

Effect of Structure on Mechanical Properties of Vinyl Ester Resins and Their Glass Fiber-Reinforced Composites

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ABSTRACT: The article describes the effect of structure of vinyl ester resins (VE) on the mechanical properties of neat sheets as well as glass fabric-reinforced composites. Different samples of VE were prepared by reacting ester of hexahydrophthalic anhydride (ER) and methacrylic acid (MAA) (1 : 1 molar ratio) followed by reaction of monomethacrylate terminated epoxy resin with glutaric (E) or adipic (F) or sebacic acid (G) (2 : 1 molar ratio). The neat VE were diluted with styrene and sheets were fabricated by using a glass mold. A significant reduction in the mechanical properties was observed by increasing the methylene content of resin backbone (i.e., sample E to G). Glass fabric-reinforced composites were fabricated by vacuum

assisted resin transfer molding (VARTM) technique. Resin content in the laminates was 50 ± 5 wt %. Increase in the number of methylene groups in the vinyl ester resin (i.e., increasing the bridge length) did not show any significant effect on limiting oxygen index (LOI) value (21 ± 1) of the laminates but tensile strength, tensile modulus, flexural strength, and flexural modulus all increased though these values are significantly lower than observed in laminates based on resin B. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 108: 1942–1948, 2008

Key words: mechanical properties; fibers; composites; thermal properties

INTRODUCTION

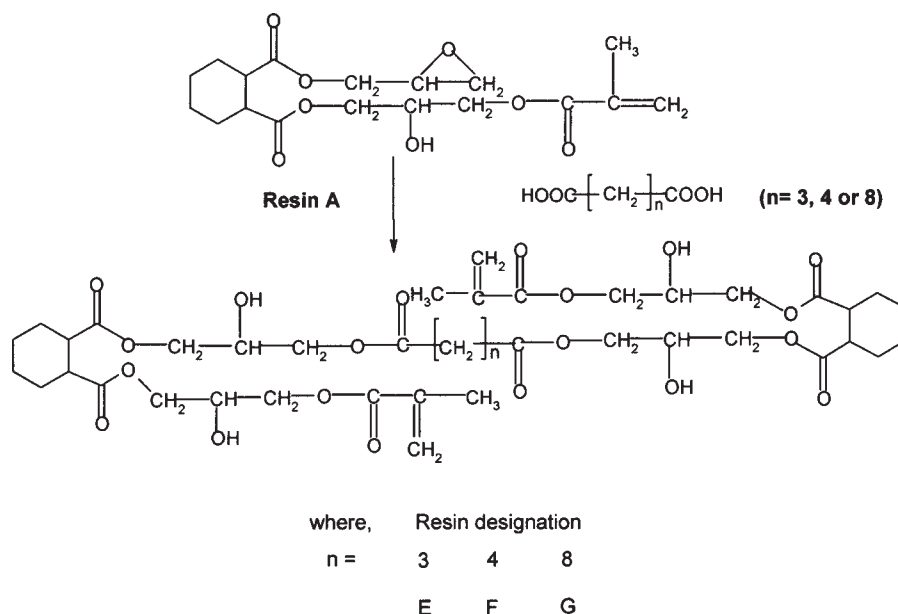
Vinyl ester resins (VE) are widely used as thermoset matrix resins to fabricate a variety of reinforced structures including pipes, tanks, scrubbers, and ducts. They are prime candidates for use in composites for transportation and/or infrastructure. This is because of their high specific strength and modulus, high corrosion resistance, good performance characteristics, and ease of fabrication. Most of these studies have been carried out on VE based on diglycidyl ether of bisphenol A (DGEBA).^{1–5} Efforts are being made to develop VE resins with better toughness characteristics, lower viscosity, and reduced shrinkage during cure and lower moisture absorption. The environmental concern has also necessitated the studies on resin formulations with low volatile evolution during processing. No studies have been reported on VE resin based on cycloaliphatic epoxy resins. It was therefore considered of interest to investigate vinyl ester resin based on cycloaliphatic epoxy as matrix resin in glass fabric-reinforced composites and evaluation of their mechanical properties

such as interlaminar shear strength, flexural strength, tensile strength, impact strength, etc.

The mechanical properties of fibrous composites, such as tensile strength and modulus, flexural strength and modulus, interlaminar shear strength, compressive strength, etc., depends on the structure of matrix resins, properties of fibers, volume fraction of fibers (V_f), and interphase. The structure of cross-linked network of VE can be altered by changing the backbone structure or end groups or the nature and concentration of reactive diluents (e.g., styrene).^{6,7} Higher interlaminar shear strength of glass fiber-reinforced laminates was observed in our earlier studies with VE containing one epoxy end group.⁶ Styrene imparts good mechanical properties (i.e., tensile strength), heat distortion resistance, and dielectric properties to cured resins.¹ However, an increase in the styrene content resulted in a decrease of tensile modulus.⁶

In our previous articles, we have reported the synthesis and characterization of methacrylate terminated cycloaliphatic epoxy resin⁸ obtained by reacting 1 : 1 or 1 : 2 molar ratio of diglycidyl ester of hexahydrophthalic anhydride (ER) and methacrylic acid (MAA). These resins have been designated as A and B, respectively. The structure of backbone of resin A was altered by reacting 2 moles of monomethacrylate terminated epoxy resin (sample A) with

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Scheme 1 Chain extension of monomethacrylate terminated epoxy resin (sample A) with dicarboxylic acids.

1 mole of glutaric (sample E) or adipic (sample F) or sebacic acid (sample G) (Scheme 1)⁹ and their mechanical properties were compared with dimethacrylate terminated epoxy resin (sample B).

In this article, we report the effect of structure of VE resin on the mechanical properties of cured VE resin containing 40 phr of styrene and glass fabric-reinforced laminates. The laminates were fabricated by vacuum assisted resin transfer molding (VARTM) technique.

EXPERIMENTAL

Materials

The epoxy resin, i.e., (diglycidyl ester of hexahydrophthalic anhydride) from Atul Limited, Vadodara, Gujarat, (available under the trade name of XR-34) was used as received. Methyl ethyl ketone peroxide (MEKP) (G. S. Chemicals, Mumbai, India), cobalt octoate (gift sample from ICI R&T Centre, Mohali), and *N,N*-dimethyl aniline (DMA) (Qualigens, Mumbai) were used as received. Styrene (Qualigens, Mumbai) was washed with 10% NaOH solution to remove inhibitor followed by washing with water several times and then dried overnight over anhydrous sodium sulfate and then distilled under reduced pressure. Epoxy compatible woven roving (density 254 g/m²) of Pilkington Fiber Glass Co. was used as reinforcement.

The dimethacrylate terminated epoxy resin (B) and monomethacrylate terminated epoxy resin (sample A) were prepared by reacting epoxy resin

and MAA (molar ratio 1 : 2 or 1 : 1, respectively) in the presence of imidazole as a catalyst at a temperature of (90 ± 5)°C. The reaction was carried out in a four-necked reaction kettle equipped with a mechanical stirrer, oxygen inlet, water condenser, and thermometer. The reaction was stopped when the acid number reached a value <10.⁸

Chain extension reaction of monomethacrylate terminated resin A was done by reacting with glutaric acid/adipic acid or sebacic acid in a molar ratio of 2 : 1 to yield bismethacrylate terminated vinyl ester resin E, F, and G, respectively.⁹

Resin characterization

The resins B, E, F, and G were used as matrix resins. Styrene (40 phr) was used as reactive diluent. A Brookfield Synchroelectric Viscometer having spindle No. 2 and 3 (RV type) was used to determine the viscosity at 20 rpm of these resins at room temperature (25 ± 2)°C.

Gel time of VE resins at room temperature in the presence of reactive diluent was determined using MEKP (0.18 phr), *N,N*-DMA (0.06 phr), and cobalt octoate (0.15 phr) as initiating system. 6 ± 1 g of VE resin containing reactive monomer was placed in a tube kept in a constant temperature water bath maintained at (25 ± 2)°C. The solution was stirred continuously. A gradual increase in temperature was observed initially followed by a sharp increase in temperature. Rise in temperature of the samples as a function of time was plotted and gel time was determined by extrapolation.

Neat resin castings

Glass plates coated with a solution of poly(vinyl alcohol) and having a Teflon spacer of the desired thickness were clamped to give a mold for casting of neat resin. The vinyl ester resin containing 40 phr of reactive diluent and MEKP (0.18 phr), *N,N*-DMA (0.06 phr), and cobalt octoate (0.15 phr) was carefully poured into the mold and precaution was taken to remove all trapped air bubbles from the solution. This assembly was left at room temperature for 12 h. Then transparent sheets of VE were removed and post curing of the sheets was done at 120°C for 2 h in an air oven. The cured samples have been designated by letter designation of resin sample followed by the letter "S" (indicating styrene as reactive diluent). For example, sample B and E diluted with 40 phr of styrene have been designated as BS and ES, respectively.

Fabrication of glass fabric-reinforced composites

The glass fabric/vinyl ester resin composites were prepared by vacuum assisted resin transfer molding (VARTM). Eight plies of 22 × 22 × 0.2 cm³ of glass fabric were placed in a steel mold. The resin, diluent, and curing agents were then introduced into the mold under a pressure assisted by vacuum. The introduction of resin was continued till bubbles stopped in the resin flowing out from the exit of the mold. The mold was then left at room temperature for 12 h and then the mold was kept in an air oven at 100°C for 4 h. The composite was then removed. Post curing of the laminate was done for 4 h under contact pressure using Carver hydraulic press for 2 h each at 100°C and 120°C. The glass fabric-reinforced laminates were designated as BSg and ESg, respectively, where "B" or "E" stands for matrix resin, "S" stands for styrene, and "g" stands for glass.

Characterization of neat sheets and composites

The resin content of the laminates was determined by heating weighed samples in a furnace at 600°C for 3 h. The glass fibers were collected and weighed.

From the difference in weights the resin content was obtained.

Density and void content of the composites were determined according to ASTM D792-66 and ASTM D 2734-70, respectively. The matrix density was measured on a sample of pure matrix formulation cured simultaneously beside the composite. The composite void content (V_v) can be calculated by the following equation:

$$V_v = 100 \times \left[\frac{\rho_t - \rho_e}{\rho_t} \right]$$

where ρ_t is the theoretical density of the composite (calculated using rule of mixture), ρ_e is the measured composite density, and V_v is the void content, volume %.

Swelling measurements of cured matrix resin was done at (22 ± 3)°C in ethanol, toluene, dimethyl sulphoxide (DMSO), *n*-butanol, ethylene glycol, and water. Cured samples were cut approximately into rectangular sizes having a weight of 0.1–0.2 g. The samples were dried at 80°C till constant weight obtained. These were then immersed in 20–25 mL of organic solvents (purified by distillation) in stoppered conical flasks. The samples were taken out at regular intervals of time, surface dried by filter paper, and weighed. This process was repeated till equilibrium swelling was obtained. The swelling coefficient Q was evaluated by using equation:

$$Q = \frac{m - m_0}{m_0} \times \frac{1}{d}$$

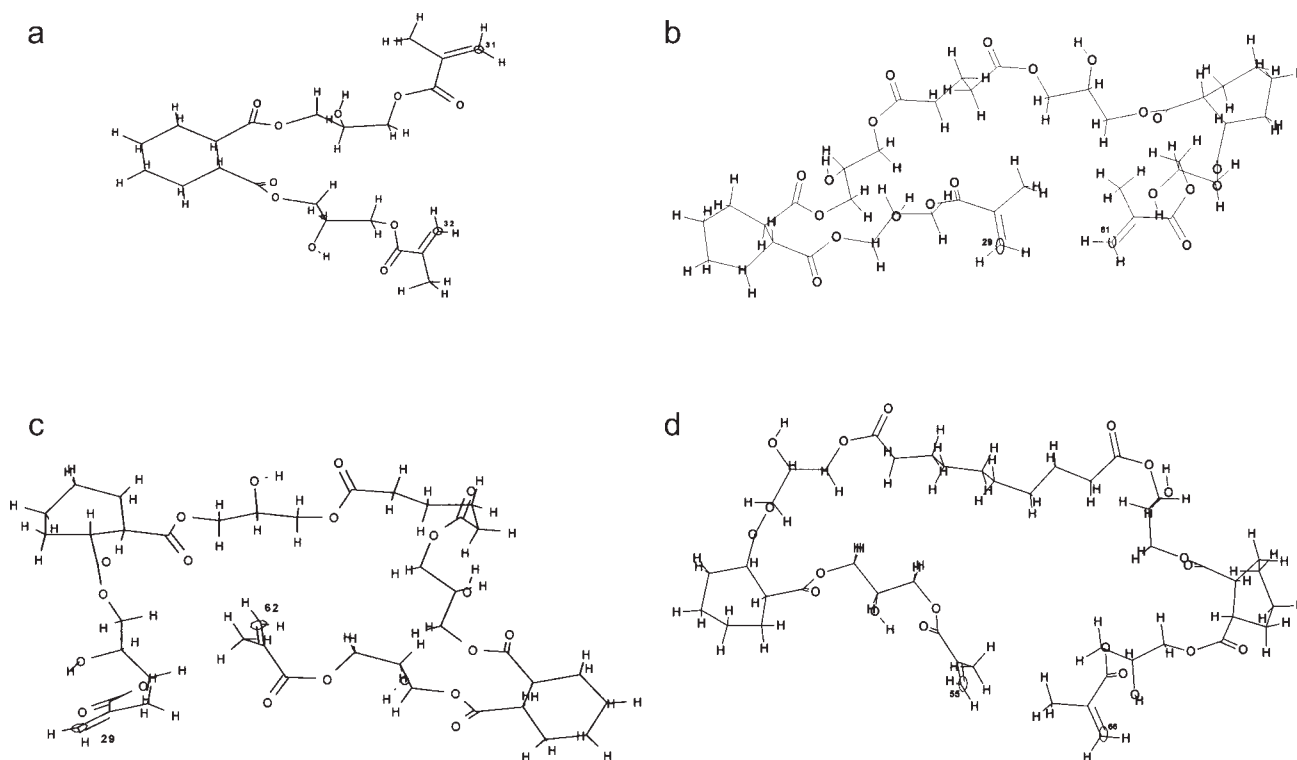
where m is the weight of the swollen polymer, m_0 is the weight of the polymer taken initially, and d is the density of the solvent.

Cured samples of VE were crushed into a powder for recording TG/DTG traces. Relative thermal stability of cured matrix resins in nitrogen atmosphere was determined using TA 2100 thermal analyzer having a 951 TG module or Perkin Elmer Pyris 6 TGA. A heating rate of 20°C/min and a sample mass of 10 ± 1 mg was used for recording TG/DTG traces. The relative thermal stability of the cured samples was evaluated by comparing the initial

TABLE I
Mechanical Properties of Neat Vinyl Ester Sheets

| Resin designation | Density (g/cm ³) | Tensile strength (MPa) | Tensile modulus (MPa) | % Elongation at break | Flexural strength (MPa) | Flexural modulus (GPa) | Impact energy (KJ/m ²) |
|-------------------|------------------------------|------------------------|-----------------------|-----------------------|-------------------------|------------------------|------------------------------------|
| BS | 1.177 | 37.2 (0.16) | 600 (70) | 3.64 (1.3) | 93.3 | 3.8 | 42.11 |
| ES | 1.223 | 2.7 (0.33) | 10 (2) | 76.5 (2.9) | – | – | 48.37 |
| FS | 1.211 | 8.6 (0.68) | 60 (9) | 45.2 (10.8) | – | – | 48.11 |
| GS | 1.186 | 2.4 (0.29) | 6 (0.2) | 82.1 (10) | – | – | 38.49 |

Figure in parenthesis indicate the variation (±) of the data.



Scheme 2 Conformational structure of (a) Resin B, (b) Resin E, (c) Resin F, and (d) Resin G.

decomposition temperature (T_i), temperature of maximum rate of mass loss (T_{max}), and final decomposition temperature (T_f).

The tensile, flexural, and interlaminar shear properties of neat sheets were determined using Zwick tensile testing machine Model Z010. Tensile testing of sheets was done according to ASTM D-638 at a cross-head speed of 1mm/min. An Instron tensile tester 5582 was used for determining tensile properties of laminates according to ASTM D 3039. An average of four specimens has been reported.

Flexural properties of the laminates and neat sheets were determined according to ASTM D-790. All samples had a width of 1.27 cm. A crosshead speed of 5 mm/min was used.

The interlaminar shear strength (ILSS) was determined according to ASTM D-2344-76. A span-to-depth ratio of 5 : 1 and a crosshead speed of 1 mm/min were used. ILSS of the samples was also determined after isothermal aging at 150°C for 240 and 500 h and at 180°C for 24 and 125 h. The effect of hygrothermal aging of the samples at a relative humidity of 80 and 100% on ILSS was also determined. These studies were carried out for a maximum of 360 h.

Hygrothermal and isothermal aging behavior was also evaluated in terms of the weight gain of composite specimens. Prior to the hygrothermal or isothermal aging, all specimens were dried to a constant weight. Hygrothermal aging of the samples

was done at 80% RH and samples were weighed at regular intervals of time after removing surface water by using a filter paper. For isothermal aging, the samples were kept at 150°C for 240 h and the change in weight was noted after regular intervals of time.

Limiting oxygen index (LOI) was determined according to ASTM D 2863-77. The impact testing machine (Model IT-042) was used to determine the impact energy of the composite and neat resin sheets. According to Izod method ASTM D 256-73 for the composites a notch angle was 45° and notch depth of 2.4 mm was used.

RESULTS AND DISCUSSION

The main objective of our studies was to investigate the effect of backbone structure on the mechanical

TABLE II
Distance Between the Two Terminal Double Bonds and Total Energy of the Molecule on the Basis of Conformational Structures

| Resin designation | Distance between terminal double bonds (Å) | Total energy (Kcal/mol) |
|-------------------|--|-------------------------|
| B | 9.1202 | 7.9749 |
| E | 4.2889 | 30.0409 |
| F | 8.0242 | 25.3878 |
| G | 6.0213 | 29.1032 |

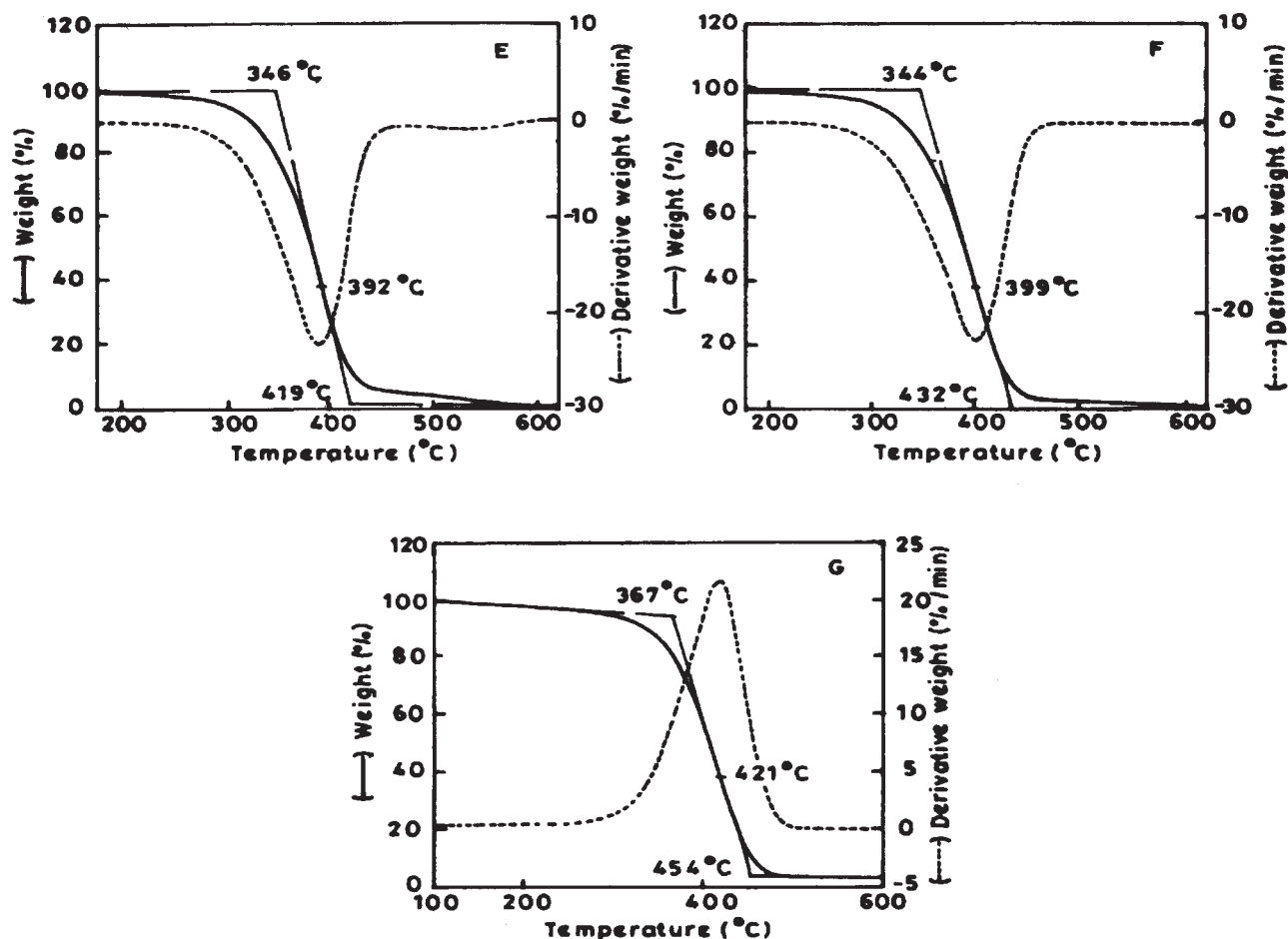


Figure 1 TG/DTG traces of cured matrix resins (a) ES, (b) FS, and (c) GS.

properties of neat sheets of VE and glass fabric-reinforced composites.

Properties of neat VE resin

The Brookfield viscosity of resin formulations BS and GS was 400 cps while a value of 350 cps was observed in ES and FS, respectively.

The gel time of resin formulation BS was 12 min and for ES, FS, and GS, it was in the range of 20–22 min. The density of cured resin samples was in the range of 1.17–1.27 g/cm³. The mechanical prop-

erties of neat sheets depended on the resin structure. The data for tensile strength and modulus of neat sheets was summarized in Table I. Lowest tensile strength and tensile modulus was in case of GS. The order of tensile strength of neat sheets was BS > FS > ES > GS. There was a significant reduction in the tensile strength by introduction of flexible units between the crosslinks. However, a significant increase in elongation at break was observed due to the introduction of flexible methylene units between the crosslinks. The order of elongation at break was GS > ES > FS > BS.

The main difference between the various vinyl ester resin samples was the presence of 3 (ES),

TABLE III
Values of Swelling Coefficients of Various Resins in Different Solvents at (22 ± 3)°C

| Solvent | Swelling coefficients Q (cm ³ /g) of resins | | | |
|-------------------|--|--------|--------|--------|
| | BS | ES | FS | GS |
| <i>n</i> -Butanol | 0.0057 | 0.2878 | 0.2665 | 0.313 |
| Ethanol | 0.1185 | 0.1085 | 0.1088 | 0.071 |
| Ethylene glycol | 0.0083 | 0.1977 | 0.140 | 0.147 |
| DMSO | 0.0929 | 1.011 | 0.851 | 0.797 |
| Water | 0.0135 | 0.1283 | 0.1244 | 0.1069 |
| Toluene | 0.0031 | 0.2177 | 0.2439 | 0.3126 |

TABLE IV
Results of Thermogravimetric Analysis of Cured Matrix Resins

| Resin designation | T_{onset} (°C) | T_{max} (°C) | T_f (°C) |
|-------------------|-------------------------|-----------------------|------------|
| BS | 360 | 416 | 446 |
| ES | 346 | 392 | 419 |
| FS | 344 | 399 | 432 |
| GS | 367 | 421 | 454 |

TABLE V
Density, Void Content, and LOI of Glass Fabric-Reinforced Laminates

| Resin designation | Density (g/cm ³) | Void content (%) | LOI |
|-------------------|------------------------------|------------------|------|
| BSg | 1.621 | 1.87 | 21.3 |
| ESg | 1.581 | 0.62 | 22.4 |
| FSg | 1.579 | 1.61 | 22.6 |
| GSg | 1.565 | 4.8 | 22.5 |

4 (FS), or 8 (GS) methylene group in the backbone of BS resin. However, the results obtained in the present studies show an abnormal behavior of resin FS. This may be due to the possibility of intra/intermolecular crosslinking. Therefore, molecular modeling was done to explore the possibility of intramolecular crosslinking reaction between the vinyl unsaturation of the resins. The conformational structure of resin B, E, F, and G are given in Scheme 2. The distance between the two terminal double bonds and total energy of the molecule are given in Table II. The end-to-end distance is higher in B and F but relatively low in E and G resin. It can therefore be proposed that in resin B and F the terminal vinyl groups within a molecule are more amenable for intermolecular crosslinking thereby leading to higher tensile strength and tensile modulus. In resin E and G, the distance between the double bonds of terminal vinyl groups are less and therefore may be involved more in intramolecular cross-bonding which may be responsible for observed lower tensile strength and tensile modulus in comparison to resin B and F.

Flexural strength and flexural modulus of the sample BS was highest. Samples ES, FS, and GS were very flexible and it was difficult to measure the flexural strength and modulus of these samples. Impact energy was higher in ES and FS sample. However, there was a marginal effect on impact strength with the change in backbone structure.

From the swelling coefficient, it can be observed that maximum swelling of samples ES, FS, and GS was in *n*-butanol while resin BS swells maximum in ethanol. However, all the networks exhibited low swelling indicating highly crosslinked systems. The vinyl ester-styrene network with a relatively high precursor molecular weight (resin ES, FS, and GS ~ 1200 g/mol) had higher swelling coefficient than the vinyl ester-styrene networks with relatively low precursor molecular weight (BS) due to the longer chains between crosslinks in the former. Despite the amount of swell, these are insoluble in common solvents and there were no extractable components. These networks swell due to the decrease in network density and/or dangling ends.

TG/DTG traces of crosslinked resins are shown in Figure 1. All the resins were stable up to 340°C and decomposed in a single step. In chain extended VE resin, an increase in bridge length resulted in an increase in onset decomposition temperature (T_{onset}) of resin samples. The maximum decomposition temperature and the final decomposition temperature were above 390°C (Table III).

Properties of composites

The density of the laminates was in the range of 1.565–1.646 g/cm³. The void content was lowest in ESg and was highest in GSg. The LOI of all the laminates was comparable and was 21.8 ± 0.5 . This was expected because flexibility or rigidity of backbone is not going to affect the burning characteristics (Table IV).

The resin content of laminates was in the range of $50 \pm 5\%$ (wt). Tensile strength of the glass fabric-reinforced laminates with resin BSg was 221 MPa. A significant reduction in tensile strength was observed by introducing flexible units between the crosslinks (resin E, F, and G). An interesting observation was that by increasing the number of methylene groups in the repeat unit, i.e., changing the resin from E to

TABLE VI
Mechanical Properties of Glass Fabric-Reinforced Laminates

| Resin designation | Resin content (wt %) | Flexural strength resin (MPa) | Flexural modulus (GPa) | Tensile strength (MPa) | Tensile modulus (GPa) | % Elongation at break | Impact strength (KJ/m ²) |
|-------------------|----------------------|-------------------------------|------------------------|------------------------|-----------------------|-----------------------|--------------------------------------|
| BSg | 46.6 | 175.4 (9.7) | 10.5 (1.1) | 221.7 (7.9) | 6.3 (0.25) | 4.7 (0.3) | 0.131 |
| ESg | 55.6 | 24 (2.3) | 1.9 (0.2) | 98.5 (10.2) | 2.4 (0.14) | 6.6 (0.9) | 2.816 |
| FSg | 53.1 | 83 (7.7) | 8.6 (0.7) | 122.0 (25.6) | 3.9 (0.13) | 7.4 (3.6) | 1.812 |
| GSg | 47.7 | 89.4 (6.7) | 7.1 (0.7) | 207.9 (7.9) | 6.3 (0.83) | 5.3 (0.7) | 2.3 |

Figures in parenthesis indicate the variation (\pm) of the data.

TABLE VII
Interlaminar Shear Strength (MPa) of the Laminates in Hygrothermal and Isothermal Conditions

| Resin designation | ILSS (MPa) | | | | | | |
|-------------------|--------------------------------------|---|-----------------|-------------------------|------|-------|------|
| | Hygrothermal aging at (25 ± 2)°C for | | | Isothermal aging (h) at | | | |
| | 0 | 168 h, 80 % R. H followed by 72 h, 100 % R. H | 360 h, 80% R. H | 150°C | | 180°C | |
| | | | 240 | 500 | 24 | 125 | |
| BSg | 14.3 | 11.56 | 6.9 | 15.2 | 15.4 | 16.8 | 13.1 |
| ESg | 3.06 | 1.56 | 2.26 | 20.5 | 17.7 | 17.4 | 18.1 |
| FSg | 5.68 | 4.44 | 3.35 | 14.0 | 20.1 | 19.5 | 19.1 |
| GSg | 14.4 | 3.3 | 5.04 | 15.4 | 15.8 | 18.8 | 18.6 |

F or G, there was an increase in tensile strength. Tensile modulus was 6.3 GPa in samples BSg and reduced significantly in sample ESg and FSg, respectively. Improvement in properties observed in laminate based on resin G could be explained in terms of localized order of octamethylene unit of different chain thereby leading to crystalline order/phases in an amorphous crosslinked network. Flexural strength in samples ESg, FSg, and GSg is reduced significantly (Table VI).

For sample ESg and FSg ILSS reduced significantly while again in sample GSg it was comparable with BSg (Table VI). In fibrous composites, the tensile properties are dominated by the properties of fiber whereas flexural and shear strength are matrix dominated. Our results indicate that flexural and ILSS properties were higher when resins had more rigidity in the backbone for example BSg resin. Interfacial adhesion depend on the interaction of fiber and matrix and it appears that the physical and chemical interaction give a higher performance, i.e., BSg resin than flexible matrix resin such as E, F, and G (Table VI).

No significant trend was observed in impact strength of glass-reinforced laminates. In laminate BSg, the impact strength was low. This may be due to strong interfacial bond. In other resins, ESg, FSg, and GSg it was comparable and higher than samples BSg (Table VI).

Effect of isothermal aging on ILSS was determined by heating the composites at 150°C for 240 h or for 500 h and at 180°C for 24 h or for 125 h. In almost all the cases, an increase in ILSS was observed on isothermal aging of laminates, this might be due to some residual curing which occurs on heating (Table VII) resulting in the enhancement of bonding between fiber and matrix.

The results of hygrothermal aging on ILSS are summarized in Table VII. There was a reduction in ILSS properties on water absorption. This may be due to the degradation of fiber-matrix interfacial bond.

An increase in weight of laminates was observed after 168 h of hygrothermal aging of samples at

(25 ± 2)°C and 80% RH. In samples BSg the water absorption was 1.84% while in samples ESg, FSg, and GSg it was 1.59%, 1.02%, and 2.58%, respectively. This moisture absorption leads to debonding of glass fibers and matrix and so a decrease in ILSS was observed. During isothermal aging, there was weight loss in the laminates. This may be due to some oxidation of polymer as isothermal aging was done at temperature 150 or 180°C in the presence of air.

CONCLUSIONS

Mechanical properties of glass fabric-reinforced laminates depend upon the formulations and it was found the incorporation of flexible units by chain extension showed no effect on LOI. With increase in bridge length, mechanical properties decrease significantly; however, with increase in the number of methylene groups in the order 2 to 8, there was increase in the tensile strength, tensile modulus, flexural strength, and flexural modulus.

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