

HALIDE FREE FIBER REINFORCED PLASTICS AS AN EFFICIENT FIRE-RETARDANT COMPOSITE MATERIAL

Rekam Manikumar^{1,✉}, T. Nageswara Rao² and Sannapaneni Janardan³

^{1,2}Department of Mechanical Engineering, GITAM School of Technology, GITAM (deemed to be University), Bangalore 561203, Karnataka, India

³Department of Chemistry, GITAM School of Science, GITAM (deemed to be University), Bangalore 561203, Karnataka, India

✉Corresponding Author: manikumar.rekam@gmail.com

ABSTRACT

The ortho unsaturated polyester resin (UPR) was successfully synthesized from propylene glycol, and diethylene glycol in the presence of acids such as phthalic anhydride and maleic anhydride using polycondensation reaction, where styrene is used as a cross-linker, which is catalysed by triphenyl phosphate and accelerated by cobalt octate. The Fire-retardant properties were incorporated into UPR using fire-retardant additives such as Aluminium trihydrate and Ammonium polyphosphate resulting in efficient fire-retardant resin. The liquid and casted mechanical properties of UPR resin are studied and The UPR composite was manufactured through the hand layup method, with the E-corrosion resistant glass fiber reinforcement with different stacking layers. The synthesized resin to the glass fiber ratio was less than other commercial resin to glass-fiber ratios. The final composite properties were found in the acceptable range.

Keywords: Fire Retardant Additives, Fire Retardant Fiber Reinforced Plastics, UPR Synthesis, FRP Mechanical Properties, FRP Flammable Tests, And Fiber Reinforced Composites.

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INTRODUCTION

Fire retardant unsaturated polyester resin is a plastic material commonly used in various industrial and construction applications. It was characterized by its ability to resist combustion and slow fire spread. FR will be achieved by incorporating fire-retardant additives into the resin, such as halogenated free or phosphorous-based compounds. These additives disrupt the combustion process, limit the amount of heat and gas produced, and slow down the spread of fire. As a result, fire retardant unsaturated polyester resin is widely used in applications where fire safety is a concern, such as in manufacturing building materials, transportation, and electrical equipment. The increasing demand for these Unsaturated Polyester resins is because of the cheap manufacturing, repairs, and fast cure with dimensional stability even can be tailor-made to meet the diverse needs of the FRP Industry. For adding different additives to reduce the flammability of the resin, the synthesis process needs to be tailored. Heat-releasing rate, peak exotherm, and low viscosity are significant factors in decreasing the flammability of the resin. So, fire retardant resin synthesis is entirely different from general-purpose resin synthesis. Shuilai Qiu *et al.* synthesized a special FR reactive glycol-based additive with a phosphorus structure. Mainly concentrated on the structural chain modification for effective enhancement of unsaturated polyester resin with FR additive to the readily available industrial resin. Some compositions achieve the UL94 Vo standard. Followed a unique additional approach for UPR to attain the UL94 Vo standard. Mechanical and fire tests are performed to analyze the composite performance with special additives and reduce the PHRT and T.H.R compared to the standard industrial resin¹. Mateus Hofmann, Abu T. Shahid *et. al.*, synthesized biobased UPR to form G.F.R.P biocomposites with a vacuum infusion process. Bio-based unsaturated polyester resin with monomers (diacids and diols) from renewable sources. This resin includes reactive diluents like styrene and hydroxyethyl-methacrylate 40% equally, synthesized with more than 50% by weight of biobased resin to the final resin. The main idea was to make a polymer prepolymer main chain with renewable sources through condensation. Explained the laminate properties obtained through the Vacuum infusion to the commercial resin laminate produced from the same vacuum infusion process. All the mechanical and

thermomechanical characterization was found competitive with a slight 10% less glass transition temperature.² Fukai Chu, Shuilai Qiu *et al.* enhance the structural relationship and interaction mechanism between the unsaturated polyester resin and fire retardants. Discussed the current problems facing the composite industries, including their poor mechanical qualities, rapid flame migration, and poor compatibility. Explained the new synthesis process with the design concept of structural chain variations (both primary and side chain) in gaseous phase mechanism and effectively +3 valence of phosphorus and sulfone groups. Concentrated on special additives designs to the UPR. Performed the tests NMR, UL94, FTIR, and GPC to evaluate the flame retardants.³ Through the processes of glycolysis and esterification, Minyan Zhang and Zhengfu Lia *et al.* created an unsaturated polyester resin with flame-retardant qualities. Used R-pet (polyethylene terephthalate), diphenylsilanediol (DDS), and propylene glycol to create the Glycolsis product. Then reacted with the maleic anhydride to form the unsaturated polyester with silicon later added, the ATH (aluminum trihydride). Formulated resins, tested with the FTIR, SEM, LOI, UL94, and H-NMR methods for FRR and Mechanical properties. FRR falls under class B with the highest impact strength and modulus of elasticity than the standard resin.⁴ Ortho UPR, also known as GP unsaturated polyester resins, are made through a condensation process using glycols, Maleic anhydride (MA), and phthalic anhydride (PA). Ortho Phthalic anhydride forms structural stiffness and is inexpensive compared to other Acids. In terms of glycols, propylene glycol (PG) resins are more important than ethylene glycol and diethylene glycol-based resins. The methyl group reduces the resin's crystallinity in PG, making it more compatible with usually employed reactive diluents.⁵ Melted an aromatic dicarboxylic acid with a polyhydric alcohol as a viscous liquid. It is common practice to use a reactive diluent, such as styrene, to reduce the reaction product's viscosity. The double bond between styrene and unsaturated polyesters produced a heterochain thermoset polymer with a rigid, three-dimensional, crosslinked structure. Methyl ethyl ketone peroxide (MEKP) is a common catalyst that initiates the curing reaction at room temperature when combined with a cobalt or cobalt-amine activator system or accelerator. UPRs also neutralize the free radicals cumene hydroperoxide and benzoyl peroxide (BPO).⁶ An inhibitor was added to the resin after it was synthesized to increase curing speed, shorten cure time, and reduce undesired colors, scents, or negative effects. Among the inhibitors are hydroquinone, 4,4-dihydroxy biphenyl, and substituted catechol.⁷ For various applications, researchers have tried to alter the corrosion, thermal, mechanical, and FR properties of UPRs. According to Parker *et al.*, isophthalic acid enhances mechanical characteristics and corrosion resistance.⁸ Watanabe *et al.* invented a two-stage production method. Which uses dimethyl terephthalate rather than isophthalic acid to address the essential improvements.⁹ Better mechanical qualities were obtained in the work of Benny Cherian, Eby Thomas Thachil, and others through the polymerization of alcohols and acids in the ratios of 30% ethylene glycol to 70% propylene glycol and MA/PA is 60/40. All mechanical properties are examined exclusively for cast resin without fiber reinforcement and all polyesterification reactions at 30 mg KOH/g.¹⁰ They also created other constructions by adjusting the concentrations of the anhydride and alcohol. Synthesis of unsaturated polyester resin attained the finest mechanical characteristics using a 60% MA/PA combination. However, higher percentages of PA-concentration resins have shown to be more resilient and flexible. Similar to DEG's increased flexibility and toughness, DEG also boosted impact strength, which was lost while loading. Ideal characteristics are seen with a 20/80 DEG/PG resin ratio with an equal mixture of MA and PA.¹¹ Diverse diols and polyols were combined with bio-derived diesters of unsaturated acids such as fumaric, succinic, and itaconic acids to create resins with a molecular weight range of Mn B480–477,000 and glass transition temperatures (Tg) that range from -30.1 °C to -16.6 °C.¹² These resins differ in their solubilities depending on the starting monomers. Yoon and his coworkers fixed UPR and then created UPR. Recycled UPR cured more quickly than pure resin, in comparison. assessed the mechanical characteristics of pure resin and mixes (composed of pure resin and recycled materials). Although the qualities of pure resin were higher, the properties of the blends depended on their composition and were shown to be useful in several application scenarios.¹³ Used Cobalt (Co) curing system with varying concentrations (from 0.05% – to 1%). The gel time slashed when the concentration of Co was from 0% to 1%. Gel time decreased to 50% in the presence of 0.05 percent Co.¹⁴ Considering cobalt acetate as an accelerator and MEKP as the initiator of the reaction, it was planned how the volume ratios of the two curing agents would affect gelation and the exothermic behavior of a UPR. shown that the start resin temperature increased when the resin's gelation started. the accelerator and

initiator absorptions had an inverse relationship with the gel time.¹⁵ The liquid system's viscosity increased at the curing temperature but reduced at higher temperatures. Based on its fragility parameter, the cured UPR's quality was projected (M_c). The UPR-MEKP system's T_g and heat resistance when M_c is lessened and T_g is increased.¹⁶ Kosar and Gomzi used an experimental and theoretical model to investigate the UPR's curing behavior.¹⁷ Dynamic and isothermal measurements were used to examine the curing system's kinetic behavior, and The two were in complete agreement (in terms of the reaction heat and kinetic parameters provided). Cylindrical molds help to measure the heat generated by the response. The fundamental cause of the superior heat produced in the mold was the differential heat conductivity between copper and glass. A significant challenge in low-temperature molding methods is controlling the resin shrinkage of residual monomers. Under the right processing circumstances, Low-profile additives (LPAs) help reduce UPR/styrene resin shrinkage, but they may also result in a higher residual styrene content. With LPAs, it is possible to conduct a systematic investigation to determine the impact of the originator system and reaction temperature on the model morphology, ultimate resin alteration, and resin shrinkage of UPR. The outcomes demonstrated that two initiators could increase the resin system's ultimate conversion, with the greatest effect at low temperatures. LPA performed better in the shrinkage control study at low (35°C) and high (100°C) temperatures than it did at intermediate temperatures (e.g., 60°C - 75°C). The eventual shrinkage of resin results in changes in the relative reaction rates between the LPA-rich and UPR-rich phases, changes in the morphology, and the formation of microvoids.¹⁸ Commercial UPRs have a mass percentage of 30% to 40% styrene. The composition of the resin determines whether resin and styrene are miscible. Styrene concentration increases the associated phase separation. The Styrene monomer regulates the mixture's phase behavior, which controls the mixture's thermal stability and mechanical qualities.¹⁹ It has been discovered that phase separation occurs in cured resin with high styrene concentrations through Dynamic Mechanical Analysis (DMA) research. The styrene content, thermal stability, and mechanical behavior influence the glass transition temperature.²⁰ According to the application, such as marble, marine, gel coats, and buttons with different proportions of PG, DEG, NPG, PA, and MA, Hildeberto Nava (poly composites) describes various high-performance, low molecular-weight resin formulations to improve the corrosion and thermal performance in composite product design.²¹ Most researchers terminated the resin synthesis process to acid values of 40 mKOH/g and used limited materials for synthesis. Liquid resin properties and resin cast properties are insufficient to add FR additives. In this present work, we reported a composite synthesized and incorporated fire-retardant properties which are in good agreement with the standard composites

EXPERIMENTAL

Materials and Methods

Propylene Glycol (PG), Diethylene glycol (DEG), Phthalic anhydride (PA), Maleic anhydride (MA), Styrene, Triphenyl phosphate (TPP), Toluene hydroquinone (THQ), Butanox M-50 (9% Active Oxygen) (MEKP), Cobalt Octoate (6%), N, N-Dimethylaniline, Aluminum trihydrate (ATH), Ammonium polyphosphate (APP) were purchased from Finer Chemicals and used directly without any further purification and 600 woven roving glass and 300 chopped standard were purchased from Owens corning.

Preparation of Ortho Unsaturated Polyester Resin

The ortho unsaturated resin was synthesized from the monomers such as glycols (PG and DEG) and acids (PA and MA) through a polycondensation reaction and the reaction was refluxed at 210°C temperature for about 9h using a temperature-controlled mantle. During our trials observed that pure maleic anhydride enhances the immediate gelling which resists the strength of the resin, so used a mixture of monomers in the respective ratio 40:60 for getting, highly compatible, cost-effective resin with good mechanical strength with the styrene cross-linker. The completion of the reaction was monitored by determining the acid value using ASTM D 4662-87 method with the help of KOH at regular intervals (20min) and the branching of the resin was observed when secondary glycols were employed in the reaction. When the acid value reached 60mg KOH/g the temperature was reduced to 140°C and added the inhibitor (THQ 1% of MA composition), and continued to the acid value up to 18 mg KOH/g. The temperature of the reaction is reduced to 80°C by cooling and 0.02 wt% of inhibitor and paraffin wax (0.2-0.3 wt%) are added to the styrene to prevent oxygen-mediated crosslinking reaction. Premature polymerization of the reaction is also prevented by using

hydroquinone. The polyester site of unsaturation crosslinked with the styrene and forms a linear polymer over copolymers. The polystyrene chain extends to the point where one polyester chain becomes unsaturated. Such crosslinks are to be two to three monomer units long. The reaction water accounted for 8% of the overall material weight. Later the raw high molecular resin was transferred to the styrene vessel shown in Fig.-1b with slow mixing of about 100 rpm. After the final resin preparation, an accelerator must be added to the resin, and the color changes from pale yellow to pale pink shown in Fig.-1c. Aluminum trihydrate (ATH) and Ammonium polyphosphate (APP) are both commonly used as fire retardants in unsaturated polyester resins. ATH (Aluminum Trihydrate) is a hydrated aluminum salt that, when heated releases water vapor and helps to suppress combustion. It also acts as a smoke suppressant by releasing CO₂ when exposed to heat. APP (Ammonium Polyphosphate) is a phosphorus-based fire retardant that forms a protective char layer on the material's surface, slowing the combustion process by insulating it. When used together, ATH and APP synergistically enhance fire resistance: ATH releases water vapor, while APP creates a char barrier. ATH is typically added directly to the resin during manufacturing, whereas APP is often incorporated as a master batch. The proportions of ATH and APP depend on the desired fire resistance level and the specific application requirements. ATH is particularly effective at reducing smoke, while APP is more efficient at slowing fire spread. Therefore, selecting the appropriate fire retardant should align with the specific fire safety standards and application needs. In a typical formulation, the filler-to-resin ratio is 1:1, with 60% ATH and 40% APP added to the polyester resin before introducing hardening agents. This method allows for the modification of various resin types in practical applications by both resin manufacturers and processing companies. The additives are first mixed using a high-speed rotation mixer at approximately 4500 RPM for about five minutes. The process continues in an ultrasonic mixer equipped with a turbine mixer. After 80 to 90 minutes of stirring, a stable and uniform resin composition with flame retardants is achieved.

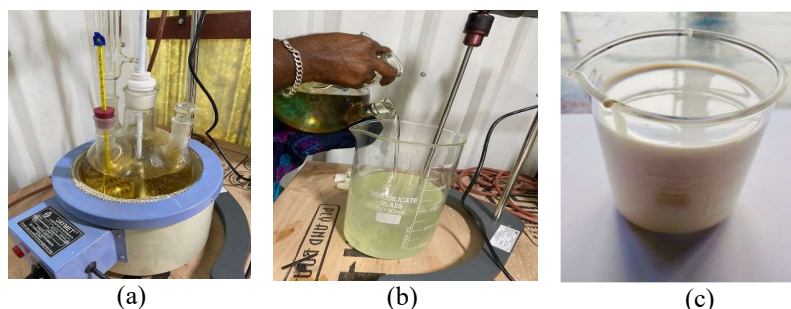


Fig.-1: (a.) the Resin at the Reactor, (b.) the Resin Transferring to the Styrene Monomer Vessel, and (c.) the Pre-Accelerated Resin

RESULTS AND DISCUSSION

Liquid Properties

After adding the preaccelerator, the resin undergoes a color change from pale yellow to creamy white, which also results in a shorter gel time and faster curing. A promotor was used to adjust the gel time and control the maximum exothermic temperature. The viscosity at room temperature (25 °C) was measured using a Brookfield viscometer with spindle two at 12 rpm, following the ISO 2555-2018 standard. Specific gravity was also determined at the same room temperature according to the ISO 2811-2016 standard. Additionally, the content of volatile components was measured following ISO 3251-2019, and the acidity test was performed using ASTM D 4662-87. Gelation time and exothermic peak temperature were recorded according to ISO 2535-2001 and ISO 584:1982 respectively. All tests were conducted on the material in its liquid state at 25 °C. The tested values of the resin were compared to those of industrial resin, revealing that the resin possessed good liquid-state properties.

Standard Resin Cast Procedure

Using the catalyst Methyl Ethyl Ketone Peroxide (M50) and the accelerator cobalt naphthenate (6% solution in styrene), the generated ortho resin was added cobalt 6 percent of 0.2 percent and initially cured at room temperature.¹⁵ To achieve a satisfactory gel time, for 100 g of Resin 6% cobalt of 0.25%, resin 1gm

of Butanox M/50 is used to measure gel time, cure time, and Peak Exotherm. For Tensile strength, Flexural strength, impact strength, HDT, and water absorption tests, separate specimens are poured into the appropriate molds. At average temperatures, the drying process took 24 hours.

Table-1: Shows the Liquid State Material Data at a Room Temperature of 25 Degrees

Properties	Test Method	Units	1	2
Appearance	-	-	Pale Yellow	Creamy White
Specific Gravity @25°C	ISO 2811:2016	-	1.09	1.25
Viscosity -Brookfield @ 25°C sp2/12Rpm)	ISO 2555: 2018	mPa (cps)	197	542
Volatile Content	ISO3251-2019	%	40	28
Acid value	ASTM D 4662-87	mgKOH/g	18	12
Geltime@25°C	ISO 2535: 2001	Minutes	44	24
Peak Exotherm Temp.	ISO 584: 1982	°C	183	120

1* is the synthesized resin data. 2* is the resin properties without additives

Test Results of Cured Cast

After curing, the specimens underwent five tests to determine their tensile strength, modulus, impact strength, hardness, toughness, elongation at break, and water absorption. Tensile strength was using a UTM machine according to ASTM D792. Flexural properties were determined using the same UTM machine with the ISO 527-4 flexural test. Hardness values according to ASTM D-2240. For heat deflection, the temperature is conferring to the ASTM D648 method. The specimens must undergo a water absorption test for seven and 28 days according to ISO62-1999 standards. Casting resin and Laminate materials undergo testing on all material attributes. (see Tables-2 and 3).

Table-2: Shows the Properties of the Cured Cast (Unfilled Cast)

Property	Units	1	2 ¹⁵	3 Commercial Resin ¹¹
Tensile strength	N/mm ²	66.36	42.5	38
Tensile Modulus	KN/mm ²	4.40	18.25	14.10
Elongation to break	%	1.92	2.5	2.4
Flexural strength	N/mm ²	98.06	-	-
Flexural Modulus	KN/mm ²	3.36	-	-
Barcol Hardness	-	84.6	87	88
HDT	°C	69.9	-	-
Water absorbtions:7dyas	%	0.21	0.25	0.21
Water absorbtions:28dyas	%	0.25	-	-

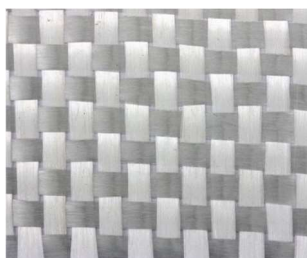
1* is the synthesized resin data. 2* and 3* are the referred from reference

Fabrication of Polymer Composite and its Testing.

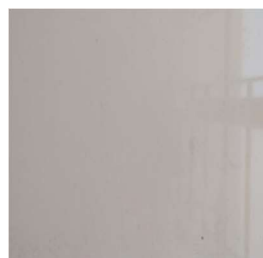
Both commercial resin and synthesized resins are laminated using the hand lamination method with the stacking arrangement of Csm300/wr600 x 5. The resin-to-mat ratio is 3.2, meaning: That 1kg of overall glass fiber consumed 3.2 kg of resin. Maintained the same reinforcement of glass fiber content to compare the results of commercial resin. The specimens are cut from the laminate using a trimming blade for the tensile specifications of ASTM D3039. Failure has happened in the span length at the load of 21.23 KN; the sample has a mean thickness of 4.38 mm and a width of 24.77 mm. The tensile strength of the sample is 182.45 Mpa. Each value represents the average over five samples, and the loading graph is displayed below(Fig.-3).



(a)



(b)



(c)

Fig.-2: (a.) CSM300 Glass Fiber *, (b.) Woven Roving600 Glass Fiber and *(c) Final Composite Laminate

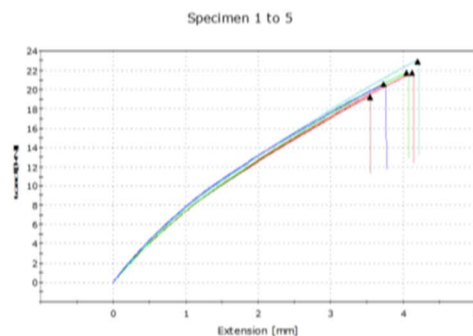


Fig.-3: Shows the Load Vs. Extension for Tensile Strength

The specimens are tailored from the laminate using a trimming blade per the dimensions of ASTM D790 for Flexural. Observed the specimen has a mean of 4.80 mm thick, a width of 11.85mm, span length failure with a load of 447.30 N, and flexural strength was 188.45 Mpa. All the values are means of 5 specimens, and the loading diagram is displayed below.

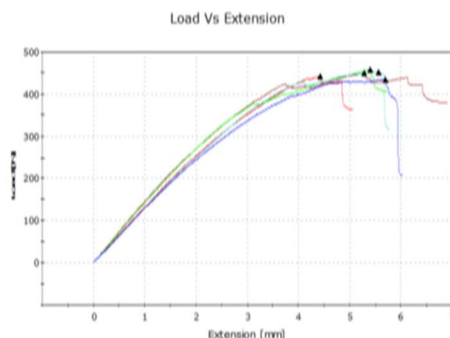


Fig.-4: Shows the Load Vs. Extension for Flexural Strength

The specimens are tailored from the laminate using a trimming blade per the dimensions of test ISO 179 for the Charpy impact test. The sample has a mean of 4.91 mm thick, a width of 8.51mm, and observed Energy (J) 5.49 with an impact resistance of J/m. The impact strength was 131.84 kJ/m². All the values are means of 5 specimens.

Table-3: Shows the Composite Mechanical Properties

Property	Unit	1	2
Density	Kg/m ³	1862	-
Tensile strength	N/mm ²	182.45	100
Tensile Modulus	N/mm ²	19084.22	-
Flexural strength	N/mm ²	188.45	170
Flexural Modulus	KN/mm ²	12097.09	9000
Impact Strength	KJ/m ²	138.73	249.92
Glass content	%	41	40
Water absorption: 7 days	mg	0.31	-
Water absorption: 28 days	mg	0.39	-

1* is the synthesized resin data. 2* is referred from reference²³

The higher crosslinking sites, Tensile strength, and modulus are higher than the other commercial resins. PG alone doesn't have a suitable property of tensile strength when PG combined with other glycols like EG and DEG found good properties. Using the DEG combination to a certain extent with the PG is recommended for almost all manufacturing processes like LRTM, Infusion, RTM, and Vacuum bagging because of less viscosity to the EG. PG with DEG Combination had more Elastic properties and a high break point over the PG with EG. Behind the 20% of DEG with the PG leads to down the tensile modulus properties sharply.¹¹ MA can be used in formulations between 50 and 70 percent of the time to improve modulus.¹⁰

Composite Flammable Properties

The Limiting Oxygen Index (LOI) method is a test used to measure the minimum concentration of oxygen required to sustain the combustion of a material. It is commonly used to evaluate the fire-retardant properties of materials and was performed by ASTM D2863. The test results are reported as the material's Limiting Oxygen Index (LOI). The UL 94 Vertical and Horizontal Flame Test, also known as the "UL 94 V Rating" test, is a standard method used to evaluate the flammability of plastic materials. The test results are then classified into three categories: V-0, V-1, or V-2. The Smoke Index Test, also known as the "Smoke Density Test" or "Smoke Development Test," is a method used to evaluate the smoke-producing characteristics of materials. The test is performed by exposing a material sample to a controlled flame and measuring the amount of smoke produced, and results are reported as a smoke index value. It is important to note that these tests only measure specific properties of materials and do not consider other fire-related factors such as heat, pressure, and the presence of different materials. The Deterioration of Visibility test, also known as the "Smoke Obscuration Test" or "Smoke Visibility Test," is used to evaluate the ability to see through smoke generated by materials when exposed to a flame. The test results are reported as a smoke obscuration value, the percentage of light transmission through the smoke. It is commonly used to evaluate the performance of materials used in construction, transportation, and other industries where smoke production is a concern. The test is typically performed by ASTM E84 or UL 723 standards. Fire resistance of rigid thermoplastics refers to the ability of these materials to maintain their structural integrity and strength when exposed to fire. Rigid thermoplastics are commonly used in construction and transportation. The melting point (DSC) of a polymer, such as a polyester resin, is the temperature at which the polymer transitions from a solid to a liquid state. One way to measure the melting point of a polymer is by using Differential Scanning Calorimetry (DSC). The melting point of polyester resins falls in the range of 150-160°C (302-320°F).

Table-4: Shows the Composite Retardant and Resistance Properties

Test type	Unit	Values
Limited Oxygen Index	%	32.50
Melting Point	°C	321.44
Horizontal Flame Test	Mm/min	0
Vertical Flame Test	Mm/min	0
Smoke Toxicity Index	-	0.91
Deterioration of Visibility due to smoke	Class	A
Laminate fire resistance (LW)	Class	A
Laminate fire resistance (CW)	Class	A

The higher the LOI value, the more difficult it is for the material to catch fire and sustain combustion. This makes the material more fire-resistant. The test results can be used to choose the appropriate materials for a specific application and to ensure that a material meets the fire safety standards.

CONCLUSION

Composites with the compositions of 40:60 (MA: PA) and 80:20 (PG: DEG) exhibit good mechanical and fire-retardant properties. In observations, it was noticed that the phthalic anhydride ratio will enhance the robust and flexible nature of composites. However, propylene glycol-based polymers are crystalline-free, less intense, flexible, and compatible with styrene when used with ethylene glycol. Even noticed that if the DEG ratio increases the resin loses its tensile strength, modulus, and water resistance. According to the regulations, the limited oxygen index test produced a satisfactory result of a 32.5 percent index, a melting point of 321.44 degrees as per the DSC method, smoke toxicity of 0.91 and class A fire resistance. The laminate performed well in every flammability testing point of 321.44 degrees, smoke toxicity of 0.91 and fire resistance of class A as per the standards. All flammable tests for the laminate showed the best properties. The adhesion properties of the material are reduced in the fire-retardant cast compared to the unfilled cast.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

Rekam Manikumar  <https://orcid.org/0000-0003-4829-6800>

T. Nageswara Rao  <https://orcid.org/0000-0003-1767-5699s>

Sannapaneni Janardan  <https://orcid.org/0000-0003-0609-6587>

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