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Original Article

Effect of surface treatment and fiber loading on the physical, mechanical, sliding wear, and morphological characteristics of tasar silk fiber waste-epoxy composites for multifaceted biomedical and engineering applications: fabrication and characterizations



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ABSTRACT

In the present study, waste of tasar silk fiber was used as reinforcement in the epoxy resin. Tasar silk fiber waste (TSFW) was pre-treated with NaOH coupling agent before reinforcing it with epoxy matrix. Treated and untreated TSFW-epoxy composites were made using the compression moulding technique. Composites were characterized for physical, mechanical and wear properties. Effect of NaOH coupling agent and fiber loading were examined on TSFW-epoxy composite. Comparatively, lower void fraction of 5.1% was obtained at 30% reinforcement of treated TSFW composite as compared to 5.8% as obtained for untreated TSFW composites. The tensile strength, flexural strength and impact strength were observed to be 68.47 MPa, 41.18 MPa, and 2.2 J for untreated TSFW where as these

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Physical
Mechanical
Wear properties
Water-absorption
SEM

properties exhibits marginal increment of 70.86 MPa, 43.52 MPa and 2.35 J respectively. Hardness of composite experienced enhancement of 6% upon using treated TSWF. Overall, 10% reduction in specific wear was observed upon using treated TSWF as compared to untreated one. Scanning electron microscope (SEM) analysis suggested that the tensile specimen undergoes ductile fracture while flexural specimen failed in brittle manner. Fiber agglomeration and large deformation of tasar silk resulted in improved strength as observed in SEM analysis.

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1. Introduction

Growing concern of carbon emission and accumulation of non-renewable waste material due to synthetic fibers have shifted the paradigm of composite materials towards biodegradable natural fibers [1]. In comparison to synthetic fibers, natural fibers are cheap, easy to process, found in abundance, attractive thermal properties, high resistive to corrosion, and have good mechanical properties [2]. Commonly known epoxy polymer is extensively used in the fabrication of composite material owing to several merits such as high moisture resistance, good thermal resistance, good quality of adhesion, and outstanding mechanical properties [3,4]. However, epoxy's low fracture toughness and high brittleness restrict its applications in real situations. Besides, blending of rubber and reinforcement of fiber with epoxy can improve the toughness of composites [5]. Moreover, several synthetic fibers have been utilized in the fabrication of composites based on the epoxy resin which showed noteworthy improvement in the mechanical and wear properties of epoxy. Somehow, this practice of reinforcement of synthetic fiber in an epoxy matrix is condemned due to numerous issues such as expensive, the hazardous effect due to the presence of polymeric content, high energy requirement in processing, low wear resistance and non-renewable [6]. This has led to the urgent replacement of synthetic fiber with natural fiber. Hemp, jute, sisal, kenaf, oil palm fiber, are some of the natural fibers which were combined with epoxy to fabricate composites. In addition, silk fiber is now becoming very popular as a reinforcing agent in the epoxy resin to achieve the desired toughness and strength [7]. The volume fraction of fiber in the composites is one of the important aspects of composites strength, which depends on both fiber and matrix physical characteristics and physical bonding [8]. Moreover, treatment of natural fiber has become very common to enhance the bonding strength of fiber-matrix interface. To do so, various types of chemical and physical treatment have been performed on the fiber [9]. Typically known chemical treatment as silane treatment employed in carbon fiber resulted in enhanced bonding between fiber and matrix leading to improved mechanical properties [10]. Bagasse fiber treated with NaOH yielded 12% higher strength as compared to untreated one in polyester based composites [11]. Cordia-dichotoma, when treated with NaOH solution gives improved impact strength and thermal properties in epoxy based composites [12]. Recently, the advantage of NaOH treated stem fiber polymer composite was demonstrated for

enhanced mechanical and wear properties [13]. The concentration of chemical solution used for treatment of fiber must be kept under certain limit and up to a certain period of time otherwise, the cellulosic bond or protein bond among the fibers gets weakened [14].

Silk fiber is mainly composed of protein with the longest fiber's length among all the natural fiber. The silk fiber is generally classified as (a) cultivated (*Bombyx mori*) and (b) wild silk (Tasar, Eri, and Muga). In the past two decades, bombyx mori silk has received greater attention as a reinforcement in composite materials due to its excellent mechanical strength, stiffness, and modulus [15]. Reinforcing mat of silk fiber in poly-ε-caprolactone (a biodegradable polymer) displayed remarkable improvement of 45% and 35% respectively in bending and tensile strength of polymer [16]. Short silk fiber-reinforced polylactic acid composite showed improved flexural and young's modulus by 5% and 25%, respectively [17]. Bonding force, breaking energy, and tensile strength of polyurethane reinforced silkworm cocoon composite were enhanced by 500%, 300%, and 150%, respectively, compared to virgin matrix [18]. The effect of silk fiber loading in the polybutylene succinate composite was investigated and it was reported that mechanical properties of the composite were enhanced remarkably till 60% of silk fiber loading without even chemical treatment of silk fiber [19]. At 20 wt. % silk fiber loading in a gelatin matrix, the mechanical properties such as modulus, tensile strength, impact strength, and bending strength of gelatin boosted by approximately 400%, 275%, 445%, and 250%, respectively [20]. The inclusion of woven silk fiber in epoxy matrix resulted in a significant enhancement in bending and tensile strength as compared to virgin epoxy [21]. Woven mat of silk fiber reinforced epoxy exhibited improvement in impact properties, ductility and breaking energy of composite at 60 wt. % fiber loading [22]. Recently, hybrid composite of TSWF -jute-epoxy was investigated and reported for improved mechanical and wear characteristics due to the inclusion of silk fiber in jute-epoxy composite [23]. Silk fiber was also reinforced with various polymers like polyester, polyolefin, polystyrene that showed encouraging results for mechanical strength but the issue of hydrophilicity of silk fiber has somehow restricted the higher loading of silk fiber in polymer composite applications [24–27]. The issue of hydrophilicity of silk fiber can be resolved by the surface treatment of silk fiber with NaOH solvent. The NaOH removes the hydroxyl group present on the surface of fiber and enhances the bonding

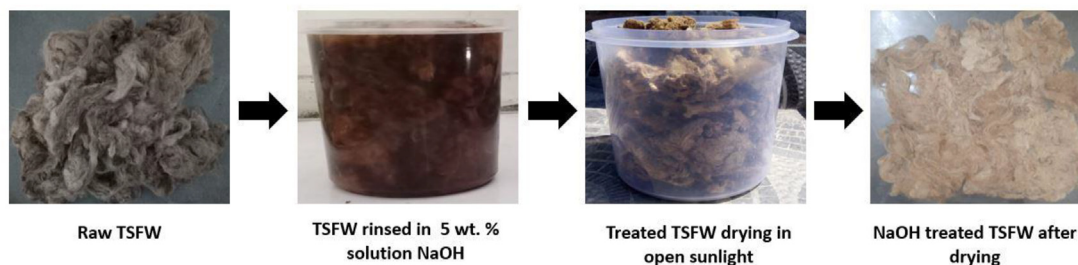


Fig. 1 – NaOH treatment of TSWF.

characteristics of fiber and matrix [28]. The outermost layer of tassar silk fiber contains sericin which holds together two main fibroin threads and is used extensively in biomedical applications. This layer of sericin, however, has a detrimental effect on the application of the composite material as the interfacial bonding between the fiber and polymer gets weakened if the sericin is not removed from the surface.

The literature revealed that most of the research in silk fiber composite revolves around mulberry silk, which is considered the strongest silk among all the known silk. Being protein, silk has been reinforced in polymer for the improvement in the mechanical strength of the composite [29]. Tassar silk consists of proteins namely: (1) fibroin, a compound of nonpolar glycine and highly hydrophobic and (2) compounds of serine, alanine, and amino acids, which are highly hydrophilic. Recently, it was reported that tassar silk has more strength than mulberry silk. The high strength of tassar silk is attributed to the structure of amino acids forming a long chain of hydrogen bonds [30].

Asia accounts for almost 80% of the total silk produced in the world. India is the second-largest producer of raw silk with tassar silk fiber having the highest share of production [31]. Tassar silk fiber is a unique variety of silk produced from the silk worm known as *Antheraea mylitta*. It is majorly used as a textile material as almost all the silk sarees manufactured in India are from tassar silk. The waste accumulated after the processing of tassar silk saree is being utilized for the making of house hold commodities such as hand bags, shawls and handicrafts [32]. Waste of tassar silk fiber has almost equal strength as the raw tassar silk contains but lacks shining and lustrous which is why discarded for further processing. The importance of TSWF as a reinforcement in the sphere of

composite is yet to be disclosed [33]. Epoxy is one of the most employed matrix materials for the fabrication of composites. It is cheap and easily available in resin form at room temperature (RT). Literature suggested that natural fibers are extensively reinforced with epoxy due to their capability of making a strong bond with fibers. Its rapid cooling makes it eligible to fabricate a wide range of composite products. It endures sufficient strength and can also be tailored for high load bearing composite [34,35]. Therefore, the present investigation focussed on the effect of TSWF on the physio mechanical and wear characteristics of epoxy. The TSWF was treated with NaOH solvent and the effect of the treatment was also investigated in the present study.

2. Experimentation

2.1. Materials

Waste of tassar silk fiber (TSFW) of density 1.3 g/cm³ was purchased from a local vendor of Bhuvneshwar, Orissa, India at a cost of \$ 4/kg. Epoxy resin of grade LY 556 and appropriate hardener of HY 951 grade was selected as matrix material of density 1.25 g/cm³ [36] which was purchased from AMTEC ester Pvt. Ltd. Delhi, India. Sodium hydroxide (NaOH) was provided by the department of chemistry, NIT Uttarakhand, India.

2.2. Treatment with NaOH

NaOH solution was prepared by adding 5 g of NaOH powder in 100 g of water to prepare 5 wt. % of NaOH solution [37]. TSFW

Table 1 – Sample composition.

| Abbreviation | Compositions |
|--------------|--|
| TSW-5 | 5 wt. % untreated TSWF-epoxy composite |
| TSW-10 | 10 wt. % untreated TSWF-epoxy composite |
| TSW-15 | 15 wt. % untreated TSWF-epoxy composite |
| TSW-20 | 20 wt. % untreated TSWF-epoxy composite |
| TSW-25 | 25 wt. % untreated TSWF-epoxy composite |
| TSW-30 | 30 wt. % untreated TSWF-epoxy composite |
| N-TSW-5 | 5 wt. % NaOH treated TSWF-epoxy composite |
| N-TSW-10 | 10 wt. % NaOH treated TSWF-epoxy composite |
| N-TSW-15 | 15 wt. % NaOH treated TSWF-epoxy composite |
| N-TSW-20 | 20 wt. % NaOH treated TSWF-epoxy composite |
| N-TSW-25 | 25 wt. % NaOH treated TSWF-epoxy composite |
| N-TSW-30 | 30 wt. % NaOH treated TSWF-epoxy composite |

Table 2 – Density of untreated and NaOH treated TSWF composites.

| Sample | Theoretical density (g/cm ³) | Experimental density (g/cm ³) | Voids (%) |
|-----------|--|---|-----------|
| TSFW-5 | 1.2523 | 1.2298 | 1.8 |
| TSFW-10 | 1.2549 | 1.2224 | 2.6 |
| TSFW-15 | 1.2572 | 1.2142 | 3.4 |
| TSFW-20 | 1.2598 | 1.2084 | 4.1 |
| TSFW-25 | 1.2621 | 1.2006 | 4.9 |
| TSFW-30 | 1.2645 | 1.1911 | 5.8 |
| N-TSFW-5 | 1.2523 | 1.2329 | 1.5 |
| N-TSFW-10 | 1.2549 | 1.2266 | 2.3 |
| N-TSFW-15 | 1.2572 | 1.2189 | 3.0 |
| N-TSFW-20 | 1.2598 | 1.2127 | 3.7 |
| N-TSFW-25 | 1.2621 | 1.2068 | 4.4 |
| N-TSFW-30 | 1.2645 | 1.2004 | 5.1 |

was soaked in the NaOH solution and left for 24 h. Fibers were then taken out from the solution and washed in distilled water. The rinsed fibers were dried in open sunlight to remove the water content present in the fiber. The fiber treatment with NaOH is shown in Fig. 1.

2.3. Composites fabrication

Fabrication started with the mixing of epoxy and hardener in the ratio of 10:1 and stirred for 15 min. TSWF was organized and pressed in a compression moulding machine to form a non-woven mat. Mould of steel of size 300 × 300 × 4 mm was used for the fabrication of composite samples. Prepared mixture of epoxy and hardener was poured in the mould followed by placing of non-woven mat of TSWF. The mould was then subjected to cold compression at a pressure of 1 MPa and left for curing at RT for 24 h. For each composition, two samples were fabricated. Samples were finally allowed to cure in the mould for 1 day and then taken out from the mould to prepare test specimens. The composition of fiber and matrix for treated and untreated TSWF composites are illustrated in Table 1.

2.4. Physical testing

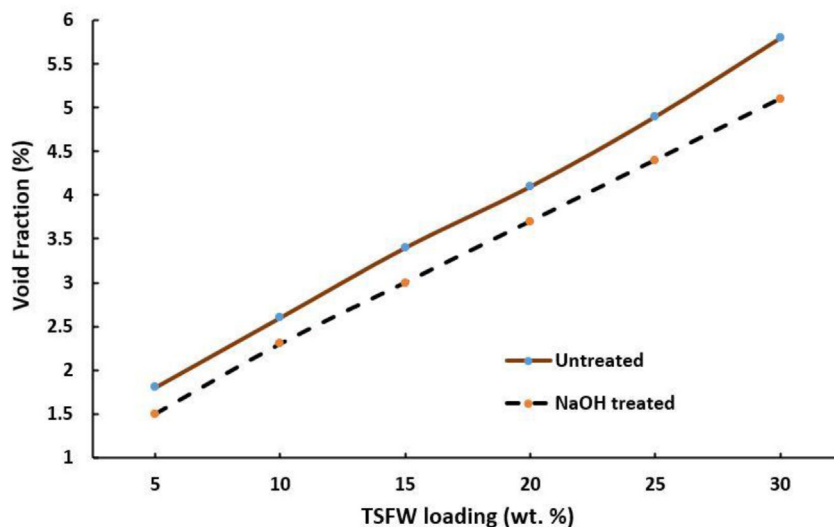
Each test was repeated three times and average of them was reported in the manuscript. Theoretical and experimental densities of the composite were evaluated by Eq. (1) and Archimedes principle of buoyancy respectively. The percentage of void fraction was calculated as per: Eq. 2

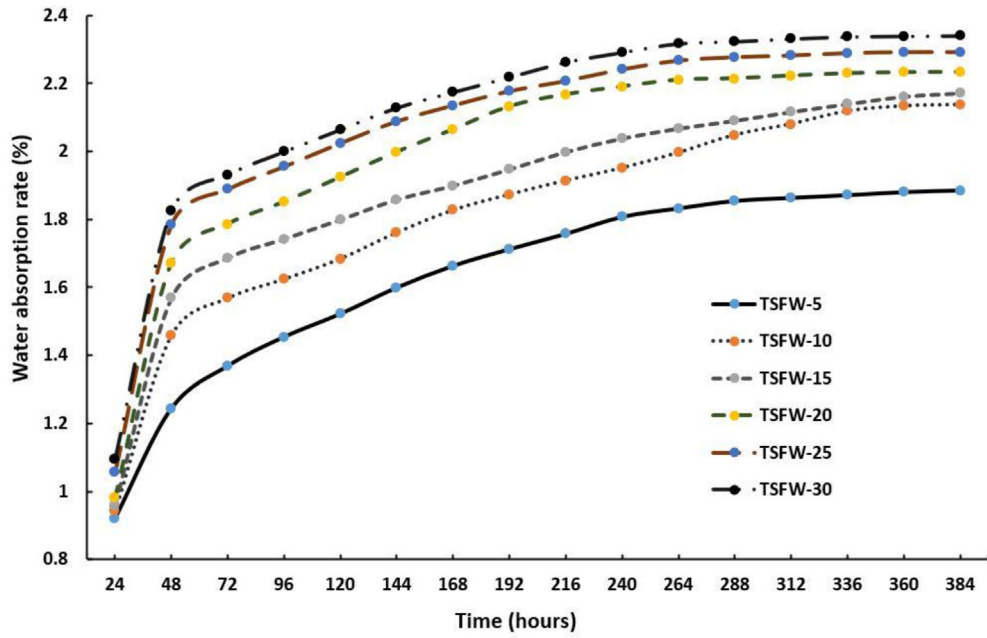
$$\rho_{th} = \frac{1}{\frac{W_m}{\rho_m} + \frac{W_{tsw}}{\rho_{tsw}}} \quad (1)$$

$$V_f = \frac{\rho_{th} - \rho_{exp}}{\rho_{th}} \quad (2)$$

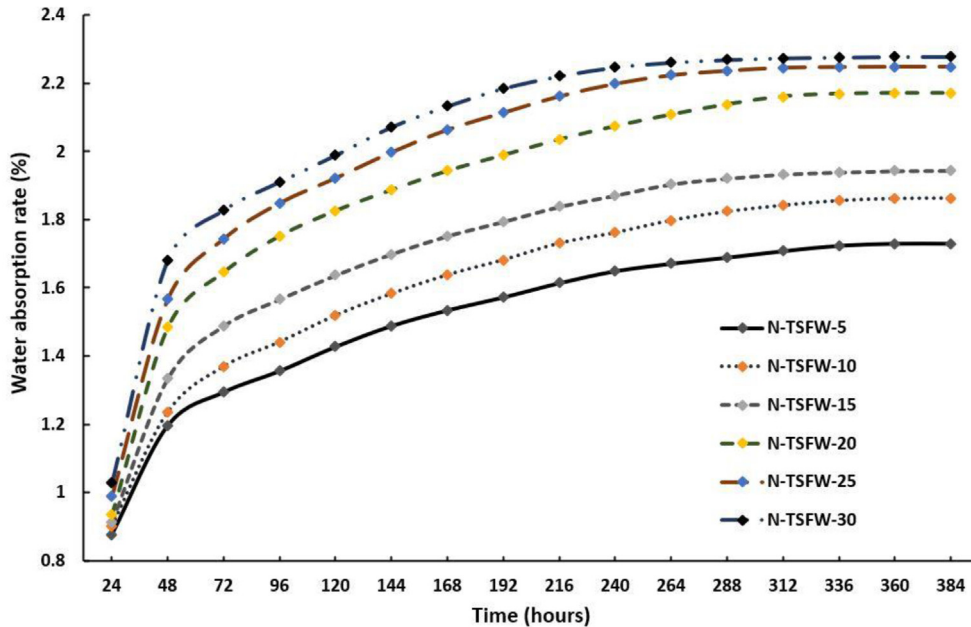
where,

ρ_{th} = Theoretical density, W_m , W_{tsw} , are the weight fraction of epoxy and TSWF respectively. ρ_m and ρ_{tsw} are the theoretical density of matrix and TSWF respectively. As per Eq. (2), V_f denotes void fraction, and ρ_{th} and ρ_{exp} are the theoretical and experimental density of the sample. The sample's water uptake was determined by immersing the sample in distilled water at RT for 16 days or till the composite gets saturates. Before and after the immersion, the weight of the sample was

**Fig. 2 – Void fraction of composites.**



(a)



(b)

Fig. 3 – Water absorption rate of (a) Untreated TFSW composite and (b) NaOH-treated TFSW composite.

monitored on a daily basis, and the water absorption rate was estimated using: Eq. (3).

$$\text{Water absorption (\%)} = \frac{\text{initial weight } (W_i) - \text{final weight } (W_f)}{\text{initial weight } (W_i)} \quad (3)$$

A computerized compression moulding machine was used to carry out the tensile and flexural test according to ASTM D

3039 [50] and ASTM: D790-0 standards [51], respectively at a crosshead speed of 2 mm min^{-1} undergoing a load of 400 KN. Ambient temperature and pressure were kept at a relative humidity of 30%. Span to thickness was kept a 16:1 for a flexural test. The impact test was carried out in digital impact testing machine AIT D 300 in compliance with ASTM: E23 [52] standard with a 2 mm of a deep notch at 45° angle. Vickers hardness of composite was tested in digital Vicker's hardness

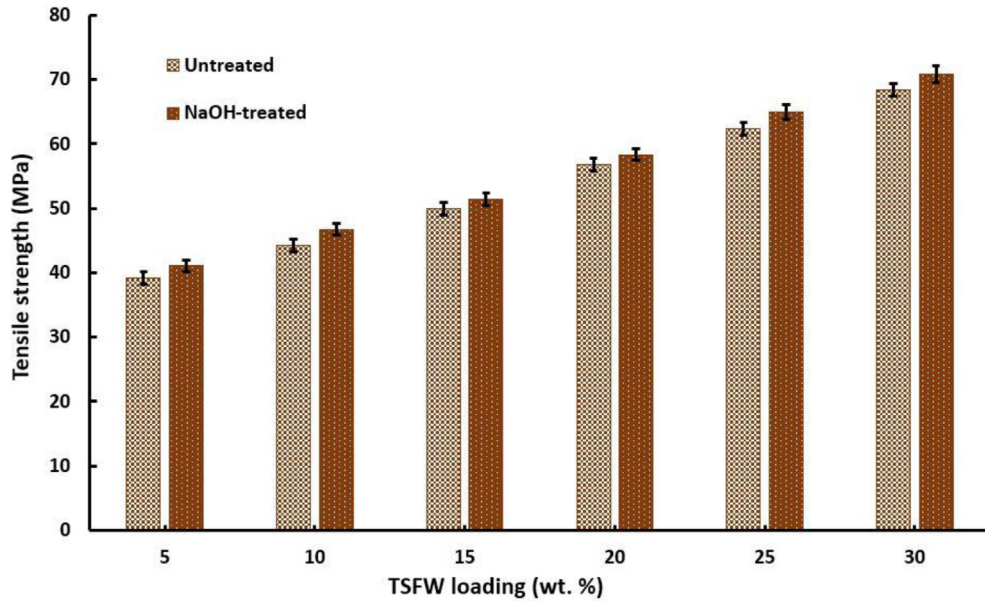


Fig. 4 – Tensile strength of untreated and NaOH-treated TFSW composite.

machine containing diamond indenter with the apical angle of 136° under the load of 1 Kgf applied on the specimen for 10 s at 10 spots.

2.5. Sliding wear characterization

The sliding wear characteristics of the composite were evaluated in accordance with ASTM G-99-17 standard [53]. The composite specimen was first polished, cleaned, and pressed against a steel disc of hardness 65 HRC. The normal load was kept fixed at 10 N and sliding velocity (3.5 m/s to 7 m/s) was varied for test specimens for the examination of sliding wear behaviour. The specific wear rate of the composite specimen was calculated as per: Eq. (4).

$$W_s = \frac{\Delta m}{\rho_{exp.T.V_s.F_n}} \quad (4)$$

where Δm represents a difference in mass before and after the wear, ρ_{exp} represents experimental density, T represents a time of wear, V_s is the sliding velocity and F_n is the normal load.

3. Results and discussions

3.1. Density and voids

The theoretical and experimental density of untreated and NaOH treated TFSW-epoxy composites are shown in Table 2. Observations reveal that experimental densities are slightly lower than theoretical densities for all the compositions due to the presence of voids in formed during the fabrication and curing process. Voids formation is the result of discontinuity present in the composite due to non-homogeneous distribution of TFSW in an epoxy matrix. It can be observed from Fig. 2

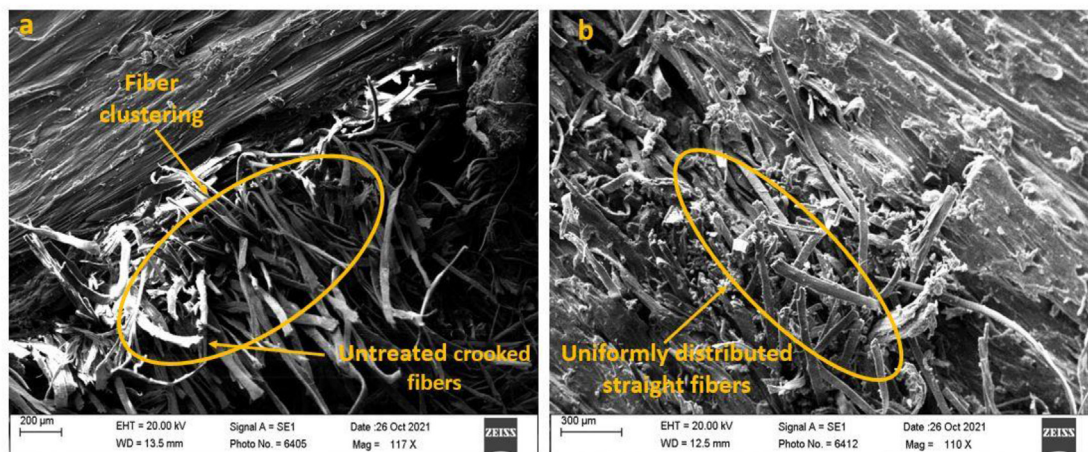


Fig. 5 – Showing tensile fractured specimen for (a) untreated TFSW (b) NaOH treated TFSW specimen.

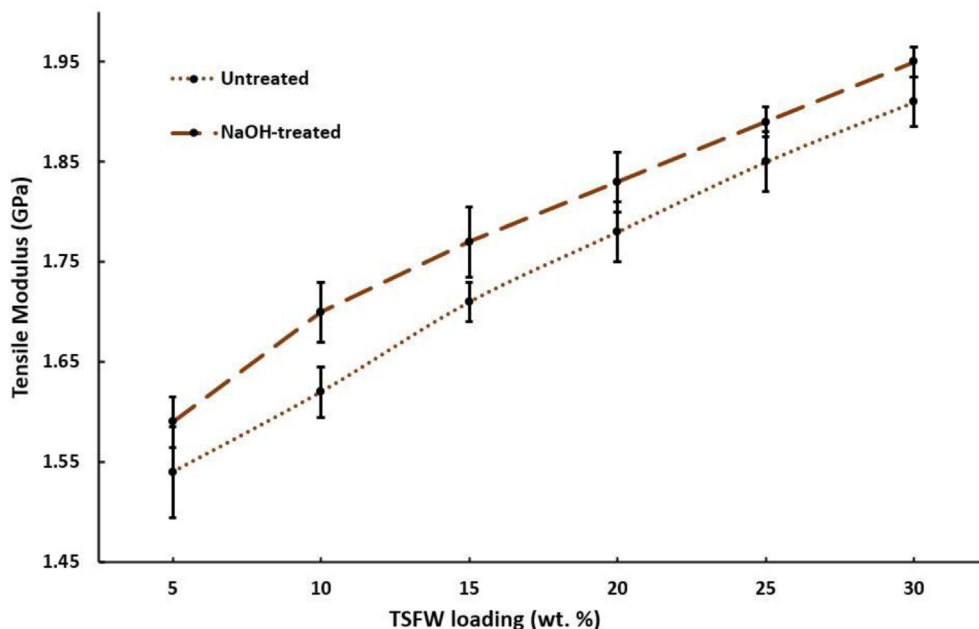


Fig. 6 – Tensile modulus of untreated and NaOH-treated tasar silk waste composite.

that as the TFSW percentage in the composite increases, the void fraction of the composite increases for both untreated and treated composites but, NaOH treated composites exhibited lower void fraction as compared to untreated composites [38]. Treated TFSW was free from unwanted impurities and showed better fiber distribution in the epoxy matrix, which could be the cause of lower void content.

3.2. Water absorption rate

The incorporation of TFSW in epoxy showed a remarkable increase in water absorption as shown in Fig. 3(a). This

increase in water uptake was expected because epoxy is hydrophobic while TFSW is hydrophilic [39]. As the TFSW percentage in epoxy increases, the amount of hydroxyl group and peptide group present in the TFSW increases, which leads to an increase in the ability to absorb the water via the formation of hydrogen bonds. In addition, a molecule of water gets stuck in the voids present in the composites which also increased the water absorption rate. The lowest water absorption rate was observed at 5 wt. % of TFSW while the highest was observed at 30 wt. %. Similar observations for water absorption rate were observed for NaOH treated composite but at a comparatively lower rate as shown in Fig. 3(b). The lower rate

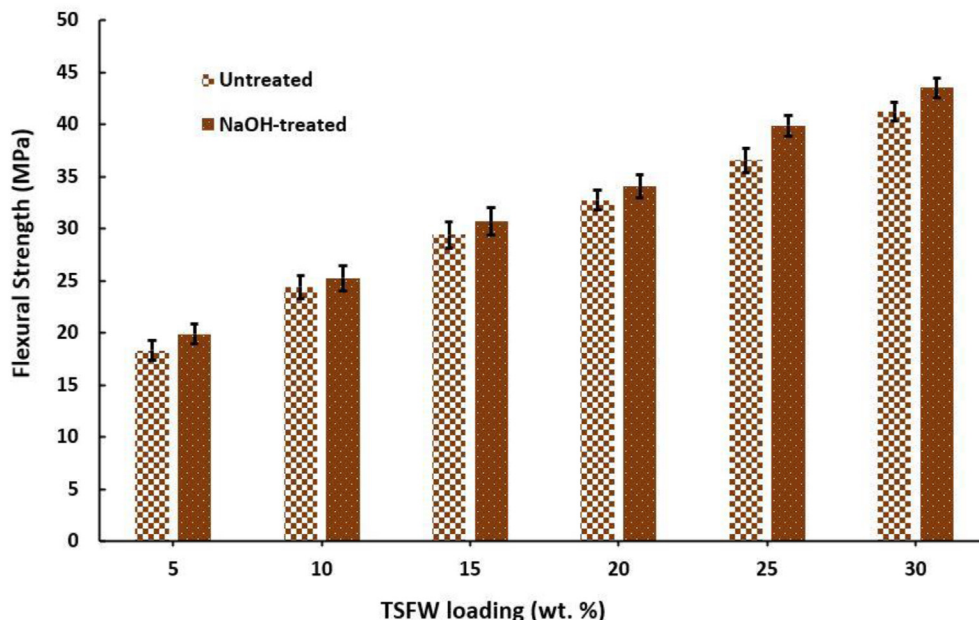


Fig. 7 – Flexural strength of untreated and NaOH-treated TFSW composite.

of water absorption is due to the low void fraction as shown in Fig. 2 which reduces the entrapment of water molecules in the voids as compared to untreated TSWF epoxy composite [40]. Moreover, the treatment of TSWF has led to the elimination of the hydroxyl group from the surfaces thus minimizing the water uptake ability of the composite.

3.3. Mechanical properties

Better mechanical properties can be achieved by enhancing the interfacial adhesion between the fiber and matrix. The improvement in the interfacial adhesion can be attained by

the surface modification of fiber with chemical treatment [41]. The TSWF in the present study was chemically modified by 5 wt. % NaOH solution for mechanical characterization. Figure 4 illustrates the comparison of tensile strength of untreated and NaOH treated TSWF-epoxy composites. Observations reveal that the incorporation of untreated TSWF significantly enhances the tensile strength of the composite from 39.19 ± 0.96 MPa at 5 wt. % to 68.47 ± 1.1 MPa at 30 wt. %. The same NaOH treated TSWF loaded epoxy composites exhibited 41.08 ± 0.93 MPa and 70.86 ± 1.26 MPa at 5 wt. % and 30 wt. % respectively. Although, the tensile strength of the composite increases but at a marginal range. Similar trend

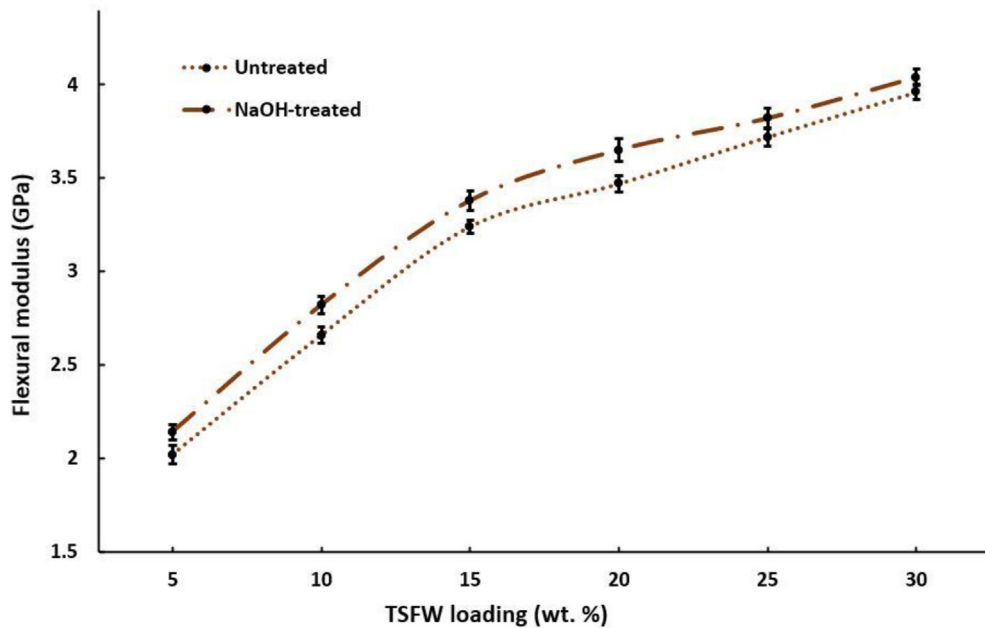


Fig. 8 – Flexural modulus of untreated and NaOH-treated TSWF composite.

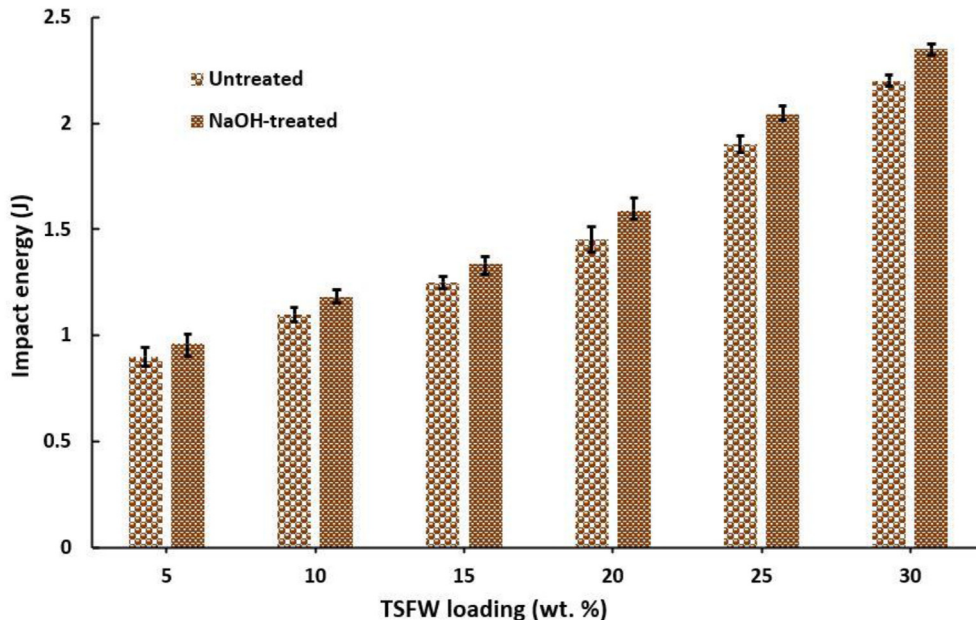


Fig. 9 – Impact energy of untreated and NaOH-treated TSWF composite.

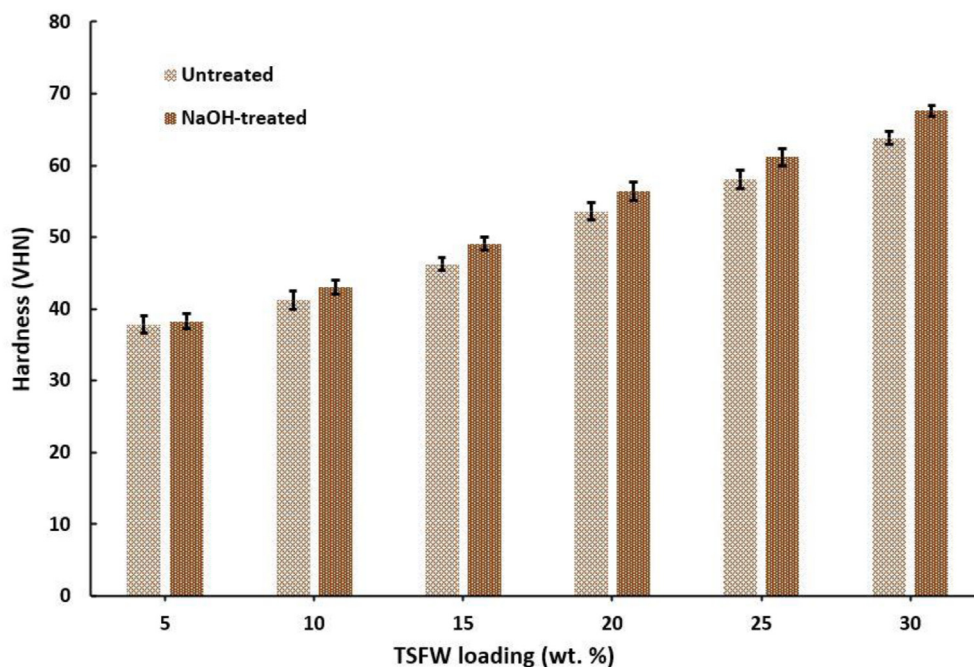


Fig. 10 – Vickers hardness of untreated and NaOH-treated TSWF composite.

was observed for alkali treated hemp fiber-cyanate ester composite [8]. The enhancement in the tensile strength is attributed to the excellent strength of TSWF. The improvement in tensile strength of NaOH treated TSWF composite was because of the removal of sericin from the surface of fiber resulted in an enhancement in the tenacity of fiber. High tenacity consequently leads to improved interfacial adhesion of fiber matrix as reflected in the results as shown in Fig. 4. Moreover, as evident from Fig. 5(b), NaOH treated TSWF fibers distributed uniformly in the matrix as compared to untreated one and avoids the clustering of fiber as observed in case of untreated TSWF as shown in Fig. 5(a). Treatment by NaOH also revealed from Fig. 5 that fiber gets straight and does not entangle during applied load thus leading to improved stress transfer.

The tensile modulus of composites showed approximately 30% enhancement for both untreated and treated TSWF composites as shown in Fig. 6. The tensile modulus was found to be 1.54 ± 0.045 GPa and 1.91 ± 0.025 GPa for untreated and 1.59 ± 0.025 GPa, 1.95 ± 0.01 GPa for NaOH treated at 5 wt. % and 30 wt. % respectively. Tasar silk has very high stiffness as compared to the epoxy resin which imparted high stiffness and restricted the mobility of the epoxy chain thus resulting in an enhancement in the tensile modulus.

The flexural strength of untreated and NaOH treated TSWF-epoxy composites has been illustrated in Fig. 7. Observations revealed that the inclusion of TSWF has shown significant enhancement in the flexural strength of the composite for both untreated and NaOH treated composite. However, noteworthy improvement in the flexural strength is slightly higher for NaOH treated TSWF-epoxy composite. The lowest flexural strength was observed at 5 wt. % as 18.32 ± 0.98 MPa and 19.89 ± 0.92 MPa for untreated and NaOH treated composites respectively and the highest was obtained

at 30 wt. % as 41.18 ± 0.88 MPa and 43.52 ± 0.95 MPa for untreated and NaOH treated composites respectively which are in good agreement with the results reported for sialne treated hemp fiber-cyanate ester composite [9]. The enhancement in flexural strength could be due to the high shear modulus of TSWF which imparted enough strength to the epoxy to bear high bending loads [42]. NaOH treated fiber eliminated the sericin compound and activated alanine and glycine compounds which are hydrophobic and have a higher affinity for making a strong bond with polymer matrix.

The flexural modulus of untreated and NaOH treated TSWF-epoxy composites is shown in Fig. 8. The trend in the flexural modulus followed a similar trend as followed in case of tensile modulus but of higher magnitude. As seen earlier (Fig. 6), the NaOH treated TSWF composite also exhibited a higher value of flexural modulus as compared to untreated TSWF composites. Since, tasar silk is one of the stiffed fiber among all the known natural fiber and thus resulted in the enhancement in flexural modulus of the composite. Improvement in the interfacial locking between treated fiber and epoxy might also be the cause of improved flexural modulus of composite [43].

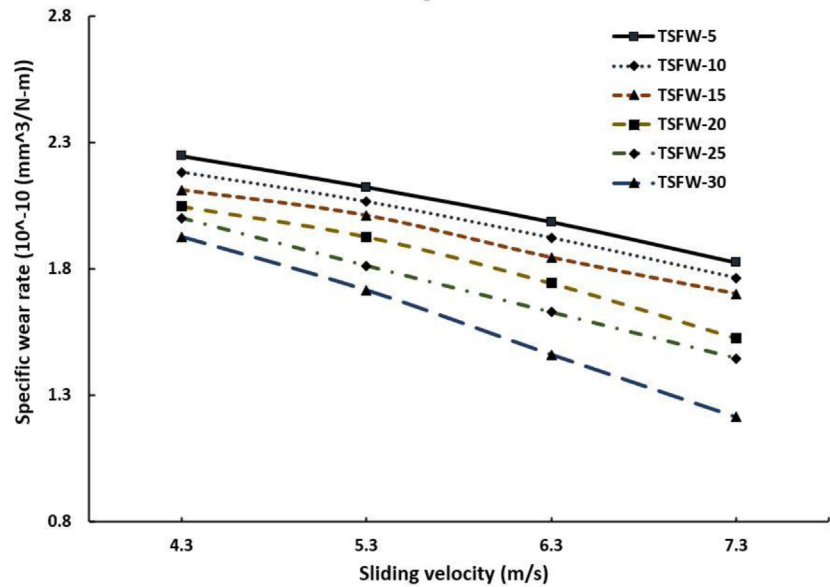
The effect of TSWF loading on the impact energy of the composite is shown in Fig. 9. The addition of TSWF waste has shown significant improvement in the impact strength of the composite as 0.9 ± 0.045 J was observed at 5 wt. % which was increased to $2.2 \text{ J} \pm 0.025$ at 30 wt. % of TSWF addition. On the other hand, NaOH treated TSWF composite exhibited 0.96 ± 0.06 J at 5 wt. % and 2.35 ± 0.03 J at 30 wt. % of fiber loading. The improvement in impact energy is in good sync with sialne treated carbon fiber-cyanate ester composite [10]. The enhancement in the impact energy is credited to the high stiffness and toughness of TSWF endowed with amide and peptide bonds [44]. These bonds are highly hydrophobic and

enable efficient fiber matrix bonding resulting in enhanced impact energy.

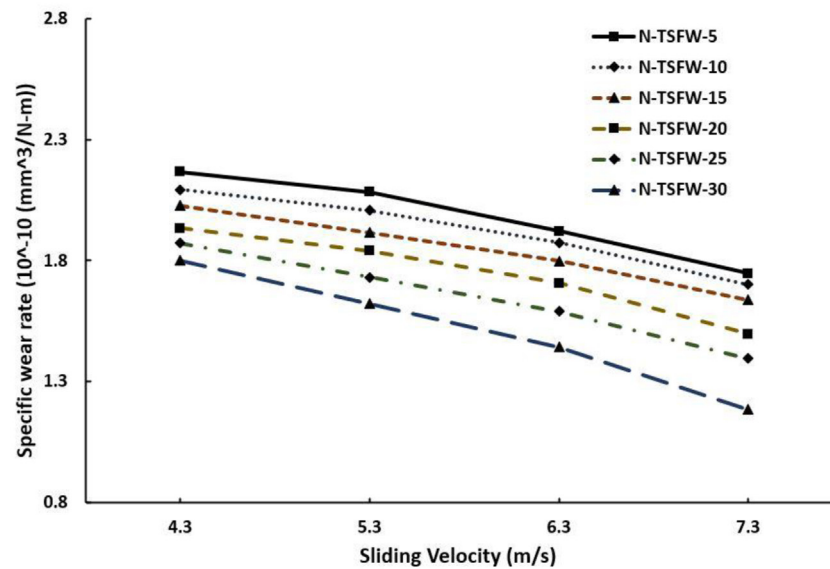
Figure 10 shows Vickers hardness of untreated and NaOH treated TSWF-epoxy composite at different fiber loading. The inclusion of TSWF in epoxy has shown significant enhancement in Vickers hardness number. At 5 wt. % of fiber loading, untreated and NaOH treated TSWF composite exhibited 37.8 ± 1.2 VHN and 38.3 ± 1.1 VHN respectively.

The hardness shoots to 63.8 ± 0.9 VHN and 67.7 ± 0.8 VHN at 30 wt. % of fiber loading for untreated and NaOH treated

composite respectively. TSWF has high tenacity than epoxy matrix which makes the fiber surface smooth [45]. As shown previously, the incorporation of jute and coir fiber in polypropylene significantly enhances the hardness of composite [46]. The smooth fiber causes the composite to act harder resulting in higher hardness. Interestingly, the improvement in mechanical properties of the composite has been obtained at every fiber loading percentage which is attributed to the efficient manufacturing technique.



(a)



(b)

Fig. 11 – Sliding wear rate for (a) untreated and (b) treated TSWF epoxy composite.

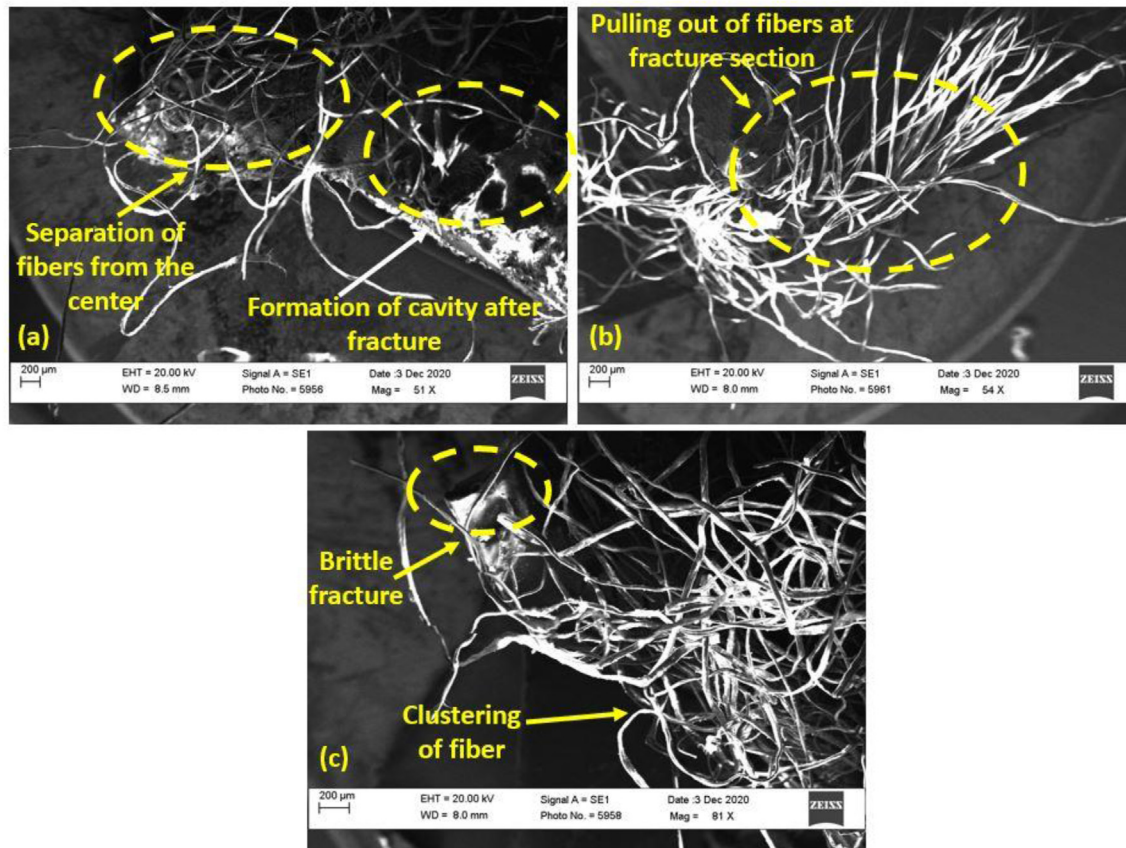


Fig. 12 – Fractograph at 30% untreated TSWF reinforcement: (a) tensile, (b) flexural and (c) impact.

3.4. Specific wear rate

Various applications where polymer composites are exposed to sliding wear are cylinder liner, automobile accessories, prosthetic joints, aerospace, and electric device contact bushes. Meanwhile, Polymer composites are making their place in sliding wear applications due to their light weight, high corrosive resistance, and easy tailorable properties. The inclusion of waste natural fiber in polymer not only enhances the wear behavior but also reduces the overall cost of the composites [47]. In view of this, untreated and NaOH treated TSWF reinforced epoxy composite was examined for sliding wear characteristics at varying velocity and constant normal load as shown in Fig. 11(a and b). Observations show that the specific wear varies between 2.247×10^{-10} mm³/N-m (highest) and 1.214×10^{-10} mm³/N-m (lowest) for untreated TSWF composite while 2.166×10^{-10} mm³/N-m (highest) and 1.184×10^{-10} mm³/N-m (lowest) for treated TSWF composite. Initially, at low velocity (4.3 m/s), the specific wear was high for all the compositions but as the velocity increased, the specific wear drops marginally to 5.3 m/s. The specific wear rate suffered a small drop as the velocity increased from 5.3 m/s to 6.3 m/s and followed by a meager drop in specific wear rate as the velocity goes beyond 6.3 m/s for all the samples. At higher sliding velocity, a low wear rate was witnessed as more fibers were exposed to abrasion caused by the

sliding disc leading to higher resistance offered by the TSWF. Thus, a higher amount of energy was required to facilitate the cutting of fibers. Hence wear rate decreases as the sliding velocity increases [48].

3.5. Fractograph of samples

The microscopic analysis of the fractured surface of the tensile specimen has been dominant with cavity formation due to the ductile fracture at the interface of the fractured zone. The cavity formation resulted from the separation of fiber from the mid and agglomeration at the corner as shown in Fig. 12(a). Fiber pull out in the axial direction was observed in the shear fracture of the flexural specimen along with ductile deformation as shown in Fig. 12(b). Clustering of fiber at mid and corner zones accompanied by brittle fracture signifies the debonding of fiber and matrix. Figure 13(a) shows the fractured zone of NaOH treated tensile specimen. It was observed that the fibers were separated at a greater distance as compared to the micrograph of the tensile specimen as shown in Fig. 12(a) resulting in the formation of a crack at the center. A little fiber breakage occurs which may be due to the hardening and roughening of fiber that took place due to the NaOH treatment.

The flexural specimen fractured along the transverse direction leading to the formation of the plane surface with the

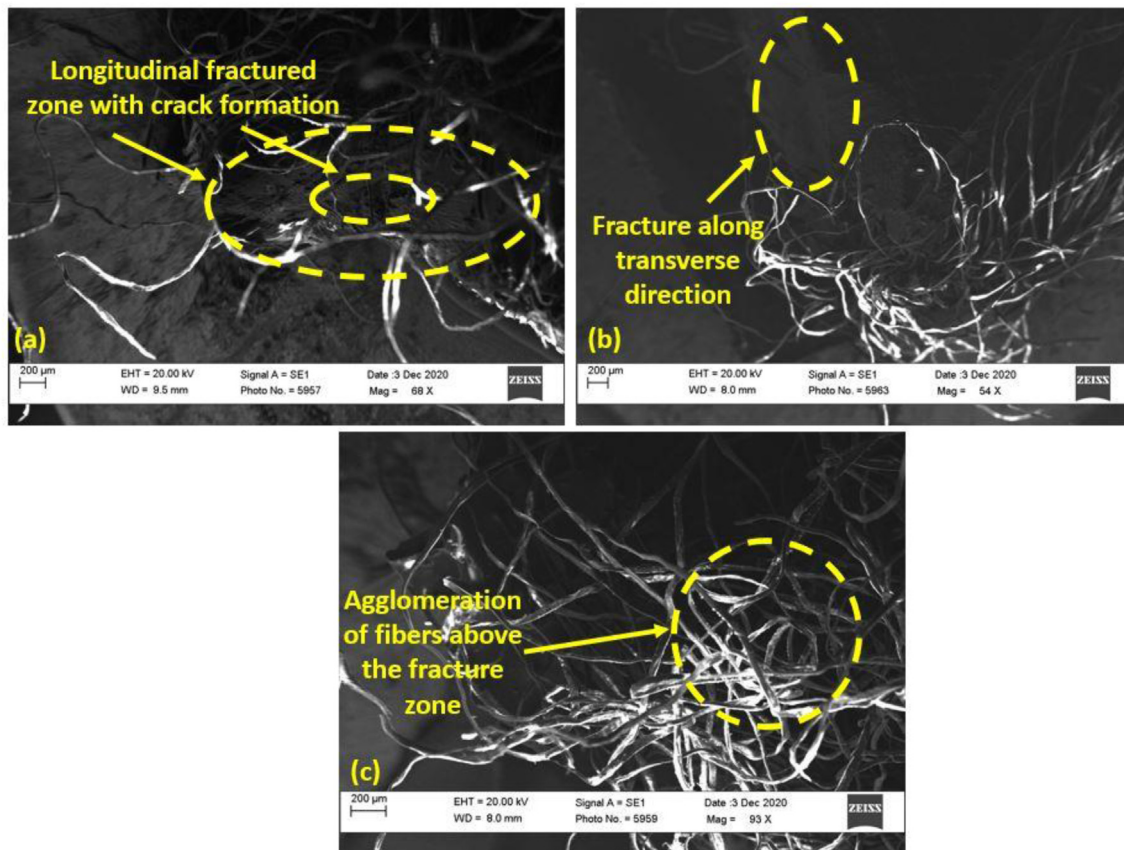


Fig. 13 – Fractograph at 30% NaOH treated TSWF reinforcement: (a) tensile, (b) flexural and (c) impact.

presence of few fibers along with bending of fibers along the perpendicular direction as observed in Fig. 13(b). Figure 13(c) shows the clustering of fiber right above the fractured surface of NaOH treated impact test fractured specimen. Bending and mingling of fiber in one another were also observed in the same specimen. Observations revealed that the fractured surfaces have undergone mostly ductile deformation due to the high elongation of the break of TSWF. However, agglomeration of fiber leads to the formation of cracks but NaOH treated specimens have minimized the crack propagation up to a certain level and resulted in twisting and winding as resistance against the external load [49].

4. Conclusions

Tasar silk fiber waste reinforced epoxy composite was successfully fabricated by compression moulding method and tested for physical, mechanical, and wear behaviour. Physical results suggested that reinforcing TSWF in the epoxy resulted in an increase in void fraction due to the increase in the theoretical density of the composite. In particular, maximum voids were obtained as 5.1 and 5.8% for treated and untreated TSWF composite respectively. Improvement in the tensile (3.47%), flexural (5.37%), impact (6.8%), and Vickers hardness (6%) were obtained because of the higher interfacial adhesion. NaOH treatment of TSWF results in a marginal increment in the mechanical strength of the composite while a

significant reduction in specific wear was observed for both untreated and NaOH treated TSWF reinforced epoxy composite. SEM analysis suggested that the high elongation of break of TSWF has been the critical factor for the enhancement in the mechanical strength. Moreover, brittle fracture, fiber pull out, clustering of fiber, and transverse failure are some of the phenomena observed in the SEM analysis. The present work may be extended for various other chemical treatment such as sodium bi carbonate, acetylation, permagnate, isocynate, benzoylation etc. For the enhancement of interfacial bonding between the fiber and matrix. Since, tasar silk has major composition of protein, its biocomposite with polylactic acid, poly vinyl chloride, or polyhydroxybutyrate can be utilized as scaffold material or tissue engineering with the advantage of lowering of cost and enhanced biocompatibility. For improved strength of the biocomposite, a natural fiber such as coir, hemp or jute can also be hybridized in the silk-biopolymer composite. The result as demonstrated in the present article may be act as a useful information for the development of tasar silk waste reinforced biopolymer composites in the near future.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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