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Research Article

Numerical and Experimental Assessment of Mechanical Properties of Biobased Epoxy Reinforced by Flax Fibres and Seashell Nanoparticles for Prosthetic Socket

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Chronic diseases such as peripheral vascular and arteriosclerosis, wars, terrorist attacks, natural disasters, and traffic collisions are the major causes of the high demand for prostheses. The inadequacy of the typically used materials at reasonable prices and the high stiffness of these materials, which can negatively influence socket-limb load transfer, imply an urgent need to find alternatives to the existing prosthetic sockets. This work aims to use renewable, low-hazard, and low-cost natural flax fibres and seashell nanoparticles as substitutes for conventional reinforcement materials for prosthetic sockets. Seashell nanoparticles of 1, 3, and 5 weight fractions and 3 layers of flax fibres were integrated into biobased epoxy. Tensile and flexural properties of modified and unmodified specimens were assessed, and the finite element technique (ANSYS-20) was utilised to analyse and evaluate the mechanical characteristics of the specimens by observing the stress and total deformation. Here, all fabricated nanocomposites provided tensile and flexural strength higher than that of additive-free biobased epoxy. In addition, hybrid nanocomposites fabricated from 3 layers of flax fibres and 3 wt% of seashell nanoparticles were revealed to have the highest mechanical properties compared with unmodified resin and biobased epoxy filled with other percentages of the reinforcement. These findings were further validated numerically in which the total deformation was shown to decrease after the addition of 3 wt% of these nanoparticles within the nanocomposites. Moreover, the antibacterial activity proved its superb antimicrobial performance against various pathogenic microorganisms, namely, *Staphylococcus aureus* and *E. coli*. Accordingly, the fabricated composite systems are suggested to be an appropriate candidate for forming prosthetic sockets. These composites have promising mechanical and antibacterial properties, and they are made of affordable and available materials, which can be a suitable option, particularly in poor countries due to the fact that advanced technologies may require a substantial amount of money for equipment, surgery fees, and medical care.

1. Introduction

The demand for prosthetic limbs is a matter of concern for a large group of people all over the world. This demand is not restricted to a specific country or geographical area even though the number of required prostheses is different from one region to another worldwide. There are many reasons behind such an urgent global need for prostheses, including war, road traffic accidents, natural disasters [1], and chronic diseases such as diabetes [1, 2]. The conventional production of prosthetic limbs involves modular components connected to a limb socket. The socket is a vital part of the assembly because of its contact with the individual's residual limb [3]. Lots of progress has been made in the design and material selection for socket fabrication. Composites formed by the incorporation of layers of cotton stockinette, Nyglass stockinette, and carbon fibres (CF) in acrylic are normally used for construction sockets. Nonetheless, the undesirable emissions of these non-sustainable materials and the disposal complications that occur after their use can be considered as a negative environmental issue. In addition to their environmental and health problems, these materials are also expensive. Therefore, when the demand for prostheses is imperative, the utilisation of available alternatives should be taken into consideration. Biomaterials, including thermoset polymers and fibres, derived from plants may afford the required alternatives [4]. Besides their abundance and accessibility as sustainable sources, plant-based fibres provide promising characteristics such as lower cost, low bulk density, low environmental impact, longevity, biocompatibility, and good mechanical properties, thereby providing composites that could have properties that cannot be achieved using synthetic fibre based composites [5–7]. Plant-based fibres are composites comprised essentially of hollow cellulose fibrils embedded in a matrix of lignin and hemicellulose [4, 8]. The cellulosic fibre-based composites exhibit environmental sustainability [9]. Some kinds of flax are considered among the strongest plant fibres which are competitive with glass fibres [10]. According to their lignocellulosic origin, they are composed of cellulose, hemicellulose, lignin, pectin, waxes, and fats [11], with an elastic modulus of $44\,kN/mm^2$ [10]. The low ecological effect during production and accessibility make flax suitable as a competitive biobased reinforcing material [12]. Flax fibres (FF) are stated to have remarkable vibration damping properties, making composite prosthetic sockets better than those made of fibreglass or CF. Besides, FF has the advantage of being a natural, renewable source and an affordable substitute for manmade fibres. Hence, lightweight sockets made of FF could be valuable for decreasing the vibrations transmitted to the body. In a recent study, Monette et al. found that the composite sockets produced completely from FF were lighter and more efficacious in reducing damping vibrations than sockets fabricated totally from CF [13].

Likewise, inorganic nanoparticles including calcium carbonate ($CaCO_3$) have positively enhanced the mechanical properties of composites thanks to the high strength and large specific surface area of these nanoadditives [14, 15]. Affordable inorganic reinforcements have been incorporated into polymers not only to reduce cost but also to enhance their mechanical behaviour. Discarded by-product waste materials such as seashell nanoparticles (SS), which are composed of high $CaCO_3$ and some organic compounds, have high thermal stability and are considered an alternative to mineral limestone [16]. SS can be obtained from the hard outer part of the sea creatures' bodies including clams and snails [17]. Studies revealed that the chemical composition of several types of SS is $CaCO_3$ (up to 99%) [17–19]. There has been a widespread tendency to reinforce polymers using SS for a number of purposes. For instance, biocomposites made of SS powder and low-density polyethylene were prepared. As a result of the increase in the reinforcement, the coefficient of friction and wear was revealed to decrease [20]. SS-reinforced polymethyl methacrylate (PMMA) biocomposite was also demonstrated to have higher compressive strength [21] and wear resistance [22] compared to the SS-free composite. Even so, SS are believed to not be utilised as intensely as other ceramic fillers in reinforcing biomaterials despite having a composition similar to human bone and teeth.

Epoxy resins have been extensively investigated for various applications, including in biomedical areas such as in dentistry [23]. Epoxy resins can provide a sound polymerised network by using various agents including amines and thiols [24]. The relentless efforts to find alternatives to conventional petroleum-based epoxy have led to widespread interest in natural-based epoxy. Biobased epoxy has characteristics such as low toxicity and cost, which can reduce the detrimental influence of nonsustainable epoxy on the environment and economy. Nonetheless, the use of these biobased materials may cause an inadequate performance of the obtained epoxy products [25]. Therefore, the purpose of this work was to appraise the influence of the incorporation of three layers of woven flax fabric (FF) and various weight fractions of SS on the mechanical properties of biobased epoxy in order to be used for prosthetic sockets. The mechanical property measurements of mono-nanocomposites and hybrid nanocomposites were presented. Neat biobased epoxy, FF-reinforced biobased epoxy, and FF-biobased epoxy hybrid nanocomposites with 1, 3, and 5 wt% of SS were tested for tensile and flexural properties. Finite element simulation was used for obtaining the stress and total deformation of the sockets. Due to the fact that the area of contact between the patient's skin and the socket can be a stimulating environment for the growth of bacteria, the antibacterial properties of the composite material that had the best mechanical properties were also evaluated. The obtained nanocomposites had tensile and flexural strength higher than those of unreinforced biobased epoxy. Furthermore, the total deformation of nanocomposites achieved from finite element analysis was shown to decrease after the addition of 1 and 3 wt% of these nanoparticles. Besides, the fabricated composite systems exhibited superb antimicrobial performance against various pathogenic microorganisms.

2. Materials and Methods

2.1. Materials. Seashell nanoparticles (SS) that have a size of 50 nm were provided from Viet D.E.L.T.A Industrial Co., Ltd. High biobased laminating epoxy resin and its hardener, and flax fibres (FF) were purchased from Entropy Resins and Shaoxing Starke Textile Co., Ltd, respectively.

2.2. Fabrication of FF/SS/Biobased Epoxy Composites. SS nanoparticles at 1, 3, and 5 wt% were mixed with biobased epoxy and ethanol using a high-speed stirrer at 35°C for 30 minutes. After ethanol evaporation, the mixture was degassed and left to cure in a silicon mould containing 3 layers of FF for 10 hours at room temperature. Afterwards, the composite substrates having a square shape of 25×25 cm were removed and cut using a computer numerical control (CNC) router machine to form specimens for morphological, mechanical, and antibacterial assessment.

2.3. Scanning Electron Microscopy (SEM). Microscopic investigations of SS, FF, pristine biobased epoxy, and its composites were conducted using INSPECT F 50 FE-SEM. Specimens were gold-coated prior to SEM imaging.

2.4. Mechanical Properties. Neat and reinforced biobased epoxy were mechanically characterized by means of tensile and flexural tests. Instron Universal Tester was used to assess the tensile strength of dog-bone-shaped specimens having dimensions of $154 \times 13 \times 5$ mm according to ASTM D638. They were mounted in the grips and pulled at a crosshead rate of 5 mm/min until failure. For flexural tests, specimens were cut according to ASTM D790 standard where specimens of dimensions $125 \times 13 \times 4$ mm were placed on two supports whereas load was applied at the centre. Samples of tensile and flexural tests are illustrated in Figure 1.

2.5. Finite Element Analysis. The finite element provides equivalent alternating stress, namely, von Mises stress and total deformation of the simulated prosthetic socket. The model of the socket used herein was previously designed and plotted in AutoCAD after cutting a positive mould fabricated from the gypsum of a transtibial amputee into horizontal discs [26]. This design's x, y, and z dimensions were taken, and the socket's geometry was exported to ANSYS 2020 R1 workbench. Pressures were applied on the sockets plane, and ANSYS was used for the assessment. The three-dimensional element was utilised for the modelling of solid constructions. It was elucidated by eight nodes that had three degrees of freedom at every node. Figure 2 represents the mesh generation of the composite sockets.

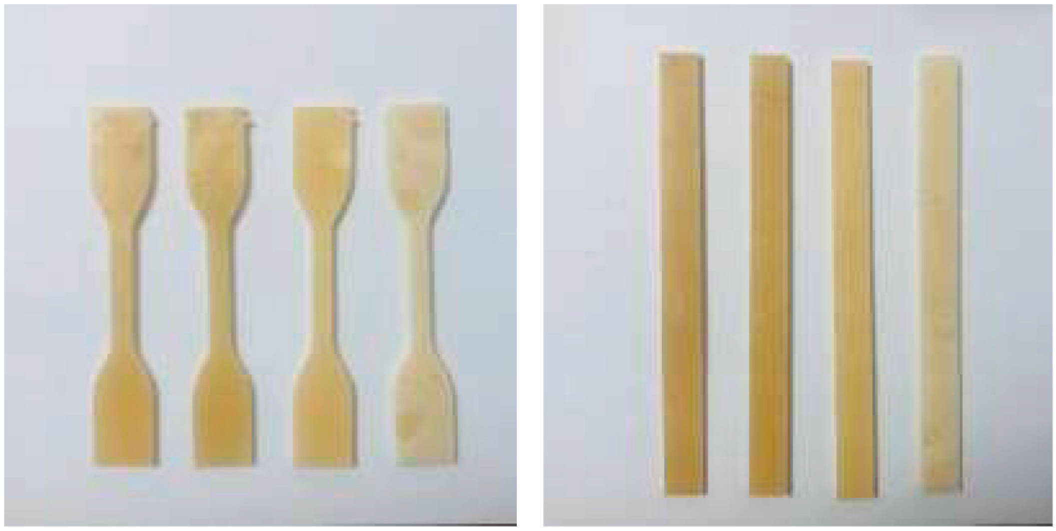
2.6. Antibacterial Activity. The antimicrobial activity of the composite that had higher mechanical properties, namely, 3 FF/3 wt% SS/biobased epoxy composite, was compared with additive-free bioepoxy samples. Antimicrobial activity was examined using an agar diffusion test against *E. coli* and *S. aureus*. Agar support was inoculated with bacteria; modified and unmodified biobased epoxy samples were placed on agar support and incubated at 37°C for 24 hours. Afterwards, the diameter of the zone of growth inhibition against the abovementioned bacteria was measured.

3. Results and Discussion

3.1. Morphology of FF, SS, and Nanocomposite. The surface morphology of SS, FF, and untreated and SS/FF-treated biobased epoxy was evaluated by FE-SEM. Photographs and SEM images of SS and the woven flax fabric are presented in Figure 3. SEM image displays the woven structure of FF that has surface roughness because of the presence of wax, lignin, oil, and other noncellulosic matters [27] while the yarn's surface was bounded together to produce a mat. On the other hand, the high surface area and energy of the miniature-sized particles led to the formation of agglomerations and clusters; these nanoparticles were shown to have rod-like shapes (Figure 3(d)). Figure 4 demonstrates how FF and SS changed the morphology of the biobased epoxy after reinforcing. While the additive-free biobased epoxy exhibited almost a flat surface, the presence of FF and 3wt % SS can be clearly seen after modification. The reinforcement was noticed to be well dispersed inside the matrix.

3.2. Mechanical Properties of Biobased Epoxy/FF/SS Nanocomposites

3.2.1. Tensile Properties. The tensile properties of the nanocomposites were largely affected by the loading of the reinforcement. The tensile strengths of biobased epoxy-FF-SS nanocomposites are shown in Figure 5. It shows that FF/SS/biobased epoxy composites had their tensile behaviour tuned according to the nanoparticles' weight fraction. FF/biobased epoxy and FF/SS/biobased epoxy nanocomposites exhibited a greater stiffness compared to the additive-free resin. The mesh-like interlocking shape in FF may make the composites containing FF withstand higher loading prior to breakage [28]. It can also be seen that the increase in the SS weight fraction led to improving the tensile strength of the nanocomposites. The nanocomposite displayed an improvement of 21.21% and 38.82% in tensile strength after the incorporation of 1 wt% and 3wt% of SS compared to reinforcement-free epoxy. Nonetheless, these values dropped after 5 wt% of SS nanoparticles had been introduced. The nature of the interface is essential in generating optimal performance for biobased epoxy/FF/SS composites. Thus, better tensile properties were accomplished when the specimens had an appropriate interface between the matrix and nanoparticles, which were achieved at 1 and 3 wt% of SS since the applied load was generally transferred to the additives through an interface. It was pointed out previously that the modulus of elasticity of epoxy-based nanocomposites could improve with nanoparticle content. Conversely, the ultimate tensile stress was revealed to be less for nanocomposites with a higher mass fraction of these nanofillers, which could be due to the aggregation of nanofillers at higher loading. The tensile stress, yield stress, and elastic modulus of the majority of the nanocomposites containing rigid nanoparticles can be enhanced with the increase in particle loading. Moreover, slips and changes in the position of the epoxy chains have occurred during the exposure of epoxy to a certain load. The structures with high cross-linked networks are more likely to have less deformation. Once nanoparticles are included in the resin, the movement of the biobased epoxy chains and their sliding on each other is restricted, resulting in a reduction in the flexibility of the composite material in general. As the SS content increases, the disentanglement and chain motion within the composites become even more difficult. This increase in constraint polymer chains accounts for the enhancement in the tensile properties of the formed composites. Some studies also found that the tensile stress was reduced with nanoparticle loading [29, 30]. This phenomenon may be attributed to the creation of voids, leading to stress concentration within the nanocomposite [30]. Moreover, the stress concentration could be a consequence of the creation of agglomerates at higher particle mass fractions [29].



(a) (b)

Figure 1: Neat biobased epoxy and FF-SS-reinforced biobased epoxy specimens were prepared for (a) tensile test, and (b) three-point bending test.

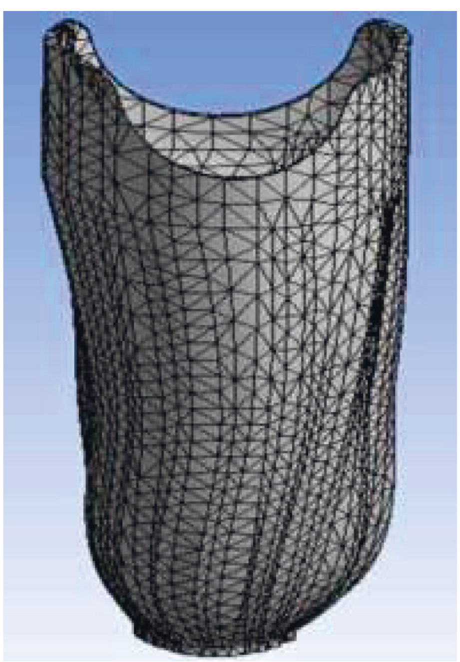


Figure 2: Prosthetic socket mesh design [26].

3.2.2. Flexural Strength. The flexural strength of bare biobased epoxy, FF-biobased epoxy, and FF/SS-biobased epoxy nanocomposites were determined by the three-point bending test. A steady increase in the flexure strength was recorded for biobased epoxy after reinforcing by FF and various mass fractions of SS (Figure 6). The maximum improvement in flexural strength was found to be around 52.8% for the composite composed of 3 wt% of SS compared to the flexural strength of the blank biobased epoxy. On the other hand, when 5 wt% of SS was included in the nanocomposite, the flexural strength dropped to a lower value than nanocomposites fabricated with 1 and 3 wt% of these fillers, yet the flexural strength was higher than that of blank biobased epoxy. Increasing the quantity of these fillers more than a particular amount has been detected to negatively affect flexural strength in previous studies. Among the factors that influence the performance of composites is the interaction between nanoparticles and matrixes. How much the density of the interphase established between two constituents relies on the interaction between fillers and matrix [23, 31]. Inter-particle distance has also a remarkable role to play in the interphase and nanocomposite characteristics. It was previously pointed out that some nanoparticles unless appropriately mixed with epoxy, could develop pores and increase the porosity of composites. The attraction between nanoparticles attributable to the van der Waals and electrostatic forces can deteriorate the performance of composites owing to irregular or inhomogeneous bonding between the components of the composite. Consequently, this could result in a reduction in the values of strength and modulus of nanocomposites with high loading of nanoparticles [31].

3.3. Simulation Results. For a designed socket, it is crucial to measure its effectiveness and capability of withstanding against forces and stresses to ensure that it does not fail prematurely during service. The finite element method may provide a great benefit in tracking the behaviour of the designed socket and how it is affected by the applied forces. The output data of experimental mechanical properties (Table 1) along with the geometry were fed to ANSYS to determine the equivalent von Mises stresses and total deformation.

3.3.1. Von Mises Stress. The finite element method and von Mises analysis have lately been utilised for forming a

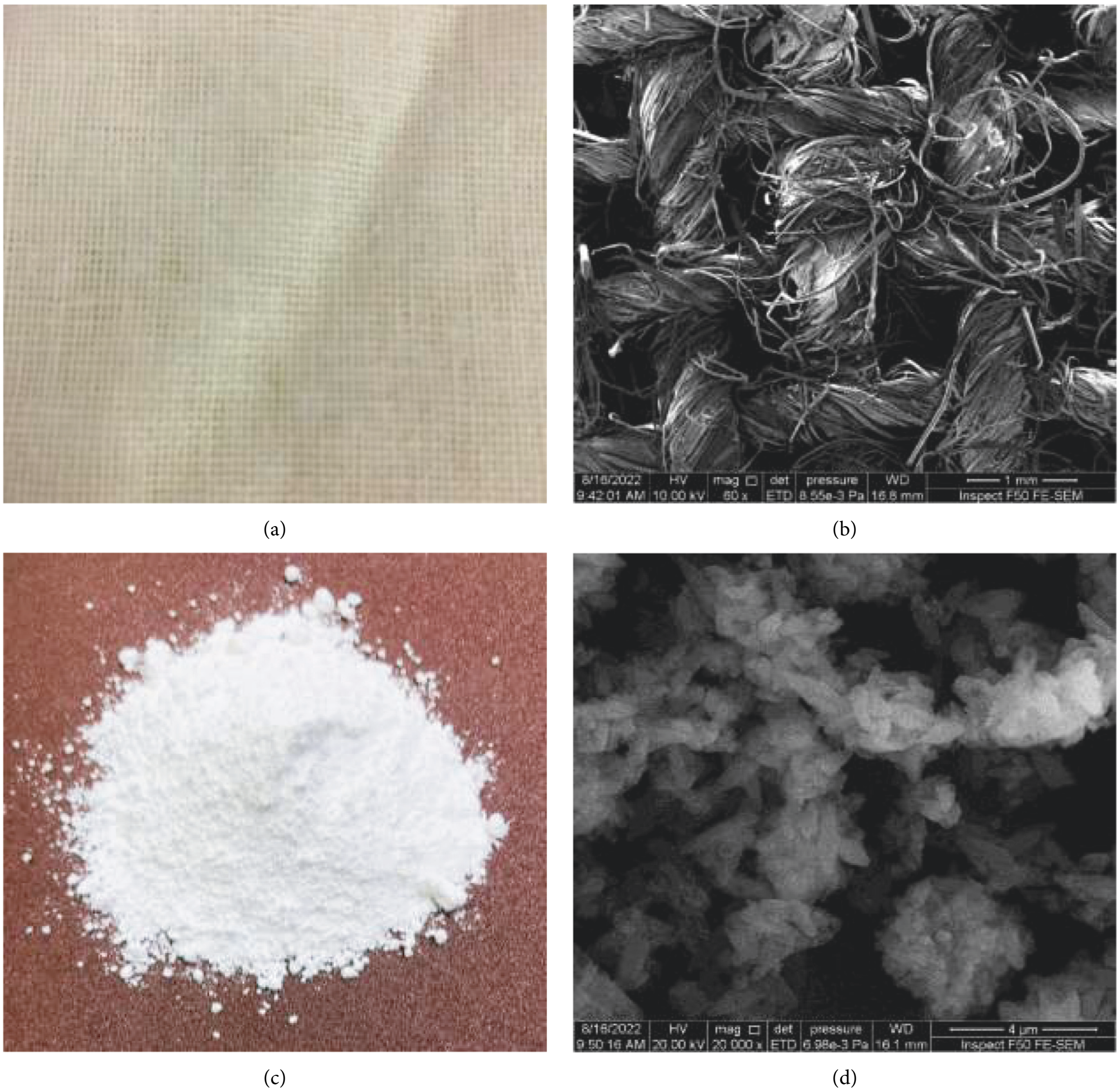


Figure 3: (a, c) Photographs of FF and SS (b and d) SEM images of FF and SS, respectively.

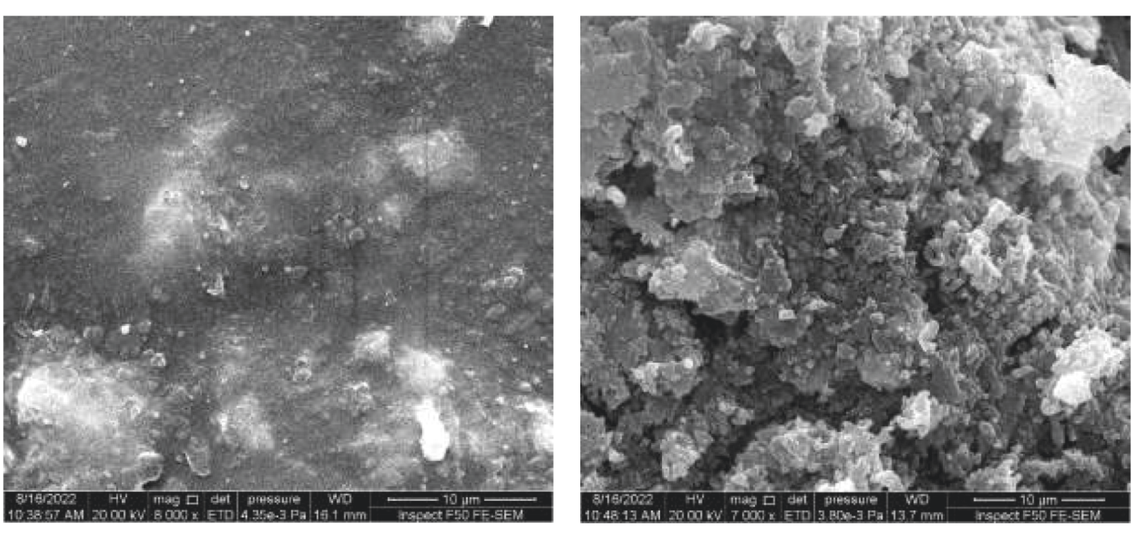


Figure 4: SEM micrographs of (a) blank biobased epoxy and (b) bioepoxy/FF/3wt%SS nanocomposite.

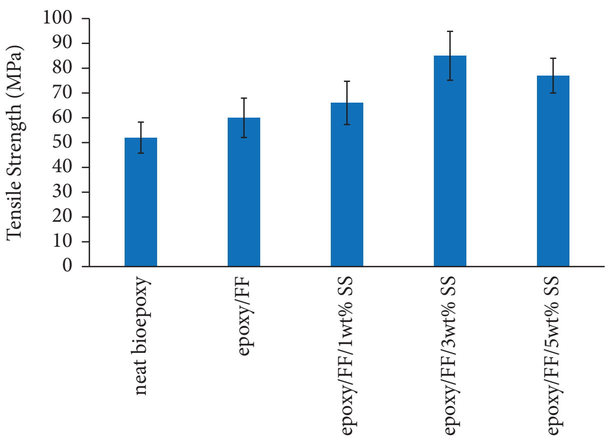


Figure 5: Tensile strength of pristine biobased epoxy and biobased epoxy reinforced by FF and various weight fractions of SS.

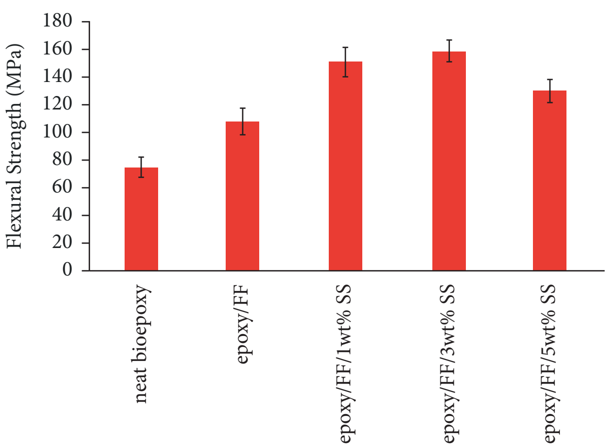


Figure 6: Flexural strength of pristine biobased epoxy and biobased epoxy reinforced by FF and various weight fractions of SS.

virtual model of biomedical-related applications and for assessing the stress distribution on essential parts [32]. Thus, a numerical analysis was carried out to measure the stresses formed in the socket as a consequence of the created interface pressure between the socket and contact body parts. Figure 7 depicts the design of composites, which demonstrates the overall estimated values and positions of the von Mises stresses through the different materials. For the fabricated composite, the findings demonstrated that the maximum equivalent von Mises stress was around 3.208 MPa. The highest value of equivalent stress was observed for the biobased epoxy/FF/5 wt% SS composite while the composite containing 1 wt% of SS had the lowest equivalent stress. The impacted area was located at the centre of the anterior side of the tibia bone. The loading of the reinforcement affected the stresses of the formed nanocomposites in which FF/biobased epoxy and FF/SS/biobased epoxy nanocomposites had higher stiffness than the additive-free resin. The mesh-like interlocking shape in FF and the proper interface between the matrix and nanoparticles as well as the rigid nature of the used nanoparticles caused the produced nanocomposites to have greater von Mises stresses compared to blank resin.

3.3.2. Total Deformation. Information about the socket's general behaviour can be obtained from the deformation evaluation. Among all unmodified and modified specimens, the maximum deformation value of the model socket was detected for biobased epoxy at approximately 3.07 mm, whereas the biobased epoxy reinforced by FF and 3 wt% SS had the lowest deformation of 0.85 mm. It was noticed that the total deformation decreased as the weight fraction of SS rose. Moreover, the highest deformations were detected in the lateral plane's centre, while the socket's basal plane had the lowest values (Figure 8). The specimens that have high cross-linked networks are more likely to have less deformation. Consequently, the presence of the rigid nanoparticles and three layers of fibres in the resin constrained the movement of the biobased epoxy chains, causing a reduction in the flexibility of the fabricated nanocomposite. The restriction of disentanglement and chain motion of epoxy can be even clearer with the increase in the SS loading. Generally, the obtained deformation values for the socket can be suitable, considering that the socket should deform when the interface pressure is applied to prevent irritation of the patient's skin during service.

3.4. Antibacterial Activity. Among the paramount issues experienced by amputee patients who use traditional prostheses are itching, skin irritation, and ulcers. Using natural materials could enhance the function of the prosthesis, which in turn improves the patient's quality of life. The antimicrobial activity of the composite that had the best results in terms of mechanical properties was therefore evaluated. This composite system was tested against *E. coli and S. aureus*. The growth of all tested pathogens was inhibited with the 3 wt%SS/FF/biobased composite (Figures 9 and 10). *E. coli* was slightly less sensitive to SS-FF-loaded biobased epoxy, while the bacteria straining *S. aureus* was more sensitive. Overall, the composite fabricated from biobased epoxy and 3 wt% SS plus 3 layers of FF had higher inhibittion zones than those of additive-free resin. The effectiveness of the composite against *E. coli* and *S. aureus* was enhanced after SS/FF incorporation by about 63% and 61%, respectively. The inhibition rate for *S. aureus* was higher than that for E. *coli* at about 4%. The thicker peptidoglycan membrane in the cell wall of *S. aureus* could lead to restriction of the diffusion and growth of microorganisms [33, 34].

It has also been reported that flax fibres extracted from various kinds and methods are characterised by the capability of reducing the growth of *S. aureus*. The antimicrobial capacity of fibres is largely associated with their chemical composition, principally lignin content, which is cross-linked with phenolic compounds in which the lignin-enriched fibres can have a great ability to decrease bacteria colonies [35]. As a result of showing potential capabilities in resisting bacterial growth, there is a tendency to use SS in

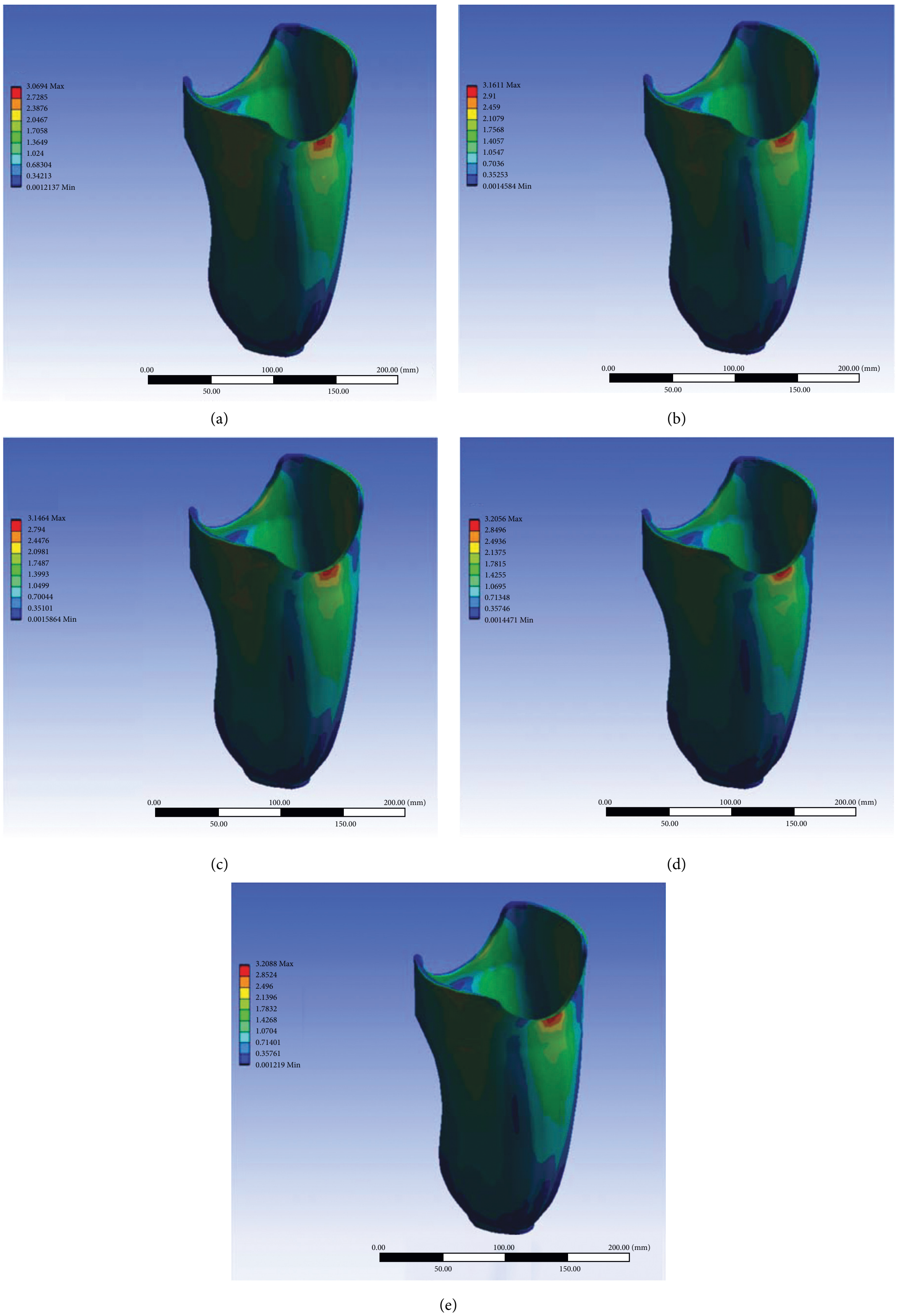
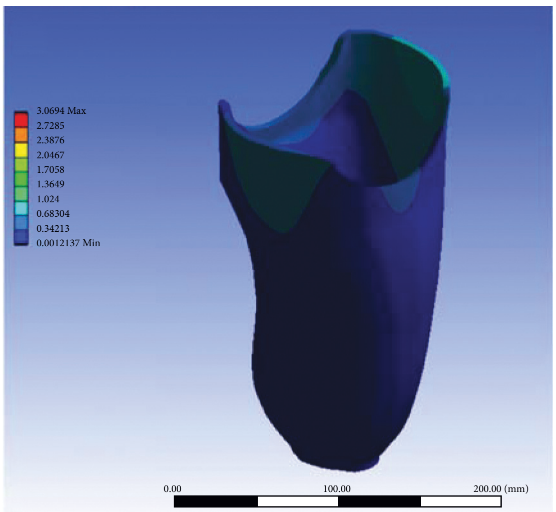


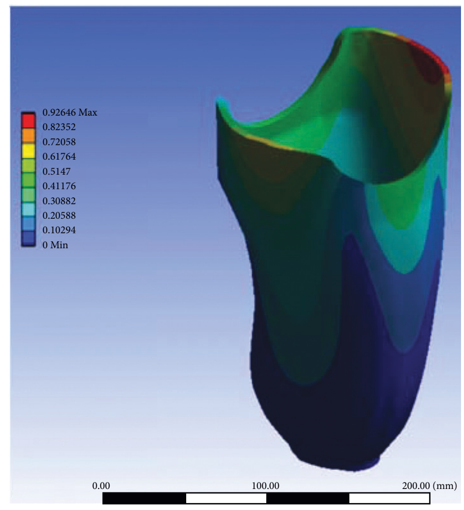
Figure 7: Contours of stress distribution of (a) pristine biobased epoxy, (b) FF/biobased epoxy composite, (c) FF/1wt % SS/biobased epoxy composite, (d) FF/3wt % SS/biobased epoxy composite, and (e) FF/5wt % SS/biobased epoxy composite.

Table 1: Measured mechanical properties of modified and unmodified biobased epoxy.

Specimen	Tensile strength (MPa)	Elastic modulus (GPa)	Poisson's ratio	Flexural strength (MPa)
Pristine biobased epoxy	52 ± 6.25	1.85 ± 0.459	0.220 ± 0.006245	75 ± 7.37
FF/biobased epoxy composite	60 ± 7.94	2.00 ± 0.465	0.328 ± 0.006248	108 ± 9.64
FF/1wt % SS/biobased epoxy composite	66 ± 8.72	2.12 ± 0.481	0.342 ± 0.006252	151 ± 10.59
FF/3wt % SS/biobased epoxy composite	85 ± 9.85	3.02 ± 0.512	0.540 ± 0.006258	159 ± 7.94
FF/5wt % SS/biobased epoxy composite	77 ± 7.1	2.55 ± 0.462	0.394 ± 0.006246	130 ± 8.33



(a)



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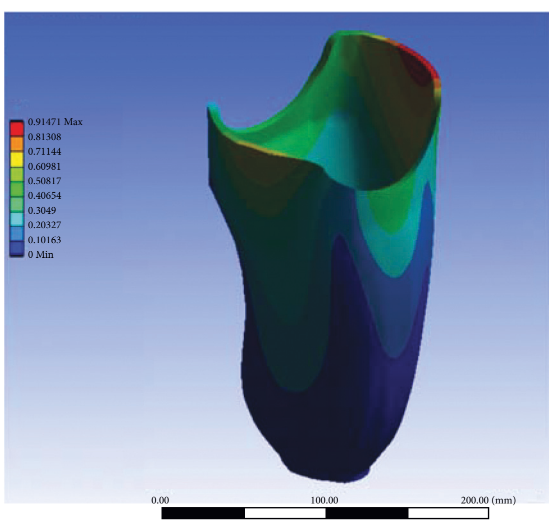

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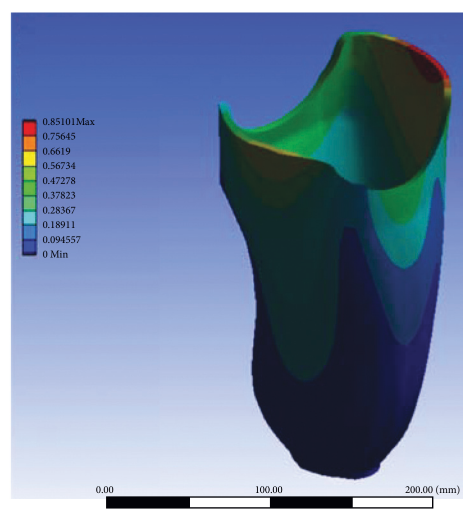
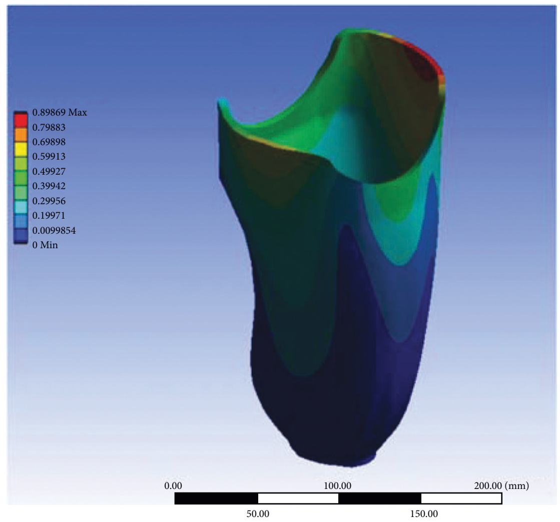


(d)

Figure 8: Continued.



(e)

Figure 8: Contours of total deformation distribution of (a) pristine biobased epoxy, (b) FF/biobased epoxy composite, (c) FF/1wt % SS/biobased epoxy composite, (d) FF/3wt % SS/biobased epoxy composite, and (e) FF/5wt % SS/biobased epoxy composite.

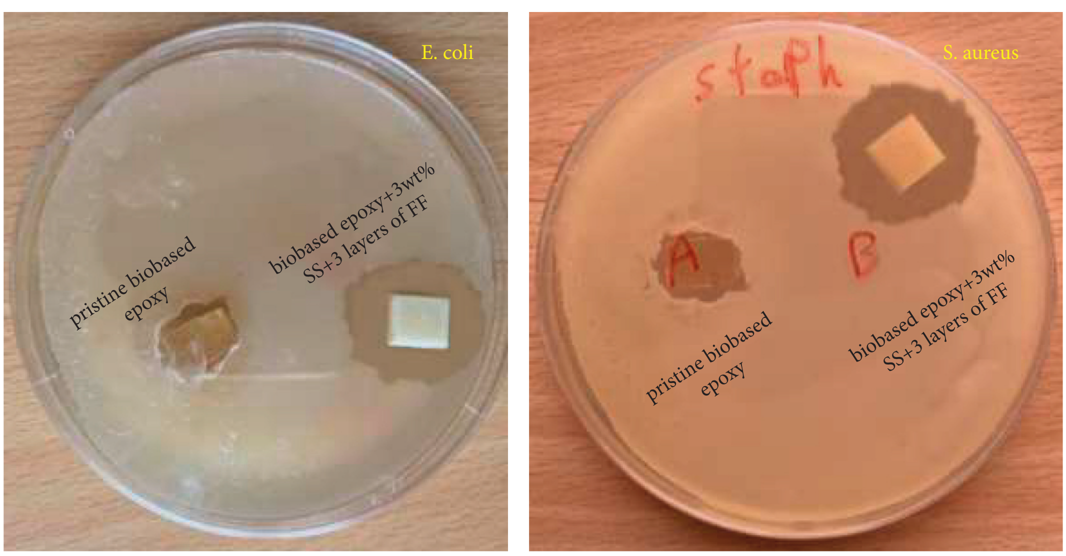


Figure 9: Zone of inhabitation of pristine biobased epoxy and 3 wt% SS-FF-loaded biobased epoxy against *E. coli* and *S. aureus*.

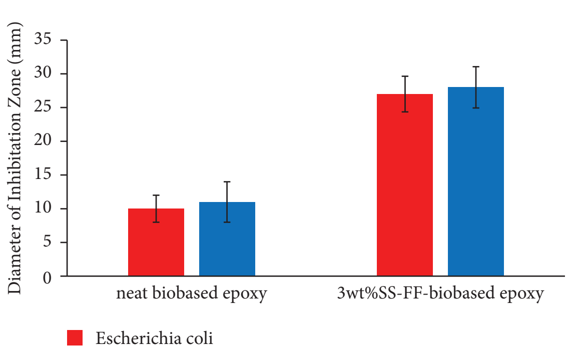


Figure 10: Antibacterial activity of pristine biobased epoxy and 3 wt% SS-FF-loaded biobased epoxy against *E. coli* and *S. aureus*.

different cosmetic applications such as skin exfoliators [36, 37]. The $CaCO_3$ component of SS can be converted to compounds appropriate for biomedical applications [38]. $CaCO_3$ derived from SS can stimulate osteogenesis, and can be used as a substrate for bone replacement [39]. It has been pointed out that calcium phosphate, the primary mineral component of the bone, can be obtained from SS [40]. In general, several hypotheses about the mechanism of antibacterial sensitivity of nanoparticles have been cited. One of which indicates that nanoparticles can interact with the surface of the bacterial cell membrane through electrochemical interactions [41]. Additionally, in as much as the miniature size, nanoparticles can easily penetrate the bacterium cell and can negatively influence cellular activity by deactivating enzymes and other biological molecules

[42]. Moreover, reactive oxygen species can be induced inside the bacterial cell by nanoparticles, resulting in cell death [43].

4. Conclusions

The practicability of fabricating a prosthetic socket from renewable materials, namely, biobased epoxy that was reinforced by plant fibres (FF) and natural-based nanoparticles (SS) was examined in this work. The influence of the incorporation of these natural hybrid reinforcements on the mechanical performance of biobased epoxy was elucidated. Both the tensile and flexural strength of biobased epoxy/FF/SS nanocomposites were revealed to gradually improve with the mass fraction of SS nanoparticles. The mean values of the tensile and flexural strengths of FF/SS/biobased epoxy nanocomposites at 3 wt% of SS were found to be by far higher than those of the blank biobased epoxy. Nonetheless, a reduction in tensile and flexural properties was seen at higher nanoparticle loading of 5 wt%, which could most likely be attributed to the inhomogeneous dispersion of SS nanoparticles within the matrix, and the formation of microvoids. The findings of the finite element method also indicated that the biobased epoxy reinforced by 3 layers of FF and 3 wt% of SS had the lowest values of total deformation. Besides, the composite system was revealed to have excellent antibacterial activity compared to that of the reinforcement-free biobased epoxy. The effectiveness of this composite system as a candidate for transtibial prosthetic sockets needs also to be addressed through the manufacturing and testing of entire prosthetic sockets under daily activity-like circumstances.

Data Availability

The research data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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