Design of Experiments Worksheet

Study Code: DOE-2026-014 — Coating Yield Optimization

Nexa Materials Laboratory | Process Development Group

Principal Investigator	Design Type	Date Issued	Revision
Dr. Amara Josef	2^3 full factorial + centre points	03 Aug 2026	Rev. 2

1. Purpose

This worksheet governs a designed experiment to identify which process factors most strongly affect coating **yield** and **tensile strength** on the Line C powder-coat cell, and to fit a first-order (plus interaction) model usable for setpoint optimization. It is the controlled record used to plan factor levels, randomize run order, execute trials, and capture raw responses before they are transcribed into the analysis workbook.

2. Background

Line C yield has averaged 87.4% over the last quarter, below the 92% target carried in the Q3 operating plan. Engineering suspects cure oven temperature and catalyst loading interact – raising temperature alone has not moved yield consistently across shifts. Rather than adjust one factor at a time, this study runs a full factorial screen across three candidate factors so that main effects *and* two-factor interactions can be estimated cleanly, with replicated centre points to check for curvature before committing to a response-surface follow-up.

Scope of this worksheet

- Defines factors, levels, and units (Section 2).
- Records the randomized run order and blank response fields for live data capture (Section 3).
- States the planned analysis method and decision rule in advance, to avoid post-hoc rationalization of the result (Section 4).
- Provides the pre-run checklist and sign-off block (Appendix).

Randomization policy

Run order was randomized *before* this sheet was printed (Section 3, seed recorded in the Appendix). Operators must execute runs in the printed order — re-sequencing to group similar setpoints defeats the purpose of randomization and will confound trial order with any drift in ambient humidity or resin lot.

1 Factors, Levels, and Responses

2.1 Factor and level definitions

Three factors were selected from the fishbone review held 22 Jul 2026 (attendees: Process Engineering, Quality, and Line C operators). Two levels each, coded −1 (low) and +1 (high), plus a centre point (0) run in triplicate to test for curvature.

Code	Factor	Description	Low (−1)	Centre (0)	High (+1)
A	Cure Temperature	Oven zone 3 setpoint	170 °C	185 °C	200 °C
B	Catalyst Loading	% w/w in resin blend	0.8%	1.1%	1.4%
C	Cure Time	Belt dwell time in oven	8 min	10 min	12 min

Units note

Temperature entered on the oven HMI in °C directly; catalyst loading is dispensed by mass on the batching scale (see SOP-BAT-07); cure time is set by belt speed, converted via the line's speed-to-dwell chart posted at Station 3.

2.2 Responses

Response	Definition	Units	Target
Y_1 — Yield	Coated panels passing visual + adhesion inspection, per 50-panel run	%	≥ 92%
Y_2 — Tensile Strength	Mean of 5 pull-test coupons per run (ASTM D638 setup)	MPa	≥ 38 MPa

2.3 Held-constant factors and assumptions

- Resin lot fixed to Lot #SYN-2291 for the full study — do not substitute lots mid-run.
- Substrate: 1.2 mm cold-rolled steel panels, single supplier batch, degreased per SOP-PRE-02.
- Ambient humidity logged but not controlled; recorded per run as a covariate (Section 3).
- Line speed upstream of the oven held at nominal 4.2 m/min regardless of cure-time setting.

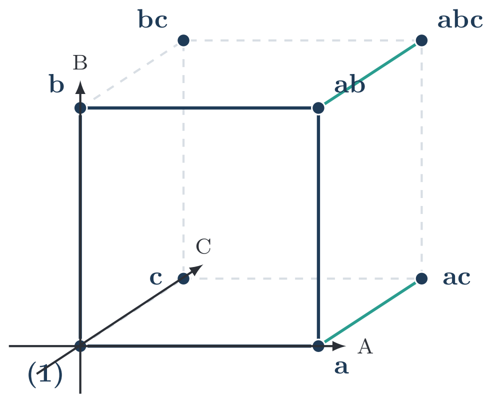
Safety constraint

Do not exceed 205 °C on zone 3 under any combination of factors — this is 5 % above the resin manufacturer's stated degradation threshold. If a computed high level from a future centre-point augmentation would cross this line, stop and consult Process Engineering before running.

2 Design Matrix and Run Order

3.1 Design geometry

The eight factorial vertices of the 2^3 cube are labelled below in standard Yates notation; each letter present indicates that factor is at its *high* level, and (1) denotes all factors low. Three additional centre-point replicates (not shown on the cube) sit at the geometric middle and are used only to test for curvature.



3.2 Randomized run sheet

Execute runs strictly in **Run Order**; **Std Order** is the Yates reference used only for analysis. Random seed: 7719 (Mersenne Twister, drawn 01 Aug 2026, witnessed by Quality). Fill Y_1, Y_2, and the operator/timestamp fields immediately after each run — do not batch-fill at the end of the shift.

Run	Std	A	B	C	T (°C)	Cat %	t (min)	Y_1 (%)	Y_2 (MPa)	Op/Time
1	7	−1	+1	+1	170	1.4	12			
2	3	−1	+1	−1	170	1.4	8			
3	10	0	0	0	185	1.1	10			
4	1	−1	−1	−1	170	0.8	8			
5	5	−1	−1	+1	170	0.8	12			
6	8	+1	+1	+1	200	1.4	12			
7	2	+1	−1	−1	200	0.8	8			
8	11	0	0	0	185	1.1	10			
9	4	+1	+1	−1	200	1.4	8			
10	9	0	0	0	185	1.1	10			
11	6	+1	−1	+1	200	0.8	12			

Worked example — how to record a response

Suppose Run 4 (all factors low) yields 44 of 50 panels passing and a mean tensile of 36.2 MPa, read at 07:42 by operator J. Okafor. Enter: Y_1 = **88.0**, Y_2 = **36.2**, Op./Time = **JO / 07:42**. Record the failing-panel defect mode in the Appendix notes field, not on this sheet.

3 Analysis Plan

Fixed in advance, per Quality's pre-registration policy for optimization studies — the method below is not to be altered after responses are observed.

4.1 Effect estimation

For each factor, the main effect is the difference between the average response at the high level and the average response at the low level, using only the eight factorial runs (centre points excluded from effect estimates, used only for the curvature test below):

$$\text{Effect}_A = \bar{Y}_{A=+1} - \bar{Y}_{A=-1}, \qquad \text{Effect}_{AB} = \frac{(\bar{Y}_{A=+1,B=+1} + \bar{Y}_{A=-1,B=-1}) - (\bar{Y}_{A=+1,B=-1} + \bar{Y}_{A=-1,B=+1})}{2}$$

Two-factor interaction effects (AB, AC, BC) and the three-factor interaction (ABC) follow the same contrast pattern from the signed table below.

Std	A	B	C	AB	AC	BC	ABC
1 (1)	−	−	−	+	+	+	−
2 a	+	−	−	−	−	+	+
3 b	−	+	−	−	+	−	+
4 ab	+	+	−	+	−	−	−
5 c	−	−	+	+	−	−	+
6 ac	+	−	+	−	+	−	−
7 bc	−	+	+	−	−	+	−
8 abc	+	+	+	+	+	+	+

4.2 Curvature check

Before trusting the linear model, compare the average of the eight factorial runs against the average of the three centre-point runs:

$$\Delta_{\text{curv}} = \bar{Y}_{\text{factorial}} - \bar{Y}_{\text{centre}}$$

If Δ_{curv} is large relative to the pooled centre-point standard deviation (rule of thumb: $|\Delta_{\text{curv}}| > 2\,s_{\text{centre}}$), a purely linear model is inadequate and this study escalates to a central-composite augmentation rather than reporting final setpoints from the factorial alone.

4.3 ANOVA skeleton

Populate after data entry into the analysis workbook (`DOE-2026-014-analysis.xlsx`); this sheet only reserves the structure so reviewers can see the plan before results exist.

Source	DF	SS	MS	F	p
A — Cure Temperature	1				
B — Catalyst Loading	1				
C — Cure Time	1				
AB	1				
AC	1				
BC	1				
ABC	1				
Curvature	1				
Pure error (centre pts)	2				
Total	10				

4.4 Decision rule

- Statistical significance: effect retained in the model if $p < 0.05$.
- Practical significance: an effect below **1.5 percentage points** of yield is treated as noise regardless of p-value — the line's shift-to-shift baseline variation is already ± 1.2 points.

- If A, B, and AB are all significant, proceed directly to a steepest-ascent confirmation run at the predicted optimum; if only main effects are significant, the centre point already approximates the optimum and no further runs are needed.
- Any run outside its factor's safety constraint (Section 2.3) is excluded from the model and flagged, not silently dropped.

⚠ Do not p-hack

Do not add, drop, or re-code runs after seeing partial results. If a run must be voided (equipment fault, wrong panel batch), record the reason in the Appendix and re-run at the *same* design point with a new randomized slot, drawn by Quality — not chosen by the operator.

4 Pre-Run Checklist and Sign-Off

5.1 Pre-run checklist

Complete in full *before* Run 1. Initial each item; do not check a box for a step you did not personally verify.

- ☐ Resin Lot #SYN-2291 confirmed on batching label
- ☐ Oven zone 3 thermocouple calibration current (< 30 days)
- ☐ Catalyst scale zeroed and tared
- ☐ Belt speed chart posted at Station 3
- ☐ Panel substrate batch matches Section 2.3
- ☐ Randomized run sheet (Section 3.2) printed, not re-sorted
- ☐ Humidity logger active at Station 1
- ☐ Pull-test rig (ASTM D638 fixture) verified with reference coupon

5.2 Voided-run log

Voided Run	Std Order	Reason	Re-run Slot

5.3 Sign-off

Role	Name	Signature	Date
Prepared by (PI)	Dr. Amara Josef		
Reviewed by (Quality)	Priya Ramanathan		
Approved by (Process Eng. Lead)	Tobias Lindqvist		

Retention

Completed worksheets are scanned into the LIMS study folder `DOE-2026-014` within 24 hours of the final run and retained per the site's 7-year quality-record policy. The original paper copy stays with the PI until the analysis report is approved.

5.4 Method references

The design and analysis conventions used in this worksheet follow standard factorial-design practice [2, 1]; the curvature-check heuristic in Section 4.2 follows the engineering handbook treatment of centre-point augmentation [3].

References

[1] George E. P. Box, J. Stuart Hunter, and William G. Hunter. *Statistics for Experimenters: Design, Innovation, and Discovery.* John Wiley & Sons, Hoboken, NJ, 2nd edition, 2005.

[2] Douglas C. Montgomery. *Design and Analysis of Experiments.* John Wiley & Sons, Hoboken, NJ, 10th edition, 2019.

[3] NIST/SEMATECH. e-Handbook of Statistical Methods. Technical Report Handbook 151, National Institute of Standards and Technology, 2012.