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Examination of the Effects of Chemical Treatments on the Mechanical, Chemical, and Physical Characteristics of *Hibiscus rosa-sinensis* Plant Fiber

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Abstract

Natural fibres in recent years have been found to contribute a great share in making composites. Hibiscus rosa-sinensis plant fibres which are natural were used in this work and have not yet been tried to make the composites so far and easily available thus making this study very necessary it aims to perform various chemical treatments and to study the physical and chemical properties of them. The methodology adopted was that literature review, problem identification, extraction of fibres, treatment of fibres, chemical and mechanical testing of fibres, results and discussions and conclusion. Fibre chemical composition tests were also done to determine the constituents of the fibres. Two chemical treatment methods (alkalinization and benzoylation) were proposed in this work. Chemical treatments were found to improve surface roughness. Using Scanning Electron Microscopy (SEM), researchers examined the fibres' structural and morphological alterations and discovered that components like pectin were largely eliminated. The structural properties of fibres were known from X-ray diffractograms, and crystallinity percentages were found for the cellulosic fibres. Fourier Transform Infrared (FTIR) spectroscopy presented the spectra of the fibres which revealed the structure of organic compounds and the presence or the absence of functional groups. It showed the partial removal of hemicellulose and lignin. Axial tensile tests were performed for the fibres to know the maximum tensile force and percentage elongation. Chemical treatments partially removed impurities in the fibres and made morphological changes. The improvements in mean tensile values and percentage elongation were found in 5% alkali-treated and benzoylation-treated fibres, 56% and 63% higher than the untreated fibres respectively.

Keywords: *Alkalinization and Benzoylation Treatments, FTIR, Natural Fibres, SEM Images, Tensile Test*

1.0 Introduction

Man-made fibres are being extensively used for their good stiffness values[1]. Owing to the problems in energy crisis and growing environmental consciousness necessitate the use of renewable and recyclable materials; a great potential can be tapped by using materials which are available in nature[2]. For many years, natural fibres

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have been used as reinforcing elements in making composites in which fibres share a major portion of the load in the composites[3-6]. Extensive use of the fibres along with polymers is affected due to low cost, economical production, biodegradability and low specific weight[6-10]. Generally, chemical treatments improve fibres adhesion with matrix materials by reducing their hydrophilic nature of, roughness and moisture absorption. A few examples are coir, flax, kenaf and sisal. However, the usage is limited due to some demerits like non-uniform mechanical properties and hygroscopic nature[11-13]. Hence it becomes necessary to treat the natural fibres to alter their structural and surface properties by chemical treatment methods to suit applications by improving their mechanical properties. Many treatment methods are being used by researchers. For wooden materials, Auto-hydrolysis and Kraft pulping methods are used to remove hemicelluloses[14]. The steam explosion method, which is a type of physicochemical modification is used for dietary food materials for the separation process[15]. Recently anti-bacterial hydrogels were attempted to prepare by some researchers by cross-linking process[16]. Hibiscus extracts can also be used to improve the antimicrobial properties of nanocomposite film[17]. Alkali treatment or mercerization being a chemical method can change the constituents of fibers[18]. Alkalinization and benzoylation treatments remove moisture content from fibres, increase their strength, remove almost all impurities adjoining the fibres and stabilize molecular orientation. Mwaikambo *et al.*[19] have performed alkali treatments on hemp, sisal and jute fibres in various concentrations. Optimal results are achieved when 4% potassium permanganate is used to chemically treat the suggested fibre with varying weight percentages to increase their mechanical quality[20]. When the fibre was dried using various methods, the physio-chemical, stability, and antioxidant values were recorded[21]. Unlike nanoparticles, which have antibacterial characteristics, cellulose does not. The nanocomposite of cellulose and zinc oxide is produced using hibiscus leaf extract[22]. A hybrid of Hibiscus Leaves Mucilage (HLM), polyvinyl alcohol pectin, and the electrospinning process, a new nanofiber mat has been developed as a scaffold for wound healing[23].

From the literature review, it is seen that many works were carried out using various natural fibres and this also may pose a demand on them. *Hibiscus rosa-sinensis* plant fibre is used in this work and this fibre is available in plenty in India. Hence this Hibiscus fibre will avoid scarcity of the natural fibres. Polymer composites using this fibre can be used in automotive applications like interior lining components of cars, instrument panels and packaging industries. The primary goal of this research is to examine the physical and chemical characteristics of the fibre after two different chemical treatments. This was accomplished by subjecting the fibres to alkali solutions of varying concentrations.

Figure (1) shows a view of the *Hibiscus rosa-sinensis* plant. It is a species of tropical hibiscus belonging to the Malvaceae family native to East Asia. It grows up to a height of 5m. Its stem is cylindrical and branched; the bark is rich with fibres. It is present in the various parts of the Indian subcontinent. *Hibiscus rosa-sinensis* plant fibres are obtained from the plant's inner bark. Fibre length is dependent on plant age and can range from 0.1 m to 1 m. The prime constituents of the Hibiscus fibre as in the other natural fibres are cellulose, hemicellulose, pectin and lignin. The amount of cellulose and the microfibrillar angle determine the mechanical properties of the fibre.



Figure 1. *Hibiscus rosa-sinensis* plant.

2.0 Materials and Methods

2.1 Extraction of Fibres

Plants of a minimum of 1m height were selected for this work. A procedure known as retting is used to remove the fibres from the plant. After trimming the stem from

branches, leaves etc., they are soaked in water for about 20 days. During the process, microorganisms start digesting the epidermis and consequently, it leads to the exposure of fibres. In this process, no chemicals are used. Normally the plant grows to a height of 2.5 m. After 20 days, they are taken out and then the barks are peeled off from the stem. After washing the barks in water several times, the fibres are separated and then dried.

2.2 Density of Fibres

About 5 g of fibre is weighed accurately using trace paper with the help of a chemical balance and is then soaked in water and taken in a measuring jar. The replacement of water is noted which is known as volume of fibre.

$$\text{Density of fibre} = \frac{\text{Weight of fibre}}{\text{Volume of fibre}}\ \text{g/cc} \qquad (1)$$

2.3 Alkali Treatment of Fibres

For two hours at 25 degrees Celsius, with a liquor ratio of 20:1, hibiscus fibres are immersed in solutions of 5% and 10% by weight of NaOH. After that, the fibres are given many washes with water to remove any alkali solution that may have stuck to their surfaces. After rinsing with water, the fibres are treated with diluted acetic acid to neutralize them. Finally, the fibres are dried for seven hours at 70 degrees Celsius[24].

Fibre – OH + NaOH → Fibre – O – Na + H_2O

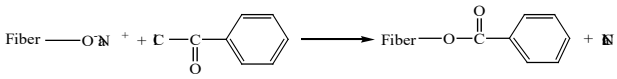
Scheme 1. Reaction of NaOH with fibre.

Alkalinization alters coordinating hydrogen bonds in the cellulose of fibre. It also removes some lipids, wax, and lignin from the fibre cell walls outside[24-26]. An additional sodium hydroxide ionizes the hydroxyl group to an alkoxide in the alkaline process[27]. The effect of this treatment is to increase the surface roughness of the fibres which will result in better mechanical interlocking with matrix materials.

2.4 The Process of Benzoylation

Composites undergo this treatment to increase their strength by decreasing the fibre's hydrophilic nature and improving its interaction with the matrix material's hydrophobic properties[27]. For 15 minutes, the suspended alkali-pretreated fibres were allowed to react with a 10% solution of sodium hydroxide and benzoyl chloride. Filtration is used to remove the fibres from the filtrate. After that, to get rid of any leftover benzoyl chloride, the fibres were immersed in ethyl alcohol for half an hour. After that, the fibres were rinsed with water many times. The next step was to dry them in an oven set at 800°C for a whole day. The hydroxyl groups on the fibres are substituted with benzoyl groups in this process. Manikandan Nair *et al.* performed fibre modification using benzoylation treatment[28].

Fibre –OH + NaOH → Fibre – $O^-Na^+ + H_2o$



Scheme 2. Reaction of Benzoyl chloride with treated fibre.

2.5 Fiber Chemical Composition Testing

Knowledge of chemical composition and surface adhesive bonding characteristics is needed for making natural fibre-reinforced composites.

2.5.1 Establishing Cellulose Concentration

About 1g of fibre sample is weighed accurately in an RB flask. To which sodium chlorite solution is added and reflexed for 1 hour. Then the residue is filtered through a weighed G3 crucible. Successive water wash is given to the crucible and is then kept in an air oven at 100±2 °C for 1 hour for drying. It is cooled in desiccators. The crucible is again weighed.

$$\%\ \text{Cellulose (by mass)} = \frac{(W3-W2)}{(W1-W)}\ X\ 100 \qquad (2)$$

In cases when,

The empty RB flask weighs (in grams) = W

When filled with fibre, the RB flask weighs W1 grams.

W2= The crucible's initial weight in grams

Finally, with the residual in g, the crucible's weight is W3.

The empty RB flask weighs (in grams) W = When filled with fibre, the RB flask weighs W1 grams.

W2= The crucible's initial weight in grams

Finally, with the residual in g, the crucible's weight is W3.

2.5. Finding the Concentration of Lignin

About 1g of the trial is evaluated accurately in an RB flask. To which 75% H_2SO_4 is added and is reflexed for 1 hour. Then the residue is filtered through a weighed G3 crucible in suction. The crucible is then kept in an air oven at 100±2 °C for 1 hour for drying and then chilled in a desiccator. The crucible is again weighed.

$$\% \text{ Lignin (by mass)} = \frac{(W3-W2)}{(W1-W)} \text{ X } 100 \quad (3)$$

Let,
The empty RB flask weighs (in grams) = W
When filled with fibre, the RB flask weighs W1 grams.
W2= The crucible's initial weight in grams
W3= The crucible's final weight in grams

2.5.3 Determination of Wax Content

About 2g of sample is weighed accurately in a weighed Whatman filter paper and is kept in Soxhlet apparatus. Petroleum ether is taken in a weighed RB flask and is fitted with the Soxhlet apparatus. The extraction is kept for 4 hours. Then the RB flask is kept in an oven at 100±2 °C for drying and then cooled in a desiccator and is again weighed.

$$\% \text{ Wax (by mass)} = \frac{(W3-W2)}{(W1-W)} \text{ X } 100 \quad (4)$$

W= Weight of filter paper in g
W_1= Weight of filter paper with fibre in g
W_2= Weight of empty RB flask in g
W_3 = Weight of RB flask with wax in g

2.5.4 Determination of Ash Content

About 2 to 3 g of sample fibre is weighed accurately in a silica crucible and is kept in a muffle furnace maintained at 650 °C for 1 hour. Then the crucible is cooled in a desiccator and is weighed.

$$\% \text{ Ash (by mass)} = \frac{(W2-W)}{(W1-W)} \text{ X } 100 \quad (5)$$

W= Weight of crucible in g
W1= Weight of crucible with fibre in g
W2= Weight of crucible with ash in g

2.5.4 Determination of Moisture Content

About 2 to 3 g of fibre sample is weighed accurately in a weighed petri dish. The dish is placed in an oven and dried at 100±2 °C for 6 hours. The dish is cooled in a desiccator and then weighed.

$$\% \text{ Moisture (by mass)} = \frac{(W1-W2)}{(W1-W)} \text{ X } 100 \quad (6)$$

W= Weight of empty dish in g
W1= Weight of the dish with fibre before drying in g
W2= Weight of dish with fibre after drying in g

2.5.6 FTIR Spectroscopy

FTIR prepares an infrared spectrum by which chemical bonds in a molecule can be detected. Infrared spectra of both untreated and chemically treated fibres were captured using a Nicolet is-10 spectrometer made by Thermo Fisher Scientific. The fibres were chopped to a length of about 2-3 mm and then ground to powder form. With the fibres, zinc-salenium crystal was used in the spectrometer to obtain the spectra. The section under "Results and Discussion" displays the fibre spectra.

2.5.7 X-Ray Diffraction Test

X-ray diffractograms were recorded on a PANALYTICAL-made X1PERT POWDER model diffractometer with generator settings of 30 Ma and 45kV and angular range from 10.00660(2Θ) to 89.99560(2Θ). The diffraction patterns were generated using a Cu Kα X-ray source. The program associated with the machine calculates the crystallinity percentage. Cellulose crystallinity can be expressed as the ratio of the amount of crystalline cellulose to the total amount of sample fibre.

2.5.8 Scanning Electron Microscopy (SEM)

SEM micrographs of raw and chemically treated fibres are presented in this work. A scanning electron microscopy of model Zeiss was used to study on surface morphology of both untreated and treated fibres. The fibres were sputtered with Gold. The tests were carried out with an accelerating potential of 3 KV for the electron beam used. The surface morphology analysis is of greater importance in characterizing structural changes that occur in chemical treatments.

2.5.9 Tensile Test

A tensile test of the fibres was carried out in the Zwick/RoellZ010 model machine with a load cell capacity of 100 N. The fibres were attached to the holder on both ends firmly to ensure a good grip. The machine makes use of testing software testXpert II and control technology testcontrol II. This test was conducted with a fibre gauge length of 50 mm at a test speed of 1mm/min according to the standard American Society for Testing and Materials ASTM D3822 (ASTM International, United States), 2001. Each fibre specimen was set to test with 20 samples of equal lengths.

3.0 Results and Discussions

3.1 Density

As discussed in the previous section, the density of the fibre was found, and its values are given in the table. The density of this fibre is almost equal to the Jute fibre shown in Table 1[25].

3.2 Chemical Composition Test

Table 2 shows the constituents of natural fibre before and after chemical treatments. There is a partial removal of lignin and other impurities.

3.3 FTIR Test

Figure 4(a) shows the presence of the OH group (O-H bond) of lignin at the stretching frequency of about 2950 cm^{-1}. There is an appearance of a steep peak of the carbonyl group of hemi cellulose (-C=O) at about 1750 cm^{-1}. A peak at about 1490 cm^{-1} is found in the spectra of untreated fibres which is attributed to the aromatic group of lignin (C=C). A trough appears at the stretching frequency of about 3280 cm^{-1}. It is evident from the Figure 4(b) that the partial removal of lignin occurs[9]. The shift of the OH group of lignin and carbonyl (C=O) of hemi cellulose to about 2920 cm^{-1} and 1710 cm^{-1}respectively is noticed. The peak intensity of the aromatic ring(C=C) of lignin is reduced to 1450 cm^{-1} compared to the intensity of the untreated fibre. The spectrum (Figure 4(c)) of 10% NaOH treated fibre also depicts the shifting of the two peaks of (OH) of lignin and (C=O) of carbonyl to about 2910 cm^{-1}and1690 cm^{-1} respectively. This shows clearly the further removal of lignin and hemicellulose by the alkali treatment. Figure 4(d) shows the presence of the benzoyl carbonyl group(C=O) at about 1700 cm^{-1} which is due to the benzoylation treatment of the fibre.

Figure 2 (a) shows the presence of the OH group (O-H bond) of lignin at the stretching frequency of about 2950 cm^{-1}. There is an appearance of a steep peak of the carbonyl group of hemi cellulose (-C=O) at about

Table 1. Density of fibres

Contents	Untreated	5% NaOH	10% NaOH	Benzoylation
Density g/cc	1.3175	1.3320	1.3537	1.3012

Table 2. Chemical compositions of fibres

Contents	Untreated	5% NaOH	10% NaOH	Benzoylation
Cellulose Content %	76.11	75.7	72.4	75.1
Lignin Content %	15.47	11.8	13.4	10.65
Wax Content %	0.43	0.22	0.13	0.16
Ash Content %	0.90	1.3	2.23	1.83
Moisture Content %	10.31	9.78	9.48	9.64

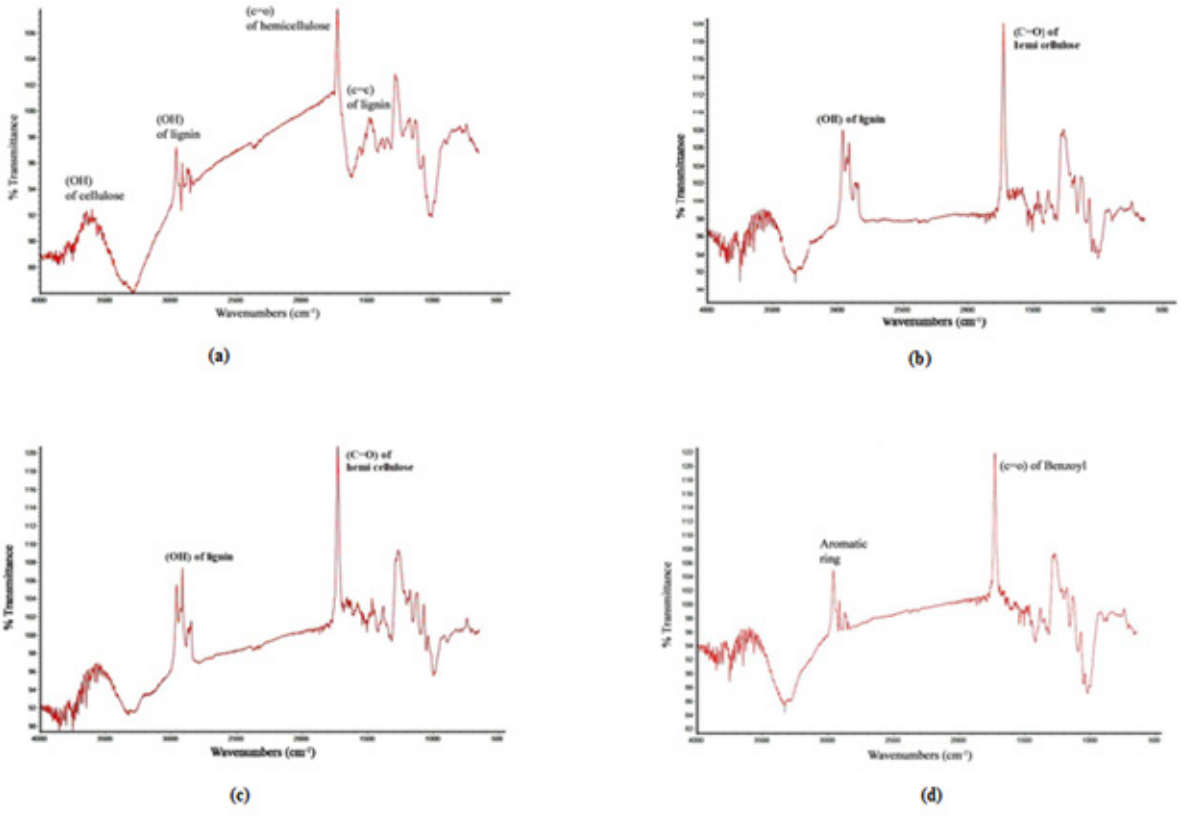
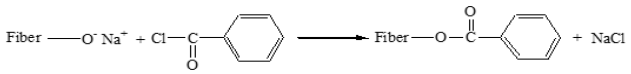


Figure 2. FT-IR spectrum of **(a)** Untreated **(b)** 5% Alkali treated **(c)** 10% Alakali treated and **(d)** Benzoylation treated fibre.

1750 cm^{-1}.A peak at about 1490 cm^{-1} is found in the spectra of untreated fibres which is attributed to the aromatic group of lignin (C=C). A trough appears at the stretching frequency of about 3280 cm^{-1}. It is evident from Figure 2(b) that the partial removal of lignin occurs[9]. The shift of the OH group of lignin and carbonyl (C=O) of hemi cellulose to about 2920 cm^{-1} and 1710 cm^{-1} respectively is noticed. The peak intensity of the aromatic ring(C=C) of lignin is reduced to 1450 cm^{-1} compared to the intensity of the untreated fibre. The spectrum (Figure 2(c)) of 10% NaOH treated fibre also depicts the shifting of the two peaks of (OH) of lignin and (C=O) of carbonyl to about 2910 cm^{-1} and 1690 cm^{-1} respectively. This shows clearly the further removal of lignin and hemicellulose by the alkali treatment. Figure 2(d) shows the presence of the benzoyl carbonyl group(C=O) at about 1700 cm^{-1} which is due to the benzoylation treatment of the fibre.

$$Fibre - OH + NaOH \rightarrow Fibre - O^{-}Na^{+} + H_2o$$

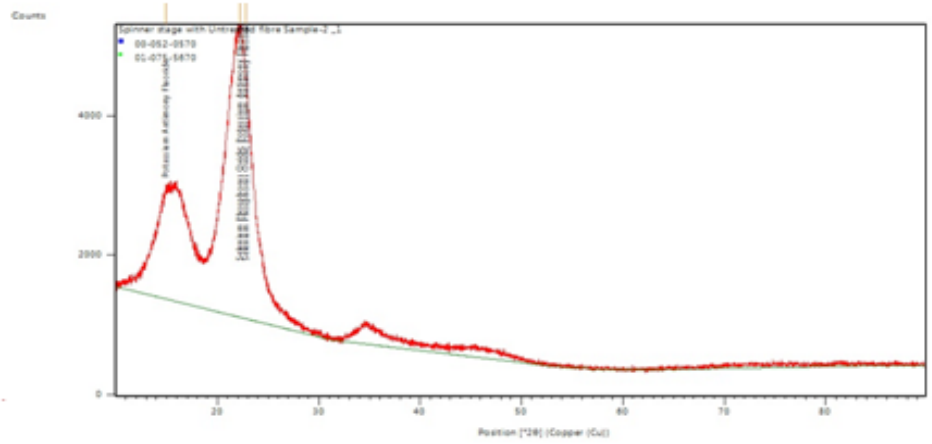


Scheme 3. Reaction of Benzoyl chloride with treated fibre

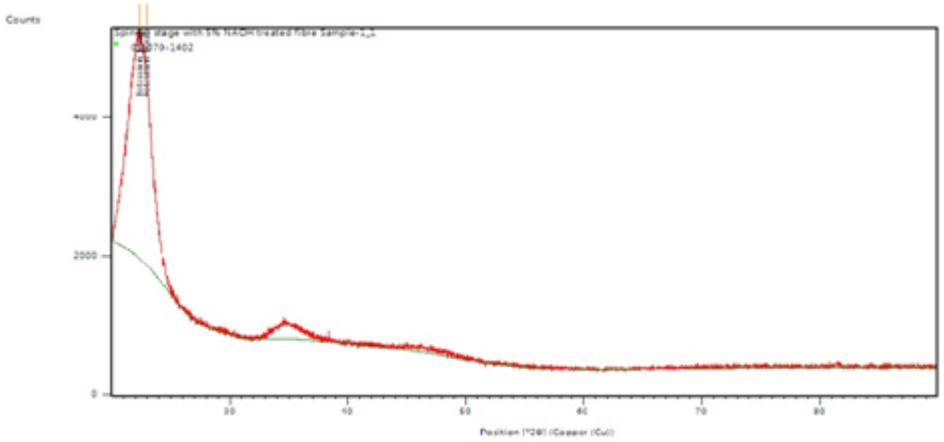
Also, there is the presence of a benzene ring at the stretch frequency of about 3000 cm^{-1} which reflects the replacement of the OH group in fibre. The peaks at a stretch frequency of 3550-3600 cm^{-1} range show the presence of the OH group of cellulose both in untreated and treated fibres. This indicates that there is no change in the chemical structure of cellulose in alkali and benzoylation treatments. But there is a change in structure physically such that from a compact packed structure of cellulose to an amorphous structure forming spaces within the cellulose molecules as mentioned in the paper by authors Kabir *et al*[18].

3.4 XRD Test

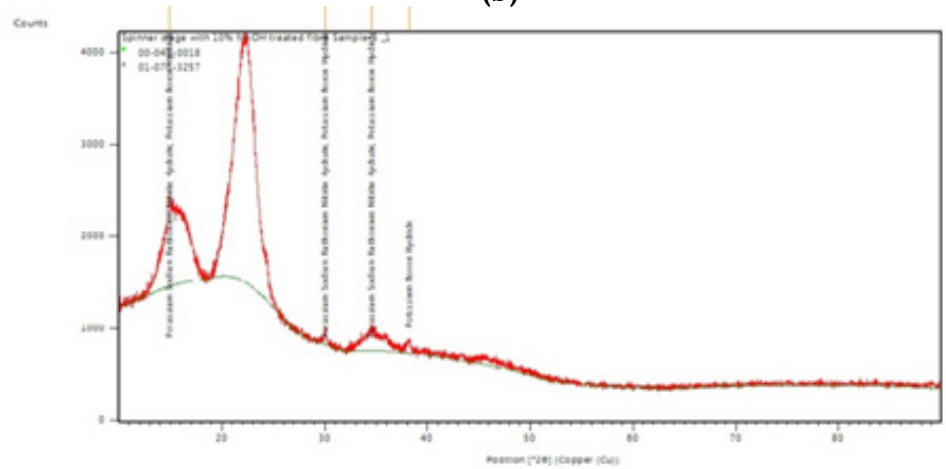
Figure 3 shows X-ray diffraction patterns of the four sample fibres. The counts at 2Θ value of 22° peak intensity represent crystalline material and 2Θ value of 18° peak intensity represent amorphous material in the cellulose. The crystallinity percentage values of the sample fibres are shown in Table 3.



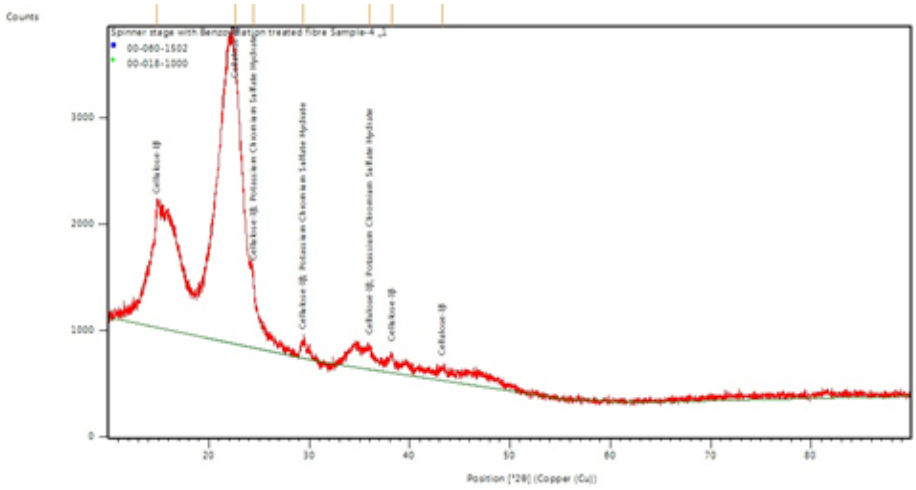
(a)



(b)



(c)



(c)

Figure 3. XRD diffraction patterns of **(a)** Untreated **(b)** 5% Alkali treated **(c)** 10% Alakali treated and **(d)** Benzoylation treated fibre.

Table: 3. Crystallinity % of fibres

S. No	Sample Fibre	Crystallinity %
1.	Untreated	47.63
2.	5% NaOH treated	33.41
3.	10% NaOH treated	43.55
4.	Benzoylation treated	43.48

The untreated fibre sample shows a higher crystallinity percentage, and the values have no relevance with tensile strengths. The 5% NaOH treated fibres have the lowest crystallinity percentage which is evident from the graph as it has diffraction peaks only at angles 8° and 34.5° the other samples have a greater number of peaks especially the highest peaks at about angle 22°. Also, the 5% NaOH-treated fibre sample has the maximum intensity of diffraction. From the results of the tensile test, the 5% NaOH treated sample has higher tensile values. And it can be seen except in the 5% NaOH treated sample that there are broader peaks in the diffraction pattern which represent the presence of non-cellulosic compounds. But compared to the 5% NaOH treatment, 10% NaOH and benzoylation treatments provided improved values which may be attributed to better orientation of crystals of cellulose.

3.5 SEM Test

The surface morphology of raw and chemically treated fibres has been examined using SEM equipment of model Zeiss. By comparing the raw and treated fibres, one may observe the structural changes that have taken place in the fibres.

Surface morphology photographs of both treated and untreated fibres are shown in Figure 4 using scanning electron microscopy. The pictures show that the treated fibres have a distinct surface morphology than the untreated ones. Impurities such as lignin and pectin blanket the raw fibres, as seen in Figure 4(a). Chemical treatments are used to eliminate fibre impurities, such as lignin and pectin, which can make the surface look rough. According to the statement cited in the article of authors Mohd Edeerozey A M *et al.*, this characteristic is needed to improve good adhesion of the fibre with matrix material even though this work is not associated with any matrix materials[29]. Figure 4(b) shows that in the 5% treated fibre, many of the impurities are removed and it gets further improvement in 10% NaOH treatment. Further, better removal of fibre impurities is observed in benzoylation-

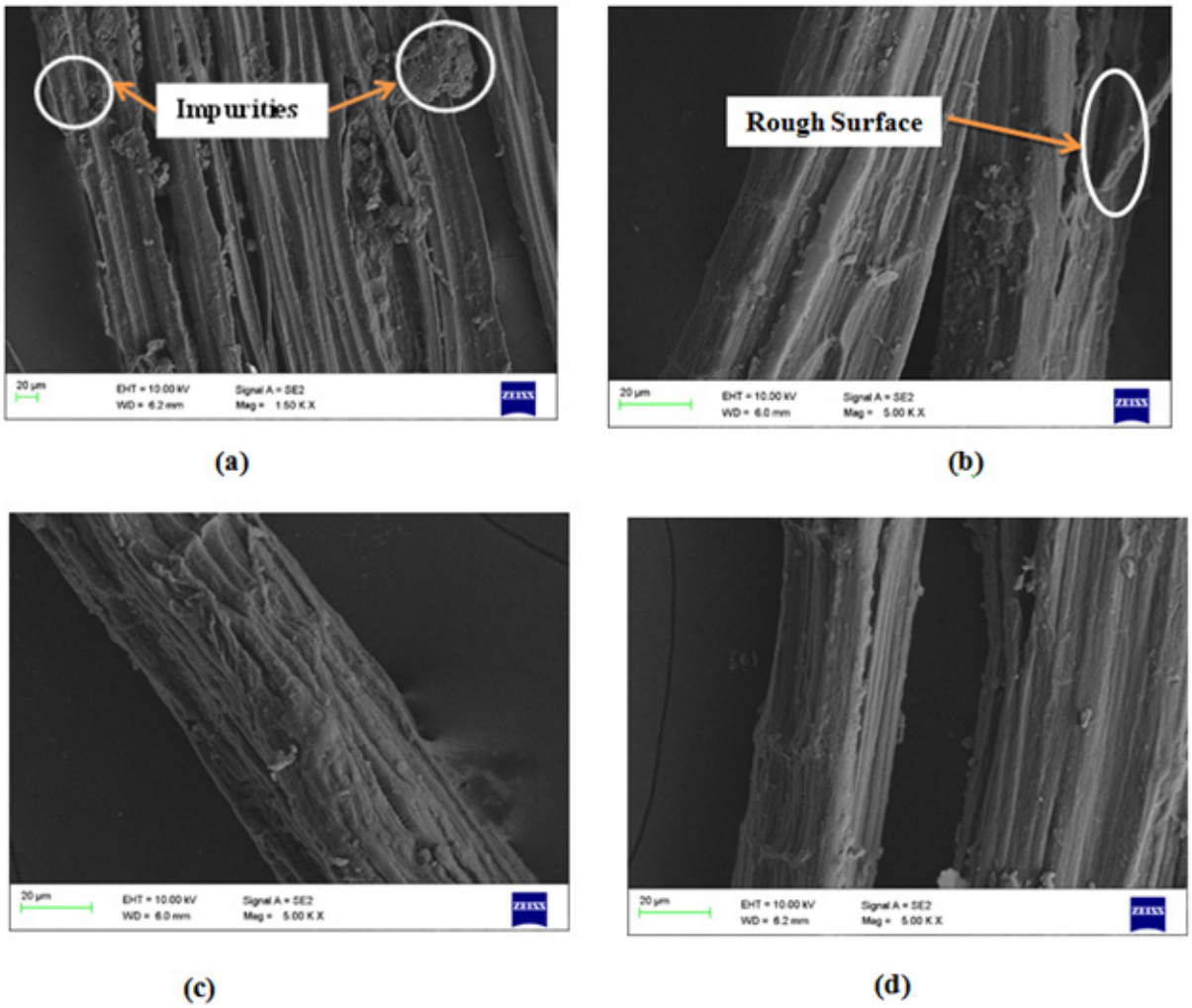


Figure 4. SEM micrographs of **(a)** Untreated **(b)** 5% alkali treated **(c)** 10% alkali treated **(d)** Benzoylation treated fibre

treated fibre as shown in Figure 4(d). Improved surface roughness is felt by touching them[29].

3.6 Tensile Test

Tensile properties for untreated, alkaline treated (5% and 10%) and benzoylation-treated fibres were obtained from force/elongation curves. Twenty samples have been tested in each case. Figure 5(a) shows the response of the untreated fibres to tensile force and elongation. The diagram reveals that maximum tensile force and elongation are 3.75 N and 5.85 % respectively. Figure 5(b) shows that the maximum tensile force and elongation for 5% alkali-treated fibres are 3.45 N and 3.8 % respectively. Similarly, these values for the other two samples can be inferred from Figures 5(c) and 5(d). They are 4.0 N and 3.1% and 3.58N and 3.46 % respectively. But more correct results can be obtained from their mean values.

The bar chart is plotted for the mean tensile forces of four sample fibres. The mean tensile force and elongation of untreated fibre are 1.79 N and 1.3% respectively. For 5% and 10% alkali-treated fibres, they are 2.07 N and 1.5% and 1.77 N and 1.5% respectively. The benzoylation-treated fibres have mean values of 1.59 N and 2.0%. Hence the treated fibres possess better tensile properties due to the removal of fibre impurities than the untreated fibres. It depicts that maximum tensile value occurred in 5% NaOH treated fibres. The value decreases in successive sample fibres. Normally partial removal of lignin and

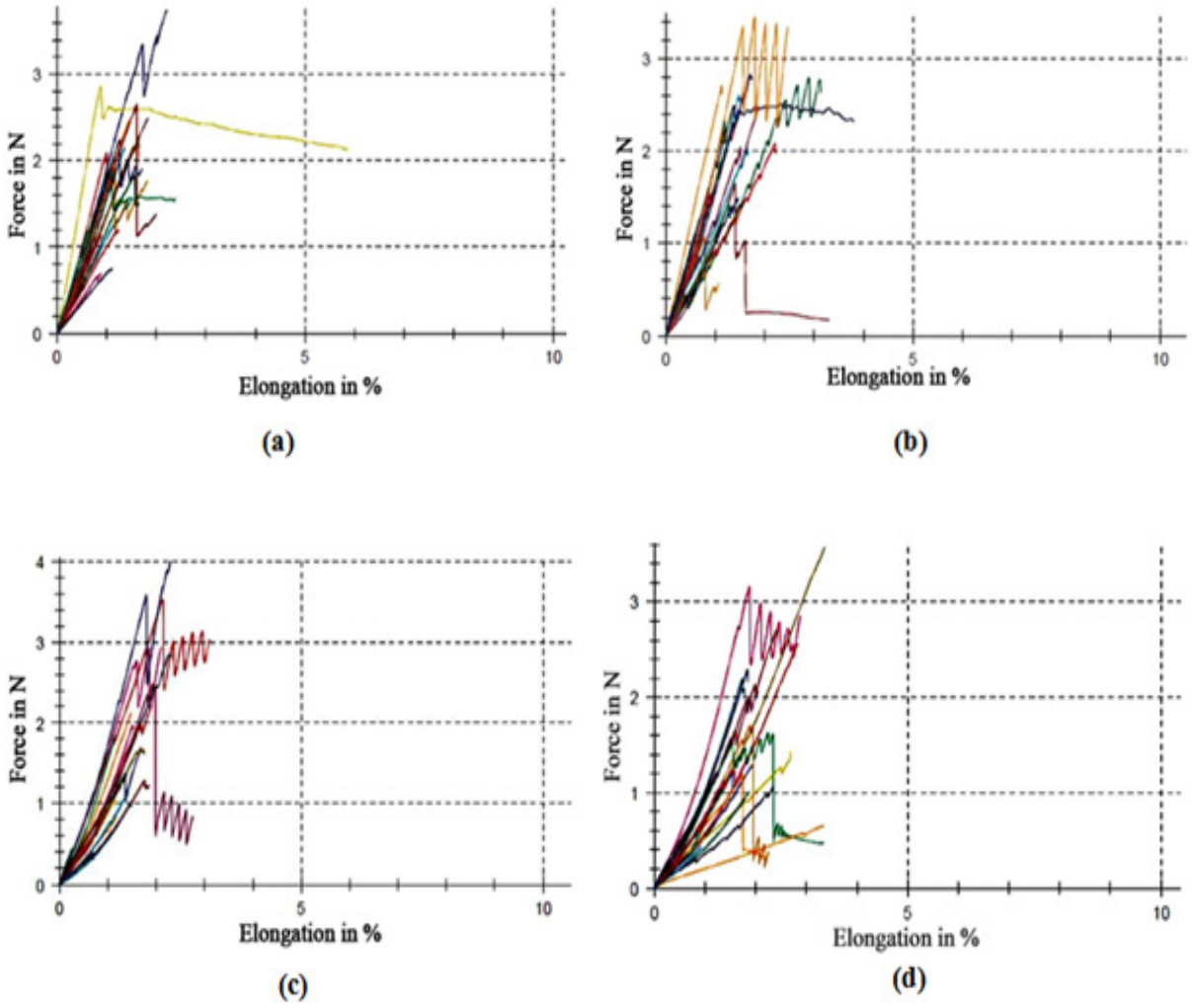


Figure 5. Effect of Tensile force on Elongation of **(a)** untreated **(b)** 5% alkali treated **(c)** 10% alkali treated and **(d)** Benzoylation treated fibres

hemicellulose occurs during NaOH treatment which makes the interfibrillar region less dense and less rigid comparatively. While tensile testing, the fibrils rearrange themselves along the direction of loading thus improving the tensile values. Figure 6(b) shows the bar chart plotted for mean elongation in % for the four samples. Generally, the structure and properties of the natural fibres are capricious[7]. The benzoylation-treated fibres show the maximum % elongation. 5% and 10% NaOH treated fibres exhibit the same value as % elongation. The fibre material is not homogeneous and hence mean tensile force has been taken to prepare the bar chart. Similarly, the mean elongation has been used to make the bar chart. The tensile strength of fibre values is depicted in Table 4.

There are improvements in tensile strengths in treated fibres compared to the untreated. 5% NaOH-treated fibres show a higher tensile value which may be due to the partial removal of impurities and improved alignment of fibrils along the loading direction. With a further increase in NaOH concentration to 10% and in benzoylation-treated fibres, there is a reduction in strength which may be attributed to damage to the fibres. These can be seen in SEM images. Table 5 shows the tensile strength of some natural fibres.

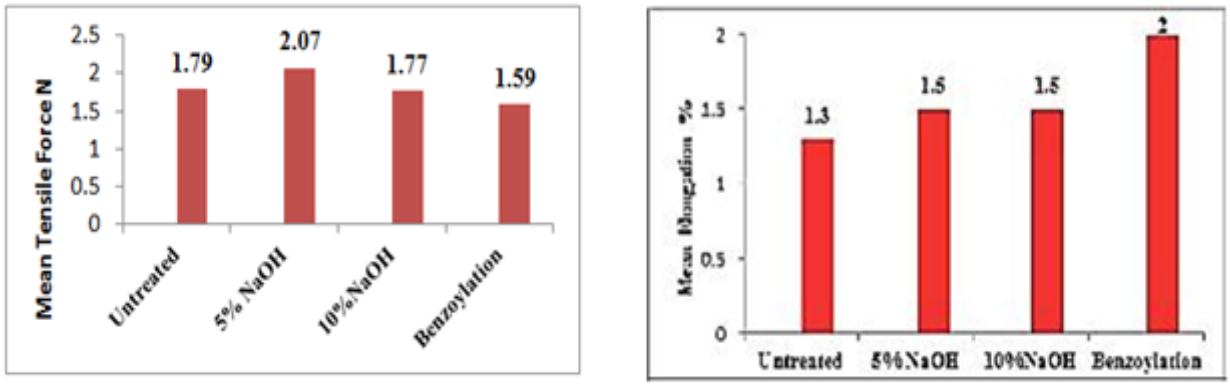


Figure 6. **(a)** The mean tensile force **(b)** The mean elongation of various fibres.

Table 4. The tensile strengths of fibres

Contents	Untreated	5% NaOH	10% NaOH	Benzoylation
Mean Diameter in mm	67.57 x 10^{-3}	54.44 x 10^{-3}	55.30 x 10^{-3}	52.97 x 10^{-3}
Mean Tensile Force in N	1.79	2.07	1.77	1.59
Tensile Strength in N/ mm^2	499.43	889.70	737.32	721.88

Table 5. The tensile strength of some natural fibres[17]

S.NO	Fibres	Tensile Strengths MPa
1.	Cotton	287-597
2.	Jute	393-800
3.	Flax	345-1500
4.	Hemp	550-900
5.	Sisal	400-700
6.	Coir	175-220

The above table shows the tensile strength values of some natural. Concerning the above values, the tensile strength values of *Hibiscus rosa-sinensis* fibres are comparable.

4.0 Conclusion

The test results have revealed that.

Chemical treatments of the fibres have been found to remove partially deleterious constituents like lignin and hemicellulose which are observed from FTIR spectra.

There is a presence of OH group of cellulose both in untreated and treated fibres.

5% NaOH treatment has aided in improving the tensile value and Benzoylation treatment has been found effective in improving the mean value of % elongation at maximum tensile force.

Chemical treatments partially remove impurities like lignin and pectin and morphological changes like surface roughness occur in the fibres. This is obvious from the SEM images.

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