

Curing studies of unsaturated polyester resin used in FRP products

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Fiber reinforced plastics (FRP) products being strong and light in weight, find extensive applications in automobiles, marine, structural and household components. Unsaturated polyester (UP) resin is a principle binder material used in FRP products. Curing of UP resin is a key for successful FRP processing. Gel time (t_{gel}) and peak exothermic temperature (θ_{peak}) are critical measures of curing of resin. t_{gel} indicates commencement of polymer cross-linking (setting) while θ_{peak} controls shrinkage and cracking of polymer. This paper aims to predict the t_{gel} and θ_{peak} during cure of UP resin. The weight percent of methyl ethyl ketone peroxide (MEKP) as catalyst, cobalt octoate (C_{oct}) as an accelerator, calcium carbonate ($CaCO_3$) as filler and glass fibers as reinforcements are used as process variables. The variations in t_{gel} and θ_{peak} are approximated by regression equations. Strong interactions are found between the process variables. The process optimization is made to achieve a desired combination of t_{gel} and θ_{peak} within the regime of experimentation. Confirmatory experiments are performed to validate the predicted results.

Keywords: Curing, FRP, Gel time, Peak exothermic temperature, Process optimization, Unsaturated polyester

Unsaturated polyesters (UP) are the macromolecules with a polyester backbone and belong to category of the thermo-set resin. These are step-growth products of saturated acids (such as phthalic or isophthalic acid) and unsaturated acids (such as maleic or fumaric acid), condensed with a dihydric alcohols. The UP resins can be easily handled in processes like hand lay up, filament winding, resin transfer moulding in the liquid form. They can also be used in moulding compounds. Moreover, they possess good mechanical and service properties; have excellent thermal stability and weather resistance. Hence, these resins are used in a number of applications like insulation coatings, fiber reinforced plastics (FRP) products, sandwich panels, sheet moulding compounds (SMC), bulk moulding compounds (BMC), pultrusion components etc.

The curing of UP resin occurs as a combination of chemical kinetics and control by diffusion. The curing reaction between the styrene monomer and UP involves a free radical chain growth polymerization. During the curing process at room temperature, generally the organic peroxides (like methyl ethyl ketone peroxide (MEKP)) are mostly used as catalysts (initiators) while cobalt salt is frequently used as an accelerator. The inhibitors such as quinones and

phenol are added to react with the initiating or propagating radicals to reduce their reactivity. This gives an induction period before the resin is cured and adds to its storage life. The success of producing a FRP product by processes like hand lay up; resin transfer moulding or resin spraying depends on the cure kinetics as well as the gel time. The interaction of chemical kinetics with physical phenomena like gelling makes the curing process more complex. Several studies deal with curing of resins in FRP products.

A substantial decrease in curing time and hardness were noted when concentration of cobalt octoate (C_{oct}) and MEKP were increased up to 0.02 wt% and 2 wt% respectively¹. The role of α -methyl styrene and Cu salts as inhibitors was emphasized to keep a low peak exotherm of UP resin curing process to prevent distortion of polymer². It was concluded that for given filler content, the gel time reduces with increase in temperature³.

A shift in the peak exo-therm was observed from 170°C to 100°C for UP resins when MEKP (0-1.6%) and C_{oct} (0- 0.2%) were introduced⁴. The gel time was found to be increased from 10 min to 300 min when 4-tert butyl catechol was changed from 0 to 0.10%, while it reduced from 54 min to 16 min when C_{oct} was varied from 0.05 to 0.8%⁵. A reduction in curing temperature (from 100°C to 30°C) was noticed when a redox system comprising of 1.5% cumene

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hydroperoxide (CHP) as initiator and 0.3% cobalt naphthenate (CONap) as promoter were added in the base resin⁶. In case of P- toluenesulfonic acid, the peak exotherm as well as heat release rate were found to be less than that of other catalysts, viz., lead dioxide (PbO₂) and zink acetate dihydrate ((CH₃COO)₂ Zn)⁷. To facilitate uniform gel time throughout the part by varying the mixing ratio of resin and catalyst, an on-line mixing method was suggested in which curing agent mixed with resin when the later entered the mould⁸. It was claimed that the gel time for the fabricated part was reduced by 20-25%.

To characterize the cure kinetics and resin viscosity for liquid composite moulding, a semi-empirical model was developed⁹. Fourier transform infrared (FTIR) spectroscopy, DSC, rheometry and ultrasonic measurements were suggested to monitor curing of resins and their results have been correlated¹⁰. For curing of UP/ styrene, it was revealed that with presence of 5 wt% of nanoclay, the tensile modulus and the fracture toughness (K_{IC}) were also improved by 30%¹¹. A styrene free curing of UP resin was claimed, wherein the UP-alkyd chains are intrinsically cured into a cross linked matrix in presence of peroxides¹². The rheological and mechanical effects of polyester based modifiers (with or without isocyanate groups) on the curing rate of epoxy/triethyl tetramine (TETA) system were investigated recently¹³. It was found that gel time was reduced in case of functionalized polyesters, indicating an accelerating effect on curing process. Significant improvements in mechanical properties like tensile modulus and abrasion resistance were found for low-cost polymeric composites like untreated sugarcane fiber and polyester¹⁴. Several basic studies on the curing of UP resin can be found in handbooks related to FRP processing¹⁵⁻¹⁷ highlighting the effect of inhibitors, initiators, accelerators, fillers and reinforcements on free radical polymerization.

A critical review of existing literature on curing behavior of liquid resins reveals that several moulding problems like incomplete mould filling, cracking of FRP mouldings, porous mouldings and longer cycle times can be attributed to the complex interaction between the ingredients. Several studies have considered the effects of process variables on mechanical, tribological and rheological characteristics of FRP product. A few studies can be attributed to curing kinetics and viscosity prediction. However, a number of experimental studies have

neglected the interaction effects among process variables to investigate their effect on process or product characteristics. To the best of our knowledge, most of the curing studies were carried out by one factor at a time (OFAT) approach and the statistical techniques like design of experiments (DOE) were rarely applied to such problems. It can also be seen that there are limited studies on parametric optimization for getting a desired response (e.g. optimal product quality characteristics). DOE facilitates systematic, efficient, cost effective planning and analysis of the experimental studies¹⁸⁻²⁰. Numerous investigations in other fields have efficiently used such studies to get optimal blend of product or process characteristics²¹⁻²³. Thus, experimental investigations based on simultaneous variations of compositions (of process variables) rather than an OFAT approach are needed to get more insight into curing of the resin. In this study, an attempt is made to develop mathematical model for UP resin curing process based on DOE technique. The wt% of catalyst (MEKP), accelerator (C_{oct}), filler (CaCO₃), and glass fibers were chosen as process variables and their effects on gel time (t_{gel}) and peak exothermic temperature (θ_{peak}) were investigated. An optimization problem was then formulated such that both these responses should fall within a given range for a better product quality and productivity.

Experimentation

Plan of experiments

The process variables and their levels selected for this study are shown in Table 1. The levels are expressed as wt% of the base resin, i.e., for 100 g of UP. The levels chosen are in agreement with existing literature¹⁻⁴ and also common in composites processing methods. As this study involved four inputs at three levels each (coded as -1, 0 and +1) and two outputs, a conventional full factorial scheme of experimental design would have resulted into 64(=4³) experiments. Since conducting large number of experiments (e.g. 64) consumes more time as well as resources, experimental design with face centered,

Table 1—Factors and their levels

Factor Name	Designation	Lowest level(-1)	Middle level(0)	Highest level(+1)
Catalyst	X ₁	1.8	2.5	3.2
Accelerator	X ₂	1.8	2.5	3.2
CaCO ₃	X ₃	0	10	20
Glass Fibers	X ₄	0	15	30

'central composite design' (CCD) was chosen for planning and analysis of experiments. This scheme is a type of response surface methodology (RSM) and for the above combination of process variables it resulted in a design of 31 experimental runs^{18,19}.

Experimental procedure

The resin used in this work was a commercial general purpose UP based on orthophthalic anhydride, maleic anhydride and propylene glycol formulated by Mechemco Industries, India under the trade name 'Mechster 9000.' The resin was found to have an average molecular weight of 1500, a styrene content of $35 \pm 2\%$ and inhibitor as 150-200 ppm hydroquinone. The resin was used as received, i.e., without removal of inhibitors. The curing system consisted MEKP, a 6% solution in dimethyl phthalate and C_{oct} , a 6% solution in styrene. Calcium carbonate ($CaCO_3$) was used in the form of the filler material while glass fibers (in the form of chopped strands) of 25 mm length and 1 mm diameter were used as reinforcements. The curing experiments were carried out at room temperature ($28 \pm 1^\circ C$) with an appropriate quantity (based on DOE) of MEKP, C_{oct} , $CaCO_3$ and glass fibers. A mild steel (MS) mould having dimensions of 60 mm $\times$ 40 mm $\times$ 68 mm was used to carry out the curing process.

The gel time of the reaction mass was found using a specially constructed softness indicator²⁴. This device had an oscillating steel plunger with a tapered end that touched the resin surface. Its other end rested against a dial gauge indicator to record the movement of the plunger (penetration depth). At gel time, the reaction mass began to turn into a stable, hard, non-workable mass which could be seen as reduction in the penetration depth. Figure 1 shows the gel time indicator fabricated for these experiments. The

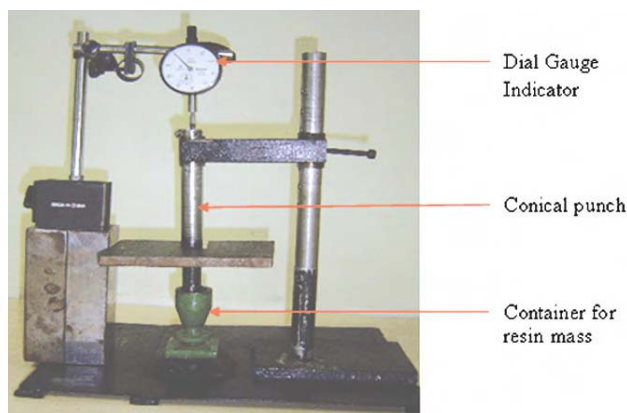


Fig. 1—Softness indicator to measure t_{gel}

exothermic temperature during curing process was monitored with a J-type of thermocouple on a continuous time scale. The peak exothermic temperature was recorded as the maximum temperature reached by reaction mass. Figure 2 shows a typical time-temperature curve during this process and the locations of t_{gel} and θ_{peak} . Table 2 shows the plan of experiments, the values of t_{gel} and θ_{peak} obtained for different parametric combinations.

Analysis of experiments

The investigations of experimental results were done using analysis of variance (ANOVA) at 5% level of significance. MINITAB 13.0²⁵ was used to analyze the results. Table 3 shows the ANOVA for t_{gel} showing contribution of different factors with the estimated regression coefficients. It (a ' p ' value less than 0.05) shows strong interactions between catalyst/accelerator (X_1X_2), catalyst/ $CaCO_3$ (X_1X_3) accelerator/ $CaCO_3$ (X_2X_3) and accelerator/glass fibers (X_2X_4). It can also be seen that these parameters/interactions have linear effect on the t_{gel} while the non-linear (curvature) effects are statistically insignificant.

The results of ANOVA, carried out for the θ_{peak} , are shown in Table 4. Here also, it can be seen that the linear effects of factors as well as interactions are statistically significant. It also shows that accelerator alone is not contributing to θ_{peak} but its interaction with the catalyst results in a decrease of the same. Strong interactions can be observed between catalyst/glass fibers (X_1X_4) as well as accelerator/glass fibers (X_2X_4).

Predictive modeling of resin curing process

In this step, the regression equations¹⁸ were developed wherein the process variables were used as predictor variables to correlate them with the t_{gel} and

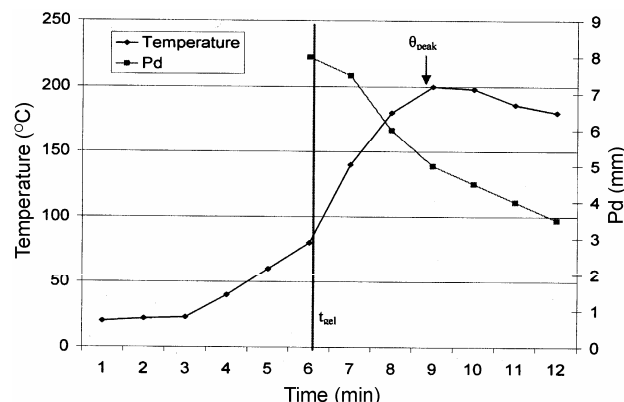


Fig. 2—Time-Temperature Plot of UP Resin Curing Process

Table 2—Experimental runs and corresponding outputs

Run order	X ₁	X ₂	X ₃	X ₄	t _{gel} (s)	θ _{peak} (°C)
1	0	0	1	0	538.6	164.25
2	1	0	0	0	332.0	183.25
3	1	1	-1	-1	211.3	210.70
4	0	0	0	0	454.7	176.19
5	-1	1	1	1	546.5	149.13
6	0	0	-1	0	370.8	188.13
7	-1	1	-1	1	431.9	172.75
8	-1	-1	-1	1	557.9	162.15
9	0	0	0	0	451.0	178.00
10	0	0	0	0	454.7	176.19
11	-1	1	-1	-1	355.5	188.25
12	0	-1	0	0	535.5	175.88
13	0	0	0	0	454.7	176.19
14	1	1	1	-1	239.1	187.13
15	0	0	0	0	453.0	179.00
16	-1	-1	-1	-1	541.9	201.13
17	1	-1	-1	1	231.3	177.13
18	-1	-1	1	1	865.7	138.01
19	0	0	0	1	477.8	159.13
20	-1	0	0	0	577.4	169.13
21	1	1	-1	1	354.5	172.25
22	1	-1	1	1	452.3	143.75
23	1	1	1	1	382.3	157.80
24	0	0	0	-1	431.6	193.25
25	-1	1	1	-1	470.1	173.87
26	0	1	0	0	373.9	176.50
27	1	-1	1	-1	503.1	196.49
28	-1	-1	1	-1	849.7	167.75
29	0	0	0	0	458.0	178.00
30	1	-1	-1	-1	282.1	220.63
31	0	0	0	0	454.0	176.60

θ_{peak}. The significant terms (based on ANOVA) were only accounted in the equations. The gel time in terms of coded units can be expressed as:

$$t_{gel} = 454.5 - 123X_1 - 80.8X_2 + 83.9X_3 + 23.1X_4 + 45.6X_1X_2 - 21.7X_1X_3 - 8.3X_2X_3 + 31.8X_2X_4 \quad \dots (1)$$

Similarly, the equation for peak temperature can be written as:

$$\theta_{peak} = 176.79 + 7.05X_1 - 11.94X_3 + 17.06X_4 - 1.57X_1X_2 - 3.44X_1X_4 + 2.44X_2X_3 + 3.56X_2X_4 \quad \dots (2)$$

The experimenter can predict the response of any combination of process variables with these equations within the regime of experimentation (i.e. -1 to +1).

Results and Discussion

The free radical polymerization mechanism of UP resin with styrene as a cross linking monomer is as

Table 3—ANOVA for t_{gel}

Term	Coef	SE Coef	T	P
Constant	454.5	4.969	91.455	0 *
X ₁	-123	3.948	-31.077	0*
X ₂	-80.8	3.948	-20.465	0*
X ₃	83.9	3.948	21.25	0*
X ₄	23.1	3.948	5.851	0*
X ₁ * X ₁	0.1	10.398	0.006	0.995
X ₂ * X ₂	0.1	10.398	0.006	0.995
X ₃ * X ₃	0.1	10.398	0.006	0.995
X ₄ * X ₄	0.1	10.398	0.006	0.995
X ₁ * X ₂	45.6	4.188	10.889	0*
X ₁ * X ₃	-21.7	4.188	-5.182	0*
X ₁ * X ₄	-0.0	4.188	0	1
X ₂ * X ₃	-48.3	4.188	-11.534	0*
X ₂ * X ₄	31.8	4.188	7.594	0*
X ₃ * X ₄	0.0	4.188	0	1

* Significant factor or interaction

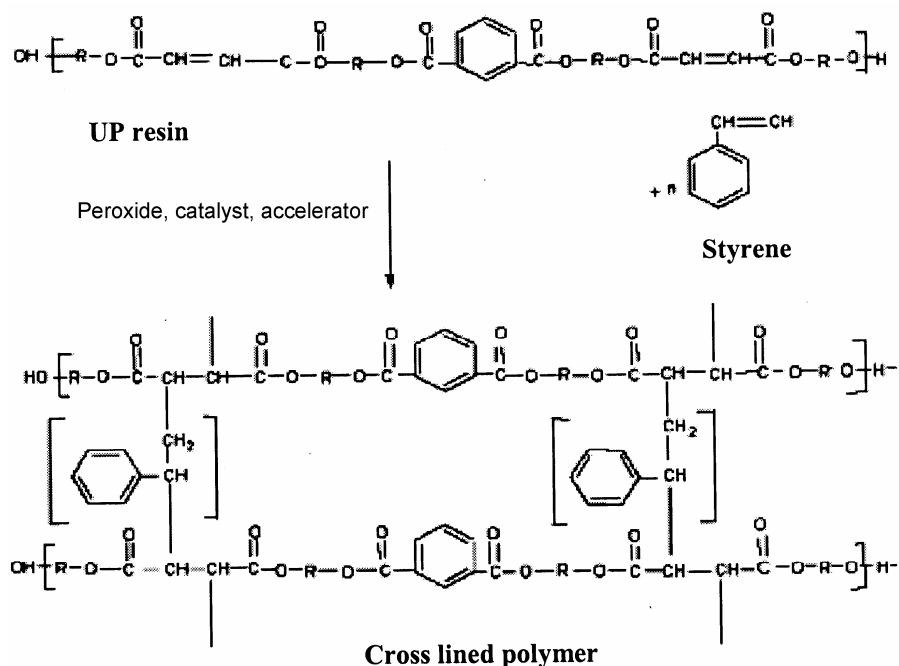
Table 4—ANOVA for θ_{peak}

Term	Coef	SE Coef	T	P
Constant	176.79	0.724	244.179	0*
X ₁	7.05	0.5753	12.261	0*
X ₂	0.3	0.5753	0.527	0.605
X ₃	-11.94	0.5753	-20.757	0*
X ₄	-17.06	0.5753	-29.657	0*
X ₁ * X ₁	-0.17	1.5151	-0.11	0.914
X ₂ * X ₂	-0.17	1.5151	-0.11	0.914
X ₃ * X ₃	-0.17	1.5151	-0.11	0.914
X ₄ * X ₄	-0.17	1.5151	-0.11	0.914
X ₁ * X ₂	-1.57	0.6102	-2.569	0.021*
X ₁ * X ₃	0	0.6102	-0.002	0.998
X ₁ * X ₄	-3.44	0.6102	-5.64	0*
X ₂ * X ₃	2.44	0.6102	3.997	0.001*
X ₂ * X ₄	3.56	0.6102	5.832	0*
X ₃ * X ₄	-0.01	0.6102	-0.012	0.99

* Significant factor or interaction

shown in Fig. 3. It shows that UP resin has reactive double carbon-carbon covalent bonds which can be cross-linked to form thermosetting material. It is done with the vinyl group molecules like styrene in presence of catalyst (like MEKP) and accelerator (like C_{oct}). This result in cross linking between the polymer chains and result the formation of a three dimensional, rigid and hard polymer. Figure 4 (a-d) shows the linear plots of t_{gel} based on Eq. (1). Based on the cross-linking mechanism (as shown in Fig. 3), following observations were recorded:

Figure 4a is a response surface graph representing an interaction between catalyst and accelerator with a

Fig. 3—Cross linking mechanism of UP resin¹⁸

decreasing trend in t_{gel} . This value was approximately 703.8 s at the lowest levels (-1,-1) of catalyst and accelerator, which reduced to 296.8 s at their highest levels (+1, +1). It can be observed that for a given level of accelerator concentration; catalyst results in a significant decrease in the t_{gel} . This is primarily because of an increase in free radicals generated due to the decomposition of catalyst by the accelerator. These free radicals initiate the exothermic copolymerization reaction. In the course of radical copolymerization, the heat evolved results in temperature rise. Consequently, the heat accumulation increases the reaction rate and polymer molecular weight rises steadily throughout the reaction. The increase of the viscosity results from the formation of macromolecules which grow until the formation of a macro network leading to an infinite viscosity. This makes the reaction mass as semi-viscid. From the plot, we can also say that for a given (fixed) level of catalyst, t_{gel} is not sensitive to accelerator. This is obvious as a fixed level of catalyst limits the generation of free radicals.

Figure 4b reveals an interaction effect between catalyst and CaCO_3 . Here the highest value of 683s was recorded for the lowest level of catalyst and highest level of CaCO_3 (-1, +1). The lowest value was observed as 269.8 s for the highest level of catalysts and lowest level of CaCO_3 (+1,-1). The increase in gel time owing to the presence of CaCO_3 can be

attributed to the retardation of the cross linking of the resin. The microgels formed cannot come close because they are impended by the presence of fillers. In this interaction, it is interesting to note that the presence of catalyst results in decrease of t_{gel} while that of CaCO_3 results in the increase of the same.

Figure 4c explains a similar trend for accelerator and CaCO_3 . Here the highest t_{gel} was found as 667.7 s for the lowest accelerator and highest level of CaCO_3 (-1, +1). Its lowest value was observed as 338.3 s for highest value of catalyst and lowest value of CaCO_3 (+1,-1).

Figure 4d depicts an increase in t_{gel} when the quantity of glass fibers increases particularly at higher levels of accelerator. The highest value of 544.2 s was observed at (-1,-1) levels of accelerator and glass fibers respectively while the lowest value of 319 s was found at (+1,-1) levels of the same. The delay in gel time due to presence of glass fibers can be attributed to absorption of heat generated in the exothermic reaction.

With regard to the peak exothermic temperature, following observations were made from Fig. 5 (a-d):

Figure 5a indicates the maximum value of θ_{peak} as 184.5°C at the highest level of catalyst and lowest level of accelerator (+1,-1). Its lowest value was observed as 167.26°C at the lowest levels of both catalyst and accelerator (-1,-1). This also can be attributed to the formation of increased free radicals at

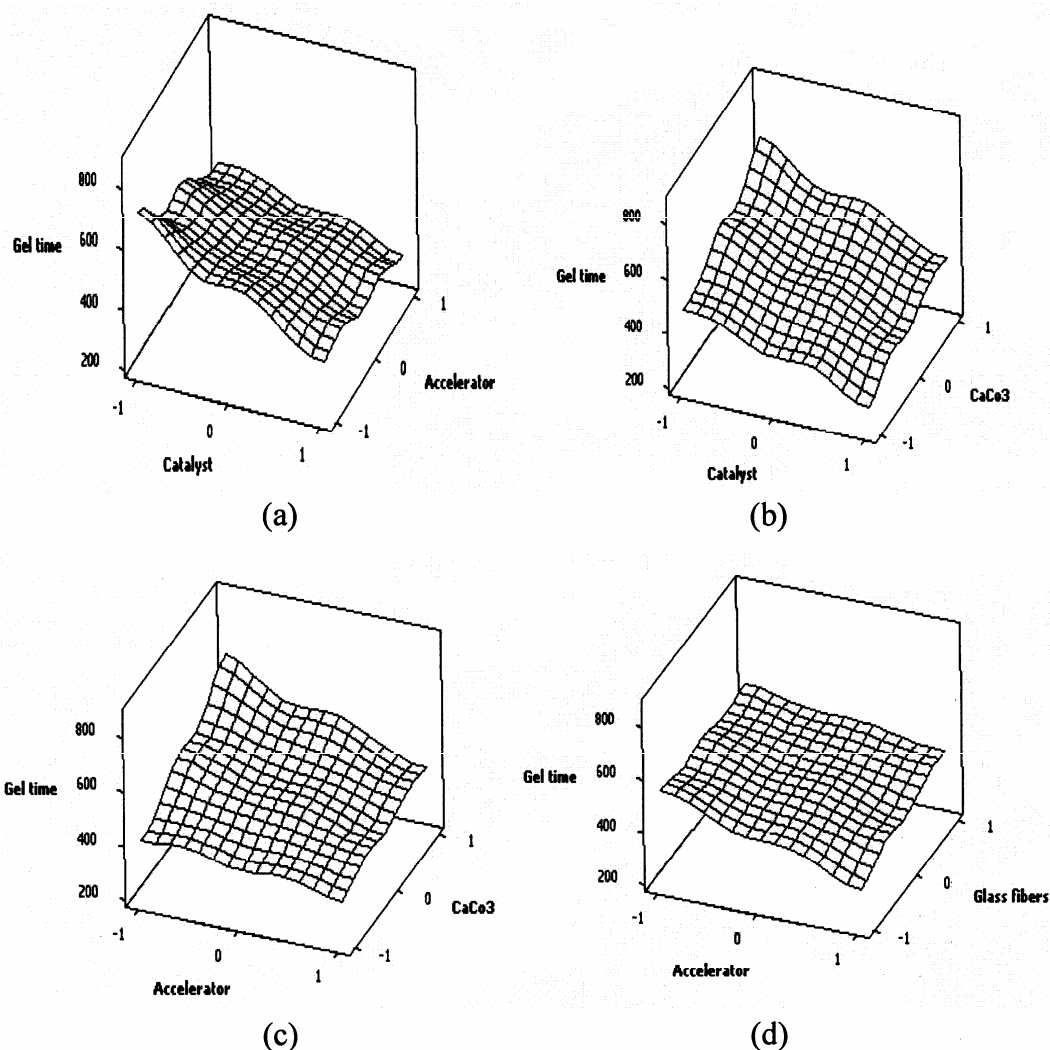


Fig. 4—Variation of gel time with process variables.

higher levels of catalyst. It can be noted that, the increase in accelerator level does not contribute significantly towards θ_{peak} at higher levels of catalyst.

Figure 5b demonstrates the interaction effect of catalyst and glass fibers on θ_{peak} . The temperature of the reaction mass increases when rate of heat generation, due to copolymerization is larger than that of heat dissipation. As the decomposition of free radicals (from catalyst) contributes towards an increase in heat accumulation, the presence of glass fibers absorbs the heat in reaction mass. This can be seen as decrease in temperature. Thus, the maximum peak temperature was recorded as 203.73°C at the highest settings of catalyst and the lowest settings of glass fibers (+1,-1). The minimum value of the same could be seen as 155.51°C at the lowest level of catalyst and the highest level of glass fibers (-1, +1).

Figure 5c depicts addition of CaCO_3 results in minimizing θ_{peak} , a typical property of heat absorbing material. It has been seen that θ_{peak} has been reduced from a maximum value of 190.26°C to 161.5°C when CaCO_3 was changed from lowest level (-1) to the highest one (+1). Further, in this interaction, the accelerator contributes towards slight increase in temperature at higher level of CaCO_3 .

Figure 5d, too, indicates a similar trend as above. Here also, an increase in glass fiber quantity resulted in reduction in θ_{peak} from 196.5°C to 155.26°C. The accelerator effect is inconsequential at higher level of glass fibers.

For all of the above observations, the responses at central points (0, 0) were in plane of the response surface. This means that, in this case, the quadratic (curvature) effects are insignificant and hence can be

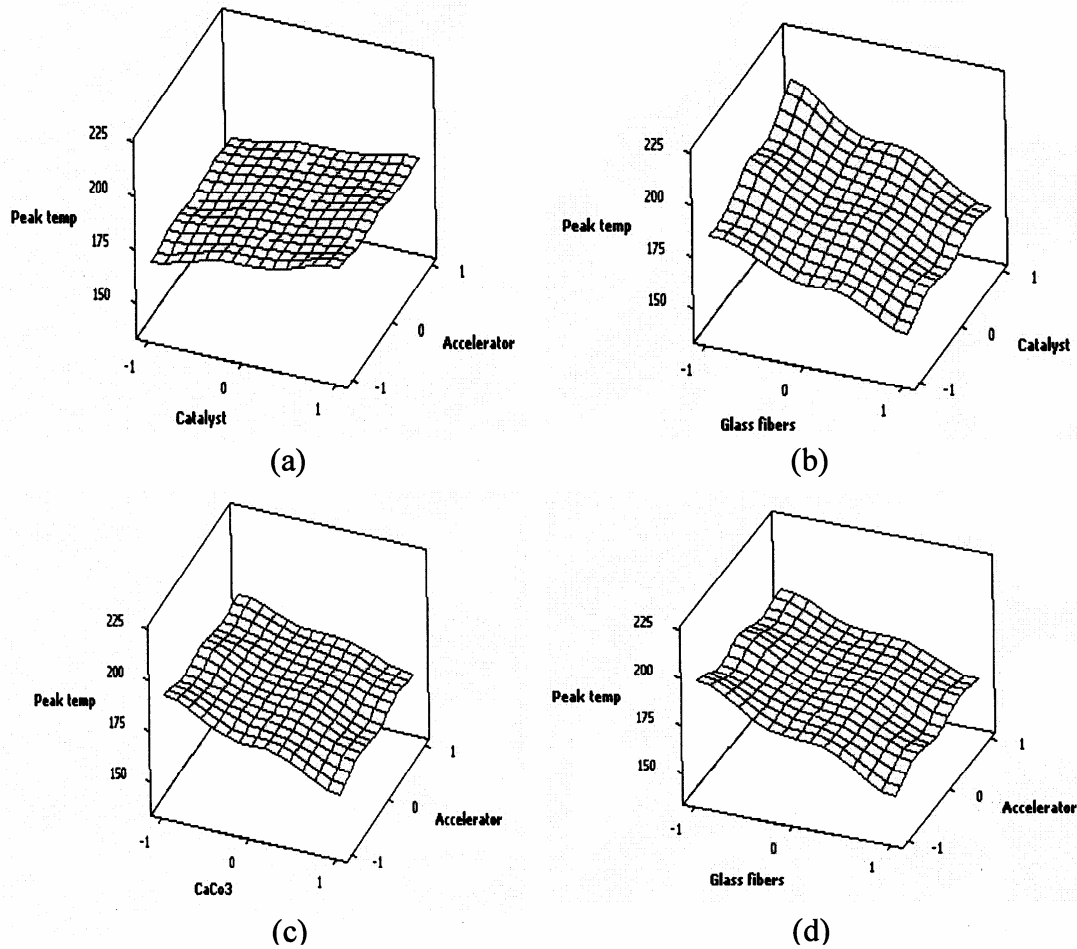


Fig. 5—Variation of peak temperature with process variables

neglected. It can be highlighted here that considering one separate factor at a time for such complex interactions may lead to a 'pseudo-convergent' prediction, as in an interaction, relationship between the response variable and a factor is influenced by level of some other factor²⁰.

Process optimization

Based on the linear regression equations for the t_{gel} as well as θ_{peak} , a response optimization procedure was carried out to find a setting of process variables for desired combination of the above responses. In context of present study, lower t_{gel} can result in defects like incomplete mould filling as at t_{gel} mobility is arrested in polymer while its higher value can increase the cycle time and reduce the productivity. Similarly, a lower θ_{peak} can cause a slower reaction rate and hence longer curing times while the higher values may result in distortion of the part due to shrinkage and cracking². Thus the values of t_{gel} and

θ_{peak} , neither too less nor too high are desirable. To achieve this, a parametric optimization with objective as 'target is the best' was performed. From the experimental findings, target values to be achieved were considered as 600 s (10 min) for t_{gel} and 170°C for θ_{peak} .

For linear regression models, the 'D' optimality is commonly used criterion¹⁸. It is done by minimizing the variance in the regression coefficients of the fitted model. An overlaid contour plot showing the feasible region corresponding to the target values of t_{gel} and θ_{peak} is shown in Fig. 6. It is formed by intersections of lower and upper bound values of the two responses. In present case, these were selected as 580 s and 620 s for t_{gel} and 168°C and 175°C for θ_{peak} . The experimenter can decide a proper combination of input variables to achieve a desired response value from such feasible region. Figure 7 shows a MINITAB response optimisation plot. It shows how different experimental settings can influence the

predicted responses. It also evaluates how well a combination of input variables satisfies the target. This is based on a composite desirability index (D) that evaluates how the settings optimize a set of responses overall. It has a range of zero to one. In present case, the ' D ' value is very close to 1 indicating a proper convergence to optimal solution.

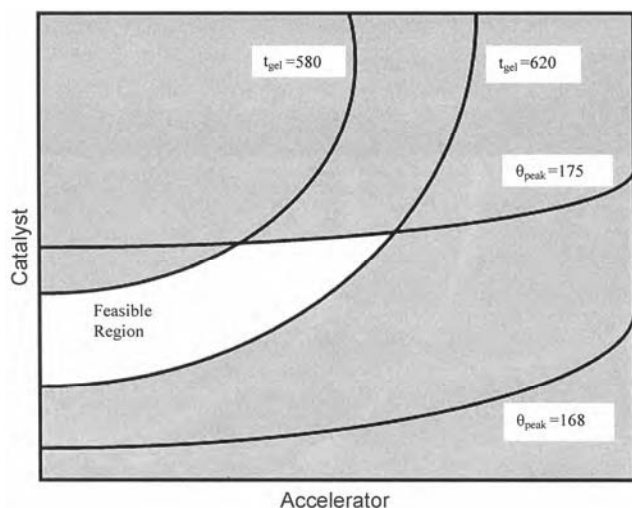


Fig. 6—Overlaid contour plot showing a feasible region of optimization.

The plot also summarizes the trend of the response when factors are changed from -1 to +1. From Fig. 7, the values of process variables X_1 , X_2 , X_3 and X_4 were respectively found in coded units as -1, -0.7573, -0.5113 and 0.3097. In actual units these were recognized as 1.8 % of MEKP, 1.97% of C_{oct} , 4.89 % of $CaCO_3$ and 19.65% of glass fibers.

Confirmatory experiments

To verify the above predictions, confirmatory experiments were performed. This was done with the new set of process variables as obtained above. Four replications were carried out for these parametric permutations. The results of confirmatory experiments are shown in Table 5. It could be seen that values of t_{gel} and θ_{peak} were in close agreement with the predictions of the optimized values. Slight variations in the response values (that are insignificant at 5% level of significance) in the neighborhood of the target values could be attributed to the elimination of insignificant terms in regression equation as well as uncontrollable noise factors (like fluctuation in room temperature, humidity etc.) during experimentation. To minimize the effect of noise factors, robust design studies like Taguchi's experimental design can be undertaken.

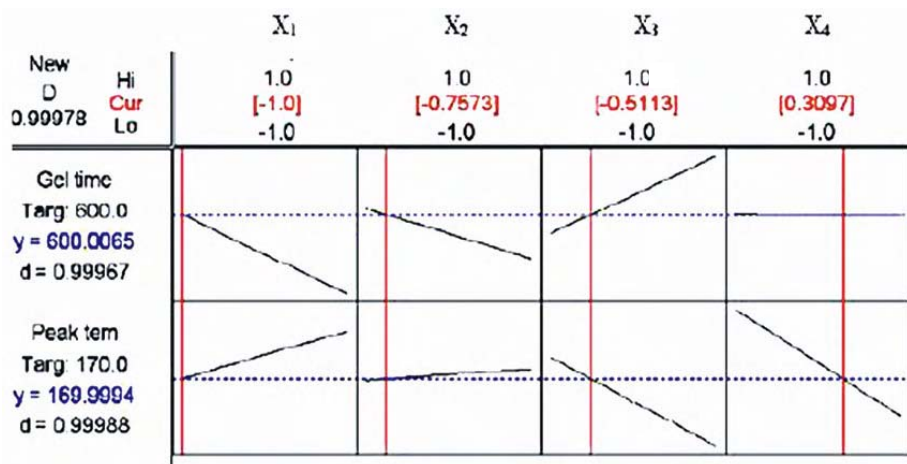


Fig. 7—Results of process optimisation.

Table 5—Confirmatory experiments

Run No.	X_1	X_2	X_3	X_4	t_{gel} (s)			θ_{peak} (°C)		
					Predicted	Experimental	Avg.	Predicted	Experimental	Avg
1	-1	-0.75	-0.51	0.30	600.22	585.00	620	171.11	178.00	176.50
2	-1	-0.75	-0.51	0.30	600.22	638.00		171.11	176.00	
3	-1	-0.75	-0.51	0.30	600.22	630.00		171.11	177.00	
4	-1	-0.75	-0.51	0.30	600.22	627.00		171.11	175.00	

Conclusions

In this study, experimental investigations (based on statistical modeling) for UP resin curing process were discussed. Gel time and peak exothermic temperature were modeled in terms of the wt% of MEKP (catalyst), C_{oct} (accelerator), $CaCO_3$ (filler) and glass fibers (reinforcements). Response surface method (RSM) was chosen to effectively plan the experiments. The following conclusions can be drawn from this study:

It was found that all the linear effect of factors and certain interactions were significant. Regression equations were developed to model significant factors and interactions. It was observed that t_{gel} increased with amount of $CaCO_3$ and glass fibers while it decreased with amount of catalyst as well as accelerator. On the other hand, θ_{peak} increased with amount of catalyst as well as accelerator while decreased with amount of $CaCO_3$ and glass fibers. The catalyst and accelerator increased the rate of reaction while $CaCO_3$ and glass fibers absorb the exothermic heat, thereby reducing the same.

As an example, an optimization problem was taken up, with a t_{gel} of 600 s (10 min) and θ_{peak} of 170°C as the target values for a good quality FRP product. The optimal setting of process variables was: 1.8% of MEKP, 1.97% of C_{oct} , 4.89 % of $CaCO_3$ and 19.65% of glass fibers. The agreement of confirmatory experiments with predicted values confirmed the validity of methodology adapted.

In this work, we also wish to emphasize that consideration of interaction effects among different process variables gives valuable information for FRP manufacturing processes (which are otherwise neglected). The application of statistical methods in such situations not only saves experimental costs but also greatly increases the odds of identifying the hard-to-find solution of nagging quality control problems.

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