Engineered Science
DOI: https://dx.doi.org/10.30919/es8d679

Investigation on the Effects of Uncarbonised, Carbonized and Hybrid Eggshell Filler Addition on the Mechanical Properties of Glass Fibre/Polyester Composites

Suhas Kowshik, Sathyashankara Sharma,* Sathish Rao U., Manjunath Shettar, Pavan Hiremath and Arjun Upadhyaya

Abstract

Waste eggshells were procured and processed to obtain uncarbonated eggshells and carbonized eggshells. Four variants of composites viz., unfilled, uncarbonated eggshell filled, carbonized eggshell filled and hybrid eggshell filled were fabricated using the hand lay-up technique. All three variants of filled composites were added 10 wt.% of eggshells. The idea of hybridizing the uncarbonated and carbonized eggshell fillers was also carried out. A mechanical stirrer was utilized for mixing the eggshells with unsaturated polyester resin. All four variants of composites were subjected to tensile and flexural strength tests as per the respective American Society for Testing and Materials (ASTM) standards. The tests showcased that all eggshell-filled composites possess better tensile and flexural strengths compared to unfilled composites. Carbonized eggshell-filled composites exhibited the highest strengths among all the composite variants considered followed by hybrid composites and uncarbonated eggshell-filled composites. The unfilled composites exhibited the least strength. The carbonized eggshell-filled composites showcased 43.9 and 34.14% higher tensile and flexural strengths in comparison with unfilled composites. Microscopic analysis of the failed specimens was conducted using a scanning electron microscope (SEM). The SEM images revealed that the eggshell filler was responsible for crack deviations and crack arrests contributing to the strength of the composites.

Keywords: Carbonized eggshell; Eggshell; Filled composites; Hybrid composite; Polyester.
Received: 13 February 2022; Revised: 20 March 2022; Accepted: 27 March 2022.
Article type: Research article.

1. Introduction

Polymer matrix composites (PMC) have a wide spectrum of applications varying from the automotive sector to construction materials to electrical industries to even children's toys. Polymer composites exhibit properties like high strength, high stiffness, lightweight, and high corrosion resistance. Composites are used in a wide range of industries including aerospace, automotive, construction, and even sporting.[1,2]

Glass fiber reinforced plastics (GFRP) are the most popular and widely used PMCs owing to the low cost and ease of availability of glass fibre.[3] Apart from having good tensile strength, they also possess superior corrosion resistance, resistance to chemicals, resistance to microbial attack, and are lightweight in construction.[4,5]

Fillers are defined as additives in a solid form that differ from the plastic matrix with respect to their composition and structure.[6] Filler materials play a vital role in the polymer industry. Filled polymer matrix composites have the potential to swap orthodox materials.[7] The major advantage associated with the usage of fillers is the reduction in the overall cost of the composites owing to the reduction in the resin requirement.[8] The filler materials used can be either organic or inorganic. Some of the commonly used inorganic filler materials are calcium carbonate, aluminum silicate, calcium sulphate, and alumina trihydrate. Replacement of inorganic fillers with organic fillers such as chicken eggshells has become a new trend among the research fraternity due to their advantageous properties like low densities, high filling levels, non-abrasiveness, renewable nature, and even cost-effectiveness.[9,10]

Chicken eggshells are comprised of a network of protein fibres, associated with crystals of calcium carbonate (96% of shell weight), magnesium carbonate (1%), calcium phosphate (1%), and also organic substances and water.[11,12] Chicken

[1] *Department of Mechanical and Manufacturing Engineering, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal 576104, Karnataka, India.*
**E-mail:* ss.sharma@manipal.edu (S. Sharma)

© Engineered Science Publisher LLC 2022

eggshell comprises an extremely regular ultrastructure which is made up of polycrystalline calcium carbonate ceramic consisting of only one polymorph, calcite.[13,14] Eggshells are potentially polluting industrial waste when not properly managed because they support microbiological action.[11] Most of the chicken eggshell wastes are disposed of in landfills without any pre-treatment and incur considerable disposal costs. The disposal of waste is a very important problem, which can cause risks to public health, contamination of waste, and pollution of the environment.[15] Hence, an environment-friendly solution to the problem is necessary.

The usage of chicken eggshells as a biofiller for polymer matrix composites is one of the solutions to the problem at hand. Various studies have showcased the effective usage of eggshells as filler to improve the properties of polymer composites.[16,17] The current study is focused on understanding the effects of three different variants of eggshell fillers on the mechanical properties of glass fibre/polyester composites.

Although there have been few studies regarding the effectiveness of eggshell filler in polymer composites, there is significant scope for research. The know-how regarding carbonized eggshell fillers is certainly limited and there is space for exploring the area. Further, knowledge of hybrid eggshell fillers, especially the hybridization of carbonized and uncarbonated eggshells is unexplored, proving itself to be a niche area of research and hence has been undertaken in the course of this study.

2. Materials and methods

Two major constituents of composite materials are matrix and reinforcement. Boron-free E-glass fibre was used as reinforcement material and unsaturated polyester was used as the matrix material in the current research work. Chicken eggshell is a potential organic filler material that has the ability to replace the widely used inorganic mineral calcium carbonate.[9,13] Hence, chicken eggshells were used as filler material in the fabrication of composites.

The methodology adopted for the research work is depicted in Fig. S1. Waste eggshells were obtained and processed to obtain uncarbonated and carbonized eggshell fillers. The processing method has been explained in the further sections of the article. The uncarbonated and carbonized eggshell fillers were utilized in the fabrication of composites. Tensile and Flexural tests of the fabricated composites were undertaken as per the ASTM standards D3039 and D7264 respectively.[8,18,19] The failed specimens were further subjected to microscopic analysis to understand and correlate the test results.

2.1 Processing of eggshells

Waste eggshells from various sources like the canteen, restaurants, *etc*. were obtained. These eggshells were then processed to obtain two variants of eggshells viz. uncarbonated and carbonized. The various activities involved in the processing of eggshells to obtain uncarbonated and carbonized eggshell fillers are depicted in Fig. S2 and Fig. S4, respectively. The eggshell fillers obtained after processing were subjected to energy dispersive spectroscopy (EDS) to understand the precise chemical composition.

2.1.1 Eggshell processing to obtain uncarbonated eggshell particles

The waste chicken eggshells obtained were first washed with warm demineralized water to remove any leftover albumen and/or other protein material and/or impurities adhered. The washed eggshells were dried for 24 hours (by natural phenomena) in order to remove the moisture content. The dried eggshells (Fig. S3(a)) were then ground with a mechanical grinder. The ground eggshells were sieved with a mechanical sieve of 45 microns grit size to obtain a particle size distribution between 45 and 37 microns. The obtained eggshell particles were collected and stored in an airtight container as shown in Fig. S3(b).

2.1.2 Carbonized eggshell filler processing

The steps involved in the processing of eggshells to obtain carbonized eggshell filler are shown in Fig. S4. The waste chicken eggshells obtained were washed with warm demineralized water to remove any leftover albumen and/or other protein material and/or impurities that adhered. The washed eggshells were dried for 24 hours (by natural phenomena) in order to remove the moisture content. In order to carbonize, the eggshells were first chipped and filled into a graphite crucible. Further, powered graphite was added into the eggshell-filled crucible and packed thoroughly. The decomposition temperature of calcium carbonate is known to be at around 900 °C. Hence, a muffle furnace was used to heat the eggshells and graphite powder-filled crucible to a temperature of 850 °C for one hour in order to obtain decent decomposition without build-up of soot on the eggshell. Later, the carbonized eggshells were ground with a mechanical grinder and sieved through 45 microns grit size sieve using a mechanical sieve to obtain a particle size distribution between 45 and 37 microns. The carbonized eggshell as shown in Fig. S5 were collected and stored in an airtight container.

2.2 Fabrication of composites

Four different variants of composites viz., Unfilled (UF), Uncarbonized Eggshell (UCES) filled, Carbonized Eggshell (CES) filled, and Hybrid (HY) were prepared. The details regarding the variants and their compositions are shown in Table 1. The hand lay-up technique was employed in the fabrication of the four variants of composites.

E-glass fibre mats consisting of chopped glass fibre strands with random orientation having 450 GSM were utilized as reinforcement. The matrix material utilized for fabrication was polyester resin. Both glass fibre and polyester resin were supplied by Mookambika Poly Products, Udupi, Karnataka, India. Methyl-Ethyl-Ketone Peroxide (MEKP) was used as the hardener and mixed in the hardener-to-resin ratio of 12:1.

 © Engineered Science Publisher LLC 2022

Table 1. Composite variants and their composition.

Composite variant	Glass fibre (wt.%)	Polyester (wt.%)	Uncarbonized eggshell (wt.%)	Carbonized eggshell (wt.%)
Unfilled (UF)	34	66	0	0
Uncarbonized eggshell filled (UCES)	34	56	10	0
Carbonized eggshell filled (CES)	34	56	0	10
Hybrid eggshell filled (HY)	34	56	5	5

Chicken eggshell fillers were mixed with polyester resin using a mechanical stirrer. Table 1 shows the proportion of mixing. Composites of 300 mm × 300 mm dimensions and an average thickness of 3 mm were prepared.

2.3 Tensile test

Mechanical properties in terms of the ultimate tensile strength (σ) and Young's modulus (E) were determined as per the ASTM D3039 standard. Each specimen was cut to the dimension 250 mm × 25 mm × 3 mm. Five specimens of each variant were subjected to the tensile test. The specimens were conditioned as per ASTM D3039 standard. The specimens were tested at a test speed of 2 mm/min using the Zwick-Roell universal testing machine. The ultimate strength of the specimen was determined by the maximum load carried before failure. The stress-strain responses were recorded to determine young's modulus for each specimen.

2.4 Flexural test

Mechanical properties in terms of flexural strength and flexural modulus were determined through a three-point bending test as per ASTM D7264. Each specimen was cut to the dimension 128 mm × 13 mm × 4 mm. Five specimens of each variant were subjected to the three-point bending test. The pre-test conditions were maintained for the specimens. The specimens were tested at a test speed of 2 mm/min using the Zwick-Roell universal testing machine. The flexural strength and flexural modulus were obtained from the testing. Further, the necessary stress-strain curves were plotted and compared.

2.5 Microscopic analysis

Microscopic analysis of eggshell filler material was performed using Energy Dispersive Spectroscopy (EDS). The microscopic analysis of the failed specimens using a scanning electron microscope (SEM) was also conducted to understand the failure mechanisms of all four variants of composites. Zeiss EVO MA18 instrument was used to perform the SEM imaging. Specimens were diced to dimensions of 10 mm length and 6 mm width in order to equip them in the specimen holder of the microscope. Further, in order to produce effective imaging, the specimen surface should be electrically conductive. Thus, the specimen surfaces were deposited with silver using a low vacuum sputtering system.

3. Results and discussions

3.1 Tensile test

The tensile test revealed that the highest average tensile strength of 118 MPa was exhibited by carbonized eggshell-filled composites followed by the hybrid composites with an average strength of 94 MPa. The uncarbonated eggshell-filled composites showcased a strength of 88 MPa and the least strength of 82 MPa was exhibited by the unfilled composites. The stress-strain curve representing the maximum recorded values concerning the four variants of composites is shown in Fig. 1. The obtained stress-strain curve indicates the brittle nature resulting in the catastrophic failure of the prepared composite as there exist no yield points before the fracture mechanism.[19] The specimens subjected to tensile testing are shown in Fig. S6. It can also be observed in Fig. 1 that carbonized eggshell-filled composites exhibit the least strain at the break while the unfilled composites exhibit the highest strain at the break. The reduction in strain with an increase in tensile stress could be due to the increase in stiffness of the composite owing to the addition of filler.[20]

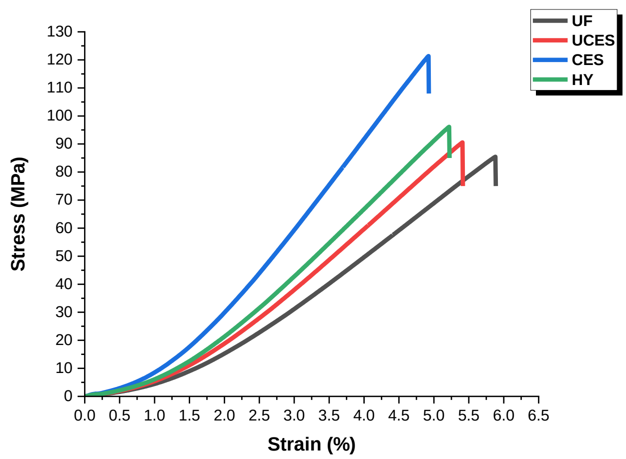

Fig. 1 Stress-strain variation of composite variants.

The variation of tensile strengths among all the composite variants is represented in Fig. 2(a). The uncarbonated eggshell-filled composite variant exhibited an 8% increased tensile strength in comparison to unfilled composites. Carbonized eggshell-filled composites exhibited 43.9% higher tensile strength in comparison with unfilled composites. Hybrid composites showcased 14.6% higher tensile strength compared to unfilled composites.

© Engineered Science Publisher LLC 2022

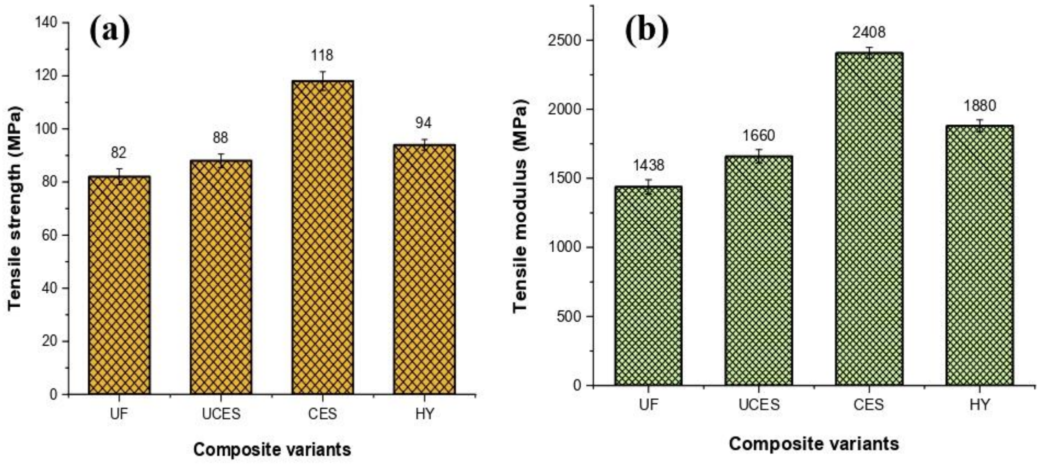

Fig. 2 Mechanical characterization of composite variants (a) Tensile strength and (b) Tensile modulus.

It is evident from the results that all the eggshell-filled composites possess higher strength compared to unfilled composites. The higher strength could be due to the transfer of tensile stress from the polyester matrix to eggshell filler material.[21] Furthermore, the whole eggshells were processed to obtain small particles of eggshells which essentially means that the surface area of the particles increased. Hence, the strengthening effect of the carbonized eggshell-filled composites can be attributed to the better-increased surface area of particles in the matrix compared to the uncarbonated eggshells.[22]

Similar studies to understand the effect of eggshell filler on polymer composites have been conducted by various researchers. However, an overall upward trend could be observed in all the studies. The improvement in strengths varied from 10 to 25%. Our study also shows a similar upward trend of increase in tensile strength of 8%. However, the higher values observed in other studies could be due to different fibres and polymer resins considered, the amount of fibre, and the resin and filler material considered. The addition of eggshell filler proved to be beneficial for the tensile strength of composites.[8,21] Studies conducted using carbonized eggshell filler have also showcased an improvement in the tensile strength of the composites. The improvement in strengths varied from 25 to 45% which is similar to the results obtained in our study.[17,21,23] However, few studies have also shown a reduction of strength due to filler addition. The reasons for the contradicting trends could be the high amount of filler leading to void creation and agglomeration of filler particles due to the non-homogeneous distribution of filler in resin.[22,24]

The variation of tensile modulus among all the composite variants is represented in Fig. 2(b). The highest average tensile modulus of 2408 MPa was exhibited by carbonized eggshell-filled composites followed by the hybrid composites with an average tensile modulus of 1880 MPa. The uncarbonated eggshell-filled composites showcased a modulus of 1660 MPa and the least tensile modulus of 1438 MPa was exhibited by the unfilled composites.

The uncarbonated eggshell-filled composites exhibited 15.4% higher tensile modulus in comparison to unfilled composites. Further, carbonized eggshell-filled composites and hybrid composites respectively showcased 67 and 30.7% higher tensile modulus in comparison with unfilled composites. The increase in tensile modulus could be attributed to the increased brittleness and stiffness of the filled composites.[25]

3.2 Flexural test

The flexural test revealed that the highest average flexural strength of 165 MPa was exhibited by carbonized eggshell-filled composites followed by the hybrid composites with an average strength of 145 MPa. The uncarbonated eggshell-filled composites showcased a strength of 136 MPa and the least strength of 123 MPa was exhibited by the unfilled composites.

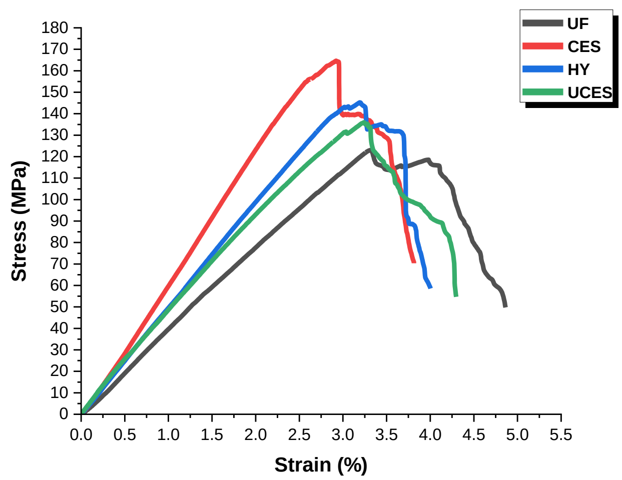

Fig. 3 Stress-strain variation comparison of composite variants.

The graph of stress vs. strain representing the maximum recorded values concerning the four variants of composites is shown in Fig. 3. The obtained stress-strain curve indicates that carbonized eggshell-filled composites exhibit the least strain while the unfilled composites exhibit the highest strain.

© Engineered Science Publisher LLC 2022

Further, it can also be observed that all eggshell-filled composite variants exhibit reduced strain in comparison to unfilled composite. The specimens subjected to flexural testing are shown in Fig. S7. The reduction in strain with an increase in stress could be due to the increase in stiffness of the composite due to the addition of eggshell filler.[26]

The variation of flexural strengths among the composite variants is represented in Fig. 4(a). The uncarbonated eggshell-filled composite variant exhibited an 11% increased flexural strength in comparison to unfilled composites. Further, carbonized eggshell filled and hybrid composites respectively exhibited 34.14% and 17.88% higher flexural strength in comparison with unfilled composites. The strengthening effect of the eggshell-filled composites can be attributed to the better-increased surface area of filler particles in the matrix.[22]

Few similar studies conducted to comprehend the effects of eggshell filler addition on the mechanical properties of composites revealed similar results with respect to flexural strength. The studies announced the improvement in the flexural strength of composites in the range of 11 to 20%. Our study has shown an improvement of 11% which shows a similar upward trend and similar values too. However, higher strengths were observed when the authors considered higher fibre content.[19,26] Further, studies considering carbonized eggshells have also shown a similar trend of a significant increase in flexural strength of 20 to 40%.[18,19] However, few studies have also shown a reduction of strength due to filler addition. The reasons for the contradicting trends could be the high amount of filler leading to void creation and agglomeration of filler particles due to the non-homogeneous distribution of filler in resin.[22,24]

The highest average flexural modulus of 5689 MPa was exhibited by carbonized eggshell-filled composites followed by the hybrid composites with an average tensile modulus of 1880 MPa. The uncarbonated eggshell-filled composites showcased a modulus of 1660 MPa and the least tensile modulus of 1438 MPa was exhibited by the unfilled composites. The variation of flexural modulus among all the composite variants is represented in Fig. 4(b). The addition of uncarbonated eggshells exhibited 14.03% higher flexural modulus in comparison to unfilled composites. Further, carbonized eggshell filled and hybrid composites respectively exhibited 52.64 and 25.48% higher flexural modulus in comparison with unfilled composites. The increase in flexural modulus could be attributed to the increased brittleness and stiffness of the filled composites.[22,25]

3.3 Modes of failure

The various modes of failure of the tensile test specimens can be explained with the help of Fig. S6. The failure modes of all the specimens according to ASTM D3039 are documented in Tables 2-5 documented below. Fig. S6(a) shows the failed test specimens of unfilled composites. Table 2 showcases the modes of failures of unfilled specimens. Fig. S6(b) shows the failed test specimens of uncarbonated eggshell-filled composites and Table 2 showcases the failure modes of the same. Further, Fig. S6(c) shows the failed test specimens of carbonized eggshell-filled composites and Table 4 represents the failure modes of the same. Fig. S6(d) shows the failed test specimens and showcases the failure modes of the same. Specimens in both unfilled and filled composites have failed at the top portion. The formation of the main chap at the top portion would have caused the stress concentration to lead to failure. The formed main chap at the top portion inhibits the further development of chaps to other portions of the specimen.[27] It is also observed that the specimens have failed at the middle portion (within the gauge length). Some specimens have also displayed angular failure in the gage region. This could be due to the anisotropic behavior of the composites owing to the usage of randomly oriented fibre.[19,28]

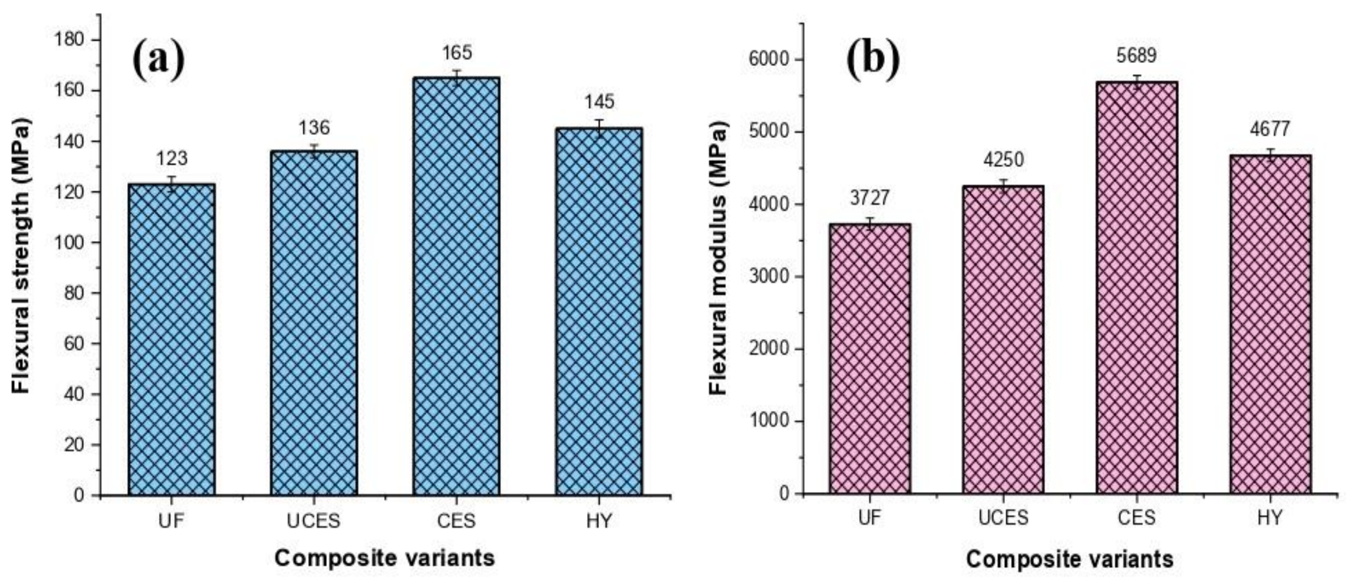

Fig. 4 Mechanical characterization of composite variants (a) Flexural strength and (b) Flexural modulus.

© Engineered Science Publisher LLC 2022

Table 2. Modes of failure of Unfilled specimens.

Specimen number (top to bottom in Fig. S6(a))	Mode of failure as per the ASTM standard	The first character (Failure type)	The second character (Failure area)	Third character (Failure location)
1	LGM	Lateral	Gage	Middle
2	LGM	Lateral	Gage	Middle
3	AGM	Angular	Gage	Middle
4	LAT	Lateral	At grip	Top
5	LAT	Lateral	At grip	Top

Table 3. Modes of failure of uncarbonated eggshell-filled specimens.

Specimen number (top to bottom in Fig. S6(b))	Mode of failure as per the ASTM standard	First character (Failure type)	Second character (Failure area)	Third character (Failure location)
1	LGM	Lateral	Gage	Middle
2	AGM	Angular	Gage	Middle
3	LGM	Lateral	Gage	Middle
4	AAT	Angular	At grip	Top
5	LGM	Lateral	Gage	Middle

Table 4. Modes of failure of carbonized eggshell-filled specimens.

Specimen number (top to bottom in Fig. S6(c))	Mode of failure as per the ASTM standard	First character (Failure type)	Second character (Failure area)	Third character (Failure location)
1	LAT	Lateral	At grip	Top
2	LAT	Lateral	At grip	Top
3	LGM	Lateral	Gage	Middle
4	LGM	Lateral	Gage	Middle
5	AGM	Angular	Gage	Middle

Table 5. Modes of failure of hybrid eggshell-filled specimens.

Specimen number (top to bottom in Fig. S6 (c))	Mode of failure as per the ASTM standard	First character (Failure type)	Second character (Failure area)	Third character (Failure location)
1	LGM	Lateral	Gage	Middle
2	LGM	Lateral	Gage	Middle
3	LGM	Lateral	Gage	Middle
4	LAT	Lateral	At grip	Top
5	LAT	Lateral	At grip	Top

3.4 Microscopic analysis

3.4.1 Energy dispersive spectroscopy (EDS)

The eggshell fillers obtained after processing were subjected to Energy Dispersive Spectroscopy (EDS) to understand the precise chemical composition. The EDS results clearly showcase the major presence of various elements such as calcium, carbon, oxygen, magnesium, and phosphorous in carbonized eggshell filler as shown in Fig. 5, and the presence of Calcium, Carbon, Oxygen, and Iron can be noted through the EDS results of carbonized eggshell filler as shown in Fig. 6.

It can be clearly observed that the amount of calcium, as well as carbon present in the carbonized eggshell filler, is higher than in the uncarbonized eggshell filler. It is known that calcium in the form of calcium carbonate is present in carbonized eggshells. During carbonization, Calcium carbonate ($CaCO_3$) upon heating to 850 °C decomposes to calcium oxide or calcite (CaO) and carbon dioxide (CO_2). Further, the carbon added in the form of graphite contributes to the reversible reaction of calcination and hence more amount of calcium can be observed with carbonized eggshells. This increase in calcium in the form of calcium carbonate would have contributed to the improvement of composite strengths. Further, the addition of graphite has increased the carbon content in the carbonized eggshell which would have further contributed to the strength of the composites.[29]

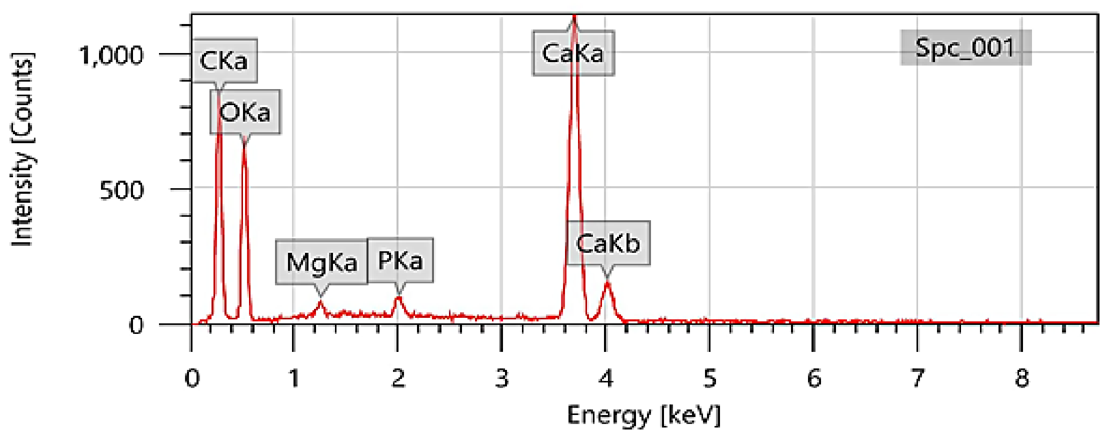

Fig. 5 EDS result of uncarbonated eggshell filler.

 © Engineered Science Publisher LLC 2022

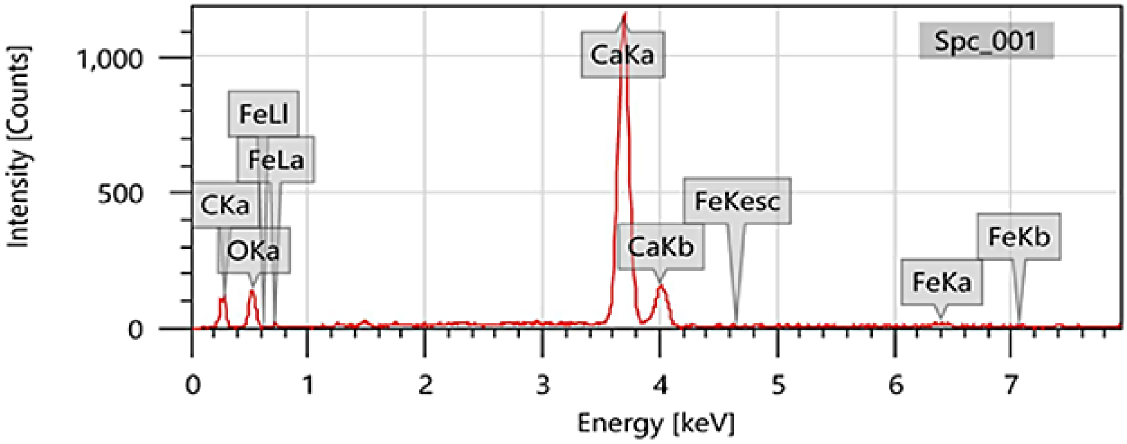

Fig. 6 EDS result showing the composition of uncarbonated eggshell filler.

Table 6. EDS results show the composition of uncarbonated and carbonized eggshell fillers.

Element	Line	Uncarbonized eggshell filler		Carbonized eggshell filler	
		Mass%	Atom%	Mass%	Atom%
C	K	28.21 ± 0.17	39.16±0.24	37.11 ± 1.00	52.35 ± 1.42
O	K	49.02 ± 0.60	51.09±0.63	52.03 ± 0.50	29.30 ± 0.28
Mg	K	0.59 ± 0.05	0.41±0.03	-	-
P	K	0.98 ± 0.04	0.53±0.02	-	-
Ca	K	21.20 ± 0.21	8.82±0.09	9.46 ± 0.16	17.78 ± 0.30
Fe	K	-	-	1.40 ± 0.15	0.57 ± 0.06
Total		100.00	100.00	100.00	100.00

3.4.2 Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) was utilized to investigate the fracture mechanisms in the four composite variants. Fig. 7(a) through (h) displays the SEM images of all variants of composites obtained from the test specimens that were subjected to the tensile test. It is quite evident from Fig. 7(a) that the crack propagation at multiple locations is not hindered in the unfilled composite specimen. This can be attributed to the absence of filler material. It was seen earlier through the composite test results that the unfilled composites exhibited the least tensile strength. The unrestricted crack propagation due to the absence of filler material can be recognized as the reason for the lower strength of the composites. Further, Fig. 7(b) showcases the poor interfacial bonding of matrix and reinforcement in the unfilled composite specimen which could be another reason for their lower strength.

Figure 7(c) clearly shows the crack restriction and deviation at multiple points along the line of failure in carbonized eggshell-filled composite specimens. This could be attributed to the presence of eggshell filler.[27,30] The presence of filler particles can also be observed in the same SEM image. Fig. 7(d) shows the adherence of chunks of polymer onto the fibre strands exhibiting good interfacial bonding between the matrix and reinforcement in an uncarbonated eggshell-filled composite specimen. The above-mentioned reasons could be attributed to the higher strength of the composite.

The crack deviation and arrest in the carbonized eggshell-filled composite are clearly revealed in Fig. 7(e). The presence of eggshell filler particles can also be observed from the image. Hence, it can be deduced that the crack deviation and arrest could be due to the addition of eggshell filler material. Also, the good interfacial bonding of the matrix and fibre in the carbonized eggshell-filled composite can be observed in Fig. 7(f). The physical bonding between matrix and filler depends on various parameters like particle size, particle shape, surface area, and so on.[28] The particles in this case are irregularly shaped which has contributed to the higher level of adhesion with the resin. Further, various studies have shown that lower particle sizes contribute to higher strength owing to the increase in surface area.[11,23] The process of carbonization of eggshell filler would have reduced the particle size and hence increased the surface area of the particles leading to better interfacial adhesion.[31] The above-mentioned reasons could have led to the higher strength of the carbonized eggshell-filled composites.

Figures 7(g) and 7(h) reveal the crack deviation and arrest due to the addition of eggshell filler material in a hybrid eggshell-filled composite specimen. The presence of eggshell particles can also be clearly seen in the SEM images. It can also be seen that the crack arrests and deviations are superior to uncarbonated eggshell-filled composites but inferior to carbonized eggshell-filled composites. The presence of

© Engineered Science Publisher LLC 2022

carbonized eggshells in addition to the uncarbonated eggshells have improved the interfacial bonding in the composite due to increased surface area and surface roughness of the eggshell particles due to the process of carbonization. Thus, it can be

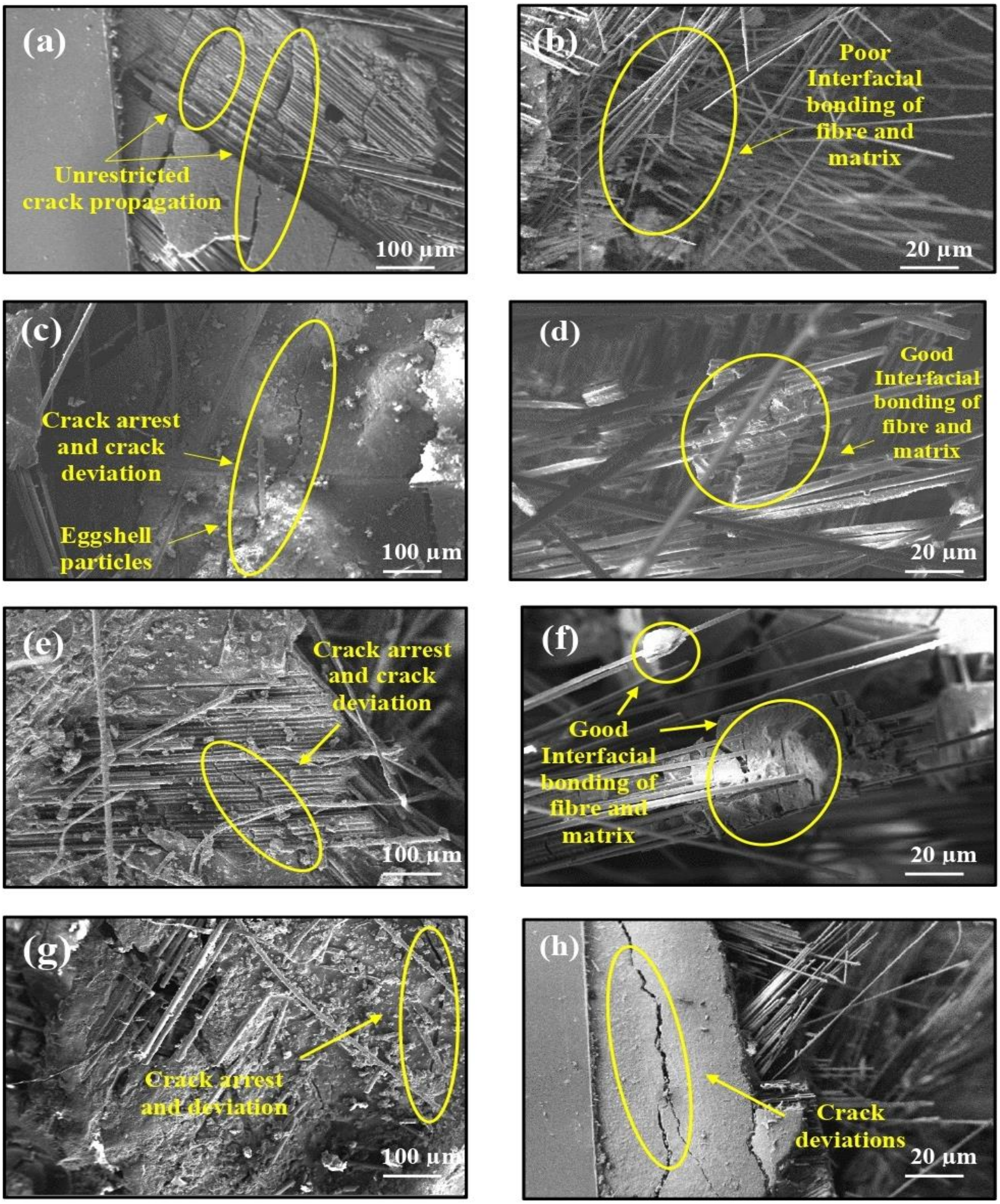

Fig. 7 SEM images of composite variants (a) Unrestricted crack propagation in unfilled composite (b) Poor interfacial bonding of fibre and matrix in unfilled composite (c) Crack arrest and deviation in uncarbonated eggshell-filled composite (d) Good interfacial bonding in uncarbonated eggshell filled composite (e) Crack arrest and deviation in carbonized eggshell filled composite (f) Good interfacial bonding in carbonized eggshell filled composite (g) Crack arrest and deviation in hybrid eggshell filled composite (h) Crack deviations in hybrid eggshell filled composites.

 © Engineered Science Publisher LLC 2022

correlated to the mechanical test results wherein hybrid composites exhibit better properties than uncarbonated eggshell-filled composites but are inferior compared to carbonized eggshell-filled composites.

It is evident from the composite test results that all the eggshell-filled composites possess superior mechanical properties in comparison with unfilled composites. The higher strength could be due to the transfer of tensile stress from the polyester matrix to eggshell filler material.[21] Furthermore, the whole eggshells were processed to obtain small particles of eggshells which essentially means that the surface area of the particles increased. Hence, the strengthening effect of the carbonized eggshell-filled composites can be attributed to the better-increased surface area of particles in the matrix compared to the uncarbonated eggshells.[22]

The eggshell filler particles are homogenously distributed which attributes to the crack arrest and deviation. Similar results have been documented by other researchers in which the authors have attributed the crack arrest to the homogenous distribution of eggshell filler particles.[32] Interfacial bonding of eggshell filler and polyester resin can also be seen in the SEM images. Interfacial bonding of filler and matrix plays a vital role in the strengthening of material.[33,34] Furthermore, our literature study revealed that the obtained tensile and flexural strengths follow the same trend as the research conducted by other researchers.[19]

4. Conclusions

In summary, four variants of composites viz., unfilled, uncarbonated eggshell filled, carbonized eggshell filled, and hybrid eggshell filled were fabricated using a hand lay-up technique in which glass fibre was used as reinforcement, unsaturated polyester was used as matrix material and eggshells were used as filler material. The composites were subjected to mechanical characterization. The results of the tensile and flexural tests showed that the addition of eggshells as filler material contributes to the improvement in the mechanical properties of glass fibre/polyester composites. SEM images showcased the crack arrests and deviations in eggshell-filled composites which proved to be a confirmation of the test results. Further, the studies also revealed that carbonized eggshell-filled composites possess the highest tensile and flexural strengths among all the variants of composites considered owing to the increased surface area of eggshells and tensile stress transfer from the matrix to filler. Carbonized eggshell-filled composites exhibited 43.9 and 34.14% higher tensile and flexural strength in comparison with unfilled composites. Thus, the addition of eggshell filler, especially carbonized eggshell filler proved to be beneficial for glass fibre/polyester composites. Although hybrid eggshell filler composites did not prove to be the ones with the highest strength amongst the variants considered, they displayed significant improvement in tensile and flexural strengths in comparison with unfilled composites. Considering the higher cost of the carbonizing process, hybrid fillers can act as a trade-off between higher-strength carbonized eggshell-filled composites and uncarbonated eggshell-filled composites. Further, the usage of eggshell fillers, especially hybrid fillers in various other resins and fibers would prove to be a good opportunity for future works.

Acknowledgments

This work was supported by the Department of Mechanical and Manufacturing Engg., MIT, MAHE, Manipal.

Conflict of Interest

The authors declare no conflict of interest.

Supporting information

Applicable.

References

[1] J. Yang, Y. Liu, S. Liu, L. Li, C. Zhang, T. Liu, *Materials Chemistry Frontiers*, 2017, **1**, 251-268, doi: 10.1039/c6qm00150e.

[2] H. Fu, H. Xu, Y. Liu, Z. Yang, S. Kormakov, D.Wu, J. Sun, *ES Materials & Manufacturing*, 2020, **8**, 3–23, doi: 10.30919/esmm5f713.

[3] S. M. Shahabaz, S. Sharma, N. Shetty, S. D. Shetty, M. C. *Engineered Science*, 2021, **16**, 26–46, doi: 10.30919/es8d553.

[4] E. Kilickap, *Expert Systems With Applications*, 2010, **37**, 6116-6122, doi: 10.1016/j.eswa.2010.02.023.

[5] D. Dixit, R. Pal, G. Kapoor, M. Stabenau, *Lightweight Ballistic Composites*, 2016, 157-216, doi: 10.1016/b978-0-08-100406-7.00006-4.

[6] A. K. Nurdina, M. Mariatti, P. Samayamutthirian, *Journal of Vinyl and Additive Technology*, 2009, **15**, 20-28, doi: 10.1002/vnl.20173.

[7] J. Sun, X. Zhang, Q. Du, V. Murugadoss, D. Wu, Z. Guo, *ES Materials & Manufacturing*, 2021, **13**, 53–65, doi: 10.30919/esmm5f450.

[8] P. Hiremath, M. Shettar, M. C. Gowrishankar, *Indian Journal of Science and Technology*, 2016, **9**, 1-6, doi: 10.17485/ijst/2016/v9i32/97024.

[9] T. Boronat, V. Fombuena, D. Garcia-Sanoguera, L. Sanchez-Nacher, R. Balart, *Materials & Design*, 2015, **68**, 177-185, doi: 10.1016/j.matdes.2014.12.027.

[10] P. Jagadeesh, Y. G. T. Girijappa, M. Puttegowda, S. M. Rangappa, S. Siengchin, *Journal of Natural Fibers*, 2020, **19**, 4132-4147, doi: 10.1080/15440478.2020.1854145.

[11] D. A. Oliveira, P. Benelli, E. R. Amante, *Journal of Cleaner Production*, 2013, **46**, 42-47, doi: 10.1016/j.jclepro.2012.09.045.

[12] M. C. Yew, N. H. Ramli Sulong, M. K. Yew, M. A. Amalina, M. R. Johan, *Progress in Organic Coatings*, 2015, **81**, 116-124, doi: 10.1016/j.porgcoat.2015.01.003.

[13] M. T. Hincke, Y. Nys, J. Gautron, K. Mann, A. B. Rodriguez-Navarro, M. D. McKee, *Front Biosci (Landmark Ed)*, 2012, **17**, 1266–1280, doi: 10.2741/3985.

[14] E. Nolasco, E. Jundt, M. M. Kramp, E. Dutta, N. Nishijima, Y. Oda, C. Xu, Y. Zhang, K. Majumder, *ES Food & Agroforestry*,

© Engineered Science Publisher LLC 2022

2020, **2**, 50–57, doi: 10.30919/esfaf1219.
[15] S. Murugan, Y. Munusamy, H. Ismail, *Plastics, Rubber and Composites*, 2017, **46**, 42-51, doi: 10.1080/14658011.2016.1260217.
[16] D. R. K. Chandrappa, M. S. S. Kamath, *Journal of Mechanical and Energy Engineering*, 2021, **4**, 335-340, doi: 10.30464/jmee.2020.4.4.335.
[17] M. Zieleniewska, M. K.Leszczyński, L. Szczepkowski, A. Bryśkiewicz, M. Krzyżowska, K. Bień, J. Ryszkowska, *Polymer Degradation and Stability*, 2016, **132**, 78–86, doi: 10.1016/j.ploymdegradstab.2016.02.030.
[18] P. Hiremath, M. Shettar, M. C. G. Shankar, N. S. Mohan, *Materials Today: Proceedings*, 2018, **5**, 3014-3018, doi: 10.1016/j.matpr.2018.01.101.
[19] R. Bhat, N. Mohan, S. Sharma, A. Pratap, A. P. Keni, D. Sodani, *Journal of Materials Research and Technology*, 2019, **8**, 3653-3661, doi: 10.1016/j.jmrt.2019.06.003.
[20] S. Owuamanam, D. Cree, *Journal of Composites Science*, 2020, **4**, 70, doi: 10.3390/jcs4020070.
[21] L. J. Shin, E., G. B. Dassan, M. S. Z. Abidin, A. Anjang, *Arabian Journal for Science and Engineering*, 2020, **45**, 5783–5791, doi: 10.1007/s13369-020-04561-z.
[22] S. B. Hassan, V. S. Aigbodion, S. N. Patrick, *Tribology in Industry*, 2012, **34**, 217–225.
[23] A. A. Hassen, M. Dizbay-Onat, D. Bansal, T. Bayush, U. Vaidya, *Polymers and Polymer Composites*, 2015, **23**, 653-662, doi: 10.1177/096739111502300908.
[24] A. Baby, S. Y. Nayak, S. S. Heckadka, S. Purohit, K. K. Bhagat, L. G. Thomas, *Materials Research Express*, 2019, **6**, 105342, doi: 10.1088/2053-1591/ab3bb7.
[25] K. Nijandhan, M. Raghunath, B. M. Kanna, P. Nithish, G. V. Prakash, E. Logesh, *Advances in Materials Research*, 2021, **5**, 107-114, 10.1007/978-981-15-8319-3_12.
[26] N. A. N. Azman, M. R. Islam, M. Parimalam, N. M. Rashidi, M. Mupit, *Polymer Bulletin*, 2020, **77**, 805-821, doi: 10.1007/s00289-019-02779-y.
[27] H. Chen, S. Wu, F. Dang, *Elsevier*, 2016, 235-238, doi: 10.1016/b978-0-12-803628-0.00022-7.
[28] C. L. Tucker, *Encyclopedia of Materials: Science and Technology*, 2001, 5726-5733, doi: 10.1016/b0-08-043152-6/00997-9.
[29] H. Wu, X. Huang, L. Qian, *Engineered Science*, 2018, **2**, 17-25, doi: 10.30919/es8d656.
[30] N. M. Abdul Wahab, M. A. Md Hanafiah, D. Samsudin, M. S. Mustafa, Z. Ahmad, S. N. Syed Ismail, *Charting the Sustainable Future of ASEAN in Science and Technology*, 2020, 435-445, doi: 10.1007/978-981-15-3434-8_37.
[31] Y. Chen, Y. Wang, T. Su, J. Chen, C. Zhang, X. Lai, D. Jiang, Z. Wu, C. Sun, B. Li, Z. Guo, *ES Materials & Manufacturing*, 2019, **4**, 31–37, doi: 10.30919/esmm5f214.
[32] M. Panchal, G. Raghavendra, A. R. Reddy, M. Omprakash, S. Ojha, *Polymers and Polymer Composites*, 2021, **29**, 897-908, doi: 10.1177/0967391120943454.
[33] O. J. Gbadeyan, S. Adali, G. Bright, B. Sithole, O. Awogbemi, *Composite Structures*, 2020, **239**, 112043, doi: 10.1016/j.compstruct.2020.112043.
[34] K. B. N. Balasubramanian, T. Ramesh, *Polymers for Advanced Technologies*, 2018, **29**, 1568-1585, doi: 10.1002/pat.4280.

Author Information

__Mr. Suhas Kowshik__ is a Senior Assistant Professor in the department of Mechanical and Industrial Engineering, Manipal Institute of Technology, Manipal, India. He holds B.E. (Mechanical), M.Tech. (Manufacturing Engineering and Technology) and pursuing Ph.D. (composite Materials) degrees. He has more than 8 years in teaching, industry and research experience. His areas of interest include characterization of polymer matrix composites, bio composites and hybrid composites. He has published 14 (Scopus indexed) research papers in reputed international journals and presented 10 research papers in international conferences.

__Dr. Sathyashankara Sharma__ is working as Professor and Head in the Department of Mechanical & Industrial Engineering, MIT, MAHE, Manipal. He holds B.E. (Industrial and Production Engineering), M.Tech. (Materials Engineering) and Ph.D. (Materials Engineering) degrees. He has more than 30 years of teaching and research experience. His area of interest includes Heat Treatment, Deformation of Metals, Material Characterization, Composite Materials. He has published more than 86 (Scopus indexed) research papers in journals and presented more than 50 research papers in conferences.

__Dr. Sathish Rao U.__ is working as Associate Professor (Sr. Scale) in the Department of Mechanical & Industrial Engineering. He holds B.E. (Mechanical), M.Tech. (Advanced Manufacturing Engineering) and Ph.D. (Composite Machining Optimization) degrees. He has more than 25 years in teaching, industry and research experience. His area of interest includes manufacturing, material characterization and design optimization of composite materials. He has published 13 (Scopus indexed) research papers in journals and presented more than10 research papers in international conferences.

 © Engineered Science Publisher LLC 2022

Dr. Manjunath Shettar *is working as Assistant Professor (Sr. Scale) in the Department of Mechanical & Industrial Engineering and Asst. Director, Student Welfare, MIT, MAHE, Manipal. He holds B.E. (Mechanical), M.Tech. (Production Engineering) and Ph.D. (Composite Materials) degrees. He has more than 12 years in teaching, industry and research experience. His area of interest includes manufacturing, material characterization and composite materials. He has published 50 (Scopus indexed) research papers in journals and presented 13 research papers in international conferences.*

Dr. Pavan Hiremath *is a Senior Assistant Professor in the department of Mechanical and Industrial Engineering, Manipal Institute of Technology, Manipal, India. He holds B.E. (Mechanical), M.Tech. (Manufacturing Engineering and Technology) and Ph.D. (Material Science and Engg.) degrees. He has more than 8 years in teaching, industry and research experience. He is one of the reviewers for many international journals and conferences. His areas of interest include characterization of polymer matrix composites, damage tolerance in advanced composites, heat treatment of metals and alloys etc. He was a part of testing team (intern) of SARAS aircraft designed and developed by CSIR National Aerospace Laboratories Bangalore. He has published 40 (Scopus indexed) research papers in reputed international journals and presented 12 research papers in international conferences.*

Mr. Arjun Upadhyaya *is a B.Tech. Mechanical Engineering student in the department of Mechanical and Industrial Engineering, Manipal Institute of Technology, Manipal, India. His contribution is crucial in getting the composite specimens tested for this research.*

Publisher's Note: Engineered Science Publisher remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

© Engineered Science Publisher LLC 2022