

Fracture toughness, flexural, and compressive properties of *Delonix regia* pod-eggshell particle reinforced virgin low-density polyethylene nanocomposites

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Abstract: The valorisation of environmental and agricultural wastes for the development of smart and hygienic cities has attracted increasing research attention. This study investigated the production of biocomposites using virgin low-density polyethylene as a matrix reinforced with eggshell, *Delonix regia* pod and their hybrid particles. The developed composites were evaluated structurally and mechanically. Results obtained reveal an interaction between the low-density polyethylene molecules and reinforcement particles causing formation of new compounds as confirmed by the X-ray diffraction analysis. Enhancements in the fracture toughness, flexural and compressive properties are attributed to the detected new compounds that improve the load bearing capacity of the composites. However, maximum fracture toughness and flexural strength were noted at 4% eggshell particle additions to the low-density polyethylene while the maximum compressive strength was affirmed at 2% eggshell particle addition. The developed materials have a potential application in engineering structures and biomedicine for bone replacement when the obtained properties are compared with properties of materials currently used in the identified areas.

Keywords: Compound, weight fraction, agricultural wastes, bio composites

1. Introduction

Fracture can be defined as the critical stress intensity factor of a sharp crack where propagation of the crack suddenly appears rapid and unlimited (Mouritz, 2012). Fracture toughness describes the resistance of materials to the propagation of flaws under an applied stress (Vaidya & Pathak, 2019). It assumes that the longer the flaw, the lower is the stress needed to cause fracture. The ability of a flaw to cause fracture depends on the fracture toughness of the material which is a key parameter that measures the resistance of a material to crack propagation after it has already initiated in a

material (Bello, 2020). Flexural properties are used as parameters to for selecting materials for a component that will carry load without bending. It is applied in automobile parts like bumper beam and bonnet and in biomedical applications like a material for a bone replacement (femur). Moreover, compressive properties are expressed by a strength and contraction when materials are subjecting to loads that have the same line of action acting towards each other. They are important selection parameters of materials in applications. Both compressive and flexural properties are considered in selecting materials for prosthesis applications.

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This study focuses on the fracture toughness of the low-density polyethylene reinforced with eggshell and *Delonix regia* pod particles in addition to compressive and flexural properties. The study is a continuation of the previous studies on the eggshell-*Delonix regia* pod particles reinforced low-density polyethylene where compressive properties and effects of crack length on fracture toughness properties of the low-density polyethylene-based composites have not been considered (Bello et al., 2022). Moreover, hybrid particle addition containing equal amounts of the eggshell and *Delonix regia* pod particles was considered in this study unlike previous study that has different amounts of both particles at each level of reinforcement (Bello et al., 2024). Knowledge of compressive and flexural properties of newly emerging materials are important part of technical considerations for selecting them for an application in engineering and biomedical structures. For instance, compressive and flexural strengths and strains are fundamental to the materials for bone replacements (Campbell et al., 2012; Choudhary et al., 2020; Wątroba et al., 2019). Therefore, the present study is the complement of the previous study to widen the applications areas of the emerging eggshell-*Delonix regia* pod particles reinforced low-density polyethylene.

Polyethylene belongs to a class of thermoplastic polymer. It is produced by additional polymerisation of ethylene. Its melting point varies from 120 to 130°C. It is ductile, resistant to chemical, nontoxic and insulating. Its fatigue and impact energy are good. Polyethylene is classified based on the density. The grouping includes low-density, very low-density, linear low-density, medium density, high density, cross linking, molecular and ultra-high molecular weight polyethylene. Low-density polyethylene is produced under high pressure condition (100-300 MPa) and a temperature range of 80-300°C. It melts in the range of 105-115°C and has a density range of 0.91-0.94 gcm³. Polyethylene possesses properties (cheap, nontoxic, chemically resistant) favourable to many applications and it is recyclable by remelting/fusing. This justifies its uses in many applications. *Delonix regia* (Caesalpiniaceae) is a stirringly decorative medium sized tree (Figure 1a) that is planted in garden in all the warmer and damper part of India, native to Madagascar. It is known as Gulmohar (Hindi) and flamboyant or Royal Poinciana (English). Pod is elongated, flat, woody, and dehiscent. Seeds are transverse and oblong (Ahmed and Nirmal, 2009). Eggshell is an important structure that forms an embryonic chamber for the developing chick and a container for packaging egg. It is discarded as wastes after hatching or consumption of its contents. Eggshell

(Figure 1b) has been ranked as a solid waste that threatens human and animal survival due to disease outbreak from its malicious odour. Research on conversion of eggshell wastes into useful materials supports United nation Sustainable Development Goal 11. Moreover, eggshell is rich in calcium carbonate which is a hard material with a potential to raise load bearing capacities when used as filler in the polyethylene. Therefore, focus of this research on compressive, flexural and effect of fracture length on fracture properties to complement the untapped areas bridging the gap that exists on eggshell-*Delonix regia* pod particle reinforced low-density polyethylene (Bello et al., 2025; Bello et al., 2023b) will expand future applications of the composites which will in turn enhance usages of eggshell and *Delonix regia* pods in producing new artefacts for societal benefits. This study will help generating a wealth from wastes and removing wastes from environments to create smart cities free from wastes nuisance through productions of innovative materials for society benefits.

2. Materials and methods

2.1. Particle productions

Delonix regia are used as ornamental tree on campus of the Kwara State University, Malete. *Delonix regia* fruits (Figure 2a) when burst release both seeds (Figure 2b) and pods (Figure 2c) used in this study. Eggshells (Figure 2d) were sourced from an eatery in Malete Market of Kwara State. *Delonix regia* pods were rinsed in water to remove dirt particles and dried at any average daily temperature of 25°C for 120 hours. The same process was repeated for the eggshells. The pods were crushed manually using mortar and pestle. Pod flakes obtained after crushing were pulverised using a disc grinder and then ball milled for 70 hours at 10 charge ratios and 92 rpm of the drum rotation.

Dried eggshells were manually crushed through repeated palm folding and opening. The particles obtained were ball milled for 70 hours using the same milling parameters with those of the *Delonix regia* pods.

2.2. Composites specimen preparation

Polyethylene without eggshell nor *Delonix regia* pod particles was prepared to serve as the control to benchmark the low-density polyethylene-based composites. 5 drops of vegetable oil were added to about 160g of the virgin low-density polyethylene and mixed thoroughly. Measurement was taken using Ohaus weighing balance with ± 0.0001 accuracies. The oil

mixed polyethylene pellets (Figure 2f) were poured into the metallic moulds, lined inside with aluminium foil, (Figure 2e) having standard shapes for compressive, flexural and fracture toughness tests. Moulds were placed in a Uniscope laboratory oven (Figure 2g) produced by Surgifriend medicals England. After the oven was switched on, the oil-mixed polyethylene was heated to 150°C at a heating ramp of 30°C per hour. Then, the moulds were brought out of the oven and the softened oil-polyethylene blend were compressed mechanically at a pressure of 2-3 N/mm². Samples were removed from the mould after cooling to room temperature at 25°C and the aluminium foil was removed from each polyethylene sample. Eggshell particles/low-density polyethylene, *Delonix regia* pod particles/low-density polyethylene and eggshell-*Delonix regia* pod particles/low-density polyethylene composites grades were produced. Amount of the low-density polyethylene remained constant at 160g while proportions of eggshell, *Delonix regia* pod and the hybrid particles vary from 2 to 12% by weight (wt%) at an interval of 2wt%.



Figure 1: Picture of (a) *Delonix regia* tree used as ornaments (b) eggshells demonstrating improper disposal of wastes

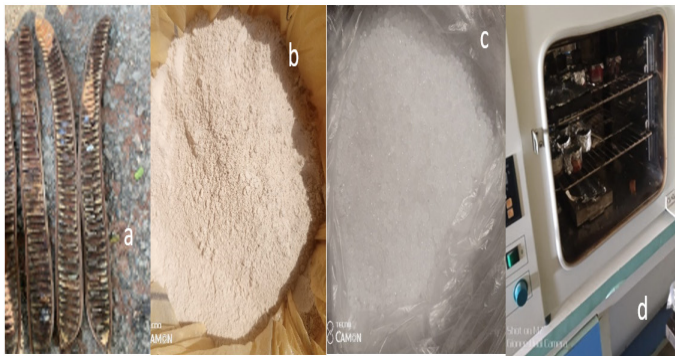


Figure 2: Pictorial demonstration of materials used and procedures for composite preparations: (a) *Delonix regia* pods (b) eggshells (c) low-density polyethylene (d) fused composite ready for compaction

Moreover, composite design formulation for the hybrid particles contains even proportion of both eggshell and *Delonix regia* particles, that is 2wt% hybrid particles contains 1wt% eggshell and 1wt%

Delonix regia pod; 4wt% contains wt% eggshell and 2wt% *Delonix regia* pod with the trend up to 12wt% with 6wt% eggshell and 6wt% *Delonix regia* pod particles as presented in Table 1. Prior to the processing, agglomerates within the particles were addressed by dissolving respective particles in ethanol followed by agitation and evaporation using magnetic stirrer/hotplate set at 110°C. Then, the agitated particles were added to the low-density polyethylene containing 5 drops of vegetables oil to make the surface of the polyethylene pellets wettable to the particle attachments. Particle-oil-low-density polyethylene mixture was thoroughly mixed before pouring into the mould lined inside with the aluminium foil. Then the same process was repeated for the eggshell/low-density polyethylene, *Delonix regia* pod/low-density polyethylene and eggshell-*Delonix regia* pod/low-density polyethylene like the pristine low-density polyethylene without any particle additions to produce composite samples (Figure 2h).

Table 1: Nanocomposite Design Formulation

S/N	Eggshell reinforced low-density polyethylene	<i>Delonix regia</i> reinforced low-density polyethylene	Eggshell- <i>Delonix regia</i> Reinforced low-density polyethylene	
Weight fractions (%)				
	Eggshell nanoparticles	<i>Delonix regia</i>	Eggshell nanoparticles	<i>Delonix regia pod</i>
1	0	0	0	0
2	2	2	1	1
3	4	4	2	2
4	6	6	3	3
5	8	8	4	4
6	10	10	5	5
7	12	12	6	6

2.3. Characterisation of the composites samples

2.3.1. X-ray diffraction analysis

Prepared low-density polyethylene-based composite and control were examined using EMPYREAN PANalytical diffractometer having Pixcel detector with Bragg-Bretano geometry that operates on continuous mode. It has automatic divergence slit type that uses Cu K α radiation which is emitted from Cu anode material at an accelerating voltage of 45 kV and a current of 40 mA. Samples were scanned with the aid of Gonio axis with 2Theta (°) position scale between 0 and 80° at a step size

of 0.0260°. Chemical formula of detected phases in the examined samples were named using X'Pert HighScore Plus software. The examination was conducted at Nigerian Geological Survey Agency, Kaduna in line with (Speakman, n,d).

2.3.1. Scanning electron microscopic analysis

Microstructural features of the pristine virgin low-density polyethylene and that of a composite were probed with the use of the Scanning Electron Microscope, Model JSM 6510A that is equipped with an energy-dispersive X-ray spectrometer (EDX). Samples were firmly fixed to the bottom of the holder using double-sided adhesive tapes and scanning was done with an electron beam that moved at 15 keV. Information on the structure of the examined sample was obtained from signals and images produced by secondary and back scattered electrons in line with (Echin, 2009). The energy-dispersive spectroscopy determines the elemental composition of the samples.

3. Mechanical tests

3.1. Flexural test

Flexural property test was performed on the prepared rectangular low-density based composite samples of 200 mm long (total length), 30 mm thick and 30 mm wide using 3-point bending method. The samples were positioned horizontally on two pivots at 120 mm apart (span length) and loaded at the centre with help of the Testomeric tester until the sample fractured. The analysis was carried out per ASTM D7264 /D7264M-15, 2015 (ASTM D7264 /D7264M-15, 2015).

3.2. Fracture toughness test

20 mm long x 10 mm thick and 30 mm wide rectangular samples for fracture toughness was notched to a depth of 12.3 mm equal to 0.41 crack length to the sample width (a/w) ratio and subjected to a single edge notch compact tension and mode I loading at 0.05 mm/min using Testomeric tester per ASTM D 5045 as reported in (Bello, 2020; Lucas et al., 2011). Before loading, a sharp pre-crack was made at the notch tip with the help of a razor blade. Fracture strengths obtained from compact tension loading were used for calculating the fracture toughness (K_{Ic}) using formula in Equations 1-2 (Perez, 2004). Symbols σ , a , π and w are fracture strength, crack length (metre), π (3.142) and sample width (metre), respectively as used in Equations (1) and (2)

$$K_{Ic} = \alpha \sigma \sqrt{\pi a} \quad (1)$$

$$\alpha = \frac{(2 + \frac{a}{w})w}{\sqrt{aw} (1 - \frac{a}{w})^3} \left([0.5 + 2.62 \left(\frac{a}{w}\right) - 7.52 \left(\frac{a}{w}\right)^2 + 8.3 \left(\frac{a}{w}\right)^3 - 3.16 \left(\frac{a}{w}\right)^4] \right) \quad (2)$$

3.3. Compressive test

Cylindrical samples of the composites and the pristine polyethylene of 1:2 diameter to height ratio were subjected to compressive loading gradually at a velocity of 1.3mm/min per ASTM D 695 using Testomeric tester (Bello et al., 2023a). The analysis was performed at National Centre for Agricultural Mechanization (NCAM), Ilorin. Stresses at yield and peak which the composites can withstand and their Young's modulus were determined automatically by the tester before sample crushing or flattening.

4. Results and discussion

4.1. X-ray diffractograms of Eggshell-Delonix regia Pod Particle Reinforced Low-Density Polyethylene

Detected peaks fitted compounds as observed in Figure 3 confirms that the polyethylene is semicrystalline material. Structural features of n-Heptadecane, Paracyclophane and 1,2- Diphenylcyclohexene belonging to the low-density polyethylene are presented in Tables 2 and 3. It is noted that the compounds appear at varied angles (2theta) with different full width half maximum (FWHM) and interplanar spacings. A monomer (ethylene) of the polyethylene belongs to alkane series which affirms the presence of Diphenylcyclohexene detected in the low-density polyethylene since both belong to alkene series.

Moreover, detection of both n-Heptadecane, and Paracyclophane which belong to alkane series could be linked to compounds of the branched chains to the primary ethylene chains. After eggshell additions to the low-density polyethylene, new compounds (Figure 4) were detected entirely which ascertain a chemical reaction between the low-density polyethylene molecules and eggshell compounds/molecules.

All detected compounds of the low-density polyethylene composites are organic except CaCN₂ (an inorganic compound) and belong to the alkyl and alkane series. CaCN₂ could be attributed to the eggshell while the alkyl and alkane series compounds are already attributed to the chemical reaction of the polyethylene and eggshell phases. The detected compounds of the polyethylene composites have different structural properties regarding the 2theta, FWHM and the interplanar (d) spacings like those of the pristine low-density polyethylene.

In additions, distinctions between corresponding phases of both pristine low-density polyethylene and the low-density polyethylene composites are discernible. Peaks at 18.7977 in Figure 3 are corresponding to peaks at 19.3051 ($^{\circ}2\theta$) in Figure 4. Likewise, are the phases at 21.5959 and 22.2175 and 37.7346 and 38.3029 ($^{\circ}2\theta$). It is noted that the corresponding phases at each pair of 2θ in Tables 2 and 4 have different FWHM. Since FWHM is a measure of peak width, the difference can be interpreted as peak broadness or narrowness. For the first corresponding phases, the FWHM are 0.5136 and 0.4280.

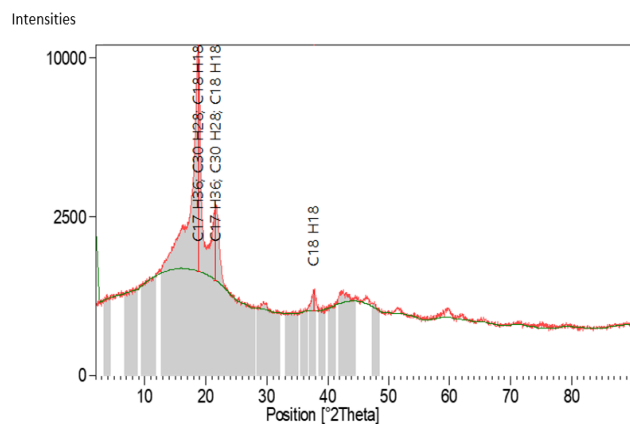


Figure 3: X-Ray Diffractograms of the Low-Density Polyethylene

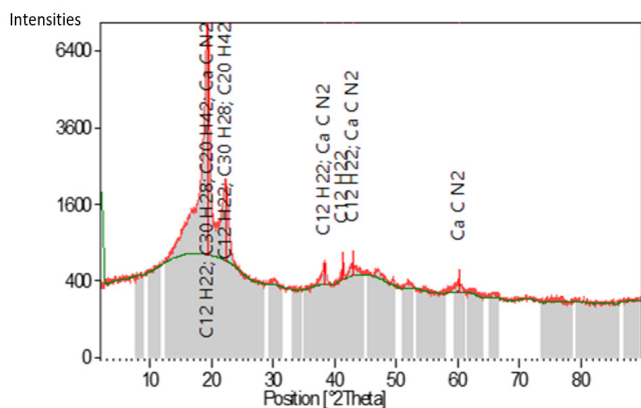


Figure 4: X-ray diffractograms of 4wt% eggshell particle reinforced low-density polyethylene

Table 2: Peak List of the Low-Density Polyethylene

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]
18.7977	9652.03	0.5136	4.72082	100.00
21.5959	2053.21	0.6278	4.11504	21.27
37.7346	302.04	0.4566	2.38402	3.13

Table 3: Pattern List of the Low-Density Polyethylene

Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
36-1590	35	n-Heptadecane	0.000	0.232	C ₁₇ H ₃₆
33-1795	31	Paracyclophane	0.000	0.400	C ₃₀ H ₂₈
27-1924	26	1,2-Diphenylcyclohexene	0.000	0.413	C ₁₈ H ₁₈

This implies that composite peak is narrower than that of the pristine polyethylene. For the second corresponding phases, the FWHM are 0.6278 and 0.6849. This ascertains that the composite peak is broader than that of the pristine low-density polyethylene. The FWHM of the third corresponding phases is the same which confirms peaks of the same width. Moreover, new peaks at 41.3702, 42.9480 and 60.2320 ($^{\circ}$ 2 θ) (Table 4) are noted with broader peaks because of their higher FWHM. Generally, it can be inferred that eggshell addition to the low-density polyethylene causes much higher degree of broadness of the phase peaks than the peak narrowness. The peak broadness and narrowness can be described by compression and tension experienced by the polyethylene molecules when eggshell particles occupy interstitial spaces between them. Such compression and tension can result in straining that can lead to an improvement in the load bearing capacities of the composites. Similar explanation is found in literature (Bello et al., 2021).

Table 4: Peak List of 4wt% Eggshell Particle Reinforced Low-Density Polyethylene

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spac- ing [Å]	Rel. Int. [%]
19.3051	6895.62	0.4280	4.59785	100.00
22.2175	1435.36	0.6849	4.00130	20.82
38.3029	243.90	0.4566	2.34994	3.54
41.3702	363.11	0.4570	2.18252	5.27
42.9480	335.27	1.2909	2.10593	4.86
60.2320	245.80	0.7410	1.53649	3.56

4.2. Microstructural features of the eggshell-Delonix regia pod particle reinforced low-density polyethylene

Pristine low-density polyethylene appears in fibrous form having closed ends (Figure 5). Structures at 1000, 2000, 3000 and 4000 magnifications as observed in Figure 5b-e, respectively, shows that the microstructure has a

continuous solid tube serving as a central body to which tiny polyethylene molecules attach. The structure is sound and free from the defect. Energy dispersive X-ray spectroscopy shows that the low-density polyethylene has carbon which is the major element and O having a very short peak. Structural differences resulting from different spatial configuration is noted with the low-density polyethylene composite. Beside a bended continuous tube, there are background continuous solid structures having different shapes (Figure 6). Structure in Figure 6 is observed to be free from defect, and such structure could be ranked to be of high integrity. In addition to C and O that are noted with the low-density polyethylene, presence of Ca is attributed to the eggshell addition to the polyethylene.

Table 5: Pattern List of 4wt% Eggshell Particle Reinforced Low-Density Polyethylene

Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
04-0223	25	2,2'-Dimethyldicyclopentyl	0.000	0.227	C ₁₂ H ₂₂
33-1795	42	Paracyclophane	0.000	0.816	C ₃₀ H ₂₈
40-1505	41	Eicosane	0.000	0.584	C ₂₀ H ₄₂
85-0920	15	Calcium Cyanamide	0.000	0.539	CaCN ₂

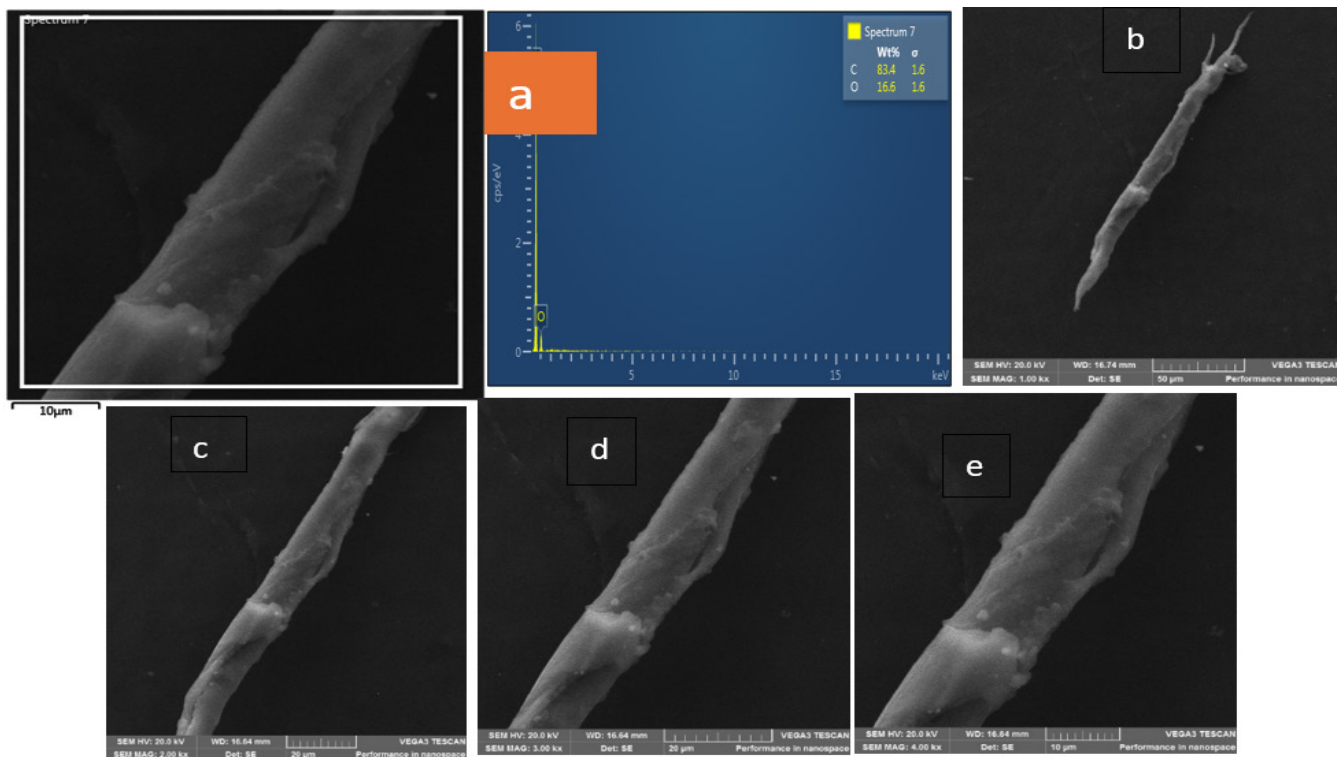


Figure 5: SEM/EDX of the pristine low-density polyethylene (a) chemical composition obtained from the area scanning of the polyethylene structure at resolution of 10 μm (b-d) images captured at different magnifications

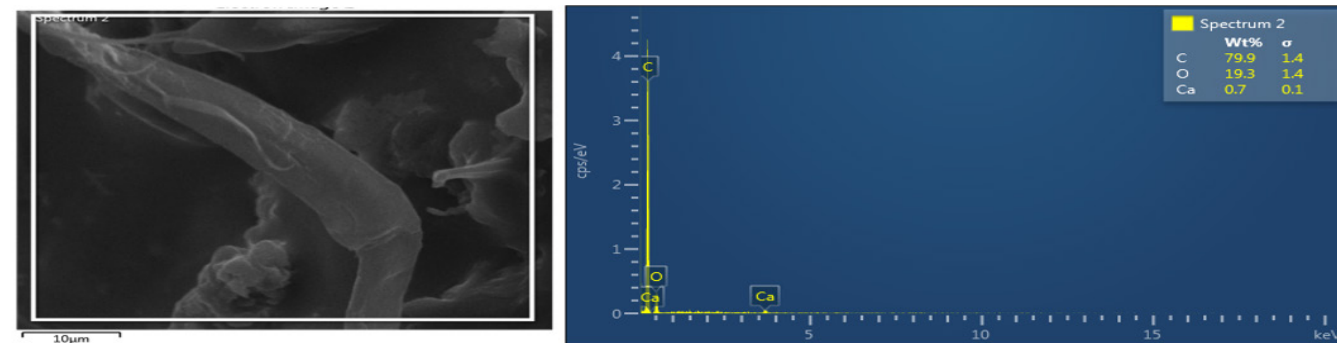


Figure 6: SEM/EDX of the *Delonix regia*-eggshell particle reinforced low-density polyethylene

4.3. Flexural properties of the eggshell-Delonix regia pod particle reinforced low-density polyethylene

Ultimate flexural strength of polyethylene-based composite is greater than that of the pristine polyethylene up to 10wt% additions (Figure 7). Moreover, maximum value of the ultimate flexural strength is noted at 4wt% of eggshell particle additions (Figure 7a), implying that addition of eggshell particles above 4wt% impairs the ultimate flexural strength. Moreover, saturation level is observed at 8wt% for the hybrid low-density

polyethylene composites and 10wt% for the *Delonix regia* pod particle addition. Moreover, 4wt% eggshell particle reinforced low-density polyethylene retains its maximum value of flexural yield strength (Figure 7b), flexural fracture strength (Figure 7c) and Young's modulus (Figure 7d) at 4wt% eggshell addition while the hybrid composites experience a decrease in the yield strength up to 4wt% followed with an increase in the yield strength at 6 and 8wt% but decrease again at higher reinforcement levels at 10 and 12wt% additions (Figure 7b). Their flexural breaking strength and Young's

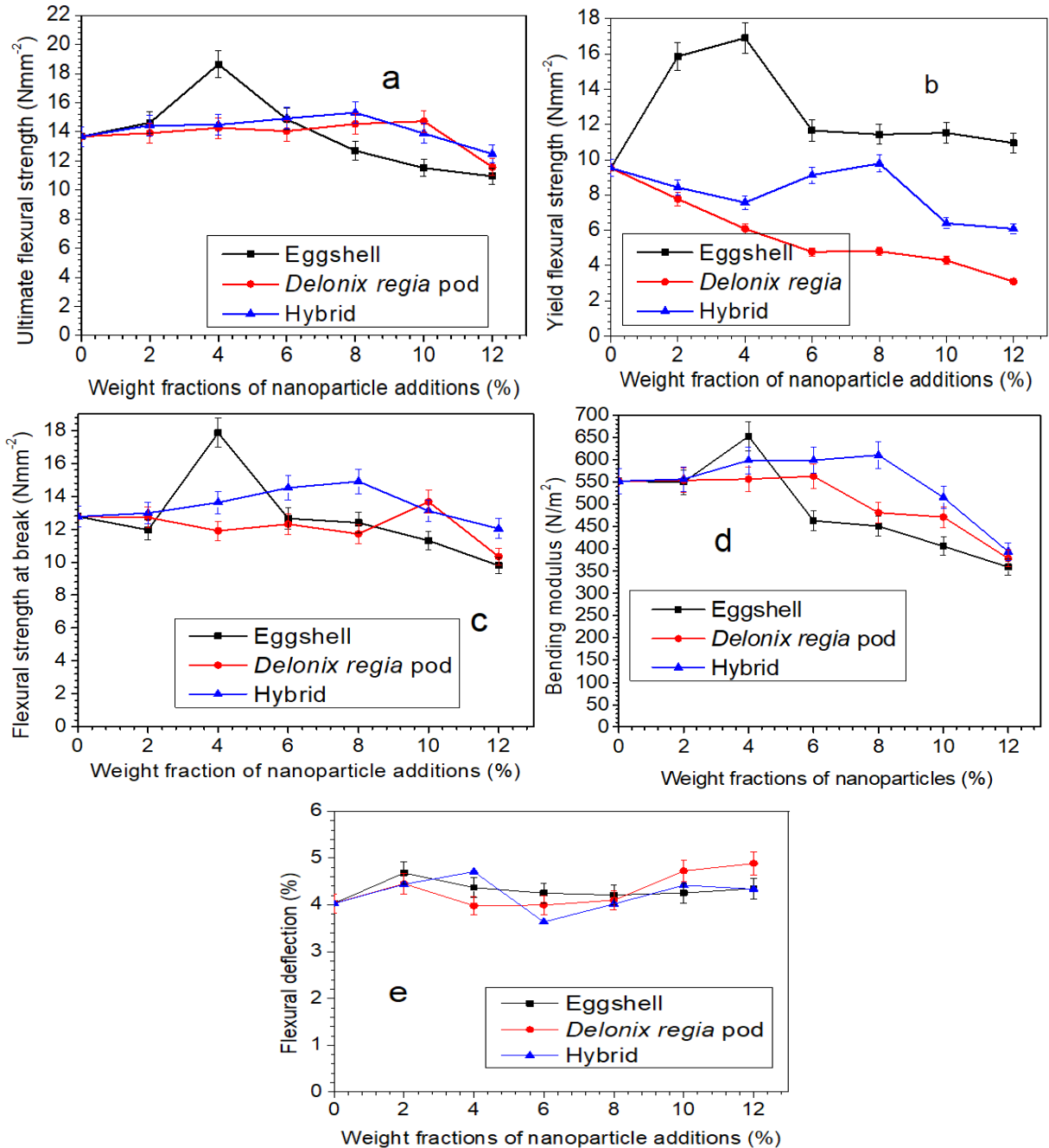


Figure 7: Flexural properties of the low-density based composites: (a) ultimate strength (b) yield strength (c) flexural fracture strength (d) bending modulus (e) flexural deflections

modulus increase up to 8wt% of particle additions whereas the *Delonix regia* pod particle reinforced low-density polyethylene suffers a decrease in the flexural strength from 8wt% of reinforcement particle additions. Increases in flexural deflections (Figure 7e) are noted except at 6wt% of all composite grades. The recorded improvement in the flexural strength due to particle additions to the low-density polyethylene agrees with literature (Bello et al., 2022; Bello et al., 2018; Sutapun et al., 2013; Yallew et al., 2016).

4.4. Fracture toughness of the eggshell-*Delonix regia* pod particle reinforced low-density polyethylene

Fracture toughness of eggshell and hybrid particle reinforced low-density polyethylene increase up to 4wt% above which there is a decrease in the fracture toughness. For the *Delonix regia* pod particle reinforced low-density polyethylene, the fracture toughness increases up to 10wt% (Figure 8a). Generally, it is observed that low-density polyethylene having 4wt% of eggshell particles also has the highest value of fracture toughness. Further study on effects of the crack length on the fracture toughness shows that the higher the crack length, the lower the fracture toughness. This explains the fact that the crack size is related to the fracture toughness of the fracture materials. That is, the higher the crack length, the smaller the resistance of the material is to the crack propagation. Although study of fracture behaviour on low-density polyethylene based composites is scarce in literature, however, the recorded improvement in the fracture toughness in this study agrees with similar findings in literature on epoxy based composites (Bello, 2020; Liu et al., 2015; Ulus et al., 2015) but contradicts the decrease in the fracture toughness reported on high density polyethylene due to particle additions in the article (Bello et al., 2023a).

4.5. Compressive properties of the eggshell-*Delonix regia* pod particle reinforced low-density polyethylene

It is observed that eggshell, *Delonix regia* pod and hybrid particles additions to the low-density polyethylene impair the ultimate compressive strength of the low-density polyethylene as Figure 9a shows. The decrease is easily seen as the wt% of the reinforcement particles increase in respects of the ultimate compressive strength. However, in the case of the Young's modulus, hybrid particles reinforced low-density polyethylene demonstrates slight increase with the weight of the particle additions increases up to 8wt% above which the decrease in the Young's modulus prevails (Figure 9b). Eggshell particle reinforced low-density polyethylene has a maximum value for the Young's modulus at 2wt% addition. Above this level of reinforcement, the decrease in the Young's modulus resumes. Furthermore, there are enhancements in the Young's modulus at 10 and 12wt% *Delonix regia* pod particles additions to the low-density polyethylene. Yield compressive strength curve of the hybrid particle reinforced low-density polyethylene appears parallel to horizontal axis from 4-12wt% particle additions implying that an increase in the yield strength is only noted at 2wt% (Figure 9c).

Delonix regia pod particle reinforced low-density polyethylene shows a slight increase in the yield strength till 6wt%. Then, the decrease in the yield compressive strength prevails afterwards while the eggshell particle reinforced low-density polyethylene displays progressive reductions in the compressive yield strength (Figure 9c). Moreso, compressive strain appears roughly parallel to the horizontal except at 2wt% which implies that the particle additions don't cause a significant change in the compressive strain of the low-density polyethylene (Figure 9d) and the compressive

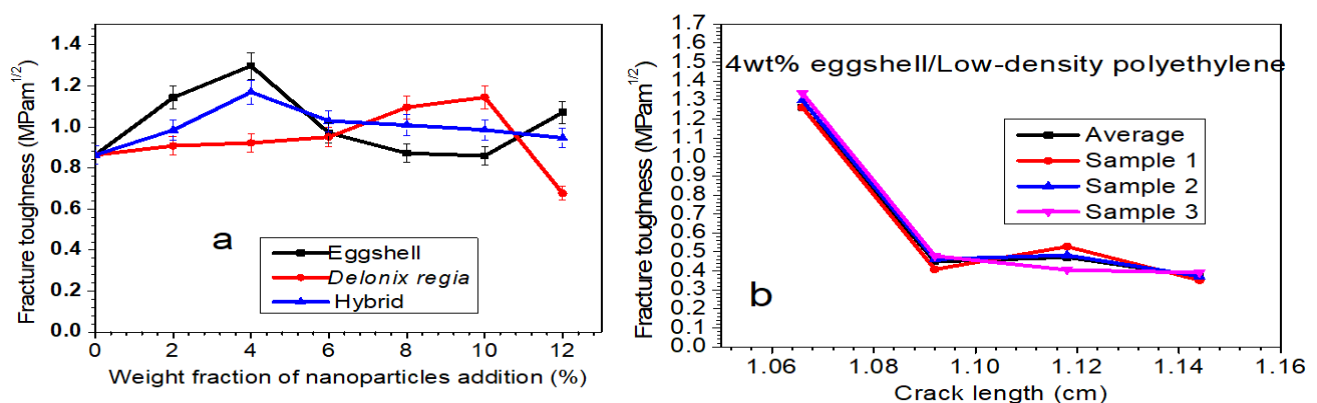


Figure 8: (a) Fracture toughness of the low-density polyethylene-based composites (b) Crack size with the fracture toughness of 4wt% eggshell particle reinforced low-density polyethylene

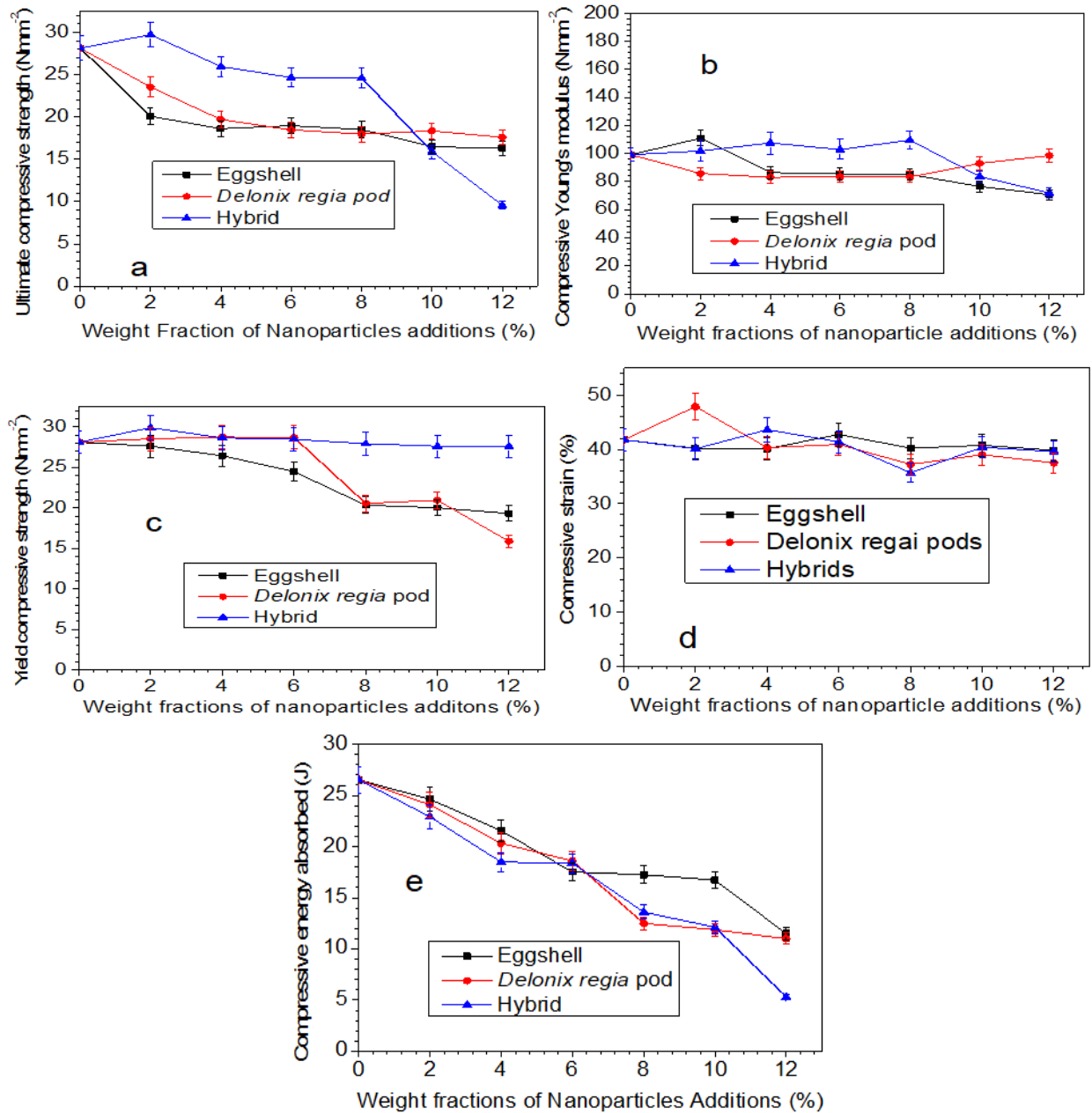


Figure 9: Compressive Properties of the Low-Density Polyethylene-Based Composites

energy absorbed for all the composites shows the same reducing trends throughout the levels of reinforcement (Figure 9e). The differences observed in the compressive properties due to eggshell particles, *Delonix regia* pod and the hybrid particle additions could be attributed to compounds that are present in each of the reinforcing particles. Eggshell contains mainly calcium carbonates which behaviour of its particles in the low-density polyethylene is expected to be varied from the *Delonix regia* pod particles which are mainly organic and that of the hybrid particle containing mixture of organic and inorganic particles.

5. Conclusions

From investigations carried out, this study has the following conclusions:

1. X-ray diffraction study affirms that the low-density polyethylene is a semicrystalline material and there is formation of new compounds due to particle additions.
2. Detection of Ca and Ca compounds in the polyethylene composites by EDX and XRD is attributed to eggshell additions to the low-density polyethylene.

3. SEM affirms microstructural variations between the low-density polyethylene and composites and the differences are linked with spatial configuration of new compounds detected in the low-density polyethylene-based composites.
4. Low-density polyethylene attains maximum values of the flexural properties at 4wt% eggshell particle addition except flexural deflection that has the maximum value at 12wt% addition of the *Delonix regia* pod particles.
5. Fracture toughness of the eggshell and hybrid particle reinforced low-density polyethylene increases up to 4wt% particle addition while *Delonix regia* pod particles reinforced low-density polyethylene has a progressive increase in the fracture toughness till 10wt% addition.
6. The ability of the low-density polyethylene-based composites to resist crack propagation decreases with the crack size or length.
7. There is a progressive reduction in the ultimate compressive strength and compressive energy absorbed as the particle additions increase.

6. Acknowledgement

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References

- Ahmed, J., & Nirmal, S. A. (2009) *An overview of delonix regia: chemistry and pharmacological profile*.
- ASTM D7264 /D7264M-15. (2015) Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials. West Conshohocken, PA: ASTM International.
- Bello, S. A., Agunsoye, J. O., Adebisi, J. A., Raji, N. K., Adeyemo, R. G., Alabi, A. G. F., & Hassan, S. B. (2018) Flexural Performances of Epoxy Aluminium Particulate Composites. *Engineering Journal, Chulalongkorn University*, 22(4), 97-107. doi:10.4186/ej.2018.22.4.97
- Bello, S. A. (2020) Fracture toughness of reinforced epoxy aluminum composite. *Composites Communications*, 17, 5-13. doi:<https://doi.org/10.1016/j.coco.2019.11.006>
- Bello, S. A., Raji, N. K., Kolawole, M. Y., Adebayo, M. K., Adebisi, J. A., Okunola, K. A., & AbdulSalaam, M. O. (2021) Eggshell nanoparticle reinforced recycled low-density polyethylene: A new material for automobile application. *Journal of King Saud University - Engineering Sciences*. doi:10.1016/j.jksues.2021.04.008
- Bello, S. A., Adeyemo, R. G., Balogun, S. W., Adebayo, M. K., & Okunola, K. A. (2022) Nanocomposites: Properties Comparison between Recycled and Virgin Low-Density Polyethylene. In S. A. Bello (Ed.), *Hybrid Polymeric Nanocomposites from Agricultural Waste* (1st ed.). USA: CRC, Taylor & Francis.
- Bello, S. A., Egbanubi, O. E., & Alabi, A. G. F. (2023a) Emerging hybrid particle-reinforced high-density polyethylene nanocomposite for bone replacement. *Polymer Bulletin*. doi:10.1007/s00289-023-04791-9
- Bello, S. A., Raji, N. K., Kolawole, M. Y., Adebayo, M. K., Adebisi, J. A., Okunola, K. A., & AbdulSalaam, M. O. (2023b) Eggshell nanoparticle reinforced recycled low-density polyethylene: A new material for automobile application. *Journal of King Saud University - Engineering Sciences*, 35(6), 406-414. doi:10.1016/j.jksues.2021.04.008
- Bello, S. A., Adebayo, M. K., Adeyemo, R. G., & Popoola, P. A. (2024) Sustainable hybrid nanoparticle reinforced low-density polyethylene: emerging materials for engineering applications. *Iranian Polymer Journal*, 33(7), 965-980. doi:10.1007/s13726-024-01307-8
- Bello, S. A., Olaitan, S. O., Adebayo, M. K., Akinwande, L. O., Kolawole, F. O., Kolawole, M. Y., & Adeyi, T. (2025) Vehicle bumper fascia prototyping using sustainable nanocomposites. *Hybrid Advances*, 10, 100488. doi:10.1016/j.hybadv.2025.100488
- Campbell, A. I., Sexton, S., Schaschke, C. J., Kinsman, H., McLaughlin, B., & Boyle, M. (2012) Prosthetic limb sockets from plant-based composite materials. *Prosthet Orthot Int*, 36(2), 181-189. doi:10.1177/0309364611434568
- Choudhary, R., Venkatraman, S. K., Bulygina, I., Senatov, F., Kaloshkin, S., & Swamiappan, S. (2020) Designing of porous PMMA/diopside bone cement for non-load bearing applications. *Journal of Asian Ceramic Societies*, 8(3), 862-872. doi:10.1080/21870764.2020.1793476
- Echin, P. (2009) *Handbook of Sample Preparation for Scanning Electron Microscopy and X-Ray Microanalysis*. US: Springer.
- Liu, Y., Qu, C.-B., Feng, Q.-P., Xiao, H.-M., & Fu, S.-Y. (2015) Enhancement in Mode II Interlaminar Fracture Toughness at Cryogenic Temperature of Glass Fiber/Epoxy Composites through Matrix Modification by Carbon Nanotubes and n-Butyl Glycidyl Ether. *Journal of Nanomaterials*, 2015, 1-6. doi:10.1155/2015/812061
- Lucas, F. M. d. S., Andreas, Ö., & Robert, A. (2011) *Handbook of Adhesion Technology*: Springer-Verlag Berlin Heidelberg.
- Mouritz, A. P. (2012). 19 - Fracture toughness properties of aerospace materials. In A. P. Mouritz (Ed.), *Introduction*

- to *Aerospace Materials* (pp. 454-468): Woodhead Publishing.
- Perez, N. (2004). *Fracture Mechanics* (1 ed.). US: Springer.
- Speakman, C. A. (n,d) Estimating Crystallite Size Using XRD (pp. 1-105): MIT Center for Materials Science and Engineering.
- Sutapun, W., Pakdeechote, P., Suppakarn, N., & Ruksakulpiwat, Y. (2013) Application of Calcined Eggshell Powder as Functional Filler for High Density Polyethylene. *Polymer-Plastics Technology and Engineering*, 52(10), 1025-1033. doi:10.1080/03602559.2013.769578
- Ulus, H., Ustun, T., Sahin, S. O., & Avci, A. (2015) *Synergistic Effect of Bnnp-CNT Hybridisation on Fracture Toughness of Carbon Fiber Reinforced Epoxy Laminates*. Paper presented at the 8th Ankara International Aerospace Conference, Ankara TURKEY
- Vaidya, A., & Pathak, K. (2019) 17 - Mechanical stability of dental materials. In A. M. Asiri, Inamuddin, & A. Mohammad (Eds.), *Applications of Nanocomposite Materials in Dentistry* (pp. 285-305): Woodhead Publishing.
- Wątroba, M., Bednarczyk, W., Kawalko, J., Mech, K., Marciszko, M., Boelter, G. & Bała, P. (2019) Design of novel Zn-Ag-Zr alloy with enhanced strength as a potential biodegradable implant material. *Materials & Design*, 183, 108154. doi:10.1016/j.matdes.2019.108154
- Yallow, T. B., Kumar, P., & Singh, I. (2016) Mechanical Behavior of Nettle/Wool Fabric Reinforced Polyethylene Composites. *Journal of Natural Fibers*, 13(5), 610-618. doi:10.1080/15440478.2015.1093576

