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Research Article

Effect of Duck Eggshell Powder as a Reinforcement on Dynamic Mechanical Properties of Epoxy Composites

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ABSTRACT

After a duckling hatches from an egg, the empty eggshell is usually discarded as waste or used as natural fertilizer due to their high calcium content. Utilization of waste materials in polymer composite is increasing day by day mainly due to environmental considerations and waste management. Owing to its rich content of calcium carbonate, renewability, and availability and cost-effectiveness, duck eggshells can be used as natural filler in polymer composite and it is future potential resource for the polymer industry (1). The present study is focused on to analyze the influence of duck eggshell powder as a filler with epoxy polymer on the dynamic mechanical behavior. Therefore, this focus is an effort to explore the potential application of waste duck eggshell powder for the first time as a natural filler in epoxy composites. Five dissimilar composites were fabricated by filling different wt% of duck eggshell powder and epoxy. The composites are manufactured by open mold method. The manufactured composite samples then were subjected to dynamic mechanical test. Specimens cut and tested in relation to ASTM standards. The test showed fluctuation behavior with a positive indication at 4% increment was filled with four independent ratios (4, 8, 12 and 16 wt. %) of eggshell powder. The results showed that duck eggshell powder (DESP) fillers' have potential usage as a natural filler to develop the thermal resistance of the epoxy polymer (EP) matrix composites, thereby reducing costs and minimizing the environmental issues caused by waste eggshells. To be specific, storage modulus, loss modulus, damping factor and phase transition temperature were analyzed through dynamic mechanical analysis (DMA). The results revealed that storage modulus of the composites decreases as the temperature increases. Although, the composite containing 12% eggshell powder attained a higher storage and loss modulus. These newly developed composite material are being initiated for a range of automotive applications.

Keywords: Duck eggshell; Epoxy; Storage Modulus; Loss modulus; Damping factor.

1. INTRODUCTION

Generally, a composite material is a material structure that can be prepared by incorporating two or more materials to obtain distinct properties that are special than the properties of individual materials. Polymer composite comprise of a resin and reinforcement medium or filler. The main advantage of use of reinforcement or filler is, it might reduce the final cost of the composite by reducing the volume of resin. Filler material plays a primary role in polymer industries. Substantial interest has been motivated in the manufacture of thermoset polymer composites by virtue of their distinct properties, which include favorable mechanical properties, thermal resistance, and a low production cost. Owing to the combination of more than one material, the properties of composite are controlled by various factors such as filler content, filler characteristics, and interfacial adhesion. This leads to the behavior of filled polymer composite to be more intricate than unfilled composite. Polymer composites have been popularly used in automotive, aerospace and construction industries as a result of their ease of fabrication, low density, economy and impressive mechanical performance. In specific, powder or particles filled polymer composites are useful for a various applications owing to their low cost, favorable physicochemical and mechanical properties, simple fabrication process, and positive effect on the surrounding atmosphere. This character of powder filled polymer composite has paid the much interest on researcher's to develop biodegradable materials.

An eggshell is a network of protein fibers, linked to crystals of calcium carbonate ($CaCO_3$), magnesium carbonate ($MgCO_3$) and calcium phosphate ($Ca_3(PO_4)_2$), in addition to organic matter and water (6). The properties could differ from one eggshell to other eggshell, even within the same species depending on the quality of eggshell. Duck eggshells contain about 94-97% $CaCO_3$ and 50.75% calcium deposits in the nature of flour (7).

Dynamic mechanical analysis is an approach employed to determine the viscoelastic behaviour of polymeric materials. Polymer composites are commonly tested to obtain precise measurements of viscoelastic properties namely elastic modulus (or storage modulus, E'), viscous modulus (or loss modulus, E″), and damping coefficient (tan Δ) using Dynamic Mechanical Analysis or DMA. The dynamic mechanical analysis method determines viscoelastic properties in relation to temperature, frequency or time. Results are generally plotted with respect to temperature or strain in the form of E', E", and tan Δ. DMA could also be applied for the purpose of quality control and product development owing to the small sample size and simple experimental procedure. Besides the viscoelastic properties, additional information like phase transition temperature can be identified. The phase transition temperature is among the most requisite property of polymer composite that describes the temperature region where the mechanical behaviour of a material changes from hard to deformable or rubbery. Viscoelastic behaviour and phase transition temperature of the final composite are two essential characteristics of a polymer composite material for product development process. Glass transition temperature Tg can be found using various measurement modes. The commonly used methods are single cantilever or dual cantilever (3 point bending), compression and torsion, but sometimes under tension and shear. Dynamic mechanical analysis utilizes smaller specimen and lower loads as compared to conventional mechanical tests, to give wide information on viscoelastic properties of a composite material. This proves that DMA is an extremely powerful approach for the prediction of practical uses of glass transition temperatures, energy dissipation and stiffness of a composite material.

2. MATERIAL AND METHODS

Epoxies are generally out-perform resins in comparison to other resin types in terms of mechanical strength and resistance to climate change in the environment, which leads to their almost exclusive use in automotive and aircraft components. For the production of composite plates, Epoxy LY-556 and hardener HY-951 were used. Duck eggshell powder was used as reinforcement filler.

2.1 Composite Fabrication. Table 1 shows the compositions of the filler and matrix used in the present work. The composites were prepared using a single plate open mold having a cavity of 50 x 13 x 3 mm to get a specimen size of required dimension. The material used for preparation of the mold is plywood. The overall dimension of the mold is 100 x 50 x 10 mm. The epoxy and hardener in the ratio of 10:1 are weighed as per the calculations and poured into the plastic bowl. The resin and hardener were stirred for about 2 minutes. Further, the eggshell powder is weighed as per the calculations and is poured into resin mixture. All the materials are thoroughly stirred manually for about 5 minutes. The whole mixture of resin and eggshell powder was flowed inside the mold cavity and is allowed to cure for 24hrs. After 24 hrs the cured composite slab was extracted from the mold and kept for 1 day for further curing outside the mold. Lastly, after 1 day, the cured composite plate was subjected to post curing for 30min at 100°C to improve ductility. After post curing, the composite slab was kept at room temperature for three to four days and specimens were cut to size of ASTM standard to conduct DMA test.

Table 1: Composite mixture

Composite Mixture	Composite slab (DESP+EP)	
	DESP (wt%)	ER (wt%)
A2	0	100
B2	4	96
C2	8	92
D2	12	88
E2	16	84

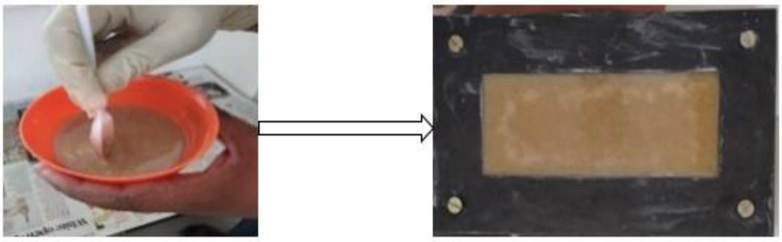

Fig. 1: Composite plate fabrication.

2.2 Experimental setup of DMA. Dynamic mechanical thermal analyzer (TA Instruments Q800) shown in figure 2 was utilized to analyze the values of storage modulus (E'), loss modulus (E"), damping factor (tan Δ), and the melting point (glass transition temperature, Tg). The range of temperature through which the properties were determined was -28^{0}C - 160^{0} **C**, at a temperature flow rate of 5^{o}C/min. Three-point bending configuration was applied to conduct forced at varying temperatures and at steady frequency of 1 Hz. The specimens were in rectangular strips with dimensions of 55 × 13 × 3 mm. Dynamic mechanical analysis was performed on 5 different composite samples under controlled atmospheric temperature and it is chronicled in table 1.

Fig. 2: Experimental setup of DMA.

3. RESULTS AND DISCUSSIONS

3.1 Storage modulus (E')

The term storage modulus is the amount of energy stored in stable material when it is deformed (4). The plots of the storage module (E') versus temperature at different eggshell powder content for different composite specimens are described in Figure 3. As can be remarked that at lower temperatures, a slight raise in the value of storage modulus can be observed on entire composites and starts decreasing as the temperature starts increasing. A prominent peak is present on entire experimental curves where a steep fall in storage modulus which is correlated to the Tg value (glass transition temperature) of a material. Generally, this abrupt fall indicates the greatest thermal working temperature of the corresponding composite. In spite of whole composite mixtures, the composition with 12% percent eggshell powder displayed largest storage modulus than other composites. This can be owing to the positive effect of the duck eggshell powder with the epoxy composite and it indicates that a small addition of duck eggshell powder percentage can contribute a high positive increment in the storage modulus values. Thus, the inclusion of duck eggshell powder content from 4% to 12% have followed in a 14.14% increase in storage modulus. This denotes that increase in duck eggshell powder content in epoxy composites could be definitely have a positive benefit on the improvement of E' on the entire compositions. This is on account of increased ductility resulting from the mixing of duck eggshell powder as reinforcement fillers (5). This will increase the effective stress transfer between duck eggshell powder and epoxy resin.

Table 2: Viscoelastic properties.

Composite Mixture	**Viscoelastic properties**		
	Storage Modulus (E') (MPa)	Loss Modulus (E") (MPa)	Tan Delta (tan Δ)
A2	2950.56	395.45	0.832
B2	3150.16	450.80	0.835
C2	2960.52	425.60	0.800
D2	3436.42	490.12	0.792
E2	3331.22	465.24	0.756

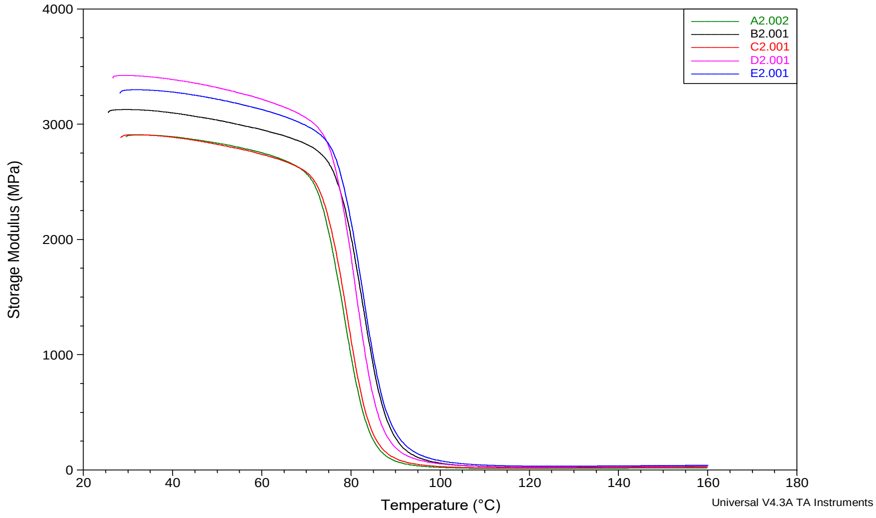

Figure 3: Combined storage modulus v/s temperature

3.2 Loss modulus (E")

The term loss modulus (E") is the total magnitude of energy dissipated by the material as heat when it is subjected to dynamic load. The variation in loss modulus values at different temperatures of duck eggshell powder epoxy composite with change of eggshell powder content are displayed in graph 4. As can be remarked in this graph that the loss modulus values are practically stable at lower temperatures and abrupt increase can be observed after about 70 °C a up to the peak point. Further, the increase in duck eggshell powder content results in widening of the peaks of the curves. Then a sudden fall is attained at very lower temperatures until reaching nearly zero which is continued until 140°C. As in all the storage modulus curves, 12% duck eggshell powder content have exhibited higher loss modulus (approximately between 70–90 °C) in comparison to other compositions. The higher value of E" at this temperature is due to increase in internal friction and softening of the composite which promotes energy dissipation (5). In comparison to 0% eggshell powder, the loss modulus of 12% eggshell powder was raised by 19.31%. Generally, the value of loss modulus diminishes at higher temperatures for all the composite systems, because of decrease in frictional resistance between the epoxy and duck eggshell powder for higher temperatures. Peak heights are significantly more with the increment of duck eggshell powder content.

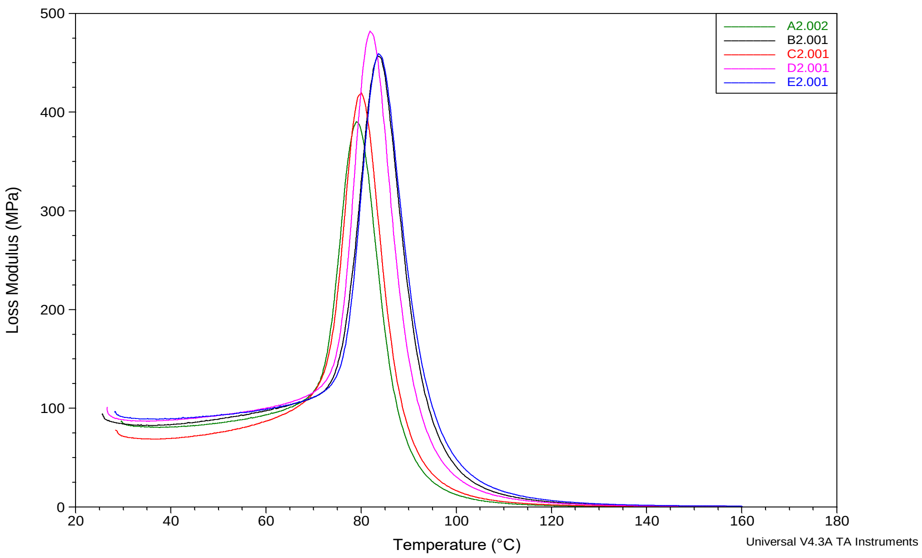

Figure 4: Combined loss modulus v/s temperature

3.3 Damping factor or Tan Δ

The term damping is the capacity of a material to withstand deformation by decreasing the amplitude of vibration in elastic and viscous phases in a polymer structure. In composites, damping factor is influenced by the inclusion of fillers. The damping factor (tan Δ) is calculated by dividing the loss modulus by storage modulus. That means:

$$Tan\,\Delta = \frac{E''}{E'} \qquad (1)$$

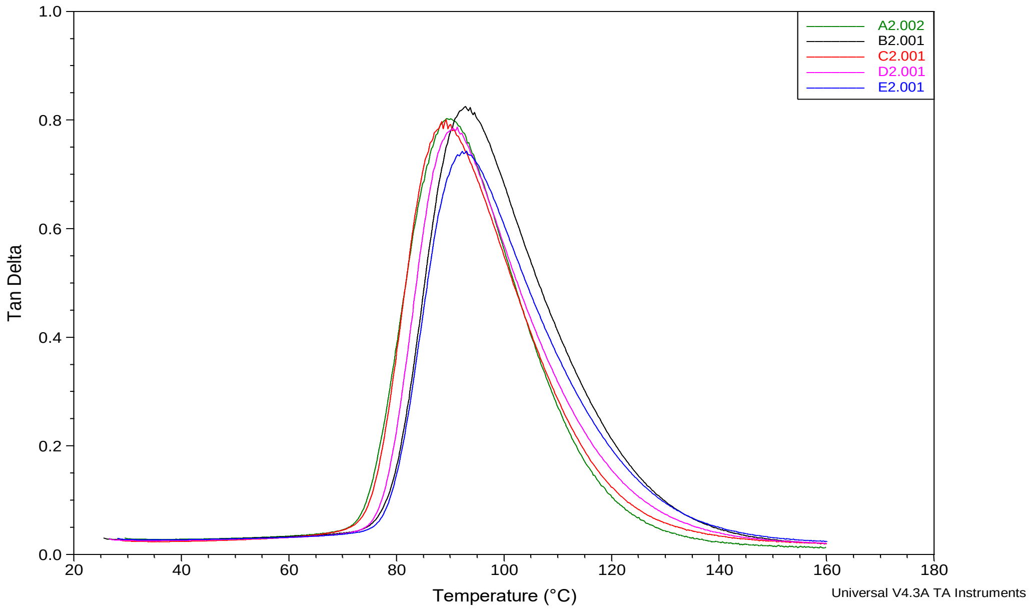

Figure 5: Combined Tan Δ v/s temperature

Figure 5 illustrates the variation of damping factor in all the compositions at various temperatures. As can be remarked in this graph that the value of damping factor is low at the beginning and maintains constant up to certain temperature (about 70 °C) temperature. In general, as the temperature raises, damping reaches a peak point (about 80 °C) in the glass transition region and then drops in the rubbery region. Furthermore, in the glassy region, whole molecular segments are chain segments in the compacted state and the deformation is principally elastic while in rubbery region, whole molecular segments are totally free to move resulting from viscous flow and the deformation is plastic (2). Therefrom, the energy dissipation is high in glassy region and low in rubbery region

(2). This could be result of decrease in thermo resistance of the composite at relatively higher temperatures on account of viscous flow in polymer chain (Rashmi Aradhya et al. 2022). Vibrations of molecular segments inside composite causes the damping in the composite system (4). However, increment in the duck eggshell powder content reduces the internal vibration which in turn leads decrease in damping value. The composite E2 showed least damping value which indicates increase in elastic behavior of the composite after mixing of duck eggshell powder content with the epoxy. Relative to the 0%, damping decreased by 9.14% for the 16%.

3.4 Phase transition or glass transition temperature

Table 3: Phase transition temperature.

Composite Mixture	Phase transition temperature (Tg)	
	Tg values by loss modulus peak (°C)	Tg values by Tan δ peak (°C)
A2	78.95	90.34
B2	83.35	92.80
C2	80.03	89.19
D2	81.82	90.42
E2	83.58	93.01

The temperature value in which a material turns from a vitreous solid and rigid to a flexible and strong elastomer is called the phase transition or glass transition temperature (Tg). The phase transition is a flexible change in the material between rubbery and glassy regions. The 'Tg' is identified by the peak in the E" curve or a peak in the tan Δ curve. In the current investigation, the phase transition temperature (Tg) for different composites was identified by two distinct modes; peak height of loss modulus curves and peak height of tan Δ curves separately. These separate values are displayed in table 3.

As can be remarked in these values that among the two modes, the temperatures (Tg) attained from the peak height of tan Δ curves are little greater than those recorded from the peak height of loss modulus curves. By taking the peak height of E" as a Tg, there is an increase in value of Tg value with the increase in duck eggshell powder content. A little boost in the value of Tg can be viewed with the increase in duck eggshell powder content. This is because of decrease in mobility in the epoxy polymer chain owing to increased powder content (3). Likewise, in comparison to the 0%, the Tg of the 16% was more than other compositions due to better interfacial compatibility. Higher the Tg values lower the energy dissipation (3).

4. CONCLUSIONS

In the course of this research work, duck eggshell powder filled epoxy composites were processed by varying the percentage of filler and the effect of filler on dynamic mechanical properties was studied. The study gives rise to the following major conclusions:

- The DMA of duck eggshell powder reinforced epoxy composites was significantly affected the addition of duck eggshell powder.
- The increase duck eggshell powder led to an improved E' with a minimal damping (tanδ) caused by the development of a rigid and tough composite. Correspondingly, increasing eggshell powder content has improved the glass transition (Tg) peak points, which implies a reduced mobility polymer molecules at the interface because of the increased duck eggshell powder content.
- At increased temperatures, as the duck eggshell powder content increased, the value of E" was increased to a highest peak. The higher duck eggshell powder resists thermal exposure while lower duck eggshell powder leads to polymer melting. The highest peak in the E" curve is due to the restriction in polymer molecules in the chains

(4).

From the primary experimental investigations, duck eggshell powder filled epoxy composites could be effectively utilized for the manufacture of components that can be functional at temperatures below, approximately, 80 °C. They can be able to make interior components of automobiles and aircrafts.

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