

Wear, morphological and thermal behavior of flax fibre reinforced epoxy composites

S M Vinu Kumar^{1,a} & Harwinder Singh²

¹Department of Mechanical Engineering, Sri Krishna College of Technology, Kovaipudur 641 042, India

²Department of Textile Engineering, Panipat Institute of Engineering & Technology, Panipat 132 102, India

Received 16 June 2022; revised received and accepted 13 October 2022

Effect of different types of chemical treatments, viz. alkali (NaOH) treatment, trimethoxymethylsilane (Silane) treatment, and combination of both alkali and silane treatment on structural and thermal properties of flax fibres has been investigated by means of scanning electron microscopy (SEM), X-Ray diffraction and thermogravimetric (TGA) analysis. Besides, flax fibre reinforced epoxy composites (FFRECs) comprising 45wt.% of fibre reinforcement have been subjected to multi-pass dry sliding wear test and experimental runs are conducted as per Taguchi design of experiment technique. Crystallinity index (CI) value of 54.85 is found for alkali-cum-silane (AST) treated fibre, which is 39.23 % and 29.79 % higher than that of untreated (UT) fibre and silane treated (ST) fibre respectively. However, a very mere difference in CI values is observed between alkali treated (AT) and AST fibres. TGA study shows that UT fibre is thermally stable up to 240°C and this stability is enhanced to 310°C owing to chemical alteration as exhibited by AST fibre. Microhardness test indicates minor improvement in the hardness property of the treated FFRECs over the untreated composites. Wear test results reveal that chemically modified FFRECs offer better wear resistance than untreated FFRECs and the mechanisms endured by the composites are exposed by SEM. Amongst the prepared FFRECs, silane-treated FFREC exhibits better wear resistance property. Furthermore, the findings of analysis of variance (ANOVA) show that applied load and sliding distance contribute significantly to wear volume loss of FFRECs.

Keywords: Alkali treatment, Chemical treatment, Dry sliding wear, Flax fibre, Silane treatment, Surface treated fibres

1 Introduction

In current days, global warming has become a serious threat, which is being faced by the environment and the world community as a whole. Therefore, to curb such menace, scientists have shifted their attention to environment-friendly and recyclable materials instead of working with synthetic materials. Researchers have started working on preparing polymer composites reinforced with natural fibres as an alternative to synthetic fibre-based polymer composites, as natural fibres own exceptional properties, like biodegradable, energy-efficient, cost-effective, environment friendly and non-toxic^{1,2}. Natural fibres are further sub-categorized into plant, animal and mineral fibres. Major elements in the cell wall of the plant fibres are: cellulose, hemicellulose and lignin. Cellulose bound with both crystalline and amorphous regions, whereas hemicellulose and lignin are only limited with amorphous regions. In general, plant-based natural fibres are extensively used as a reinforcement in the automotive industry, owing to their unique lightweight and high fibre strength properties³.

In the current research work, flax fibre is used as a reinforcement material, and like all other natural fibres, flax fibres too are hydrophilic by nature, resulting in poor interfacial bonding strength. This severely affects the stress transfer between matrix and fibres, and thus mechanical properties of the composites reduce including their durability characteristics^{4,5}. In this regard, several physical and chemical surface modification methods are practised with the intention to enhance the interfacial fibre-matrix bonding of composites. Some of the widely accepted chemical modification techniques are alkalization, silane, acid treatment, peroxide, etc. These chemical modifications introduce an additional third layer which enhances the interfacial bonding between the filler and the matrix⁶.

Researchers have been continuously exploring the influence of various chemical treatments on morphological, mechanical, tribological, thermal, and interfacial bonding strength of the natural fibres and their composites. Fiore *et al.*⁷ confirmed in their investigation that, sodium bicarbonate treatment of sisal fibre had made the interfacial bonding stronger and led to improvement in mechanical properties of the sisal-

^aCorresponding author.
E-mail: vinukmr1988@gmail.com

epoxy composites. Gang⁸ revealed that chemical modification of the wood fibre by means of a coupling agent has formed a strong bond between wood fibre and polyimide. Thus, the wear performance of wood fibre reinforced polyimide composites improved. Liu *et al.*⁹ concluded in their experimental study, that the mechanical and tribological properties of the corn stack composites had improved tremendously owing to the silane treatment of corn stack fibres, as treatment strengthen fibre-matrix bonding. Wu *et al.*¹⁰ explained the improvement in the thermal degradation and tensile strength of sisal fibre as a result of tetraethylorthosilicate sol-gel and silane coupling agent treatment of sisal fibre. Rajeshkumar *et al.*¹¹ confirmed that the treatment of *Phoenix* sp. fibre with 15 % NaOH solution has improved the morphological and wear resistance properties of the fibre and its composites. Thus, these composites were suggested as friction materials for automobile applications. Komal *et al.*¹² observed, that banana fibre treated with alkali (NaOH) solution had a promising effect on the thermal and mechanical properties of its composites. Behera *et al.*¹³ showed that the combined effect of alkali-glutamic acid treatment had improved the crystallinity index of the sisal fibre. Besides, study also confirmed the improvement of wear, thermal, and microhardness properties of the sisal fibre and its composites due to the effect of chemical alteration of the fibre. Abdul Karim *et al.*¹⁴ determined that the treatment of short bamboo fibre with 5% sodium carbonate solution for 6 h had enhanced mechanical properties of bamboo-polyester composites, as the concentration of sodium carbonate was optimized, which was sufficient enough for the removal of amorphous material from the bamboo fibre surface. Samaei *et al.*¹⁵ noted a significant improvement in the morphological, thermal, mechanical, and acoustical performance of the NaOH-treated kenaf fibre compared to untreated one.

Literature survey on the treated flax fibre shows that few studies are reported in which NaOH and silane treatments of flax fibres were carried out. Studies are yet to be explored particularly on flax mats of different woven structures. Therefore, the focus of the present research work is to treat the flax fibre (irregular basket-type woven structure) with NaOH, trimethoxy-methylsilane (silane), and combination of both alkali (NaOH) and silane to enhance the interfacial bonding between the fibre and the matrix. This was ascertained from the experimental findings of the structural, and

thermal properties of untreated and treated flax fibres and wear performance of their composites.

2 Materials and Methods

2.1 Materials

The epoxy resin (grade VBR 8912) and hardener (VBR1209) were procured from Vasavibala Resin Private Limited (Chennai, Tamilnadu, India) and used as the matrix material. Flax yarns were supplied by local vendors and weaved into irregular basket woven fabric using a tabletop loom (Make: ERGO G2). Sodium hydroxide and silane used for the chemical treatment of the flax fibres, were supplied by Sigma Aldrich Pvt. Ltd.

2.2 Flax Fibre Surface Treatment

Woven flax fabric was carefully washed with distilled water for the removal of dirt and other unwanted impurities present on its surface. UT woven flax fabric was then allowed to dry at 28°C.

2.2.1 Alkali (NaOH) Treatment

The woven flax fabric was immersed in the 5 wt.% concentration of NaOH (alkali) solution at room temperature (28°C) for a duration of 60 min. In the next step, alkali treated (AT) woven flax fabric was carefully washed in a distilled water containing few drops of acetic acid to ensure the easy removal of any NaOH from the fibre surfaces. Finally, AT fabric was placed inside the oven for drying at 65°C for 24 h.

2.2.2 Silane Treatment

The woven flax fabric was dipped in the silane solution which was prepared by dissolving 0.1 % silane in an acidified distilled water. The flax fabric was immersed in this silane solution at room temperature (28°C) for 60 min. After that, silane-treated flax fabric was kept inside the oven for drying at 65°C for 24 h.

2.2.3 Combination Treatment (Alkali-cum-Silane)

In the combination treatment, flax fabric was treated with both sodium hydroxide and trimethoxy-methylsilane coupling agent. First, the flax fibres were chemically modified with NaOH solution followed by coating with silane coupling agent as described in the above section 2.2.2. Once the treatment of the fibres was done, alkali-cum-silane treated (AST) flax fabric was subjected to dry process inside the oven at 65°C for 24 h.

2.3 Composite Preparation

In this study, 45wt.% of fibre content has been considered while preparing the flax fibre reinforced epoxy composites (FFRECs), because at this weight

fraction, these composites exhibited better physico-mechanical properties than other composites as also reported in our previous study⁹. For the fabrication of FFRECs, a simple hand layup technique was used followed by the curing of laminates in compression moulding machine. Initially, lower steel mold (dimension 250 mm × 250 mm × 4 mm) was coated with silicon spray over which teflon sheet was covered simply to facilitate the easy removal of the cured laminates. At an outset, the pre-calculated quantity of epoxy resin and hardener were mixed thoroughly in the weight ratio of 1:2 and poured over the four layers of the flax fabric which had been stacked one upon the other in the lower mold. For ensuring an even distribution of matrix material over the fabric, a steel roller approximately weighing 4 kg was employed. Further to improve the epoxy flow through the flax fabric, the lower mold was closed with upper mold and the entire mold unit was subjected to compression pressure (250 psi) in a compression moulding machine for 24 h. Once the curing was done, composites were post-cured for another 12 h. Finally, prepared composite laminates were cut into appropriate sizes for various testing characterization.

2.4 Testing Methods

2.4.1 Scanning Electron Microscopy (SEM)

SEM (Japan-Model-JEOL-6480LV) test was carried out to examine the surface topography of both untreated and chemically treated flax fibres.

2.4.2 X-Ray Diffraction (XRD)

In this X-Ray diffraction test, was used to determine crystallinity of both untreated and chemically treated flax fibres. Fibres were scanned for the range (2θ) of 0°–70° at scanning speed of 7 deg/min, where θ indicates the angle of diffraction.

2.4.3 Thermogravimetric Analysis (TGA)

Thermal stability of the untreated and treated flax fibres was studied using TGA curves obtained from the TGA instrument (NETZSCH STA 2500 Regulus, German). In this process, fibre samples within a proper scale of 5-10 mg were heated at a rate of 10°C/min from room temperature (28°C) to 600°C.

2.4.4 Vickers Microhardness Test

In accordance with ASTM E384 standard, microhardness properties of the FFREC were studied using micro indentation tester at a test load of 350 g. At each loading condition, five to six indentations were made on the surface of the composite sample and average value was taken to attain the accuracy of the

microhardness value.

2.4.5 Dry Sliding Wear Test

FFRECs were subjected to sliding wear test on a Pin on disc test rig (DUCOM Instrument Pvt Ltd.) which is in accordance with ASTM G99. The composite specimens of dimension 6 mm × 6 mm × 3.5 mm were glued to the stainless-steel pin of diameter 6 mm which is mounted by means of specimen holder. The specimen was brought into contact of the rotating stainless-steel disc (EN31 grade, 62 HRC, Ra 0.25-0.30 μm) running at a specific speed. Wear tests were performed for both untreated and chemically treated FFRECs for the selected control factors as detailed in Table 1. In this test, mass of the FFREC specimens before and after the wear were recorded using digital electronic balance (accuracy of 10⁻⁴ g). By using following equation, wear volume loss (WVL) of the composites was determined:

$$WVL = \frac{\Delta M}{\rho} \quad \dots (1)$$

Where ΔM is the mass loss due to wear test (g); and ρ , the density of the composite (g/cm³).

2.4.6 Experimental Design

Using MINITAB 16 software, Taguchi design of experiment (DOE) was generated to study the major

Table 1 — Control factors and their levels

Control factors	Level1	Level 2	Level 3	Level 4
Chemical treatment (CT)	UT	AT	ST	AST
Applied normal load (AL), N	10	15	20	25
Sliding velocity (SV), m/s	1.0	1.5	2.0	2.5
Sliding distance (SD), m	600	800	1000	1200

Table 2 — DOE using L₁₆ Taguchi orthogonal array

Run	Chemical treatment	Applied normal load, N	Sliding velocity m/s	Sliding distance, m
1	UT	10	1.0	600
2	UT	15	1.5	800
3	UT	20	2.0	1000
4	UT	25	2.5	1200
5	AT	10	1.5	1000
6	AT	15	1.0	1200
7	AT	20	2.5	600
8	AT	25	2.0	800
9	ST	10	2.0	1200
10	ST	15	2.5	1000
11	ST	20	1.0	800
12	ST	25	1.5	600
13	AST	10	2.5	800
14	AST	15	2.0	600
15	AST	20	1.5	1200
16	AST	25	1.0	1000

UT-untreated, AT-alkali treated, ST-silane treated and AST- alkali-cum-silane treated.

outcome of the input-control factors on the wear response. Dry sliding wear tests were conducted on FFREC specimens in accordance with Taguchi's L_{16} orthogonal array design as shown in Table 2. The Taguchi design of experiment was mainly intended to study the effect of several operating factors on the wear performance of the FFRECs. Wear experiments were conducted as per the control factors varied at four levels as indicated in the Table 1. In this optimization technique, wear data so obtained was transformed into a signal-to-noise (S/N) ratio. Considering the fact that experiments require lesser wear rate, the smaller wear rate is the better characteristic and hence it was selected to find out the S/N ratio. It has been computed using following

equation:

$$S/N = -10 \times \log \frac{1}{x} (\sum y^2) \quad \dots (2)$$

where signal to noise ratio is indicated by S/N; and the count of observation and observed date are denoted by 'x' and 'y' respectively. Moreover, contribution of the individual factors was found out from the analysis of variance (ANOVA) study.

3 Results and Discussion

3.1 Surface Morphology of Fibres

SEM micrographs of the surfaces of untreated and chemically treated flax fibres are shown in Figs 1 (a)-

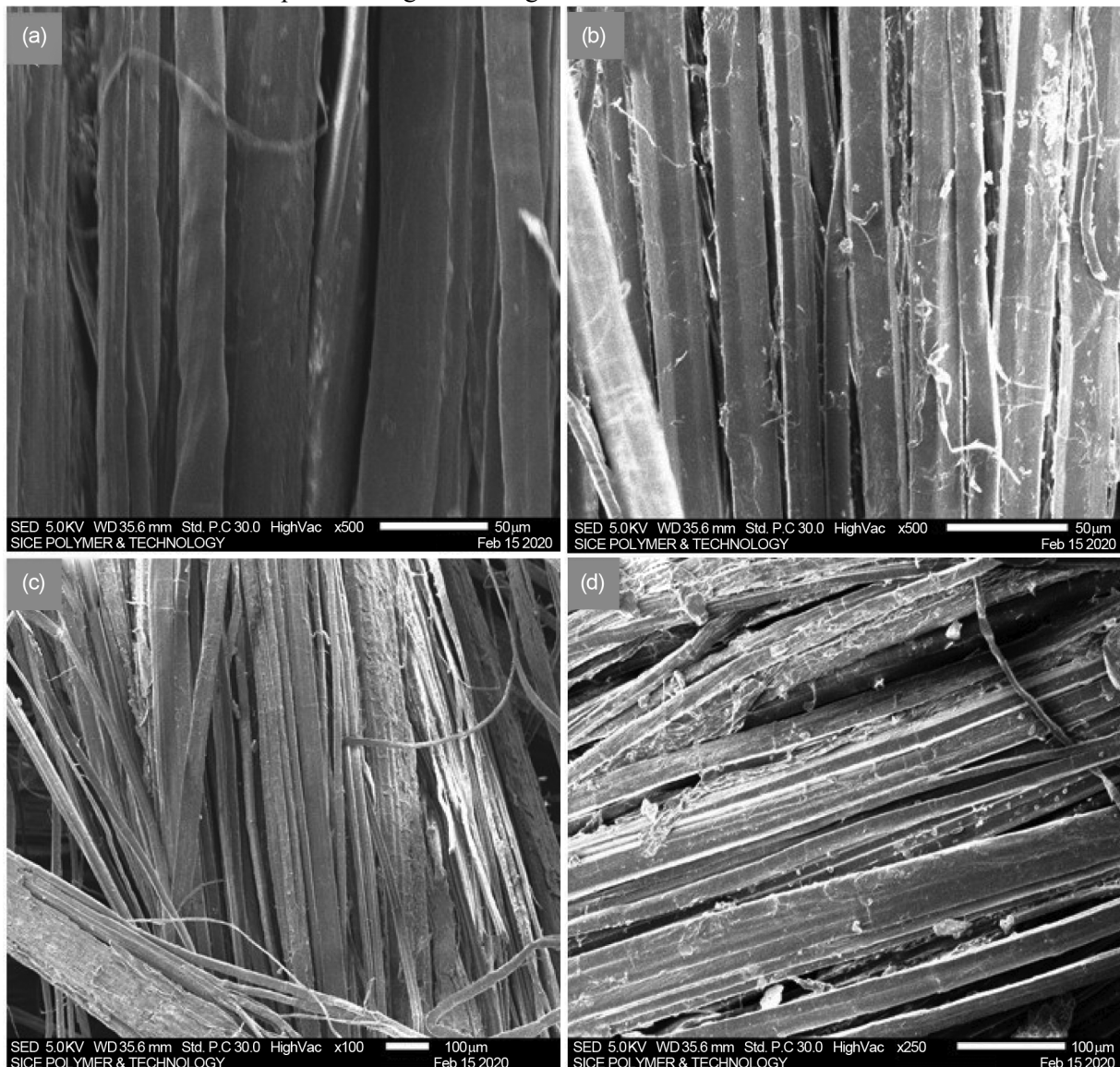


Fig. 1 —SEM images of (a) UT, (b) AT, (c) ST and (d) AST flax fibres

(d). It is clearly evident from Fig. 1(a) that flax fibres exhibit inferior interfacial adhesion strength due to the presence of lignin, wax, oil, silica, pectin, and non-cellulosic content¹⁶. UT flax fibres are quite smooth in nature due to the existence of lignin and pectin contents^{10,12}. However, lignin, wax, and other unwanted contaminants are evicted from the fibre's surface after the alkali treatment and consequently the surface roughness of the fibre increases, whereas the diameter of the fibre decreases as indicated in Fig. 1(b). Hence, this structural modification of the flax fibres may enhance the fibre-matrix bonding. From Figs 1 (c) and (d), it is evident that, ST and AST fibres exhibit few ridges on the surface of flax fibres owing to the elimination of wax and non-cellulosic contents. Besides, silane coating deposition can be observed on the fibre surface which may likely facilitate the strong interlocking of fibre and epoxy resin.

3.2 XRD Analysis

XRD analysis provides the data pertaining to the effectiveness of the chemical treatment of the fibre in terms of crystallinity and structural features. XRD diffraction patterns of the untreated and chemically treated flax fibres are depicted in Fig. 2. The crystallinity index (CI) and crystallinity percentage (%) of the raw and chemically treated flax fibres have been calculated using the following equations:

$$I_c = \frac{I_{002} - I_{am}}{I_{002}} \quad \dots (3)$$

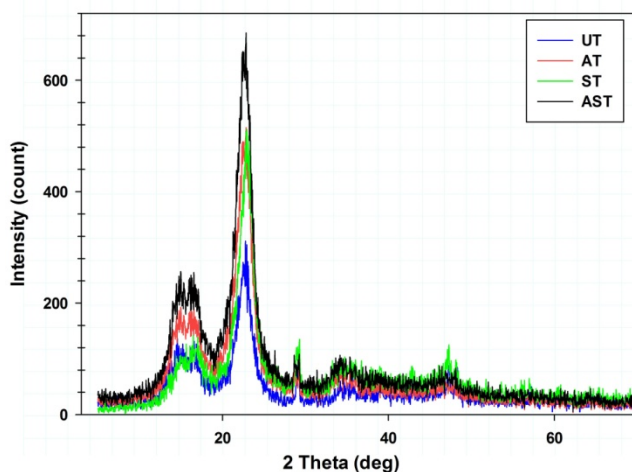


Fig. 2 — XRD patterns of UT, AT, ST and AST flax fibres

$$\text{Crystallinity \%} = \frac{I_{002}}{I_{002} + I_{am}} \times 100 \quad \dots (4)$$

where I_{002} and I_{am} stand for maximum and minimum intensity peaks at the crystalline and amorphous materials at 2θ scale respectively.

From the X-ray diffractograms, it can be seen that chemically modified flax fibres exhibit higher intensity of the crystallographic plane and amongst the processed fibres, AST fibres show maximum intensity. The crystallinity index (CI) and crystallinity percentage of the unmodified and chemically modified flax fibres are given in Table 3. It is clearly understood from Table 3 that, chemical treatment has improved CI of flax fibres and amongst all the test fibres, AST flax fibres record highest value of CI followed by AT and ST flax fibres. The main reason for such an improvement of CI in treated fibres is owing to the reduction in the amorphous elements, namely hemicellulose, lignin and wax impurities¹⁷. Kumar and Anand¹⁸ studied that chemically modified fibres are of higher degree of crystallinity that is attributed to the presence of rigidity in the cellulose. Behera *et al.*¹³ and Mohanta *et al.*¹⁹ reported similar observations during the chemical modifications of sisal and luffa cylindrica fibre respectively. Obtained CI values for the various chemically treated flax fibres are compared with the other raw and treated natural fibres, as indicated in Table 4.

3.3 Thermal Behavior of Treated and Untreated Flax Fibre

The chemical constituents of natural fibres decide the thermal degradation behavior of their composites. Thermal degradation rate of fibre constituents (lignin, cellulose, hemicellulose, wax, and other impurities) could be very well understood by performing TGA study¹⁹. From the TGA curve (Fig. 3), it is clearly evident that, early stage of degradation with a weight loss of 3.5-7.3 % is observed below 100°C, which may be attributed to the loss of moisture absorbed in the fibre. In the subsequent stage, at the temperature range (210-265°C) breakdown of hemicellulose and

Table 3 — XRD data of untreated and chemically treated flax fibres

Fibres	Crystallinity index (CI)	Crystallinity, %
UT	39.384	67.75
AT	54.521	68.73
ST	42.861	67.93
AST	54.852	69.05

Table 4 — Comparison of thermal and crystallinity index characteristics of chemically treated flax fibres with other natural fibres

Fibre	Raw/chemically treated	Thermal stability °C	Maximum thermal degradation, °C	Crystallinity index (CI)	Reference
Flax	Raw	245	375	39.38	Present Work
	5 wt.% NaOH	285	395	54.52	
	Silane treated	280	390	42.86	
	Alkali-cum-silane treated	310	410	54.85	
Sisal	Raw	225	371	50.41	5
	5 wt.% NaOH	235	376	63.14	
<i>Ficus religiosa</i> root	Raw	235	325	42.92	20
	5 wt.% NaOH	245	356	48.64	
<i>Acacia planifrons</i>	Raw	250	350	65.38	21
	5 wt.% NaOH	265	385	74.76	
<i>Thespesia populnea</i> bark	Raw	245.4	323.76	48.17	22
	-	-	-	-	
Cotton shell	Raw	-	317	48.96	23
	5 wt.% NaOH	-	332	63	
Banyan tree root	Raw	230	358	72.47	24
	5 wt.% NaOH	230	368	76.35	
<i>Prosopis juliflora</i> bark	Raw	217	331	46	25
	-	-	-	-	
Maize tassel	Raw	220	350	85.48	26
	5 wt.% NaOH	230	367	88.41	
<i>Tridax procumbens</i>	Raw	195	250	34.6	27
	5 wt.% NaOH	223	280	40.85	
<i>Kigelia africana</i>	Raw	300	440	59	28
	5 wt.% NaOH	300	425	60	
<i>Calotropis gigantea</i> fruit	Raw	265	280	36	29
	5 wt.% NaOH	282	317	39.8	
<i>Coccinia grandis</i>	Raw	213	351.6	52.17	30
	5 wt.% NaOH	220.6	360.1	57.64	

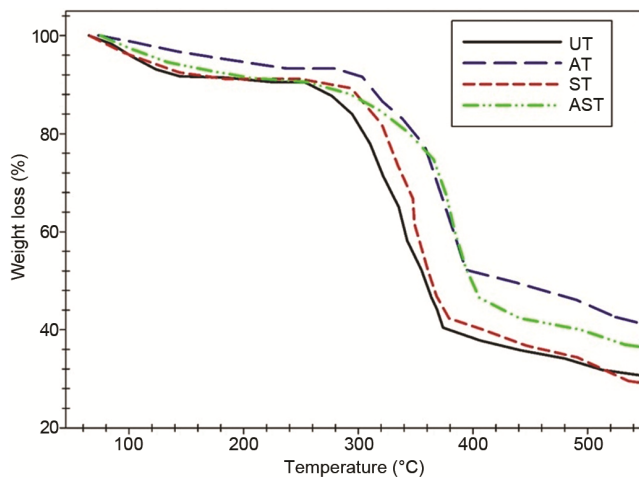


Fig. 3 — TGA curves of UT, AT, ST and AST flax fibres

glycosidic linkage of cellulose takes place^{13,19}. From the TGA curve, it is interesting to note that the highest thermal degradation is suffered by the untreated fibres

above 380°C, but this value is shifted to 405°C, 390°C, and 430°C for alkali, silane, and alkali-cum-silane treated flax fibres respectively. From this analysis, it can be deduced that chemical alteration of fibres has induced a positive effect. Mainly thermal stability of the fibres has enhanced due to the expulsion of the contaminations from the fibre surface³¹. Thermal stability and maximum thermal degradation of the various chemically treated flax fibres in comparison with the other raw and treated natural fibres are presented in Table 4.

3.4 Microhardness Test

Alkali treated (AT) FFREC exhibits the highest hardness value of 19.21 HV followed by AST-FFREC, ST-FFREC and UT-FFREC with a hardness value of 18.88 HV, 16.14 HV, and 14.88 HV respectively. This slight improvement in the hardness property of the treated FFREC may be attributed to the wettability

characteristics prevailing between chemically modified flax fibre and epoxy materials³².

3.5 Dry Sliding Wear Properties of FFREC using Taguchi Optimization Technique

In this research work, Taguchi optimization method has been adopted mainly to analyse the experimental data pertaining to the wear properties of FFREC. Signal to Noise (S/N) ratio plays a pivotal role to understand the impact of the control factors on the output response (wear loss). For minimizing the wear of FFREC it has been set to ‘smaller-is-better’ characteristics. The control factor having minimum S/N ratio value indicates highest wear volume loss (WVL) and vice-versa. Computed value of S/N ratio of WVL at various combinations of input factors has been portrayed in Table 5. Maximum WVL of 26.945 mm³ and minimum WVL of 2.257 mm³ are exhibited by UT FFREC for the experimental runs four and one respectively and its combinations of the input factors which are responsible for the WVL are given in Table 1. The S/N ratio-response of FFREC is shown in Table 6 and main effect for S/N ratio with respect to the control factors is presented in Fig. 4. The silane-treated FFREC shows maximum wear resistance when the samples are operated at the sliding speed of 1 m/s under the influence of 10 N of applied load to cover sliding distance of 600 m (Fig 5). Table 6 conveys the influencing factors affecting the wear performance of the FFREC in terms of the delta value, and its higher value means, the higher

contribution of the factor. Applied load with the value of delta 12.66 is the most significant factor contributing to the wear loss of the FFREC followed by sliding distance (4.38), sliding velocity (3.91) and chemical treatment (2.59) respectively.

Figure 4 clearly illustrates that chemically treated FFREC possesses a higher mean of S/N ratio than UT FFREC, which clearly implies that UT FFREC exhibits higher wear volume loss (WVL) than treated FFREC. The lowest WVL is shown by ST FFREC followed by AST FFREC. Interestingly ST FFREC displayed intermediate WVL.

Table 5 — Wear volume loss (WVL) and its corresponding S/N ratio

Run	WVL, mm ³	S/N ratio
1	2.257	-7.0706
2	8.751	-18.8412
3	17.882	-25.0483
4	26.945	-28.6096
5	3.189	-10.0731
6	11.345	-21.0961
7	12.444	-21.8992
8	15.555	-23.8374
9	3.687	-11.3335
10	6.489	-16.2463
11	8.865	-18.9536
12	13.634	-22.6925
13	7.856	-17.6003
14	4.256	-12.5800
15	10.889	-20.7398
16	12.114	-21.5836

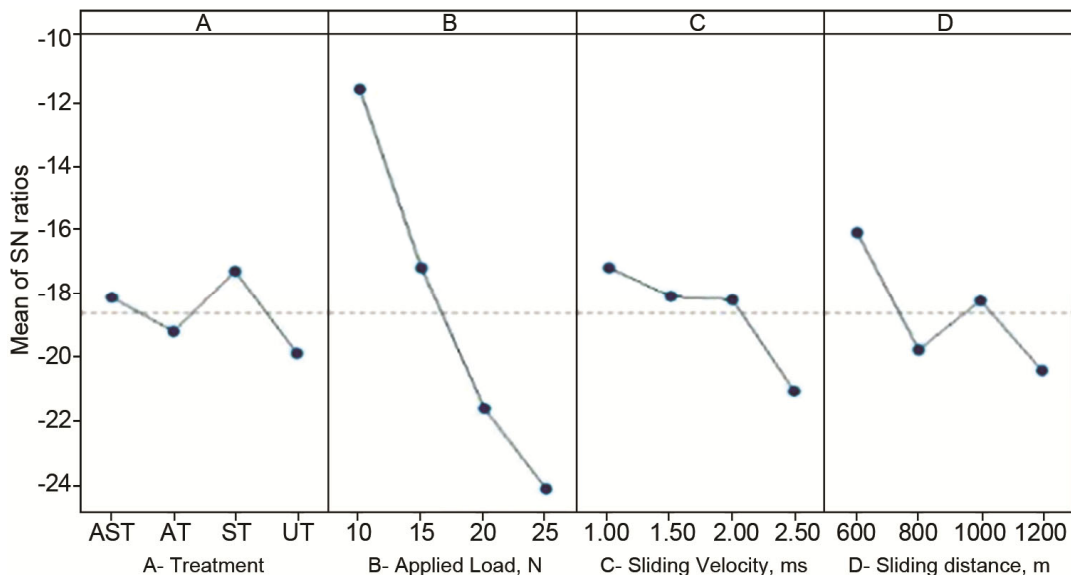


Fig. 4 — Variation of S/N ratio of WVL of FFREC with respect to the controllable factors

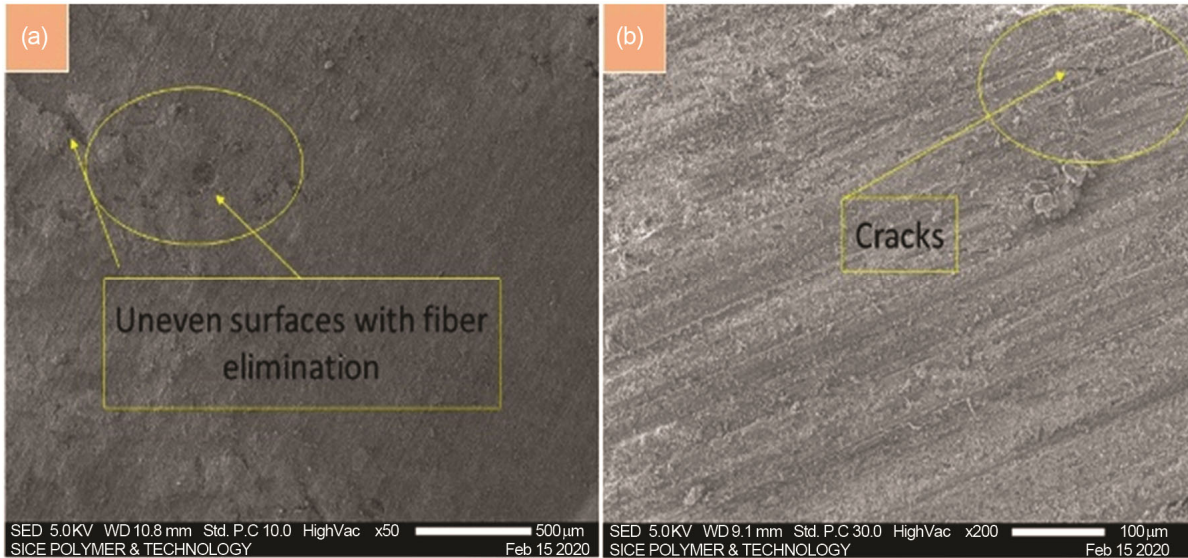


Fig. 5 — Worn-out SEM images of (a) UT and (b) ST FFREC

Table 6 — Response table for S/N ratio

Level	Chemical treatment (CT)	Applied load (AL)	Sliding velocity (SV)	Sliding distance (SD)
1	-18.13	-11.52	-17.18	-16.06
2	-19.13	-17.19	-18.09	-19.18
3	-17.31	-21.66	-18.20	-18.24
4	-19.81	-24.18	-21.09	-20.44
Delta	2.59	12.66	3.91	4.38
Rank	4	1	3	2

Table 7 — Analysis of variance for S/N ratios

Source	DF	Adj. SS	Adj. MS	F	P
Chemical treatment (CT)	3	15.83	5.276	0.31	0.820
Applied load (AL)	3	370.51	123.503	7.22	0.069
Sliding velocity (SV)	3	34.55	11.516	0.67	0.623
Sliding distance (SD)	3	45.75	15.249	0.89	0.536
Residual error	3	51.30	17.102		
Total	15	517.94			

Elimination of wax and other impurities as a consequence of alkali and alkali-silane treatment increases the fibrillation process causing the fibre surface to get rougher and so the contact area between the fibre surface and matrix material increases. As a result, their interfacial bonding strength gets enhanced. Hence, chemically modified FFRECs are found to be superior in wear resistance than untreated FFREC. This may be the reason for the minimum WVl of the treated FFREC than that of UT FFREC²². Furthermore, Fig. 4 envisages that the S/N ratio

decreases with an increase in the applied load, sliding velocity and sliding distance, which consequently leads to an increase in the WVl. This can be ascribed to an increase in temperature of the sliding interface with the increase in the aforementioned factors. As a consequence, thermal softening of the matrix-fibre reinforcement takes place which may be accountable for the poor wear resistance of FFREC^{13,23}.

3.5.1 Analysis of Variance and Effect of Control Factors

MINITAB 16 has been employed for ANOVA investigation to understand the influence of control factors on WVl. ANOVA results are tabulated in Table 7. The effect of each input control factor on WVl is given by P value. The lesser the P-value, the higher is the effect of the control factors on the WVl.

From Table 7, it can be confirmed that the applied normal load (P=0.069) and sliding distance (P=0.536) contribute the highest significant effect on wear performance (WVl) of the FFREC. Factors like sliding velocity and chemical treatment have least effect on the WVl.

3.6 Worn Morphology of FFREC

Figure 5 shows the worn-out SEM images of the UT and ST FFREC. With increase in the sliding velocity, the wear rate of FFREC is increased and results in uneven surfaces, as observed in the Fig. 5(a). The formation of uneven surface may be due to the severe damage of the composite layers¹³. UT FFREC experiences higher wear rate with

increase in the sliding velocities owing to the detachment of flax fibre as a result of poor interfacial bonding between fibre and matrix. However, interfacial bonding is enhanced by chemical modification of fibre and hence treated FFREC shows comparatively better wear resistance as compared to untreated FFREC. When FFREC is subjected to dry sliding wear at higher load and sliding velocity, temperature at the sliding interface is increased, resulting in higher wear rate¹⁶. Due to this process, thermal softening or crack occurs at the fibre-matrix³⁴ which could be the main reason for the poor wear behaviour of FFREC, as indicated in Fig. 5(b).

4 Conclusion

This experimental study investigates the effect of various chemical modifications on the morphological, thermal, microhardness and tribological behaviour of flax fibre and FFRECs. SEM pictures clearly expose the removal of waxy and other contaminants from the fibre surface through chemical treatments. NaOH treated fibre exhibits the best results. Chemical-treated flax fibres exhibit better thermal stability than UT flax fibres. The highest rate of thermal degradation is experienced by AST fibre followed by AT, ST and UT flax fibres. Chemical modification also improves crystallinity index of flax fibres. Amongst the chemically-altered fibres, AST fibre exhibits the highest crystallinity index. The highest value of microhardness shown by AT FFREC followed by AST FFREC, ST FFREC and UT FFREC. The best optimal combination of control factors that offer maximum wear resistance are: ST FFREC, applied load of 10 N, sliding velocity of 1.0 m/s, and sliding distance of 600 m. Tribological wear test results based on Taguchi DOE show that the wear resistance properties of the FFREC have improved owing to the chemical modification of the flax fibres. The FFREC wear performance follows the order:

ST FFREC > AST FFREC > AT FFREC > UT FFREC.

ANOVA results convey that applied load and sliding distance are the most significant factors affecting WVL. However, influences of sliding velocity and chemical treatment on WVL are minor. The prime reason for micro cracks and matrix erosion of the FFREC is attributed to thermal softening as exposed by SEM.

References

- Nayak S & Mohanty J, *Compos Commu*, 18 (2020) 19. doi:10.1016/j.coco.2020.01.006.
- Shettahalli Mantaiah V K, *J Nat Fibers*, 19 (2022) 15415. doi: 10.1080/15440478.2022.2060404.
- Nirmal U, Hashim J & Ahmad M M, *Tribol Int*, 83 (2015) 77. doi:10.1016/j.triboint.2014.11.003.
- Fiore V, Di Bella G & Valenza A, *Composites [Part B]: Eng*, 68 (2015)14. doi: 10.1016/j.compositesb.2014.08.025.
- Singh H & Chatterjee A, *Indian J Fibre Text Res*, 46 (2021) 275.
- Behera S, Gautam R K, Mohan S & Chattopadhyay A, *J Nat Fibers*, 19 (2021) 6134. doi: 10.1080/15440478.2021.1904483.
- Abdul Karim M R, Tahir D, Hussain A, Ul Haq E & Khan K I, *Plast, Rubber Compos*, 49 10 (2020)425. doi: 10.1080/14658011.2020.1768336.
- Samaci S E, Mahabadi H A, Mousavi S M, Khavanin A, Faridan M & Taban E, *J Ind Text*, 51 (2020) 8601. doi: 10.1177/1528083720944240.
- Kumar S V, Kumar K S, Jailani H S & Rajamurugan G, *Mater Res Express*, 7 (2020)085302. doi: 10.1088/2053-1591/abaea5.
- Vardhini K J, Murugan R & Rathinamoorthy R, *Indian J Fibre Text Res*, 44 (4) (2019) 459.
- Rajeshkumar G, Hariharan V & Scalici T, *J Natures Fibre*, 13 (6) (2016) 702. doi.org/10.1080/15440478.2015.1130005.
- Mohanta N & Acharya S, *J Compo Mater*, 50 (22) (2016)3117. doi: 10.1177/0021998315615654.
- Oza S, Ning H, Ferguson I & Lu N, *Compos [Part B]: Eng*, 67 (2014) 227. doi: 10.1016/j.compositesb.2014.06.033.
- Abdul Karim M R, Tahir D, Hussain A, Ul Haq E & Khan KI, *Plast*, 49 (10)(2020) 425. doi. org 10.1080/14658011.2020.1768336.
- Rashid B, Leman Z, Jawaid M, Ghazali M, Ishak M & Abdelgnei M, *Wear*, 380 (2017)26. doi: 10.1016/j.wear.2017.03.011.
- Kumar S, Patel V, Mer K, Gangil B, Singh T & Fekete T, *J Nat Fibers*, 16 (4)(2020) 762.
- Vardhini K, Murugan R & Rathinamoorthy R, *Indian J Fibre Text Res*, 44 (4) (2019)459.
- Kumar R & Anand A, *Mater Res Express*, 6(1) (2019) Article ID 015309. doi: 10.1088/2053-1591/aae69b.
- Mohanta N & Acharya S, *J Composite Mater*, 50 (22) (2016)3117. doi: 10.1177/0021998315615654.
- Arul Marcel Moshi, Ravindran D, SundaraBharathi S R, Indran S & Suganya Priyadarshini G, *Int J Biol Macromol*, 142(2020) 212. doi: 10.10 2019.01.02416/j.ijbiomac.2019.09.094.
- Senthamaraiakannan P, Saravanakumar S S, Sanjay M R, Mohammad M & Siengchin S, *Mate Lett*, 240(2019)221. doi.org/10.1016/j.matlet.2019.01.024.
- Kathirselvam M, Kumaravel A, Arthanarieswaran V P & Saravanakumar S S, *Carbohydr Polym*, 217 (2019) 178. doi.org/10.1016/j.carbpol.2019.04.063.
- Rajkumar R, Manikandan A & Saravanakumar S S, *Int J Polym Anal Ch*, 21 (2016) 359. doi.org/10.1080/1023666 X.2016.1160509.

- 24 Ganapathy T, Sathiskumar R, Senthamarai kanna n P, Saravanakumar S S & Khan A, *J Biol Macromol*, 138 (2019) 573. doi.org/10.1016/j.ijbiomac.2019.07.136.
- 25 Saravanakumar S S, Kumaravel A, Nagarajan T, Sudhakar P & Baskaran R, *Carbohydr Polym*, 92(2013) 1928. doi.org/10.1016/j.carbpol.2012.11.064.
- 26 Maepa C, Jayaramudu J, Okonkwo J, Ray S S, Sadiku E & Ramontja J, *Int J Polym Anal Ch*, 20(2) (2015)99. doi: 10.1080/1023666X.2014.961118.
- 27 Vijay R, Lenin Singaravelu D, Vinod A, Sanjay M R, Siengchin S, Jawaid M, Khan A & Parameswaranpillai J, *Int J BiolMacromol*, 125 (2019) 99. doi.org/10.1016/j.ijbiomac.2018.12.056.
- 28 Ilangovan M, Guna V, Prajwal B, Jiang Q & Reddy N, *Carbohydr Polym*, 236 (2020)Article ID 115996. doi.org/10.1016/j.carbpol.2020.115996.
- 29 Narayanasamy P, Balasundar P, Senthil S, Sanjay M R, Siengchin S, Khan A & Asiri A M, *Int J Biol Macromol*, 150(2020) 793. doi.org/10.1016/j.ijbiomac.2020.02.134.
- 30 Senthamarai kanna n P & Kathiresan M, *Carbohydr Polym*, 186 (2018) 332. doi:10.1016/j.carbpol.2018.01.072.
- 31 Oza S, Ning H, Ferguson I & Lu N, *Compos [Part B]: Eng*, 67 (2014) 227. doi: 10.1016/j.compositesb.2014.06.033.
- 32 Webo W, Masu L & Maringa M, *Adv Mater Res-Switz*, 12 (2018)48.
- 33 Rashid B, Leman Z, Jawaid M, Ghazali M, Ishak M& Abdelgnei M, *Wear*, 380 (2017) 26. doi: 10.1016/j.wear.2017.03.011.
- 34 Kumar S, Patel V, Mer K, Gangil B, Singh T & Fekete G, *J Nat Fibers*, 18 (2) (2019) 192. doi: 10.1080/15440478.2019.1612814.