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Study of Mechanical Properties of Alkali Treated Smilax Zeylanica Fiber/Sisal Fiber/Vinyl Ester Hybrid Composites under Wet Condition

Athijayamani AYYANAR [1] *, Ramkumar GP [2], Alavudeen AZIZ BATCHA [3], Thiruchitrambalam MANI [4]

[1] *Department of Mechanical Engineering, Government College of Engineering, Bodinayakanur 625 582, Tamilnadu, India*
[2] *Department of Mechanical Engineering, Karaikudi Institute of Technology and Management, Karaikudi 630307, Tamilnadu, India*
[3] *Department of Mechanical Engineering, Kalasalingam University, Srivilliputhur 626128, Tamilnadu, India*
[4] *Department of Mechanical Engineering, Vel Tech Multi Tech Dr.Rangarajan Dr.Sakunthala Engineering College, Chennai 600062, Tamilnadu, India*

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Mechanical properties of vinyl ester hybrid composites reinforced with alkali treated Smilax zeylanica and sisal fibers were studied at wet condition in the present communication. Hybrid composites were fabricated by using a simple hand lay up technique based on three different fiber loading of 25, 35, and 45 wt.% with alkali treated fibers. Hybrid composite specimens were then subjected to the water absorption test to observe the behaviours of composite specimens at wet condition under mechanical loads such as tensile, flexural and impact. The water absorption test was carried out in two ways in the distilled water environment at room temperature. The first way test was conducted for 10 days to observe the percentage of water particle absorption of hybrid composites. The second way test was performed for 5 days to determine the mechanical properties of hybrid composites at wet condition to observe its durability when they are used in outdoor applications. Mechanical properties of hybrid composite specimens at wet conditions were compared with the dry composite specimens. Experimental results showed that the percentage of the water particle absorption in the alkali treated hybrid fiber composites is lower as compared to the untreated hybrid fiber composites. Mechanical properties of alkali treated hybrid fiber composites at wet condition are slightly reduced as compared to the treated hybrid fiber composite at dry condition. As a result, it is observed that the resistance to the penetration of the water particles is higher for the alkali treated smilax zeylanica and sisal fibers reinforced vinyl ester hybrid composites. The fracture surfaces of the hybrid composite specimens were examined by scanning electron microscope to understand the effects of water absorption on the mechanical properties.

Keywords: smilax zeylanica fibers, sisal fibers, vinyl ester, mechanical properties, scanning electron microscopy.

1. INTRODUCTION

The fillers in the form of fibers and particles from various plants, vegetables, fruits, seeds and the other agricultural residues represent an inexpensive, readily available, and abundant source of renewable and biodegradable lignocelluloses materials [1, 2]. Recently, new composite materials using bio renewable resources are developed following the philosophy of sustainable development that is helpful to more and more materials. The renewable resources-based parts and products are manufactured in recommencing interest due to ecological concerns. Therefore, natural cellulose fillers (fibers and particles) and biodegradable polymers are considered as environmentally safe and suitable alternative to synthetic fillers [3, 4].

Many industries like structural, automotive, and packaging have been shown their serious and enormous interest in the development of composite materials filled with natural cellulose fibers. Natural cellulose fibers present some well known advantages like low cost and lower density over the synthetic fibers such as glass, carbon, and aramid. Natural cellulose fibers are biodegradable, renewable, less abrasive to the processing equipment, harmless, and specific mechanical properties [5, 6].

A number of studies have been carried out on the natural cellulose fiber reinforced polymer composites. The properties of these composites mainly depend on fibers and their chemical compositions. All natural cellulose fibers are hydrophilic in nature due to hydroxyl groups. Though, the main disadvantage of natural cellulose fibers with the polymer composites is the poor compatibility with the hydrophobic polymer-matrix. This behaviour of natural cellulose fibers leads to the formation of a weak interface with the polymer matrix, which results in poor mechanical properties [7, 8]. Moreover, the transfer of applied load from the polymer matrix to the fiber is determined by the level of the interfacial adhesion, which is achieved either by the fiber and the polymer matrix modification with physical and chemical treatments or by use of additives like beaching and chlorination [9, 10].

Generally, natural cellulose fillers like fibers and particles have a high moisture absorption tendency, which is one of the main factors affecting the overall properties of the natural cellulose fillers reinforced polymer composites [11, 12]. Therefore, the usage of natural cellulose fibers as

* Corresponding author. Tel.: +91-9865906160; fax: +91 0456 224512.
E-mail address: *athimania@gmail.com* (A. Athijayamani)

reinforcing agent has been limited by their weakness to moisture absorption due to their hydrophilic in nature. Moisture absorption of natural cellulose fiber reinforced polymer composites results in the formation of micro-cracks at the fiber-matrix interface due to the swelling of the fibers which can reduce the dimensional stability and mechanical properties [13, 14]. From the literature survey, it is identified that no studies focused on the mechanical properties of Smilax zeylanica and sisal fiber reinforced vinyl ester hybrid composites at wet condition. Therefore, this study will focus on the preparation of Vinyl Ester (VE) composite with alkali treated Smilax Zeylanica Fibers (SZFs) and Sisal Fibers (SFs) by a simple hand lay-up technique and the estimation of their mechanical properties at wet condition. In order to study the prospective of use of these composites in humid environmental conditions or outdoor applications, the water absorption test is performed on these alkali treated composite at room temperature. The fracture surfaces of the composite specimen after test are examined by Scanning Electron Microscope (SEM).

2. EXPERIMENTAL DETAILS

2.1. Materials

The well-grown stems of Smilax zeylanica plants (*Smilacaceae*) were cut and collected from Panangudi village near Sivaganga District, Tamilandu, India. The SZFs were peeled off carefully from the stem using a sharp blade and then soaked in pond water for two weeks for removal of the foreign materials. Subsequently, the fibers were separated and thoroughly washed in fresh running water and also allowed to dry in the sunlight. Finally, the SZFs are used as reinforcing agents as received condition for the preparation of polymer matrix composite.

Sisal fibers were extracted from the well-grown leaves of the sisal plant (*Agave sisalana*) as in our previous study [15]. The extracted sisal fibers are also used as reinforcing agent with SZFs as received condition for hybridization.

The polymer resin matrix used for this study was a commercially available vinyl ester resin, (Satyen Polymers Pvt. Ltd, Bangalore) supplied by GVR Enterprise, Madurai, Tamilnadu, India. The vinyl ester resin matrix was mixed with curing catalyst (Cobalt naphthenate 6 %) at a concentration of 0.01 w/w of the matrix for curing. Methyl ethyl ketone peroxide was used as an accelerator for resin matrix.

2.2. Alkali treatment

The alkali treatment process of SZFs starts with immersing the fibers in alkaline solution. Therefore, 5% sodium hydroxide solution was initially taken in a glass tray and then completely dried SZFs and SFs were immersed in this solution about two hours at room temperature. After that, they are rinsed with distilled water to remove the excess alkaline solution stick to the SZFs and SFs. Finally, the treated fibers were again allowed to dry at sunlight.

2.3. Reasons for alkali treatment of fibers

Natural cellulose fillers (fiber and particles) derived from bio resources like plants, vegetables, fruits and seeds are not only renewable and biodegradable, but also have several other advantages such as abundantly available, low cost, lightweight, and higher specific properties [16]. Natural cellulose fibers are used as a suitable alternative for the synthetic fillers like glass in the polymer composites and their applications can be found in various fields such as automotive, construction, packaging, and furniture. Although several advantages, natural cellulose fibers also possess some disadvantages like high moisture absorption tendency, incompatibility and wettability with some polymer resin matrices in the preparation of composite materials [17 – 19]. To resolve these problems of natural cellulose fibers, some physical and chemical treatments employed for surface modification are suggested by several material researchers. Among various treatments, alkali (sodium hydroxide) treatment is one of the most effective and simplest methods in the surface modification of natural fibers [20 – 23].

Alkali treatment removes a certain amount chemical element such as lignin, hemicellulose, pectin and oils covering the external surface of the fibers. Moreover, the important effect of alkali treatment on natural cellulose fibers is increasing surface roughness, which can be achieved by the disruption of the hydrogen bonding presented in the structure of the fibers. Therefore, a strong interfacial adhesion (mechanical interlocking) can be obtained between the fibers and the polymer resin matrix with improved properties like physical and mechanical [24]. With aim of developing polymer hybrid composites having better performance, the alkali treated Smilax zeylanica and sisal fibers reinforced vinyl ester hybrid composite was prepared in this study and their mechanical properties were determined at wet condition (water absorption) to investigate the influence of alkali treatment on the resistance of water absorption experimentally.

2.4. Composite preparation

A simple hand lay-up technique [25] was used to produce the SZF/SF/VE composite plates; a steel mould with size of $150 \times 150 \times 3$ mm was used to prepare the composites. Alkali treated SZFs and SFs with different weight percentages of 25, 35, and 45 wt.% were used to prepare VE hybrid composites. Both the fibers were mixed with the resin matrix at 1:1 ratio. Hybrid composite plates were cast at room temperature and allowed to cure for 24 hours. Finally, the cured composite plates were removed carefully from the mould box and cut as per the ASTM standards for water absorption and mechanical testing.

2.5. Water absorption test of the hybrid composites

Water absorption test at two parts was carried out in a distilled water environment to evaluate the mechanical properties and the sorption behaviours of alkali treated SZF/SF/VE hybrid composites. Water absorption tests are conducted according to ASTM 570 at the room temperature. The effects of 10 days water immersion on the sorption behaviour of the treated hybrid composites were studied in the part based on the weight percentages of SZFs and SFs. After immersion for the particular time, the hybrid composite specimens were taken out from the water baths and hybrid composite specimens were cleaned using the dry cloth to make sure the removal of the water molecules from the surface of the hybrid composite specimens. The hybrid composite specimens are weighted and again immersed in the same water baths. For 10 days of water immersion, the percentage of the water absorption is measured by the weight difference between the hybrid composite specimens before and after immersion using the following equation:

$$W_{up}(\%) = \frac{W_{at} - W_{ao}}{W_{ao}} \times 100 , \quad (1)$$

where W_{ao} is the weight of the hybrid composite specimen before soaking into the distilled water environment (grams), and W_{at} is the weight of the composite specimens after soaking into the distilled water environment (grams). In the second part, the effects of 5 days water immersion on the mechanical properties of alkali treated fiber composites are studied based on the weight percentages of SZFs and SFs and compared with the treated hybrid composites at dry condition.

2.6. Mechanical testing

For tensile property, the composite specimens were tested on a computerized universal testing machine. Tensile strength values of the hybrid composites were observed in accordance with ASTM D638 – 10 standard at a crosshead speed of 5 mm/min. The flexural tests were conducted for the hybrid composite specimens by the three-point bending method on the same universal testing machine. The flexural strength values were determined according to ASTM D790-10 procedure at a crosshead speed of 5 mm/min. In impact test, the strength of the samples was determined using an Izod impact test machine having the hammer with a weight of 300 Joule. Hybrid composite specimens were tested at impact load according to the procedure of ISO 180. Totally, five hybrid composite specimens were tested to get an average value at all tests.

2.7. SEM Study

An SEM machine (Model-Hitachi S-3000N) was used to examine the fracture surface of hybrid composite specimens after tensile, flexural and impact tests.

3. RESULTS AND DISCUSSION

3.1. Water absorption behaviour of alkali treated hybrid composites

The percentage of water absorption according to the hybrid fiber content during distilled water immersion of alkali treated SZF/SF/VE hybrid composite with an increase in soaking time is depicted in Fig. 1. It is observed that the rate of increase of water absorption decreases with the immersion time for all hybrid composites. The hybrid composites with 45 wt.% hybrid fiber content shows higher the percentage of water absorption as compared to 25 wt.% and 35 wt.% hybrid composites. It may be due to the hybrid fibers contain abundant polar hydroxide groups, which result in a high water absorption level of hybrid composites at 45 wt.%. It may also be due to the nature of the hybrid fibers, voids formation inside the hybrid composites lead to the initiation of micro cracks in the composite surface, which results in the penetration of water molecules to the composite material by capillary action [26].

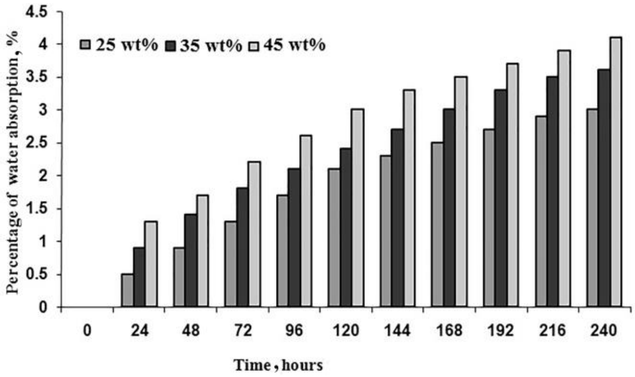
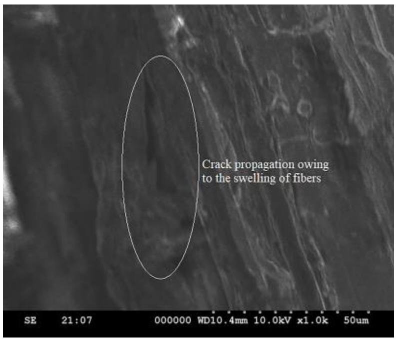


Fig. 1. Variations in water absorption percentage according to the immersion time for alkali treated SZF/SF/VE hybrid composites

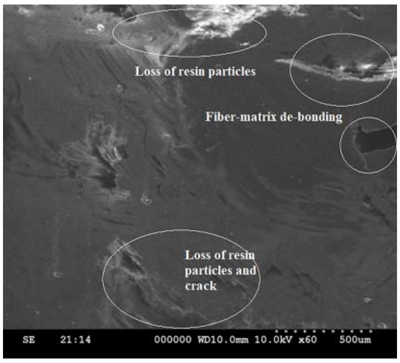
The surface of the natural cellulose fibers becomes rough after treating by alkali solution and leads to better interfacial adhesion between the fibers and the matrix. Therefore, the voids and gaps between the fiber and the matrix are reduced. Hence, the movement of the water molecules in the structure of the composites is controlled. The SZFs and SFs can absorb more water molecules than the VE resin matrix, because they are hydrophilic in nature. Although the polymer composites are prepared with the alkali treated natural cellulose fibers, the water molecules are penetrated into the composite specimens through the cut sides by a diffusion process. For alkali treated SZF/SF/VE hybrid composites, the diffusion coefficients can be calculated using the water absorption data. The SEM micrographs of the surface of the alkali treated hybrid composite specimens after distilled water immersion are presented in Fig. 2 a and b. The crack propagation owing to the swelling of the fibers and loss of resin particles due to the diffusion of the water molecules is observed from the Fig. 2 a. Losses of resin particles and fiber-matrix de-bonding on the surface of the composite specimens are also identified from the Fig. 2 b, which leads to the crack formation on the surface of the fiber reinforced polymer composite samples. Therefore, composite specimens fail quickly due to the penetration of water molecules.

3.2 Diffusion coefficients

To observe the behaviour of water absorption of the SZF/SF/VE hybrid composites, the tests were carried out by immersing the hybrid composite specimens in a water bath (distilled, at room temperature) during a time stage until the saturation level was reached. The penetration of water molecules into the composites was formed by three different mechanisms. The water particles are entered into the polymer composites reinforced with the natural cellulose fibers by the different mechanisms such as Fickian law, Relaxation control, and non-Fickian law. The entering of water particles into the polymer matrix composites has been followed by Fickian and non-Fickian diffusion laws [27]. Several researchers have been reported the diffusion of water particles in the fiber reinforced polymer composites carried out by the Fick's law [28, 29]. Stevulova et al. [30] have evaluated the diffusion mechanisms by the shape of the sorption curve as given in Eq. 1.



a



b

Fig. 2. SEM micrographs of the fracture surface of alkali treated SZF/SF/VE hybrid composite specimens after distilled water immersion

Moreover, the diffusion mechanisms can be classified as Fickan (n = 0.5), Relaxation control (n > 0.5), and non-Fickan (0.5 < n < 1) according to the value of the angular coefficient. The water absorption data are used by the Eq. 2 for the analysis of the diffusion mechanism according to the Fick's law as follows:

$$\frac{W_{at}}{W_{a\infty}} = kt^n ; \tag{2}$$

$$\log\left(\frac{W_{at}}{W_{a\infty}}\right) = \log(k) + n\log(t), \tag{3}$$

where $W_{a\infty}$ is the amount of water particle at the equilibrium stage during water immersion and W_{at} is the amount of water particle at time t during water immersion; the terms 'n' and 'k' are constants i.e., angular and linear coefficients and there are calculated from slope and intercept of plot of log ($W_{at}/W_{a\infty}$) vs log (t). The diffusion curve fitting log plot of '($W_{at}/W_{a\infty}$) versus time)' for alkali treated hybrid composites is illustrated in Fig. 3. The values of linear and angular coefficients for the alkali treated SZF/SF/VE hybrid composite are given in Table 1. From Table 1, it can be seen that the values of angular coefficient 'n' are very close to n > 0.5. Consequently, it is proved that the diffusion of water particles into the hybrid composites is followed non-Fickian diffusion law. The relationship between the data obtained during water absorption and immersion time is obtained by the value of $R^{2,}$ which is the coefficient of determination and found to be a good agreement. Besides, the coefficient of diffusion 'D' (parameter of the non-Fickian diffusion law) is the ability of the water particles to enter inside the hybrid composite samples and can be determined using the following equation:

$$D = \frac{\pi Z^2 h^2}{16 W_{a\infty}^2}, \tag{4}$$

where 'h' is the thickness of hybrid composite specimens and 'Z' is the initial linear part of the experimental water absorption curve.

$$\theta = \frac{W_{a\infty} - W_{at}}{\sqrt{t_2} - \sqrt{t_1}}. \tag{5}$$

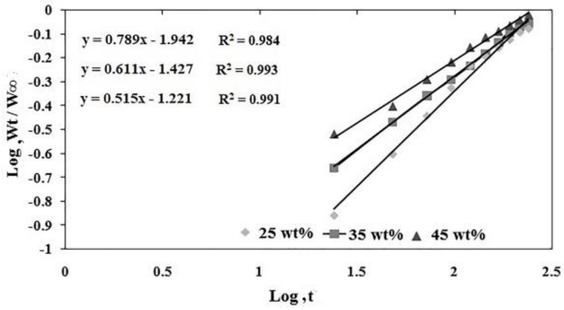


Fig. 3. Diffusion curve fitting plot for alkali treated SZF/SF/VE hybrid composites

Table 1. The obtained values of constants for all SZF/SF/VE hybrid composites in distilled water absorption

Hybrid composites SZF/SF/VE, wt.%	Angular (n) coefficients, liner (k) coefficients and coefficients of determination (R^2)		
	n	k	R^2
25	0.789	1.942	0.984
35	0.611	1.427	0.993
45	0.515	1.221	0.991

The diffusion curve fitting plot of W_t versus time$^{0.5}$ for hybrid composites was presented in Fig. 4.

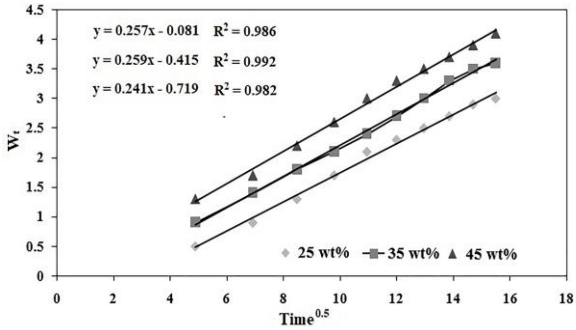


Fig. 4. Diffusion curve fitting plot W_t and *(time)*$^{0.5}$ for alkali treated SZF/SF/VE hybrid composites exposed to the distilled water environment

Table 2 shows the values of the co-efficient of diffusion 'D' obtained from the plot of W_t versus time$^{0.5}$ for alkali treated SZF/SF/VE hybrid composites. The results showed that the values of ‘*D*’ for all hybrid composites are almost the same. Moreover, the values of ‘*D*’ increase with increasing hybrid fiber content. Hence, it is evident that the water absorption process in the alkali treated SZF/SF/VE hybrid composite is taken by non-Fickian diffusion law.

Table 2. The values of ‘*D*’ for alkali treated SZF/SF/VE hybrid composites

Hybrid composites, wt.%	Z	k	R^2	$D \times 10^{-12}$, m^2s^-
25	0.257	0.081	0.986	0.076592
35	0.259	0.415	0.992	0.077913
45	0.241	0.719	0.982	0.079781

3.3. Effect of water absorption on the tensile strength of alkali treated hybrid composites

The tensile strength versus hybrid fiber weight percentage of alkali treated SZF/SF/VE hybrid composite specimens at wet condition is illustrated in Fig. 5.

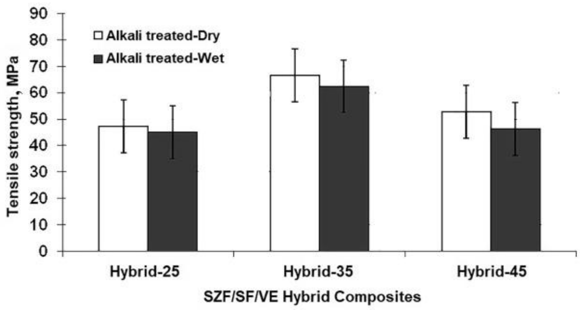


Fig. 5. Variations in tensile strength of alkali treated CGF/AFF/PF hybrid composites at wet condition

It is observed that for treated hybrid composites at dry condition, the tensile strength is found to increase significantly as the hybrid fiber content increased until 35 wt.%. The maximum tensile strength is observed as 66.7 MPa at 35 wt.% hybrid composite. After this increment, the tensile strength drops considerably on the further addition of SZFs and SFs at 45 wt.%. The tensile strength of the alkali treated hybrid fiber composites decreased with exposure to the distilled water immersion. The tensile strength of 35 wt.% of the treated hybrid composite at wet condition was 62.5 MPa. It is 6.3 % lower than that of 35 wt.% of the treated hybrid composite at dry condition. The tensile strength of alkali treated hybrid composite at wet condition is slightly lower as compared to the treated fiber composite at dry condition. Due to the better mechanical interlocking and interfacial adhesion, the formation of micro cracks and gaps between the regions of the fiber-matrix area is reduced. Therefore, the penetration of water particles into the interface may control, resulting in the reduced percentage of water absorption. Moreover, the decreasing tensile strength of alkali treated hybrid composite after distilled water immersion may be due to the formation of hydrogen bonding between the treated fibers and water molecules. The SEM micrograph of fracture surface of alkali treated 35 wt.% hybrid composite at wet condition is illustrated in Fig. 6. Fig. 6 shows the resin matrix fracture, crack propagation, and de-bonding between the fiber and the resin matrix. The crack propagation by the resin matrix fracture leads the failure of the composites. It is also observed from Fig. 6 that the fractures with the brittle nature of most of the SZFs and SFs were identified during the observation due to the alkali treatment of fibers. The brittle nature of the fracture shows the best mechanical interlocking between the fibers and the matrix.

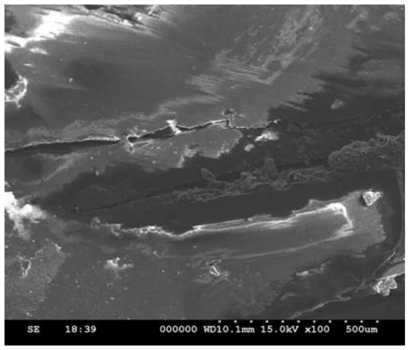


Fig. 6. SEM micrograph of fracture surface of hybrid composite specimen (35 wt.%) after tensile test

3.4. Effect of water absorption on the flexural properties of alkali treated hybrid composites

Alkali treated SZF/SF/VE hybrid composites at wet condition are tested at flexural load by three point bending procedure and their results are presented in Fig. 7. From Fig. 7, it is observed that the hybrid composite specimens at wet condition show the decreasing trend when compared with the hybrid composite specimens at dry condition.

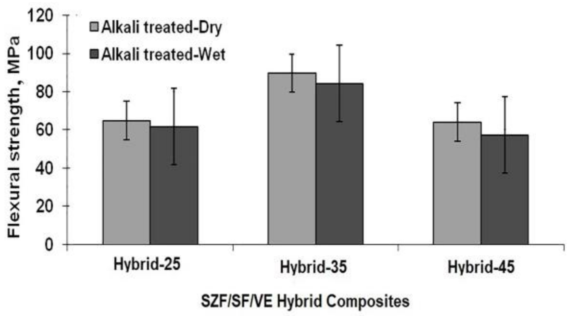


Fig. 7. Variations in flexural strength of alkali treated SZF/SF/VE hybrid composites at wet condition

Moreover, at both the conditions the hybrid composite with 35 wt.% of fiber content showed the maximum flexural strength value. However, the flexural strength of 35 wt.% of hybrid composite at wet condition is lower by 6.1 % as compared to hybrid composite at dry condition. The reason may be due to the swelling of the hybrid fibers which can be created by the penetration of water particles in the region between the fiber and the polymer matrix. Hybrid composite with 45 wt.% at wet condition shows the reduction of 10.4 % in flexural strength when compared to the hybrid composite at dry condition. The flexural strength values of hybrid composite at wet condition are slightly varied than that of hybrid composite at dry condition. Hence, the percentage of reduction in the flexural strength is less at the wet hybrid composites. The reason for this less reduction is that the entering of water particles into the interfaces of composites is controlled by better interfacial adhesion between the fiber and the matrix. Due to the surface modification, hydrophilic nature of the fibers is changed to a certain level, as a result, the decreasing of penetration of water particles into the hybrid composites. The unstructured compounds from the surface of fiber bundles are the partially removed by the surface modification, which decreases the hydrogen bonding formation in the fiber structure. Hence, the number of hydrogen bonding tending to react with water particles is decreased [31]. From the SEM illustration, the separation of the fibers from the resin matrix and fiber pull out can be observed on the fracture surface of the alkali treated SZF/SF/VE hybrid composite (45 wt.%) at wet condition, as shown in Fig. 8.

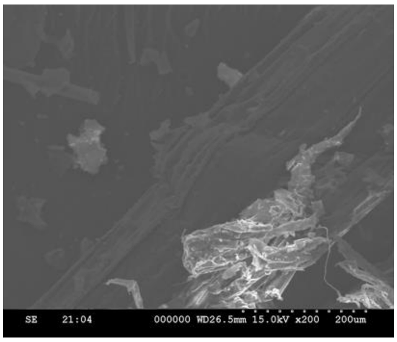


Fig. 8. SEM micrograph of the fracture surface of alkali treated SZF/SF/VE hybrid composite (45 wt.%) after the flexural test

3.5. Effect of water absorption on the impact strength of alkali treated hybrid composites

Fig. 9 illustrates the results of impact test carried out on the SZF/SF/VE hybrid composites. It can be seen from Fig. 9 that impact strength of all alkali treated hybrid composite specimens slightly decreased due to the immersion in the distilled water environment.

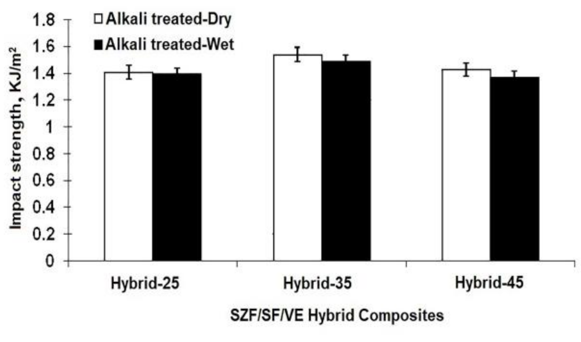


Fig. 9. Variations in impact strength of alkali treated SZF/SF/VE hybrid composites at wet condition

It may due to the penetration of water particles inside the hybrid composite specimens and they are damaging the interfacial region of the fibers and the polymer matrix. Even though the polymer composite materials are reinforced with alkali treated natural cellulose fibers, they have some voids and channels during manufacturing. These voids and channels present in the composites allow the penetration of water particles inside the composite materials and cause fiber swelling. After that, a weak interfacial adhesion is created between the fibers and the polymer matrix due to poor chemical bonds, consequently, the composites are attaining the quick failure during loading with reduced property levels. For the 35 wt.% hybrid composite at wet condition the impact strength is lower than that for 35 wt.% hybrid composite at dry condition. The reasons may be due to the entering of high amounts of water particles, which causes swelling of the fibers by breaking of chemical bonds. Subsequently the de-bonding between the fiber and the polymer matrix is created, as a result, a decrease in the impact strength of the hybrid composites.

4. CONCLUSIONS

Water absorption behaviour and their effects on the mechanical properties of alkali treated SZF/SF/VE hybrid composites have been studied based on the content of SZFs and SFs. Water absorption tests were conducted in two ways. In the first way, the hybrid composite specimens were immersed in distilled water environment for 10 days to understand the water absorption behaviour of the alkali treated SZF/SF/VE hybrid composites. It was identified that the percentage of water absorption is lower at alkali treated hybrid composites as compared to the untreated hybrid composites. The hybrid composites containing 45 wt.% of hybrid fiber content shows higher the percentage of water absorption as compared to composites containing 25 wt.% and 35 wt.% of fiber content, respectively. Moreover, the hybrid composites were immersed at same water path for 5 days to observe their mechanical properties at wet condition. It is observed that the water absorption affects the mechanical properties of the alkali treated SZF/SF/VE hybrid composite. It is proved by the reduction of the mechanical properties of alkali treated hybrid composites at wet condition as compared to the hybrid composites at dry condition. The percentage of reduction of mechanical properties at wet hybrid composites was slightly lower than the dry hybrid composites. Hybrid composites with 35 wt.% of fiber content showed the maximum mechanical properties at both dry and wet conditions. From the 10 days distilled water immersion test, it is observed that the water absorption process in the alkali treated SZF/SF/VE hybrid composites is taken by non-Fickian diffusion law

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