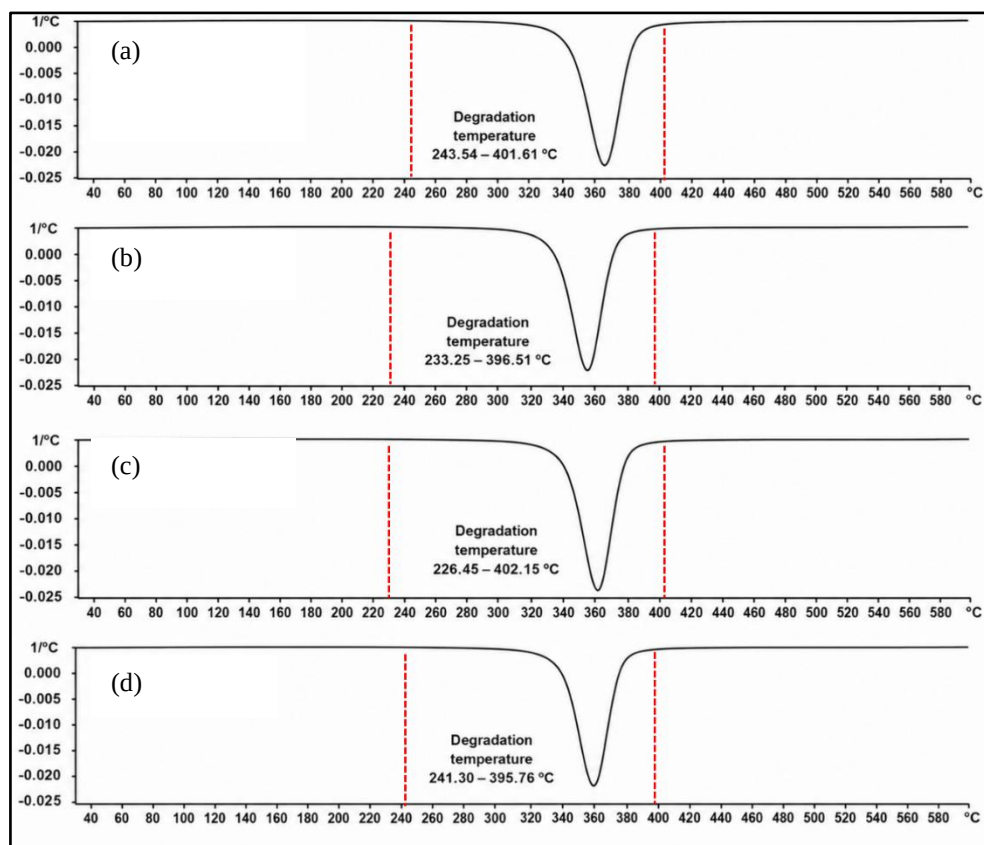


Figure 3. Derivative thermogravimetry (DTG) curve of (a) untreated (UC) and boric acid-treated cellulose (C-BAs) samples at different temperatures (b) 25 °C (c) 40 °C, and (d) 55 °C

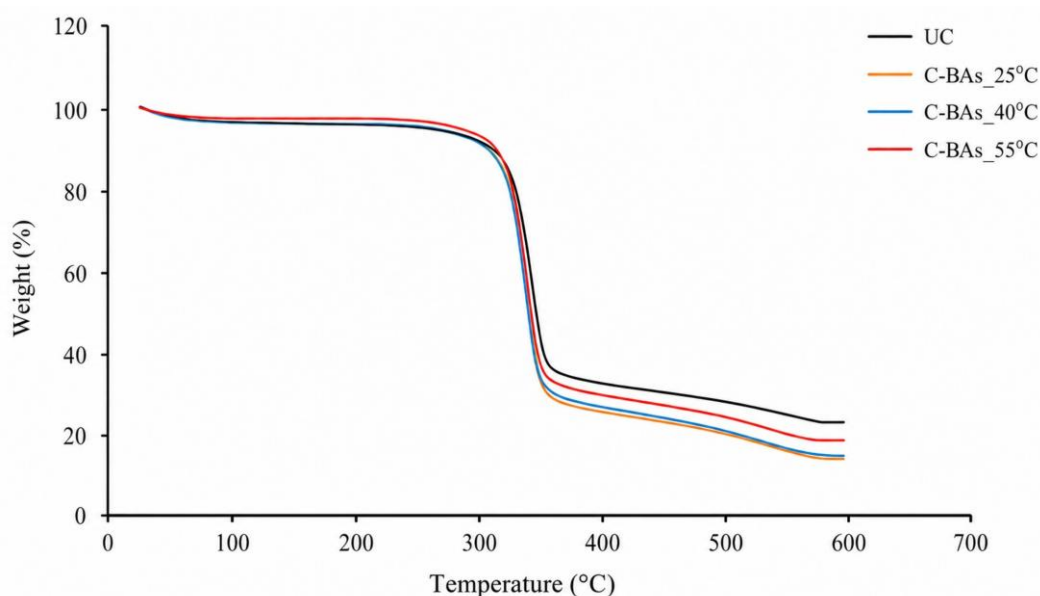


Figure 4. Thermogravimetric analysis (TGA) curve of both untreated (UC) and boric acid-treated cellulose (C-BAs) samples at different temperatures

4. Conclusion

Treating cellulose with 3% (w/v) BA at 25 °C for 2 h showed improved thermal stability. Based on FTIR analysis, treated cellulose showed a significant reduction in free hydroxyl groups, indicating successful crosslinking through esterification and efficient water elimination, which are critical for thermal stability. Furthermore, the cellulose structure remained intact, preserving its characteristic backbone bands. The XRD patterns are similar across all samples; however, the diffraction intensities are lower than those of the untreated cellulose sample. Although TGA revealed that untreated cellulose retained the highest char yield, the C-Bas_25 °C-treated sample still demonstrated reasonable thermal

performance, with slight reductions attributed to partial amorphisation or catalytic effects of borate groups. This study will serve as a basis for future work to determine optimal conditions for chemical modification of cellulose.

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Declaration of Competing Interests

The authors declare no conflicts of interest.

Credit Author Contribution Statement

Dzun Noraini Jimat: Methodology; Conceptualisation; Analysis; Visualisation; Supervision; Writing – review & editing
Nur Aleeya Maisara: Methodology; Data curation; Writing - original draft.
Sarina Sulaiman: Investigation; Visualisation; Writing – review & editing.

Availability of Data and Materials

Data sharing is not applicable to this article as no new datasets were generated or analysed in this study.

Generative Artificial Intelligence Disclosure

No generative artificial intelligence tools were used in the preparation of this manuscript. All intellectual content is original and solely attributable to the authors. AI-based tools were used solely for language editing and grammar refinement.

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