Increasing the hardness of a two-component PU clear coat

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TL;DR

An eight-run fractional factorial design was used to identify the factors controlling the pendulum hardness of a two-component polyurethane clear coat. Curing temperature, curing time, and catalyst level each increased hardness. Hardener type had no measurable effect. The catalyst effect depended on curing temperature. It raised hardness substantially at 60 °C but had essentially no effect at 80 °C. Two operating points produced comparable hardness, near 86 to 91 oscillations: 80 °C with 1 % catalyst, or 60 °C with 3 % catalyst, both at 60 minute curing time.

Background

The hardness of an existing two-component polyurethane clear coat was to be increased without changing the base formulation. Four factors that can be varied in processing and formulation were considered candidates: curing temperature, hardener type, catalyst level and curing time. The response was pendulum hardness, reported in oscillations, where a higher count indicates a harder film.

The objective was to determine which of the four factors control hardness, whether their effects are independent, and what settings maximize hardness within the current formulation.

Results

Main effects

Three of the four factors increased hardness. The estimated effect is the change in hardness when a factor moves from its low to its high setting, averaged over the other factors.

FACTOR	LOW → HIGH	EFFECT (OSCILLATIONS)
Curing temperature	60 → 80 °C	+19.0
Curing time	30 → 60 min	+16.5
Catalyst level	1 → 3 %	+14.0
Hardener type	A → B	+1.5

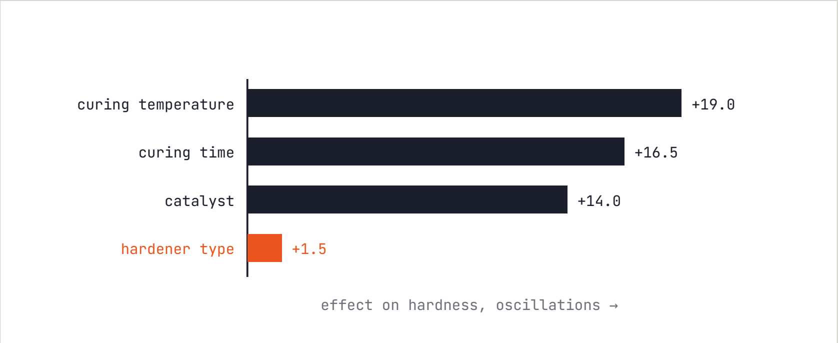

Figure 1. Main effects on hardness, in oscillations. Curing temperature, curing time, and catalyst level each raise hardness by 14 to 19 oscillations. Hardener type (orange) moves it by 1.5 oscillations and is treated as inactive.

Hardener type changed hardness by 1.5 oscillations, within the noise of the measurement, and is treated as inactive.

Temperature–catalyst interaction

The catalyst effect depended on curing temperature. Averaged over the remaining factors, the mean hardness in each combination of the two factors was:

	CATALYST 1 %	CATALYST 3 %
60 °C	45.0	77.5
80 °C	82.5	78.0

At 60 °C, increasing catalyst from 1 to 3 % raised hardness by 32.5 oscillations. At 80 °C the same increase changed hardness by −4.5 oscillations, effectively no change. At the higher curing temperature the film is already near its achievable hardness, leaving little for the catalyst to contribute.

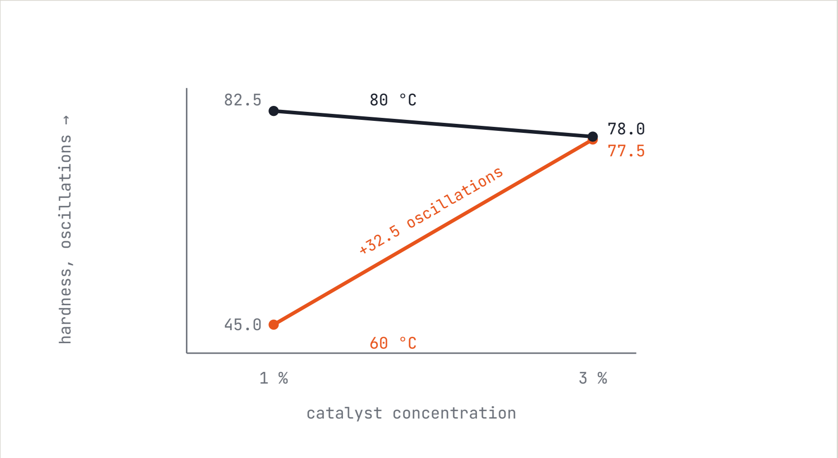

Figure 2. Curing temperature × catalyst interaction. At 60 °C, raising catalyst from 1 to 3 % increases hardness by 32.5 oscillations; at 80 °C the response is essentially flat. The lines are not parallel, which is the interaction.

Operating points

With the 60 minute curing time, two factor settings produced comparable predicted hardness:

ROUTE	TEMPERATURE	CATALYST	PREDICTED HARDNESS (OSCILLATIONS)
Higher temperature, low catalyst	80 °C	1 %	≈ 91
Lower temperature, more catalyst	60 °C	3 %	≈ 86

Conclusion

Curing temperature, curing time, and catalyst level control the hardness of the clear coat. Hardener type does not. Because catalyst is effective only at the lower curing temperature, target hardness can be reached either by curing at 80 °C with 1 % catalyst or by curing at 60 °C with 3 % catalyst.

Curing at 60 °C with 3 % catalyst for 60 minutes is recommended. It yields approximately 86 oscillations, comparable to the 80 °C route, at a curing temperature 20° lower, with corresponding savings in energy and reduced thermal load on the substrate. Hardener type can be selected on cost or availability without affecting hardness.

Appendix

A. Experimental design

A two-level fractional factorial design was selected to estimate the effects of the four factors. A full two-level factorial design would require $2^4 = 16$ runs. A half fraction ($2^{4-1} = 8$ runs) was used instead, halving the experimental effort while retaining the ability to estimate all four main effects clear of one another and of all two-factor interactions.

The design is of Resolution IV. It was constructed by assigning the fourth factor to the three-factor interaction of the first three (generator $D = A \cdot B \cdot C$), which gives the defining relation $I = ABCD$. The factors and their coded levels were:

FACTOR	CODE	LOW (-1)	HIGH (+1)
Curing temperature	A	60 °C	80 °C
Hardener type	B	type A	type B
Catalyst level	C	1 %	3 %
Curing time	D	30 min	60 min

A fractional design is appropriate at a screening stage, where the aim is to find which of several factors have large main effects. Most of the information is carried by the main effects and a few two-factor interactions, while three- and four-factor interactions can reasonably be assumed negligible.

B. Runs and measured response

Eight panels were prepared, one per run, each cured under the indicated combination of settings, and measured for pendulum hardness.

RUN	A TEMP	B HARDENER	C CATALYST	D TIME	HARDNESS (OSC)
1	60 °C	type A	1 %	30 min	33
2	60 °C	type A	3 %	60 min	87
3	60 °C	type B	1 %	60 min	57
4	60 °C	type B	3 %	30 min	68
5	80 °C	type A	1 %	60 min	88
6	80 °C	type A	3 %	30 min	72
7	80 °C	type B	1 %	30 min	77
8	80 °C	type B	3 %	60 min	84

Mean hardness was 70.75 oscillations.

C. Confounding (alias structure)

In a Resolution IV design each main effect is aliased only with three-factor interactions, which are assumed negligible, so the four main effects are estimated cleanly. The two-factor interactions, however, are aliased in pairs and cannot be separated within the eight runs:

- AB = CD (temperature × hardener with catalyst × time)
- AC = BD (temperature × catalyst with hardener × time)
- AD = BC (temperature × time with hardener × catalyst)

Because hardener type was found to be inactive as a main effect, any interaction involving hardener is expected to be negligible. On that basis each aliased pair is attributed to its non-hardener member: AC = BD is read as temperature × catalyst, AD = BC as temperature × time, and AB = CD as catalyst × time. This attribution is an assumption, not a measurement; the complementary half fraction, or the full sixteen-run design, would confirm it directly.

D. Effect estimates

Each estimable effect was calculated as the difference between the mean response at the high and low levels of the corresponding contrast.

TERM	INTERPRETATION	EFFECT (OSC)
A	Curing temperature	+19.0
D	Curing time	+16.5
C	Catalyst level	+14.0
AC = BD	Temperature × catalyst	-18.5
AD = BC	Temperature × time	-5.0
B	Hardener type	+1.5

TERM	INTERPRETATION	EFFECT (OSC)
AB = CD	Catalyst × time	-1.0

E. Identification of active effects

The eight runs are unreplicated, so no independent estimate of pure experimental error is available. Active effects were therefore identified from the distribution of the effect estimates. Four effects (A, AC, D, C; magnitudes 14.0 to 19.0) are clearly separated from the remaining three (AD, B, AB = CD; magnitudes 1.0 to 5.0). The three small effects were pooled to form an error estimate with three degrees of freedom, and the four large effects were retained in the model.

F. Analysis of variance

The reduced model contains the four active terms; the three negligible terms are pooled as residual error.

SOURCE	SUM SQ	DF	MEAN SQ	F	P
Curing temperature (A)	722.0	1	722.0	38.3	0.009
Temperature × catalyst (AC)	684.5	1	684.5	36.3	0.009
Curing time (D)	544.5	1	544.5	28.9	0.013
Catalyst level (C)	392.0	1	392.0	20.8	0.020
Residual (pooled)	56.5	3	18.83		
Total	2399.5	7			

The model accounts for 97.6 % of the total variation ($R^2 = 0.976$, adjusted $R^2 = 0.945$). The residual standard deviation is 4.3 oscillations.

G. Model

The fitted model, in coded factor levels (–1 at the low setting, +1 at the high setting), is:

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Hardness = 70.75 + 9.5·A + 7.0·C + 8.25·D - 9.25·(A·C)
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where A is curing temperature, C is catalyst level, and D is curing time. Each coefficient is one half of the corresponding effect. The negative AC coefficient quantifies the reduction in the catalyst effect as curing temperature increases.

H. Predictions

Predicted hardness at the two recommended operating points, both at the high curing-time setting (60 min):

- 80 °C, 1 % catalyst: 90.75 oscillations
- 60 °C, 3 % catalyst: 85.75 oscillations

The temperature × catalyst cell means reported under Results summarize the interaction underlying these predictions.

I. Limitations

- The data are illustrative and were generated without random error or replication. The error estimate is obtained by pooling small effects rather than from repeated measurements, so the significance tests should be read in that light.
- The two-factor interaction aliasing is resolved by assumption (an inactive hardener), not by measurement. The complementary half fraction, or the full sixteen-run design, would resolve it directly.
- The fitted model is valid only within the tested factor ranges (60 to 80 °C, 1 to 3 % catalyst, 30 to 60 minutes). Extrapolation beyond these ranges is not supported by the data.