Animal fibre characterisation and fibre loading effect on mechanical behaviours of sheep wool fibre reinforced polyester composites

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Abstract

The quest to design and develop environmentally friendly, sustainable and low-cost materials has tremendously increased attention on natural fibre reinforced polymer (FRP)/bio-composites more than synthetic or conventional counterparts. Therefore, this study presented animal fibre characterisation and influence of various fibre loadings on mechanical properties of sheep wool fibre reinforced polymer (SWFRP) composites. The sheep wool fibres (SWF) characterisation was carried out using X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), scanning electron microscopy (SEM) and non-contact surface roughness machine. The functional group and chemical bond of the SWF was analysed using FTIR techniques. The crystallinity index and thermal stability of the SWF were characterised, using XRD and TGA techniques, respectively. Furthermore, the SWFRP composites were fabricated using compression moulding technique and a varying weight percentages of 20, 30 and 40 wt%. The composite plates were cut into test samples according to ASTM standard methods for their mechanical (tensile, flexural and impact) behaviours to be extensively analysed. The surface morphology of the fractured samples were examined with aid of a SEM. From the result obtained, it was evident that the SWF exhibited crystallinity index of 30%, functional groups of amide-I and amide-III, thermal stability up to 230 ^{o}C and average surface roughness of 0.716 μm. The SWFRP composite recorded a significant increased tensile strength properties when fibre loading was increased from 20 to 40 wt%. The optimum 40 wt% SWFRP composite sample recorded better flexural and impact strength, when compared with other counterparts. This was attributed to a better fibre-matrix interfacial adhesion, as established from its SEM micrographs. Summarily, the SWF are suitable reinforcements for polymer composites and consequently, the SWFRP composites are potential environmentally friendly, sustainable and low-cost materials for engineering applications.

Keywords: Sheep wool fibres (SWF), characterisation, XRD, FTIR, TGA, mechanical properties, microscope analysis.

1. Introduction

Polymer is one of the important materials in engineering application, due to the low density, ease of manufacturing at a low cost. Different types of polymers and their composites are developed to obtain good mechanical and physical properties. These composites are effective materials for industrial applications [1]. Composites fabricated from synthetic fibres possess excellent mechanical properties and they are relevant in both medium and high load applications, whereas natural fibre reinforced polymer (FRP) composites are good for low load bearing applications. Another advantage of natural FRP composites is that they are excellent biodegradable materials [2]. Research community have made numerous successful attempts to use a variety of natural fibres, such as jute, banana, sisal, coir, kenaf, Palmyra and hemp for fabricating several bio-composites for low load carrying applications [3]. Jowar, sisal and bamboo fibres reinforced polyester composites have been fabricated and their modulus properties were compared [4]. It was observed that jowar reinforced polyester composite was superior to the other two. The waste rubber material has been used as a reinforcement material for making polyester and epoxy resin composites. It was reported that an addition of waste rubber altered the density and thermal conductivity properties [5]. The water absorption influenced the tensile properties of the composites made by waste newspaper reinforced polyester resin [6]. Rice straw fibre has been reinforced with polyester resin. Both tensile and impact properties of the pure polyester resin and reinforced composites were tested. It was observed that addition of rice straw fibre provided 1.6 and 18 times better tensile and impact properties [7]. Unidirectional composites have been prepared from sansevieria natural fibre reinforced polyester resin, through conventional method. A better tensile and impact resistances were recorded [8]. Composites have been fabricated by randomly placed coir fibre in polyester resin. From the results obtained, better flexural properties than the pure polyester were recorded [9]. The fibre length influenced the physical and mechanical properties of the composites made with banana fibre and polyester resin. The composites possessed a lower strength than the pure polyester [10]. Similarly, the weight percentage of short fibre influenced the dynamic mechanical behaviours of the composites made with banana fibre and polyester resin [11]. The characteristics of mechanical and moisture absorption have been studied on natural FRP composite. A 50 wt% of sisal fibre was added to epoxy composite reinforced with banana fibre. It showed an increase in mechanical and decreased in water absorption properties [12-14]. Manufacturing of composite using hand lay-up technique is one of the easiest way to produce the polymer composites. Many of the researchers focused on this method to fabricate different kinds of polymeric composites, using various natural fibres and resins, as reinforcement and the matrix materials, respectively [15].

Furthermore, there are scarce attempts whereby animal fibres were used as reinforcement in natural FRP composites. For instance, animal fibre was used to produce composite materials for soundproof applications in buildings [16]. To protect the environment from pollutions and to increase the value of fibre, animal fibre reinforced polymeric composites have been fabricated and studied [17-19]. Wool is a natural fibre obtained from the sheep, because of having some excellent properties, such as a very good insulation and low flammability [20]. It was reported that the sheep wool fibres (SWF) reinforced polymer composites showed very good insulating properties, when compared with other natural fibres [21]. Wool fibre produced minimum impact to the environment. Therefore, this study produced wool fibre into useful products that are more eco-friendly to the nature and environment [22].

Epoxy resin has been reinforced with SWF, tensile and flexural properties of the composites were tested. It was observed through the scanning electron microscope (SEM) that there was breakage of fibres, mainly because of uneven distribution of fibres which produced a poor adhesion bonding between fibre and the matrix. It was further reported that adding higher weight percentage of fibre produced a decrease in the tensile strength [23]. Xin et al. [24] investigated the flexural properties of the composites which were prepared by combining wool fibre and polyester resin. It was reported that uneven distribution of fibres led to fibre pull-out from the matrix, permanent deformation and breakage of fibres as well as appearance of dendrite-like structure. Fourier transform-infrared spectroscopy (FTIR) spectra of the *Sansevieria cylindrica* fibres reinforced polymer composites has been reported [25]. The fibre bands of 1510 and 1612 cm^{-1} indicated lignin and hemicelluloses of C=O bonds. The X-ray diffraction (XRD) reports indicated that the two peaks were identified at 2θ = 15.4° and 22.5°, which reflected the presence of cellulose I and IV. The thermo-gravimetric analysis (TGA) of kenaf and rice husk FRP composites has been reported [26]. The kenaf fibre composite recorded a higher thermal stability than the rice husk fibre counterpart, due to higher cellulose content in the kenaf when compared with the rice husk fibre.

The objective of this present work on fabrication of bio-composites via compression moulding technique, using SWF and polyester resin as reinforcement and matrix materials, respectively. Besides, the mechanical properties of the fabricated composites with different fibre loadings (wt%) were studied, in addition to their characterisation using XRD, FTIR, TGA and SEM.

2. Materials and methods

An attempt was made to fabricate the sheep wool fibre reinforced polymer (SWFRP) composites by using polyester resin reinforced with SWF. The mechanical behaviours of the fabricated composite samples were studied.

2.1. Materials

Indian SWF and isophthalic unsaturated polyester resin were used as reinforcement and matrix materials, respectively. The sheep wool was collected from local areas (Fig. 1), in/around Virudhunagar region, India. Dust in the reinforcement material was washed with water and dried in a room temperature to remove some moisture present. It was cut into small pieces of equal measurement of average length of nearly 20 mm. The polyester resin and its additional ingredients: catalyst (MEKP) and accelerator (CN) were used to fabricate the SWFRP composites. The matrix material and other chemicals were purchased from M/s VB Pvt. Ltd, Madras, TN, India.

Fig. 1. SWF used as a reinforcement.

2.2. Fabrication of composites

The compression moulding technique was employed to prepare the composite plates. Initially, the steel mould of 300 x 125 x 3 mm was cleaned manually, using cloth material. Wax was applied on the interior face of the mould to easily remove the fabricated composites without any damage. The SWF length of 20 mm were randomly arranged in the mould cavity. The polyester resin, 2% MEKP and 2% CN as catalyst and accelerator, respectively were stirred properly in a glass beaker with manual stirrer and poured over the fibres in the mould cavity. Then, the mould was closed manually and compressed by using a universal testing machine. The compressed mould was kept in a universal testing machine for the next 24 hours at a constant load and room temperature. The prepared composites were sized into appropriate dimensions required or recommended to study the mechanical properties in accordance to the ASTM standards [28]. Similarly, the SWFRP composites of three different fibre loadings/wt% of 20, 30 and 40 wt% were prepared by combining the SWF and the polyester resin, using the same procedures. The fabricated composite plate is shown in Fig. 2.

Fig. 2. Fabricated SWFRP composite plate.

2.3. SWFRP composite sample characterisation

2.3.1. X-ray diffraction

X-ray spectrum of the SWF was observed to understand the crystallinity nature of the SWF. The peaks in the spectrum of sheep wood fibre (SWF) were studied, using Bruker XRD SSD160 with Cu-Kα monochromatic radiation at an angle of incidence of 5 °/min in 2θ =10°- 80° (2θ range), where θ represents the angle of diffraction of X-rays within a crystalline material, known as Bragg angle [27].

2.3.2. Fourier transform-infrared spectroscopy

The FTIR analysis was performed to study the functional group modifications on the surface of SWF, using PES RXI FTIR spectrometer in the range of 400 to 4000 cm^{-1} at the rate of 32 scans/min and 2 cm^{-1}, as a resolution [27].

2.3.3. Thermo-gravimetric analysis

The thermal stability of the SWF was carried out in a thermo-gravimetric analyser. The initial readings were captured using JS thermal analyser STA 449 F3 in the range 28 – 600 °C at the heating rate of 10 °C/min in Ni atmosphere.

2.3.4. Surface roughness

The surface roughness of the SWF was measured using a 3D non-contact profiler (Talfsurf CCI MP, UK). Three samples were measured in three different locations on each sample and the measured average values are recorded to support repeatability, reliability and accuracy. The average mean values of the three different locations of all the three samples were reported. Surface roughness was measured for a minimal fibre length of 50 mm and it was carried out along the length of the fibre.

2.3.5. Morphological structure and diameter of the SWF

The surface morphology of the SWF was studied, using JEOL M-6390 SEM and the diameter of SWF was measured in a particular position with suitable magnification and the SWFRP composite samples were examined at different magnifications.

3. Mechanical Testing

3.1. Tensile test

The tensile test for the randomly oriented SWFRP composites was done in accordance with ASTM D638 standard, using a universal testing machine with a maximum load of 400 kN [29-30]. The tensile test rectangular samples with dimension of 200 x 20 x 3 mm (Fig. 3) were cut from the composite plates, using a reciprocating blade cutter. The SWFRP composites of three different fibre contents of 20, 30 and 40 wt% were tested. In each wt% composition, three identical samples were selected to carry out the test and the average value was recorded.

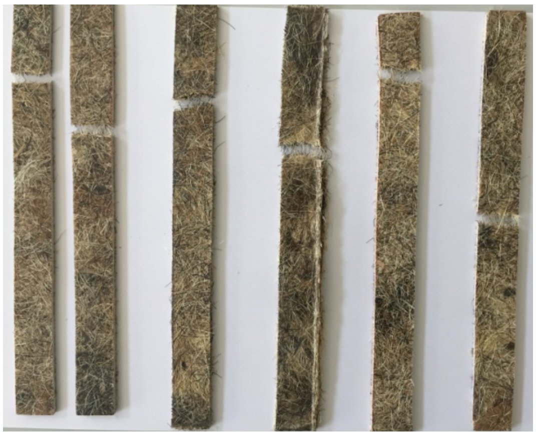


Fig. 3. SWFRP composite samples after tensile test.

3.2 Flexural test

The flexural responses of all the SWFRP composite samples with varied fibre loadings were obtained according to the ASTM D790 standard [29-30]. The dimension of the sample was 127 x 13 x 3 mm (Fig. 4). The flexural test was conducted on a universal testing machine, using three-point bending method. In each wt% composition, three identical samples were selected to carry out the test and the average value was recorded.

Fig. 4. SWFRP composite samples after flexural test.

3.3. Impact test

Impact test of an un-notched composite samples with a dimension of 65 x 13 x 3 mm was performed according to the ASTM D256 standard [30]. All the SWFRP composites with different fibre loadings were tested. In each wt% composition, three identical samples were selected to carry out the test and the average value was obtained. The flexural fractured samples are shown in Fig. 5.

Fig. 5. SWFRP composite samples after impact test.

4. Results and discussion

4.1. X-ray diffraction

The X-ray diffractogram of the SWF is shown in Fig.6. The XRD peaks intensities values were recorded from 9° to 80° of counter angle. The first peak was observed at 2θ = 9.8° and second peak value was observed at 2θ = 21.36°. The first and second peaks were specifically corresponding to the planes of [002] and [004], respectively. A minor peak was observed at 2θ = 16.46°. The crystallinity index value of the SWF was calculated, using Eq.1 [31-32].

$$\mathrm{CI} = \frac{I_{9.8^o} - I_{16.46^o}}{I_{9.8^o}} x\ 100 \quad (1)$$

Where $I_{9.8°}$ denoted major peak intensity of the SWF and $I_{16.46°}$ indicated that the minor peak intensity of the SWF. The two peaks indicated the presence of protein (keratin) in the SWF [32]. The CI value of the SWF was measured at 30.21%. The CI value of the SWF was greater than that of the chicken feather barb with CI at 24.8% [33] and peacock feather barb fibre has CI at 15.6% [34].

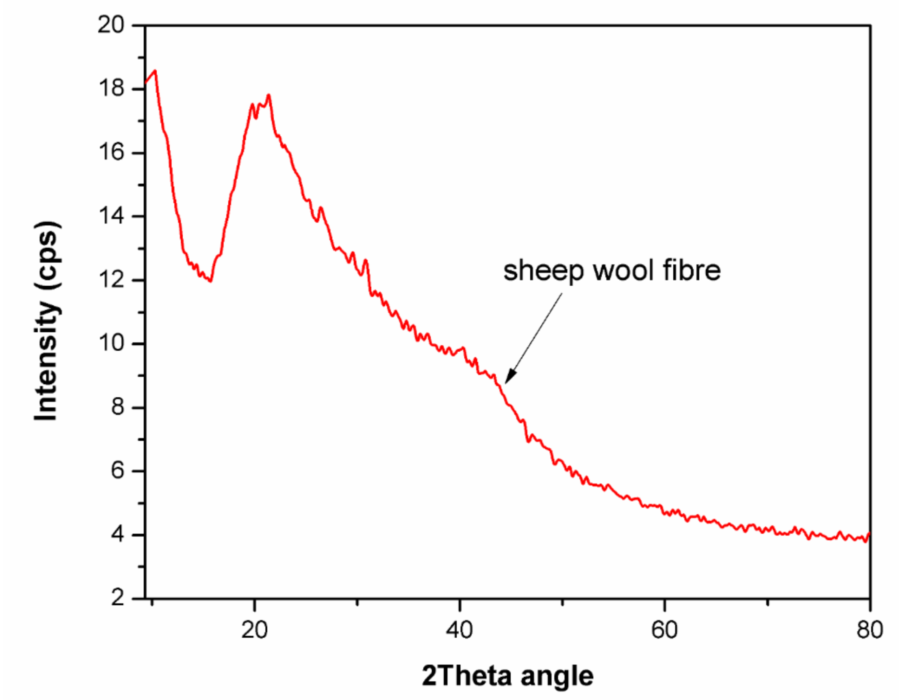


Fig. 6. X-ray Diffraction curve of the SWF used.

4.2. Fourier transform-infrared spectroscopy

The functional groups of chemical composition of the SWF was characterised, using FTIR analysis technique. Fig.7 shows the FTIR spectrum of the SWF. From Fig. 7, it was observed that the major sharp peaks were observed at 825.53, 1035.77, 1180.44, 1232.51, 1506.41, 1606 and 2962.66 cm^{-1}. The peak at 1606 cm^{-1} can be attributed to amide-I band with C=O stretching vibration and it corresponds to C=O band elastic vibration [34-36]. The peak at 1232.51 cm^{-1} indicated the amide-III band with C-N stretching more over the CNH group, as indicated at this peak with N-H plane bending. The peak at 1180 cm^{-1} can be traced to presence of (S=O) cysteic acid and the observed peak at 1525 cm^{-1} can be attributed to the C-N-H band of bending deformation. The secondary amide of N-H bending and C-N wagging were observed. The peak at 825 cm^{-1} was due to the presence of C-S stretching vibration and the peak at 2962.66 cm^{-1} can be ascribed to C-H band vibration [34,37,38].

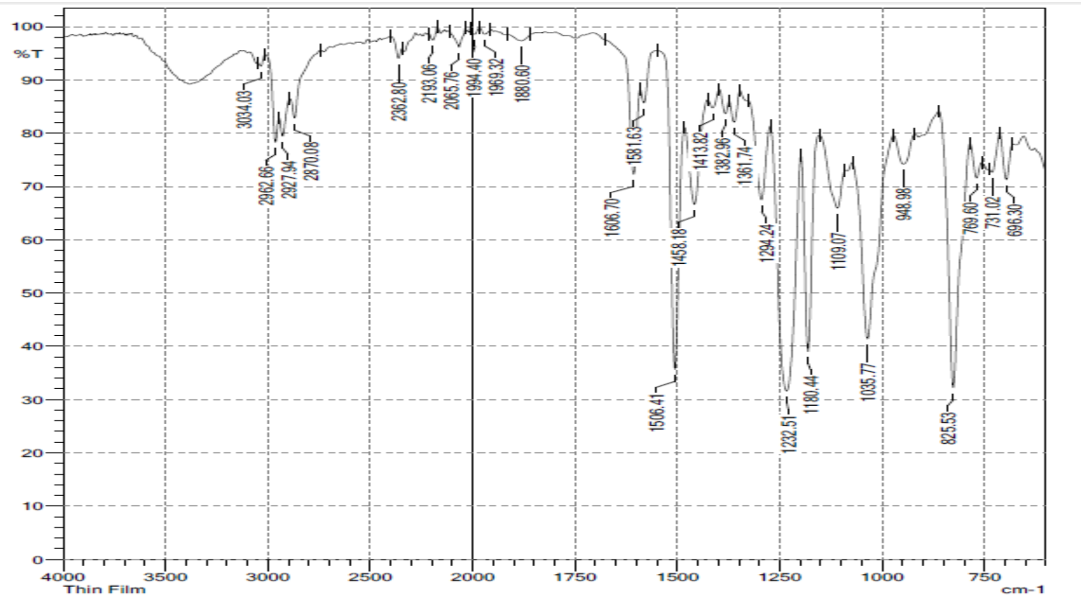


Fig. 7. FTIR curve of the SWF used.

4.3. Thermo-gravimetric analysis

The thermal stability of the SWF was analysed, using TGA, as depicted in Fig. 8(a). It was observed that three stages of mass loss have occurred in the SWF during heating. The first stage of mass loss occurred due to evaporation of water molecules from the fibre, and it took place when the temperature ranged from 30 to 160 °C. At this stage, SWF has exhibited 18% of mass loss [34,39]. Similar to the feather fibre, there were three types of water molecules: free water, chemically bonded and loosely bonded water. The second stage of mass loss occurred when the temperature ranged from 230 to 360 °C. The main reason for having this mass loss was due to the degradation of chain molecules of it protein (keratin). Almost 70% of mass loss was recorded at this stage. The last degradation of the SWF was observed from 350 to 520 °C. At this stage, the exothermic reaction started. Fig. 8(b) shows the derivative thermogram of the SWF. It depicts that the first inflection point was created at 74.2 °C. At this temperature, the maximum degradation has occurred. The main inflection point appeared at 314.2 °C and this stage recorded the maximum degradation of the fibre. From the TGA study, it was observed that the SWF was thermally stable up to 230 °C [34,40,41], before huge degradation began.

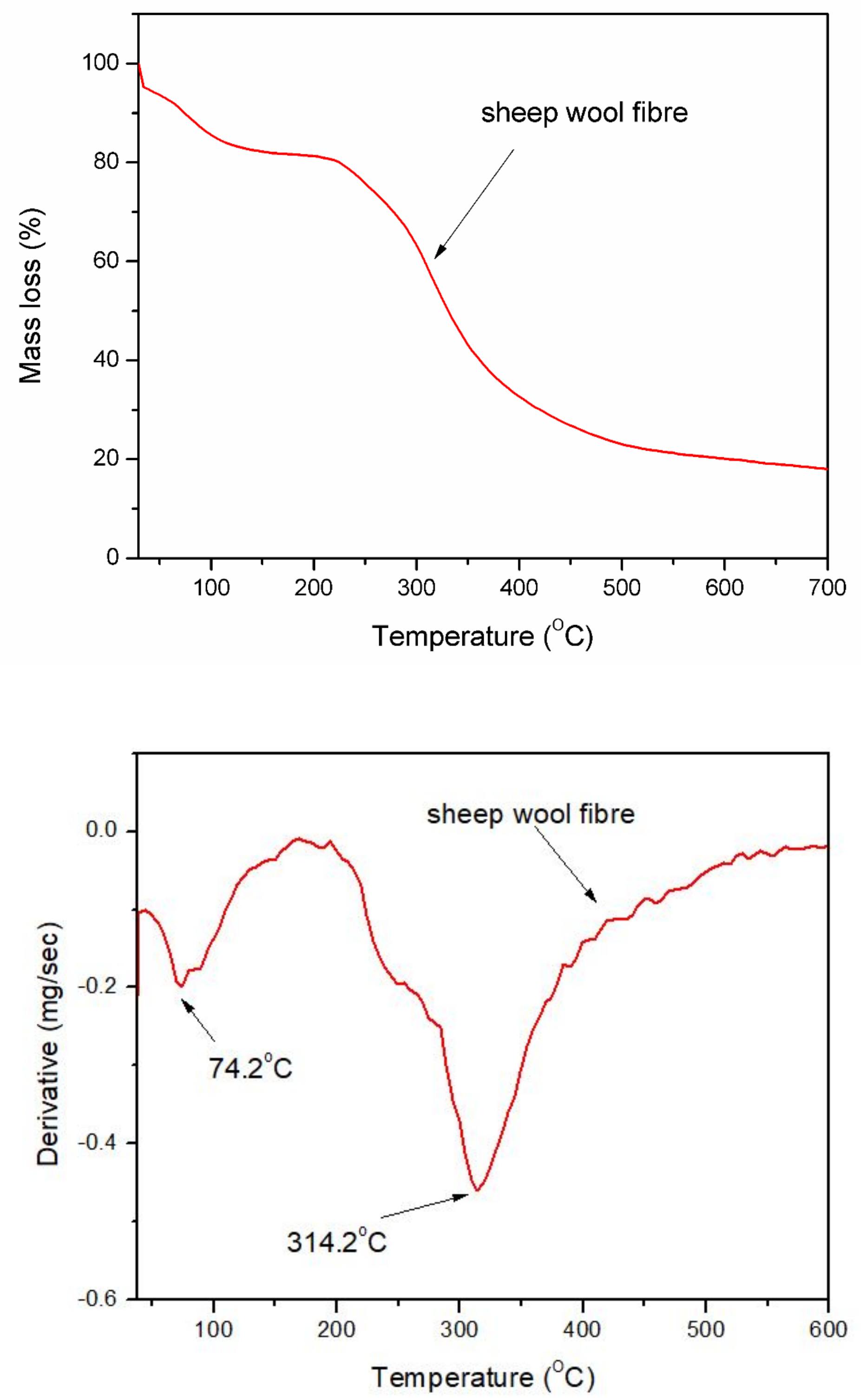


Fig. 8. (a) TGA curve and (b) derivative thermogram of the SWF used.

4.4. Surface roughness

The roughness of SWF surfaces is directly related to the fibre-matrix interfacial bonding. The surface texture of the SWF is shown in Fig. 9. Fig. 9(a) and (b) show the 3D surface texture and 2D plane in XY direction with surface value in the line diagram of the SWF, respectively. In addition, Fig. 9(b) depicts the positive and negative values of surface roughness of the SWF. It was observed that SWF possessed more uneven surfaces present on their outer layers. The 3D images display that rough surfaces occurred along the SWF length. The rough fibre surfaces are mostly preferred during the fabrication of polymer composites, because they enhance mechanical bonding with polymer resin to the extent that the fibres will not easily pull out from the polymer matrix. The average surface roughness (R_a) value of the SWF was 0.716 µm. Therefore, the higher surface roughness indicated more contaminants on the fibre surfaces.

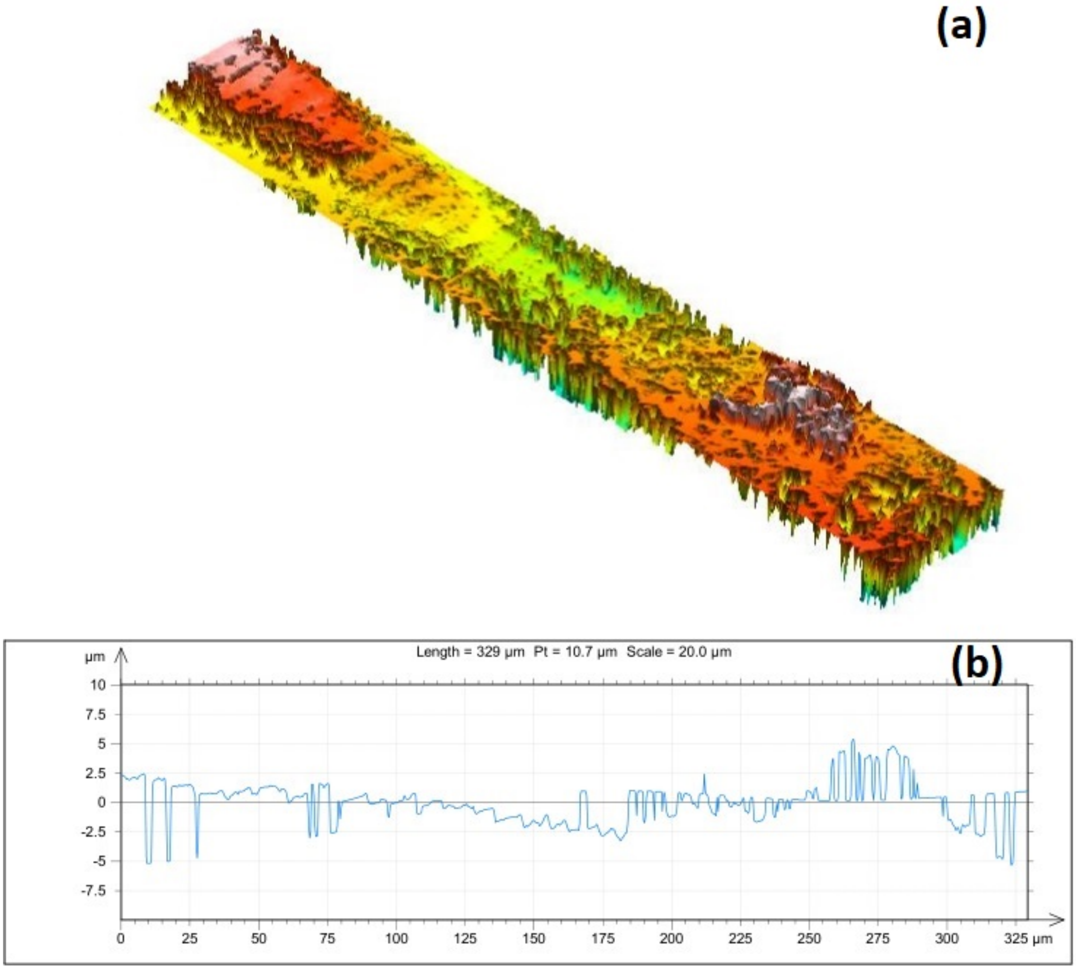


Fig. 9. Surface roughness of the SWF used, showing its (a) 3D surface texture and (b) 2D XY plane line diagram of surface roughness measurement.

4.5 SEM of the SWF surface morphology

The surface topography of the SWF was examined and analysed, using SEM technique. Fig. 10 shows the SEM image of a longitudinal view of the SWF. It depicts that the fibre has more uneven rough surfaces on its outer surfaces and also observed that some impurities and more wave lines occurred around the fibre outer area. This study observed that the irregular surface texture fibre provided a better interfacial adhesion with the matrix within a polymer composite system. The diameter of the SWF was measured using longitudinal fibre SEM images. The linear measurement of the SWF diameters was recorded for five images, before an average value of 0.161 mm was obtained.

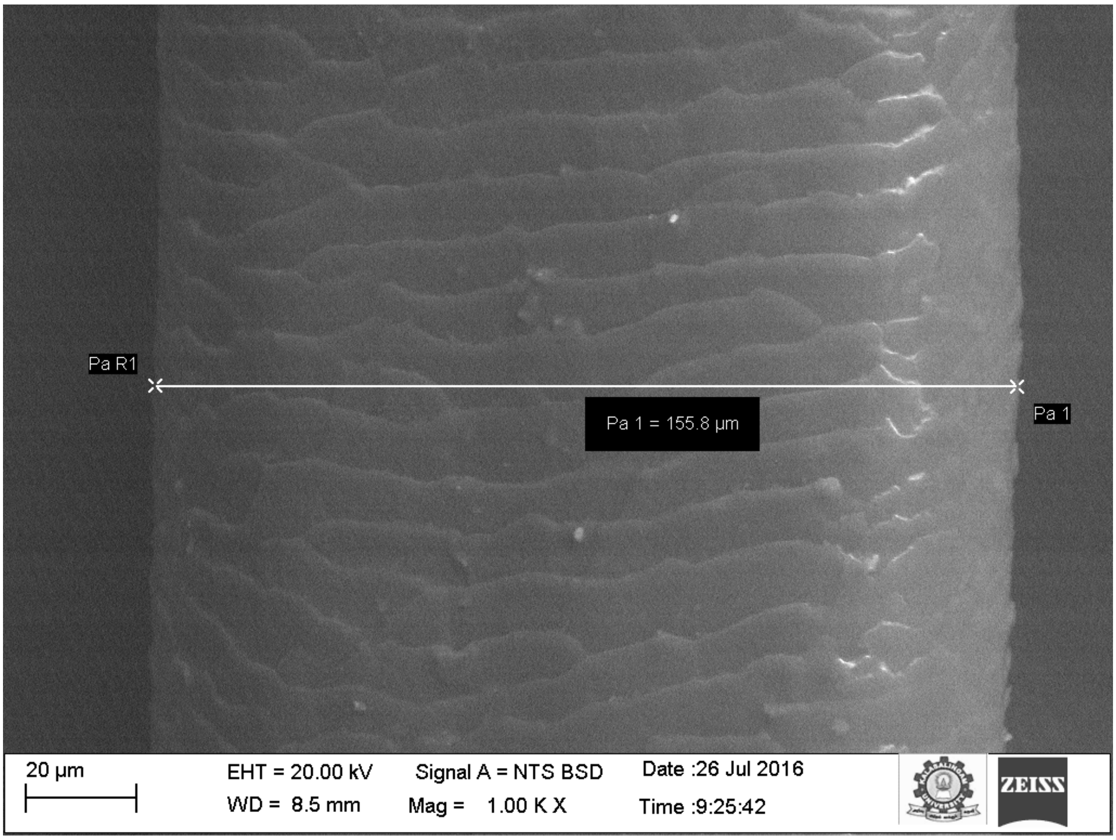
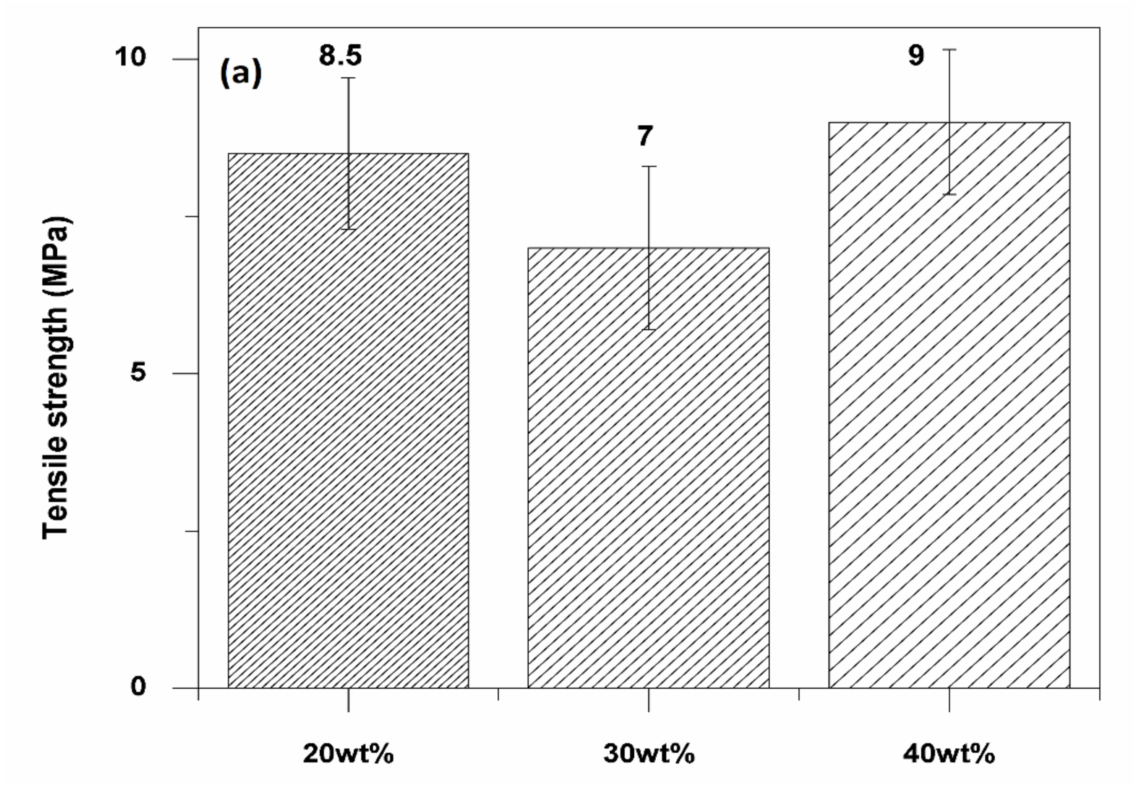


Fig. 10. Surface morphology of the SWF used.

4.6. Mechanical properties of the SWFRP composites

4.6.1. Tensile strengths

The tensile experiment was conducted for 20, 30 and 40 wt% SWFRP composite samples. Five samples were tested for each category and their average values were reported. The tensile strengths of the 20, 30 and 40 wt% SWFRP composites were obtained at 8.5, 7.0 and 9.0 MPa and their corresponding tensile moduli were measured at 1.55, 1.43 and 1.34 GPa, respectively. From Fig. 11(a), it was observed that the highest tensile strength was obtained with 40 wt% SWFRP composites. This can be attributed to a better interfacial bonding between the 40 wt% SWF and the polyester polymer matrix with best load transfer from the matrix to fibres. However, the tensile strengths of the SWFRP composites were lower than that of the pure resin. The pure resin recorded tensile strength of 18.88 MPa. Moving forward, the lowest tensile strength was obtained from the 30 wt% SWFRP composite. The reason behind its lower strength could be presence of a poor fibre-matrix interfacial bonding and many micro-voids. The tensile moduli of the SWFRP composites were gradually decreased from a lower fibre content to higher wt% SWFRP composites, as depicted in Fig. 11(b). This trend was observed due to the strain variation of the SWFRP composites. Strain is directly related to the tensile modulus of a material. The strain values of the SWFRP composites were measured at 1.91, 2.08 and 2.55% for 20, 30 and 40 wt% SWFRP composites, respectively. The nature of the fracture was ductile, due to higher elongation of the SWFRP composite materials.



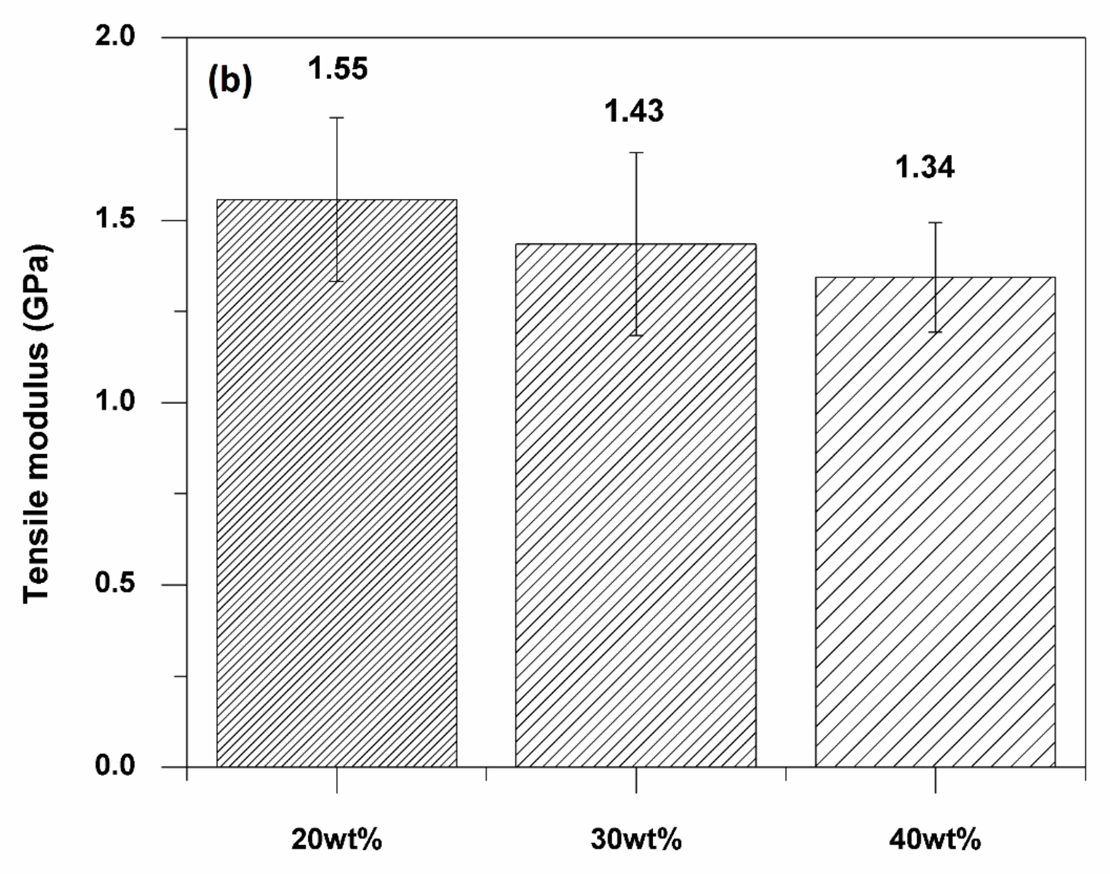


Fig. 11. Tensile properties of the various fibre loaded SWFRP composite samples, showing their tensile (a) strengths and (b) moduli.

4.6.2. Flexural strengths

The flexural strengths of all the SWFRP composites are shown in Fig. 12. It was observed that the 40 wt% SWFRP composite exhibited the highest flexural strength of 25 MPa, while the 30 wt% SWFRP composite recorded the lowest value of 20 MPa. When compared with the resin, the flexural strengths of SWFRP composites were lower. The pure resin has flexural strength of 29.45 MPa. This can be attributed to the poor dispersion of fibres and poor strength of the single fibre. The single fibre strength also plays a major role in the mechanical properties of the SWFRP composites.

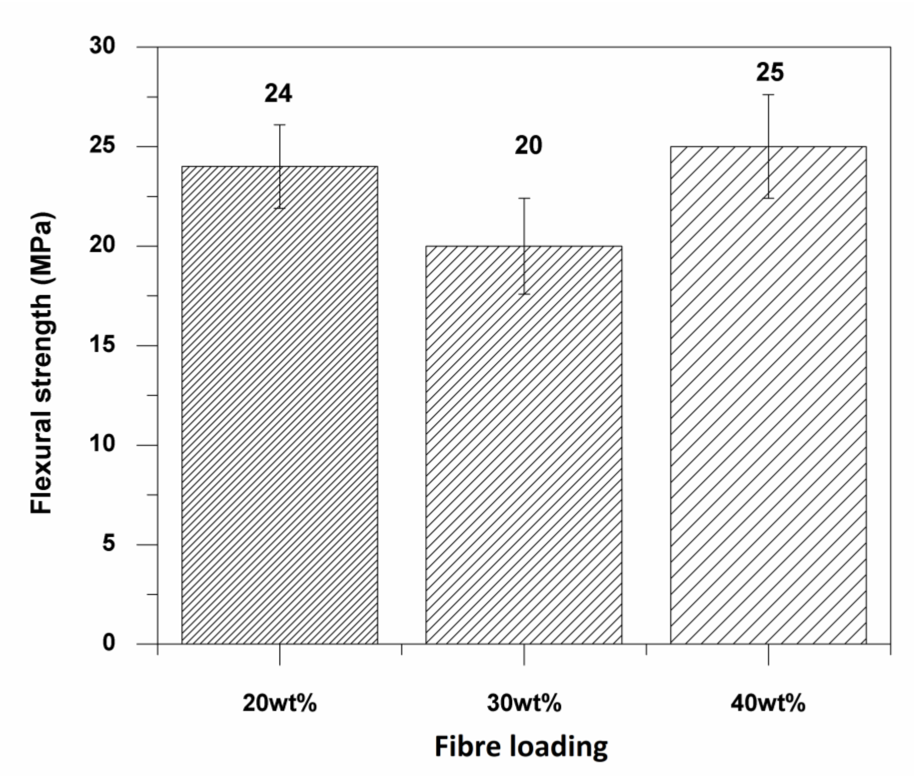


Fig. 12. Flexural strength of the various fibre loaded SWFRP composite samples.

4.6.3. Impact strengths

The impact behaviours of all the SWFRP composites are shown in Fig 13. The impact strengths of the SWFRP composites increased when fibre loading was increased from 20 to 40 wt% in the SWFRP composite system. The main reason behind this exhibition can be attributed to more energy absorption nature in a large quantity or fibre content based composites. Similar trend has been reported [42], where human hair fibres were used as a reinforcement and obtained an increased trend of impact strength with an increased fibre loading from 3 to 5 wt%.

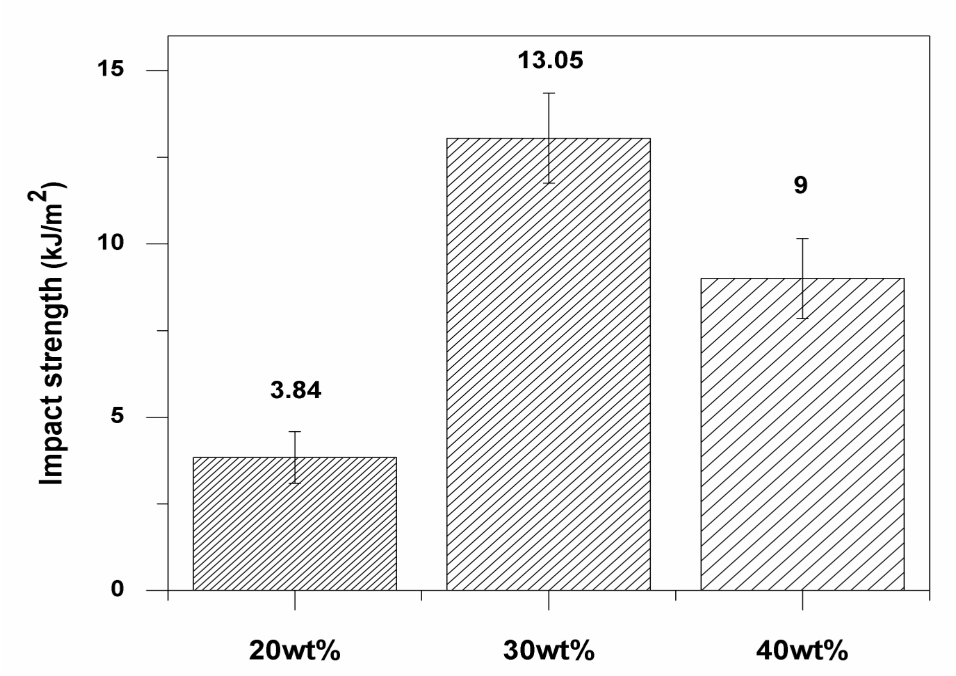


Fig. 13. Impact strength of various fibre loaded SWFRP composite samples.

4.7. Morphological analysis of the SWFRP composites

The tensile fractured samples were considered for damage analysis and morphological characterisation of the SWFRP composites. Figs. 14(a)-(c) show the SEM images of 20, 30 and 40 wt% SWFRP composite samples, respectively. Fig. 14(a) evidently shows more resin-rich area, due to less amount of fibre weight percentage. Also, it shows some voids and fibre pull-out were present on the 20 wt% SWFRP composite sample. Moreover, matrix crack formed in the 20 wt% SWFRP composite sample. Fig. 14(b) shows that the orientation of fibre was random and more numbers of micro-voids and some fibre pull out were present. Presence of voids reduced the mechanical strength of the polymer composite. This is the main reason behind the record of a lower tensile strength with 30 wt% SWFRP composite sample. The poor interfacial bonding supported fibre pull out during tensile test. The magnitude of the composite tensile strength decreased, due to occurrence of more fibre pull-out damage. Fig. 14(c) shows that more fibres were loaded and accumulated in the 40 wt% SWFRP composite sample. Consequently, more numbers of loosely separated fibres were visible. By comparing with both Figs. 14(a) and (b), Fig. 14(c) depicts a lower void content within the 40 wt% composites, thus an enhanced strength was obtained. More also, Fig. 14(c) shows a fibre-to-fibre contact and a strong SWF-polyester interfacial adhesion. The strong interfacial bonding exhibited a better load transfer from matrix to more fibres, during loading conditions.

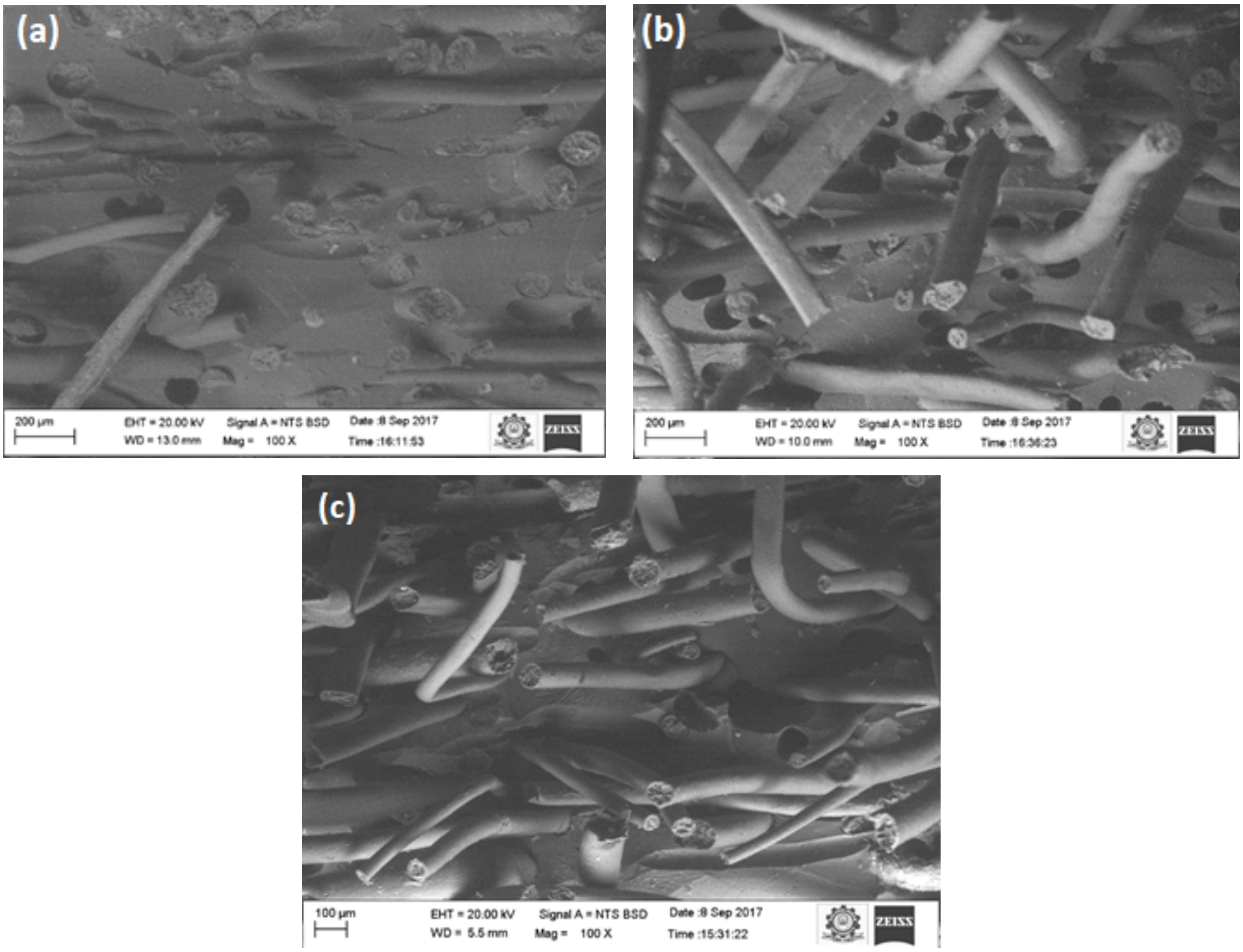


Fig. 14. SEM images of the tensile fractured surfaces of (a) 20, (b) 30 and (c) 40 wt% SWFRP composite samples.

5. Conclusions

The following conclusions were highlighted based on the characterisation of the SWF and mechanical performances of the various fibre loaded SWFRP composite samples.

- The crystallinity index of the SWF was observed around 30% and the functional groups of amide-I and amide-III were observed in the SWF.
- The thermal stability of the SWF has three stages of degradation with temperature. The SWF has thermal stability up to 230 °C, before a significant mass loss occurred.
- The SWF surface morphologic images confirmed that the fibre has more uneven rough surfaces along the length of fibre, with an average value of 0.716 µm. Therefore, based on this study, SWF are suitable reinforcements for polymer composites.

- The SWFRP composites were successfully fabricated, using compression moulding and the SWFRP composite samples were subjected to various mechanical tests. The tensile and flexural strengths of the SWFRP composites were comparatively lower than the pure polyester resin. However, the tensile properties of the SWFRP composites were increased when the fibre loading was increased from 20 to 40 wt%. The highest tensile strain was observed from the 20 wt% SWFRP composite sample and the lowest tensile strain was recorded by the 40 wt% SWFRP composite.
- The higher flexural and impact strengths of the 40 wt% SWFRP composite sample were obtained, when compared with other two counterparts. Therefore, the optimum mechanical properties were obtained with 40 wt% SWFRP composite sample, due to a better fibre-matrix interfacial adhesion, as confirmed from its micrographs. However, the SEM images of the fractured samples confirmed that the fabricated SWFRP composite samples possessed some voids.

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