

DYNAMIC MECHANICAL AND STATIC ANALYSIS OF KEVLAR FIBER REINFORCED EPOXY COMPOSITES INCORPORATED WITH EGG SHELL PARTICLE

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Received: March 29, 2023; Revised: December 25, 2023; Accepted: January 16, 2024

Abstract

Research on composite material has been carried out stringently by using various reinforcing elements in the matrix with an intend to replace traditional monolithic materials eventually. In this regard, the Calcium-rich egg shell, which is abundantly available as a solid waste, can be used as a possible filler material for reducing material cost, improving processability, and refining the requisite properties of composite for specific applications with simultaneous reduction of the waste. In this study, the static and dynamic mechanical characteristics of eggshell particle-filled Kevlar/epoxy composites have been investigated. Through stirring with the aid of an Ultrasonicator, different weight percent of NaOH-treated eggshell particles (0, 10, 20 wt. %) of 90 micron sizes are mixed with epoxy to fabricate the composite using the traditional hand-layup method. In comparison to the hybrid composites, the mechanical properties of Kevlar/epoxy (0 wt. % filler) composite demonstrate superior tensile and flexural strength and modulus. However, when the Thermogravimetric analysis (TGA) and the Dynamic mechanical analysis (DMA) are accomplished for evaluating the thermal deterioration and viscoelastic characteristics, the hybrid composites demonstrated improvement in overall damping properties including storage modulus and loss modulus along with the thermal stability characteristics. Also, the elongation before failure is more for composite with 10 wt. % (71%) as well as 20 wt.% (18%) filler content with substantial improvement in Inter Laminar Shear Strength. Thus, the presently prepared composite is recommended for weight-sensitive and high-performance engineering applications under elevated temperatures and intense dynamic loading.

Keywords: Kevlar Fiber Composite, Eggshell Particle, Mechanical Properties, DMA, TGA

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DOI: <https://doi.org/10.55766/sujst-2024-01-e0892>

Suranaree J. Sci. Technol. 31(1):010273(1-10)

Introduction

Fiber reinforced polymer (FRP) composites are widely used in high-performance industries owing to their design flexibility to satisfy the manufacturing demand together with superior specific toughness and strength. The FRP composites are highly resistance to break by brittle cracking. Kevlar is one of the fibers that is widely used for fabrication of impact-resistant structures because of its damage tolerance, good impact performance and high degree of toughness (Oliwa, 2020). It is a strong synthetic fiber with very high heat resistance capability. Developed by DuPont, Kevlar has many applications along with racing sails, bicycle tires to bullet proof vests considering its diverse inherent properties. Kevlar is used as protection in armor, cryogenics and personal protection (Shuai *et al.*, 2020). It is also used in sports equipment like helmets and shoes etc. In certain places Kevlar is used as a substitute for light aircraft construction. In Kevlar FRP composites the main reinforcing element i.e., high-strength continuous Kevlar fibers are placed in a matrix or resin while the matrix works as a binder, protecting the fiber and distributing the load across the fibers (Naser *et al.*, 2019). Use of these synthetic fibers in FRP composites have been proved to have improved tensile, flexural as well as impact strength for specific applications (Rassiah *et al.*, 2021). High performance composites of Kevlar 29 and Cocos nucifera sheath (CNS) with different compositions have been used in the areas where thermal stability and rigidity are of utmost importance (Naveen *et al.*, 2019). The composite with 75 to 25 ratio of Kevlar to CNS shows good thermal stability along with char residue, loss modulus (E''), damping factor and storage modulus (E') as compared to the other composites (Naveen *et al.*, 2019). Various fillers are also added during the fabrication of FRP composites that not only minimizes organic content but also yields in weight and cost reduction and improvement in strength, stability, stiffness and thermal resistance. The use of nano silica as a filler in CKFRE (Carbon Kevlar Fiber Reinforced Epoxy) composites improved flexural and tensile strength due to the addition of nano-silica particles, owing to superior adherence to the epoxy/fiber matrix for attaining high mechanical and dynamic performance as required qualities (Alsaadi *et al.*, 2018). Numerical analysis of hybrid Kevlar/epoxy composites revealed that the stresses and deflection values decrease with increase in number of layers, fiber orientation and composite thickness and found suitable for aircraft body application with an improvement in mechanical properties (Abu *et al.*,

2011). The interfacial shear strength of composites has been seen to be improved owing to the functionalization of Kevlar fiber with phosphoric acid treatment (Li and Xia, 2010; Peigang *et al.*, 2020). It has been revealed that the FRP hybrid composites with higher percentage of fiber loading exhibit better mechanical properties (Richard *et al.*, 2019). FRP composites with Kevlar/glass hybrid structure have shown better results in terms of specific mechanical strength, flexural and impact energy (Valença *et al.*, 2015). With the integration of nano carboxyl functionalized multi-wall carbon nano tubes (MWCNT) in Kevlar/epoxy composites have shown improvement in overall characteristics (Muzammil *et al.*, 2019).

Natural fillers are increasingly used every now and then owing to their availability in nature, easy processing and adherence capability. The shell of a chicken egg is a waste product from the poultry industry and the home kitchen which is produced in large quantity worldwide. If the egg shells are not recycled properly, it may lead to a potential pollution problem in coming days. Chicken egg shell may be turned into a value-added product because it comprises 95 percent calcium carbonate an (Hassan *et al.*, 2012). The impact of filler particle size on the processing, tensile characteristics, morphology, and thermal degradation of poly (vinyl chloride)/chicken egg shell particle composites has been investigated (Murugan *et al.*, 2017). It has been revealed that in comparison to the larger filler particle size, smaller filler particle size enhanced the tensile modulus and strength of these composites. When compared to the larger filler particle size of the composite, the thermal stability of the finer filler size was found to be better. When compared to coatings manufactured using commercial calcium carbonate filler, egg shells obtained from aviculture waste has been readily utilized in acrylic coatings having greater water resistance, mechanical and thermal properties, as well as resistance to freeze–thaw cycles (Yew *et al.*, 2013). Egg shell filler reinforced epoxy composites using un-boiled and boiled egg shells have been investigated for the erosion wear behaviour. When compared to pure epoxy, the use of egg shells as a filler improved the wear resistance. Erosion resistance was negatively affected by exposure to various environmental conditions. Un-boiled egg shell filler composites outperform cooked egg shell filler composites in terms of wear resistance (Panchal *et al.*, 2018). The fine-sized egg shell filler improves the overall mechanical strength. The inclusion of filler enhanced the tensile and compressive strength of the

material. The egg shell as a filler can be used towards a valuable composite material for sustainable development (Lee *et al.*, 2020). Tensile characteristics of biodegradable composite films based on PPC (polypropylene carbonate) and ESP (egg shell particle) was found to be greater than a clean PPC matrix, and they added on with ESP concentration up to 4% before falling. The composite films outperformed the matrix in terms of thermal stability (Feng *et al.*, 2014). The carbonized egg shell addition depicted an enhancement in tensile, flexural and impact properties to some level and after that it dropped (Baby *et al.*, 2019). Addition of Hemp fiber commit to enhance the load bearing capabilities of epoxy resin composites and the dispersion of egg shell filler increased the composites thermal stability (Inbakumar and Ramesh, 2018). It is also seen that when a laminate is subjected to increasing thermal loading, TGA (thermogravimetric analysis) is often used to examine the thermal disintegration of the composites. The ability of a material to tolerate mechanical deformation at higher temperatures is measured by its thermal strength. The TGA was used by distinct researchers to study the heat bearing capability of hybrid synthetic as well as natural FRP composites (Jesuarockiam *et al.*, 2019). It has been reported that the Kevlar hybrid composite could perhaps replace the Kevlar/epoxy polymeric structure at elevated temperatures, according to TGA and dynamic mechanical study results. The thermal stability of glass fiber composite with incorporation of MWCNT has enhanced drastically (Rasana *et al.*, 2019). Moreover, while applying continuous loads, DMA (dynamic mechanical analysis) is broadly used technique for measuring viscoelastic properties such as storage modulus (E'), loss modulus (E''), and damping factor ($\tan \delta$). Numerous attempts have been made by combining various synthetic and natural fibers or fillers to improve these properties and the dynamic mechanical analysis of composite exhibited an enhancement in E' , E'' , $\tan \delta$ values as well as the glass transition temperature (Adak *et al.*, 2018). However, the largest storage and loss modulus are seen for higher glass fiber loading, whereas the lowest damping factor is observed for higher natural fiber loading (Atiqah *et al.*, 2018). In a hybrid palm/epoxy composite with PLSS (palm leaf stem stalk) filler the DMA result indicates improvement in storage modulus, loss modulus and damping factor as compared to only palm and epoxy composite. By adding filler, improved protection from swelling and degradation is reported (Kar *et al.*, 2020). The modulus of polyurethane shape memory polymer (SMP) is rather low. As a result,

sliced strand-mat glass fibers must be used as reinforcing elements for construction of SMP composites. Composites with a 15% fiber loading (regarded as critical fiber loading), offer the most improvement in properties. This is due to the glass fiber reinforcement, which usually improves the vibration damping properties of the product (Khiyon *et al.*, 2019).

According to the findings of the literature review we note that, researchers have attempted to use egg shell as filler material for improving the characteristics of diverse FRP composites. However, the properties of Kevlar fiber polymer matrix composite filled with egg shell particle is yet to be explored. Though many research work is available on Kevlar fiber reinforced composites owing to its popularity in engineering applications, the DMA and the TGA of Kevlar-epoxy composite filled with egg shell particle is yet to be discovered. The fundamental goal of this study is to fabricate a new class of Kevlar fiber reinforced composite laminates filled with three distinct weight percentage (0%, 10%, and 20 wt.%) of powdered egg shell and study the mechanical characteristics, viscoelastic behaviour through DMA and the thermal behaviour in terms of TGA so as to justify its applicability.

Materials and Methods

The details of the methodology implemented in the present research is portrayed graphically in Figure 1.

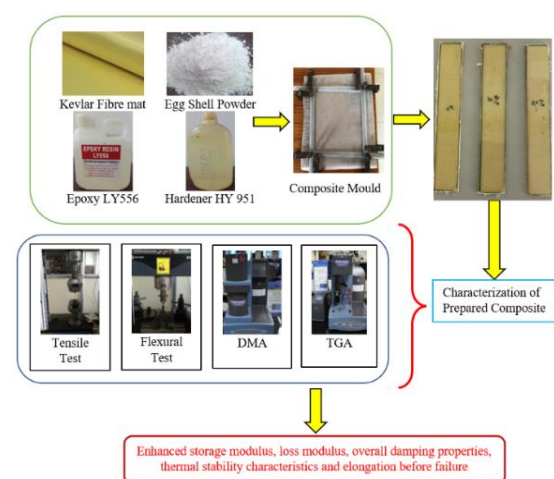


Figure 1. Details of the methodology used in the present analysis

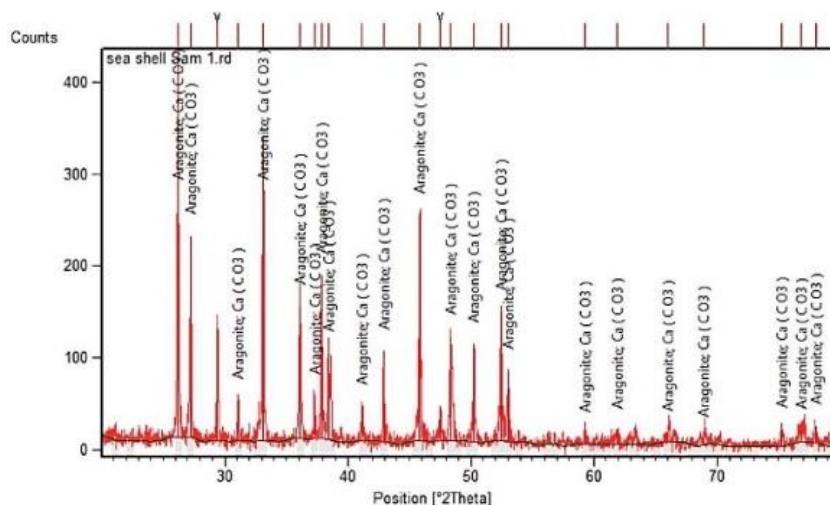


Figure 2. XRD of egg shell sample particle

Materials

Kevlar-29 para-aramid fabric (thickness: 0.3 mm, density: 1.44 gm/cm³) is employed as the fibre in this study and procured from Hayael Aerospace India Pvt Ltd, Chennai, India. Fibers treated with 5% NaOH for several hours has shown to improve fiber's compatibility with epoxy, tensile strength, cellulose content and the flexural strength (Rout *et al.*, 2016; Yudhanto *et al.*, 2021)). Therefore, the present Kevlar-29 fiber mats are cut with a cutter into desirable size (200mm×200mm), treated with 5% of NaOH solution for 2 hrs, rinsed completely with distilled water and dried in sunlight prior to using as reinforcement for the fabrication of composite. The matrix component is used as epoxy resin LY-556 and hardener HY-951 and obtained from Herenba Instruments and Engineers, Chennai, India. The egg shell is collected locally, washed, dried in sunlight, subsequently powdered and sieved to below 90 micron and used as a bio-filler after alkali treatment (5% NaOH). The x-ray diffraction (XRD) technique is used to obtain the crystallographic structure of the currently obtained egg shell particle and the result is portrayed in Figure 2. Abundancy of calcium carbonate (CaCO₃) in the form of aragonite with JCPDS number 01-075-0476 is observed.

Fabrication of Composite

The composites are made by using traditional hand lay-up approach because it is a cost-effective and simplest open molding method. A mould made of wood of size (150mm×60mm×5mm) is used to

fabricate the composites. Teflon sheet is used over the wooden mould and then silicon spray is applied on both the sides to avoid sticking to the surface of the mould and for smooth surface finish. Epoxy resin and egg shell particle with required wt.% are stirred for 10 mins using Ultrasonicator (Make: LABMAN, 2.5L, 100W, Heating power - 100W, Frequency: 40kHz) for accomplishing a homogeneous blend. After that, the blend is combined with the hardener in a ratio of 10:1 during stirring before being applied over the alkali treated bi-directional fiber mat with one layer over another. For specimen preparation, five layers of fibre mat are utilized. A roller is used to remove entrapped air and surplus resin. Finally, the mould is pressed at room temperature with a load of roughly 30kg weight and removed after 24 hours.

Composites with three different wt.% of filler content (0, 10 and 20) are prepared as mentioned in Table 1. For mechanical testing and thermal investigation, the samples are cut with the help of a cutter according to the ASTM standards (Figure 3).

Table 1. Composite designation

Designation	Composition		
	Epoxy (wt.%)	Kevlar fiber (wt.%)	Egg shell filler (wt.%)
C1	70	30	0
C2	60	30	10
C3	50	30	20

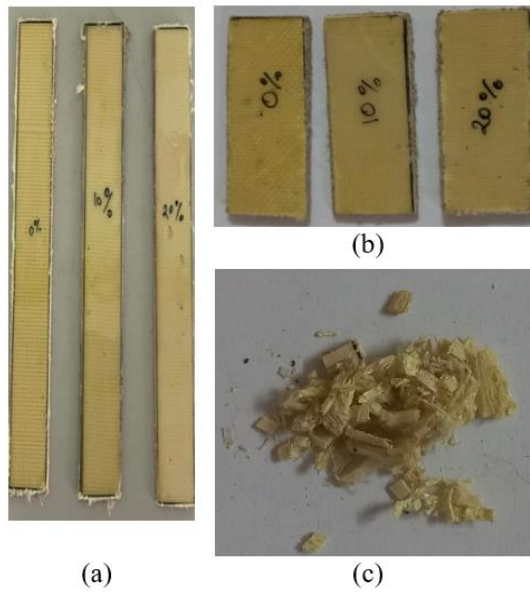


Figure 3. Samples for (a) mechanical characterization, (b) DMA and (c) TGA

Experimentation

As mentioned earlier, the presently prepared composites are tested for their tensile, flexural, viscoelastic and thermal stability characteristics.

Tensile and Flexural Test

An Instron 3382 Universal Testing Machine (UTM) present at Department of Mechanical Engineering, VSSUT, Burla, India has been employed to perform the tensile test with ASTM D 638-compliant samples and maintaining 2 mm/min cross head speed. Similarly, the flexural strength refers to a material's ability to resist bending before it reaches its breaking point and acquired via a three-point bending test performed using Instron 3382. The test specimens are fabricated as per ASTM D 790-99 standard and a 2 mm/min cross head speed is applied during experimentation. Each sample during tensile (150mm×20mm) and flexural (130mm×25mm) testing is tested thrice, with the average of the results used to determine the tensile and flexural properties of the sample. The flexural stress (σ) and the Inter Laminar Shear Strength (ILSS) is obtained via the following equation:

$$\sigma = \frac{3FL}{2bt^2} \text{ and } ILSS = \frac{3F}{4bt} \quad (1)$$

where, F = maximum load in tension, b = width of the specimen, L = distance between the supports

(mm), t = thickness of the specimen and P = maximum load in bending.

Dynamic Mechanical Analysis (DMA)

The dynamic mechanical analyzer (DMA Q800 TA, at LARPM, CIPET, Bhubaneswar, India) is utilized to evaluate the influence of adding egg shell filler by acquiring the viscoelastic properties according to ASTM D 5026 standards. The dimensions of the test samples are taken as 65mm×25mm×3mm. The three-point bending is carried out at 1 Hz frequency on the prepared laminates under a controlled loading condition. The analysis is accomplished from temp range of 25°C to 150°C maintaining a 10°C per minute heating rate. The loss modulus (E''), storage modulus (E'), and $\tan \delta$ of the composite samples are acquired.

Thermogravimetric Analysis (TGA)

Using a TGA Q50 available at LARPM, CIPET, Bhubaneswar, India, the thermal behaviour of Kevlar/epoxy with egg shell filler composites is investigated for TGA according to ASTM E 1868 standard. The powdered composites (5-10mg) are deposited according to the standards and the experiment is carried out in a nitrogen environment at temperatures ranging from 25°C to 600°C at a pace of 10°C each minute.

Results and Discussion

The results of the aforementioned tests are discussed in the following lines. Figure 4 (a) and (b) shows the arrangement of machine during tensile test and flexural test respectively.



Figure 4. Loading arrangement for (a) Tensile test; (b) flexural test

Tensile Test Results

The tensile properties for the present Kevlar/epoxy with different wt.% of egg shell particle filler are shown in Table 2 and the corresponding tensile strengths of each composite samples are shown graphically in Figure 5. From the test outcome it is found that the unfilled composite depicts marginally more tensile strength than filled composites with the tensile strength of composite having 20 wt.% of egg shell filler is higher in comparison to that for 10 wt.% of filler. This behaviour may be attributed to inadequate adherence of the matrix to the fiber and filler particles, as well as the effect of filler particle agglomeration in the matrix body which may have resulted in filler clumping impact and subsequently non-uniform stress transfer from matrix to fiber.

Table 2. Tensile test results of Kevlar/epoxy composites with egg shell filler

Composite Type	Max load (KN)	Young's modulus (GPa)	Extension (mm)
C1	10.505	6.505	5.084
C2	11.43	5.283	8.728
C3	12.78	3.153	6.01

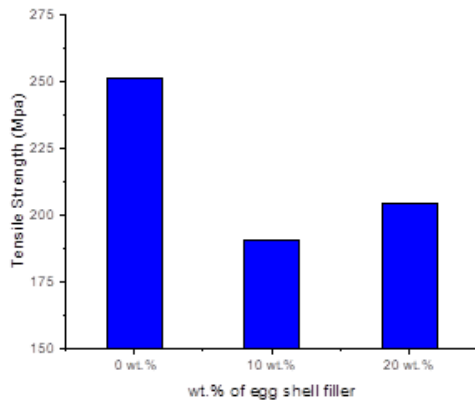


Figure 5. Tensile strength of egg shell filled Kevlar/epoxy laminated composites

Moreover, reduction in elastic modulus (refers to its stiffness at the elastic point) is noticed with increasing filler wt.%. The tensile modulus for unfilled composite is 6.5 GPa, for composite with 10 wt.% filler it is 5.28 GPa and for 20 wt.% filler it is 3.15 GPa. The decrease in tensile modulus refers to increase in resistance of material to deformation. However, it is worthy to mention that the elongation

before failure is maximum for composite with 10% filler content (71% more compared to no filler).

Table 3. Flexural test results of Kevlar/epoxy composites with egg shell filler

Composite Type	Flex modulus (GPa)	Flex strength (MPa)	ILSS (MPa)
C1	15.317	194.29	1.3889
C2	8.265	179.59	1.4880
C3	9.889	189.86	1.8720

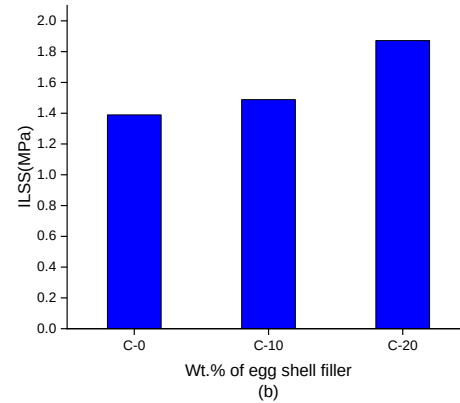
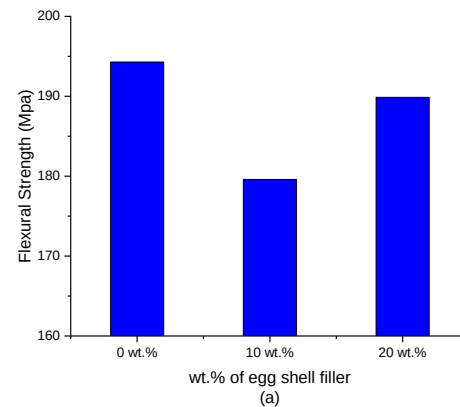


Figure 5. (a) Flexural strength and (b) ILSS of egg shell filled Kevlar/epoxy laminated composites

Flexural Test Results

The results of the flexural test for the presently prepared composites are presented in Table 3 and the corresponding flexural strengths of each composite with corresponding ILSS values are shown graphically in Figure 6. The specimens fail during three point-bend tests because of the commencement of cracks inside the matrix region

that further increases along the fibre axis and eventually reaches the laminate. In line with the tensile test results with the addition of filler, the flexural strength of the composite is found to be reducing. The bending strength for composite with 20 wt.% filler is 189.86 MPa which is less than unfilled composite (194.29 MPa) as well as the composite filled with 10 wt.% egg shell filler (179.59 MPa). However, significant improvement in ILSS (7.14% and 34.78% for 10 wt.% and 20 wt.% egg shell filler in comparison to the Kevlar/epoxy composite with no filler) has been noticed due to incorporation of egg shell particle as filler material.

Dynamic Mechanical Analysis (DMA)

The damping capacity of Kevlar/epoxy and Kevlar/epoxy with egg shell filler composite is analyzed with DMA. The composite releases and absorbs some energy under dynamic loading condition. The energy absorbing capacity means the stiffness or rigidity of the composite and the energy releasing capacity means the material's damping capacity. The damping factor ($\tan \delta$), loss modulus (E''), and storage modulus (E') are acquired in order to determine damping and stiffness of the presently prepared composite samples.

Storage Modulus (E')

In sinusoidal flexural deformation, the ratio of stress in phase with strain is the capability of the material for storing energy elastically and regarded as E' . The distinction in storage modulus of Kevlar/epoxy composite with and without egg shell filler is shown in Figure 7. In the glassy zone, the unfilled Kevlar/epoxy composite has a storage modulus of 3189 MPa, which increases to 6081 MPa and 6833 MPa for 10 wt.% and 20 wt.% of filler, respectively. This indicates that the gradual improvement of the composite stiffness to the maximum for 20 wt.% filled composites. During the glass transition, the E' value starts to decrease because the stiffness of the matrix is affected by the rise in temperature that results in matrix's molecular mobility and degradation in fiber/matrix adhesion. During the rubbery plateau phase, there is no such considerable change found in E' value when the temperature is increased. Based on the observations, it is inferred that with the inclusion of egg shell filler, the E' value has changed significantly in glassy region and glass transition region, while there is no variation found in E' value due to egg shell filler in the rubbery region, which is evident from the overlapping of plots. The composites have a higher E' value at temperatures ranging from 50 to 70°C.

Loss Modulus (E'')

For a viscoelastic material like the currently prepared Kevlar/epoxy composite filled with egg shell particle, the loss modulus (E'') is defined as the amount of thermal energy released per cycle in a sinusoidal loading condition (Jesuarockiam *et al.*, 2019). Figure 8 outlines the influence of adding egg shell filler on E'' values of Kevlar/epoxy composites put through temperature loading. It has been noticed that with in the glass transition temperature range the E'' value increases, reaches a maximum, and then decreases for all laminates irrespective of the filler. The overlapping of curves at higher temperatures (over 110°C) demonstrates that the addition of egg shell particle has no effect on viscose dissipation at higher temperature. The rise in E'' value signifies the increased damping capacity (Adak *et al.*, 2018) of the presently prepared composites.

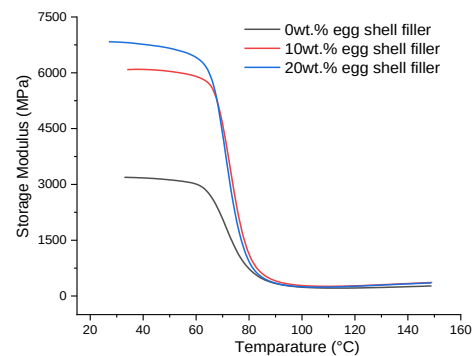


Figure 7. Effect of egg shell filler on Storage Modulus (E') of Kevlar/epoxy laminated composites

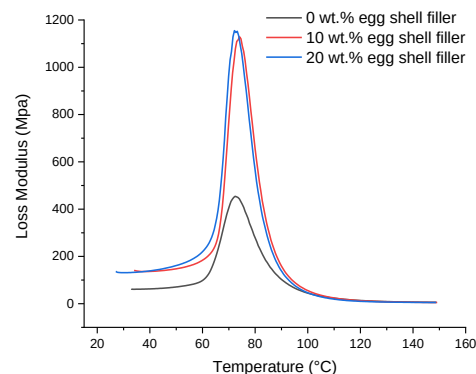


Figure 8. Effect of egg shell filler on Storage Modulus (E') of Kevlar/epoxy laminated composites

Tan Delta ($\tan \delta$)

The Tan delta value denotes the mechanical damping factor, which is decisive by the adhesive characteristics of the fiber and matrix. The composites having lower damping value of $\tan \delta$ represent improved interfacial adhesion with higher load bearing capacity and higher value at glass transition temperature range represent weak interfacial adhesion with low load bearing capacity. The plot of the laminated composites are shown in Figure 9. From the plot it is clearly seen that, below the glass transition temperature range the damping is low which may be due to the fact that the polymeric chain is frozen in this region. Lower damping values are also noticed in the rubbery zone, may be due to unrestricted molecular mobility. In glass transition region, the value increases as the molecular mobility of the polymeric chain increase. The value for Kevlar/epoxy is found to be (0.30) lowest for no filler case signifying less molecular mobility and energy dissipation. This is owing to the high adherence between the fibre and the matrix, which slows the polymeric chain's mobility. With the addition of filler, the value increases to a maximum of 0.60 for the composite with 20 wt.% filler content, which shows the higher energy dissipation and more viscos behaviour (Atiqah *et al.*, 2018; Kar *et al.*, 2020).

Thermogravimetric Analysis (TGA)

For the prepared egg shell filled Kevlar/epoxy composite, TGA analysis is performed, and the result is shown in Figure 10 that clearly demonstrates the impact of adding egg shell filler on the thermal stability characteristic. From the thermogram it is observed that the initial mass loss occurs at 100-200°C which indicates the presence of moisture content and other compounds in composites. The second stage of mass loss or major mass loss is due to the disintegration of epoxy and hardener which is reported at temperatures above 300°C. The third phase of mass loss is observed beyond 600°C as a result of splitting of Kevlar fibre hydrogen bonds with egg shell filler. Subsequently, the composite maintains a mass loss up to 800 °C. It is noticed that as the egg shell particles are added, the thermal stability of the composites improved. The char percentage and degradation temperature are shown in Table 4. The heat stability of Kevlar/epoxy composites 10 wt.% and 20 wt.% has been claimed to be superior, as evidenced by the equivalent char residue production. The final decomposition temperature of Kevlar/epoxy with 0,

10 and 20 wt.% filler is 570.52°C, 572.48°C and 573.13°C, respectively which shows the incorporation of egg shell particle exhibited better thermal stability. The hybrid composite with 10 wt.% filler demonstrates the highest major degradation temperature (MDT) as well as final degradation temperature (FDT) as compared to other composites.

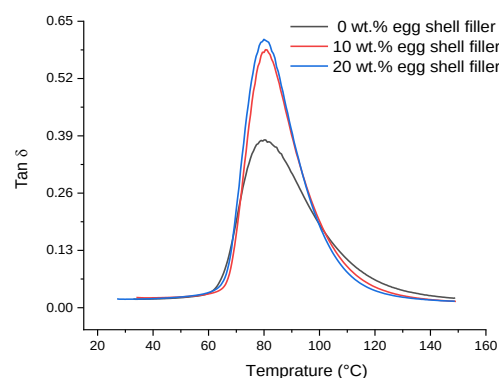


Figure 9. Effect of egg shell filler on value of Kevlar/epoxy laminated composites

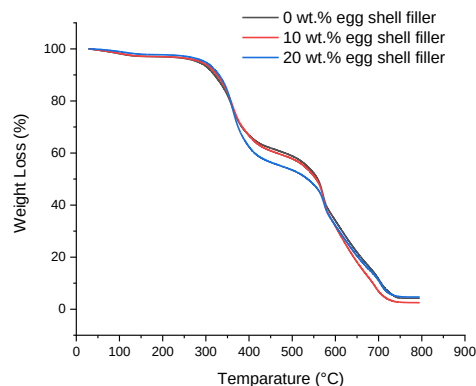


Figure 10. Effect of egg shell filler on thermal stability of Kevlar/epoxy laminated composites

Table 4. Char residue and thermal degradation of different wt.% of Kevlar/egg shell composites

Composit e Type	MDT (°C)	Wt. loss	FDT (°C)	Char residue (%)
C1	361.20	36.50	570.52	19.45
C2	361.85	38.05	572.48	16.66
C3	360.55	42.86	573.13	13.74

Conclusions

The dynamic mechanical and static property of the Kevlar/epoxy composites with different wt.% of egg shell filler have been investigated. The fabrication of hybrid composites using chemically treated Kevlar fiber reinforcement in polyester resin filled with egg shell particle is readily done by simple hand lay-up technique. The tensile strength (24.15% and 18.67% for 10 and 20 wt.% filler, respectively) and the tensile modulus (23.16% and 30% for 10 and 20 wt.% filler, respectively) has been reduced slightly due to the incorporation of egg shell as filler. However, the damping factor has been improved significantly (56% and 60% for 10 and 20 wt.% filler, respectively). Also, the elongation before failure is more for composite with 10 wt.% (71%) as well as 20 wt.% (18%) filler content in comparison to without filler alongside improvement in Inter Laminar Shear Strength. Moreover, the addition of egg shell particle increased the thermal stability besides the viscoelastic properties of the composites. The highest viscoelastic characteristic and stability under thermal environment of Kevlar/epoxy composites is seen for laminate with 20 wt.% filler content. The Thermal stability of Kevlar/epoxy composite found to be superior which is evidenced by the maximum char residue production. For Kevlar/epoxy composite with 10 wt.% of egg shell filler, the major degradation temperature and final degradation temperature is found to be maximum. Overall, from the present research it may be inferred that, the overall damping characteristic as well as the thermal stability property of the Kevlar/epoxy composite have been enhanced significantly at the cost of slight reduction in tensile and flexural property, but with improvement in elongation before failure owing to the incorporation of egg shell filler. Thus, the presently prepared composite can be effectively used in weight sensitive and high-performance engineering application under elevated temperature as well as intense dynamic loading.

Acknowledgement

The authors acknowledge the financial assistance provided by TEQIP-III, VSSUT, India under "Collaborative Research and Innovation Scheme" grant via. reference no. VSSUT/TEQIP/158/2021 dated 18/03/2021.

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