The Tensile and Flexural Properties of Treated and Untreated Sisal Fibre-Epoxy Resin Composites

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Abstract-- The influence of chemical treatment of sisal fibres through the subsequent processes of mercerisation (alkali treatment), then silane treatment and eventually acid hydrolysis on the fibres were investigated. The effect of the treated fibres on the tensile and flexural properties of their composites with epoxy resin were also studied. Scanning electron microscopy examination of the treated and untreated fibres showed that the subsequent processes of chemical treatment enhanced the removal of surface impurities and therefore increased the roughness of the fibre surfaces. It was concluded that this would avail an increased area on the fibre for interlocking with matrices and would therefore enhance adhesion of the two. Consistent to this conclusion, subsequent testing of treated fibre reinforced composites gave rise to higher values of tensile and flexural strength and stiffness than the untreated fibre reinforced composites.

Index Term-- mercerisation, silane, sisal, tensile, flexural, acid hydrolysis, sisal-epoxy composites

1.0 INTRODUCTION

Natural fibre composites are emerging as realistic alternatives to replace glass reinforced composites in many applications [1, 2]. Natural fibres such as sisal, hemp, kenaf, flax, jute and banana are usually combined with a plastic polymer to form a composite [3]. Further, growing environmental awareness has also triggered a paradigm shift towards designing materials compatible with the environment [4]. Because of increasing environmental consciousness and legislated requirements, the replacement of the traditional composite structure usually made of carbon, glass or aramid fibres is becoming important [5]. Composites derived from natural fibres now maintain a balance between economics and the environment, thus allowing them to be considered for applications in many industrial fields like automotive, electronics, biomedicine, cosmetics and the packaging industry [6].

Apart from low cost and acceptable values of specific strength, the other advantages of natural fibres include sequestration of carbon dioxide, biodegradability and low density [7]. Natural fibres serve as reinforcement by enhancing the strength and stiffness of matrices and by further reducing the weight of the resulting composite structure. The use of natural fibres typically reduces the weight of the resulting composite by 10% and lowers the energy needed for their production by 80%, while the cost of the composite components is 5% lower than the comparable synthetic fibre reinforced component [3]. The properties of natural fibres vary with their source and treatment. Mechanical properties depend on whether the fibres are taken from the plant stem or leaf, the soil and climate of the plant location, the age of the plant and the extraction process (retting) adopted to collect the fibre from the plant [8].

Natural fibres are hydrophilic while the matrices are usually hydrophobic in nature. The surfaces of natural fibres, therefore, need to be modified for them to be compatible with the hydrophobic matrices [5, 9, 10, 11, 12]. Whilst, filler materials facilitate the enhancement of the mechanical properties of matrices before reinforcement with fibres [13], they are more important for the purposes of increasing the bulk and, therefore, handling of the resulting composites [14]. Generally, the tensile and flexural properties of composites are markedly improved by adding fibres to a polymer matrix since fibres have much higher values of strength and stiffness than those of the matrices [13]. In general, the higher the content of the reinforcing fibre, the higher the performance of the resulting composites [14]. Therefore, the effect of fibre content on the tensile and flexural properties of fibre reinforced composites is of particular interest and significance for many researchers [15].

The vacuum infusion method of fabricating composites has been adopted in this work in order to take advantage of its benefits of reducing porosity in the resulting composite and also to ensure consistency in the composite manufactured. The method also supports proper wetting of the reinforcing fibre, which gives rise to better and stronger fibre/matrix interfacial bonding and the attendant improvement in the mechanical properties of the fabricated composites [15].

Sisal fibres were selected for use in this work due to their easy availability. Sisal is a tropical plant that is fibrous in nature, besides being reasonably strong compared to other natural fibres. When embedded in a matrix, the resulting composites have a wide variety of applications such as in the motor vehicle industry. The main advantages of sisal fibre for reinforcement include: reasonable strength compared to most synthetic fibre composites, lower cost, ease of cutting, light weight, abundant availability and environmental benefits [16].
In this paper, the effect of chemical treatment on sisal fibres is investigated. The effect of this treatment on the tensile and flexural properties of their composites with epoxy resin is also looked into.

2.0 EXPERIMENTAL DETAILS

2.1 Materials
Epoxy Epolam 2015 resin and Epolam 2014 hardener were obtained from AMT composites of South Africa. The mix ratio of the resin and the hardener according to the data sheet obtained from the company was 100:32. Sisal fibres in a bundle of 10kgs were obtained from Mogotio farm, Nakuru County, Republic of Kenya. The reagents which were used in this work were: sodium hydroxide, 3-glycidyloxypropyltrimethoxy-silane, methanol and hydrochloric acid (HCL). Sodium hydroxide was provided by Minema chemicals (Pty) limited while 3-glycidyloxypropyltrimethoxy-silane, methanol and hydrochloric acid were provided by Sigma Aldrich limited, all of South Africa.

2.2 Preparation of Treated Sisal Fibres
The sisal fibres were mercerised by immersion in 5% sodium hydroxide solution for 20 hours. The fibres were then washed with distilled water in order to remove the sodium hydroxide from them and then further immersed in 1% acetic acid in order to neutralise any remaining sodium hydroxide. This was followed by silane treatment in which the silane radical group was attached onto the sisal fibres. This involved immersion of the alkali treated fibres in a silane solution made up of 5% of 3 glycidyloxypropyltrimethoxy-silane diluted in a 95% aqueous solution of methanol, the later in order to hydrolyse the silane and make it active [17]. This treatment was then followed by the immersion of the fibres into 67.5% solution of hydrochloric acid for 1 hour [20]. Following onto this, the treated fibres were washed with deionized water and dried in an oven at 45°C for 24 hours.

2.3 Manufacture of Composites
The composites were manufactured using the vacuum infusion method using the equipment shown in Figure 1.

The vacuum infusion system consists of the vacuum pump, resin trap and air-tight clamping devices. This method of composite manufacture supports proper wetting of the reinforcing fibres, which gives rise to better and stronger interfacial bonding of the fibre and the matrix. Both treated and untreated sisal fibre-epoxy resin composites were manufactured using this method.

The sisal fibres were cut using a pair of scissors and straightened using a comb in order to avoid bunching of fibres which would otherwise minimise the wetting of the fibres with resin and, therefore, reduces the efficacy of reinforcement. These cut fibres were then weighed using an electronic balance with an accuracy of ± 0.5 gm and grouped into various masses corresponding to different fibre weight fractions. A thin layer of wax was smeared onto the base of a square glass mould of 50 cm by 50 cm. The wax ensures that the composite can be easily removed from the mould box after curing. Pre-determined weights of sisal fibres were placed in the glass mould with their longitudinal direction aligned to one another. The fibres were then covered with peel ply and infusion mesh simultaneously. The
peel ply was made of polyester material and is used to wick away slight excesses of resin. The infusion mesh was made of a plastic material and it aids the resin to efficiently flow throughout the fibres. The bleeder cloth was then laid near the tube exiting to the vacuum so that excess resin could be sucked through the tube to the resin trap. The spiral tubing were then both connected, one to the tube from the resin beaker and one to the vacuum. Thereafter, a vacuum bag was used to cover the entire casting. A tacky tape was then used to secure the vacuum bag onto the mould. The vacuum pump motor was switched on and the tube leading to the resin storage container temporarily closed off using a G-clamp in order to avoid suction of air into the fibres before creating a vacuum. A break of one hour was allowed with the vacuum pump running as the resin was being prepared. The resin and the hardener were then measured in their appropriate ratios using an electronic balance of accuracy ±0.5 gm and subsequently mixed using a spatula. Air entrapment in the resin was eliminated through puncturing of air bubbles with a sharp needle. After the break, the resin suction pipe was placed into the resin container and the closing G-clamp on the pipe removed in order to allow suction of the resin onto the fibres. The sisal fibre composites were subsequently cured in air for 24 hours. After this air curing, the composites were further cured in an oven at 80ºC for four hours as recommended by the supplier of the resin in order to produce composites with excellent mechanical properties. The reinforcement was varied from 0 to 15 wt % in the different composites moulded. A casting of pure epoxy resin was also done to provide baseline properties for the properties of the composites.

2.4 Tensile Testing

The tensile strength and stiffness were determined according to ASTM D 3039 test standard specification. The test specimens were cut from both the cast composites and the epoxy resin using a 3000 series CNC router machine. The specimens were cut to dimensions of 250 mm length, 25 mm width and 3 mm thickness. Ten specimens were tested for all composite and epoxy samples and the average values of the tensile strengths and epoxy samples and the average values of the tensile strengths and moduli of elasticity calculated for each of the specimens.

2.5 Scanning Electron Microscopy (SEM)

The surface morphologies of the composites at various fibre weight fractions for both the treated and untreated sisal fibre-epoxy resin composites were analysed using the electron microscopy technique. The composite surfaces were analysed using the Zeiss Environmental SEM (ESEM: model EVO HD 15, operating at 20 kV), where the specimens were gold sputter coated using Quorum-150R ES model thin film coating equipment. The coating was applied in order to enable the specimens to become easily visible. The treated and untreated sisal fibres were scanned as well.

2.6 Flexural Testing

A three-point bending test was used to obtain values of flexural strength and stiffness. This was done in accordance to ASTM D 790 – 02 test standard specifications. In this test, a simply supported beam with span to thickness ratio of 16:1, with a centre loading support span, developed for design application was used. Specimens of dimensions 48 mm length, 3 mm thickness and 12.7 mm width were cut from the cast composites using a 3000 series CNC router machine. Testing was conducted in a displacement control mode with a cross head speed of 1.3 mm/min, as specified in the standard.

3.0 RESULTS AND DISCUSSION

3.1 SEM Results of Untreated and Treated Sisal Fibres

The untreated and treated sisal fibres which were subjected to scanning electron microscopy gave rise to the images being shown in Figures 2 and 3.
From the images it is clear that the cross sectional dimensions of the treated sisal fibres (180.6 μm) are smaller than that of the untreated sisal fibres (286.6 μm). Typically a reduction in the cross sectional dimensions of the reinforcing fibres implies an increased number of reinforcing fibres per given cross section of matrix and, therefore, better mechanical properties.
3.2 Tensile Test Results
The tensile fracture surfaces of the tensile specimens were viewed on a scanning electron microscope. Five scans were done for each specimen with the representative results shown in Figures 4 to 10.
It is clear from the images in these figures that the untreated sisal fibre-epoxy resin composites have higher incidences of fibre pull-out than those for the treated sisal fibre-epoxy resin composites. It is also evident that the pull out holes in the untreated sisal fibre-epoxy resin composite are larger than in the treated sisal fibre-epoxy resin composites. This is possibly due to fibre bunching which led to poor interfacial interaction between the bunched fibres and the resin and therefore, a weaker interfacial bond. This phenomenon is however, not evident in treated fibre reinforced composites. Further, the unreinforced epoxy resin sample exhibited a smooth fracture surface. Fibre pull-out is a result of ineffective bonding between the fibres and the matrix, which therefore, leads to an ineffective load transfer between the fibres and the matrix and non-optimal reinforcement. It is evident from the foregoing that treatment of the sisal fibres enhanced the interfacial bond strength between the fibres and matrix and is thus expected to give rise to composites with higher mechanical properties.
The tensile properties of pure epoxy, treated and untreated sisal fibre-epoxy resin composites are shown in Table 1. The results at 0 wt% represent the results for pure epoxy.

<table>
<thead>
<tr>
<th>weight % of the reinforcing fibre</th>
<th>Tensile strength of pure epoxy, treated and untreated sisal fibre-epoxy resin composites</th>
<th>Tensile modulus of pure epoxy, treated and untreated sisal fibre-epoxy resin composites</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Untreated sisal fibre-epoxy resin composites</td>
<td>Untreated sisal fibre-epoxy resin composites</td>
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<tr>
<td></td>
<td>Treated sisal fibre-epoxy resin composites</td>
<td>Treated sisal fibre-epoxy resin composites</td>
</tr>
<tr>
<td>Range (MPa)</td>
<td>Average (MPa)</td>
<td>CV (%)</td>
</tr>
<tr>
<td></td>
<td>Range (MPa)</td>
<td>Average (MPa)</td>
</tr>
<tr>
<td>Epoxy (0)</td>
<td>37.00 - 47.57</td>
<td>42.59 ±3.71</td>
</tr>
<tr>
<td>5</td>
<td>37.00 - 47.57</td>
<td>42.59 ±3.71</td>
</tr>
<tr>
<td>10</td>
<td>48.73 ±4.79</td>
<td>52.01 ±2.99</td>
</tr>
<tr>
<td>15</td>
<td>51.54 ±5.62</td>
<td>61.59 ±3.91</td>
</tr>
</tbody>
</table>
The results in Table 1 clearly show that, at 5 wt%, 10 wt% and 15 wt% of the reinforcing fibres, the treated sisal fibre-epoxy resin composites exhibited improvement in average tensile strength over the untreated sisal fibre-epoxy resin composites by 19.72%, 6.73% and 19.50%, respectively. The increase in the average tensile strength for the untreated and treated sisal fibre-epoxy resin composites over the values for the pure epoxy for the three percentage weights of 5 wt%, 10 wt% and 15 wt% are seen in the table to significant at -29.68%, 14.42% and 21.01%, and -15.80%, 22.12% and 44.61%, respectively.

The standard deviations of the tensile stiffness for both the untreated and treated composites are small, all falling within the range of 170.49 and 446.62. Furthermore, the co-efficient of variation for both the untreated and treated composites are small ranging from 5.70% to 15.33%. This implies that there was not much scatter in the experimental data obtained. The coefficients of variation are less than 50% (insignificant) implying that all the values were closer to the mean.

The tensile modulus for the treated sisal fibre-epoxy resin composite showed improvement over the values obtained for untreated sisal fibre-epoxy resin composites for the same reason noted in analysing a similar trend for tensile strength. It is evident that the change in properties of treated natural fibres leads to a change in the properties of their composites. The chemical treatment of natural fibres modifies the fibre surface to making them less hydrophilic and, also improving their surface roughness, and the attendant improvement of tensile properties [17]. Mohanty et al. [5] noted that a suitable coupling agent could be used to improve the compatibility between hydrophilic fibres and the hydrophobic matrices in order to improve the fibre-matrix interfacial bond. This is was achieved in the present work through silane treatment of natural fibres. This treatment creates a bond between the hydroxyl groups in the natural fibre with silane which reduces the hydrophilicity of the fibres. This in turn leads to the establishment of strong links between the fibres and polymer matrices, and the attendant improvement in the mechanical properties of their composites. Figures 11 and 12 show plots of the variation of the average tensile strength and elastic modulus with weight fractions of the reinforcing fibre for the treated and untreated sisal fibre-epoxy resin composites.
The plotted curves in Figure 11 clearly show the presence of minimum and critical fibre weight fraction for both the treated and untreated sisal fibre-epoxy resin composites. Furthermore, the plotted curve for the untreated sisal fibre-epoxy resin composite shows saturation in the average tensile strength beyond 11 wt%. The minimum strength of the reinforcing fibre for the treated and untreated sisal fibre-epoxy resin composites is 35 MPa and 30 MPa, respectively, corresponding to a minimum fibre weight fraction of 5%. Further, the critical fibre weight fraction for both the treated and untreated sisal fibre-epoxy resin composite are 7.6 wt% and 9 wt%, respectively. At the critical fibre weight fractions, the values of strength for both the treated and untreated sisal fibre-epoxy resin composites are the same at 42 MPa. It is also evident that the curve on the right side has two different gradients. This is a sign of reducing effectiveness of reinforcement with increasing weight percentage of the reinforcing fibres. The advent of reduced effectiveness of reinforcement occurs at 10 wt% for both the treated and the untreated sisal fibre-epoxy resin composites. The correlation co-efficient for both curves is 1. This implies a perfect curve fit to the experimental data plotted in both cases.
Fig. 12. A plot for the average values of tensile elastic modulus versus reinforcing fibre weight percentage for treated and untreated sisal fibre-epoxy resin reinforced composites

The plotted curves in Figure 12 clearly show the presence of minimum fibre weight fraction for both the treated and untreated sisal fibre-epoxy resin composites. Furthermore, the plotted curve for both the treated and untreated sisal fibre-epoxy resin composite shows a gradual decline in the average tensile modulus beyond 12 wt%. This gradual decline is a sign of reducing effectiveness of reinforcement with increasing percentage of the reinforcing fibres which is expected to be the result of fibre bunching and uneven fibre distribution. The minimum values of stiffness of the treated and untreated sisal fibre-epoxy resin composites are 2800 MPa and 1700 MPa, respectively, corresponding to 5 wt% of the reinforcing fibres. The curve for treated sisal fibre-epoxy resin composite has no critical weight fraction. It is also evident, as was the case for the curves of strength shown in Figure 11, that the right end curves in Figure 12 have two different gradients. This is a sign of reduced effectiveness of reinforcement. The advent of reducing effectiveness of reinforcement with increasing percentage of the reinforcing fibres occurs at 10 wt% for both the treated and the untreated sisal fibre-epoxy resin composites. The correlation coefficient for both curves is 1, implying a perfect curve fit to the experimental data plotted in both cases.

3.3 Flexural Test Results

Flexural tests were performed to evaluate the strength and stiffness of the pure epoxy resin, as well as treated and untreated sisal fibre-epoxy resin composites. The average values obtained are shown in Table 2. The results at 0 wt% represent the values for pure epoxy resin.
## Table II
Flexural strength and Flexural Modulus for pure epoxy resin as well as treated and untreated sisal fibre-epoxy resin composites

<table>
<thead>
<tr>
<th>Fibre weight (%)</th>
<th>Flexural strength of treated and untreated sisal fibre-epoxy resin composite</th>
<th>Flexural modulus of treated and untreated sisal fibre-epoxy resin composite</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Untreated sisal fibre-epoxy resin composites</td>
<td>Treated sisal fibre-epoxy resin composites</td>
</tr>
<tr>
<td>Range (MPa)</td>
<td>Average (MPa)</td>
<td>CV (%)</td>
</tr>
<tr>
<td>Epoxy (0)</td>
<td>64.21-82.38</td>
<td>69.85±5.71</td>
</tr>
<tr>
<td>5</td>
<td>47.33-87.15</td>
<td>51.02±12.54</td>
</tr>
<tr>
<td>10</td>
<td>98.22-109.47</td>
<td>101.00±4.27</td>
</tr>
<tr>
<td>15</td>
<td>105.73-130.43</td>
<td>116.30±8.53</td>
</tr>
</tbody>
</table>


The results in Table 2 clearly show that the average flexural strength and stiffness of the treated sisal fibre-epoxy resin composites were higher than those for the untreated sisal fibre-epoxy resin composites, for the same fibre weight fractions of 5 wt%, 10 wt% and 15 wt%, respectively, with the increment from the untreated to the treated composites at these percentage weights being 14.56%, 22.22% and 69.36%, respectively, for flexural strength. The increment in values of flexural stiffness from the untreated to the treated composites of the order 13.04%, 9.35% and 14.66% for the same percentage weights, respectively. The increase of the average flexural strength for the untreated and treated fibre reinforced composites over the values for the pure epoxy resin for the three percentage weights of 5 wt%, 10 wt% and 15 wt% are seen in the table to be significant at -26.96%, 44.60% and 66.50%, and -16.32%, 76.74% and 181.99%, respectively.

The increment in the values of flexural stiffness of both the untreated and treated composites over the values for the pure epoxy resin for the three percentage weights are seen in the table to be significant at -16.66%, 5.80%, 14.98% and 38.79%, respectively. The improvement in flexural properties can be attributed to the improved matrix/fibre interfacial properties as a result of the fibre treatment. The standard deviations for the flexural strength are small ranging from 4.27 to 12.54, while those for flexural modulus are also small ranging from 307.41 to 552.04. This implies that there was not much scatter in the experimental data obtained. The values of co-efficient of correlation for both flexural strength and flexural stiffness are also small, all ranging from 5.65% to 17.33%. Since the values of co-efficient of correlation are less than 50%, it implies that the values obtained are closer to the mean and that there is no much scatter.

A graphical representation of the values of flexural strengths as well as the flexural moduli is shown in Figures 13 and 14, respectively.

![Graph showing flexural strength vs fibre weight fraction](image)

**Fig. 13.** A plot of the average values of flexural strength versus fibre weight fraction percentage for treated and untreated sisal fibre-epoxy resin reinforced composites

The equation for the flexural strength is given by:

\[ \sigma_f = -0.0905w_f^3 + 2.8856w_f^2 - 14.445w_f + 69.85 \]

The correlation coefficient is 1.

The equation for the flexural modulus is given by:

\[ \sigma_f = -0.138w_f^3 + 3.446w_f^2 - 17.546w_f + 69.85 \]

The correlation coefficient is 1.
Starting with an initial decrease, both Figures 13 and 14, clearly show a continuous increase of the tensile strength and stiffness with increasing fibre weight percentage. Suradi et al. [25] reported similar observations with empty fruit bunch fibres. Furthermore, higher values were recorded for the treated sisal fibre-epoxy resin composites than for the untreated sisal fibre-epoxy resin composites for the same reasons given in analysing a similar trend for the tensile test results. The coefficient of correlation in all the curves plotted in Figures 13 and 14, is equal to 1. This implies perfect polynomial curve fits to the experimental data plotted in all the cases. From Figure 13, it can be observed that the minimum flexural strength of the reinforcing fibre weight fraction percentage for both the treated and untreated sisal fibre-epoxy resin composites is 5 wt% while the corresponding values of strength at this point are 55 MPa and 50 MPa respectively. The critical reinforcing fibre weight fraction percentages of flexural strength for the treated and untreated sisal fibre-epoxy resin composite were 6.5 wt% and 7 wt% respectively. The corresponding values of flexural strength values at these points was 60 MPa for both sets of composites. For both treated and untreated sisal fibre-epoxy resin composites, it is evident that the curves on the right of 5 wt% has two different gradients. This is a sign of reducing effectiveness of reinforcement with increasing weight percentage of the reinforcing fibres. The advent of the reduced effectiveness of reinforcement occurs at 10 wt%.

The values of flexural strength and stiffness of both the treated and untreated sisal fibre-epoxy resin composites were higher than the corresponding values for tensile strength and stiffness. The difference arises from the nature of the stress and strain states in tension and bending which are not the same. With a tensile test, the maximum tensile stresses are experienced throughout the entire volume (and surface area) of the test piece; in bending (where the sample sees tensile stress above the neutral axis and compressive stresses below), the maximum tensile stresses are conversely concentrated in a small region on the top surface above the neutral axis. Accordingly for similar sized test pieces, the tensile sample sees the maximum stresses throughout its entire gauge length, i.e., over a much larger volume than the corresponding bend sample.

**CONCLUSIONS**

1. Fibre surface treatment leads to an increase in the tensile and flexural properties of the treated sisal fibre-epoxy resin composites as compared to the untreated sisal fibre-epoxy resin composites.
2. There are higher incidences of fibre pull-out in the untreated sisal fibre epoxy-resin composites than in the treated sisal fibre-epoxy resin composites.
3. The values of the flexural strength and stiffness are higher than the values of tensile strength and stiffness at the same weight fractions.
4. Incidences of reduced rate of increase in the reinforcing effect with increasing weight percentage of the reinforcing fibres are evident beyond a given weight percentage for both tensile and flexural strength and stiffness.

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REFERENCES