Physicochemical characteristics of chemically treated bagasse fibers
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Abstract: The present paper reports effect of chemical modification of bagasse fibers by using oxidizing agents like potassium permanganate and potassium dichromate. The fiber modification was assessed by several techniques like Fourier Transfer Infrared Spectroscopy (FTIR), Thermo Gravimetric Analysis (TGA), and X-Ray Diffraction (XRD) which provide evidences for the desired structural changes. Scanning electron microscope (SEM) used to analyze the microstructure of the samples. Our studies reveal decrease in the crystallinity index of samples after treatment with dichromate solution implying effective removal of polar impurities. Raw fibers had crystallinity index of 72% while permanganate and dichromate treated fibers showed 47% and 50%, respectively.

Subjects: Composites; Materials Processing; Polymers & Plastics; Surface Engineering-Materials Science

Keywords: Bagasse fiber; chemical modification; cellulose fiber; composites

1. Introduction
The development of new materials derived natural fibers has been focus currently due to the environmental concerns over the use of synthetic polymers. Natural fibers play a prominent role in composite material manufacturing due to their beneficial properties like biodegradability and its sustainable benefits. Sugarcane is one of the promising biomass energy in the world. It produces two types of biomasses, namely, Bagasse and sugarcane trash (Chokshi & Gohil, 2021; S Chokshi et al., 2020; Li et al., 2007; De Paiva et al., 2019; Shyam Kumar et al., 2021; Vidyashri et al., 2019). The residue left after refining process of sugarcane is bagasse and it is the largest plant residue. Sugarcane trash is the field residue remaining after harvesting. The bagasse has 40%–50% moisture content and it is a mixture of hard fiber, soft fiber and pith with hygroscopic property.
It mainly consists of Cellulose, Lignin, Hemicellulose, Pentosan, Wax, and Minerals. This composition depends on the harvesting method and maturity or variety of the sugarcane. Bagasse finds usage as fuel due to its good calorific value (Alonso Pippo et al., 2011). Such fibers are currently being used as substitute for the synthetic fibers in the form of reinforcements and fillers, while forming the materials, due to their low cost, light weight, low density, good modulus of elasticity, and eco-friendly processing method. Such reinforced fibers are reported to have wide applications in the field of automobiles, constructions, electrical insulation, packaging, and other domestic applications (Deepa et al., 2020; Mahmud & Anannya, 2021; Manimaran et al., 2018; Sagar Chokshi et al., 2020; Yashas Gowda et al., 2018).

Bagasse like other natural fibers being polar and when mixed with nonpolar hydrophobic matrix leads to the difficulty in dispersion of the fiber in the polymer matrix. This is attributed to the short fiber composite, fiber dispersion, fiber orientation, and fiber length, and fiber matrix adhesion. This causes the mismatch of hydrophilic natural fiber and thermoplastic hydrophobic polymer matrix that decreases the interfacial bonds which leads to reduced mechanical property and increased water absorption tendency (Wirawan & Sapuan, 2018). In spite of natural fiber possessing several economic and ecological advantages, it is found to have high moisture adsorption and poor dimensional stability, which makes them inefficient in long-term composite applications. The fiber was also prone to have microbial attack on humid climate and yellowing in sunlight that make end user experience unpleasant.

The presence of large number of hydrophilic group on the cellulose fiber limits their application in automotive and other allied industries. The disadvantages of both natural fibers and their polymer composites were partially addressed by the suitable functionalization of natural fibers. Through functionalization it is possible to alter the property for the targeted applications. When the proper modification and manufacturing process is used, Sugarcane bagasse (SCB) showed improved mechanical property such as Impact strength, Tensile strength, Hardness, Flexural strength, and Flexural modulus. SBC can be easily treated and modified with variety of chemicals and reagents, which could help in the preparation of new composite materials with improved properties (Loh et al., 2013). Bagasse and similar plant fibers mostly composed of cellulose that has relatively high modulus. The reinforcing property is affected by the dimension of the fiber. Commonly, Bagasse has a length 1.2 mm and diameter of 15 µm giving an approximate aspect ratio of 50 (Hajiha & Sain, 2015). Figure 1 shows the sample of raw bagasse fiber.

The reinforced composites prepared from bagasse and its performance depend on the factors like chemical composition, physical property, cell dimension, structure, and microfibrillar angle, which contribute to their mechanical properties. Bagasse has approximately cellulose content of 50% and hemicellulose in 25% and lignin about 25%. More specifically, it has about 50% of α-cellulose and 30% of pentosans. It also contains pectin around 0.6–0.8% and extractives of 1.5–9.0%. Bagasse has some advantage when compared to other agricultural plant residues like rice straw or wheat straw which has 17.5% and 11% ash content,
respectively. Also in comparison to other agricultural residues bagasse found to have rich solar energy reservoir due to its high yield and annual regeneration capacity that are essential to meet large material demand for sustainable development (Aminudin et al., 2017; Prasad et al., 2020). The physical properties of bagasse includes the structure, fiber dimension, strength, and defects. Table 1 shows the physical and mechanical properties of bagasse fiber (Gurunathan et al., 2015). Over the years extensive work has been carried out for the chemical modification of natural fibers by using sodium hydroxide, potassium hydroxide, hydrogen peroxide, benzoyl peroxide, acetyl chloride or acetic anhydride, multifunctional silanes, and potassium permanganate to name a few. In effect, the objectives of all these treatments have been to reduce the presence of polar impurities and to achieve better adhesion with matrix material of the composites (Bartos et al., 2020; Kalia et al., 2009; Madhu et al., 2019; Mahesha et al., 2016, 2019; Ryszard et al., 2020; Saw & Datta, 2009; Le Moigne et al., 2018b). We recently observed potassium permanganate is an effective treatment method but suffers from disadvantage of unwanted colouring of fibers that require additional treatment with oxalic acid to remove the colour (Madhu et al., 2019; Mahesha et al., 2016; Vidyashri et al., 2019). In this context, it worthwhile to study the behavior of potassium dichromate that is a strong oxidizing agent but relatively lesser when compared to potassium permanganate. In this paper, we describe the fiber property modification using sodium hydroxide pretreatment that is followed by potassium dichromate solution. The results have been compared with potassium permanganate treatment under similar experimental conditions.

2. Experimental

2.1. Materials and methods

Bagasse is collected from the native sugarcane juice shops. NaOH pellets are used for the pretreatment of bagasse fiber. Reagents like potassium permanganate (KMnO₄) and potassium dichromate (K₂Cr₂O₇) were obtained from commercial suppliers. The bagasse was dried in sunlight for a period of 7 days by distributing it on the water resistant sheet. The fibers were completely dried off to avoid the accumulation of fungi on bagasse fiber surface.

2.2. Extraction and processing

The sugarcane was crushed with a small-scale extractor to remove the juice and the left out residue called as bagasse, which is used for the functionalization process. Pitch, which is the inner soft core of bagasse was removed manually to obtain the outer hard rind of Bagasse. The bagasse was soaked in water for 24 hours to remove the sugar traces and dried in sunlight for 7 days to remove the traces of water, if any.

2.2.1. Pre-treatments

Alkaline treatment: 25 g of bagasse was immersed in 10 % NaOH solution for 5 hours and rinsed with distilled water in order to achieve the neutral pH. After rinsing the fiber was dried for 2 days or until constant weight was observed.

| Physical properties | Values |
|---------------------|--------|
| Density (g/cc)      | 0.55–0.70 |
| Microfibrillar angle (°) | 10–22 |
| Diameter (μm)       | 300–400 |
| Tensile strength (MPa) | 170–290 |
| Young’s modulus (GPa) | 15–19 |
| Aspect ratio (L/D)   | 100–140 |
| Elongation at break (%) | 3–7 |
3. Chemical treatments

Potassium permanganate treatment (KMnO₄): 1 gm of 10% NaOH pretreated bagasse was taken along with the 10% of 5 mL KMnO₄ in a round bottom flask fitted with condenser and heated on water bath for 8 hours. Then washed with water to attain the neutral pH and further washed with oxalic acid to remove the MnO₃ impurities. It was dried to remove moisture contents.

Potassium dichromate treatment (K₂Cr₂O₇): 1 gm of pretreated bagasse was taken along with the 10% of 5 mL K₂Cr₂O₇ solution in a round bottom flask fitted with condenser and heated on water bath for 8 hours. Then washed with water to attain the neutral pH. It was dried to remove moisture content completely. Representative images of processed fibers are given in Figure 2(a–d).

4. Characterization techniques

Thermo-Gravimetric Analysis (TGA) of the oxidized bagasse fiber was performed by TGA (Model TG/DTA 200) using an average 5 mg sample heated from room temperature to 800°C at the rate of 10°C/min in an inert N₂ atmosphere using platinum pan. Morphology of the raw and chemically treated bagasse fiber was examined using Scanning Electron Microscopy (SEM). Initially, the sample was sputtered and the analysis was done in vacuum using SEM (ZEISS EVO 18) on an aluminium stub. To analyze the crystallinity of the oxidized fiber X-Ray Diffraction (XRD) was used. The sample was scanned in 2θ range between 0 and 90° range. The crystallinity index was calculated using the formula (1)

\[
Ic = \frac{I_{cry} - I_{am}}{I_{cry}} \times 100
\]

Where \( I_{cry} \) indicates the lattice diffraction of the crystalline peak and \( I_{am} \) indicates the intensity corresponding to the diffraction of the amorphous peak (Bansal et al., 2016).

Figure 2. (a) Raw fiber, (b) 10% NaOH-treated bagasse, (c) 10% KMnO₄-treated bagasse, and (d) 10% K₂Cr₂O₇-treated bagasse.
Figure 3. TGA of raw fiber.

Figure 4. TGA Plot of 10% KMnO$_4$-treated fiber.

Figure 5. TGA of 10% K$_2$Cr$_2$O$_7$ fiber.
Table 2. Thermal stability of raw and chemically treated fibers

| Serial no. | Sample          | T_{20\%} | T_{\text{max}} | Y_{c} |
|------------|-----------------|----------|----------------|-------|
| 1          | Raw fiber       | 260      | 340            | 16    |
| 2          | KMnO₄           | 240      | 355            | 25    |
| 3          | K₂Cr₂O₇        | 120      | 360            | 12    |

5. Results and discussion

Thermo-gravimetric analysis (TGA): This technique is used for the analysis of the thermal stability. Sample weighing 2 mg was placed in a platinum pan and tests were performed using the programmed temperature of 10°C/min. Figures 3–5 represents the TGA curves of the raw fiber and the chemically treated fibers. At 50–70°C, there occurs 2–4% weight loss which is due to the loss of volatile contents (Ghetti et al., 1996). At temperatures from 230°C onward, there is gradual loss of cellulose and hemicellulose and more precisely at the temperature range from 345 to 365°C there seems loss of cellulose and 300–320°C occurs the loss of hemicellulose. Decomposition temperature beyond 400°C is attributable for lignin (Megiatto et al., 2007). Thermal stability can be seen up to 230–250°C, which is termed as maximum processable temperature until which weight loss is negligible. Temperature greater than 230°C leads to the reactions like dehydration and, decarboxylation which involves C–C, C–H, and C–O bond breakage which is in line with the thermal characteristics already described for many natural fibers (Frollini et al., 2013).

\( T_{20\%} \) decomposition temperature of dichromate treated fibers observed at relatively lower temperature of about 120°C, which might be due to the presence of trapped moisture. Given, improved thermal stability at higher temperature range the early weight loss may be due to trapped water between layers of fibers. Drastic reduction in weight happens beyond 310°C, which corresponds to the decomposition of lignin in the material. Complete decomposition of the material occurs around 475°C–500°C temperature range (Balaji et al., 2020). Table 2 shows the \( T_{20\%} \) decomposition temperature where 20% weight loss occurs, \( T_{\text{max}} \) decomposition temperature at maximum decomposition rate and \( Y_{c} \) is the residual char yield at 450°C. Among the treated fibers the \( T_{\text{max}} \) value of K₂Cr₂O₇-treated fibers found to be better and showed enhanced thermal stability.

6. X-ray diffraction analysis

Figures 6–8 show the X-ray diffraction patterns of raw and chemically treated bagasse fibers.

The result reveals that the crystallinity index of the chemically treated fibers decreased when compared to the untreated raw fibers. Therefore, the chemical treatment was efficient in removing impurities like lignin and hemicellulose contents. In the Figure 10 and 11, there
occurs a prominent peak at 30° which might account for the bonding of chromium ions with the fibers after dichromate treatment. The result obtained by XRD was shown in the Table 3 (Pereira et al., 2014).

7. Scanning electron microscope

Initial observation shows the raw fiber where there is no fiber breakage. After the treatment of the bagasse with KMnO₄ and K₂Cr₂O₇, It is possible to observe clearly the modifications of the surface areas of the bagasse fiber. So, the NaOH pretreatment before functionalization breaks the lignin structure along with that it hydrates and swells the cellulose thereby reducing the crystallinity (Fasanella et al., 2018).

| Sample                             | Crystallinity index Iₐ (%) |
|------------------------------------|----------------------------|
| Raw fiber                          | 73%                        |
| 10% KMnO₄-treated fiber            | 47%                        |
| 10% K₂Cr₂O₇-treated fiber          | 50%                        |

Figure 7. XRD plot of 10% KMnO₄-treated fiber.

Figure 8. XRD of 10% K₂Cr₂O₇ fibers.

Table 3. Crystallinity index values of treated fibers

https://doi.org/10.1080/23311916.2021.2014025
The SEM morphology with different magnifications of untreated fibers (Figure 9) shows the presence of large amount of extractives, waxes, lignin, and hemicellulose which provides the fiber continuous covering layers for the cellulosic material. These show poor binding capacity to hydrophobic resin systems. Whereas the treated fibers shows the breakage of lignin and XRD gives the promising proof for the removal of extractives and cellulose bundle, which gives roughness to the fiber (Figure 10). Such a modified surface facilitates enhanced interfacial bonding between the fiber and the matrix. K₂Cr₂O₇-treated fibers shows enhanced roughness on their surface and the breakage of microfibrils was to a large extent in comparison with KMnO₄ treated fibers (Figure 11) (Rezende et al., 2011).

8. Fourier-Transfer infrared spectroscopy
FT-IR spectra of both raw and treated bagasse fiber were given in Figures 12-14. The peak around 3100 cm⁻¹-3700 cm⁻¹ corresponds to the hydrogen-bonded OH stretching. FT-IR provides promising proof for the increase the peak intensity after chemical treatment. The peak around 2900 cm⁻¹ because of C-H stretching vibration (Cerqueira et al., 2011). The adsorption peak at 1729 cm⁻¹ and 1712 cm⁻¹ accounts for the vibrational stretching of carbonyl groups.
(\(C = O\)). The peak about 1200 cm\(^{-1}\) assigned for C-O stretching of acetyl group present in the hemicellulose decreased on the chemical treatment. The stretch at 1030 cm\(^{-1}\)–1050 cm\(^{-1}\) accounts for the C-O stretching of Hemicellulose and lignin (Figure 13) (Simão et al., 2016). These results gave explanation for the reduction of Hemicellulose and lignin content upon chemical treatment. There exists a peak at 611 cm\(^{-1}\) which implies on treatment with \(K_2Cr_2O_7\) there is a possibility of Cr-O-C bond due to the formation of the chromium complex (Figure 14) (Le Moigne, Otazaghine, Corn, Angellier-Coussy, Bergeret, Le Moigne, Otazaghine, Corn, Angellier-Coussy, Bergeret et al., 2018a). These results need further validation and optimization and would likely prove useful for finding new treatment method of chemical modification of fibers.
9. Conclusions
The following important outcomes are derived through the investigation of physicochemical properties such as crystalline character, morphology, chemical constituents, and thermal stability of bagasse fibers

- The crystallinity index of the sugarcane bagasse fibers decreases upon alkali pretreatment followed by KMnO₄ and K₂Cr₂O₇ treatments. Hence, the chemical treatments are effective in removing the impurities, lignin, and hemicellulose contents.
- The removal of hemicellulose, lignin, and wax contents in treated fibers was identified by the SEM analysis. FTIR analysis show removal of lignin and related impurities.
- Thermal stability of the chemically treated fibers higher when compared to the raw fibers. Among treated fibers, 10% K₂Cr₂O₇ treatment showed enhanced thermal stability and surface roughness, which could be useful for composite applications.
- Reduced crystallinity index implied good opportunity to explore composite preparation with polymers like polyethylene and polypropylene that has similar crystallinity index values.

Funding
The authors received no direct funding for this research.

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Disclosure statement
No potential conflict of interest was reported by the author(s).

Citation information
Cite this article as: Physicochemical characteristics of chemically treated bagasse fibers, Roopa Prabhu, Sharad Ganesh, GT Mahesha & K Subrahmanya Bhat, Cogent Engineering (2022), 9: 2014025.

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