Materials Research Express

PAPER • **OPEN ACCESS**

Mechanical and wear behaviour of pulverised poultry eggshell/sisal fiber hybrid reinforced epoxy composites

To cite this article: I O Oladele *et al* 2020 *Mater. Res. Express* **7** 045304

Materials Research Express

OPEN ACCESS

RECEIVED
18 February 2020

REVISED
16 March 2020

ACCEPTED FOR PUBLICATION
1 April 2020

PUBLISHED
14 April 2020

PAPER

Mechanical and wear behaviour of pulverised poultry eggshell/sisal fiber hybrid reinforced epoxy composites

I O Oladele[1,5], B A Makinde-Isola[1], A A Adediran[2,3,5], M O Oladejo[1], A F Owa[4] and T M A Olayanju[2]

[1] Department of Metallurgical and Materials Engineering, Federal University of Technology, PMB, 704, Akure, Ondo State, Nigeria
[2] Landmark University Waste to Wealth and Energy Initiative Group, PMB 1001, Omu-Aran, Kwara State, Nigeria
[3] Department of Mechanical Engineering, Landmark University, PMB 1001, Omu-Aran, Kwara State, Nigeria
[4] Department of Materials and Metallurgical Engineering, Federal University Oye, Ekiti State, Nigeria
[5] Authors to whom any correspondence should be addressed.

E-mail: iooladele@futa.edu.ng **and** dladesoji@gmail.com

Keywords: poultry eggshells, sisal fiber, eco-friendly, biodegradable

Abstract

This study investigates the effects of calcined and uncalcined eggshell particles (ESP) and sisal fiber (SF) on the mechanical and wear properties of eggshell particles/sisal fiber reinforced epoxy composites. Egg shell was processed to obtain calcined and uncalcined egg shell particulate of −43 μm and sisal fibers was extracted by soil retting and treated with NaOH. The composite was developed using the hand lay-up method by blending the selected materials in predetermined proportions. SEM/EDS and XRD of the egg shell particles were carried out while mechanical and wear properties of the developed composites were evaluated. SEM images of the fracture surfaces were also examined. The results showed that eggshell particles contain $CaCO_3$ and the uncalcined ESP/SF reinforced epoxy composite showed improved properties than the calcined ESP/SF composites in most of the tests performed. Also, it was observed that most of the tests gave the best results at different weight fractions. However, 3 wt. % emerged as the weight fraction with optimum values.

1. Introduction

Due to environmental pollution and petroleum depletion, development of composites using natural fibers has been given great attention from scientists and researchers globally recently (Faruk *et al* 2014, Dinesh *et al* 2019). Natural fibers such as sisal and kenaf have been successfully applied in both thermoplastic and thermosets matrices (Jeencham *et al* 2014, Fiore *et al* 2015). Natural fibres and particles are used as reinforcement in polymer matrix composites due to their abundant availability, ease of manufacturing and being less aggressive to manufacturing tools, sustainability and biodegradability compared to the synthetic fibres (Ahmad *et al* 2015, Daramola *et al* 2019). Automobile industries also find the need to lower fuel consumption by lowering the weight of automobiles and reducing dependency on non-renewable resources, such as petroleum based polymers and to source for their replacement by using natural materials which has little or no effect in the environment. Also, hybrid composites from synthetic and natural reinforcements have been developed immensely but not much has been done in the area of using the blend of animal waste with vegetable fibres.

Sisal fibre is one of the hardest natural fibre that is obtained from the leaves of the plant *Agave sisalana*. It is widely cultivated in India, Brazil, East Africa and Indonesia. It has a very good durability and strength and it is also one of the most extensively cultivated hard fibres which makes it easily available (Samuel *et al* 2012, Sahu and Gupta 2018). Reports shows that sisal fibre consists of 43%–56% cellulose, 21%–24% hemicellulose, 7%–9% lignin and 0.6%–1.1% ash (Favaro *et al* 2010). Its high cellulose content makes it to be hydrophilic in nature and hence, difficult compatibilty with hydrophobic polymer matrix usually ensue (Kamaraj *et al* 2018). Thus, chemical treatments are carried out to improve compatibility between the fibre and the polymer matrix (Manoj Kumar *et al* 2019). Alkali treatment was found to be simple and highly effective method for the treatment of sisal fibre (Cai *et al* 2016). It removes lignin, pectin, and hemicelluloses of the fiber leaving high quality fibers with

increased surface roughness which allows better fiber-matrix interface adhesion, fiber fitness, longevity and reduces the diameter of the fibers (Oladele *et al* 2010, Karthikeyan and Balamurugan 2012). The treatment of sisal fiber using alkali (NaOH) however, reduces the composites variability on tensile behavior, decreases stiffness and tensile strength (Silva 2003, Favaro *et al* 2010). Sisal fibres are used as reinforcing material like glass fibres with epoxy. The commercial use of sisal in composites development has increased due to its strength, low density, environmental friendliness and cost-effectiveness. Generally, it can be said that egg shells are of no economic value (Zieleniewska *et al* 2016). However, they are rich in minerals and are therefore regarded as a source of the calcium as one of the predominant constituents (Oliveira *et al* 2013). About several hundred thousand tons of egg shell wastes are produced annually worldwide by the food processing industries and, the utilization cost as well as impact on environment of this waste is high. Wastes can be well and sustainably managed in innovative ways by turning them into useful materials (Dodson *et al* 2012, Tiimob *et al* 2015). Egg shell waste is principal sources of calcium carbonate about 95% and 5% of organic materials such as sulphated polysaccharides, collagen and other proteins and filling of polymers by calcium carbonate improves its mechanical properties and heat stability (Cheung *et al* 2009, Zieleniewska *et al* 2016). These characteristics justify egg shell as a good material for inexpensive, light weight, and low load bearing composite applications as needed in the automotive industry, homes, offices and factories (Amba *et al* 2014). The choice of epoxy matrix in this study is due to its known and excellent adhesion, chemical and heat resistance, good mechanical properties and very good electrical insulating properties. Though they are more expensive than polyester and vinyl ester, they produce stronger and more temperature resistant composite parts/materials (Dagwa *et al* 2015). Natural fiber composites have been studied by many researchers, among them, Oladele *et al* (2014) investigated the effect of water on treated and untreated sisal fibre reinforced polypropylene composites for use in ceiling applications and the result showed that the chemical treatment enhanced the mechanical and water absorption properties of the composites. Easwara *et al* (2017) who carried out a comparative study of the impact strength characteristics of treated and untreated sisal fiber reinforced polyester composites to study the effect of the fibre thickness on the impact strength characteristics for fibre volume of 10, 15, 20, 25 and 30%. Random orientation of fibres was adopted and the specimens were fabricated by using manually operated hot compression moulding technique. The results obtained from the study have shown that impact strength increases with increase in the thickness of the fibre. Araya *et al* (2019) investigated the mechanical properties of sisal reinforced composites as function of epoxy- matrix and they observed that 30 wt% of sisal fiber-reinforced composites have the maximum tensile and flexural strength of 85.5 MPa and 85.79 MPa respectively and the impact strength has been found to be maximum for 40 wt% sisal fiber which is 24.5 kJ m^{-2}. As the result show, and compared with other researcher findings, the mechanical properties are acceptable as substitutes for applications demanding low-cost engineering applications such as automotive internal parts including interior door panel, back seat and body panels. Stanislav and Miroslav (2017) carried out a study on the effect of egg shell microparticles on the mechanical properties of epoxy resin to determine the influence of the filler content in the structure of the composite and he observed that there was enhancement in the tensile strength, elongation at break and hardness and a good wettability between the microparticles and the polymer was observed from the SEM micrograph. Senthil and Madan (2015) studied the mechanical properties and water absorption of egg shell polyester composites as a function of egg shell powder and it was observed that the addition of egg shell powder to the polymer leads to decrease in the tensile strength and modulus of elasticity, while an increase in hardness, % elongation at break and flexural strength was observed. Water absorption behavior of the composites was also investigated and they observed that there was increase in the water absorption property of the composites with increase in exposure time and filler content. In this research, the poultry eggshell was from animal source with ceramic based composition while sisal fibre was from plant source containing celluloses, hemicelluloses and lignin as major constituents which are polymeric in nature. Hence, the research tends to promote the development of natural ceramic-natural polymer based hybrid reinforced epoxy composites for automobile application where biodegradable materials are highly desirable. Selected properties of some vegetable fibres were as shown in table 1 to substantiate the choice of sisal fibre.

2. Materials and method

2.1. Materials

The materials used in this study were Bisphenol A diglycidyl ether epoxy resin (commercial grade), diethylene triamine curative (hardener), eggshell, *Agave sisalana* leaves, sodium hydroxide and distilled water. The epoxy resin and the amine curative were procured from Orkila Chemicals, Ikeja, Lagos State, Nigeria while eggshell and sisal fibre were sourced and acquired from farmland in Akure, Ondo State, Nigeria.

Table 1. Comparison of mechanical properties of natural fibers Naveen *et al* (2018); Pandey *et al* (2010); Guranathan *et al* (2015).

Fiber	Diameter (μm)	Density (g cm^{-3})	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
Sisal	50–200	1.2	460–855	15.5	8
Henequen	—	1.4	500	13.2	4.8
Coir	100–450	1.2	140.5–175	6	27.5
Cotton	—	1.21	250–500	6–10	7
Bamboo	—	0.6–1.1	140–800	11–32	—
Oil palm	240	0.7–1.55	248	3.2	—

Table 2. Formulation of the composites.

Sample (wt.%)	Resin (g)	Hardener (g)	Egg shell (g)	Sisal fiber (g)
Control	120.0	60.0	—	—
3	116.4	58.2	3.6	1.8
6	112.8	56.4	7.2	3.6
9	109.2	54.6	10.8	5.4
12	105.6	52.8	14.4	7.2
15	102.0	51.0	18.0	9.0

2.2. Extraction and treatment of sisal fibre

Sisal fibre was extracted from the plant leaves using soil retting method after which the fermented leaves were disinterred, washed and then sun dried for 5 days. The fibers gotten after extraction from the leaves were treated by putting them in a solution of 2 M NaOH for 4 h at 45 °C. Then the fibers were washed under continuous stream of water and distilled until the complete removal of NaOH residue. Subsequently, the mercerized fibers were dried in the Sun for 2 days during the dry season and chopped to obtain 10 mm size. This method of extraction have been found to produce strong fibres due to the absence of stress induction from beating which is associated with decorticated fibres (Oladele *et al* 2014).

2.3. Egg shell particles

The egg shell was collected and washed thoroughly in water and dried for 2 days in sun. The egg shell was divided into two portions, a portion was left uncalcined and the other portion was put into a crucible and calcined at 900 °C for 1 h to obtain calcined egg shell. The obtained egg shell was ball milled to obtain egg shell powder and particle size analysis was carried out on different portions of egg shell particle to obtain the required particle sizes of undersize (−43 μm). This method was based on the work by Anjali *et al* (2017).

2.4. Development of the composite samples

The composite was developed using the open mould hand lay-up method by incorporating the particles and fibres into the epoxy matrix from 3 to 15 wt% ESP/SF. The epoxy resin and hardener were added in the ratio 2:1. Homogeneous mixture of the epoxy resin, the hardener and the ESP/SF for each test sample was achieved by manually mixing the composition with a glass rod stirrer for 2 min in a polymeric container. The homogeneous mixtures were thereafter introduced into respective moulds designed for each property to be investigated and allowed to cure in air and removed after curing. The cured samples were then tested according to ASTM standards. The formulation and the amount of constituents used were as shown in table 2.

2.5. Characterization and evaluation of the developed composites

2.5.1. XRD spectrum

X-Ray diffraction (XRD) pattern of the eggshell particles were carried out to determine the phases present in the particulate by taking measurements within the range of $2\theta = 10$–$90°$ using a Bruker D2 Phaser® diffraction machine, with a copper K_α radiation source. The machine was operated at generator settings of 30 kV and 20 mA at a temperature of 25°C an the patterns were analysed using PANalytical (v3.0e) X'pert Highscore software.

2.5.2. Flexural test

Three points bending test was used to evaluate the flexural strength of the samples according to ASTM D790. The tests were performed in a universal tensile testing machine Instron series 3369 model. The length, width and thickness of the specimen were 120, 15 and 3 mm, respectively. The tests were carried out using a displacement

control rate at 10 mm min^{-1}. The test speed was 5 mm min^{-1} over a span of 65 mm. Three samples were tested for each composition and the average value was used as the representative values.

2.5.3. Tensile test

The tensile tests were conducted according to ASTM C1557 standard on a universal testing machine Instron series 3369 model. The specimens with dimension 90 × 10 × 5 mm dumbell shape were used. The test was conducted at a crosshead speed of 5 mm min^{-1}. In each case, three samples were used and the average values were reported.

2.5.4. Impact test

An impact test was carried out on the sample using a Charpy impact testing machine in accordance with ISO 179. Samples were cut into the impact test dimension of 80 × 10 × 3 mm and notched at the centre. Samples were placed horizontally on the machine, maintaining a distance of 60 mm between lines of supports. The initial reading of the gauge was taken and then a suspended handle that swings and fractures the sample was released. The final reading was taken after the sample has fractured. For each sample, three test pieces were tested. The average value was taken as the representative value.

2.5.5. Hardness test

Hardness test was conducted on the specimen using a Shore D hardness tester. The samples were placed on the stand of the tester and indented. Three values were obtained by indenting the samples in three different places and the average value was used.

2.5.6. Wear test

The wear procedure follows the standard CS-10 Calibrase or H-16 calibrade. The wear test was carried out with Taber Abrasers, Model ISE AO16. The standard load used was 500 g and a revolution of 150 rpm. Centre hole of 10 mm was made on the sample so as to fix the test piece on the machine. The sample was secured to the instrument platform which is a motor driven at a fixed speed and the values were recorded. Each specimen was a flat and round disc of approximately 100 mm^2 and a standard thickness of approximately 6.35 mm. Wear resistance was measured using the weight difference before and after abrasion (weight loss technique). Care was taken to remove loose particles adhering to specimens during testing, especially prior to weighing. The weight losses of each of the samples were determined using equation (1):

$$\text{Weight loss} = \text{Final weight} - \text{Initial weight of sample} \tag{1}$$

2.5.7. Microscopy characterization

The SEM/EDS of the eggshell particulate and morphological characterization of the composite fracture surface was carried out using EVO MA 15, Carl zeiss SMT. The samples were gold sputtered to improve electrical conductivity.

3. Results and discussion

3.1. SEM/EDS of calcined and uncalcined eggshell particles

Plates 1(a) and (b) shows the SEM images of the calcined and uncalcined eggshell particles. It can be seen from these images that 1(a) contains larger particle sizes than 1 (b). These observed particle sizes can be attributed to the presence of $Ca_2Fe_7O_{11}$ and $CaCO_3$ in image (a) of the calcined eggshell with irregular rhombohedral shape while image (b) which indicates fine particles may be due the presence of calcite crystals ($CaCO_3$) as the only major constituent in the uncalcined eggshell particles. This was in agreement with previous results obtained by Owuamanam (2019) and Boronat *et al* (2015).

The EDS analysis as displayed in table 3 shows that the major constituents of both the calcined and uncalcined eggshell particles are Ca, Al, Mg and O while it was observed that the calcined eggshell particles contain some traces of Fe. This analysis showed that calcination reduced the Ca content and increase the O content compared to the uncalcined eggshell.

3.2. XRD of calcined and uncalcined eggshell particles

The x-ray diffraction patterns obtained for the calcined and uncalcined eggshell particles are shown in figure 1. The diffraction peaks suggested a crystalline phase showing the main material of ES to be calcium carbonate phase in the form of calcite ($CaCO_3$). The major XRD intensity peak is found at 2θ angle of 29.4° and the minor peaks occur at 23.2°, 31.5°, 36.1°, 39.6°, 43.3°, 47.7°, and 48.7° for both ES and LS as also reported in the

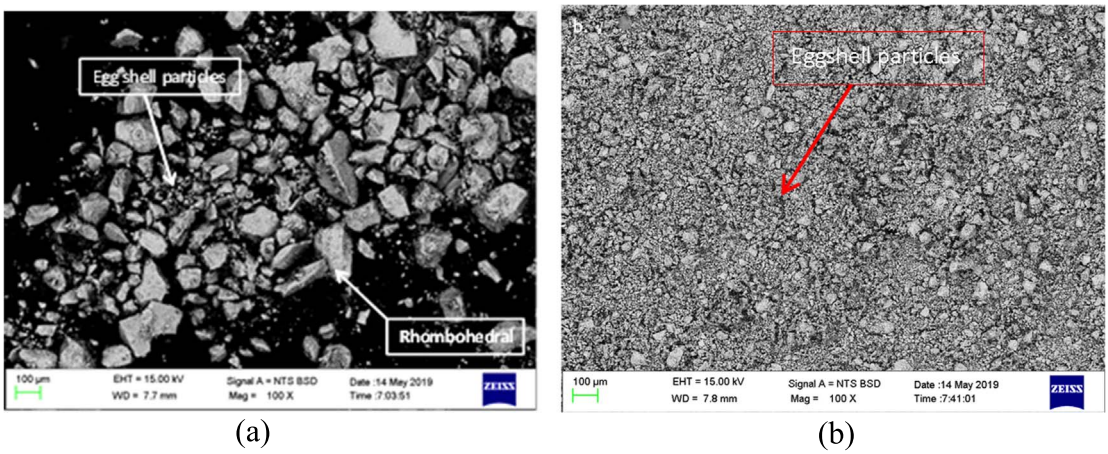

Plate 1. SEM image of (a) calcined, (b) uncalcined eggshell particles.

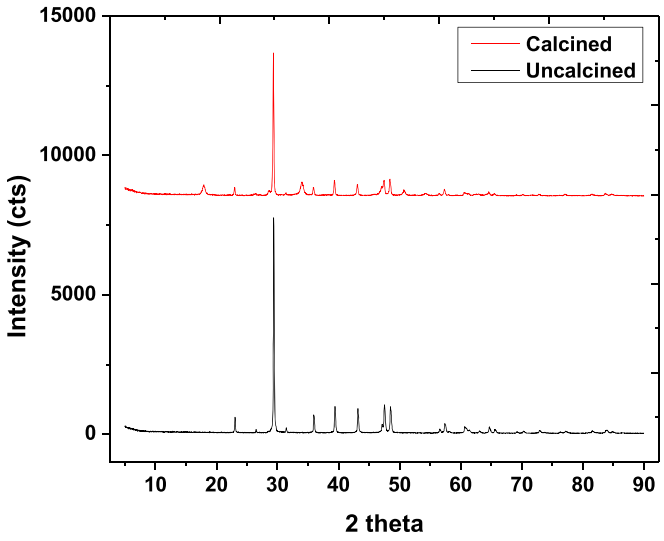

Figure 1. XRD pattern of the calcined and uncalcined eggshell particles.

Table 3. Composition based on EDS analysis of calcined and uncalcined eggshell particles.

%wt. comp.	Ca	Al	Mg	O	Fe	K	Na	Si
Calcined	62.2	0.4	0.5	34.6	2.3	—	—	—
Uncalcined	77.4	0.4	0.24	12.9	—	8.6	0.4	0.06

literature (Rahman *et al* 2014, Tiimob *et al* 2016, Owuamanam 2019). The XRD of calcined and uncalcined eggshell particles are shown in figure 1. The result shows that most of the peaks confirm the presence of $CaCO_3$ in both the calcined and uncalcined eggshell particles. However, the calcined eggshell particles show the presence of Fe in the form of $Ca_2Fe_7O_{11}$. It was also observed that the intensity of $CaCO_3$ in uncalcined eggshell particles is higher than that of calcined particles. These results were in agreement with the analysis of the SEM/EDS in table 3. The elemental compositions of the particulate eggshell were responsible for the compounds formed and detected by XRD analysis.

3.3. Mechanical properties

Figure 2 shows that there was enhancement in the maximum flexural strength of the calcined composites from 6–15 wt% with 15 wt% having the highest flexural strength of 35.82 MPa while for the uncalcined composites, the 9 wt% uncalcined ESP/SF has the highest flexural strength with a value of 36.76 MPa. This implies that the presence of the egg shell and sisal fiber in the epoxy matrix aided better properties in both processes. Alkaline

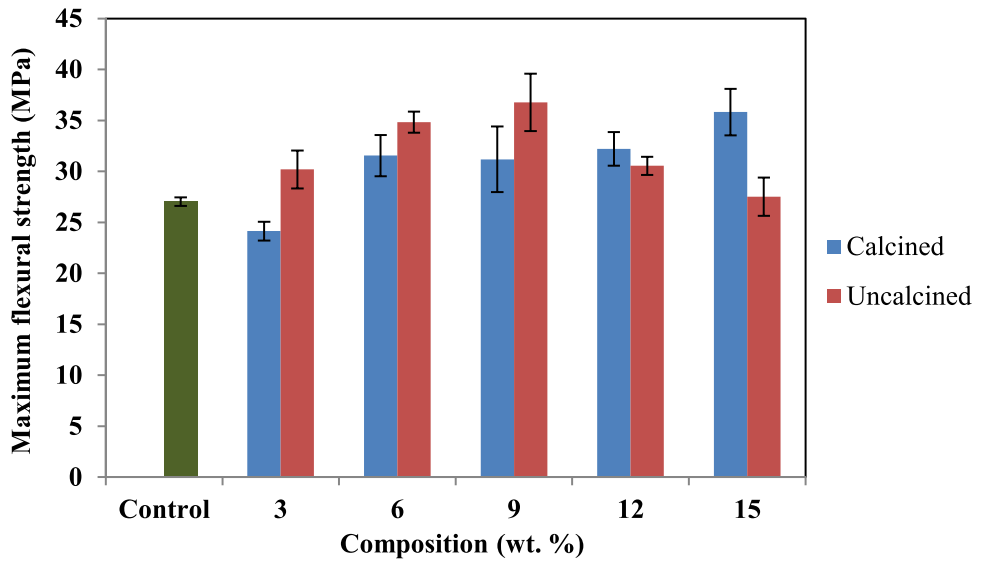

Figure 2. Variation of maximum flexural strength with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

treated sisal fiber has been discovered to be good potential for improved mechanical properties in polymer composites development (Oladele *et al* 2014). However, since sisal fiber is common to both processes, the observed results were due to the influence of the two different eggshell particles on the formulations. From the plot, it was observed that the uncalcined ESP/SF epoxy composite showed better enhancement than the calcined ESP/SF composite in the lower wt% range of 3–9 wt% and this is preferable to the calcined ESP/SF reinforced epoxy composite which was better within the range of 12–15 wt%. Comparing the calcined and uncalcined ESP/SF reinforced epoxy composite, 9 wt% uncalcined ESP/SF reinforced epoxy composite has the highest flexural strength (Kumaran *et al* 2019). The enhancement may be due to the presence of fine particles of $CaCO_3$ as well as organic membranes which contains some functional groups such as hydroxyl, amine and carboxylic groups. These functional groups enhance hydrogen bonding with the epoxy resin in the uncalcined ESP/SF reinforced epoxy composites. This was in agreement with the findings of Owuamanam (2019) and Apalangya *et al* (2014). The observed result was also in accordance with Panneerdhassa *et al* (2016) when they investigate the properties of luffa fiber and ground nut particle hybrid reinforced epoxy composites.

From figure 3, it was observed that the flexural modulus of the calcined ESP/SF epoxy composites increases as the ESP/SF increases from 3–9 wt% and reduced gradually from 12–15 wt% (Dinesh *et al* 2019). It also shows that 9 wt% reinforcement gave the best properties for calcined ESP/SF with a flexural modulus of 327.77 MPa. The uncalcined ESP/SF reinforced epoxy composite samples shows enhancement from 3–15 wt% as ES/SF increases with the composite containing 15 wt% uncalcined ESP/SF having the highest flexural modulus of 371.35 MPa. This trend is in agreement with Kolawole *et al* (2019). The result showed that there was optimum enhancement in both the calcined and uncalcined ESP/SF composites, however, the uncalcined ESP/SF composite showed better enhancement than the calcined ESP/SF composites. This may be due to the fine $CaCO_3$ particles of the uncalcined ES particles as shown in plate 1 which leads to higher flexural modulus.

Figure 4 shows that there was no enhancement in the maximum tensile strength of the composites for all weight fraction used. This may be due to improper transfer of tensile load from the matrix to the reinforcements (Kolawole *et al* 2019). This suggests that the developed composite may not be suitable for tensile strength loading application. However, the uncalcined samples show better enhancements compared to the calcined ones. Previous works have shown that the use of natural limestone fillers causes reductions in tensile strengths of composites when compared to the unreinforced polymer matrix due to the agglomerates serving as stress concentration regions in the composite (Boronat *et al* 2015, Owuamanam 2019).

The variation of tensile modulus with calcined and uncalcined ESP/SF composites was shown in figure 5. The result showed that there was enhancement in the tensile modulus of the developed composites from 3–9 wt% for both calcined and uncalcined ESP/SF. It was also be observed that the composite containing 9 wt% composites gave the highest tensile modulus for both the calcined and uncalcined ESP/SF with a value of 964.76 MPa and 1007.98 MPa, respectively. The enhancement in the tensile modulus may be due to good bonding between the ESP/SF reinforcements and the epoxy matrix. It was observed from the result that the uncalcined eggshell gave better enhancement than the calcined eggshell due to the reasons stated in figure 3. The decrease in tensile modulus from 12–15 wt% may be due to insufficient wetting of the filler at higher

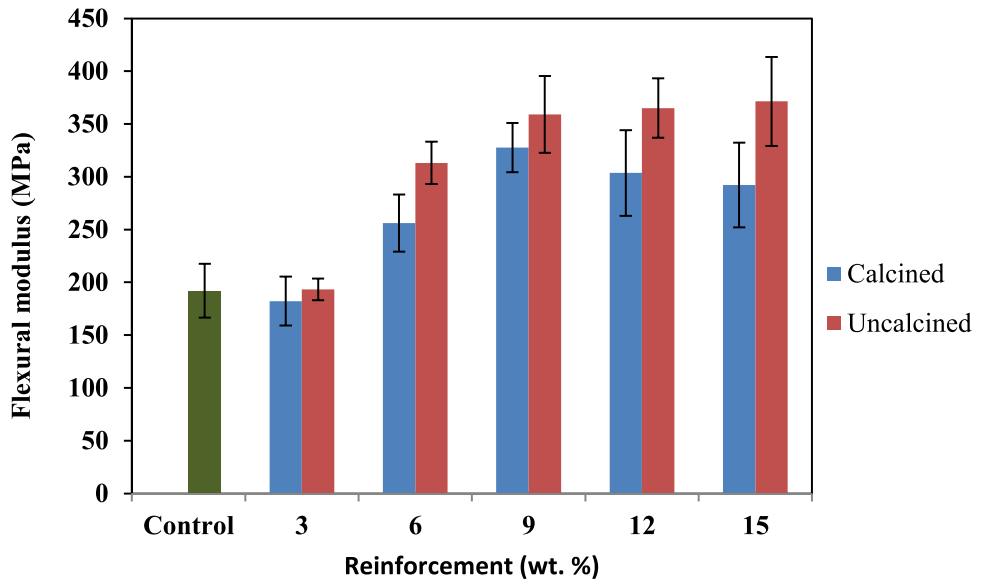

Figure 3. Variation of flexural modulus with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

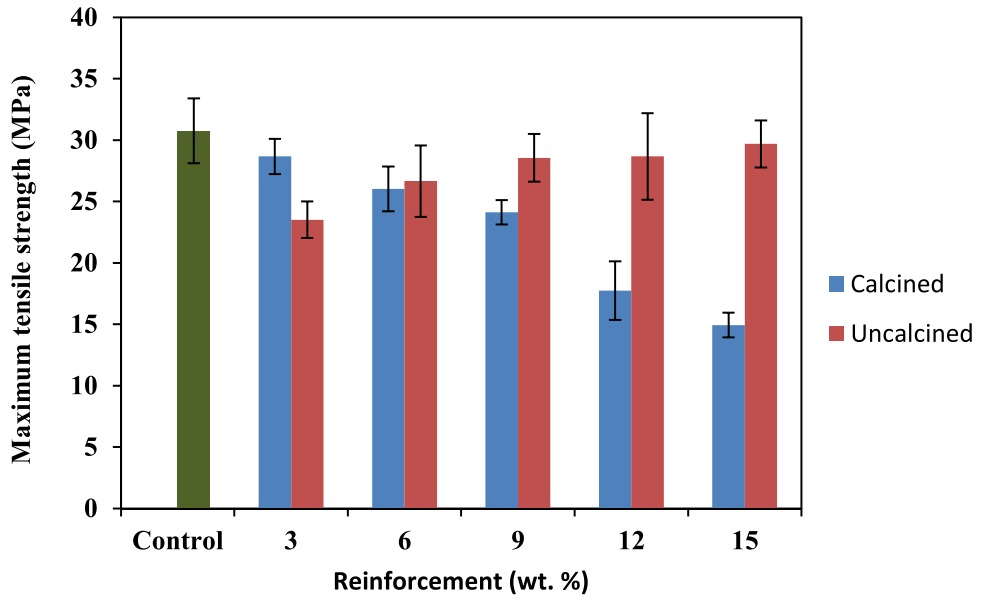

Figure 4. Variation of maximum tensile strength with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

concentration (Kolawole *et al* 2019) or fiber agglomeration (Pickering *et al* 2016). This observation highlights the fact that the incorporation of fillers into the polymer matrix improves the stiffness of the composites within a specified optimum values (Kolawole *et al* 2019).

The variation of impact strength with calcined ESP/SF and uncalcined ESP/SF reinforced epoxy composites was shown in figure 6. The results show that there was enhancement in the impact strength of the developed composites and as the eggshell/sisalfiber increases the impact strength decreases in both the calcined and uncalcined ES/SF samples. This is in accordance with the findings of Hassan and Aigbodion (2015). It was also observed that the composite samples containing 3 wt% ESP/SF for both calcined and uncalcined ESP/SF have the highest impact strength with values of 23.16 and 18.633 J m^{-2}, respectively. However, the composite samples containing calcined ESP/SF performed better than those containing uncalcined ESP/SF. This can be attributed to the volatile matters and moisture that are given off during carbonization as well as the presence of $Ca_2Fe_7O_{11}$. The observation was in line with the submission. On the other hand, the results showed that the incorporation of low weight fraction of ESP/SF from 3–6 wt% increases the ability of epoxy to absorb energy by

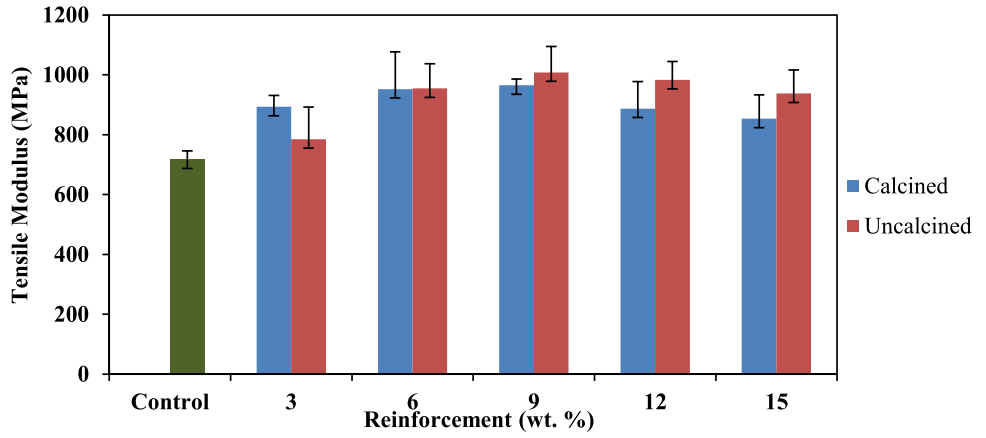

Figure 5. Variation of tensile modulus with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

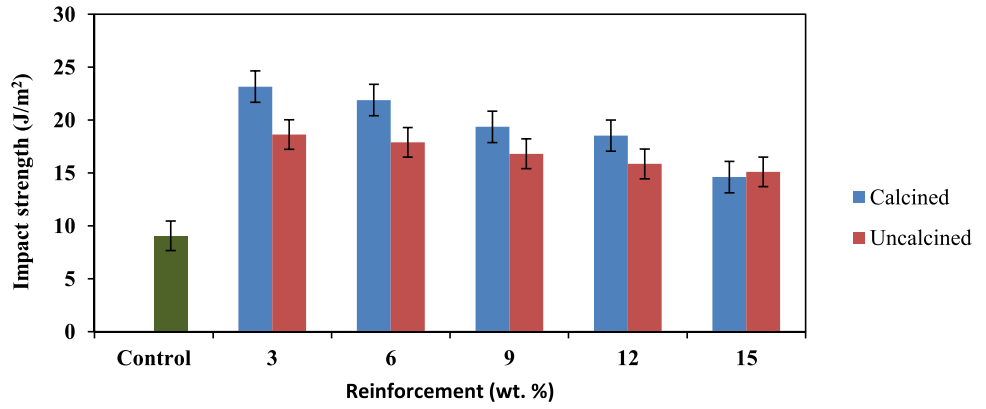

Figure 6. Variation of impact strength with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

increasing toughness. This was in agreement with the findings of Teboho *et al* (2018). The reduction may be as a result of accumulated particles in the composites, which reduces the energy absorbing capacity (Chen and Evans 2008, Owuamanam 2019). As the loading of reinforcement increases, the ability of the composites to absorb impact energy decreases due to the reduction in the ratio of the matrix to particles.

The variation of hardness with the calcined and uncalcined ESP/SF composites was as shown in figure 7. For both calcined and uncalcined samples, initial increase from 6–9 wt% was followed by a decrease from 12–15 wt%. In the calcined samples, it was observed that the hardness was only enhanced for 6 and 9 wt% ESP/SF composites while the uncalcined composites shows enhancement from 6–15 wt%. For the calcined and uncalcined composites, 9 wt% ESP/SF have the highest hardness values of 67.87 HS and 72.25 HS, respectively. The hardness increases with an increase in the mass of the reinforcement used for both the calcined and uncalcined ESP/SF based composites. This was in agreement with the findings of Oladele *et al* (2014) in which it was stated that high density enhances higher material hardness. It was observed that the uncalcined ESP/SF showed better properties compared to the calcined samples.

The variation of wear index with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites was as shown in figure 8. The results showed that there was enhancement in the wear index of both the calcined and uncalcined ESP/SF hybrid composites. This improvement was due to the reduction in coefficient of friction resulting from low friction force. Low frictional force has been reported to be due to the presence of natural fibre which reduced the contact area of resin to abrasion disc (Ramesh *et al* 2014, Shalwan and Yousif 2014). The epoxy resin shows a high wear rate because of the very soft nature of epoxy molecules. Lower wear index depicts high wear resistance. It was also observed for both the calcined and uncalcined ESP/SF that as the reinforcement weight fraction increases the wear index increases from 3–15 wt%. Nevertheless, calcined ESP/SF hybrid composites possess the most improved wear resistance in all variations considered compared to the uncalcined ESP/SF hybrid composites. This improved wear resistance may be due to the

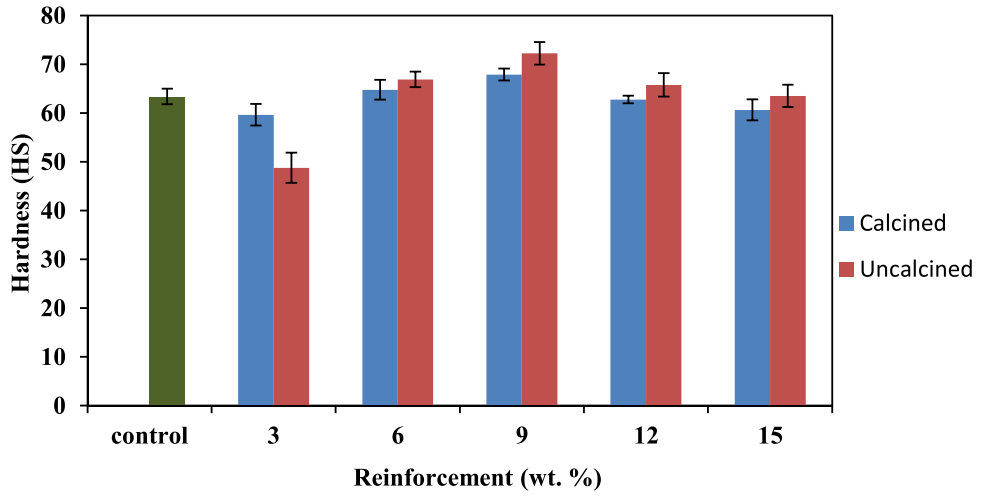

Figure 7. Variation of hardness with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

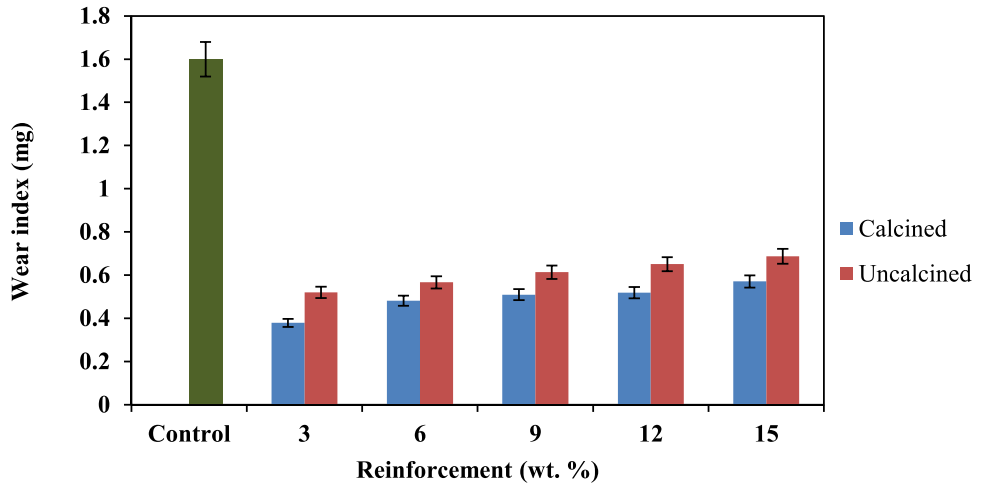

Figure 8. Variation of wear index with calcined and uncalcined eggshell particles/sisal fiber hybrid reinforced epoxy composites and control.

presence of $Ca_2Fe_7O_{11}$. Sample with 3 wt% reinforcement gave the best wear resistance for both calcined and uncalcined samples with values of 0.38 and 0.52 mg, respectively.

3.4. Scanning electron microscopy

Plates 2–4 shows the SEM images of the fractured surfaces of 3, 9 and 15 wt% calcined and uncalcined ESP/SF composites. These were the weight fractions with the optimum properties for the developed composites. Plate 2(a) revealed a well dispersed calcined eggshell particles and sisal fiber within the epoxy matrix with a crack along the fractured surface. The particles and the fiber distribution were not obvious while plate 2(b) revealed noticeable distribution of both the uncalcined eggshell particles and the sisal fiber which depicts agglomeration. This may contribute to the reason why this sample did not emerge the sample with the best properties. Plates 3(a) and (b) revealed more visible dispersion of reinforcements in the matrix compared to plate 2 due to high reinforcement content within the matrix. Plate 3(a) revealed the distribution of the calcined eggshell particles and sisal fiber within the epoxy matrix. The distribution of the reinforcements within the epoxy was similar to what was noticed in plate 2(b) but agglomeration was more highly pronounced in this sample. This may also contribute to the reason why it does not emerge as the best sample in any of the properties. The distribution of the uncalcined eggshell particles and sisal fiber was displayed in plate 3(b). The reinforcements were well dispersed in the epoxy and there is no evidence of fiber/particle touching that characterized agglomerated samples. This was responsible for the emergence of the sample as the best sample in most of the properties considered. This result show good interfacial adhesion and a relatively uniform dispersion of the reinforcements

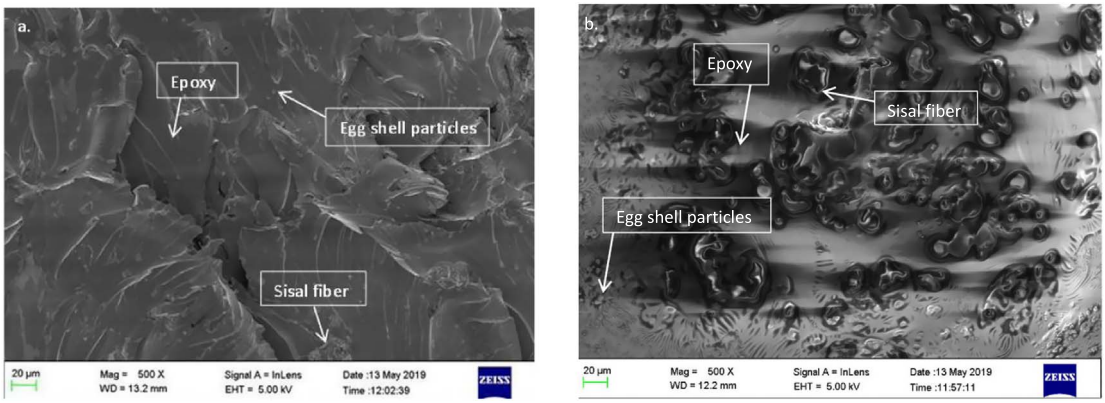

Plate 2. SEM image of 3 wt% (a) calcined ESP/SF, (b) uncalcined ESP/SF reinforced epoxy composite.

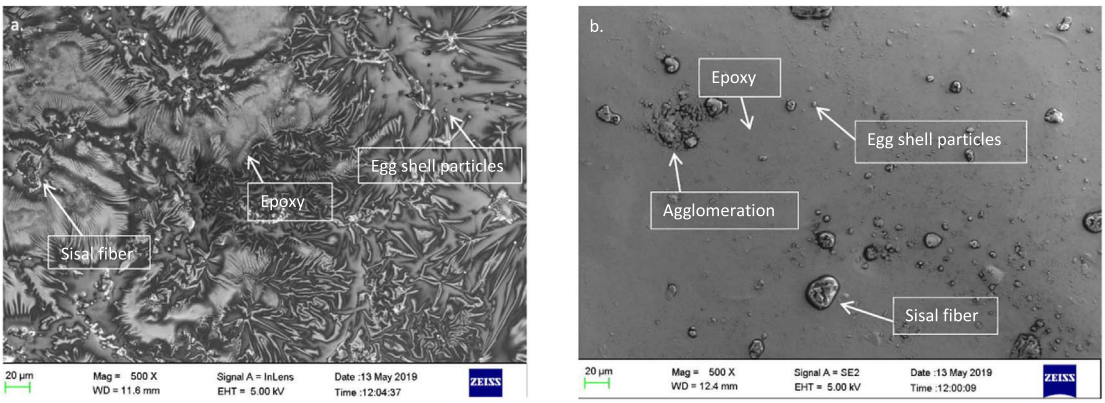

Plate 3. SEM image of 9 wt% (a) calcined ESP/SF, (b) uncalcined ESP/SF reinforced epoxy composite.

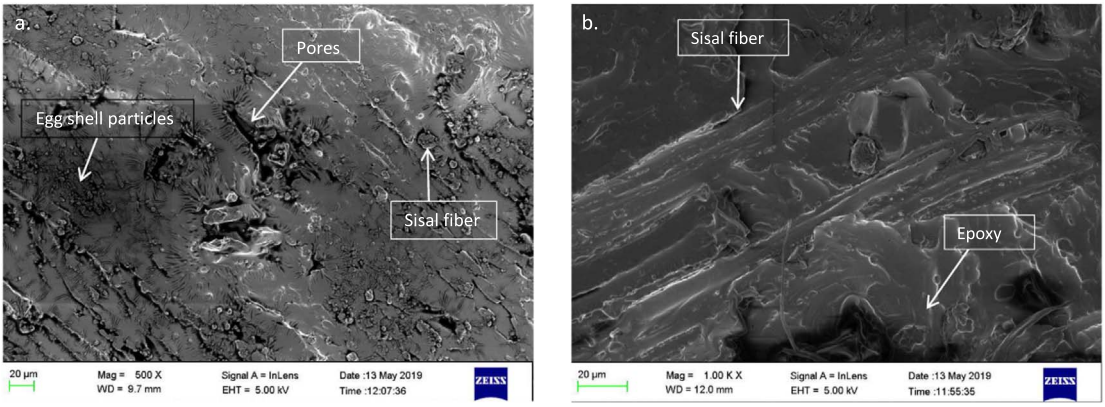

Plate 4. SEM image of 15 wt% (a) calcined ESP/SF, (b) uncalcined ESP/SF reinforced epoxy composite.

in the matrix. Fiber treatments has been reported by Oladele *et al* (2014) to significantly improve adhesion at the fiber/matrix interface and also lead to ingress of the matrix into the fibers, obstructing pullout of the cells. Also, plates 4(a) and (b) revealed similar features as was noticed in plate 3 with more conspicuous distribution of reinforcements in the epoxy. However, cracks were noticed within the epoxy matrix in both images in addition to the traces of agglomeration. Hence, these contribute to the reason why they were not the best in most of the properties considered.

4. Conclusion

Analyses of the processed eggshells revealed that calcination reduced the amount of Ca and increase that of O in eggshells. Hence, high $CaCO_3$ was present in uncalcined eggshell while Fe and $Ca_2Fe_7O_{11}$ was present in calcined eggshell. The mechanical properties were improved by both eggshells. Flexural properties, tensile modulus and hardness were enhanced by uncalcined eggshell particle based composites while impact and wear resistance were improved by the calcined eggshell particles based composites. The optimum weight fractions for the utmost improved properties of the developed composites from uncalcined eggshell particles were from 9 wt% while that of calcined eggshell particles were from 3 wt%.

Acknowledgments

The authors wish to acknowledge the African Academy of Sciences Grant No [ARPDF 18-03] for providing the financial support to carry out the Scanning Electron Microscopy and x-ray Diffraction analysis for this work. Also, the services of Dr M O Bodunrin of the Department of Materials and Metallurgical Engineering, Federal University of Technology Akure, Nigeria was also appreciated. A A Adediran appreciates Landmark University Centre for Research Innovation and Development (LUCRID) for their support.

ORCID iDs

A A Adediran https://orcid.org/0000-0001-9457-1071

References

Ahmad F, Choi H S and Park M K 2015 A review: natural fibre composites selection in view of mechanical, light weight, and economic properties *Macromol. Mater. Eng.* **300** 10–24

Amba C, Pragnya R V and Umasankar V 2014 The micro structural and mechanical property study of effects of egg shell particles on the aluminum 6061 *12th Global Congress on Manufacturing and Management, GCMM 2014. Procedia Engineering* **97** 961–7

Anjali R M, Shivani Bhandari A P, ArpitSaxena S, Nitin Kumar N C, Dheeraj G and Sah P L 2017 Fabrication and mechanical testing of egg shell particles reinforced Al-Si composites *International Journal of Mathematical, Engineering and Management Sciences* **2** 53–62

Apalangya V, Rangari V, Tiimob B, Jeelani S and Samuel T 2014 Development of antimicrobial water filtration hybrid material from bio source calcium carbonate and silver nanoparticles *Appl. Surf. Sci.* **295** 108–14

Araya A B, Anthony N S, Mark K, Yanxi L and Daniel T R 2019 Mechanical properties of sisal-epoxy composites as functions of fiber-to-epoxy ratio *AIMS Materials Science* **6** 985–96

Boronat T, Fombuena V, Garcia-Sanoguera D, Sanchez-Nacher L and Balart R 2015 Development of a biocomposite based on green polyethylene biopolymer and eggshell *Mater. Des.* **68** 177–85

Cai M, Takagi H, Nakagaito A N, Li Y and Waterhouse G I 2016 Effect of alkali treatment on interfacial bonding in abaca fiber-reinforced composites *Composite Part* A **90** 589–97

Chen B and Evans J R G 2008 Impact and tensile energies of fracture in polymer-clay nanocomposites *Polymer (Guildf)* **49** 5113–8

Cheung H Y, Ho M P, Lau K T, Francisco C and David H 2009 Natural fibre-reinforced composites for bioengineering and environmental engineering applications *Composites: Part* B **40** 655–63

Dagwa I M, Adama K, Gadu A and Alu N O 2015 Tensile and hardness property evaluation of kaolin tensile and hardness property evaluation of kaolin/sisal fibre fibre epoxy composite *Nigerian Journal of Technology* **34** 750–60

Daramola O O, Akinwekomi A D, Adediran A A, Akindote-White O and Sadiku E R 2019 Mechanical performance and water uptake behaviour of treated bamboo fibre-reinforced high-density polyethylene composites *Heliyon* **5** 1–6

Dinesh S, Kumaran P, Mohanamurugan S, Vijay R, Singaravelu D L, Vinod A, Sanjay M R, Siengchin S and Bhat K S 2019 Influence of wood dust fillers on the mechanical, thermal, water absorption and biodegradation characteristics of jute fiber epoxy composites *J. Polym. Res.* **27** 1–9

Dodson R J, Hunt J A, Parker L H, Yang Y and Clark H J 2012 Elemental sustainability: towards the total recovery of scarce metals *Chemical Engineering Process* **51** 69–78

Easwara P G L, Keerthi G B S and Velmurugan R 2017 Comparative study of impact strength characteristics of treated and untreated sisal polyester composites *11th International Symposium on Plasticity and Impact Mechanics, Implast 2016 Procedia Engineering* **173** 778–85

Faruk O, Bledzki A K, Fink H P and Sain M 2014 Progress report on natural fiber reinforced composites *Macromolecular Material Engineering* **299** 9–26

Favaro S L, Ganzerli T A and Carvalho A G V 2010 Chemical, morphological and mechanical analysis of sisal fiberreinforced recycled high-density polyethylene composites *Express Polymer Letters* **4** 465–73

Favaro S L, Ganzerli T A and De CarvalhoNeto A G 2010 Chemical, morphological and mechanical analysis of sisal fibre-reinforced recycled high-density polyethylene composites *Polymer Letters* **4** 465–73

Fiore V, Di Bella G and Valenza A 2015 The effect of alkaline treatment on mechanical properties of Kenaf fibers and their epoxy composites *Composite Part B Eng* **68** 14–21

Guranathan T, Smita M and Sanjay K N 2015 A review of the recent developments in biocomposites based on natural fibres and their application perspectives *Applied Science and Manufacturing* A **77** 1–25

Hassan S B and Aigbodion V S 2015 Effects of eggshell on the microstructures and properties of Al–Cu–Mg/eggshell particulate composites *Journal of King Saud University—Engineering Sciences* **27** 49–56

Jeencham R, Suppakarn N and Jarukumjorn K 2014 Effect of flame retardants on flame retardant, mechanical, and thermal properties of sisal fiber/polypropylene composites *Composite Part B Eng.* **56** 249–53

Kamaraj M, Santhanakrishnan R and Muthu E 2018 An experimental investigation on mechanical properties of SiC particle and sisal fibre reinforced epoxy matrix composites *IOP Conf. Ser.: Mater. Sci. Eng.* **402** 012094

Karthikeyan A and Balamurugan K 2012 Effect of alkali treatment and fiber length on impact behavior of coir fiber reinforced epoxy composites *Journal of Scientific & Industrial Research* **71** 627–31 hdl.handle.net/123456789/14634

Kolawole S A, Zakariyaw A A and Omoniyi A O 2019 Preparation and characterization of epoxy filled snail shell thermoset composite *Direct Research Journal of Chemistry and Material Science* **6** 8–13

Kumaran P, Mohanamurugan S, Madhu S, Vijay R, Lenin Singaravelu D, Vinod A, Sanjay M R and Siengchin S 2019 Investigation on thermo-mechanical characteristics of treated/untreated Portunus sanguinolentus shell powder-based jute fabrics reinforced epoxy composites *J. Ind. Text.* (https://doi.org/10.1177/1528083719832851)

Manoj Kumar G, Uthranarayan C, Jones Joseph Jebaraj D and Keerthana S 2019 *Exploration of Flexural, Impact and Morphology Test of Sisal Glass Fiber Reinforced Epoxy Composite Incorporated with Molybdic Acid* **1362** 012016

Naveen J, Jawaid P, Amuthakkannan P and Chandrasekar M 2018 Mechanical and physical properties of sisal and hybrid sisal fiber-reinforced polymer composites *Woodhead Publishing Series in Composites Science and Engineering* **21** 427–30 In book: Mechanical and Physical Testing of Biocomposites, Fibre-Reinforced Composites and Hybrid Composites

Oladele I O, Omotoyinbo J A and Adewara J O T 2010 Investigating the effect of chemical treatment on the constituents and tensile properties of sisal fibre *Journal of Minerals & Materials Characterization & Engineering* **9** 569–82

Oladele I O, Omotoyinbo J A and Adewuyi B O 2014 Mechanical and water absorption properties of sisal-fibre-reinforced polypropylene composites for ceiling applications *The West Indian Journal of Engineering* **37** 29–35

Oliveira D A, Benelli P and Amante E R 2013 A literature review on adding value to solid residues: egg shells *J. Clean. Prod.* **46** 42–7

Owuamanam S I 2019 Fabrication and characterization of bio-epoxy eggshell composites *A Thesis Submitted to the* College of Graduate and Postdoctoral Studies, Department of Mechanical Engineering University of Saskatchewan Saskatoon

Pandey J K, Ahn S H, Lee C S, Mohanty A and Misra M 2010 Recent advances in the application of natural fiber based composites *Macromolecular Materials and Engineering* **295** 975–89

Panneerdhassa R, Gnanavelbabub A and Rajkumarc K 2016 mechanical properties of luffa fiber and ground nut reinforced epoxy polymer hybrid *Composites. Procedia Engineering, 12th Global Congress on Manufacturing and Management, GCMM 2014* **97** 2042–51

Pickering K L, Efendy M A and Le T M 2016 A review of recent developments in natural fiber composites and their mechanical performance *Composites Part A: Applied Science and Manufacturing* **83** 98–112

Rahman M M, Netravali A N, Tiimob B J and Rangari V K 2014 Bioderived 'green' composite from soy protein and eggshell nanopowder *ACS Sustain. Chem. Eng.* **2** 2329–37

Ramesh M, Sri Ananda Atreya T, Aswin U S, Eashwar H and Deepa C 2014 Processing and mechanical property evaluation of banana fiber reinforced polymer composites *Procedia Engineering* **97** 563–72

Sahu P and Gupta M K 2018 *Mechanical, Thermal and Morphological Properties of Sisal Fibres* **455** 012014

Samuel O D, Agbo S and Adekanye T A 2012 Assessing mechanical properties of natural fibre reinforced composites for engineering applications *Journal of Mineral and Material Characterization and Engineering* **11** 780

Senthil J and Madan R P 2015 Preparation and characterization of reinforced egg shell polymer composites *International Journal on Mechanical Engineering and Robotics (IJMER)* **3** 2321–5747

Shalwan A and Yousif B F 2014 Influence of date palm fibre and graphite filler on mechanical and wear characteristics of epoxy composites *Mater. Des.* **59** 264–73

Silva R V 2003 Composite based on polyurethane resin derived from castor oil and vegetable fibers *(PhD Thesis)* São Carlos: Univ of São Paulo

Stanislav P and Miroslav M 2017 Polymeric particle composites based on filler from hen egg-shells *Engineering for Rural Development, Jelgava* **24** 175–80

Teboho C M, Mokgaotsa J M, Tshwafo E M, Linda Z L, Oriel M T and Sandile P S 2018 Sugarcane bagasse and cellulose polymer composites *Sugarcane: Technology and Research* **12** 225–241

Tiimob B, Apalangya V, Samuel T, Jeelani S and Rangari V 2015 Synthesis, characterization and *in vitro* cytotoxicity assessment of eggshell-derived β-$CaSiO_3$ nano biomaterial *British Journal of Applied Science & Technology* **8** 180–93

Tiimob B, Jeelani S and Rangari V 2016 Eggshell reinforced biocomposite-an advanced 'green' alternative structural material *J. Appl. Polym. Sci.* **133** 1–10

Zieleniewska M, Leszczynski M K, Szczepkowski L, Bryskiewicz A, Krzyzowska M, Bien K and Ryszkowska J 2016 Development and applicational evaluation of the rigid polyurethane foam composites with egg shell waste *Polym. Degrad. Stab.* **132** 78–86