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Thermo-mechanical properties of sodium chloride and alkali-treated sugarcane bagasse fibre

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The experimental characterization of mechanical and thermal properties of treated and raw sugarcane bagasse fibre has been studied. The bagasse fibres are treated with sodium chloride (NaCl) and sodium hydroxide (NaOH) solutions. The NaOH treated fibres show better structural and thermal properties than other two types. SEM image of alkali-treated fibres reveals that the bundles of fibres are mainly composed of thin parenchyma cell walls. The fibres are joined with each other which improves the mechanical properties. The statistical analysis is also performed using ANOVA one-factor method. From ANOVA, the significant difference between the dependent parameters and the various chemical treatments are determined. The results show that the NaCl and NaOH treated fibres significantly improve the mechanical properties and thermal stability.

Keywords: Alkaline treatment, Bagasse fibre, Crystallinity index, Mechanical properties, Sugarcane fibre, Thermo-mechanical properties

1 Introduction

Sugarcane is major agricultural crop grown in sub-tropical and tropical countries like India, China, Thailand, Australia, and Brazil[1-3]. Brazil is the massive sugarcane producer and provides 25% of total production in the world. Among 200 countries from the world, India is the second largest manufacturer of sugar[1-3]. The bagasse is an agricultural residue obtained after crushing and separation of juice from sugarcane. This waste bagasse of 540 million tons is generated every year throughout the world[1-3]. The bagasse mainly consists of 25% lignin, 25% hemicelluloses, and 50% cellulose. The bagasse is used to manufacture value-added substances such as ethanol, food ingredients and other chemical components. These types of substances also offer environmental, strategic and economic advantages for sugarcane manufacturing companies[4].

From past decades, the chemical, physical, thermal and biological treatments on sugarcane bagasse fibre were conducted with the help of liquid hot water and aqueous ammonia[5, 6]. Among the various traditional treatment process, hot water and alkaline pretreatment were used for determining the fractions of lignin, cellulose and hemicellulose. In hot water pretreatment, there is no additional chemicals used. But it is operated in liquid state at elevated temperature. However, the alkaline pretreatment increases the digestibility of cellulose. It is mainly used for lignin solubility as compared with hot water treatment[6, 7].

NaOH pretreatment improves cellulose surface and decreases crystallinity[8]. In another study[9], the bagasse was treated with 10% of sulphuric acid (H_2SO_4) solution at 120 ºC for 10 min, and then bleached with NaCl and acetic acid under the vacuum condition. The wax, lignin, pectin and hemicellulose residuals were removed by this treatment. Asagekar and Joshi[10], soaked bagasse in water at a proportion of 1:50 for three months, then pre-treated the bagasse samples at 90 ºC for one hour. Finally, the bagasse was chemically modified with 0.1 N NaOH solution at boiling water temperature for 4 hours. From the results, it was found that there is no significant difference, and specific trend was observed.

From the literature survey, it is concluded that boiling, bleaching and alkaline treatment not only reduces the effect of lignin but also damages the fibre crystallinity. Therefore, a new processing method has been introduced in this study to improve the bagasse properties as compared to traditional process. In this process, the extraction of sugarcane fibre has been

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carried out using salt solution (NaCl) and combined (NaCl and NaOH) treatment [9-10]. The main objective of this work is to examine the effect of salt solution and combined treatment on mechanical, thermal and morphological properties of extracted sugarcane bagasse fibre.

2 Materials and Methods

2.1 Sugarcane Bagasse Fibre Extraction

Sugarcane bagasse was collected in the form of rolled press stalk from various juice shops. The collected bagasse was washed with fresh tap water to remove impurities and other sugar residues, which is then denoted as sugarcane raw sample (SRS). The salt water solution was prepared with NaCl and fresh tap water in the ratio of 1:5 at 6.5 *p*H. The sugarcane fibres were soaked in NaCl solution for 2 days in atmospheric condition. Then dried in sunlight for 8 hours to remove the residual moisture content. This is considered as salt solution treatment (SST). After the SST, the bagasse was treated with 0.1 N NaOH solution under boiling temperature for 4 hours to remove lignin content. This treatment process is denoted as combined effect of salt and alkaline treatment (SAT).

2.2 Chemical Composition of Sugarcane Fibre

The chemical constituents of sugarcane, such as lignin, cellulose, and hemicellulose of SRS, SST and SAT fibres were measured using ASTM D1106-56, D1103-55T and D1104-56 respectively. The standard deviations were also estimated by performing ten measurements for every sample. The cellulose, lignin and hemicellulose contents were evaluated using the following equations[11, 12].

$$\text{Cellulose (\%)} = \frac{\text{Weight of oven dry cellulose residue}}{W \times P} \times 100 \quad \ldots(1)$$

$$\text{Lignin (\%)} = \frac{\text{Weight of oven dry lignin residue}}{W \times P} \times 100 \quad \ldots(2)$$

$$\text{Hemicellulose (\%)} = \frac{\text{Weight of oven dry hemicellulose residue}}{W \times P} \times 100 \quad \ldots(3)$$

where W is the original weight of oven dry fibre sample; and P, the proportion of moisture-free content.

2.3 Fibre Colour and Cleanliness

The colour of treated and raw sugarcane bagasse was examined by using UV-1800, UV-VIS spectrophotometer (Shimadzu, Kyoto, Japan). This test was performed under the controlled illumination. The sugarcane bagasse sample was rotated at 90º within the measurements and the mean value was considered. Simultaneously, the extracted fibres were placed on micro dust trash analyzer (Hollingsworth Uster, MDTA 3, Poland) to determine the cleanability[11-13]. Both the tests were carried out at 30±2 ºC temperature, and 65±2% relative humidity.

2.4 Fibre Linear Density

The fibre linear density of treated and raw thin cylindrical sugarcane bagasse fibres was measured. The test was performed according to ASTM D 1577-07 standard. The bundles of sugarcane fibres were arranged in longitudinal direction. The fibre linear density of extracted sugarcane fibre was calculated using the following equation:

$$T_d = \frac{10000 \times W}{L \times N} \quad \ldots(4)$$

where T_d is the fibre linear density; L, the length of fibre bundle (m); N, the number of fibres in bundle; and W, the weight of fibre bundle (kg).

2.5 Mechanical Properties

The various mechanical properties, such as tensile, flexural and torsional rigidity were also measured. The tensile test of single fibre bundle (250 × 2 mm) was performed in Tinius Olsen (H-5kN capacity, Universal testing machine, USA). This test was performed with cross head speed of 2 mm/min, according to ASTM D 3379-75 standard.

In flexural test, the single fibre was converted into ring shape, hanged and loaded by a rider. The fibre was placed in the shear test set up and maximum load of 30 g (0.2943 N) was applied. The circular dimensions change at its lower end and flexural rigidity measured[10]. Torsional rigidity test was performed using torsional pendulum consisting of a disc connected with fibre bundle[10].

2.6 FTIR Study

FTIR spectra of treated and untreated sugarcane fibres were examined using FTIR spectrometer (Perkin Elmer Spectrum 2, USA). The spectra were recorded in potassium bromide (KBr) matrix in transmission mode between 4000 cm^{-1} and 400 cm^{-1}. The resolution of 1 cm^{-1} and scan rate of 32 scans/min. also considered.

2.7 XRD Study

X-ray diffraction (XRD) of the extracted sugarcane bagasse fibre was performed using Rigaku Ultima IV X-Ray diffractomter. The operating parameters are 40 kV voltage, 5º–80º with step size of 0.05º diffraction angle and 40 mV intensity. The entire tests were carried out three times to assure reproducibility. The degree of crystallization of the sugarcane fibre was calculated using the following equation:

$$\text{Degree of crystallization} = \frac{I_{002} - I_{Amorph}}{I_{002}} \times 100 \quad \ldots(5)$$

where I_{002} is the maximum intensity of the diffraction; and I_{Amorph}, the diffraction intensity at 18º.

2.8 Thermal Characterization

Thermogravimetric analysis (TGA) and differential scanning calorimeter (DSC) test were performed for both treated and untreated sugarcane fibres. The DSC and TGA tests were conducted in Perkin Elmer DSC 8000 and TGA 4000 respectively. The 5 mg of sample was heated from 28 ºC to 600 ºC at the rate of 10 ºC/ min under nitrogen atmosphere[9].

2.9 Morphological Properties

The longitudinal surface of treated and untreated sugarcane bagasse fibres was sputtered with gold ion for 90 s. The gold ion sputtering converts the material as conducting medium. The microstructures of both treated and untreated fibres were examined in scanning electron microscope (FE-SEM, Hitachi, Europe) under 15 kV (vacuum condition).

2.10 Statistical Analysis

The statistical calculations were carried out for both treated and raw sugarcane bagasse fibre samples. From one way ANOVA method and SPSS software, the significance of results was tested under 95% confidence level.

3 Results and Discussion

3.1 Effect on Chemical Composition

Table 1 presents the chemical composition of treated and raw sugarcane bagasse. The raw sugarcane bagasse has 46.78% of cellulose, 22.36% of lignin and 24.21% of hemicellulose. During the salt solution treatment process, the depolymerization of lignin and hydrolysis of hemicellulose fractions are facilitated. Similarly, in combined effect the lignin content gets dissolved in alkali. The substantial lignin content is removed during the combined treatment[11]. The combined treatment reduces the residual hemicellulose and lignin contents. From this effect, 76.03% of cellulose in sugarcane fibre is observed in comparison with raw bagasse (Table 1). The combined effect also decreases the lignin and hemicellulose content from 22.36% to 9.25% and from 24.21% to 8.74% respectively. It is concluded that both salt solution and combined effect remove lignin and hemicellulose content.

3.2 Effect on Colour and Cleanliness

The colour and cleanliness of both types of fibres are depicted in Table 2. The collected raw bagasse sample has very light yellow colour scheme. The raw

Table 1 — Chemical composition of treated and raw sugarcane sample

Sample (code)	Cellulose %	Hemicellulose %	Lignin %	Solid yield, %
Raw (SRS)	46.78±0.21	24.21±0.84	22.36±1.67	100
NaCl treated (SST)	61.57±1.23	9.48±1.07	10.54±1.87	62.84
Combined NaCl + NaOH treated (SAT)	76.03±1.26	8.74±0.97	9.25±1.04	68.11

Table 2 — Properties of treated and raw sugarcane fibre

Parameter	SST	SAT	SRS
Fibre colour	Light yellow	Dark yellow	Very light yellow
Fibre cleanliness	Clean surface	Clean surface	Presence of dirt particles
Linear fibre density, tex	28.48±(2.36%)*	35.52±(2.54%)*	27.76±(2.08%)*
Crystallinity index, %	35.19±(1.74%)*	63.15±(1.17%)*	28.95±(2.04%)*
Flexural rigidity, Nm^2	3.48×10^{-6} ±(2.11%)*	4.32×10^{-6} ±(2.07%)*	3.42×10^{-6} ±(1.87%)*
Torsional rigidity, Nm^2	3.15×10^{-6} ±(1.89%)*	4.67×10^{-6} ±(1.96%)*	3.56×10^{-6} ±(2.11%)*
Tensile strength, MPa	238.7±(2.01%)*	289.7±(2.16%)*	208.4±(1.05%)*
Elongation at break, %	6.33±(1.48%)*	7.02±(1.08%)*	5.28±(0.54%)*
Modulus of Elasticity, GPa	18.06±(3.23%)*	21.6±(3.17%)*	16.32±(1.54%)*

*Values within bracket denote the uncertainty values.
SST – Salt solution treatment, SRS– Sugarcane raw sample, and SAT –Salt and alkaline treatment.

sample is mainly consists of dust particles. After NaCl treatment, the colour of bagasse changed into light yellow. This change in colour is due to the chemical reaction. Simultaneously, NaOH treatment converts bagasse in dark yellow. This is due to the presence of high cellulose content which is observed in Table 1.

3.3 FTIR Spectral Analysis

FTIR spectra of SST, SAT and SRS sugarcane fibres are shown in Fig. 1(a). FTIR spectra of SST and SAT fibres show peaks at wavelengths 3500-3000 cm^{-1}, due to –OH stretching. It describes the hydrophilic capacity of the fibres. The band at 3000-2800 cm^{-1} relates to C-H asymmetric stretch bonds. This explains the presence of aliphatic bonds in cellulose. The peaks at 1650 cm^{-1} and 1770 cm^{-1} shows the bending mode or C=O stretch of absorbed water. The various peaks at 1345 cm^{-1} and 1450 cm^{-1} signify CH_2 asymmetric and symmetric bending respectively[11, 14]. The peak at 1120 cm^{-1} corresponds to C-H in plane deformation. This shows lignin or stretching of cellulose glucose ring. The peak at 1185 cm^{-1} relates to C-O-C symmetric stretching. A peak at 1070 cm^{-1} is attributed to C-O-C pyranose ring skeleton. The peak at 1025 cm^{-1} is assigned to C-O-C (ether linkage), which depicts small amount of hemicellulose and lignin. Peak at 890 cm^{-1} corresponds to β-glucosidic network in cellulose fibre[11, 15]. The tow peaks nearly at 890 cm^{-1} and 1450 cm^{-1} are contributed to amorphous regions of cellulose. These peaks are also integrated with β-glucosidic network. From these, it is observed that hemicellulose and lignin are removed (Fig. 1). An important peak at 1750 cm^{-1} in raw bagasse spectra is assigned to uronic ester or acetyl groups of hemicellulose. The ester network shows the ferulic and p-coumeric acids of hemicellulose or lignin[11]. This peak is completely vanished in the SST and SAT samples which shows the removal of lignin and hemicellulose. Peaks at 1525 cm^{-1} and 1260 cm^{-1} in SRS sample shows the aromatic C=C stretch bond of lignin (aromatic rings). In SST and SAT spectra, the peaks at 1525 cm^{-1} and 1260 cm^{-1} do not exist. This signifies that the lignin content is reduced and cellulose is separated[14, 15]. The peak at 650 cm^{-1} relates to C-O-H out of plane bending in cellulose fibre. The increase of wavenumber at 890 cm^{-1} in both SST and SAT spectra shows the typical cellulose.

3.4 XRD Curves Study

The crystallinity index of cellulose fibre is very important parameter to determine the structural properties. XRD curves of SST, SAT and SRS sugarcane bagasse fibres are shown in Fig. 1 (b). Different peaks are observed on XRD curves of SRS, SST and SAT fibres at same diffraction angles with dissimilar intensities. XRD spectra of SST and SAT present four different peaks at 15.8º, 22.11º, 28.54º and 34.7º. These peaks are attributed to (110), (200), (111) and (210) crystallographic planes of monoclinic lattice. All these types of planes are the characteristic curves of native cellulose. It is clearly observed that SST and SAT samples are crystalline in nature. The peak intensity at 15.8º and 22.11º increases with NaCl and combined treatment[11]. It is evident from the XRD curve, that on NaCl and NaOH treatment, the cellulose crystalline structure does not change. The crystallinity index of SST (35.19%) and SAT (63.15%) fibres are higher than raw bagasse (28.95%), as shown in Table 2. The higher value of crystallinity index clearly signifies the substantial removal of hemicellulose and lignin from raw sugarcane (Table 1).

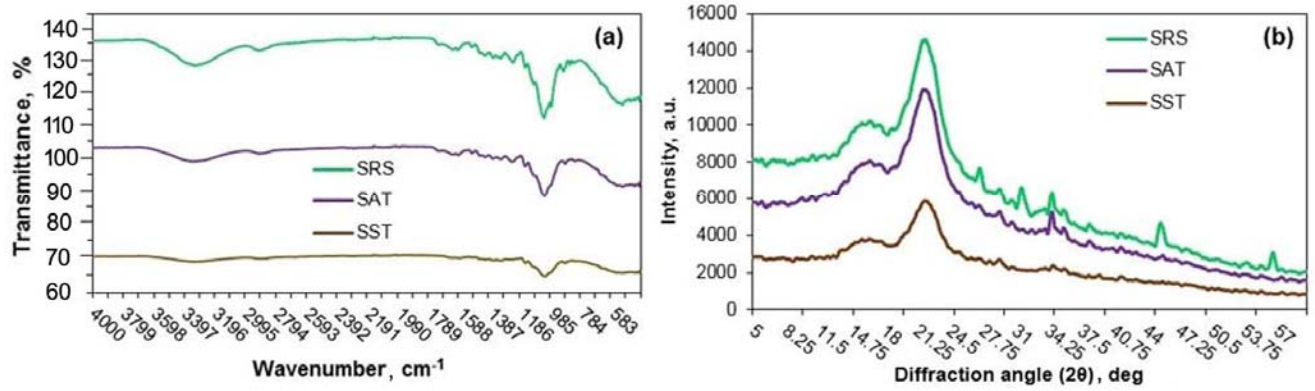

Fig. 1 — Treated sugarcane bagasse fibre structural characteristics curves (a) FTIR and (b) XRD

3.5 Effect on Linear Fibre Density

The gravimetric test has been conducted for five bundles of fibre and mean value is observed in Table 2. It is noticed that SAT bagasse fibre has improved linear fibre density by 27.56% as compared with SST and SRS. This describes the absence of lignin, as also supported by FTIR spectra and higher crystallinity index.

3.6 Effect on Mechanical Properties

The various properties of treated and raw sugarcane fibres, such as flexural, torsional rigidity, tensile strength, elongation-at-break and modulus of elasticity are also shown in Table 2. The stress-strain graph for both treated and untreated fibres are shown in Fig. 2. From the stress-strain graph, the stress differs first linearly, then quasi-linearly with increment in strain rate till the maximum value. Finally, immediate decrease is observed in stress without damaging on fibre. The SAT fibres has higher tensile strength, elongation, modulus of elasticity, flexural and torsional rigidity, and showing improvement by 39.01, 32.95, 24.44, 26.31 and 31.18% respectively as compared to raw sugarcane fibre. This effect is due to the presence of high cellulose content observed in its chemical composition. It is also noticed that SAT fibre samples have quite higher value of flexural and torsional rigidity than traditional treatment. Both treated and untreated sugarcane have lower torsional and flexural rigidity, and hence can be applied in nonwoven type of fabrics[10]. Table 3 shows the comparison of mechanical properties of sugarcane fibres with other natural fibres. There is a slight improvement in tensile properties of SAT sugarcane fibre as compared to cotton and jute.

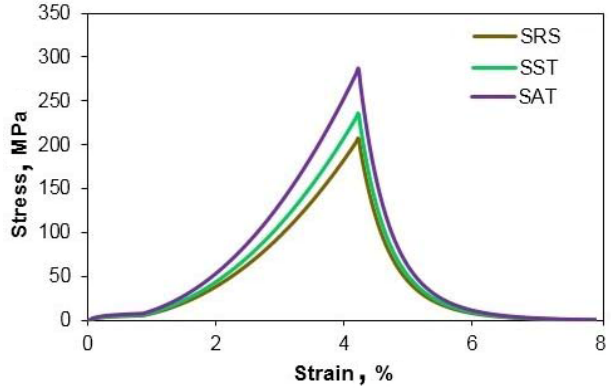

Fig. 2 — Stress-strain graph of treated and untreated sugarcane bagasse fibre

3.7 Thermal Properties

Figures 3 (a) and (b) show TGA and DSC curves respectively for both treated and untreated sugarcane fibres. The TGA curves [Fig. 3 (a)] show the weight loss occurred in both treated and untreated sugarcane fibres from 28 ºC to 300 ºC. This effect is due to the evaporation of moisture content. The fibre is dried before the analysis but elimination of water content is difficult due to hydrophilic nature[23]. From temperature 350 ºC to 500 ºC, the treated and raw bagasse shows thermal stability. From above 500 ºC, the treated and raw bagasse samples indicate large amount of mass loss. This is due to the degradation of

Table 3 — Comparison of mechanical properties of sugarcane fibre with other natural fibres[16-22]

Fibre	Tensile strength MPa	Modulus of elasticity, GPa	Elongation at break, %
SAT	289.7	21.6	7.02
Cotton	287 - 800	5.5 - 12.6	3 - 10
Hemp	690	60 - 70	1.6 - 4
Jute	393 - 780	13 - 30	1.9
Flax	345 - 1500	23.9 - 27.6	1.6 - 3.2
Ramie	400 - 938	44 - 128	1.2 - 8

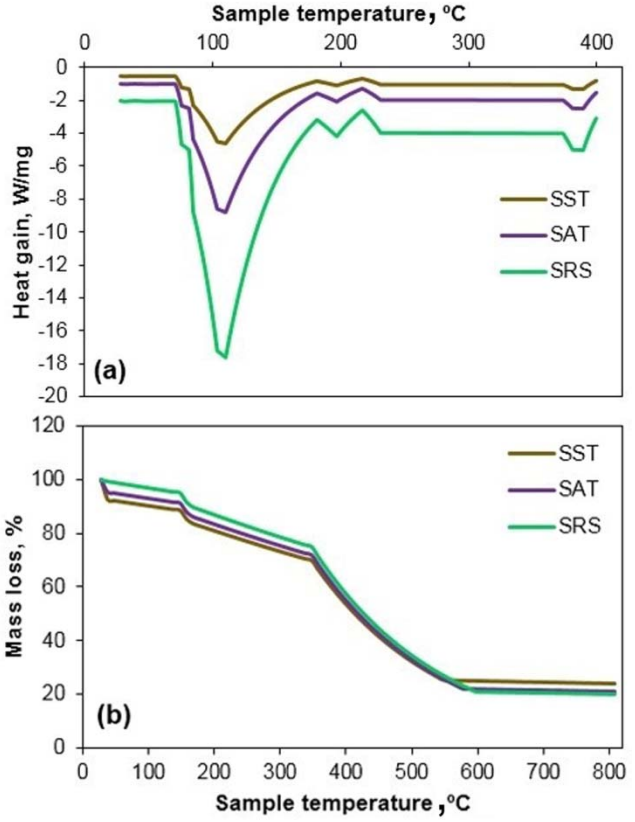

Fig. 3 — Treated sugarcane bagasse fibre thermal characteristics curves (a) DSC and (b) TGA

both cellulose and hemicellulose contents from sugarcane. After 550 ºC, the degradation also occurs with breakage in protolignin bonds[24]. The decomposition temperature of SST, SAT and SRS sugarcane fibres are 402 ºC, 408 ºC and 440 ºC respectively [Fig. 3 (a)]. This is because of the reduction of hemicellulose and lignin from SST and SAT fibres, observed in chemical composition.

From DSC curves [Fig. 3 (b)], it can be observed that the peaks of treated and untreated sugarcane fibres are displayed at around 250 ºC. This signifies the degradation of cellulose from fibre. The peaks between 400 ºC and 450 ºC also relates the breaking of chemical bonds of protolignin[16]. The raw sugarcane shows an endothermic peak at 120 ºC due to burning. The endothermic peaks lies 30 ºC and 140 ºC for SST due to water vaporization. The second peak appears at 370 ºC and 390 ºC, associated with degradation of cellulose[25]. In combined effect, endothermic peaks are appeared between 30 ºC and 120 ºC. The second peak lies at 365-385 ºC which degrades the cellulose and hemicellulose. This mainly occurs due to the formation of anhydro glucose and volatile substances[23].

3.8 Effect on Morphological Properties

The microstructure of sugarcane fibre is examined using SEM to determine the effect of salt solution and combined treatments. Figure 4 (a) shows SEM image of sugarcane fibres obtained after salt solution treatment. The sample shows fibre bundles having smooth surface after the removal of lignin, hemicellulose and pectin. It is found that average diameter of fibre (25 μm) is lower in the raw bagasse. Figure 4 (b) shows the morphology of sugarcane fibre treated with both NaCl and NaOH solution. It is also observed that bundle of fibre is mainly composed of thin parenchyma cell walls. The fibrils are joined with each other, which improves the mechanical properties. The SEM image of raw sugarcane bagasse fibre [Fig. 4 (c)] shows rough structure, dirt and bundles of thick walled parenchyma cells which has lower tensile, flexural and torsional rigidity.

3.9 Statistical Analysis

Statistical analysis was done by employing the one-parameter ANOVA technique to all experimental outcomes with different treatment process. The sum of squares, degrees of freedom, mean sum of square, fisher modulus and probability are shown in Table 4. The mean sum of squares can be defined as the ratio of sum of squares to degrees of freedom[26]. F-value also indicates the ratio between experimental error and mean sum of square. In this design, f-value can be applied as approximate perceptive to relative effects on factor. If the value of F is lower than critical value of F, then the effects will become insignificant. The independent parameters in the analysis is indicated by all SST, SAT and SRS fibres. The dependent parameter is tensile strength at failure for 10 samples under each group. The same method is also followed for elongation, modulus, fibre density, crystallinity and rigidity. Table 4 shows that the fisher modulus of all the parameters is greater than F_{crit} (4.01). The p-value of the dependent parameters is lower than the selected p-value (0.05). From both F and p value, it is found that there is a significant difference in dependent parameters between the chemical treatments.

The values of residuals are presented in Fig. 5 which indicates the statistical hypothesis control. Figure 5 (a) shows the Henry straight line which signifies the uniform distribution of residual values. The values of

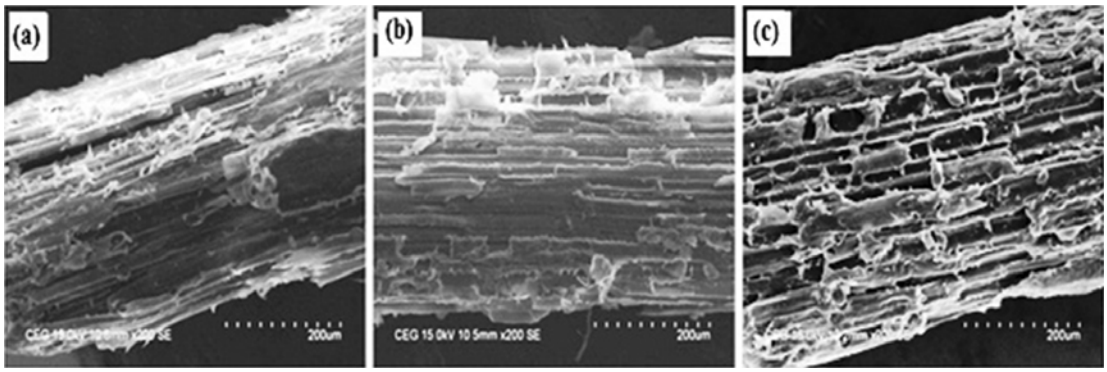

Fig. 4 — Morphology of sugarcane bagasse fibre (a) SST, (b) SAT and (c) SRS

residual against the modified ones and displayed distribution around zero randomly are given in Fig. 5 (b). Thus, the residual graphs prove that there is no conflict between hypothesis of statistics and ANOVA one factor method. Therefore, it is observed that residual is good fit to experimental data analysis.

Table 4 — Variance analysis of various properties versus different fibre treatment under confidence level of 95% for 30 samples

Source of variance	Sum of squares	Degree of freedom	Mean sum of squares	F	P	F_{crit}
Linear fibre density						
Total	6375.3	59				
Groups (between)	3737.07	1	3737.07	82.15	1.04×10^{-12}	4.01
Groups (within)	2638.22	58	45.48			
Tensile strength						
Total	871282.8	59				
Groups (between)	833319.7	1	833319.7	1273.14	3.67×10^{-41}	4.01
Groups (within)	37963.14	58	654.54			
Elongation						
Total	3519.21	59				
Groups (between)	1255.39	1	1255.39	32.16	4.71×10^{-7}	4.01
Groups (within)	2263.81	58	39.03			
Modulus of elasticity						
Total	2594.08	59				
Groups (between)	192.84	1	192.84	4.66	0.03	4.01
Groups (within)	2401.24	58	41.40			
Crystallinity index						
Total	20875.07	59				
Groups (between)	11674.01	1	11674.01	73.59	6.62×10^{-12}	4.01
Groups (within)	9200.99	58	158.64			
Flexural rigidity						
Total	5851.25	59				
Groups (between)	3603.75	1	3603.75	92.99	1.17×10^{-13}	4.01
Groups (within)	2247.5	58	38.75			
Torsional rigidity						
Total	5999.22	59				
Groups (between)	3723.54	1	3723.54	94.75	1.21×10^{-13}	4.01
Groups (within)	2275.68	58	41.25			

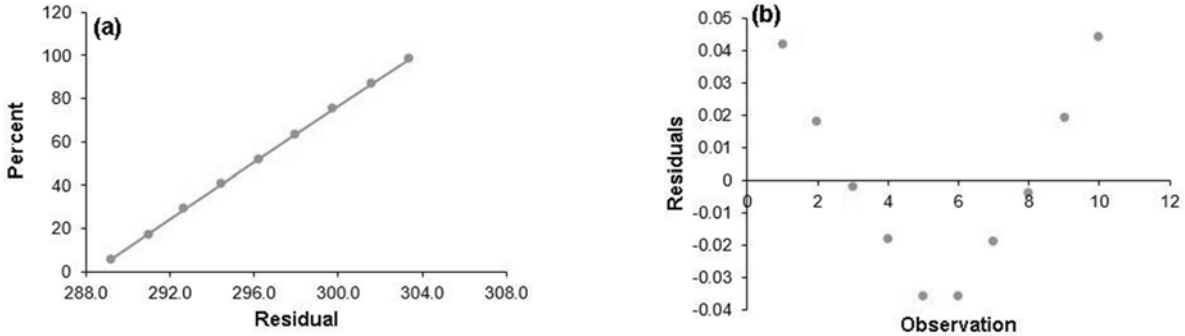

Fig. 5 — Residual plot for stress in MPa at SAT (a) Henry straight line and (b) randomly distributed residuals

4 Conclusion

The following conclusions are drawn:

4.1 The combined treatment (SAT) is more environmental friendly process, which eliminates the hemicellulose and lignin content from the fibre structure.

4.2 Both SST and SAT fibres enhance the mechanical properties and thermal stability as compared to raw sugarcane fibre.

4.3 The morphology of SAT fibre reveals that fibrils are mainly consist of thin parenchyma cells, and joined with each other.

4.4 From the statistical analysis, it is observed that the chemical treatment significantly affects the mechanical properties and thermal stability of sugarcane fibre.

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