Finite element analysis and experimental approaches of mono and hybrid nanocellulosic composites under tensile test

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Abstract

Sisal nanofibres and Rice husks nanosilica particles are valuable materials which are used in many applications around the world. Polymer composites with nanocellulosic fibres as reinforcing materials are widely used due to their high strength to weight ratio and their strong tensile properties. This study focused on the tensile properties of mono and hybrid nanocellulosic polymer composites using both experimental and the finite element method. The experimental method was used to validate the finite element analysis results. The experimental and numerical results show that the tensile properties of both the mono and the hybrid composites increase up to an optimal point, beyond which they begin to reduce. It was also evident that the hybrid composites had higher tensile properties than the mono composites.

1. Introduction

Nanocellulosic fibres have demonstrated their importance in the recent years by triggering an interest in their production in order to replace petroleum based materials [1, 2]. Nanocellulosic fibres and nanoparticles are obtained from natural sources such as: plants, animals and bacteria [3]. The nanocellulosic fibres and nanoparticles are used in polymer composites as fillers [1–4]. The benefits of nanocellulosic fibres and nanoparticles when compared to the synthetic ones include: biodegradability, increased energy recovery, good thermal and acoustic properties, acceptable specific strength properties, low density and low cost [1, 5].

Cellulose is a semicrystalline polycarbohydrate whose constituents are anhydroglucose units bonded to 1,4-glycosidic bonds [3]. There are three hydroxyl functional groups at each of the anhydroglucose units that can form hydrogen bonds that contribute to the increased stiffness of cellulose chains [3]. In order to increase accessibility to superfine cellulose nanofibris (CNF) or nanocrystals (CNC), the breakdown of the cross-linked elements in raw cellulose fibre (lignin, cellulose, pectin, and hemicellulose) is usually required [6]. In the cellulose nanocrystals (CNC), the native cellulose surfaces are modified by breaking down the β-(1,4)-glycosidic bonds that stabilizes the nanowhisker suspension through electrostatic repulsion to prevent fibril aggregation [6].

Even though nanocellulosic fibres and nanoparticles obtained from plants have benefits, their main limitation is related to the hydrophilic nature of cellulose [7]. In order to reduce the hydrophilicity of the nanocellulosic fibres and particles, these nanocellulosic fibres and particles are chemically modified in order to make them compatible with the hydrophobic matrices [7]. In order to reduce the aforementioned incompatibility, the reinforcement surface can be chemically modified [7, 8]. Chemical treatments increase interfacial adhesion with hydrophobic matrices such as epoxy and decreases water absorption. Among the reported chemical methods, mercerization, silanization, oxidation, esterification, grafting of branched, hyperbranched polymers stand out [7–10].

This study extracted nanocellulosic fibres and nanosilica particles from sisal fibres and rice husk, respectively, due to their ease of availability.
Rice husks are obtained from the rice-milling process and are usually treated by burning. The main component of rice husk includes cellulose, lignin, and ash with the content dependent on the variety, climate, and geographic location of its growth. Sisal fibres on the other hand, are obtained from the leaves of the plant agave sisalana.

Epoxy resin is one of the most versatile thermosetting polymers with a wide range of industrial applications, mainly as a matrix to make composite materials in many industries, including aerospace, automotive, marine, etc. The drawbacks of epoxy resins include brittleness and poor resistance to crack propagation because of their high degree of chemical crosslinking.

Several studies which have focused on nanocellulose and nanosilica are illustrated hereunder:

Collazo-Bigliardi et al. did a comparative study on the extraction of cellulose fibres and cellulose nanocrystals (CNC) from coffee husks and rice husks. The authors used alkali, bleaching and acid hydrolysis treatment. Thereafter, the microstructural changes were analysed, and further the sizes and aspect ratios were determined. The study found that the aspect ratios were greater than 10. The study also noted that the elastic modulus obtained from 1 wt% coffee husk CNC/epoxy composites increased by 121%, while that from 1 wt% rice husk CNC/epoxy composites increased by 186%.

Pham et al. used nanosilica from rice husk as filler in epoxy resin using the novel method of thermal treatment. The particle size distribution ranged from 40–80 nm. This study found out that the fracture toughness of epoxy increased by 16.3% with the addition of 0.07 phr of rice based nanosilica.

Mor'an et al. studied the feasibility of the use of acid hydrolysis, chlorination, alkaline treatment and bleaching in extracting cellulose from sisal fibres. The authors thereafter characterized the extracted cellulose using methods such as scanning electron microscopy among others. This study found that the aforementioned extraction procedures produced purified cellulose.

Mamat Razali et al. used the acid hydrolysis method using hydrochloric acid to derive cellulose nanocrystals (CNC) from Malaysia Indica rice straw. This study found that there was an increase in the strength of Kevlar by 300% upon the addition of 1 wt% of CNC to epoxy composites. In addition, the elastic modulus increased by nearly 3 fold.

Roszowska-Jarosz et al. obtained nanocellulose fibres from pressed lignin using chemical extraction method. The authors then prepared nanocomposites using epoxy resins in the range of 0.5% to 1.5% w/w. The resulting composites were subjected to strength tests and it was noted that there was satisfactory improvement in the mechanical properties.

Wang et al. carried out an excellent review on the methods used to obtain nanocellulose including the common method of acid hydrolysis. The authors further identified the benefits and shortcomings of each method. The use of nanocellulose in thermosets and thermoplastics were also dealt with.

Kargarzadeh et al. studied the structure of cellulose fibre. The authors went further to indicate the existing methods of extraction of various kinds of nanocellulose such as cellulose nanofibres, cellulose nanocrystals among others. The authors also indicated the conditions which are required for the extraction of nanocellulose from various natural sources.

Deepa et al. used a combination of various chemical treatments such as alkaline treatment, bleaching and acid hydrolysis to extract nanocellulose from 5 different lignocellulosic sources namely; banana, sisal, kapok, pineapple leaf and coir. This research provided an avenue for the potential sources of nanocellulose from the aforementioned plants.

Suhot et al. provided an excellent review on the physical, mechanical, and thermal behavior of rice husk (RH) as a fibre for reinforcing various synthetic polymers, based on recent studies, conducted between 2017 and 2021.

Ayswarya et al. used rice husk ash (RHA), modified rice husk ash (MRHA) and nanosilica to form different composites using epoxy resins and, thereafter, to study their mechanical, dynamic mechanical and thermal properties of the resulting composites. The results exhibited improvement of tensile strength of about 20% over pure epoxy when nanosilica filled epoxy was used. Other studies on nanoparticles from rice husk were done by Moosa et al., Conradi et al., Nguyen et al., and Jumahat et al.

Xue et al. reported on a novel type of regenerated cellulose composite fibre reinforced with cellulose nanofibrils (CNFs) and nanosilica. This study found that there was an improvement of tensile strength of 47.46%.

From the reviewed literature, it is evident that the determination of mechanical properties of nanocomposites through the use of finite element method (ABAQUS modeling), have not been been considered before. Coincidentally, Suhot et al. in their review paper, covering research between 2017 and 2021 recommended the development of finite element method to compare the mechanical properties of rice husks from experimental works. This study therefore explores the novel idea of the use of Finite Element Analysis (FEA) to determine mechanical properties of nanocomposites through ABAQUS modeling. This study used...
both the numerical simulations and the experimental approach to perform the tensile test. The numerical results were validated using the experimental results.

2. Experimental procedure

The experimental design adopted for this study was full factorial which was determined using the Minitab software. The experimental design involved varying the percentage fibre volume fraction ($\nu_f$) during the fabrication of mono and hybrid composites. The tensile mechanical properties for mono and hybrid composites were investigated. The fibre volume fraction was varied in increasing steps until the optimum fibre volume fraction for use in the fabrication of test pieces was obtained. Sisal nanofibres and rice husk nanosilica particles have been chosen as fillers for this study, as they can be easily found.

The extraction of sisal nanofibres and rice husk nanosilica particles were considered first. These nanofibres and nanosilica particles were then used to form their mono composites separately with epoxy resins, and, subsequently combined together to form hybrid composites with epoxy resins. The characterization of these composites in terms of their mechanical properties was, then considered. Relevant ASTM standards were used in this study.

2.1. Materials

In this study, commercial bi-functional epoxy resin (LR 20), with its hardener (LH-281), was considered as a binder. The epoxy matrix was considered for use in this work due to the fact that they have superior adhesive properties and are resistant to environmental degradation [22]. The epoxy matrix was used as a single component naturally cured formation.

Furthermore, sisal nanofibres and rice husk nanosilica particles were used to make the reinforcement that was used in this study. The raw material for rice husk nanosilica particle production is rice husks. Rice husk is the outermost cover of the rice grain produced after milling and constitutes about 20%–30% by weight of the rice grain [2].

2.2. Extraction of sisal cellulose nanofibres

Sisal cellulose nanofibres were extracted using the standard acid hydrolysis procedure [13, 15]. The acid hydrolysis procedure was deemed convenient for this study since the chemicals which are required to perform this procedure could be found easily.

2.3. Extraction of rice husk nanosilica particles

Nanosilica particles from rice husks for use in this study were obtained using the standard method [2, 16, 19]. Rice husks were mixed with 5% sodium hydroxide and stirred for 1 h. They were then filtered, and thereafter washed with distilled water in order to remove the sodium hydroxide from them [19]. Following onto this, they were immersed in 1% acetic acid to remove any remaining sodium hydroxide. Subsequently, they were immersed into 67.5% solution of hydrochloric acid for 5 h [2]. Following onto this, they were washed with de-ionized water and dried in an oven at 45°C for 24 h in order to ensure that they were dry. Subsequently, they were calcinated in a furnace at 500°C for 4 h [2, 16, 19], then crushed in a ball mill in order to obtain nanosilica particles [19].

2.4. Composite fabrication

2.4.1. Fabrication of sisal nanofibre reinforced epoxy resin specimens

An electronic balance with an accuracy of ±0.5 g was used to weigh sisal nanofibres. Masses which had the same fibre volume fractions were grouped together. A rectangular mould measuring 50 cm by 100 cm was smeared with a thin layer of wax, which ensures that there is ease of removal of the composite after curing. The steel mould was covered with predetermined sisal nanofibres corresponding to different fibre volume fractions which had been spread in a longitudinal direction. The electronic balance was further used to measure the matrix and its hardener in the appropriate ratio recommended by the manufacturer. A spatula was thereupon, used to mix the matrix and the hardener. A sharp needle was used to remove air from the matrix by puncturing the air bubbles. Thereafter, the nanofibres were spread with resin. Curing of the resulting nanocomposite was done for 24 h. A total of 154 samples were fabricated using this method.

2.4.2. Fabrication of rice husk nanosilica particles reinforced epoxy resin composites

The same procedure adopted to fabricate sisal nanofibre reinforced-epoxy resin composites as described in section 2.4.1 of this article, was adopted to fabricate the rice husk nanosilica particle reinforced epoxy resin composites.
2.4.3. Fabrication of sisal/rice husk hybrid reinforced epoxy resin nanocomposites

The same procedure which was adopted to fabricate sisal nanofibre reinforced-epoxy resin composites as described in section 2.4.1 of this article, was adopted to fabricate the sisal/rice husk hybrid reinforced epoxy resin nanocomposites with the exception being that the two reinforcements were used in conjunction.

2.5. Scanning electron microscopy

The surfaces of the nanocellulosic fibres from sisal and nanosilica particles from rice husk were analysed using the Zeiss Environmental SEM (ESEM: model EVO HD 15, operating at 30 kV), where the specimen was 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.

2.6. Experimental approach of tensile test

ASTM D 3039 was used for this approach with the relevant dimensions stated in the standard being applied for this experimental approach.

Tabs were manufactured and cut from a woven glass fibre/epoxy resin composite with a thickness of 1.5 mm. The tabs were bevelled 7 mm away from the gauge length at an angle of 30º to avoid stress concentration at the gauge length during testing. The overall lengths of the specimen were 250 mm, and their gauge lengths were 136 mm.

The tab lengths were 57 mm and were mounted on both ends that were to be gripped, as shown in figure 1. After the specimens were prepared, tests were carried out using an MTS 793 servo-hydraulic 100 N load cell, computer-controlled, screw drive, multipurpose testing machine. A test strain rate of 5 mm min⁻¹ was used for all the specimens as specified in the standard.

2.7. Finite element analysis for mono composites

Mono composites are composites that are composed of only one fibre and the matrix. This study used the sisal nanofibres and the rice husk nanosilica particle separately in conjunction with epoxy resins to form mono composites. In finite element analysis, the monocomposites were created in the property module. The property module has the ‘material manager’ in which the properties of the materials which are used to form the monocomposites are created. The material properties were determined experimentally and, thereafter, input in the material manager for properties of the different materials under consideration.

The property module also has the ‘composite layup manager’, in which the properties of the composite are input. These composite properties include the ply name, regions, material, thickness, orientation angle and the integration point.

The materials were selected from the material manager, which already had the properties of the fibres and the matrix that had been input earlier. The fibre volume fractions of the composites were used to define the thickness of both the fibres and the matrix. The orientation angle for the fibre was kept at 0º while that of the matrix was kept at 90º, in the same way as was done experimentally. Only two plies are selected for mono composites since only the fibre and the matrix is under consideration.

The ‘query information’ tab was selected, and the composite model was shown by selecting the ‘ply stack plot’ option.

Figure 1. Schematic diagram of a tensile specimen (mm).
2.8. Finite element analysis for hybrid composites

Hybrid composites are composed of at least two fibres and a matrix. In this study, the sisal nanofibre and rice husk nanosilica particles were considered in combination with the epoxy matrix in order to form the hybrid composite.

The same procedure used to model mono composites was adopted in modelling hybrid composites, with the distinction being that, in hybrid composites, at least three plies are selected. For this study, the hybrid composites had three materials, and, hence three plies were selected.

Another distinction between hybrid composites and monocomposites lies in the fact that in the hybrid composites, at least three orientation angles are considered. In this study, both the sisal nanofibre and rice husk nanosilica particle were oriented at 0º while the epoxy matrix was oriented at 90º.

2.9. Finite element analysis of tensile properties of composites

ABAQUS/CAE version 2018 was used to perform the tensile simulations. Tensile simulations were done to determine how strong the composites were and how much they could be stretched before they broke. The dimensions used in the simulations were similar to those which had been used in the experimental approach. This was deemed necessary for comparative purposes. These dimensions were determined using ASTM D 3039 standards.

The following procedure was used in ABAQUS/CAE software to obtain the composites’ tensile properties. For example, the procedure used to determine the tensile strength of 60% sisal nanofibre reinforced epoxy resin composites is illustrated in the following procedures.

| Property/Material                  | Sisal nanofibre | Epoxy |
|------------------------------------|----------------|-------|
| Longitudinal tensile strength (MPa) | 700            | 80    |
| Longitudinal compression strength (MPa) | 650            | 120   |
| Transverse tensile strength (MPa)  | 700            | 80    |
| Transverse compression (MPa)       | 650            | 120   |
| Longitudinal shear (MPa)           | 500            | 70    |
| Transverse shear (MPa)             | 500            | 70    |
| Axial Young Modulus (E1), (MPa)    | 12,000         | 3,400 |
| In-Plane Youngs Modulus (E2), (MPa) | 12,000         | 3,400 |
| Poissons ratio                     | 0.1            | 0.4   |
| In-Plane Shear Modulus, (G12), (MPa) | 11,000         | 1,240 |
| Transverse Shear Modulus, (G13), (MPa) | 11,000         | 1,240 |
| Transverse Shear Modulus, (G23), (MPa) | 11,000         | 1,240 |

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2.9.1. Part module

The ABAQUS/CAE software adopted for this study was used to create the composite layup part within the software. The dialogue box ‘create part’ was selected, and ABAQUS/CAE software opened an option allowing for the description of the created part. The modelling space which was used for this study was kept at 3D default and deformable type. The base feature was selected for a shell shape with planar type for this study. The approximation size was increased to 200 mm for the accommodation of the design dimensions. The units used were millimetres. The dimensions of the part created were 250 × 25 × 3 mm.

2.9.2. Material property module

Upon selection of the property on the module toolbar, ABAQUS/CAE allowed the assignment of material property by clicking on the ‘create material’ option.

For a single fibre ply material under mechanical elasticity, an elastic material behaviour was selected. The type of elasticity was set to ‘Laminar’ with the longitudinal modulus (E1), transverse in-plane modulus (E2), and transverse out-plane modulus (E3) values defined. Other material properties which were defined included: The in-plane shear modulus (G12), the out-of-plane shear modulus (G23 and G13), and the Poisson’s ratio, \( \nu_{12} \).

Furthermore, damage for fibre-reinforced composites (Hashin damage) was selected, and the required values were input.
The following values shown in Table 1 were used to create the material definition for sisal nanofibre and epoxy resin.

The 'create composite layup' manager was used on the site bar with the initial ply set at 2 to account for the sisal nanofibre and the epoxy resin with element type set at the conventional shell.

2.9.3. Assembly module
For the creation of instance under the assembly part module, the instance type was kept at 'dependent' as the entire model was designed as a single composite though made of different constituents. The whole part was, therefore, considered as a single part in the same coordinate.

2.9.4. Step module
In Abaqus software, the initial step is usually already created and cannot be modified. Additional steps were created on static general, which were then assigned to the load process and later on the boundary conditions. In addition, the field output request was used with the domain set to 'composite layup'. The output variable of stress components and invariants, total strain components and Hashin failure criteria for both fibre and matrix in tensile and compression were selected. The selected output variables allowed the analyses of the results at the end of the model designed to be compared for failure.

2.9.5. Interaction module
A coupling constraint was created at the upper edge of the cross-section from the interaction module.

2.9.6. Load and boundary conditions module
The 'Load' from the module toolbar in Abaqus/CAE allows the application of load and boundary conditions. The load manager was used to create a concentrated force in the upward direction.

To set the fixed supports condition, the boundary condition 'Encastre' was applied to the lower edge of the cross-section. This boundary condition was selected as it ensures no displacement and rotation in the global X, Y and Z-directions, making the supports fully fixed. The second type of boundary condition that was applied was the displacement/rotation boundary condition. This boundary condition was applied to the upper part of the cross-section since there will be an upward movement in this part.

2.9.7. Meshing the model
On the mesh module, the global seeds option allowed the possibility of modification of the default approximate global size. The global size was set at 0.65 mm, resulting in a fine mesh with 14,630 elements and 58,520 nodes.

From the top mesh toolbar, the element type was selected as shell family, with all the element controls kept at default. The geometrical order was quadratic with full integration. The mesh part was selected, and the model meshed as per the seeding of 0.65 mm which had been selected in the seed part.

2.9.8. Job creation and analysis submission
At this stage, the job for each model was created and submitted for analysis using the job creation manager.
2.9.9. Results visualization
With job creation selected on the access toolbar, the design created through Abaqus with all composite constituent properties and mechanical loading imparted to it, the submission option allowed for the generation of a visualisation result.

An example, the tensile strength for 60% sisal nanofibre reinforced epoxy resin composite is shown in figure 2. The tensile strength in ABAQUS software is indicated by the magnitude of the maximum principal stress. The results from ABAQUS software are usually indicated at a 75% average default setting, as shown in figure 2.

3. Results

3.1. Scanning electron microscopy (SEM) results
The Scanning Electron Microcopy (SEM) images for both the sisal nanocellulosic fibres and rice husk nanoparticles are shown in figures 3 and 4.

It is very clear in figure 3 that the acid hydrolysis procedure of nanocellulose extraction was very effective in removing hemicellulose, lignin, pectin and wax since the average diameters of the nanofibres were 5 μm. It is
also evident from figure 4, that the rice husk nanosilica particles had average diameters of 10 μm implying the effective attack of chemical agents in disrupting the internal structure of the material while removing the non-cellulosic components. Other authors reported different fibre sizes depending on the plant fibre used. As an example, water hyacinth fibre was 25–50 μm in size [23].

The van der Waals forces and hydrogen bonds that act in the crystalline zones of the cellulose provide high resistance to acid attack, while the amorphous regions are disordered and prone to acid hydrolysis. Thus, acid hydrolysis removes the cellulose fibres of the amorphous zones and reduces the fibre size to nanometric scale [24]. The average aspect ratio of the sisal nanofibres was 35.5. Kallel et al [25] reported that the aspect ratio of cellulose nanocrystals [CNC] extracted from different materials usually vary from 10 to 70, although higher values have been obtained from garlic straw (L/d = 80) [25]. Silv’erio et al [26] reported that cellulose nanocrystals could be considered as a good reinforcing material if their aspect ratio exceeds a value of 10. In our case, the average aspect ratio of the sisal nanofibres exceeded 10, implying that they were good reinforcing material.

3.2. Results of tensile strength for sisal nanofibre reinforced polymer composites
The effects of fibre reinforcement on epoxy resins are best shown in direct tensile tests. Tensile strength results of sisal nanofibre reinforced polymer composites are presented in table 2.

From table 2, it can be observed that the lowest tensile strength was recorded for the unreinforced specimens at 0 v.f%. The corresponding tensile strength value at 0 v.f% was 1.115 MPa for the experimental approach and 1.061 MPa for the FEA approach. In both the FEA and experimental approaches, the tensile strength was then seen to increase gradually with fibre additions until an optimum tensile strength was recorded. In the experimental approach, optimum tensile strength of 3.478 MPa was obtained at 9.040 v.f%, whereas, for the FEA approach, the optimum tensile strength was recorded as 2.794 MPa at 8.164 v.f%. This increase in tensile strength with fibre addition up to the optimum point can be attributed to an increase in adhesion between the fibres and the matrix as the fibre content increases. The difference in the optimum points between the FEA and the experimental approach can be attributed to the fact that the two approaches have different accuracy levels.
3.2.1. Comparison of experimental and FEA results of tensile strength for sisal nano fibre reinforced epoxy resin composites

A graph of tensile strength values versus fibre volume fraction of sisal nano fibre reinforced epoxy resin composites showing both FEA and experimental results is presented in figure 5.

From figure 5, it is evident that at around 5 υf%, both the FEA and experimental plots intersected and recorded the same value of tensile strength. This point of intersection also corresponds to the optimal fibre volume fraction for the FEA approach. At this point of intersection, the corresponding tensile strength value was 3 MPa. The optimum fibre volume fraction for the experimental approach was found to be around 9 υf%. It is also evident that the tensile strength in both the FEA and experimental approaches increases steadily up to the optimal point, beyond which the tensile strength begins to reduce gradually. The differences in the optimum points between the FEA and experimental approaches are attributed to the accuracy differences between the two methods.

3.2.2. Validation of FEA results of tensile strength for sisal nano fibre reinforced epoxy resin composites

The percentage errors of the results obtained using the FEA approach in relation to the results obtained using the experimental approach have been computed in table 3. The experimental approach was used as the reference method.

It can be seen from table 3 that the deviation between FEA and experimental results is insignificant and are in close agreement for 41.67% of the results since the margin of error is within the acceptable engineering percentage of error of ±5%. For 58.33% of the results, the margin of error falls outside the acceptable engineering percentage error of ±5%. The reasons for these deviations could be attributed to the fact that both the experimental and numerical methods have different accuracy levels.
### 3.2.3. Results of tensile strength for rice husk nanosilica particle reinforced epoxy resin composites

The results of tensile strength of rice husk nanosilica particle reinforced epoxy resin composites are presented in table 4. These results are based on both the FEA and experimental approaches.

From table 4, it is evident that the unreinforced matrix had the lowest tensile strength value in the FEA approach. In the results showing the experimental approach, a slight decrease in tensile strength with fibre additions was observed at reinforcement volume fractions \( \nu_f < 0.999 \). Beyond 0.999 \( \nu_f \%), the inclusion of fibres in the matrix improved its mean tensile strength from a value of 1.096 MPa in unreinforced specimens to a peak value of 2.721 MPa at a fibre volume fraction of 6.966 \( \nu_f \%), beyond which there was a decrease in tensile strength with successive fibre additions. In the FEA approach, the peak tensile strength was recorded as 2.522 MPa at 5.031 \( \nu_f \)%.

### Table 4. Tensile strength values for rice husk nanoparticle reinforced epoxy resin composites.

| Fibre volume fraction (\( \nu_f \)% | FEA (MPa)     | Experimental (MPa) |
|-----------------------------------|---------------|--------------------|
| 0.000                             | 1.061         | 1.115 ± 0.107     |
| 0.999                             | 1.364         | 1.096 ± 0.168     |
| 1.974                             | 1.338         | 1.291 ± 0.206     |
| 2.975                             | 1.561         | 1.518 ± 0.175     |
| 3.935                             | 2.146         | 1.964 ± 0.372     |
| 5.031                             | 2.522         | 2.521 ± 0.358     |
| 5.818                             | 2.484         | 2.625 ± 0.230     |
| 6.966                             | 2.432         | 2.721 ± 0.169     |
| 7.758                             | 2.398         | 2.507 ± 0.165     |
| 8.340                             | 2.373         | 2.523 ± 0.185     |
| 9.124                             | 2.341         | 2.448 ± 0.279     |
| 10.285                            | 2.296         | 2.268 ± 0.317     |

### Table 5. Error margin of experimental and FEA results.

| Fibre Volume Fraction (\( \nu_f \)% | FEA (MPa) | Experimental (MPa) | % Error |
|-----------------------------------|-----------|--------------------|---------|
| 0.000                             | 1.061     | 1.115              | -4.843  |
| 0.999                             | 1.364     | 1.096              | 24.453  |
| 1.974                             | 1.338     | 1.291              | 3.641   |
| 2.975                             | 1.561     | 1.518              | 2.833   |
| 3.935                             | 2.146     | 1.964              | 9.267   |
| 5.031                             | 2.522     | 2.521              | 0.040   |
| 5.818                             | 2.484     | 2.625              | -5.371  |
| 6.966                             | 2.432     | 2.721              | -10.621 |
| 7.758                             | 2.398     | 2.507              | -4.348  |
| 8.340                             | 2.373     | 2.523              | -5.945  |
| 9.124                             | 2.341     | 2.448              | -4.371  |
| 10.285                            | 2.296     | 2.268              | 1.235   |

### 3.2.4. Comparison of experimental and FEA results of tensile strength for rice husk nanosilica particle reinforced epoxy resin composites

A graph of tensile strength versus fibre volume fraction of rice husk nanosilica particle reinforced epoxy resin composites showing both experimental and FEA results is presented in figure 6 hereunder.

Figure 6 shows how the tensile strength varies with the reinforcement volume fraction in rice husk nanoparticle reinforced-epoxy resin composites in both the FEA and experimental approaches. The composite tensile strength was found to be dependent on the fibre content in both the experimental and FEA approaches. The graph shows that both the FEA and the experimental plots intersect at around 5 \( \nu_f \)% and 10 \( \nu_f \)% fibres, hence giving the same tensile strength values of around 2.1 MPa and 2.3 MPa, respectively. It is also evident that the optimum tensile strength, in both the FEA and experimental approach, was recorded at around 8 \( \nu_f \)% fibres. The corresponding tensile strength at the optimal fibre volume fraction was 2.5 MPa for the FEA approach and around 2.7 MPa for the experimental approach.
3.2.5. Validation of FEA and experimental results of tensile strength for rice husk nanosilica particle reinforced epoxy resin composites

The percentage errors of the results obtained using the FEA method in relation to the results obtained using the experimental approach have been computed in table 5.

It can be seen from table 5 that the deviation between experimental and FEA results is insignificant and are in close agreement for 58.33% of the results since the margin of error is within the acceptable engineering percentage of error of ±5%. For 41.67% of the results, the margin of error falls outside the acceptable engineering percentage of error of ±5%. These deviations are attributed to the same reasons stated in section 3.2 of this paper.

3.3. Results of tensile strength for sisal/rice husk hybrid reinforced-epoxy resin composites

Table 6 shows the tensile strength values for sisal/rice husk hybrid reinforced epoxy resin nanocomposite obtained using the FEA and experimental methods.

Table 6. Tensile strength values for sisal/rice husk hybrid reinforced epoxy resin nanocomposite.

| Fibre volume fraction (\( \nu \)%) | FEA (MPa) | Experimental (MPa) |
|---------------------------------|-----------|--------------------|
| 0.000                           | 1.061     | 1.115 ± 0.107      |
| 0.999                           | 2.004     | 1.856 ± 0.168      |
| 1.974                           | 2.292     | 2.170 ± 0.206      |
| 2.975                           | 2.834     | 2.686 ± 0.175      |
| 3.935                           | 3.339     | 3.184 ± 0.372      |
| 5.031                           | 3.500     | 3.342 ± 0.358      |
| 5.818                           | 3.405     | 3.790 ± 0.230      |
| 6.966                           | 3.275     | 3.104 ± 0.169      |
| 7.758                           | 3.192     | 3.344 ± 0.165      |
| 8.340                           | 3.134     | 3.416 ± 0.185      |
| 9.124                           | 3.059     | 3.254 ± 0.279      |
| 10.285                          | 2.956     | 3.136 ± 0.317      |

3.2.5. Validation of FEA and experimental results of tensile strength for rice husk nanosilica particle reinforced epoxy resin composites

The percentage errors of the results obtained using the FEA method in relation to the results obtained using the experimental approach have been computed in table 5.

It can be seen from table 5 that the deviation between experimental and FEA results is insignificant and are in close agreement for 58.33% of the results since the margin of error is within the acceptable engineering percentage of error of ±5%. For 41.67% of the results, the margin of error falls outside the acceptable engineering percentage of error of ±5%. These deviations are attributed to the same reasons stated in section 3.2 of this paper.

3.3. Results of tensile strength for sisal/rice husk hybrid reinforced-epoxy resin composites

Table 6 shows the tensile strength values for sisal/rice husk hybrid reinforced epoxy resin nanocomposite obtained using the FEA and experimental methods.

From table 6, it is evident the lowest tensile strength values in both the FEA and experimental approaches were recorded for the unreinforced tensile specimens at 0 \( \nu \)%). In FEA and experimental approaches, these unreinforced specimens exhibited tensile strength values of 1.061 MPa and 1.115 MPa, respectively. The tensile strength was then seen to increase with fibre additions. The highest tensile strength was recorded as 3.790 MPa at 5.818 \( \nu \)% for the experimental approach and 3.500 MPa at 5.031 \( \nu \)% for the FEA approach. The increments of the FEA results over the values obtained for the unreinforced tensile specimens were 88.88%, 167.11%, 220.92%, 195.38% and 178.61% at 0.999 \( \nu \)%, 2.975 \( \nu \)%, 5.818 \( \nu \)%, 8.340 \( \nu \)% and 10.285 \( \nu \)%, respectively. The increments of the experimental results over the values obtained for the unreinforced tensile specimens were 66.46%, 140.90%, 239.91%, 206.37% and 181.26% at the same fibre volume fractions as those for the FEA results.
### 3.3.1. Comparison of FEA and experimental results of tensile strength for sisal/rice husk hybrid reinforced epoxy resin nano composites

A graph of tensile strength versus fibre volume fraction of sisal/rice husk hybrid reinforced epoxy resin composites showing both FEA and experimental results is presented in figure 7.

From figure 7, it is evident that in both the experimental and FEA approaches, there is an increase in tensile strength up to the optimum point. This increment up to the optimum point with fibre additions is a sign of a better adhesion of the fibres to the epoxy matrix as the reinforcement increases. It is also evident that the tensile strength begins to reduce gradually with more fibre additions in both the FEA and experimental approaches. This reduction in tensile strength may be attributed to insufficient compaction due to poor workability at high fibre contents. Furthermore, it is evident that both the FEA and the experimental plots intersect at around 6 $\nu_f$%. At this point of intersection, the corresponding tensile strength is around 3.4 MPa.

### 3.3.2. Validation of FEA results of tensile strength for sisal/rice husk hybrid reinforced epoxy resin nano composites

The percentage errors of the results obtained experimentally in relation to the results obtained using the FEA approach have been computed in table 7.

From table 7, the deviation between FEA and experimental results is insignificant in close agreement for 50% of the results since the margin of error is within the acceptable engineering percentage of error of $\pm 5\%$. For 50% of the results, the margin of error falls outside the acceptable engineering percentage of error of $\pm 5\%$. These deviations could be attributed to the differences in the accuracy levels of the two methods used.

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**Table 7. Error margin of experimental and FEA results.**

| Fibre volume fraction ($\nu_f$%) | Hybrid FEM | Hybrid E | % Error |
|---------------------------------|------------|----------|---------|
| 0.000                           | 1.061      | 1.115    | -4.843  |
| 0.999                           | 2.004      | 1.856    | 7.974   |
| 1.974                           | 2.292      | 2.170    | 5.622   |
| 2.975                           | 2.834      | 2.686    | 5.510   |
| 3.935                           | 3.339      | 3.184    | 4.868   |
| 5.031                           | 3.500      | 3.342    | 4.728   |
| 5.818                           | 3.405      | 3.790    | -10.158 |
| 6.966                           | 3.275      | 3.104    | 5.509   |
| 7.758                           | 3.192      | 3.344    | -4.454  |
| 8.340                           | 3.134      | 3.416    | -8.255  |
| 9.124                           | 3.059      | 3.254    | -4.823  |
| 10.285                          | 2.956      | 3.136    | -4.829  |
3.4. Comparison of tensile strength results of mono and hybrid composites

Figure 8 shows a graph comparing the tensile strength of sisal nanoﬁbre reinforced epoxy resin composites, rice husk nanosilica particle reinforced epoxy resin composites, and sisal/rice husk hybrid reinforced epoxy resin nanocomposite.

It is evident from the graph in figure 8 that the hybrid composites have higher tensile strength values than the monocomposites. It is also evident that the sisal/rice husk hybrid reinforced epoxy resin composites had tensile strength values, which were almost closer to those of the sisal nanoﬁbre reinforced epoxy resin composites intersection point being around 7.8 υf% and 8.5 υf%. Furthermore, the values of tensile strength for rice husk nanoparticle reinforced epoxy resin composites were the lowest. The behaviour depicted in the graphs could be attributed to the difference in compaction between the different ﬁbres and the epoxy matrices.

3.5. Analysis of variance (ANOVA) for tensile strength

Table 8 shows a summary of variances of the mono and hybrid composites under experimental and the FEA approaches, whereas table 9 shows the analysis of variance between and within groups.

From table 8, the results from sisal/rice husk hybrid reinforced epoxy resin nanocomposite done using the experimental approach had the highest variance value of 0.754, while the rice husk nanosilica particle reinforced epoxy resin composites done using the FEA approach had the lowest value of the variance of 0.268. The amount of variance indicates the inﬂuence that independent variables have on the dependent variables. Therefore, it follows that the inﬂuence of the independent variables on the dependent variables in sisal/rice husk hybrid reinforced epoxy resin nanocomposite done using the experimental approach was higher than those in rice husk nanosilica particle reinforced epoxy resin composites done using the FEA approach.

Furthermore, From table 9, the values of the sum of squares (SS), degree of freedom (df), mean square (MS), F-statistic, the P-value and the F critical are indicated. The variability between the groups was 9.503, while the variability within the groups was 32.985. The degree of freedom between groups was 5, while that within groups was 66. The mean square between groups was 1.900, whereas that within groups was 0.500. The F statistic test and the P statistic test indicate that the test is signiﬁcant since each group has a mean that differs signiﬁcantly from the overall group mean.

4. Discussion

4.1. Fibre volume fraction effects on tensile strength

From ﬁgure 8, it was observed that sisal/rice husk hybrid reinforced epoxy resin nanocomposite had higher tensile strength values than the sisal nanoﬁbre reinforced epoxy resin composites and the rice husk nanosilica particle reinforced epoxy resin composites at the same fibre volume fractions. This higher tensile strength of the hybrid composite than the mono composites can be attributed to a higher adhesion to the epoxy matrices. This high adhesion in the hybrid composite results arises from the fact that the hybrid composite provides a larger surface area than the mono composites.

The reduction in tensile strength at higher reinforcement volume fractions for all the composite samples as depicted in ﬁgures 5, 6 and 7 of both the FEA and the experimental results may be attributed to insufficient

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**Table 8. Summary of variances in mono and hybrid composites.**

| Groups                                          | Count | Sum  | Average | Variance |
|-------------------------------------------------|-------|------|---------|----------|
| Sisal nanofibre reinforced epoxy resin composites (Experimental) | 12    | 32.665 | 2.722   | 0.655    |
| Sisal nanofibre reinforced epoxy resin composites (FEA) | 12    | 30.152 | 2.513   | 0.383    |
| Ricehusk nanoparticle reinforced epoxy resin composites (Experimental) | 12    | 24.404 | 2.034   | 0.426    |
| Ricehusk nanoparticle reinforced epoxy resin composites (FEA) | 12    | 24.412 | 2.034   | 0.268    |
| Sisal/ricehusk hybrid reinforced epoxy resin nanocomposite (Experimental) | 12    | 35.304 | 2.942   | 0.754    |
| Sisal/ricehusk hybrid reinforced epoxy resin nanocomposite (FEA) | 12    | 34.050 | 2.8375  | 0.512    |

**Table 9. Analysis of variance between and within groups.**

| ANOVA Source of variation | SS     | df | MS    | F      | P-value | F crit |
|---------------------------|--------|----|-------|--------|---------|--------|
| Between Groups            | 9.503  | 5  | 1.900 | 3.803  | 0.004   | 2.354  |
| Within Groups             | 32.985 | 66 | 0.500 |        |         |        |
| Total                     | 42.488 | 71 |       |        |         |        |
compaction due to poor workability at higher fibre contents. The higher fibre contents increased the number of voids and, consequently, improper impregnation of fibres into the matrix. The overall effect is to make the composite which is already weak in tension, even weaker when a direct tensile load is applied to it.

The general observation for unreinforced epoxy resin specimens is that tensile stress increases on loading until the first crack stress is reached. The matrix cracks beyond the cracking stress and can no longer carry any stress. Sisal nanofibre reinforced epoxy resin composites, rice husk nanosilica particle reinforced epoxy resin composites, and sisal/rice husk hybrid reinforced epoxy resin nanocomposite continued to carry extra loads even after the matrix had cracked. This finding implies that the composites have a post-cracking ductility behaviour, accompanied by multiple matrix cracking.

The composite tensile strengths as depicted in figures 5, 6 and 7 increased with fibre additions up to the optimal points, beyond which there is a decrease in the tensile strengths for both the experimental and the FEA results. The fibre balling-up phenomenon at fibre volume fractions exceeding the optimal point enhances the incidence of improper impregnation of fibres into the matrix, which explains why the rate of increase is seen to decrease at reinforcement volume fractions exceeding the optimal point.

The composite failure was by several cracks running perpendicular to the direction of loading followed by fibre fracture at higher fibre volume fractions in all the composites meaning that, the fibre lengths used were greater than the critical fibre length, as explained by Hale [27]. The occurrence of multiple fractures indicates that the fibre contents incorporated into the matrix had also exceeded the critical/optimum fibre volume fraction [27].

The scatter observed in the experimental results can be attributed to some of the problems encountered during the fabrication process. A lot of imperfections such as non-uniformity in fibre distributions were inevitable; this is because the evenness of fibre distribution was judged just by looking with the naked eye. The total effect of all inclusions and micro-cracks is to make the stress-strain relation non-linear. They also act as stress concentrations, thus lowering the tensile strength.

The important factors affecting the tensile strengths of sisal nanofibre reinforced epoxy resin composites, rice husk nanosilica particle reinforced epoxy resin composites, and sisal/rice husk hybrid reinforced epoxy resin nanocomposites have been found to include reinforcement volume fractions, incorporation technique and compactness of the fibres into the matrix.

Upon comparing the FEA tensile strength results with the experimental tensile strength results, it was established that the margins of error of 41.67% of the results obtained from sisal nanofibre reinforced epoxy resin composites were within the acceptable engineering percentage error limit of ±5%. For the rice husk nanosilica particle reinforced epoxy resin composites, 58.33% of the results were within the acceptable engineering error limit of ±5%. For the sisal/rice husk hybrid reinforced epoxy resin nanocomposites, 50% of the experimental results were within the acceptable margin of error limit of ±5%. The deviations from the results could be explained by the fact that both the FEA and experimental approaches have different accuracy levels.

5. Conclusions

1. The tensile properties of all the composites depicted an increase with fibre additions up to a certain optimum point beyond which they began to fall gradually with further fibre additions.

2. The increase in tensile properties of mono composites was about 2 times lower in magnitude in comparison to hybrid composites.

3. The important factors affecting the tensile strengths of sisal nanofibre reinforced epoxy resin composites, rice husk nanoparticle reinforced epoxy resin composites, and sisal/rice husk hybrid reinforced epoxy resin nanocomposites have been found to include reinforcement volume fractions, incorporation technique and compactness of the fibres into the matrix.

4. Acid hydrolysis is effective in removing the non-cellulosic materials in order for the cellulose to be extracted.

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Declarations

Availability of data and material
All data generated or analysed during this study are included in this published article.

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There is none to be declared

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