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Experimental Study on the Mechanical Properties of Glass Fiber Reinforced Vinyl Ester Composites

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Abstract : This manuscript deals with the fabrication of glass fiber reinforced vinyl ester composite and experimental study on the mechanical properties. In this study glass fiber-reinforced vinyl ester composites with 40:60 ratios were fabricated in laminated specimens using hand lay-up technique and the specimens are subjected to the investigated as per the ASTM standards. Methyl ethyl ketone peroxide (MEKP) is used as catalyst. Cobalt naphthanate were used in the resin to cure at room temperature. The promoter was used to enhance and improve properties of the resin. The curing behavior was investigated for the sample produced in varying proportion of resin mixture and cured at room temperature for 24h and at 100°C for 3h. By incorporating the curing behavior the tensile, compression, flexural and impact strength found to be improved comparatively in heat treated specimen. Micro structural characterization was carried out to examine the morphology in reinforced samples using scanning electron microscope. The radiography test was done to examine the internal structure of the molded specimen.

Keywords: Glass fiber reinforced vinyl ester composite, Hand lay-up technique, Methyl ethyl ketone peroxide (MEKP), Radiography.

1. Introduction

Polymeric composites have high quality and solidness, light weight, and high corrosion protection resistance. This sort of composite is utilized as a part of the best assorted variety of composite applications because of its focal points, for example, low density, good thermal and electrical protector, simplicity of creation with minimal effort [1,2] Thermoplastic are reversible and can be reshaped by application of heat and pressure. Molecules of thermoplastic do not cross-link and hence they are flexible and re-formable. Thermoset materials are brittle and offer greater dimensional stability, better rigidity, chemical, electrical, and solvent resistance. The most common resin materials used in thermo-set composites are epoxy, polyester, phenolics, vinyl ester, and polyimide [3-5]. Glass fiber reinforced polymers are proven and successful alternatives that have numerous advantages over traditional reinforcement methods, giving structures a longer service life. GFRP is permanently resistant to chemical acids and alkaline bases, therefore extra concrete cover, anti-shrink additives, and even cathodic protection are not required [3,4]. GFRP significantly improves the longevity of engineering structures where corrosion is a major factor. Vinyl ester resins are becoming increasingly important in new industrial applications such as coatings, printed circuit boards, metal foil laminates, building materials,

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automotive parts and fiber-reinforced composites [6-8]. The properties of such resins can be improved by incorporating vinyl type reactive diluents such as styrene to upgrade the resin systems as matrix materials for advanced composites. It is interesting to investigate the effect of the styrene content on the properties of glass-fiber reinforced composites [9-12]. This research work deals with the synthesis, characterization and curing behavior of vinyl ester resin and the study the effect of styrene content on the properties of glass-fiber-reinforced vinyl ester composites.

2. Materials and Sample Preparation

2.1 Materials

VBR 4508 grade vinyl ester is used as the matrix material. It is a medium reactive vinyl ester based on Bisphenol-A. The VBR 4508 vinyl ester incorporates the workability and curing properties. The physical properties of matrix material are tabulated in the Table 1.

Table 1 Properties of VBR 4508 Grade Liquid Vinyl Ester Resin

Appearance	Pale yellowish clear liquid
Viscosity at 25°C in centipoise(cp)	300-400
Specific gravity at 25°C	1.04-1.06
Acid value,mg of KOH	07/11/17
Gel time at 25°C, minutes	15-35
Peak Exotherm under insulated condition for $100 \mathrm{gm}$ mixture, ${}^{\circ}C$	160-170

Discontinuous random E- glass fiber shown in figure 1 is used as reinforcement and it is a low alkali glass with typical composition is tabulated in the Table 2.



Figure 1 Discontinuous random E- glass fiber

Table 2 Composition of Discontinuous random E-glass Fiber

Sio ₂	Al ₂ O ₃	CaO	MgO	B_2O_3	Na ₂ O	K ₂ O
52–56	12–15	21–23	0.4–4	4–6	0–1	Trace

2.2 Fabrication of the Composites

The fabrication of composites was performed using hand-lay-up method using E glass as reinforcement and Vinyl ester polymer as a matrix. According to the weight calculation Vinyl ester resin is mixed with an accelerator (Cobalt naphthanate), Promoter (Dimethylaniline) and catalyst Methyl ethyl ketone peroxide (MEKP) are mixed in varying proportion shown in Table 3 stirred continuously for 10 minutes. These mixed vinyl ester resin is used to fabricate the composites using Discontinuous random E glass fiber in 60:40 ratio [12,13] shown in Table 4. In the entire study composites are lay up as laminates using wooden die shown in (Figure 2) made as per specimen requirement according to the American Society for Testing and Materials standard [3-4].

Table 3 Varying proportions for Sample 'A' and 'B'

Proportion of Resin for Sample 'A'		Proportion of Resin for Sample 'B'		
Vinyl ester	10.38gram	Vinyl ester	12.93gram	
cobalt-10%	1.04gram	Cobalt-1.5%	0.19gram	
Promoter-10%	1.04gram	Promoter-1.5%	0.19gram	
Catalyst(MEKP)-10%	1.04gram	Catalyst(MEKP)-1.5%	0.19gram	
Total	13.50gram	Total	13.50gram	

Table 4 Typical Formulation of Sample 'A' and 'B'

Required Weight for Testing -	22.5 gram
Resin 60% (22.5*60/100)	13.5 gram
Fiber 40% (22.5*40/100)	9 gram
Total Gram	22.5



Figure 2 Wooden Die for Lay up the Laminates

After the lay-up process, the produced composites are then cured and heat treated in Muffle furnace according to sample designation [12] shown in table 5.

Table 5 Curing of the Laminated composites

Sample designation	Curing time
A1	Normal cooling at room temperature for 8hours
A2	Normal cooling at room temperature for 24hours and Heat treated at 100°C for 3hours
B1	Normal cooling at room temperature for 8hours
B2	Normal cooling at room temperature for 24hours and Heat treated at 100°C for 3hours

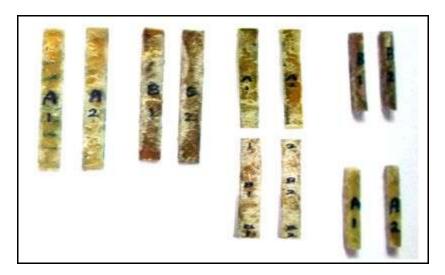


Figure 3 Samples for Testing

3. Sample characterization and Testing

The mechanical behavior such as tensile strength, compression, flexural and impact are characterized on flat specimen shown in (Figure 3) of E glass and vinyl ester composites.

3.1 Tensile Test

The tensile test is performed on the produced specimen using the Universal Testing Machine (UTM) according to ASTM procedures D 3039/3039M [3, 4]. This test method determines the in pane tensile properties of polymer matrix composites. The test piece was sized to the dimension of 250 mm \times 25 mm (length x width x thickness) with end tabs at both ends, by maintaining the cross head speed of 1 mm/min at room temperature.

3.2 Compressive strength

The compressive strength test method determines compressive properties of polymer composite materials by applying combined end-loading and shear-loading using a combined loading compression is designed for polymer matrix composite. The compressive strength was analyzed according to the ASTM 1708 [3,4,6]. The specimens were neatly polished at the sides and corners with help of metallographic polishing machine to the size of 80mm x 20mm x 20mm (length x width x thickness). The tests were carried out with the cross head speed of 1.33 mm/min.

3.3 Flexural test

The flexural test measures the force required to bend a beam under three point loading conditions. This test was carried out as per ASTM 7264 [3,4,7] pertained to testing of plastics, the size of the flexural test pieces were $150 \text{ mm} \times 20 \text{ mm} \times 3 \text{ mm}$ (length \times width \times thickness) with the cross head speed of 2 mm/min.

3.4 Impact strength (Izod and Charpy):

The impact strength was analyzed for all the proportions according to the standard ASTM E23 in Izod Mode Method. The dimensions of the samples 75.00 mm x 10.00 mm x 10.00 mm (Length \times width \times thickness). The impact strength was analyzed for all the proportions according to the standard ASTM E23 [4,6,7,14] in Charpy Test Mode Method. The dimensions of the samples 55.00mmx10.00 mmx10.00mm (Length \times width \times thickness).

3.5 Microstructure characterization

Microstructure portrayal thinks about were led to strengthened examples. This is refined by utilizing checking electron magnifying instrument. The composite examples were metallographically cleaned preceding

examination. The SEM micrographs of the composite were acquired utilizing the examining electron magnifying lens. The images were taken in backscattered electron (BSE) mode according to requirement. Infinitesimal examinations to inspect the morphology, molecule estimate and micro structure were finished by a 6480 LV filtering electron magnifying instrument (SEM) outfitted with a vitality dispersive X-beam (EDX) identifier. The fractured Specimens were examined using scanning electron microscope (SEM) model EVO MA15 to find out inter bonding of Samples[14,15,16].

4 Results and Discussion

4.1 Tensile test

The mechanical characterization on E glass fibre reinforced vinyl ester composites are produced to find the effect on the tensile strength, compression strength, flexural strength and impact strength [12,13,18]. The tensile test specimens of glass fibre reinforced vinyl ester composites are shown in Figure 4(a) and 4(b), and the properties are reported shown in Table 6.



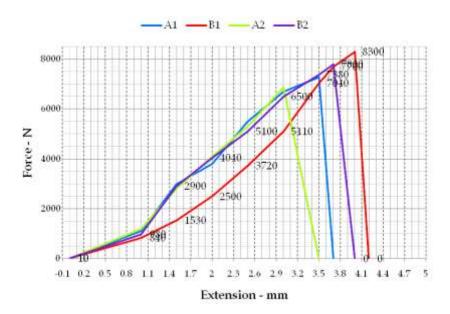
Figure 4(a) Tensile Samples A and B before testing

Figure 4(b) Tensile Samples A and B After testing

Table 6 Tensile properties of E glass vinyl Ester composites

Sampl e	Load N	Yield Strength N/mm ²	Young's Modulus N/mm ²
A1	7280	84.85	7909
B1	8300	96.74	7596
A2	6890	80.30	8555
B2	7800	90.91	7979

From the graphical representation of tensile strength, Graph 1, the sample B1 showed a maximum tensile strength of about 96.74 N/mm² in normal cooling condition for 24 hours and the proportion of the resin also influences in tensile strength. It can be observed from the result that the tensile strength of the composites has great influence in resin proportion [17].



Graph 1 Load vs. Extension curves for E glass vinyl ester specimens.

4.2 Compression Strength

A compressive behavior of vinyl ester composites are under crushing loads. The specimen is compressed and deformation at various loads is recorded. The Compressive strength samples of the glass fibre reinforced vinyl ester composites are shown in Figure 5, and the properties are reported shown in Table 7.

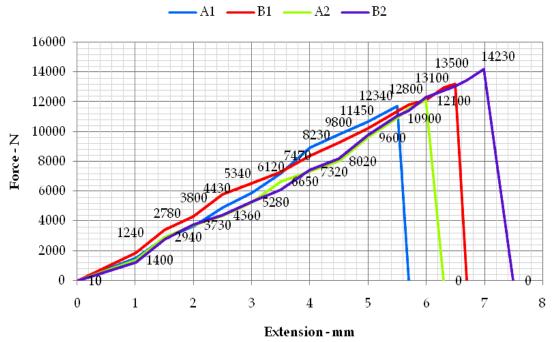


Figure 5 Compression strength test samples

Table 7 Compression properties of E glass vinyl Ester composites

Sample	Load N	Compressive Strength N/mm2	Deflection mm
A1	11700	195	5.5
B1	13200	210	6.3
A2	12100	188	6
B2	14230	216	6.9

Like tensile behavior, compressive strength of the vinyl ester composite also varied with the composition and curing time. From the Graph 2 it is observed that the sample B2 shows the maximum compressive strength of 216 N/mm² in Normal cooling at room temperature for 24hours and Heat treated at 100°C for 3hours and as like tensile strength the resin composition of Sample B also influence the properties [6,14].



Graph 2 compressive strength curves for E glass vinyl ester specimens

4.3 Flexural Strength

From the experimental characterization of vinyl ester composites the Flexural test is to define the properties of a material to withstand the bending before reaching the breaking point. The Flexural strength samples of the glass fibre reinforced vinyl ester composites are shown in Figure 6(a) and 6(b) and their properties are tabulated in Table 8.

Table 8 Flexural properties of E glass vinyl Ester composites

Sample	Load N	Flexural Strength N/mm ²	Deflection mm
A1	16700	260	5.5
B1	16200	227	6.3
A2	17220	267	6
B2	16480	236	6.9

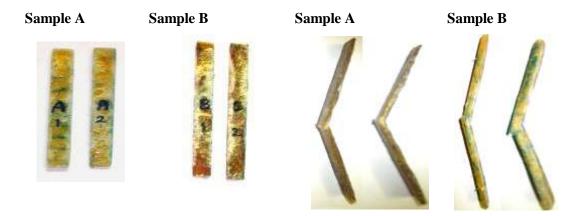
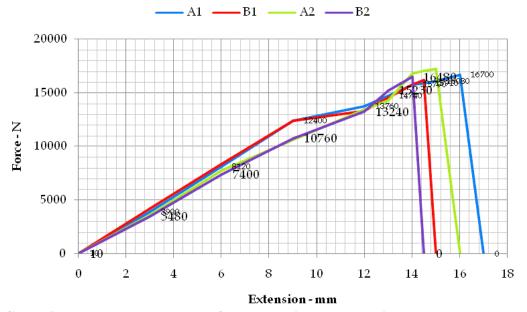


Figure 6 (a) Test sample A and B Before Testing

Figure 6 (b) Sample A and B After Testing

From the experimental characterization of E glass vinyl ester composites sample A2 shows the maximum flexural strength of 267 N/mm², however the Sample A1 showed the flexural strength of 260 N/mm² comparatively. This is due to the influence of composition of the resin mixture and the normal curing for 8hrs. From the investigation of E glass vinyl ester composites has a good flexural elastic property due to the normal curing in room temperature and proper resin mixture [13, 17, 18]. The graphical representation of the samples was shown in Graph 3.



Graph 3 Flexural strength curves for E glass vinyl ester specimens

4.4 Impact strength (Izod and Charpy)

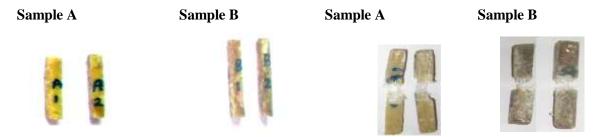


Figure 7(a) samples for Charpy before test Figure 7(b) charpy test samples after test

Thus the notch impact strength of the given specimen was found by Charpy impact test energy absorbed Value for Sample $A = 10.0 \, J$, Sample B = 5.0 J Izod Impact Test.

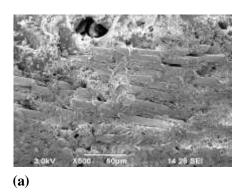


Figure 8(a) Izod test samples before the test Figure 8(b) Izod test samples after the test

Thus the notch impact strength of the given specimen was found by Izod impact test energy absorbed value for Sample A=10.0~J, Sample B=6.0~J. From the notched Charpy impact test and Izod test shown in figure 8(a) and 8(b) conducted on E glass fiber reinforced vinyl ester composites it was found that sample A absorbed more impact energy, when compared to Sample B of E glass fiber reinforced vinyl ester composites shown in figure 7(a) and 7(b). The Sample-A E glass fiber reinforced vinyl ester composite absorbed impact energy of about 10.0 J during rapid loading, whereas Sample-B of E glass fiber absorbed only of about 5.0 J. In Izod test also the same impact energy of 10.0 J was absorbed in Sample-A. The energy absorption capacity of the Sample A is due to the curing time and resin mixture, which make the fiber more ductile and elastic during impact loading [5, 18, 19].

4.5 Microstructure Analysis

The surfaces of the specimens were examined using Scanning Electron Microscopy (SEM). This is done in order to evaluate structural characterization and to examine the morphology. From the analysis of the samples A and B it shows the debonding of resin with E glass and small cracks, pits were also involved. From (figure-10) SEM graph representing that, small pits found in figure 10(a) the Sample A of E glass vinyl ester composites. When compared with normal curing the surface morphology is good in heat treated vinyl ester composites. The quality of the interface between fibre and matrix seems to be good [16,18]. There is no fibre pull outs and delamination of the fibres in this sample A. From (figure-11) SEM graph representing that, small pits and pull outs of the fiber are found due to poor bonging between fiber and matrix. In heat treated sample B there is no impression of fibre pull outs. The Quality of the interface good in heat treated E glass vinyl ester composites [15, 16,19].



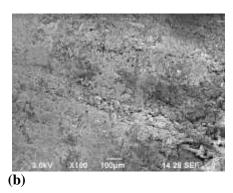
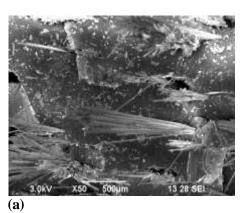


Figure 10 SEM graph Representing (a) Sample A cured at room temperature for 8hours (b) Sample A cured at room temperature and heat treated at 100°C for 3 hours.



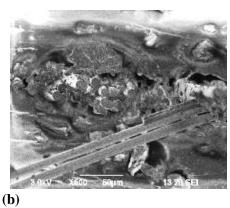


Figure 11 SEM graph Representing (a) Sample B cured at room temperature for 8hours (b) Sample B cured at room temperature and heat treated at 100°C for 3 hours.

4.6 Non-Destructive Testing (radiography):

In radiographic techniques, the internal structure of a moulded part is examined by impinging a beam of radiation on one of its surfaces and recording the intensity of the beam as it emerges from the opposite surface. Conventional radiography uses x-rays (in the range of 7–30 keV). The dimensions of the samples shown in (figure-12) 200 mm x 20mm x 8mm (Length \times width \times thickness) [1,18]. Form Radiography testing shown in (fig 13) it was found that there are many small gas holes acceptable level in Sample a of E glass vinyl ester composites. There is no significant defect found in the sample B of Eglass vinyl ester composites shown in Table 9. Form this result it was concluded that there is no internal cracks and voids are found the E glass vinyl ester composite materials [1,18]



Figure 12 Specimen for Radiography test



Figure 13 Radiography (X-Ray) Report

Table 9 Radiography Test Report

Sl.No	Radiography No.	Sample	Thickness (mm)	Film Size (inch)	Observation	Remarks
1	KC Grade	A	8	10x4	Gas Hole	Acceptable
2	KC Grade	В	8	10x4	No significant Defect	

5. Conclusion

E glass fiber reinforced vinyl ester composites were prepared and mechanically characterized. Two different Percentages of curing agent were added to the vinyl ester resin and the effect on the laminate properties was determined. The microstructure and NDT of E glass were investigated. The conclusions which can be drawn are as follows.

- The incorporation of varying percentages of the curing agent in vinyl ester resin has good effect on curing characteristics in E glass vinyl ester composites.
- The tensile strength and compressive strength for both, the sample B shows the increasing strength of E glass vinyl ester composites. By reducing the curing mixture proportions in the vinyl ester resin and curing in room temperature for 8hours gives increasing tensile and compressive strength.
- The flexural strength increased in sample A of E glass vinyl ester composites. By increasing the curing mixture proportions in the vinyl ester resin and curing at room temperature for 8hours gives increasing flexural strength.
- The impact strength increased in sample A of E glass vinyl ester composites. By increasing the curing mixture proportions in vinyl ester resin and curing at room temperature for 8 hours makes the fibre more ductile and elastic during impact loading. From the results the impact damage is reduced in Sample-A compared to Sample-B.
- The microstructures of the E glass vinyl ester composites consisting of small pit and pull out of fibres in Sample B. When compared with normal curing the surface morphology is good in heat treated vinyl ester composites. The quality of the interface between fibre and matrix seems to be good in Sample A.
- The NDT radiography test was performed and found that there are many small gas holes acceptable level in Sample A of E glass vinyl ester composites. There is no significant defect found in the sample B. There is no internal cracks and voids found in E glass vinyl ester composite materials due to the quality of the interface between fibre and matrix.
- Thus, from the mechanical characterization and microstructure it is concluded that the sample A of E glass fibre vinyl ester composite showed advantages over Sample B. This is because of curing mixture proportions and normal curing at room temperature.

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