E-Glass/phenolic matrix/APP laminate as a potential candidate for battery casing of e-vehicle - experimental investigations

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Abstract

The prime purpose of the current research is to investigate the influence of Intumescent flame retardant (IFR)-Ammonium polyphosphate (APP) on the mechanical properties of Phenolic resin/E-Glass hybrid configurations and suitability of hybrid configuration for battery casing in E-vehicle. Varying weight fractions viz. 2, 4, 6, 8, 10 wt% of APP were used to fabricate Phenolic resin/E-Glass/APP polymer composites. Tensile, flexural, low velocity impact, and quasi static indentation tests were conducted and analyzed. Among the developed hybrid configurations 6 wt% APP filled laminate shows the better tensile and flexural properties than other configurations. It is understood from low velocity Izod impact test, falling weight impact test and quasi static indentation tests that APP filled hybrid configurations have more energy absorption. 6 wt% APP filled laminate has high deformation in elastic phase and low deformation in plastic phase. Morphology and fracture mechanism of polymer laminates are also presented and discussed. It is concluded from the investigations that E-Glass/Phenolic matrix/APP laminate is found as a potential candidate for battery casings of E-vehicle.

1. Introduction

Polymer matrix composites (PMC) became inevitable in the progressive engineering applications due to their enhanced mechanical, thermal and electrical properties, corrosion proof nature, recyclability and tailorability [1]. Fiber reinforced polymer matrix composites replace most of the conventional engineering metals like aluminium and steel due to its light weight, ease of fabrication and cost efficiency. They are inexorable material in transport, aerospace, and space vehicles. E-Glass is a most commercially used high end fiber that is characterized by its unique properties like temperature stability (up to 250 °C), electrical insulation and dimensional stability under varying chemical reactions, humidity [2, 3]. Similarly, phenolic resin matrix possesses enhanced temperature stability up to 350 °C and hence mostly used for the fabrication of laboratory countertops, fireproof panels [4]. The hybrid configuration of phenolic resin/E-Glass is found as a good choice of polymer composites for fire-prone or hazardous applications. The secondary reinforcement of hybrid configuration using APP is a suitable method of limiting the flammability due to its increased char formation and low toxic fumes [5].

Naik [6] investigated tensile behavior of Epoxy/glass fiber composite at high strain rate and disclosed that it takes brittle fracture due to poor matrix-reinforcement and interfacial bonding. Zuleyha Aslan [7] reported that flexural, compression and buckling capacity of E-Glass laminate is greatly affected by critical delamination. Cagla Balaban [8] investigated low velocity impact behavior of E-Glass composites and reported that increase in impact energy directly enhances the damage area and crack propagation.

In polymer matrix composites, defects, flaws, and strength reductions occur due to accumulated stress concentrations (improved cross link density) and load repeatability. This phenomenon was reported in [9, 10].
Table 1. Properties of E-Glass fabrics, phenolic resin matrix and APP

| Properties                  | E-Glass polymer mats | Phenolic resin matrix | Ammonium polyphosphate |
|-----------------------------|----------------------|-----------------------|------------------------|
| Density (g cm$^{-3}$)        | 1.95                 | 0.9                   | 2.8                    |
| Tensile strength (MPa)       | 3050–3600            | 18–34                 | —                      |
| Elastic modulus (GPa)        | 54–66                | 35–48                 | —                      |
| Elongation at break (%)      | 4.7                  | 1–2                   | —                      |
| Maximum service temperature (°C) | 320                   | 280–350               | 280                    |
| Molar mass (g mol$^{-1}$)    | —                    | —                     | 65.8                   |
| Isoelectric point (pH)       | —                    | —                     | 7.2                    |
| Poisson ratio                | —                    | —                     | 0.31                   |

The aforementioned issue can be overcome through the secondary reinforcement of PMC with additional fillers that are conventionally tailored to the required properties [11, 12]. Various fillers or additives like nano clays [13–15], single and multi-walled carbon nano tubes [16–18], graphene [19–21], SiO$_2$ [22] and fire-retardant additives like ammonium polyphosphate, aluminium trihydrate and aluminim hypophosphates [23] have been attempted. Significant improvements in mechanical properties and fire performance have been reported in the past research. However, increase in filler density up to 20 wt% is preferable in both economical and manufacturing perspective [24, 25]. Inclusion of intumescent FR additive in polymer is expected to greatly improves flame resistance and modifies the mechanical properties.

Khalili et al [26] investigated the effect of APP, alumina trihydrate (ATH) on the natural fiber composite and studied its synergistic on the mechanical, fiber performance. They revealed that APP reinforcement significantly improves flame resistance and mechanical properties than ATH reinforced composites. Sheng Yang [27] investigated artificial thermal aging of APP at 50 °C, 75 °C and 100 °C for 1000 h and expressed that thermal aging remarkably reduces the degree of polymerization. Afrouzian [28] analyzed damage tolerance of glass fiber reinforced composites and reported that inclusion of nano filler significantly influences the mechanical behavior.

The sustainability of electric vehicles (EV) in automotive industries depends purely on light weight and cost-efficient alternative composite materials. The current research aims to develop a polymer composite which could be an alternative material for EV battery casing. Secondary reinforcement of APP into Phenolic resin/E-Glass hybrid configuration is hypothesized to yield superior mechanical, thermal properties, and fire retardancy. It is well understood from the above literature that a limited study on APP is available. No research has been conducted on investigations of Phenolic resin/E-Glass/APP polymer laminated composite and its suitability in EV application.

2. Materials and methods

2.1. Materials

The base polymer matrix, plain woven E-Glass mats were purchased from Hindustan composites, India. Phenolic resin matrix and auxiliary filler reinforcement were purchased from AYPOLS Ltd, India. The fine grade of ammonium polyphosphate with the average particle size of 45 μm (98.9% purity) was purchased from CF composites, India. Table 1 depicts the properties of E-glass fabrics, phenolic resin matrix, and ammonium polyphosphate as received from the supplier. Thin E-Glass/Phenolic resin/APP hybrid composite laminates of 3 ± 0.3 mm were fabricated from plain woven roving E-Glass mats of 440 GSM, phenolic resin matrix with hardener and ammonium polyphosphate (APP) fillers of 45 micron size.

Firstly, three fin blender was used to mix 2 wt% of APP fine fillers with phenolic resin matrix. The temperature and speed of blending were maintained as 50 °C and 450 rpm respectively. The mixing was carried out for 20 min to prepare a homogeneous polymer composite. As phenolic resin matrix generally creates air bubbles, the homogeneous mixer of phenolic resin/APP was put into vacuum chamber at 0.25 bar for 20 min to degas and eliminate air bubbles. Before starting the hand layup reinforcement of E-Glass fabric mats, required amount of wax was applied over the mould for the purpose of easy removal of laminates after compression moulding. E-Glass fabric mat was then placed on the mould and phenolic resin (catalyst mixed) was applied evenly over the mat. In the same way, five more E-Glass fabric mats were reinforced and placed one over another. Six layers hybrid polymer laminate was then put into compression moulding machine and 10 bar pressure was applied for 6 h. Compressed laminate was then taken out of compression mould and cured at room temperature for 24 h and 50 °C in hot air oven for 2 h.
In the same way, 4 wt%, 6 wt%, 8 wt% and 10 wt% of APP were also used to prepare different hybrid configurations. All these synthesized E-Glass/Phenolic resin/APP laminated composites had a total of six layers with thickness of $3 \pm 0.3$ mm. Table 2 shows the details of hybrid configurations developed for investigations.

To investigate the mechanical and micro structural properties, abrasive jet machine was used to cut the samples as per ASTM standards. Abrasive jet cutting parameters were set as suggested in [29] to avoid structural damages and delamination to the edges of samples. The entire step by step method of preparation of hybrid laminates was depicted in figure 1.

2.2. Evaluation of Mechanical Properties

Dumb bell-shaped tensile samples as per ASTM D638-03/ISO 37 standard were tested using Instron® 5582 universal tensile testing machine (100 kN load cell). The test condition was room temperature having 23°C and 50% humidity. In each hybrid configuration, three samples were tested, and the average values were reported for the discussion.

The flexural specimens of $130 \text{ mm} \times 13 \text{ mm} \times 3 \text{ mm}$ were prepared as per ASTM D-790 standard. Three point bending tests were carried out at cross head speed of $0.5 \text{ mm min}^{-1}$ in Instron 4486 universal testing machine.

Eventually all the automotive components have a threat of sudden impact through accidents. It is essential to study the behavior of the polymer composite at sudden impact loads. Hence, Izod impact samples as per ASTM

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**Table 2** Formulations of hybrid composite constituents

| Sample       | E-Glass (wt%) | Phenolic resin matrix (wt%) | APP (wt%) |
|--------------|---------------|-----------------------------|-----------|
| E56P44 (Neat Sample) | 56            | 44                          | 0         |
| NAPP2        | 56            | 42                          | 2         |
| NAPP4        | 56            | 40                          | 4         |
| NAPP6        | 56            | 38                          | 6         |
| NAPP8        | 56            | 36                          | 8         |
| NAPP10       | 56            | 34                          | 10        |
D256 were prepared. The impact test was done using AIT-300N impact tester with pendulum swing of 1600 mm and striking hammer of 18.7 kg.

Specimens of 100 mm × 100 mm × 3 mm as per ASTM D7136/D7136M-15 standard were prepared for falling weight impact (IFW) tests. The specification of the machine is: impact weight = 1.25 kg, maximum falling height = 50–6000 mm and d90 type of nose. IFW tests were carried out by varying the dropping height with the constant weight of 1.25 kg. Figure 2 represents the samples used in testing and their respective ASTM standard dimensions.

Quasi-static indentation test was done to determine the mode of collapse and energy absorption capacity of the polymer samples. The test samples were prepared as per ASTM D6264 (140 mm × 140 mm × 3 mm) and the indentation test was carried out using Instron 3382 (hemispherical indenter, 100 kN load cell). The length and diameter of the indenter is 145 mm and 12.5 mm respectively. The laminate sample was fixed into the clamp tightly by arresting all degrees of freedom and the specimen was crushed by the indenter at a low cross head speed of 0.5 mm min⁻¹ between two parallel plates. The average values of test results such as displacement of indenter with respect to force and energy absorption capacity of hybrid samples in both elastic and plastic regions were recorded using Instron testing machine.

2.3. Field emission scanning electron microscopy (FESEM)

Field emission scanning electron microscopy analysis was done to study the microstructure and damage modes of the test samples. FEI Quanta 400 F Field Emission microscope (Up to 100 000 000,00x magnification) with the accelerating voltage of 0.2–30 kV, maximum image processing resolution up to 3584 × 3094 pixel and thermal field emission gun was used to capture the microscopic image of the unfractured and fractured samples. Initially the SEM samples were cleaned thoroughly and mounted on the stub by using adhesive followed by gold coating to avoid scattering of electrons. The current SEM analysis was focused to study the dispersion of APP into matrix...
as well as the microstructure after damage. Microscopic images of fractured samples and unfractured samples with different magnifications and directions were taken and discussed in detail.

3. Results and discussions

3.1. Morphology study

The morphology of APP filled Phenolic resin/E-Glass laminates is shown in figure 3. Figures 3(B), (C), (D) show the 6 wt%, 8 wt%, and 10 wt% APP filled laminates, while figure 3(A) shows the neat Phenolic resin/E-Glass laminate. It is noticed that 6 wt% of APP loaded composite shows more even distribution and dispersion of APP into the Phenolic resin/E-Glass composite. 8 wt% and 10 wt% laminates show the clumps of fillers and aggregates.

Though the agglomeration of fillers into the matrix is very common in composite materials, but it is to be avoided to have the improved material properties. Yasser Zare [30] reported that very smaller filler particles produce aggregation/agglomerations in the composite. It can be reduced by introducing small content of large sized fillers along with preferred fillers. Elizabeth et al [31] reported that Ligand-functionalized nanoparticles in polymer materials greatly reduces the agglomeration, poor dispersion rates along with self-assembling, crack healing properties. Bernardo [32] reported that foaming or hydrating the nano particles significantly enhances yield strength and modulus up to 15% and limits the fracture toughness.

3.2. Tensile properties

Tensile behavior of APP filled hybrid polymer composites and unfilled samples were tested and presented in figures 4(A) and (B). It shows the mean result of three tested samples in each group. The deviation bar at data points refers the standard error of the measurement. From the tensile tests, the tensile strength of neat sample E56P44 is observed as 234.27 MPa, while it is 268.53 MPa, 303.42 MPa, 324.56 MPa, 284.21 MPa, 249.16 MPa for 2, 4, 6, 8, 10 wt% APP filled phenolic/E-Glass laminates respectively. The tensile strength is the resistance to plastic deformation of the composite. The maximum strength of 324.56 MPa is obtained at 6 wt% APP reinforcement which is 38.55 % higher than neat sample. The composite has become more brittle when wt% of APP is increased to 8 wt% and 10 wt%. Also noticed that these composites have more clumps of fillers and
aggregates as evidenced in SEM micrographs. Because of these reasons that 8 wt% and 10 wt% laminates have low tensile, flexural properties than 6 wt% laminate.

The elastic modulus of neat sample is 41.032 GPa, while it is 44.62 GPa, 46.326 GPa, 47.815 GPa, 48.503 GPa, and 49.865 GPa for 2, 4, 6, 8, 10wt. % respectively. Interestingly noticed that the elastic modulus has not been decreased at 8 wt% and 10 wt%. The increased elastic modulus of the composite with the loss of ductility can be noticed in heat-treating polymers. The presence of low porous aggregates in the composite can also increase the stiffness. The larger proportion of coarse aggregates may also lead to high elastic modulus \[13, 33\]. This is what observed from microscopic analysis in the current investigations. 6 wt% APP filled phenolic/E-Glass laminate shows the greatest tensile strength with the increased stiffness, because of presence of lower proportion of coarse aggregates in the composite. It can be noted that the larger proportion of aggregates

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**Figure 4.** The effect of wt% of APP fillers in (A) Tensile stress vs. strain curve (B) Breaking load and tensile modulus (C) Comparison of elastic modulus with Halpin-Tsai theoretical modulus.
are witnessed in both 8 wt% and 10 wt% of APP loading. This is the reason for having higher stiffness, but lower tensile strength (more brittle) in 8 wt% and 10 wt% of APP loading.

The experimental results were compared with the Halpin–Tsai model. Halpin–Tsai is a most common model used for estimating the elastic modulus of particulate reinforced composites [34]. The elastic modulus of the composite is found as

\[
E_c = E_m \left[ \frac{(1 + \eta \phi_f)}{(1 - \eta \phi_f)} \right]
\]

\[
\eta = \frac{m - 1}{m + 1}
\]

Where, \(E_f\) is elastic modulus of fiber material, \(E_m\) is elastic modulus of base matrix, \(\zeta\) is geometric parameter, \(\phi_f\) is fiber volume, \(I\) is a parameter related to filler shape \((I = 2 \zeta)\) and \(m = E_f / E_m\).

For the current research, \(E_m\) is the elastic modulus of APP filled phenolic resin matrix, while \(E_f\) is elastic modulus of E-Glass material. Figure 4(C) shows the comparison of elastic modulus estimated from the Halpin–Tsai model and experimental results.

3.3. Flexural properties

Figures 5(A) and (B) depicts three-point bending (flexural) behavior of fabricated hybrid samples. The result trend observed from flexural tests is similar to the result trend of tensile tests. Flexural strength of neat sample
E56P44 is observed as 215.61 MPa, while it is 232.86 MPa, 254.5 MPa, 285.18 MPa, 245.56 MPa, 236.15 MPa for 2, 4, 6, 8, 10 wt% APP filled phenolic/E-Glass laminates, respectively. The flexural properties get increased by the addition of APP up to 6 wt% of filler loading. It is observed that NAPP6 possess excellent flexural strength of 285.18 MPa, which is 32.2 % higher than the neat sample. But it is decreased for 8 wt% and 10 wt% APP added samples. Similarly, flexural modulus of 0, 2, 4, 6, 8, 10 wt% APP filled hybrid laminates are reported as 13.49 GPa, 15.65 GPa, 16.05 GPa, 16.86 GPa, 17.93 GPa and 19.16 GPa respectively. Patrick Lim [29] and Katsoulis et al [35] has also reported the similar trend of loss in flexural strength due to higher loading of APP and melamine cyanurate fillers into Epoxy/E Glass composite.

3.4. Low velocity impact tests
Izod impact test results of hybrid configurations are depicted in figure 6. The results reveal that the higher loading of APP enhances impact energy absorption capacity of the laminates. The energy absorption is found almost proportionate to the wt% of APP.

6 wt% APP reinforced Phenolic resin/E-Glass laminate results 41% of increase in energy absorption, while the maximum impact energy is resulted by 10 wt% sample (NAPP10). NAPP10 shows 69.5 % of increase in energy absorption than neat laminate. From the results it is evidenced that inclusion of APP into Phenolic resin/E-Glass laminates significantly improves the toughness of the sample. Energy absorption of toughened
laminates is in the order of 13%, 26%, 41%, 54%, 69.5% for 2 wt%, 4 wt%, 6 wt%, 8 wt% and 10 wt% respectively. This reveals that the drastic increase in impact toughness is achieved by 8 wt% and 10 wt% addition of APP into Phenolic resin/E-Glass laminates.

Falling weight impact test was conducted on 10 mm × 10 mm × 3 mm Phenolic resin/E-Glass/6 wt% APP laminate (NAPP6). The impact energy of the laminate against the free falling of 1.2 kg at varying height of 2 to 4 m was observed. The results of the test are shown in figure 7. The impact energy absorbed by the laminate is 27, 31, 36, 39 and 45 J at 2, 2.5, 3, 3.5 and 4 m of height.

3.5. Quasi-static indentation test results

Quasi static indentation response of the fabricated laminates was investigated, and the energy absorbed in elastic region and the total energy absorbed at the fracture of sample were recorded. The energy absorbed in plastic energy was then computed from these two readings.

Figure 8 shows the energy absorption in elastic region as well as plastic region. The elastic energy is a recovered deformation energy to be obtained just before fracture. The plastic energy is the energy consumed during the failure of the sample, which never be recovered again. The energy absorption in elastic phase and plastic phase of neat laminate (E56P44) is 4.8 J and 4.1 J respectively. This indicates that the neat laminate has higher deformation in the elastic region and hence could have absorbed more energy in the elastic phase. While neat laminate possesses maximum energy of 8.9 J, the total energy absorption in APP reinforced polymer composite is almost linearly increased according to weight percentage of filler up to 8 wt%. The total energy in APP filled Phenolic resin/E-Glass/APP laminates is 11.14 J, 13.19 J, 14.35 J, 16 J ad 14.2 J for 2 wt%, 4 wt%, 6 wt%, 8 wt% and 10 wt% respectively. The total energy in 10 wt% filled APP is found lower than 8 wt% laminate. Interestingly noticed that the energy absorption in plastic phase of 2 wt%, 6 wt%, 8 wt% and 10 wt% are higher than elastic phase. The primary failure mechanism in any composite is matrix failure that caused by brittle behavior. The micro cracks are generally formed in elastic phase of the loaded composite. These micro cracks accumulate together to form a macro crack in a localized damage area. With this failure plane, delamination is induced and progressed to fracture of the matrix of the composite. The toughening mechanism of Phenolic resin/E-Glass/APP laminates is understood by noticing the energy absorption in elastic and plastic phases. It indicates that APP fillers can mitigate the macro crack formation and progress of the delamination in the plastic phase.

3.6. Field emission scanning electron microscopy

Fractured tensile sample of 6 wt% APP filled Phenolic resin/E-Glass laminate is shown in figure 9. The rough fractured surface of matrix, broken glass fiber and phenolic matrix are clearly seen in figure 9(A). Crack formation and propagation of crack in matrix due to the progressive load is also evidenced in figure 9(B). In the study of crystalline structure and fracture surface analysis, filler and matrix interactions plays major role and directly influences overall mechanical properties of the hybrid laminates [36]. Figure 9(C) shows voids and smooth matrix surface. It is clearly evidences that fiber fracture has occurred due to the filler debonding and nano void formation caused by induced stress along with energy dissipation [37]. In the smooth surface region, a
good interfacial bonding between matrix and reinforcement is observed. Whereas in rough surface region, poor bonding due to the agglomeration is observed. Crack blowing mechanism is also observed in the samples, where new cracks were formed before existing crack probation. These phenomena were common in the particle reinforced composite configurations [38]. Similarly, figure 9(D) shows enhanced fractures occurred at APP fillers and fibers. This must be due to high stress concentration at APP fillers. Figure 9(E) shows the fracture of fibers and pull outs. The pull-outs are seen in weak matrix-fiber interface of the laminates. Figure 9(F) indicates the superior bonding between fiber and matrix due to the uniform dispersion of APP fillers.

4. Conclusions

E-Glass/Phenolic matrix filled with various proportion of Ammonium Polyphosphate were fabricated using compression moulding. Investigations on tensile and flexural properties reveal that reinforcement of 6 wt% into E-Glass/Phenolic matrix (NAPP6) yields the better tensile strength, elastic modulus, flexural strength and flexural modulus than any other filler concentration. Low velocity impact tests and quasi-static indentation tests reveal that energy absorption is increased by addition of APP fillers into the composite. As APP filled laminates
have high energy absorption, they can delay the crack propagation. APP reinforcement serves to have higher mechanical properties and fire-retardant characteristic. The findings of this research pave the way to select E-Glass/Phenolic matrix/APP laminates as a potential candidate for battery casings of E-vehicle.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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