EFFECTIVE SAVINGS IN PRODUCTION TIMES AND COST THROUGH MONITORING OF THE EFFECT OF ADDITIVES AND TEMPERATURE ON THE GEL TIMES OF POLYESTER RESIN.

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ABSTRACT

Using a simple experimental analysis, the effect of catalyst, accelerator, inhibitor and temperature on gel times of polyester resin is fully exposed. Particular use is made of a benzoyle-peroxide-amine system with hydro quinone as inhibitor. Results show that only by careful planning, using the in-depth knowledge of gel times, can there be a saving in production times and prevention of material wastes during lamination.

Key words: Effective-savings; Production-times-and-cost; In-depth-knowledge-of-gel-times

1. INTRODUCTION:

It is a known fact that most resin manufacturers supply polyester resins which contain a proportion of accelerator in them. These resins have a storage life of several months at shop temperature and are used for cold cure applications requiring only the addition of the correct proportion of catalyst immediately before use [1]. The cure of a polyester resin commences as soon as the catalyst is added with the speed of cure dependent on:

(a) the quantity or percentage of accelerator initially present in the polyester resin from manufacturers,
(b) The prevailing room temperature*of the resin-mix during lamination.
(c) Whether or not polyester resin contains inhibitor [2, 3].

In cases where a certain time frame is desired to be achieved as cure deadline, the addition of accelerator at a given constant temperature in the percentage so as to meet this deadline becomes the controlling arm in production [4]. A minute error in the accelerator-to-resin percentage quantity required to achieve this purpose can either (a) bring about cure before the desired time/deadline which explains the reason for majority of factory materials wastes running into millions of Naira whereby resins have cured before they can be applied to fibre reinforcements during lamination or (b) bring about delay in which case resins remain uncured minutes or hours after lamination has ceased. This also can cause tremendous wastes by way of down-time periods during handlay-up processes where applications of a second coat of resin would have to wait for the already applied coat to cure, And, in machine-applied resin injection processes, gross distortions and resin-dripping culminating in total failure of the lamination process itself [5,6,7].

The purpose of this work is to fully expose, by means of experimental data, the overwhelming need for laminators (whether engaged in small scale or industrial level) to properly calculate and tabulate accurate accelerator-to- resin percentage mix taking into consideration their workshop temperatures for particular work-piece based, of course, on the calculated time it takes staff to laminate such work piece (in cases of handlay-up processes) and machines (for resin injection processes). It is intended to act as a powerful means of eliminating entirely the colossal wastes involved in resin materials during laminations and huge financial losses due to undesirable down-time periods.

2.EXPERIMENTAL PROCEDURE

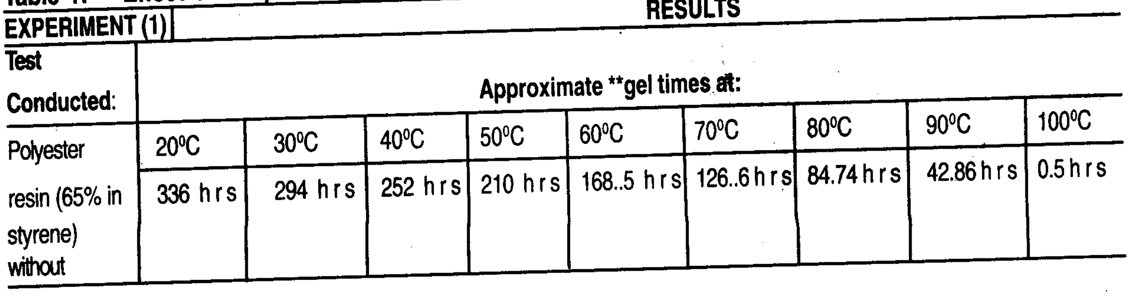
Materials

The following experiments were conducted in polyester resin matrix (containing 65% styrene) using hydroquinone as inhibitor; dimethyl aniline as accelerator and benzoyle peroxide as catalyst (see tables 1-6)

Test Method

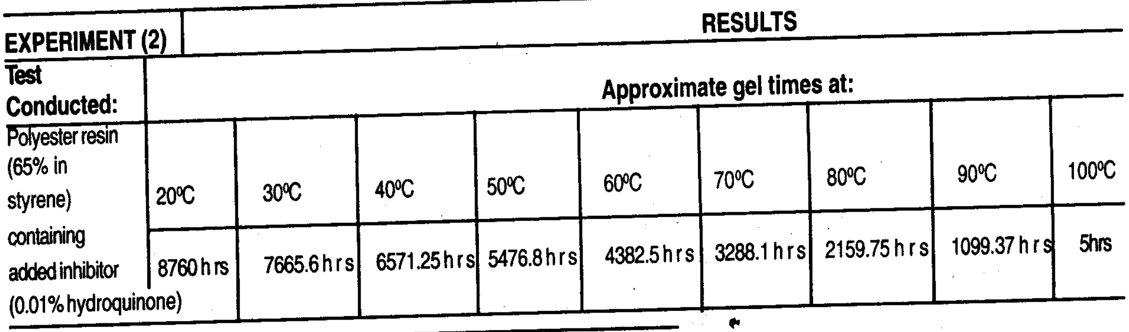
Six different tests were conducted each at temperatures of 20°C, 30°C, 40^0C, 50^0C, 60°C, 70°C, 80°C, 90°C and 100^0C respectively. These temperature ranges are considered to fall within the ranges obtainable in any workshop or factory. The approximate gel times in each case were noted and recorded under the chosen temperature points as in tables 1-6

Table 1: Effect of temperature only on gel times of (OCF E-600)polyester resin.



EXPERIMENT (1)	RESULTS								
Test Conducted:	Approximate **gel times at:								
Polyester resin (65% in styrene) without	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
	336 hrs	294 hrs	252 hrs	210 hrs	168..5 hrs	126..6 hrs	84.74 hrs	42.86 hrs	0.5 hrs

Table 2: Effect of inhibitor and temperature on gel times of (OCF E-600) polyester resin.



EXPERIMENT (2)	RESULTS								
Test Conducted:	Approximate gel times at:								
Polyester resin (65% in styrene) containing added inhibitor (0.01% hydroquinone)	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
	8760 hrs	7665.6 hrs	6571.25 hrs	5476.8 hrs	4382.5 hrs	3288.1 hrs	2159.75 hrs	1099.37 hrs	5hrs

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**This is the time it takes for the resin-mix to cure to that level where it barely exists between the solid and liquid states - that is: the transition stage when it can no longer flow or be applied as liquid even though it cannot actually be called a solid. The onset of gel is measured by the inability of the laminator to use the resin-mix any more as a laminating fluid.

Table 3: Effect of inhibitor, catalyst and temperature on gel times of (OCF E-600) polyester resin.

EXPERIMENT (3)	**RESULTS**								
Test Conducted:	**Approximate gel times at:**								
	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
Polyester resin (65% in styrene) containing added inhibitor (0.01% hydroquinone) and catalyst (1% benzoyle peroxide)	168 hrs	147 hrs	126 hrs	105 hrs	84 hrs	63 hrs	42 hrs	21 hrs	0.08 hrs

Table 4: Effect of inhibitor, catalyst, accelerator and temperature on gel times of (OCF E-600) polyester resin

EXPERIMENT (4)	**RESULTS**								
Test Conducted:	**Approximate gel times at:**								
	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
:Polyester resin (65% in styrene) containing added catalyst (1% benzoyle peroxide), inhibitor (0.01% hydroquinone) and accelerator (0.5% dimethyl aniline)	15mins	13.375mins	11.75mins	10.125mins	8.5mins	6.875	5.25	3.625	2mins

Table 5: Effect of decrease in the percentage of accelerator added to composition in experiment IV

EXPERIMENT (5)	**RESULTS**								
Test Conducted:	**Approximate gel times at:**								
	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
Polyester resin (65% in styrene) containing added catalyst (1% benzoyle peroxide), inhibitor (0.01% hydroquinone) and accelerator (0.125% dimethyl aniline)	60mins	53.5mins	47mins	40.5mins	34mins	27.5mins	21mins	14.5mins	8mins

Table 6: **Effect of a further decrease in the percentage of accelerator added to composition in experiment 6.**

EXPERIMENT (6)	RESULTS								
Test Conducted:	Approximate gel times at:								
Polyester resin (65% iin styrene) containing added catalyst (1% benzoyle peroxide), inhibitor (0.01% hydroquinone) and accelerator (0.0625% dimethyl aniline)	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
	120mins	107mins	94mins	81mins	68mins	55mins	42mins	29mins	16mins

3. RESULTS AND DISCUSSION

Table 1: Experiment 1:

We see here that as the temperature increases the time it takes for polyester resin, OCF E-600 (containing 65% styrene) and no inhibitor in it to gel decreases that is: the higher the temperature, the quicker the gel. (fig. 1)

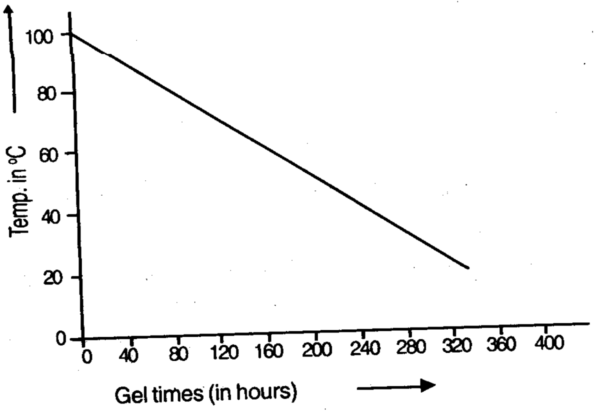


Fig 1: *Effect of temperature only on gel times of (OCF E-600) polyester resin*

Table 2: Experiment 2:

The additional influence of inhibitor (only about 0.01 % hydroquinone) to that of the temperature seems here to rather slow the speed of gel about ten times that obtained with the effect of temperature alone. At 100°C with the effect of temperature alone, we notice that it took only about 30 minutes to gel but with the added effect of inhibitor, gel time rose to 5 hours (see tables 1 & 2).

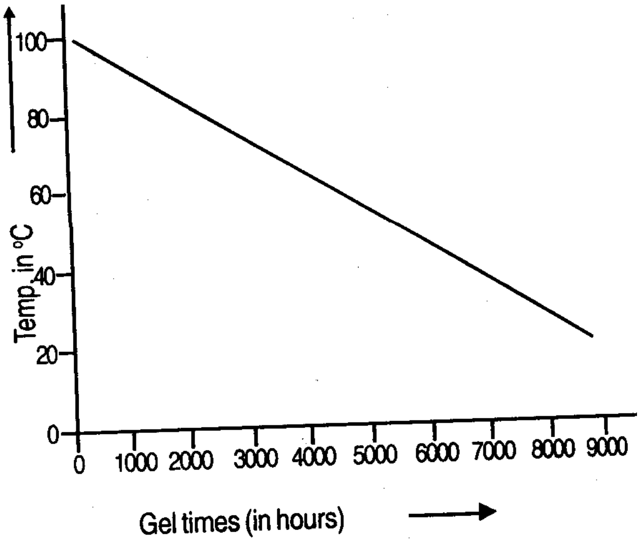


Fig. 2: *Effect of Inhibitor and Temperature on gel times (OCF E-600) polyester resin.*

Table 3: Experiment 3:

In experiment 3, a third parameter was introduced: 1% benzoyle peroxide, which is a catalyst, the effect of which speeds up the tendency to gel by 62.5 times from where the temperature / inhibitor combination left it. The graph of this behaviour is shown is fig. 3. (See also tables 2 and 3).

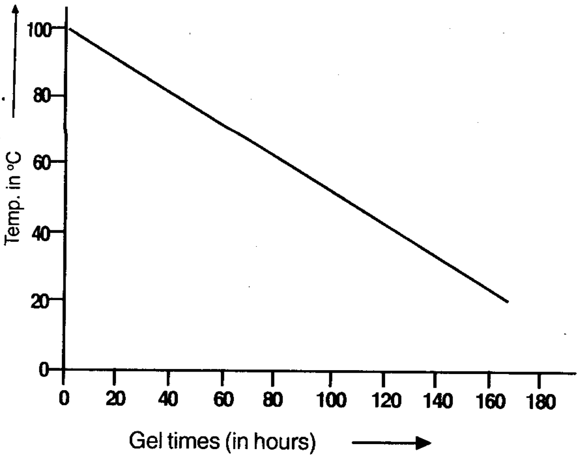


Fig. 3: *Effect of Inhibitor, Catalyst and Temperature on gel times of (OCF 3-600) polyster resin.*

Table 4: Experiment 4:

A further speed-up of the tendency to gel was achieved by the addition of an accelerator: 0.5% dimethyl aniline. However, this time the speed-up was only about 2.4 times the original value. (See tables 3 and 4 and fig. 4).

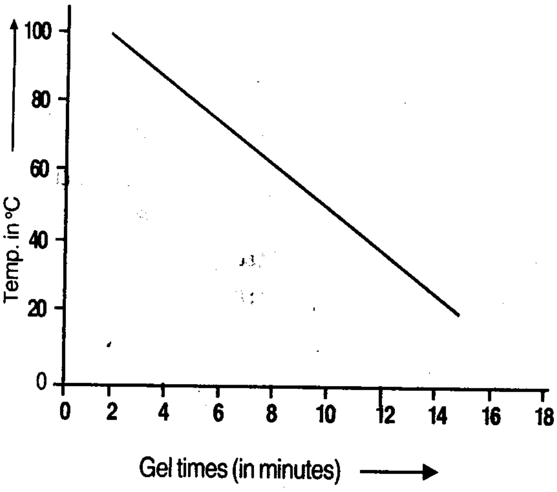


Fig. 4: Effect of Inhibitor,Catalyst, Acceleraor and Temperature on gel times of (OCF E-600) polyester resin

Table 5: Experiment 5 and Table 6: Experiment 6:

So far, we have monitored the effect of four parameters: temperature, accelerator, catalyst and inhibitor on gel times of polyester resin-mix. The first three of these we can use to hasten the speed of gel as we may desire and the last: inhibitor, we can employ in lowering or retarding the tendency to gel at wish.

Having so done, we can now employ this knowledge to obtain what 1 shall call: **Appropriate Available Time** for use in our lamination jobs/processes. Hence, tables 5 and VI are results of experiments tailored to achieve this purpose. Firstly, we recognize that of the three possible parameters we can use to alter gel times to suit our needs, the increase/ reduction of accelerator is the most convenient for the following reasons:

a The rate at which it speeds-up the tendency to gel is comparatively low when compared to those of temperature or catalyst.

b Unlike temperature or catalyst,

speed- up of gel times by addition of multiples of quantities of accelerator is found to be linear, consistent and give results that are predictable.

The two experiments: experiment 5 and 6 in tables 5 and 6 prove this. In these two experiments (see tables 5 and 6), we see that first: by reducing the accelerator used in experiment 4 down to a quarter of its original value, the gel time of 2 minutes obtained in experiment 4 at 100°C can be increased to four times its original value; that is 8 minutes in experiment 5 - which is a more convenient time- frame employable in lamination processes. Similarly, a further increase to 16 minutes was achieved. (experiment VI) by further halving the quantity of accelerator added. We see that 16 minutes is a more reasonable and useful time-frame in workshop lamination terms than 8 minutes; and 8 minutes is .even much more reasonable than 2 minutes! Why? ... This is because there is little or nothing one can do within 2 minutes before the resin-mix gels and becomes inapplicable.

Hence, with these experimental results, further multiples can be developed therefrom to meet factory/workshop needs at different prevailing temperatures provided the factory/workshop management has taken time to study and calculate the necessary time her employee laminator can spend conveniently on the job in question. This second time also which I have decided to call: Calculated Time to complete the work varies from one laminator to another depending on the experience and expertise of the individual involved.

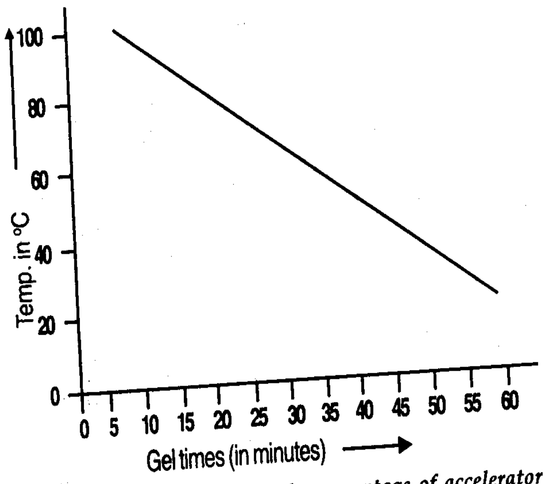


Fig. 5: *Effect of decrease in the percentage of accelerator added to composition in experiment 4.*

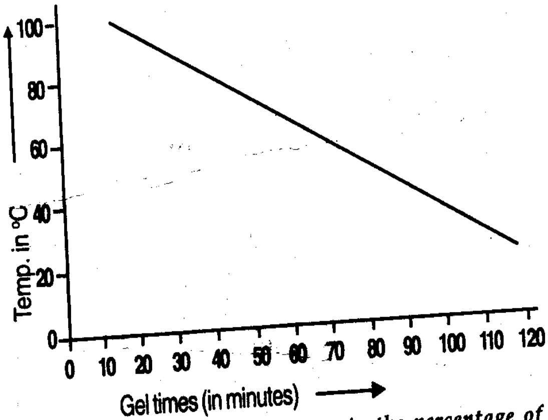
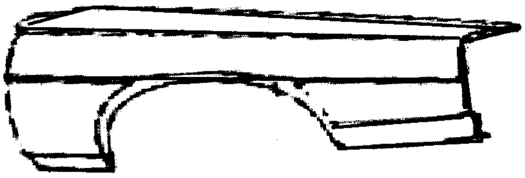
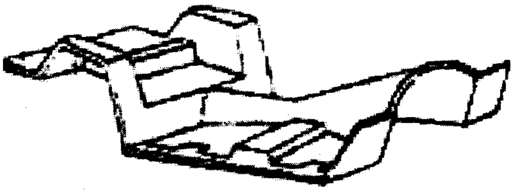


Fig. 6: *Effect of further decrease in the percentage of ccelerator added to composition in experiment 4.*

Need For Accurate Calculations During Lamination. As a specific example consider the lamination in polyester resin of two car body components: (1) two sides of a car fender and (2) the floor pan.



Right-Side Car Fender.



Floor Pan.

For each of the two fenders, in handlay· up, it may take between 5 and 7 minutes to add a new layer of mat to the already existing or cured one by a laminator And, for the required thickness of the fender, it may take a total of three or four different additions of mats to complete the lamination of a fender. This is in addition to time taken by special poinl5 such as curvature points and edges. Hence, we may have a total estimated about 7 × 3 = 21 minutes (assuming use of three layers of mat), plus say, I minutes for the special points- giving grand total of 31 minutes. This actually means that it takes about 10.3 minutes to handlay-up each layer of mat and about 1hr 2 minutes to produce the two fenders by hand lay-up in the workshop by laminator.

In resin injection process this may be about half the same time taken or even much less per machine. And, for the floor pan of a car, we may have between and twenty times this total time taken in handlay-up and resin injection respectively because of the enormous curvature and convolutions involved. However, the exact time that can be taken depends heavily on the expertise of the laminator or operator in question. In conclusion, therefore, we see that the time taken during lamination processes (whether handlay-up or resin injection) to place, add or laminate a new layer of mat to the already existing cured layer is often relatively small For the handlay-up of fenders discussed above it is about 10.3 minutes which should be much less for resin-injection process. For the lamination of a car floor pan, it may take about say (10× 10.3 = 103 minutes) at the minimum to add a full new layer of mat to the entire surface of the floor pan by handlay-up which again should be less using resin injection process.

Our use of experimental results of tables 1-6 becomes practical, meaningful and of great value when we first calculate the necessary times required for a new layer of mat to be laminated onto an already cured layer in our workshops, factories, etc., for the particular body component we are producing taking into account the expertise or ability of our laminator or operator. It may not even be car body component - it can be any product - including roofing sheets! For the particular examples discussed, applying the above table, we observe that the laminating times of 10.3 minutes and 103 minutes suggest that out of the first four tables, we are likely to be concerned only with tables 3 and 4 depending on the operating temperatures of our workshops. Table iv would normally not apply because it does not make much sense keeping a workshop at temperatures between 90°C and 100°C just to create an enabling environment for lamination. However, table iv can comfortably accommodate the lamination of the fender components at 40°C being a normal workshop temperature (compare 10.3 minutes to *the available* 11.7 minutes!). This shows an optimal matching of ***Calculated Time* to *Appropriate Available Time***. In order to accommodate the calculated lamination time for the floor pan, two approaches would be needed: (a) the lamination of the entire surface of the floor pan would have to be broken into two halves meaning 51.5 minutes for each half (b) the percentage of accelerator (dimethyl aniline) added to composition could be reduced to a quarter of the original quantity (i.e. 1,4 of 0.5% dimethyl aniline which is 0.125%). This would give us about 60 minutes *available time* to work with before cure of the resin at 20°C and about 53.5 minutes at 30°C workshop temperatures. (See table 5). Alternatively, further reductions in the percentage of accelerator added to the composition by weight could be undertaken to achieve the lamination of a full one layer of mat to the surface of the floor pan at a stretch. (See table 6: 107 minutes *available lamination time* at workshop temperature of 30°C Compare this with the *calculated time* to do the work: 103 minutes. However, care must be taken to ensure that the percentage of accelerator so added does not become small in relation to the entire composition to the point that measurement errors by weight would arise.

What Happens When Accurate Calculations Are Not Done Before Laminations

When accurate calculations of the time required to produce a body component by laminations of any process (handlay-up or resin injection) is not carried out, one of the following two main, problems may arise: the first is the possibility of the resin already mixed with laminating ingredients (catalyst, accelerator, etc) curing before it is completely utilized. This can result in enormous waste because the cure in polyester resin being a thermoset plastic is non-reversible and the cured materials can never ever be made to return to their fluid state. The unfinished layer of mat being laminated usually result in laminate products with rough surfaces, resin starved and jagged points / areas. The second is a situation where the lamination has been concluded long before the cure process starts. A down-time of so many minutes and sometimes hours would need to be wasted while waiting for that first layer of mat to cure before the next can be applied. The length of time to wait, however, is dependent on the percentage of accelerator in the resin mix.

Comparatively, however, in the two examples, already discussed above, calculations showed that a fender requiring 10.3 minutes to laminate one layer of mat to it at workshop temperature of 40°C when allocated a time period of 11.75 minutes (from the tables) would be comfortably completed on schedule with an extra time of 1.45 minutes before cure begins. This time can be used by the laminator for a short rest before the next layer. Similarly, with regards to the floor pan which required 103 minutes for one layer of mat to be added by lamination an *available time* of 107 minutes (taken from the tables) at 30^0C workshop temperature means that an extra four minutes allowance before cure begins a good balance between the risk of cure before finish of lamination and that waste in down-time period during waiting.

4. CONCLUSION

From the two examples given above, we can easily discover the immense benefits accruable to us when we spend time to calculate: (1) The time required to laminate one layer of mat a work piece at our own workshop temperatures and (2) the need to use the above tables (or multiples derived therefrom) in selecting *Appropriate Available Time* (AAT) before cure for use along with our *Calculated* Time (CT) I any work piece we may find ourselves engaged in.

In conclusion, therefore for effective savings in production times (that is elimination of undue lengthy down periods) and cost (materials waste) factory directors, managers or foremen must recognize the overwhelming to undertake accurate calculation as aforestated and to paste same at visible points along their production lines staff handlay-up laminators and resin injection machine operators to study abide by them.

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