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Chemical, Structural and Morphological Characteristics of Sugarcane Bagasse Fibers Under Different Alkaline Treatment Durations

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Abstract. *This study investigated the influence of alkaline treatment duration on the chemical composition, structural characteristics, and morphology of sugarcane bagasse (SCB) fiber, a widely available lignocellulosic byproduct of the sugar industry. SCB fibers were treated with 1.0 M sodium hydroxide (NaOH) for 30 min, 1 h, 2 h, and 4 h, with untreated fibers serving as the control. Changes in chemical composition were determined through analysis of extractives, Klason lignin, holocellulose, and cellulose contents, while structural and morphological modifications were characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and optical microscopy. Increasing alkali treatment duration progressively reduced hemicellulose content from 19.39% to 15.09% after 2 h and Klason lignin content from 20.29% to 4.39% after 4 h, indicating effective removal of non-cellulosic constituents. In contrast, cellulose content increased slightly from 44.94% in the untreated fiber to 46.72% after 2 h of treatment. FTIR analysis revealed a marked attenuation of lignin- and hemicellulose-associated absorption bands at 1730, 1600, 1510, 1460, 1245, and 834 cm^{-1}, whereas cellulose-related bands at 1425 and 1030 cm^{-1} remained largely unchanged. XRD results showed that the crystallinity index (CrI), calculated using the Segal method, increased from 37.78% for untreated SCB to a maximum of 49.80% after 2 h of treatment before decreasing to 45.65% after 4 h, suggesting that excessive alkali exposure may partially disrupt the ordered cellulose structure. Optical microscopy further confirmed progressive surface roughening and fiber defibrillation with increasing treatment duration. Although all measurements were conducted as single determinations and should therefore be interpreted as relative comparisons, the combined results indicate that a 2 h alkaline treatment provides the most favorable balance between non-cellulosic component removal and cellulose crystallinity enhancement. These findings highlight the potential of optimally treated SCB fibers for applications in biocomposites, papermaking, and biodegradable products, although further studies on mechanical, thermal, and end-use performance are required.*

Keywords: *sustainability; biomass; crystallinity; natural fiber; agricultural waste; polymer composites.*

Type of the Paper: Regular Article.

1. Introduction

Composite materials are generally composed of a base matrix reinforced with one or more

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types of fiber. The global natural fiber-reinforced polymer composite market continues to expand, with its market volume estimated at approximately 3.7 million tonnes in 2022 and projected to grow at a compound annual growth rate (CAGR) of more than 9% over the 2022–2027 period [1]. Synthetic fibers impose a heavier environmental burden and require more energy-intensive processing, which often involves hazardous substances such as sulfuric acid and petroleum derivatives. These environmental and health concerns continue to drive research toward more sustainable, environmentally friendly reinforcing materials [2].

Plant-derived natural fibers are increasingly favored for their recyclability, biodegradability, sustainability and cost-effectiveness, with appropriate surface modification further improving their compatibility with polymer matrices [3,4]. By converting agricultural residues into usable reinforcing materials, natural fibers add value to otherwise discarded biomass and support waste-valorization and circular-economy strategies [5]. Natural fibers such as SCB, kenaf, bamboo and jute also offer competitive strength-to-weight ratios, making them attractive for lightweight structural applications, while their abundance and low cost enhance the economic feasibility of large-scale manufacturing [6–9].

Despite these advantages, natural fibers possess intrinsic limitations that restrict their direct use in composites, including high moisture absorption, hydrophilicity, limited durability, poor interfacial adhesion with hydrophobic matrices and batch-to-batch variability [10]. Non-cellulosic impurities such as lignin, hemicellulose, waxes and pectin weaken fiber-matrix bonding, reducing tensile strength and long-term durability, which underscores the importance of surface modification for improving fiber-matrix adhesion [11].

Agriculture is among the largest sources of global waste and open burning or improper disposal of residues such as SCB contributes to serious air- and soil-pollution problems while releasing pollutants including smoke, NO_2, SO_2 and CH_4 [12]. Global sugarcane production reached approximately 1.95 billion tonnes in 2023, generating an estimated 540–585 million tonnes of bagasse annually, yet SCB remains underutilized relative to its potential as an industrial raw material for green concrete, bio-asphalt, biomass energy and cellulosic fiber applications [7,13,14]. Converting this abundant residue into a high-value reinforcing fiber therefore offers both an environmental and an economic benefit.

Alkaline treatment (mercerization) using sodium hydroxide is one of the most widely used chemical methods for purifying natural fibers. NaOH cleaves the ester linkages between lignin and hemicellulose and removes acetyl and uronic substitutions, thereby dissolving amorphous non-cellulosic material [15,16]. The outcome depends on both NaOH concentration and exposure time. For bagasse, Anggono et al. [15] treated fibers with 10% vol. NaOH at 60–70 °C for 2, 4 and 6 h: weight loss increased from 40.5% to 45% and 57.75%, FTIR showed that lignin- and

hemicellulose-related bands had already disappeared after 2 h with no further structural change at 4 and 6 h, and the intensity of the main XRD peak at 22° decreased with treatment time, which was attributed to further cellulose loss, while SEM showed partially decomposed fibers after 4 h. These conditions are considerably more severe than the 1.0 M NaOH used in the present study. For other natural fibers, moderate treatment improved crystallinity and fiber properties: the optimized 60 min treatment of Perotis indica fibers with 5% NaOH raised the crystallinity index from 48.3% to 55.43% [17], tensile strength of banyan aerial root fibers peaked at 120 min and declined with longer immersion [18], and bamboo fibers showed an optimum at 4 wt.% NaOH and 12 h [19]. The effects of NaOH treatment on the chemical, mechanical and thermal properties of natural fiber composites have also been reviewed [20]. However, these studies differ in fiber source, NaOH concentration, temperature and analytical coverage, so the optimal treatment duration for SCB under mild 1.0 M NaOH cannot be inferred from them. Insufficient treatment leaves residual impurities on the fiber surface, whereas excessive treatment may reduce crystallinity and weaken the fiber.

To address this gap, the present study evaluated the effect of alkaline (1.0 M NaOH) treatment duration for 30 min, 1 h, 2 h and 4 h, compared against an untreated control on the chemical composition, functional-group profile, crystallinity and surface morphology of SCB fiber. The findings aim to identify the treatment duration that best balances the removal of non-cellulosic impurities with the preservation of cellulose crystallinity, supporting the development of alkali-treated SCB as a sustainable lignocellulosic reinforcement for bio-composite, papermaking and biodegradable-material applications.

2. Materials and methods

2.1. Materials

SCB used in this study was obtained as waste from a local vendor selling fresh sugarcane juice in Sungai Ara, Penang, Malaysia. Analytical-grade sodium hydroxide (NaOH) was used to prepare a 1.0 M treatment solution.

2.2. Preparation and Alkaline Treatment of SCB Fibers

The collected SCB was cleaned and repeatedly washed with water until a neutral pH (pH 7) was achieved, to eliminate contaminants and reduce the risk of fungal contamination; the outer rind was removed manually during this cleaning and washing stage (Fig. 1(a)). The cleaned bagasse was oven-dried at 60 °C for 1 h and subsequently air-dried for 24 h (Fig. 1(b)), giving rind-free SCB (Fig. 1(c)). The dried bagasse was divided into five groups (Table 1): an untreated control (Sample A) and four samples soaked in 1.0 M NaOH (Fig. 1(d)) for 30 min, 1 h, 2 h and 4

h (Samples B–E, respectively) (Fig. 1(e)). For each treatment duration, 15 g of SCB fiber was immersed in 500 mL of 1.0 M NaOH (fiber-to-liquor ratio of 1:33.3, w/v) at room temperature, while all other parameters were kept constant. After treatment, samples were washed with distilled water until neutral pH was restored, to arrest further alkaline action, and oven-dried at 60 °C for 24 h (Fig. 1(f)) prior to characterization.

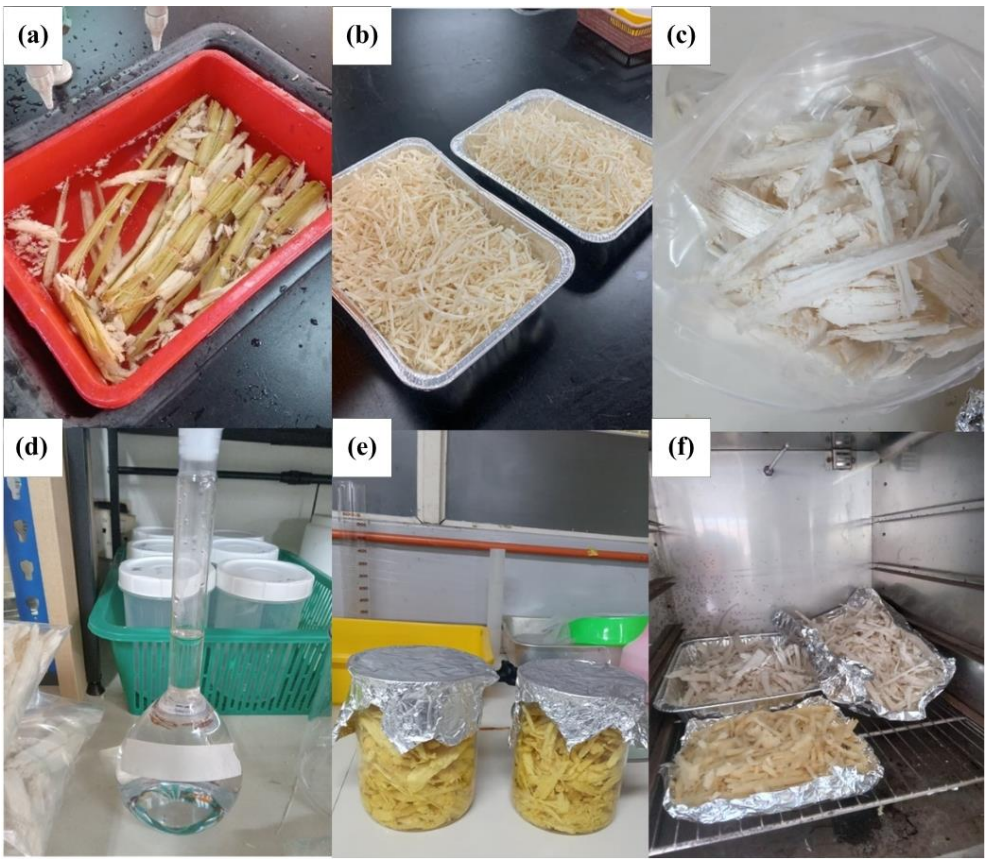


Fig. 1. Sample preparation: (a) cleaning and washing of SCB, (b) oven-drying of cleaned SCB, (c) rind-removed SCB (untreated), (d) preparation of 1.0 M NaOH solution, (e) soaking of SCB in 1.0 M NaOH and (f) oven-drying of alkali-treated SCB.

Table 1. Sample codes and corresponding alkaline soaking durations.

Sample	Soaking time
A	Untreated (control)
B	30 min
C	1 h
D	2 h
E	4 h

2.3. Chemical Composition Analysis

The chemical composition of untreated and treated SCB was determined following TAPPI methods: extractive content (TAPPI T 204 cm-97), Klason (acid-insoluble) lignin (TAPPI T 222 om-98) and cellulose content (TAPPI T 9 m-54, with modification as described below). Extractive content was quantified by Soxhlet extraction of oven-dried samples with toluene–ethanol (2:1 v/v; 267 mL toluene : 133 mL ethanol) for 4–5 h until the extract became colorless, followed by solvent

recovery by rotary evaporation at 85 °C and oven-drying of the residue at 105 °C. Holocellulose was isolated from the extractive-free residue by the sodium chlorite–acetic acid method [21], using $NaClO_2$ (4.5 g in total) and acetic acid in a 75 °C water bath for 3 h. Cellulose was determined from the holocellulose by acid hydrolysis with 1.3% sulfuric acid in a boiling water bath for 2 h (modified from TAPPI T 9 m-54). Hemicellulose was calculated as the difference between holocellulose and cellulose content. Klason lignin was determined gravimetrically after hydrolysis of the extractive-free material with 72% H_2SO_4 at lower than 20 °C, followed by dilution and 4 h of reflux. All components were calculated relative to the oven-dry weight of the sample analyzed (untreated or treated SCB, respectively). Acid-soluble lignin, ash and other minor components were not determined, so the components do not sum to 100% and the reported lignin values represent Klason lignin only. Each determination was performed once per sample.

2.4. Characterizations

FTIR spectra were recorded on a Perkin Elmer Spectrum One spectrometer fitted with an ATR accessory over 4000–400 cm^{-1}, using 16 scans. XRD patterns were recorded using an Empyrean Alpha 1 diffractometer (Malvern Panalytical) with Ni-filtered Cu Kα radiation (λ = 1.5406 Å) operated at 40 kV and 30 mA over 2θ = 10–90° with a step size of 0.02° and a scan rate of 2 °/min. The crystallinity index (CrI) was calculated by the Segal method from the intensities at 2θ ≈ 22° (I_{200}) and 2θ ≈ 18° (I_{am}) as shown in Eq. (1) [22]. The surface morphology was examined using a Leica EMSPIRA 3 digital stereo optical microscope at 5× magnification; samples were sectioned to fit the stage and secured with adhesive tape under uniform LED illumination, and representative images were captured for each sample. FTIR spectra and XRD patterns were each recorded once per sample and are used for qualitative and relative comparison.

$$\mathrm{CrI}\ (\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100 \quad (1)$$

3. Results and Discussion

3.1. Chemical Composition

The chemical composition of the untreated and NaOH-treated SCB samples is summarized in Table 2. The extractive content decreased steadily from 5.22% (Sample A) to 0.87% (Sample E), consistent with the effective solubilization and removal of low-molecular-weight, non-structural components such as waxes, fats, resins and phenolics through cleavage of ester bonds within the cell-wall matrix, a trend previously reported for alkali-treated lignocellulosic biomass [15,16].

Table 2. Chemical composition (%) of untreated and alkaline-treated SCB samples.

Sample	Cellulose (%)	Hemicellulose (%)	Klason Lignin (%)	Extractives (%)	Holocellulose (%)
A	44.94	19.39	20.29	5.22	64.33
B	45.20	18.28	18.60	3.20	63.48
C	45.96	16.49	11.14	2.26	62.45
D	46.72	15.09	7.20	1.39	61.81
E	46.10	16.33	4.39	0.87	62.43

Note: Values are from single determinations and are used for relative comparison among treatment durations; differences of about 1% or less are not interpreted.

Klason lignin content decreased markedly and continuously from 20.29% (Sample A) to 18.60%, 11.14%, 7.20% and 4.39% for Samples B, C, D and E, respectively. This progressive delignification is consistent with the cleavage of ether and ester linkages in the lignin–carbohydrate network by NaOH [16,23]. Because lignin acts as a barrier restricting cellulose accessibility, its removal is essential for applications such as biofuel production and bio-composite reinforcement.

Hemicellulose content decreased from 19.39% (Sample A) to 15.09% (Sample D) and was 16.33% in Sample E; because the determinations were not replicated, the small difference between D and E is not interpreted as a real change. Substantial hemicellulose removal has been reported for bagasse under alkaline conditions, for example up to 75.64% with 5% NaOH assisted by freeze–thaw and hemicellulose decreased with increasing NaOH concentration in autoclaved bagasse, although those studies used higher NaOH concentrations or temperature than the present work [24,25].

Cellulose content increased slightly from 44.94% (Sample A) to 46.72% (Sample D), reflecting relative enrichment after removal of non-cellulosic material rather than new cellulose formation. The value in Sample E (46.10%) is similar to that of Sample D; because the determinations were not replicated, this small difference cannot be distinguished from experimental variation, and the compositional data do not provide evidence of cellulose degradation after 2 h.

3.2. FTIR Analysis

FTIR spectra of the untreated and alkaline-treated SCB samples are shown in Fig. 2. The peak at 1030 cm^{-1}, assigned to C–O stretching of primary alcohols in cellulose and the peak at 1425 cm^{-1}, assigned to CH_2 bending vibrations of cellulose, showed no major change across treatment durations, indicating that alkaline treatment did not substantially alter the cellulose backbone; the improved visibility of these bands is instead attributed to the removal of surrounding lignin and hemicellulose rather than an actual increase in cellulose content [26,27].

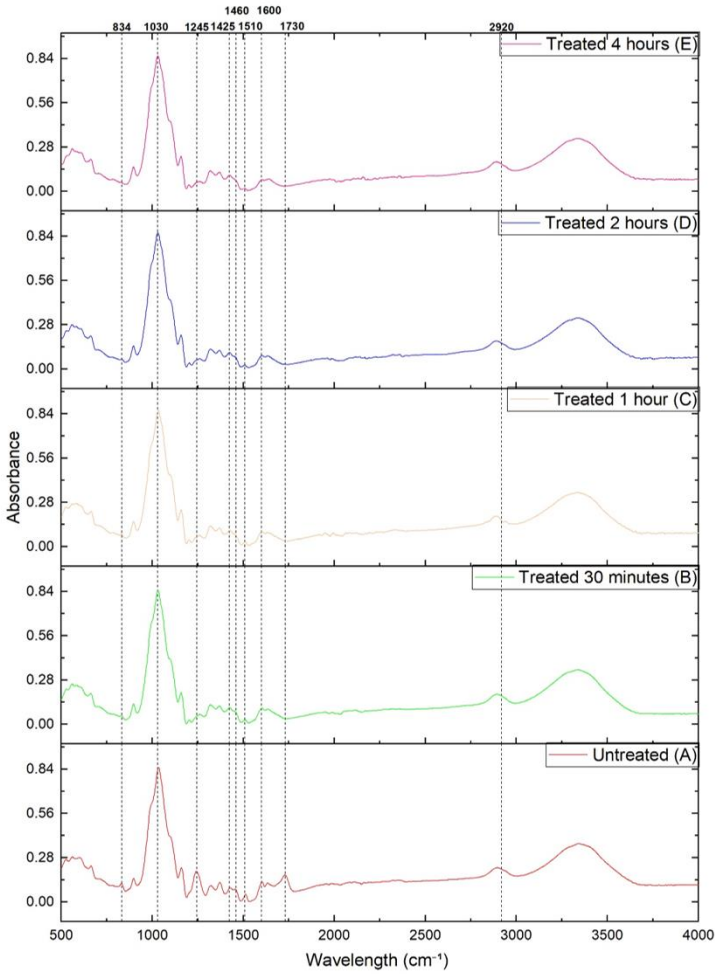


Fig. 2. The FTIR result of all the samples at different soaking time.

The absorption bands at 1730 cm^{-1} (C=O stretching of uronic ester and acetyl groups in hemicellulose) and 1245 cm^{-1} were already markedly attenuated after 30 min and were weakest in the 2 h and 4 h samples, indicating cleavage of ester linkages and solubilization of hemicellulose. The lignin-associated aromatic bands at 834 cm^{-1} (C–H out-of-plane bending), 1510 and 1600 cm^{-1} (aromatic C=C skeletal and stretching vibrations) and 1460 cm^{-1} (C–H bending) also decreased after alkaline treatment, consistent with the cleavage of ether and ester bonds within the lignin structure [28,29]. The aliphatic C–H stretching band at 2920 cm^{-1} showed only a slight decreasing trend, attributed to the removal of surface films and extractives. The FTIR results indicate that the spectral changes mainly reflect the removal of amorphous, non-cellulosic components, with no major change in the cellulose-related bands [30,31]. Similarly, Anggono et al. [15] reported that lignin bands (1508 and 1233 cm^{-1}) and the hemicellulose carbonyl band (1723 cm^{-1}) were already absent after the shortest treatment (2 h), with no further spectral change at 4 and 6 h. FTIR alone, however, cannot exclude changes in cellulose chain length.

3.3. XRD Analysis

Cellulose can adopt several polymorphic forms (cellulose I–IV). Conversion of cellulose I

to cellulose II is characterized by diffraction peaks near 12° and 20° [32]. For bagasse, it required about 15% NaOH in conventional mercerization and 5% with freeze–thaw assistance, both higher than the ≈ 4% (1.0 M) used here [24]. Consistently, no such peaks were observed in any sample (Fig. 3). CrI was calculated by the Segal method, which is empirical and sensitive to background and noise; CrI values are therefore used for relative comparison only [33,34].

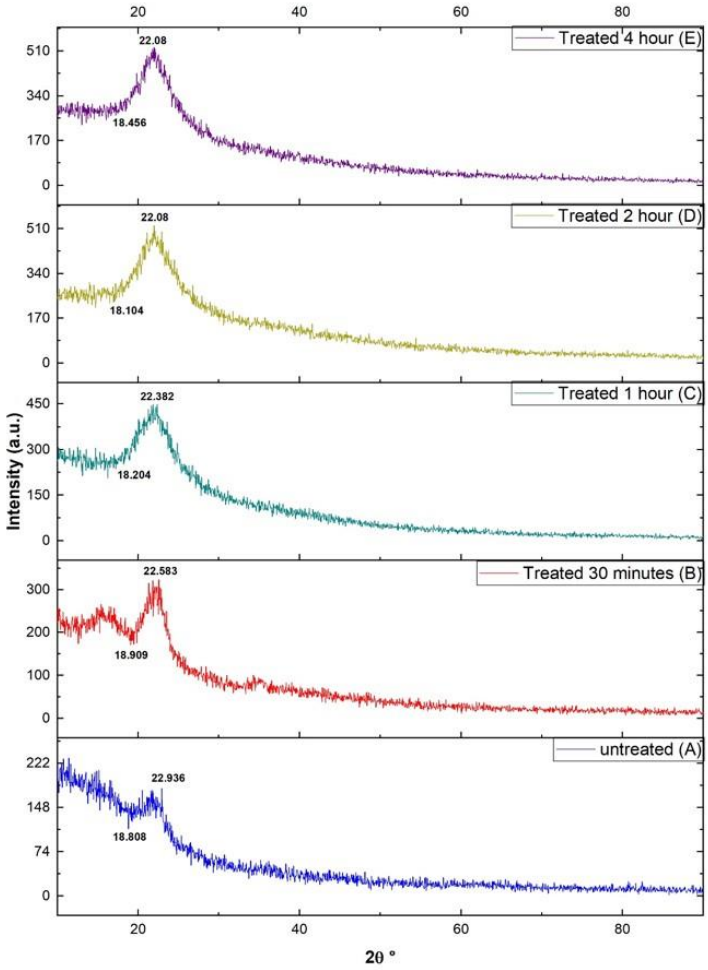


Fig. 3. The XRD result of all samples at different soaking time.

The CrI values obtained from the XRD patterns are listed in Table 3. The CrI increased from 37.78% (Sample A) to 43.61% (30 min), 43.75% (1 h) and a maximum of 49.80% (2 h), which can be attributed to the removal of amorphous, non-cellulosic material and the consequent relative enrichment of ordered cellulose [15]. Beyond 2 h, the CrI declined to 45.65% after 4 h. Although still higher than the untreated control, this decline suggests that prolonged NaOH exposure may reduce relative crystallinity, for example through swelling and disruption of hydrogen bonding. Because CrI was obtained from a single pattern and the Segal method depends on background intensity, small differences (e.g., between 30 min and 1 h) should be interpreted with caution. A decrease in the intensity of the main 22° reflection with longer NaOH treatment has also been reported for bagasse, although under more severe conditions (10% vol. NaOH, 60–70 °C, 2–6 h)

and without calculation of a crystallinity index [15].

Table 3. Crystallinity index (CrI) of untreated and alkaline-treated SCB samples.

Sample	I_{200} (crystalline intensity)	I_{am} (amorphous intensity)	CrI (%)
A	180	112	37.78
B	321	181	43.61
C	448	252	43.75
D	502	252	49.80
E	517	281	45.65

3.4. Morphological Analysis

Optical micrographs of the untreated and alkaline-treated SCB fibers are presented in Fig. 4. The untreated fiber for Sample A in Fig. 4 displayed a smooth, compact surface with clearly defined rectangular cellular structures, consistent with an intact lignin–hemicellulose matrix binding the cellulose-rich fibril bundles. After 30 min of treatment (Sample B), the fiber surface became visibly less dense, with increased inter-fiber separation suggesting the onset of amorphous-matrix removal. Progressive morphological change continued after 1 h (Sample C), where the original cellular pattern became less distinct and the underlying fibrillar structure was increasingly exposed.

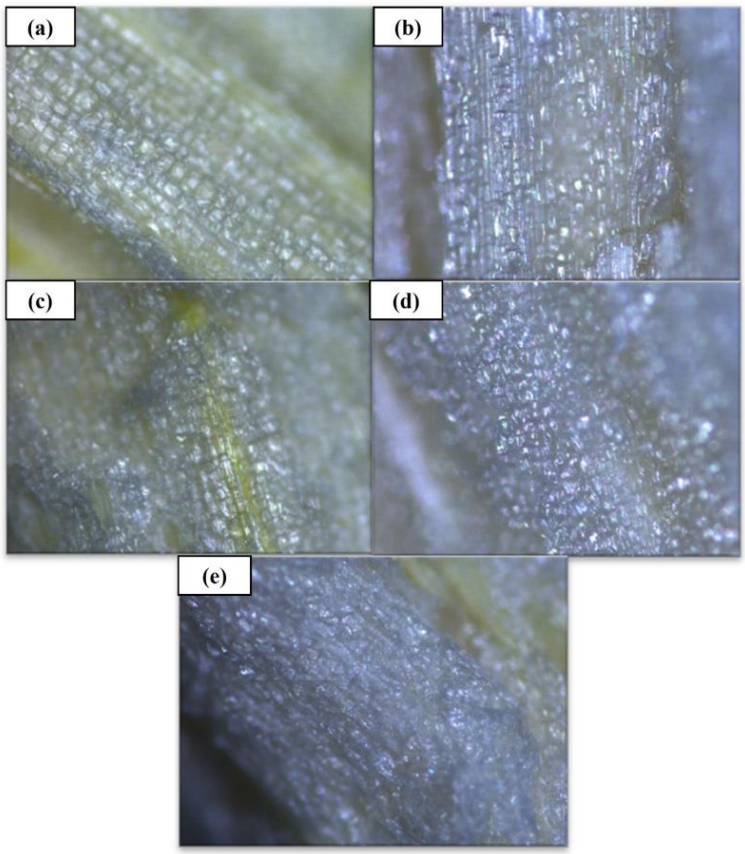


Fig. 4. Optical micrographs (5× magnification) of (a) untreated SCB (Sample A) and SCB treated with 1.0 M NaOH for (b) 30 min, (c) 1 h, (d) 2 h and (e) 4 h.

Samples D (2 h) and E (4 h) showed rougher, less compact surfaces with loss of the original regular cellular pattern, consistent with removal of lignin, hemicellulose and other non-cellulosic components. Removal of amorphous constituents and separation of fiber bundles after alkali treatment has likewise been observed in other natural fibers [17,35]. At 5× magnification, however, the optical micrographs only allow a qualitative comparison, and the apparent differences between Samples D and E should be confirmed by higher-resolution imaging such as SEM.

4. Conclusions

This study evaluated the influence of alkaline treatment duration using 1.0 M NaOH (30 min, 1 h, 2 h, and 4 h) on the chemical composition, structural characteristics, and morphology of SCB fiber. Increasing treatment duration progressively reduced lignin and extractive contents and, up to 2 h, decreased hemicellulose content, while slightly increasing the relative cellulose content. FTIR analysis confirmed the removal of non-cellulosic constituents through the attenuation of lignin- and hemicellulose-associated absorption bands, whereas cellulose-related bands remained largely unchanged. XRD results revealed that the CrI increased from 37.78% in untreated SCB to a maximum of 49.80% after 2 h of treatment before declining to 45.65% after 4 h, indicating that excessive alkali exposure may partially disrupt the ordered cellulose structure. Optical microscopy further demonstrated progressive surface roughening and fiber separation with increasing treatment duration. Among the treatment conditions investigated, 2 h of alkali treatment produced the highest crystallinity and a favorable degree of non-cellulosic component removal, as supported by the chemical composition, FTIR, XRD, and optical microscopy analyses. However, because all measurements were conducted as single determinations and the mechanical, thermal, and interfacial properties of the fibers were not evaluated, this treatment duration should be considered the most promising condition within the tested range rather than a definitive optimum. Overall, alkali-treated SCB fiber demonstrates considerable potential as a sustainable, low-cost lignocellulosic reinforcement for biocomposites, papermaking, biodegradable packaging, and other value-added industrial applications. Future research should incorporate replicated experiments and statistical analyses to validate the observed trends, together with comprehensive evaluations of composite mechanical performance, including tensile, flexural, and impact properties. In addition, thermal stability should be assessed using TGA, while the suitability of treated SCB as a precursor for biochar and activated carbon production should be explored to further expand its industrial utilization and value-added applications.

Data availability statement

Data will be made available on request.

CRediT authorship contribution statement

Nur Inas Nadira Mohamed Harith: investigation, validation, visualization, writing; **Zahran Hafiraj**: writing, editing; **Mageswari Manimaran**: writing; **Mohd Nurazzi Norizan**: research concept, writing, methodology, investigation, validation, visualization, funding acquisition, supervision; **Che Ku Abdullah**: methodology, visualization; **Anton Kuzmin**: research concept, methodology.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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