

Modification of Sugarcane Bagasse Fiber: A Comparative Study of Alkali and TEMPO Treatments

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ABSTRACT

Bagasse fiber, a byproduct of the sugarcane industry, has the potential to be a valuable resource in various industrial applications due to its abundance. This study evaluates the effects of 6% NaOH and TEMPO treatments on the structural, mechanical, and thermal properties of sugarcane bagasse fibers. Sugarcane bagasse fibers were analyzed using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), tensile testing, Fourier Transform Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA). The results indicate that fiber treatment using NaOH significantly increases the Crystalline Index (CI) from 50.95% to 58.38%, while TEMPO oxidation achieves 52.34%. The mechanical testing reveals that tensile stress increases by 85.26 MPa (6% NaOH) and 141.10 MPa (TEMPO), showcasing superior fiber compatibility with TEMPO. FTIR analysis attests to the removal of hemicellulose and lignin, as well as modifications in functional groups, while TGA demonstrates enhanced thermal stability with NaOH-treated fibers showing greater resistance to thermal degradation compared to TEMPO-treated fibers. These findings underscore the potential of chemical treatments to optimize sugarcane bagasse fiber

properties for sustainable applications. By optimizing its natural characteristics through targeted chemical modifications, this study provides valuable insights into utilizing bagasse fiber as a renewable alternative, reducing reliance on synthetic fibers and mitigating environmental impacts.

Keywords-alkali; bagasse fiber; Fourier Transform Infrared Spectroscopy (FTIR); tensile test; TEMPO; thermogravimetric analysis (TGA)

I. INTRODUCTION

Sugarcane is a key commodity in Indonesia, particularly in the sugar production industries, playing an important role in both the economy and agriculture. By the end of 2024, Indonesia's sugar production reached 2.460 million tons, an increase of 190,000 tons (10%) from 2023's 2.27 million tons. However, sugarcane processing generates significant waste, with bagasse accounting for 35-40% of the milled sugarcane weight [1]. Despite its high potential as an industrial raw material, bagasse remains underutilized. Proper processing can enhance its value in sustainable green energy, as demonstrated in previous studies exploring its applications in green concrete [2], bio asphalt [3], and biomass energy [4]. Sugarcane bagasse can also be utilized as a natural fiber, offering a sustainable alternative to synthetic fibers. Natural fibers derived from plant-based organic materials are increasingly favored due to their abundance [5] and growing environmental awareness [6]. These fibers offer several advantages, including recyclability, biodegradability, sustainability, and cost-effectiveness [7, 8]. Their potential has led to increased interest in modifying natural fibers through nanomaterial integration or chemical treatments. Proper modification enhances composite polymer performance, making them more suitable for various applications.

Sugarcane bagasse fiber, a promising material for composite polymer development, is primarily composed of cellulose, lignin, and hemicellulose [9]. Its crystallinity index ranges from 50% to 90% [10]. Chemical treatments, such as alkalization, effectively remove lignin and hemicellulose, increasing cellulose yield [11] and enhancing crystallinity [12]. NaOH treatment breaks down the complex fiber structure, improving cellulose purity. Additionally, TEMPO oxidation facilitates the formation of carboxyl groups, further modifying natural fibers for enhanced functionality [13, 14]. Both alkalization using NaOH and oxidation using TEMPO have different impacts on bagasse fiber properties. Since NaOH and TEMPO treatments influence bagasse fiber properties differently, this study aims to compare their effects. Various analyses were conducted, including SEM for surface morphology, XRD for crystallinity, tensile testing for mechanical properties, FTIR for functional groups, and TGA for thermal degradation.

II. MATERIALS AND METHODS

A. Materials

The study used bagasse from sugarcane waste supplied by a sugar factory in Kebon Agung, District of Malang, Indonesia. TEMPO was obtained from Shanghai Xinglu Chemical Technology, China and NaOH was supplied by CV. Makmur Sejati, Indonesia. Additional materials used were aluminum foil, a sieve, an oven, a spatula, an analytical balance, and a glass beaker.

B. Preparation and Chemical Treatment of Bagasse Fiber

Bagasse fiber was washed four times with distilled water to remove impurities and then soaked in a 6% NaOH solution. After 4 h, the fiber was rinsed with distilled water and then dried in an oven at 60 °C for 24 h. TEMPO treatment was conducted by dissolving 0.1 g NaBr, 3.1 g NaClO, and 0.016 g TEMPO in 300 mL of distilled water. Bagasse fiber (1 wt%) was added and stirred for 1.5 h, 0.1 M HCl was also added, while 0.5 M NaOH was gradually added to maintain a pH of 10. The mixture was homogenized for 15 minutes followed by washing with water until it became neutral. Treated fibers were dried for 4 h at 70 °C in an oven.

C. Methods

1) Morphology Analysis of Bagasse Fiber

Surface morphology of bagasse fibers was analyzed using SEM (FEI Inspect S50, Japan). Before observation, fibers were coated with a thin gold film. Imaging was conducted at an accelerating voltage of 5 kV.

2) Crystallinity Analysis of Bagasse Fiber

Crystallinity was assessed using XRD (PANalytical X'Pert PRO, UK). Specimens (10 mm length) were analyzed with CuK α radiation ($\lambda = 1.54060 \text{ \AA}$) at 30 mA and 40 kV. Scanning diffraction angles 2θ were collected through diffractograms from 10° to 90° at 25 °C. The CI was calculated using:

$$CI = \frac{I_{22} - I_{18}}{I_{22}} \times 100\% \quad (1)$$

3) Functional Group Analysis of Bagasse Fiber

Functional groups were identified using FTIR (Shimadzu Prestige-21, Japan). For accurate data to be obtained, spectral scans were performed in the wavenumber from 400 to 4000 cm^{-1} [15].

4) Thermal Degradation Analysis of Bagasse Fiber

Thermal degradation analysis was conducted using TGA, which utilizes argon gas heating to assess thermal decomposition. Material weight loss during temperature increase is recorded, generating a decomposition curve [16]. In this study, a Simultaneous Thermal Analyzer (STA 6000, PerkinElmer, USA) was used. Testing was conducted at a heating rate of 10 °C/min at a temperature range between 25 °C and 800°C.

III. RESULTS AND DISCUSSION

A. Surface Morphology Analysis

The treatment of 6% NaOH and TEMPO affects the sugarcane bagasse fibers, facilitating the removal of cellulose from lignin and hemicelluloses, which can be seen in the difference in fiber color, as displayed in Figure 1. Treatment

using 6% NaOH and TEMPO makes fiber clearer and results in smaller debris.

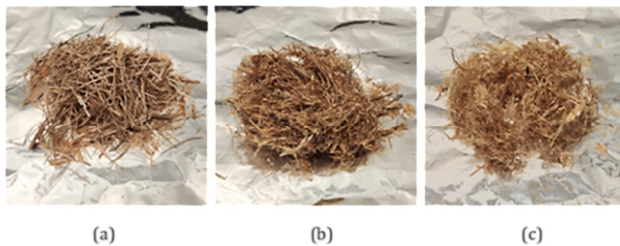


Fig. 1. Sugarcane bagasse fiber (a) control, (b) 6% NaOH, and (c) TEMPO treatment.

The SEM observation indicates that bagasse fibers are split after 6% NaOH alkalization and TEMPO treatment, as shown in Figures 2 (b) and Figure 2 (c), respectively. The fiber is cleaner from impurities after treatment, lignin and hemicellulose are separated, leaving only cellulose fibers. Alkali treatment can remove up to 90% of acetyl groups and 20 to 30% of lignin from bagasse [17, 18]. The bagasse fiber surface that has been treated is split so that the fibers look separated from each other and produce a rougher fiber surface.

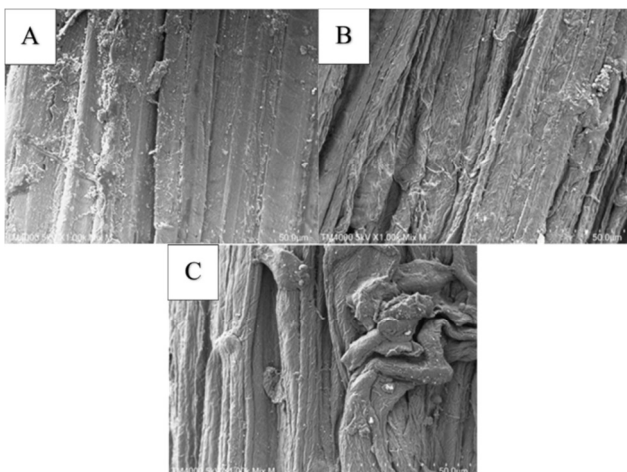


Fig. 2. Surface of bagasse fiber (a) without treatment, (b) NaOH 6%, and (c) TEMPO treatment.

B. Crystallinity Analysis

The NaOH and TEMPO treatment affects the structure of bagasse fiber, proven by the diffractogram evidenced in Figure 3. The diffraction peaks of cellulose are 15.7° (at crystallographic plane 101), 22.5° (at crystallographic plane 002), and 34.7° (at crystallographic plane 040), which are similar to/complying with [19]. Using (1), the treatment of NaOH and TEMPO increases the CI of fiber to 58.38% and 52.34%, respectively, while bagasse control has a CI of 50.95%.

Treatment using 6% NaOH likely removed hemicellulose and lignin, and thus improved the regularity of crystalline regions in cellulose. In addition, the effective reduction of the

lignin and hemicellulose content leads to the removal of amorphous components [20] and increases the proportion of cellulose [21], so the crystallinity index shows clearer peaks through XRD analysis. In contrast, TEMPO oxidation modifies the fiber surface, enhancing reactivity and compatibility with polymer matrices. TEMPO-treated fiber had a crystallinity value of 52.34%, slightly higher than the control fiber, but lower than the NaOH-treated fiber. Increased CI of natural fiber was also reported in Mendong fiber [22], and Sansevieria fiber [23] after being treated by 7.5% and 5% NaOH, respectively. Another study also indicates a slightly changing CI of bagasse fiber without changing the latter's microfibril angle after being treated with 4% NaOH [24]. TEMPO oxidation resulted in structural modifications to the cellulose without significant removal of lignin or hemicellulose. The CI of cellulose fiber was also reported to have increased after TEMPO treatment in pineapple leaves' fiber from 73.8% to 84.3% [25]. However, in TEMPO-oxidized cellulose fiber from spent coffee ground CI was reduced from 49.9% to 37% due to the combination of TEMPO with the disintegration process [26]. After TEMPO treatment, the peak at 34.5° sharpened, while the effect on crystallinity was less than the alkaline treatment [27, 28]. TEMPO with oxidation reaction enhances the order of the crystal surface, improving the arrangement of molecules in the transversal direction, and making the (040) peak more defined.

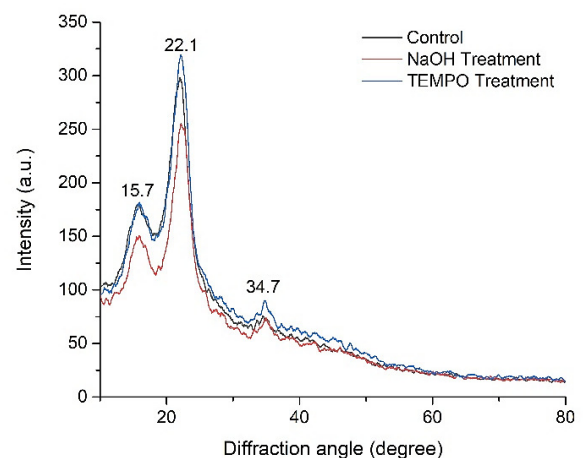


Fig. 3. Diffractogram of chemical-treated bagasse fiber.

C. Mechanical Properties

The mechanical properties of bagasse fiber were tested by the fiber tensile tester, with the results being depicted in Figure 4. Bagasse fiber without treatment (control) has a tensile strength of 45.48 MPa, which increased after 6% NaOH treatment to 85.26 MPa. NaOH increases the tensile strength and strain of fiber by removing amorphous organic matter and increasing crystallinity [29]. TEMPO treatment provides the highest increase in tensile strength with a value of 141.10 MPa. Additionally, treatment using 6% NaOH and TEMPO produces a higher modulus of elasticity, increasing it from 3372.41 MPa to 3691.12 MPa, and 4495.86 MPa, respectively, as can be seen in Figure 5.

Although NaOH treatment results in higher CI, TEMPO treatment has higher tensile strength and elastic modulus because NaOH treatment damages the fiber bundle, as portrayed in Figure 2 (b). Thus, the bundle integrity is reduced resulting in lower tensile strength. The same phenomena are described in pulp fibers [23], ramie fibers [30], and pineapple leaf fibers [31].

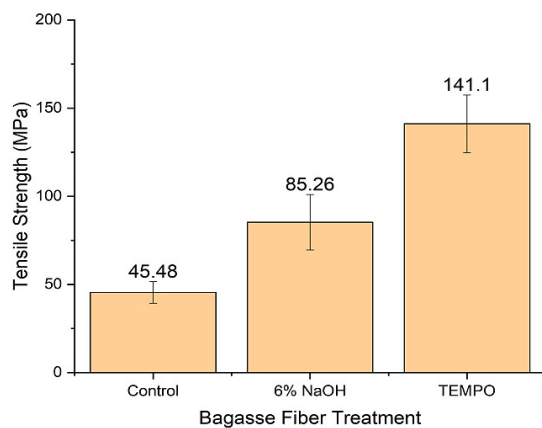


Fig. 4. Tensile strength of treated bagasse fiber.

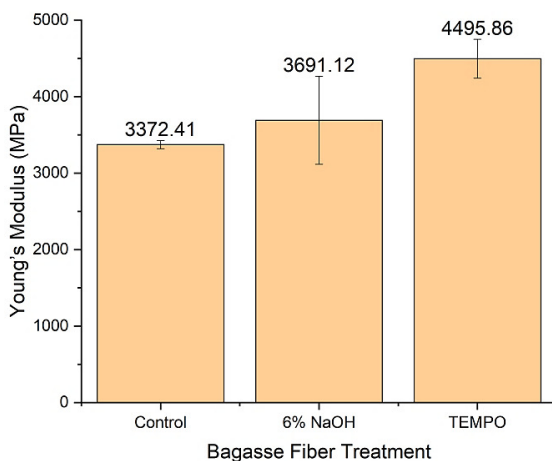


Fig. 5. Young's modulus of treated bagasse fiber.

One-way Analysis Of Variance (ANOVA) indicates that 6% NaOH and TEMPO treatments have a significant effect on the tensile strength of bagasse fibers (P -value = 0.00724). Tukey's test further reveals that TEMPO treatment significantly enhances bagasse fiber tensile strength compared to the control fiber, while NaOH treatment results in an increase that is not statistically significant.

D. Functional Group Analysis of Bagasse Fiber

The results of the functional groups analysis of bagasse fiber using FTIR are shown in Figure 6. At wavenumber 3319 cm^{-1} , O-H stretching occurs, indicating an increase in the content of hydroxyl groups [32], which is attributed to the presence of cellulose after the disappearance of lignin and

hemicellulose [33]. The stretching of the C-H bond occurs at 2895 cm^{-1} , and is associated with methyl and methylene groups in cellulose [34]. At the wavenumber of 1729 cm^{-1} the stretching of the C=O bond of esters occurs [35], which can be derived from hemicellulose or lignin, especially if the material has not been fully purified. After treatment, this peak disappears, indicating that these components are removed. Lignin degradation after chemical treatment is shown at wavenumbers 1597 cm^{-1} and 1504 cm^{-1} , with the stretching of the C=C bond of aromatic groups in lignin. At wavenumber 1051 cm^{-1} the stretching of the C-O-C Glycosidic group in cellulose is depicted. The wavenumbers of 896 cm^{-1} and 668 cm^{-1} are related to the β -glycosidic groups in cellulose. This peak remains or slightly increases at TEMPO, indicating that the β -glycosidic bond is not disrupted.

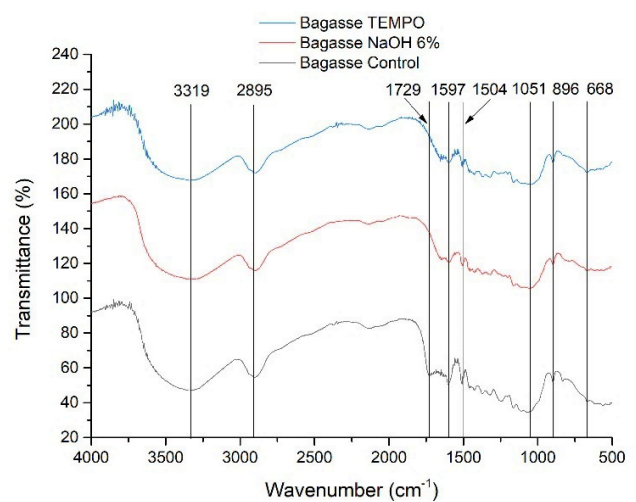


Fig. 6. FTIR analysis of bagasse fiber with added treatment.

E. Thermal Degradation Analysis of Bagasse Fiber

Figure 7 presents the thermal degradation analysis of bagasse fiber using TGA. Bagasse fiber demonstrates 3 stages of thermal degradation. Stage 1 starts from $25\text{ }^{\circ}\text{C}$ to $191\text{ }^{\circ}\text{C}$, where de-volatilization begins, which corresponds to the release of light volatiles and water content [36]. Treated bagasse fiber with 6% NaOH and TEMPO experienced a mass loss of 7.6% and 8.3%, respectively. The mass loss at this stage is due to water evaporation and hemicellulose decomposition. Material that has not evaporated completely will continue in stage 2 as the main thermal degradation.

Stage 2, taking place between $191\text{ }^{\circ}\text{C}$ and $400\text{ }^{\circ}\text{C}$, represents the primary thermal degradation phase. The peak degradation temperatures for untreated bagasse fiber and those treated with NaOH and TEMPO are $342\text{ }^{\circ}\text{C}$, $322\text{ }^{\circ}\text{C}$, and $339\text{ }^{\circ}\text{C}$, respectively, with corresponding mass losses of 50.95%, 56.3%, and 58.8%. The degradation rates are 8.1%/min, 8.2%/min, and 9.6%/min, respectively. These results indicate that NaOH treatment shifts the degradation peak to a lower temperature ($322\text{ }^{\circ}\text{C}$), making the fibers degrade more easily at a lower temperature than the untreated fibers ($342\text{ }^{\circ}\text{C}$). However, the overall degradation process is more controlled,

resulting in a slower degradation rate (8.2%/min) compared to the TEMPO-treated fibers (9.6%/min). TEMPO treatment, in contrast, leads to a degradation temperature similar to the untreated fibers (339 °C), but causes a higher mass loss (58.8%) and a faster degradation rate, indicating accelerated decomposition.

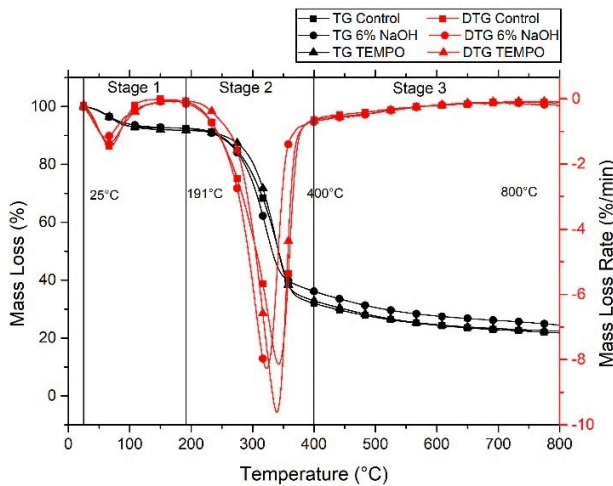


Fig. 7. DTG curve of bagasse fiber with added treatment.

Stage 3, occurring between 400 °C and 800 °C, represents the final decomposition phase, where degradation is slower and involves the release of residual material that was not combusted in the previous stage. The mass loss in this stage was 11.7% for NaOH-treated bagasse and 10.6% for TEMPO-treated bagasse.

The total thermal degradation of bagasse treated with 6% NaOH was 75.6%, leaving an ash content of 24.4%, while TEMPO treatment resulted in a higher degradation of 77.7%, leaving 22.3% ash. The 2.1% difference suggests that TEMPO treatment leads to greater overall thermal degradation, whereas NaOH treatment enhances thermal stability by increasing residual ash content. Despite the lower degradation temperature in stage 2, NaOH-treated fibers exhibit better thermal stability due to their higher residual ash content and lower total mass loss. This suggests that NaOH treatment removes amorphous components and increases cellulose crystallinity, leading to a more controlled degradation process. This result aligns with the increasing thermal stability of NaOH-alkalized *Sansevieria* fiber [23] and kenaf fiber [37]. In contrast, TEMPO-treated fibers degrade more rapidly, with higher mass loss, making them less thermally stable overall. This result is in line with the studies reported in [38], where TEMPO-treated cotton linter fiber results in higher fiber degradation, and in [39], where it was shown that wheat straw fiber is sensitive to thermal degradation after TEMPO treatment.

This study provides a direct comparative evaluation of NaOH alkalization and TEMPO oxidation treatments on sugarcane bagasse fiber, offering new insights into their impact on crystallinity, mechanical performance, and thermal stability. While previous research has individually explored NaOH for

lignin and hemicellulose removal to enhance crystallinity and thermal resistance, and TEMPO oxidation for improving fiber surface functionalization, a direct comparison of these treatments on bagasse fiber has not been performed. This study bridges that gap by systematically contrasting alkalization and oxidation, revealing a trade-off between structural rigidity and mechanical flexibility. NaOH treatment significantly increases crystallinity (from 50.95% to 58.38%) and thermal stability, whereas TEMPO oxidation enhances tensile strength (from 45.48 MPa to 141.10 MPa). The multi-dimensional analysis using SEM, XRD, FTIR, TGA, and tensile testing provides a holistic understanding of how these treatments alter sugarcane bagasse fiber at both structural and functional levels. Furthermore, the findings demonstrate that NaOH-treated bagasse fiber is better suited for thermally stable composites, while TEMPO-treated fiber exhibits enhanced mechanical properties for advanced biopolymer applications. By addressing the lack of comparative assessments and identifying key structural-mechanical trade-offs, this study contributes to the optimization of bagasse fiber utilization, supporting its application as a sustainable alternative to synthetic fibers in environmentally friendly material development.

IV. CONCLUSION

This study compares NaOH and TEMPO oxidation treatments on sugarcane bagasse fiber, offering new insights into their effects on crystallinity, mechanical performance, and thermal stability. By distinctly contrasting alkalization and oxidation, the findings contribute to the advancement of sustainable waste utilization applications.

The treatment of 6% NaOH and TEMPO on sugarcane bagasse fibers revealed significant improvements in structural, mechanical, and thermal properties. Morphological analysis confirmed effective lignin and hemicellulose removal, with NaOH-treated fibers exhibiting clearer fiber separation. The crystallinity analysis showed an increase in the Crystalline Index (CI) from 50.95% (control) to 58.38% (NaOH) and 52.34% (TEMPO), while the crystallite size increased to 80.02 nm (NaOH) but decreased to 30.32 nm (TEMPO), indicating distinct structural changes. The mechanical testing demonstrated that TEMPO-treated fibers achieved the highest tensile strength (141.10 MPa) and Young's modulus (4495.86 MPa), outperforming NaOH-treated fibers. The Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed lignin and hemicellulose removal, with TEMPO-treated fibers retaining β -glycosidic bonds. The Thermogravimetric Analysis (TGA) exhibited better thermal stability in NaOH-treated fibers (total degradation: 75.6% vs. 77.7% for TEMPO), attributed to the removal of thermally unstable components. Overall, TEMPO treatment enhances mechanical performance, while NaOH treatment improves thermal stability, demonstrating the potential of these modifications for advanced material applications.

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