Comparative study of FESEM, EDS, FTIR on alkali treated and untreated fiber of Aerva Tomentosa (Bui)

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Abstract

The Amaranthaceae family plant, Aerva Tomentosa, grows abundantly in western Rajasthan, India. In the production of bio composites, the Aerva Tomentosa (Bui) can be used successfully as natural fibers. In this study, Aerva Tomentosa (Bui) fibers were extracted from the Bui plant and treated with an alkaline solution containing 5% NaOH. The investigation involved morphological and chemical characteristics of the extracted untreated Aerva Tomentosa fibers (ATF(0)) and extracted alkali treated Aerva Tomentosa fibers (ATF(1)). According to an analysis of ATF(0) and ATF(1) using Fourier-transform infrared (FTIR) spectroscopy, both ATF(0) and ATF(1) included functional groups for cellulose, hemicellulose, and lignin. The elimination of extractives from the surface of the fibers isolated from Aerva tomentosa is demonstrated by FESEM and EDS analysis. The surface of untreated fibers becomes smoother following treatment, as evidenced by FESEM images taken at various resolutions.

Keywords: Bio Composites, Natural Fiber, Aerva Tomentosa.

Introduction

They use natural fibers extracted from plants as reinforcement in a composite structure not new throughout documented history used by multiple civilizations. Natural Fibers are the best choice to replace synthetic polymer fiber for low cost biodegradability, eco-friendly, and sustainable use of hazardous waste materials for our environment. The natural is characterized into three categories depending on their origin; Plant-based, Mineral based, and Animal-based [1,2]. Natural fiber has constituent contents and hierarchal (tree-like) structure variations across various categories of plants and species.

From the geometrical and chemical considerations of natural fibers, analysis of the varying fiber structure and natural polymer constituents, an investigation towards developing more robust, structurally performing bio composites for today and tomorrow’s needs [3,4]. According to B. Clair et al., The major constituents (Cellulose, hemicellulose, lignin, and pectins etc.) vary and depend on the source of the fibers, growing conditions, plant age, and digestion processes [1,4].

Extraction of fiber from the plant (Aerva Tomentosa)

In recent years, we have needed a natural fiber which fiber biodegradable and not Hazardous for the environment and can replace synthetic fiber. Considering the above points, we have selected the desert plant Aerva Tomentosa from Amaranthaceae family for study [5,6]. Aerva Tomentosa plants have the following desirable characteristics:

- **Hardy and firm stem up to a length of 80 cm.**
- **Convenient plant shape for harvesting.**
- **Stable flower color.**
- **The long shelf life of cut flowers.**

So, that desert cotton (Bui/Aerva Tomentosa) is a perfect choice for making bio composites of the plant Aerva Tomentosa fibers [7,8]. As shown in Figure 1.

**Figure 1:** Aerva Tomentosa (Bui) Plan

**Extraction of Bui Fibers**

Plant (Aerva Tomentosa) stems of 20 to 30 cm lengths were
collected from various places in Bikaner, particularly Shobhasar village. Then the collected stem was dried in the standard atmosphere for a week and in the oven. At 60°C after, dried stems were dipped in water for 6-7 weeks to extract the fibers in sufficient amounts [1,9]. Figure 2 & Figure 3 depicts untreated and treated Bui Fiber.

The comparison between treated crude fiber and untreated elementary fiber shows that the surface of the treated fiber is smoother than untreated fiber. The weak hydrogen bond is braked, and a new bond is formed between sodium ions, clearly shown in the SEM image described above at different resolutions.

These tiny crystals are formed due to a 5% NaOH solution reaction with crude natural fiber as below chemical reaction [10]. According to Kabir et al. and other researchers, NaOH treatment removes practically all non-cellulose components [1,11-13]. The probable reaction involved in alkali treatment is shown in equation 1.

\[
\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}2\text{O} \quad (1)
\]

A. SEM Analysis

A single fiber of Aerv a Tomentos a (untreated and treated) was visualized with the help of an SEM instrument in SE mode. The SEM images of treated Bui fiber and untreated Bui fibers are taken at a different resolutions & magnifications. SEM images of treated and untreated fibers are shown in Figure 4 (a), (b), (c), and Figure 5(a), (b), (c). The surface of the treated Bui fiber is smoother than untreated fiber [14,15], according to A. Gholampour et al., due to the weakness of hydrogen bond breaks by the NaOH (alkali) treatment and C=O bond stretches so that the Na+ ions replace the hydrogen bond [3,11,16].

It is very clearly seen in Figure 5 (a), (b) & (c), some tiny crystals appeared in the SEM image. The formation of -O - -Na+ / Na+ bond proved by EDS investigation of ATF(0) and ATF(1).
Figures 4(a), (b) and (c) are FESEM images of untreated Bui fibers at different resolutions. In these images it is very clear that untreated Bui fibers having lot of extractives like Si etc. as proved in EDS analysis also. In ATF(0) FEEM images some pores and caves also seen in these FESEM images. In sequence Figure 5 (a, b, & c) shows the FESEM images of treated Bui fibers labelled as ATF(1). Form ATF(1) SEM images shows very smooth surface, and some tiny crystals appears at the surface of fibers ATF(1). And also proved by EDS analysis of fibers.

![Figure 5 (a) Treated Bui Fiber](image)

![Figure 5 (b) Treated Bui Fiber](image)

![Figure 5 (c) Treated Bui Fiber](image)

Figures 5 (a), (b) and (c) are SEM images of treated fiber of plant Aerva Tomentose at a different resolution.

**B. EDS Analysis**

The EDS peaks in Figure 6 a & Figure 6 b shows that Carbon (C) and Oxygen (O) are the primary elements as well as both the untreated and alkali treated Aerva Tomentosa Fibers contains a modest quantity of Ca. According to the EDS study, alkali-treated fiber has a lower carbon content and a higher oxygen content than untreated fiber \[17,18\]. The value of oxygen carbon ratio for ATF(0) was found to be 0.30 while for ATF(1) 0.33. Table 1 demonstrates that the O/C ratio was higher in ATF(1) as compared to ATF(0), demonstrating that the chemical treatment eliminates lignin from the surface of the fiber. The absence of the element Na in ATF(0) contrasts with the presence of sodium in ATF(1), and the presence of Si in ATF(0) while Si wasn't found in ATF(1) indicates that the alkali treatment was successful on Aerva Tomentosa fibers.

![Figure 6 a: EDS spectrum of ATF(0)](image)

![Figure 6 b: EDS spectrum of ATF(1)](image)

**Table 1:** The Atomic concentration (%) for ATF(0) & ATF(1)

<table>
<thead>
<tr>
<th>Element(s)</th>
<th>Carbon (C) %</th>
<th>Oxygen (O) %</th>
<th>Calcium (Ca) %</th>
<th>Sodium (Na) %</th>
<th>Silica (Si) %</th>
<th>O/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>ATF(0)</td>
<td>76.06</td>
<td>23.31</td>
<td>0.86</td>
<td>0</td>
<td>0.58</td>
<td>0.3</td>
</tr>
<tr>
<td>ATF(1)</td>
<td>73.75</td>
<td>25.06</td>
<td>0.37</td>
<td>1.88</td>
<td>0</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Figure 6-8: EDS spectra of untreated and treated Aerva Tomentosa fibers.
C. FTIR Spectrum

FTIR spectrum of untreated and treated fiber is shown. A bond stretching of various important bonds is shown in the table. The FTIR data or spectrum analysis shows that a bend stretching occurs in the 1650 cm\(^{-1}\) to 1700 cm\(^{-1}\). This shows that the stretching in the C=O bend group occurs after treatment, and the fiber surface becomes smooth [19].

According to FTIR spectrum, Figure 7 a & Figure 7 b analysis from the Perkin Elmer’s Spectrum Version 10.4.00 analysis results that the hydrogen-bonded OH stretching between the 3333.33 cm\(^{-1}\) for untreated fiber and 3330.80 cm\(^{-1}\) for treated fiber occurs due to presence of cellulose and hemicellulose one another peak at 2923.2 cm\(^{-1}\) for untreated and 2920.43 cm\(^{-1}\) for treated fiber this peak shows the C-H bond stretching [9,20]. According to Netra et al., C=O bond stretching occurs in alkali-treated fiber at 1740.5 cm\(^{-1}\), CH\(_2\) symmetrical bonding shows between the range 1417.98 cm\(^{-1}\) untreated fiber and 1420.48 cm\(^{-1}\) for treated fiber it shows that lignin’s some fraction of part remains in fiber after treatment also [11]. An anti-symmetrical bridge C-O-C stretch/C-O stretch at 1245 per cm for untreated and 1241 cm\(^{-1}\) for alkali-treated fiber [21]. The \(\beta\)-glycosides linkage at treated fiber. Pyranose ring also presents at 554.13 cm\(^{-1}\) for untreated and 562.00 cm\(^{-1}\) for treated fiber [22,23].

FTIR spectra of treated and untreated elementary natural fiber show the spectrum smoothness between 1640 cm\(^{-1}\) to approximately 2840 cm\(^{-1}\) in treated fiber while slight stretching bend in untreated fiber spectra [9,20,23]. FTIR spectra show that the range 2250 cm\(^{-1}\) carbonyl group presents and at 1500 cm\(^{-1}\) peak shows doublet so that the spectrum of before treated fiber has peaks in large number 3500 cm\(^{-1}\) to 1000 cm\(^{-1}\) but after treatment changes occur in this range [24]. And one more thing is that it is not precisely possible to characterize the chemicals or functional groups present in lignin [25,26]. From all the above discussion, it is clear that treated fiber is more efficient and smoother rather than compared to untreated fiber.

Conclusion

Research indicates that ATFs may be widely used in the creation of bio composites because they have shown excellent chemical and morphological qualities. The investigation's findings are summarized as follows:

- The reduction in hemicellulose, lignin, wax, and other impurities, as shown by the chemical composition study, enhanced the cellulose content in fibers.
- The reduction of these contents from the ATF(0) surface was also confirmed by the FTIR study.
- After alkali treatment treated fiber surface seems smoother rather than for ATF(0) confirmed by FSEM study.
- Extractives like Si completely removed after alkali treatment found in EDS investigation.

Examining these elements may make ATFs more effective overall and suitable for use in a variety of technical applications.

References

https://commons.wikimedia.org/wiki/File:Aerva_tomentosa.JPG.


