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Experimental determination of the mechanical behavior of glass fiber reinforced polypropylene composites

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Abstract

Thermoplastic composites have been widely used in structural and engineering applications, due to their high specific strength and stiffness, high strain to failure, better impact strength, shorter processing cycle time, infinite shelf life, and recyclability. This paper discusses the influence of the forming pressure and coupler concentration on the mechanical behavior of glass fiber reinforced polypropylene composite laminates. The Design of Experiments' (DOE) full factorial approach was adopted for conducting the composite laminate fabrication experiments. The thermoplastic composite laminates were fabricated in a hot compression molding machine, using the film stacking technique. This is an innovative approach to develop thermoplastic composite laminates, using the available low cost raw materials, instead of high end prepreg materials. As per the ASTM standard, the tensile and flexural tests were carried out, in order to evaluate the influence of the parameters on the mechanical behavior of the composite laminates. The Tensile and flexural strengths of the thermoplastic composite laminates were the responses measured to identify the most influencing parameter. The experimental results show that the increase in forming pressure and coupler concentration initially increases both the mechanical properties, and then decreases the properties of the composite laminates. Compared to the coupler concentration, the forming pressure greatly improves both the tensile and flexural properties. Using the Scanning Electron Microscope (SEM), a morphological analysis was carried out to observe the bonding between the matrix and reinforcement.

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Keywords: Thermoplastic composites; Film stacking; Mechanical characterization; Scanning electron microscope; Design of experiments

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1. Introduction

Continuous glass fibre reinforced thermoplastic composites (GFRTP) have been introduced, as structural materials for high performance aerospace and other industrial applications, due to their high strain to failure, high specific strength and specific stiffness, better impact tolerance, short processing cycle time, infinite shelf life of prepreg, and recyclability. Recently there is a growing interest in the use of thermoplastic resins as matrix materials instead of the thermosetting matrix. These composites with good mechanical properties have attracted large-volume markets of automotive industries. Bus bumpers, seat back structures, etc., are some of the automobile components made of glass fibre/polypropylene composites, due to their ease of fabrication, light weight and economy.

The Glass fibre (GF) reinforcement and polypropylene (PP) matrix system is a commonly employed material combination, due to its high availability as well as low procurement costs. Fibre reinforced thermoplastic composites may be processed by conventional methods, such as compression moulding, and offer improvements in mechanical properties over unreinforced ones. The main difficulty in the processing of GFRTP composites is the high melt viscosity and poor wettability of fibers with thermoplastics. Hence, the use of polypropylene grafted maleic anhydride (PP-g-MAH) as an interfacial coupler is highly recommended and has been vastly reported in the literatures.

Etcheverry and Barbosa [1] investigated the enhancement of the mechanical properties of glass fiber reinforced polypropylene, by adhesion improvement, and revealed that the strength and toughness increase three times and the interfacial strength duplicates in PP/GF composites prepared with in-situ polymerized fibers. Rijsdijk et al. [2] investigated the influence of MAH-modified PP (m-PP) on the mechanical properties of continuous GF/PP composites, and showed an increase in both the longitudinal and transverse flexural strength up to 10 wt% m-PP. The ultimate tensile strength, hardness and modulus values of the modified PP composites were higher, compared to the values of Carbon Fibre reinforced PP composites [3]. The results of Lopes and Sousa [4] indicated that the intercalation effect and mechanical properties, especially the modulus, tensile strength and impact strength, were enhanced by increasing the content of MA, using maleated PP with higher graft efficiency. The effect of temperature [5] and stitching parameters [6] on the mechanical properties of glass/polypropylene woven composites have been investigated. Many researchers [7-11] studied the mechanical behavior of the glass fibre reinforced thermoplastic composites.

López et al. [12] studied the preparation and mechanical properties of PP/PP-g-MA/Org-MMT nanocomposites with different MA contents. Reis et al. [13] studied the flexural behavior of hybrid laminated composites and suggested that compared with the full glass fibre reinforced PP laminates, the hybrid composites have economical, ecological and recycling advantages. Varatharajan et al. [14] presented a comparison of the mechanical properties of glass fibre reinforced thermoplastic and thermoset composites, and suggested that thermoplastic composites exhibit higher order impact resistance.

The main parameters which affect the consolidation quality of the composites are forming pressure, forming temperature, holding time and coupler concentration. The quality of consolidation affects the mechanical properties of composite laminates, and is measured by the interfacial bonding strength and the absence of the void content. In this work, to improve the quality of consolidation of composite laminates, the forming pressure and coupler concentration are taken into consideration by keeping the other two parameters as constant. The purpose of this experimental study is to investigate the influence of the forming pressure and coupler concentration on the tensile and flexural behavior of GFRTP composites, and to obtain the best parameter values being defined by the maximum mechanical properties enhancement achieved. The Design of experiments' (DOE) full factorial approach was adopted for conducting the composite laminate fabrication experiments. The composite laminates were prepared in a compression molding machine using the film stacking technique, by varying the coupler concentration from 5wt% to 10wt% and by varying the forming pressure from 4MPa to 9MPa. PP-g-MAH was used as a coupler for the better adhesion of the GF and PP matrix. This is an innovative approach to develop the thermoplastic composite laminates, using the available low cost raw materials, instead of high end prepreg materials. Very few works have been reported in this area, and it leads to the further study of these materials. To access qualitatively the interfacial bonding between the fibre and the matrix, the SEM micro-structural analysis was carried out on the composite specimens.

2. Experimental details of the GFRTP composite laminates

2.1. Design of Experiments

The experiments were conducted based on the full factorial design approach of the Design of experiments. The full factorials examine every possible combination of factors at the levels tested. Thus, in the full factorial design, the full information about the main as well as the interaction effects was obtained. The control factors such as the forming pressure and coupler concentration were identified as important variables that may influence the mechanical behavior of the thermoplastic composite laminates by keeping the forming temperature and holding time as constant values. The factors and their levels selected are presented in Table 1.

Table 1. Selection of factors and levels

Factors	Levels		
	I	II	III
Forming Pressure (MPa)	4	7	9
Coupler concentration (wt %)	5	8	10

The total number of experimental runs for the full factorial design is indicated in Table 2.

Table 2. Total experimental runs

Exp. Runs	Forming pressure (MPa)	Coupler concentration (wt %)
1	4	5
2	4	8
3	4	10
4	7	5
5	7	8
6	7	10
7	9	5
8	9	8
9	9	10

2.2. Fabrication of thermoplastic composite laminates

An isotatic polypropylene homopolymer with a 0.6mm thick film (ρ = 0.94 g/cm3) was used as the polymer matrix, and an E-glass fiber (ρ = 2.55 g/cm3) woven fabric (610 gsm) was used as the fibrous reinforcement. In this work, the commercial grade PP-g-MAH pellets (Mw = 9.1 by GPC, 8-10 wt. % MAH) were used as the coupler. The properties of the raw materials are given in Table 3. Differential Scanning Calorimetry (DSC) was used to characterize the polypropylene with regard to the thermal properties. Based on the DSC test result shown in Fig. 1, the forming temperature for the fabrication of the thermoplastic composite laminates was set as 190°, which is well above the melting temperature of the polymer matrix.

Table 3. Properties of raw materials

Properties	Tensile strength (MPa)	Density (g/cm3)	Tensile modulus (GPa)	Yield strength (MPa)
Polypropylene	39.5	0.94	2	36.5
Glass fibre	1750	2.55	70	---

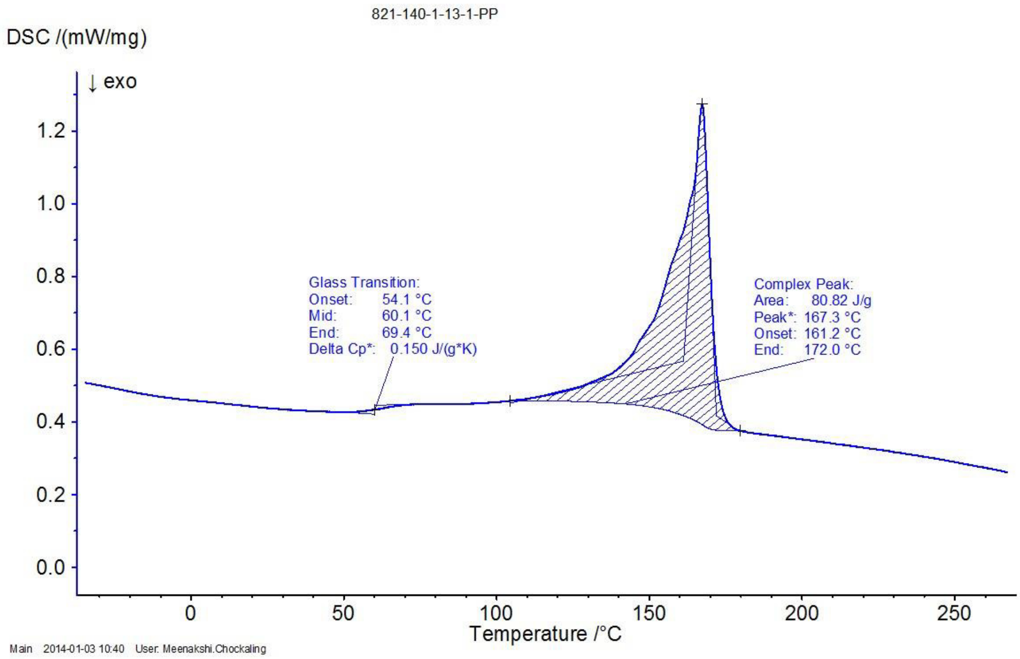


Fig.1. Differential scanning calorimetry of Polypropylene film

Fig. 2. GFRTP Composite Laminate

The thermoplastic composite laminates' fabrication experimental runs were carried out according to DOE's full factorial approach, as listed in Table 2. The GFRTP composite laminates were prepared based on the film stacking technique, using a hot compression molding machine. The PP film and GF woven fabric were stacked one over the other for 3.5 mm thickness. The stacking sequence has four layers of GF woven fabric and five layers of PP film. To improve the interfacial bonding between the PP film and GF woven fabric, PP-g-MAH pellets were distributed evenly between them in all the layers with 5wt%, 8wt% and 10wt% concentrations respectively. The stacked alternate layers were placed in between platens of 100 Ton hydraulic press, and electrically heated to a temperature of 190°C.

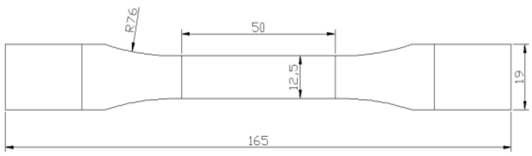
During heating, the molten matrix may stick to the contact surface of the heated platens. In order to avoid this, it is advisable to sandwich them between Aluminium (Al) sheets which will enhance the quality of the surface finish of the final thermoplastic composites. The releasing agent was applied to the Al sheets to avoid the sticking. Afterwards, a forming pressure of 4 MPa was applied to the stacked layers for 10 min, by maintaining the forming temperature at 190°C. Then, the material was allowed to cool in the mold to room temperature. The same procedure was repeated for the forming pressures of 7 MPa and 9 MPa to get the next sets of GFRTP composite laminates. The GFRTP composite laminate (Fig. 2) with a fibre weight fraction of 50% was prepared with 250×250x3.5 mm size.

2.3. Mechanical Characterization

After the fabrication of GFRTP composite laminates, test samples were cut to the required sizes, as prescribed in the ASTM standards, for tensile and flexural tests with a diamond cutter. The mechanical properties of the composites were tested by using a 5 Ton universal testing machine. During the tests, the load was measured by means of an electronic load cell, and the displacement was measured by a linear variable differential transducer. The experiments were performed with a crosshead speed of 1.5 mm/min.

2.3.1. Tensile test

The standard followed for the tensile test is ASTM D 638. The Fig. 3 shows the dog bone shape tensile specimen. The tensile testing was carried out at room temperature with the test speed of 1.5 mm/min, to determine the tensile behavior of the composite specimens.



All dimensions are in mm

Fig. 3. Tensile specimen

2.3.2. Flexural test

The standard followed for the flexural (three-point bending) test is ASTM D-790 (80 x 12.5 x 3.5 mm), and the test was conducted at room temperature to characterize the consolidation quality. Equation (1) is used to calculate the Flexural strength of the composite laminate from the peak load of the specimen.

Table 4 gives the tensile and flexural strength values of the designed experimental layout.

Table 4. Experimental tensile and flexural strength

Exp. Runs	Forming Pressure (MPa)	Coupler Concentration (wt%)	Tensile Strength (MPa)	Flexural Strength (MPa)
1	4	5	94	42
2	4	8	109	56
3	4	10	104	51
4	7	5	123	76
5	7	8	128	91
6	7	10	126	69
7	9	5	108	51
8	9	8	113	64
9	9	10	97	51

3. Results and discussion

3.1. DOE analysis of GFRTP composites

In this work, a commercially available software package (MINITAB 16) was used for the computation work. The Tensile and Flexural strengths of the GFRTP composite specimens were analyzed, to study the effects of the parameters. The main effects at all levels, on the tensile and flexural strengths, are calculated and listed in Table 5. Based on the tensile and flexural experimental results, the optimal level setting for the better mechanical behaviour is obtained at $(\text{Forming pressure})_2(\text{Coupler concentration})_2$; i.e., Forming pressure is set at 7MPa and Coupler concentration is set at 8wt%.

The influence of the forming pressure and coupler concentration on the tensile and flexural strengths of the GFRTP composite is shown in Figs. 4 and 5 respectively. It is observed that an increase in the forming pressure and coupler concentration initially increases both the mechanical properties, and then decreases the properties of the composite laminates. Compared to the coupler concentration, the forming pressure greatly improves both the tensile and flexural strengths.

Table 5 Response Table for Tensile and Flexural Strength

Levels	Tensile Strength		Flexural Strength	
	Forming Pressure (MPa)	Coupler Concentration (wt%)	Forming Pressure (MPa)	Coupler Concentration (wt %)
1	102.33	108.33	49.67	56.33
2	**125.67**	**116.67**	**78.67**	**70.33**
3	106.00	109.00	55.33	57.00
Delta	23.34	8.34	29	14
Rank	1	2	1	2

The optimal setting is $(\text{Forming pressure})_2(\text{Coupler concentration})_2$ based on Tensile strength and Flexural strength

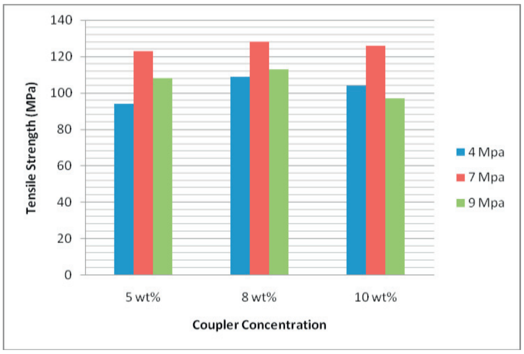


Fig. 4. Tensile strength of the GFRTP Composites

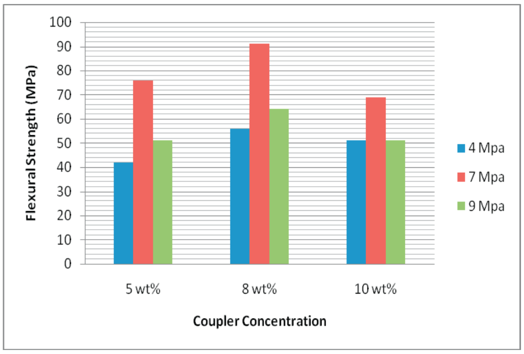


Fig. 5. Flexural strength of the GFRTP Composites

From the tensile study, it is revealed that an increase in the forming pressure improves the tensile strength by 23.34%, while an increase in the coupler concentration improves it by 8.34%.

From the flexural study, it is revealed that an increase in the forming pressure improves the flexural strength by 29%, while an increase in the coupler concentration improves it by 14%. Here, the property improvement is most dramatic in flexural behavior. This improvement in both properties indicates the improved interfacial bonding between the GF and PP, and also the reduced void content in the composite laminate.

3.2. Analysis of Variance (ANOVA)

The purpose of ANOVA is to find the significant factor statistically. It gives a clear picture as to how far the process parameter affects the response and the level of significance of the factor considered. The ANOVA values for both the tensile and flexural strengths are calculated, and listed in Tables 6 and 7 respectively. The quantity R^2, Coefficient of determination, is used to judge the adequacy of the developed model. The R^2 is 88.85% and 95.13% for the present investigation of tensile and flexural strengths, which shows the high correlation that exists between the experimental and predicted values.

Table 6. ANOVA Table for tensile strength

Source	DoF	Seq SS	Adj SS	Adj MS	F	% Contribution
Forming Pressure	2	944.67	944.67	472.33	14.03	0.016
Coupler Concentration	2	128.67	128.67	64.33	1.91	0.262
Error	4	134.67	134.67	33.67		
Total	8	1208.00				

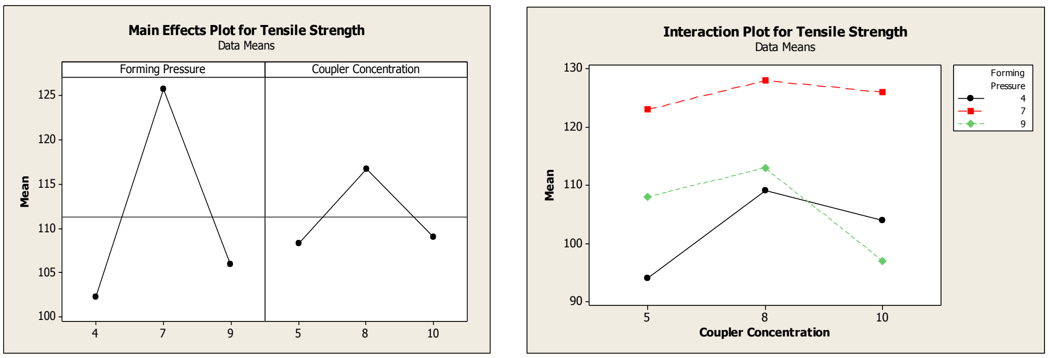
S = 5.80230 R-Sq = 88.85% R-Sq(adj) = 77.70%

Table 7. ANOVA table for flexural strength

Source	DoF	Seq SS	Adj SS	Adj MS	F	% Contribution
Forming Pressure	2	1417.56	1417.56	708.78	30.89	0.004
Coupler Concentration	2	374.22	374.22	187.11	8.15	0.039
Error	4	91.78	91.78	22.94		
Total	8	1883.56				

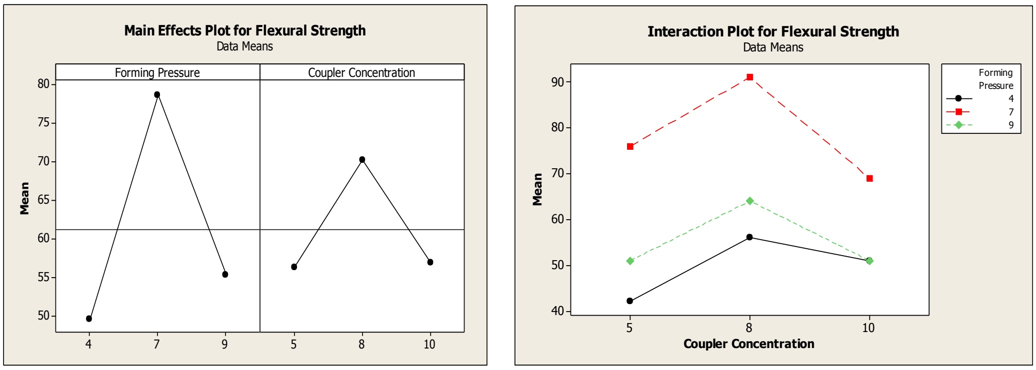
S = 4.79004 R-Sq = 95.13% R-Sq(adj) = 90.25%

The main effects and interaction for the tensile and flexural strengths are plotted in Figs. 6 and 7 respectively. In the Figs. 6 and 7, the curvature of the forming pressure and coupler concentration shows a peak in the middle, indicating that the optimum is located inside the selected region. It reveals that the tensile strength and flexural strength initially increase and then decrease with increasing forming pressure and coupler concentration.



(a) (b)

Fig 6 (a-b). Main effects and interaction plots for Tensile strength



(a) (b)

Fig 7 (a-b). Main effects and interaction plots for Flexural strength

The ‘F’ test is carried out to study the significance of the process parameter. The high ‘F’ value indicates that the factor is highly significant in affecting the response of the process. In this investigation, the forming pressure is a highly significant factor, and plays a major role in affecting the tensile and flexural strength of the GFRTP composite laminates. However, coupler concentration has little significant effect on the responses.

The residuals are examined, using the normal probability plots of the residuals and the plots of the residuals versus the predicted response which are shown in Figs. 8 and 9 for tensile and flexural strengths respectively. They revealed that the residuals generally fall in a straight line implying that the errors are distributed normally.

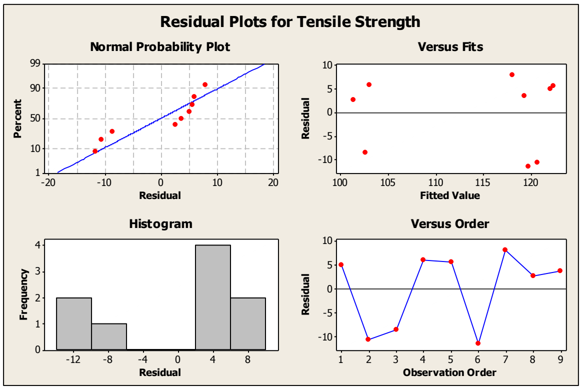


Fig. 8. Residual plots for Tensile strength

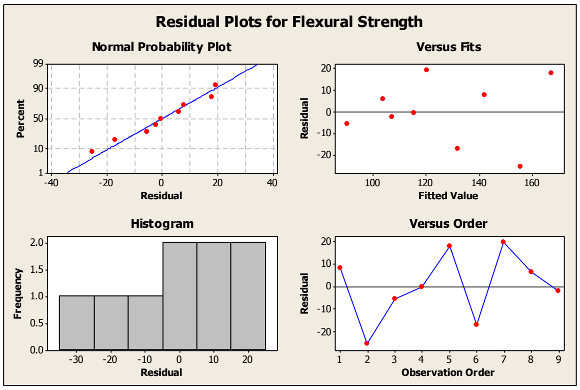
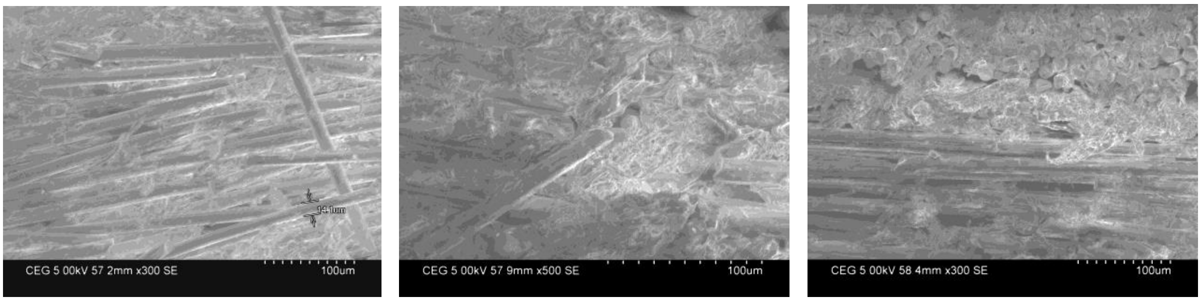


Fig. 9. Residual plots for Flexural strength

One of the most important objectives of experiments related to fabrication is to achieve the desired mechanical properties of the optimal parameters. Here, the goal is to maximize the mechanical properties. The optimum process parameters are found to be a forming pressure of 7 MPa and a coupler concentration of 8 wt%. The optimized tensile and flexural strength values are 128 MPa and 91 MPa.

3.3. Microstructural analysis using Scanning Electron Microscope

An interfacial property known as fiber–matrix interaction was observed, using the scanning electron microscope. The GFRTP composite specimens prepared with 7MPa forming pressure are considered for the micro-structural analysis, because of their higher mechanical properties’ achievement. Fig.10 (a) shows the SEM micrograph of a 7MPa-5wt% GFRTP composite laminate, and it shows a comparatively low polymer wetting and interfacial adhesion of the GF surface with PP matrix. In contrast, the SEM micrograph of the 7MPa-8wt% GFRTP composite laminate (Fig.10b) shows improved wetting of the glass fibers, and therewith good bonding to the matrix in the GF/PP system. This leads to a good load transmission between the fiber and the matrix, which in turn, leads to higher mechanical properties. Fig.10 (c) shows the SEM micrograph of the 7MPa-10wt% GFRTP composite laminate. It shows the presence of some voids, and comparatively low polymer wetting in the GF/PP system. Simply increasing the amount of coupler concentration will not result in further increase in fibre/matrix bond strength. Because, at higher (10wt %) weight fractions of PP-g-MAH, a decrease in tensile and flexural strengths was observed. Hence, the addition of PP-g-MAH seems to cause deterioration in the matrix and/or interphase properties and consequently in the mechanical properties of the composite laminates.



(a) (b) (c)

Fig. 10. SEM micrographs of (a) 7MPa-5wt% GFRTP laminate (b) 7MPa-8wt% GFRTP laminate (c) 7MPa-10wt% GFRTP laminate

4. Conclusion

In this investigation, the influence of the forming pressure and coupler concentration on the mechanical behavior of glass fiber reinforced polypropylene composite laminates was studied. The following conclusions are derived from this study:

- It is observed that an increase in the forming pressure and coupler concentration initially increases both the mechanical properties, and then decreases the properties of the composite laminates.
- Compared to the coupler concentration, the forming pressure greatly improves both the tensile and flexural strengths.
- From the tensile study, it is revealed that an increase in the forming pressure improves the tensile strength by 23.34%, while an increase in the coupler concentration improves it by 8.34%.
- From the flexural study, it is revealed that an increase in the forming pressure improves the flexural strength by 29%, while an increase in the coupler concentration improves it by 14%.
- The property improvement is most dramatic in flexural behavior.
- This improvement in both the properties indicates the improved interfacial bonding between the GF and the PP, and also the reduced void content in the composite laminate.
- It is observed from the experimental study that the fabricated thermoplastic composite laminates have shown better mechanical properties than the laminates of a similar nature discussed in various literatures.
- The optimum process parameters are found to be a forming pressure of 7 MPa and a coupler concentration of 8 wt%. The optimized tensile and flexural strength values are 128 MPa and 91 MPa.
- The SEM micrograph of the 7MPa-8wt% GFRTP composite laminate shows improved interfacial bonding between the GF and PP, which in turn, leads to a better mechanical properties' improvement in the composite laminate.

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