# A STUDY OF RESIN TRANSFER MOLDING PROCESS USING EXPERIMENTAL DESIGN METHOD

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**SUMMARY** : Statistical design of experiments was used to investigate the effect of resin transfer molding (RTM) process variables on mechanical properties of the products. Two variables, the injection pressure and mold temperature, were investigated at this stage of research. The experiment was conducted following the $3^2$ design. Since the RTM parts were molded using two batches of resin, the results were treated as a two-block problem. The short beam shear strength and flexural strength were measured to evaluate the mechanical properties. Among the two variables, the injection pressure is found to have a significant effect on both properties.

**KEYWORDS**: Resin transfer molding, composite manufacturing, statistical design of experiment, mechanical properties, molding temperature, pressure.

## INTRODUCTION

Resin transfer molding (RTM) with textile preform is considered as one of the most efficient processes for manufacturing composite components. In RTM, a dry preform of fiber reinforcements is placed in a cavity in a mold, the mold is closed, resin is introduced under pressure into the preform and cured to form a composite part. Even though RTM process is relatively straight forward and consists a few simple steps, the quality of the final products has been found to be dependent on a number of variables [1-4] such as injection pressure, vacuum assistance, temperature of the mold, fiber and resin, curing temperature, fiber architecture, volume fraction, resin viscosity, etc.. To optimize processes involving many variables, statistical design and analysis of experiment is a suitable approach.

Statistical design and analysis of experiment is increasingly used in composite manufacturing processes [5-7]. As known, this approach is particular useful in identifying the key factors among many apparent variables and in understanding their effects and inherent interactions in the process. Furthermore, this approach can lead to processing models used in process optimization.

In this work, statistical design of experiments was used to investigate the effect of resin transfer molding (RTM) process variables on mechanical properties of the products. The experiment was conducted in two stages. The first stage was a screen procedure which identified the variables having a significant effect on the results. These variables were then investigated using full factorial experiment design at the second stage. This paper presents the results of the second stage experiment.

## EXPERIMENTAL DETAILS

A Multiflow RTM machine by Liquid Control was used for resin injection. The machine is pneumatically controlled under an air pressure of 90 psi. The actual injection pressure can be regulated as desired but a minimum of 55 psi is required to drive the injection system. The mold used in this study is of rectangular shape and made of aluminum. The mold consists of three parts: the top plate, base plate and specimen window. Figure 1 shows the schematic of the mold. The mold has a cavity of 9”x5”x0.157”. The locations of the inlet and outlet ports are also shown.

A polyester resin Derakane-411-350 by Dow Chemical was used in RTM process. A non-crimp type 0/90 glass by J.B.Martin (St-Jean-Sur-Richelieu, Quebec) was used for reinforcement. Each composite laminate contains seven layers of fabric, which yields an average fiber weight fraction of 0.63 and a fiber volume fraction of 0.43.

After the fiber preform was placed in the mold, the mold was sealed and immersed in a water bath maintained at the desired temperature with an accuracy of ±1°C. A minimum of 30 minutes was allowed for mold temperature to reach equilibrium before injection. Vacuum was applied prior to injection to remove air and released as soon as resin was flowing out the outlet port. The mold was then transferred into an air oven for post cure at 90°C for four hours.

Two mechanical properties, the interlaminar shear strength (ILSS) and the flexural strength, were measured according to ASTM standards. It should be noted that the ILSS measured by short beam shear of fabric composites gives good indication of quality of the laminates such as fiber/resin wetting and porosity level but may not be used as the ILSS value as to unidirectional composites. For each plate, ILSS was measured with ten specimens and the flexural strength with five specimens. The mean value and the standard deviation for each plate are presented in Table 1.

## EXPERIMENTAL DESIGN

Our earlier study [8] using screen experiment investigated ten RTM process variables including injection pressure, hydrocheck pressure, mold temperature, post cure temperature, valve position after filling, stroke length, vacuum level, type and number of layers of reinforcement and resin to catalyst ratio. The composite properties examined were the short beam shear strength, flexural strength, glass transition temperature, damping ratio and the flexural storage modulus. It was found that the injection pressure, mold temperature, post cure temperature and the type of reinforcement had significant effect on most of these properties. Since no significant interaction was found between the former two and latter two variables, only the injection pressure and mold temperature were selected for further study.

A full factorial experiment was conducted with two variables, each at three levels, i.e. following the $3^2$ design. This yields a total of 9 treatment combinations of process variables and their levels, as shown in the first two columns in Table 1. The three levels are denoted as –1,0 and 1.

Three composite plates were manufactured under each treatment, giving a total of 27 composite plates. Composite plates were actually manufactured and tested over two time periods and the experiment can be considered as a two-block problem. At first, 9 plates corresponding to 9 treatments were manufactured using a newly received resin with a viscosity of 650 MPa-s measured at 25°C. Three months later, 18 more plates, 2 replicates for each treatment, were manufactured using the same resin. The resin viscosity, however, had increased to 859 MPa-s. In Table 1, block 1 corresponds to the plates made of lower viscosity resin whereas block 2 to the plates made of higher viscosity resin.

## RESULTS AND DISCUSSIONS

When only two or three variables are considered, the effect of variables and their interactions can be easily seen from the plots of marginal means. Figure 2a. presents the mean shear strength versus pressure at three temperature levels. Figure 2b is for the flexural strength.

In Figure 2, the first striking phenomenon is the effect of block itself. In the plots for block 1, the lines are much apart and across each other, suggesting strong temperature effect and interaction between pressure and temperature. In the plots for block 2, however, the lines are relatively close and tend to lie parallel to each other, indicating a smaller temperature effect and a weak interaction between temperature and pressure. Also, there is a clear trend of reduced strength with increasing pressure.

It has been observed that higher resin viscosity reduces void contents [1,2] particularly when fiber volume fraction is not very high [1]. In our study, however, the variation in viscosity between the two blocks is not very large and, therefore, we suspect that there might be other reasons responsible for the discrepancy between the results of two blocks. Firstly, block 1 has no replication whereas block 2 has one replication and therefore the latter yields a more consistent result. Secondly, block 2 exhibited an overall greater strength than that of block 1, which might also be attributed to the improved skill of the experimentalist because tests for block 2 were conducted after the tests for block 1 had been completed.

Another issue revealed by Figure 2 is the importance of a proper range for a variable. If the range of pressure were selected as from 70 to 80 psi, we would have concluded that pressure has a small effect on strength and therefore missed direction for optimization.

Since the results of block 1 were not reliable, further analysis was carried out only on the results of block 2. The response surface of a mechanical property to process variables was obtained through regression analysis. For $3^2$ design, the full regression model contains 9 terms:

$$Y = b_0 + b_1 T + b_2 T^2 + b_3 P + b_4 P^2 + b_5 TP + b_6 TP^2 + b_7 T^2 P + b_8 T^2 P^2 \tag{1}$$

Where T is the normalized temperature and P is the normalized pressure. The real values are related to the normalized ones as

$$T = (\overline{T} - 40)/15, P = (\overline{P} - 70)/10 \tag{2}$$

The response surfaces corresponding to the 9-term regression model are shown in Figure 3. The regression coefficients for the two mechanical properties are listed in Table 2.

The significance of each term in Eq.1 may be examined through analysis of variance according to the ANOVA table. Tables 3 and 4 are the standard ANOVA tables for ILSS and flexural strength, respectively. A variable is considered significant when its F-value, calculated by dividing the mean square value of the variable to that of error, is greater than the statistical F-value, $F_{cr}$. For the considered case, $F_{cr}$ =5.12 corresponds to 95% confidence interval. For ILSS, only the linear pressure term P is significant at 95% confidence interval and a second order interaction term $TP^2$ is significant at 75% confidence interval. The regression model therefore can be simplified as

$$Y1 = 43.62 - 4.48P - 2.83TP^2 \tag{3}$$

For the flexural strength, both the linear and quadratic pressure terms are found to be significant and regression model yields

$$Y2 = 402.04 - 21.77P + 61.99P^2 \tag{4}$$

Statistical analysis shows that, between the two variables, the injection pressure has a greater influence on mechanical properties. In the range of 60-80 psi, a lower pressure yields higher strengths. Within the temperature range under study, the effect of mold temperature exhibits only in a second order interaction term on ILSS. No significant effect has been found on the flexural strength.

The reduced mechanical properties at higher injection pressure may be attributed to fast mold filling and fiber wash. The mold filling time in the present study varied from about 20 second to a minute. It was observed that with increasing injection pressure, the mold filling time reduced whereas fabric distortion increased. A short mold filling time can lead to insufficient wetting and therefore a poor adhesion between fiber and resin and even void. This certainly will affect the ILSS value. On the other hand, the fabric distortion would have a greater impact on the flexural strength. The weak effect of pre-molding temperature observed might also be the result of fast filling such that the resin temperature was hardly affected during RTM. On the other hand, the resin state (block number) has a very strong impact on mechanical properties. The resin with a higher viscosity (block 2) yields a higher strength and more consistent results. Apart from a slower mold filling and hence a longer wetting time resulted by a higher viscosity, the improved skill of experimentalist in RTM process might also contribute to the improvement in quality of the product.

The effect of injection pressure on the ILSS and flexural strength observed in this study in principle agrees with the finding by Hayward and Harris [1]. They reported that varying injection pressure between 50 to 400 kPa (7 psi to 58 psi) resulted in little variation in ILSS and a slight decrease in flexural strength. Their investigation, however, was conducted using non-washing reinforcement such as satin weave fabrics and the injection pressure was much lower than the range of the present study. The lack of effect of mold temperature on ILSS and flexural strength observed in this study is also in line with the result of Hayward and Harris. These authors reported that the mold temperature in the range between 20 to 40°C had no effect on ILSS and flexural strength for samples later subjected to a post cure at 45°C.

## CONCLUSIONS

Among the two variables investigated, the injection pressure is found to have a significant influence on the ILSS and flexural strength. A lower pressure generally yields a higher strength. Within the temperature range under study, the mold temperature has a weak effect on ILSS through a second order interaction term but has insignificant effect on the flexural strength. This work also demonstrates that the effect of experiment block, which corresponds to resin batch and sequence in RTM molding in this study, may also have an impact on the results and hence should be taken into consideration in the analysis.

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Table 1 RTM Processing Variable Setting and Mechanical Properties of the Composites

| Temperature | | Pressure | | Interlaminar Shear Strength (MPa) | | | | Flexural Strength (MPa) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| (°C) | Level | (psi) | Level | Block 1 Mean | (S.D.) | Block 2 Mean | (S.D.) | Block 1 Mean | (S.D.) | Block 2 Mean | (S.D.) |
| 25 | -1 | 80 | 1 | 23.1 | (1.7) | 43.4 | (3.3) | 275.0 | (18.3) | 459.6 | (25.9) |
| | | | | | | 38.2 | (5.7) | | | 401.8 | (77.1) |
| 25 | -1 | 70 | 0 | 32.2 | (8.1) | 36.1 | (5.5) | 403.4 | (57.9) | 326.0 | (83.3) |
| | | | | | | 41.7 | (3.7) | | | 493.9 | (44.1) |
| 25 | -1 | 60 | -1 | 31.4 | (3.2) | 42.6 | (3.8) | 382.0 | (40.0) | 532.6 | (22.0) |
| | | | | | | 47.8 | (1.9) | | | 529.3 | (25.3) |
| 40 | 0 | 80 | 1 | 37.2 | (4.8) | 34.6 | (3.8) | 303.7 | (76.9) | 369.0 | (49.9) |
| | | | | | | 40.8 | (4.5) | | | 515.5 | (66.3) |
| 40 | 0 | 70 | 0 | 27.0 | (3.9) | 44.3 | (2.5) | 301.3 | (62.2) | 437.7 | (65.5) |
| | | | | | | 43.0 | (4.3) | | | 366.3 | (69.7) |
| 40 | 0 | 60 | -1 | 26.0 | (4.9) | 45.3 | (2.8) | 268.3 | (56.4) | 436.0 | (76.1) |
| | | | | | | 48.0 | (1.4) | | | 535.6 | (22.5) |
| 55 | 1 | 80 | 1 | 32.9 | (4.6) | 39.3 | (2.0) | 298.3 | (68.0) | 447.9 | (16.6) |
| | | | | | | 43.6 | (2.7) | | | 419.0 | (151.1) |
| 55 | 1 | 70 | 0 | 37.2 | (1.6) | 40.8 | (3.1) | 371.5 | (81.3) | 362.4 | (28.0) |
| | | | | | | 48.5 | (1.3) | | | 522.2 | (37.0) |
| 55 | 1 | 60 | -1 | 39.9 | (1.5) | 43.4 | (2.2) | 523.8 | (64.7) | 570.3 | (41.5) |
| | | | | | | 45.9 | (2.7) | | | 564.9 | (70.2) |

Table 2 Regression Coefficients In Equation 1

| | $B_0$ | $B_1$ | $B_2$ | $B_3$ | $B_4$ | $B_5$ | $B_6$ | $B_7$ | $B_8$ |
|---|---|---|---|---|---|---|---|---|---|
| ILSS | 43.62 | 2.86 | -1.84 | -4.48 | -1.45 | 0.29 | -2.83 | 2.60 | 2.68 |
| Flexural Strength | 402.0 | 16.2 | 24.1 | -21.8 | 62.0 | -8.5 | -6.3 | -36.8 | 2.6 |

Table 3 ANOVA Table For Interlaminar Shear Strength (ILSS)

| Factor | Sum of Square (SS) | Degree of Freedom | Mean Square (MS) | F-value | p-value |
|---|---|---|---|---|---|
| Temperature (L) | 11.4465 | 1 | 11.44653 | .952271 | .354640 |
| Temperature (Q) | .0107 | 1 | .01068 | .000888 | .976873 |
| Pressure (L) | 90.8050 | 1 | 90.80501 | 7.554336 | .022531 |
| Pressure (Q) | .4378 | 1 | .43780 | .036422 | .852882 |
| T(L) by P(L) | .6844 | 1 | .68445 | .056941 | .816742 |
| T(L) by P(Q) | 21.2817 | 1 | 21.28167 | 1.770485 | .216048 |
| T(Q) by P(L) | 17.9920 | 1 | 17.99202 | 1.496809 | .252228 |
| T(Q) by P(Q) | 6.3606 | 1 | 6.36056 | .529153 | .485455 |
| Error | 108.1823 | 9 | 12.02025 | | |
| Total SS | 257.2010 | 17 | | | |

*L= linear term and Q= quadratic term

Table 4 ANOVA Table For Flexural Strength

| Factor | Sum of Square (SS) | Degree of Freedom | Mean Square (MS) | F-value | p-value |
|---|---|---|---|---|---|
| Temperature (L) | 1715.06 | 1 | 1715.06 | .326832 | .581525 |
| Temperature (Q) | 2660.50 | 1 | 2660.50 | .506998 | .494484 |
| Pressure (L) | 25740.95 | 1 | 25740.95 | 4.905333 | .054021 |
| Pressure (Q) | 16230.76 | 1 | 16230.76 | 3.093020 | .112493 |
| T(L) by P(L) | 573.76 | 1 | 573.76 | .109338 | .748467 |
| T(L) by P(Q) | 107.23 | 1 | 107.23 | .020434 | .889480 |
| T(Q) by P(L) | 3615.48 | 1 | 3615.48 | .688985 | .427978 |
| T(Q) by P(Q) | 5.83 | 1 | 5.83 | .001111 | .974135 |
| Error | 47227.90 | 9 | 5247.54 | | |
| Total SS | 97877.47 | 17 | | | |

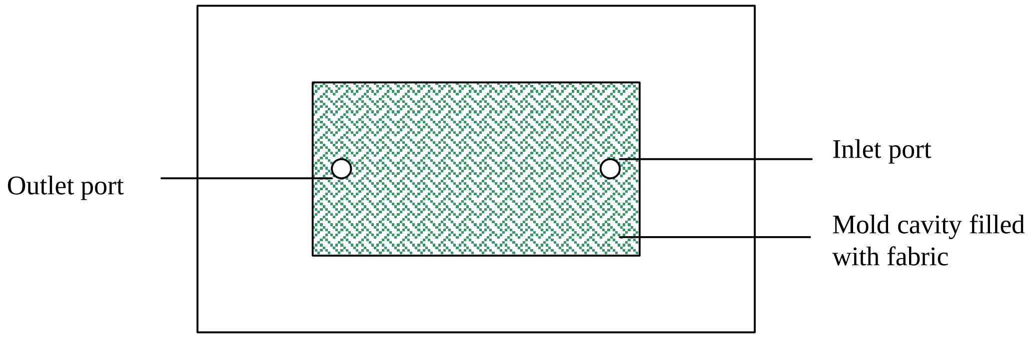


Fig.1 The schematic of the mold. The mold is of rectangular shape with a cavity of 9”x5”x0.157”.

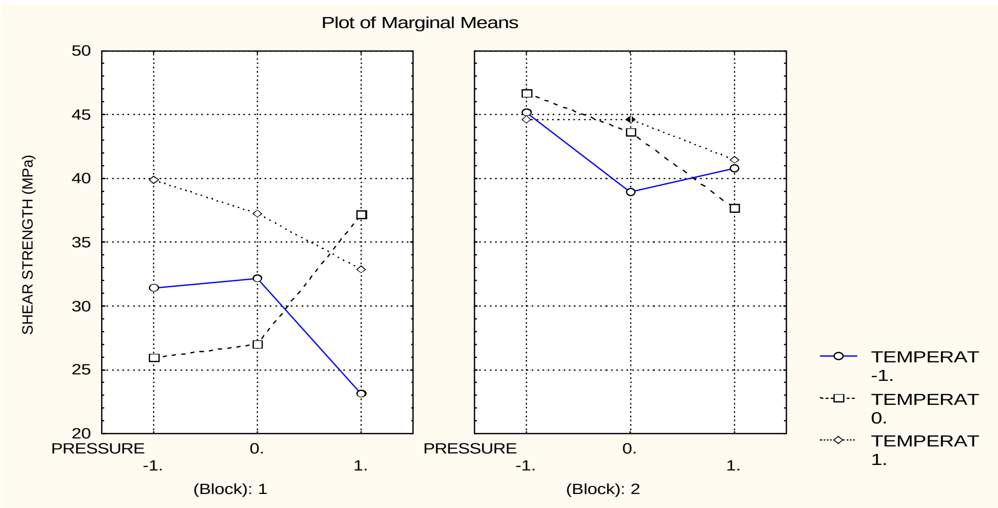


Fig.2a The marginal mean plot for the interlaminar shear strength.

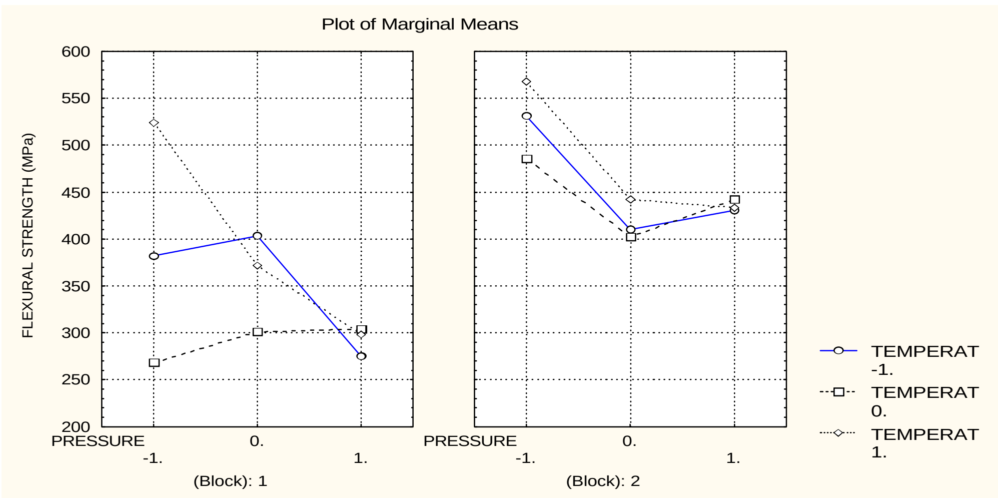


Fig.2b The marginal mean plot for the flexural strength.

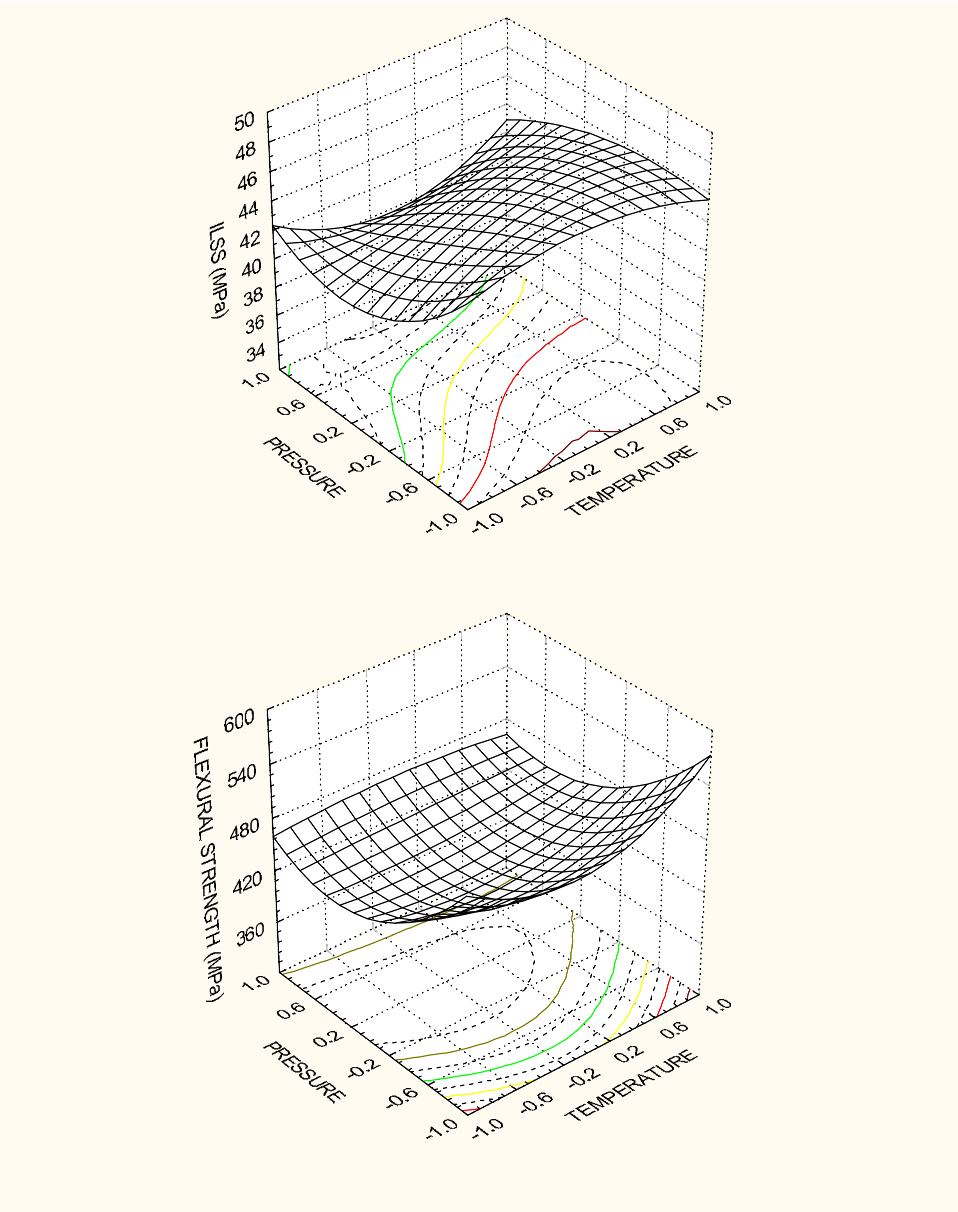


Fig.3 Response surfaces corresponding to the 9-term regression model.
Upper: the interlaminar shear strength; Lower: the flexural strength.