Novel coatings development – the importance of including auxiliary responses

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Agenda

- Industrial Research Methodology
  - What are auxiliary responses?
- Example 1 - New Resin Design for Architectural Coatings
- Example 2 - Protective Coating
- General Observations

DMADV

	Key Questions
Define	What are the goals of the project?
Measure	What are the critical to quality characteristics? Are the measurement processes suitable?
Analyze	What factors can we change to make improvements?
Design	What factor combinations lead to optimum performance?
Verify	Will the product work in the real world?

Critical for success

- Impactful, clear
- Fast, efficient
- Cost effective, robust

Simplified Process Map – Automotive Basecoat Development

Make Resin

- X – monomer types
- X – level of each monomer
- X – initiator type
- X – feed rates
- N – stir speed
- y – rheology
- y – particle size

Make Basecoat

- X – co-resins
- X – solvents
- X – additives
- X – pigments
- X – order of addition
- N – temperature
- y – rheology
- y – surface tension

Spray Basecoat

- X – air pressure
- X – flow rate
- X – temperature
- X – humidity
- X – substrate
- N – basecoat age
- y – atomization
- y – transfer efficiency

Apply Topcoat

- X – topcoat type
- X – time after basecoat
- N – topcoat batch
- N – technician
- y – wetting
- y – penetration

Cure coatings

- X – oven temperature
- X – time after topcoat
- X – orientation
- N – airflow
- N – oven type
- N – contaminates
- y – rheology during cure
- y – substrate temp. profile

Measure

- Y – appearance
- Y – colour
- Y – humidity resistance
- Y – adhesion
- y – thermomechanicals
- y – microscopy

KEY:

X = Controllable Variable
Y = Main Metric(s)
N = Noise Variable
y = Auxiliary response

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Make Resin → **Make Basecoat** → **Spray Basecoat** → **Apply Topcoat** → **Cure coatings** → **Measure**

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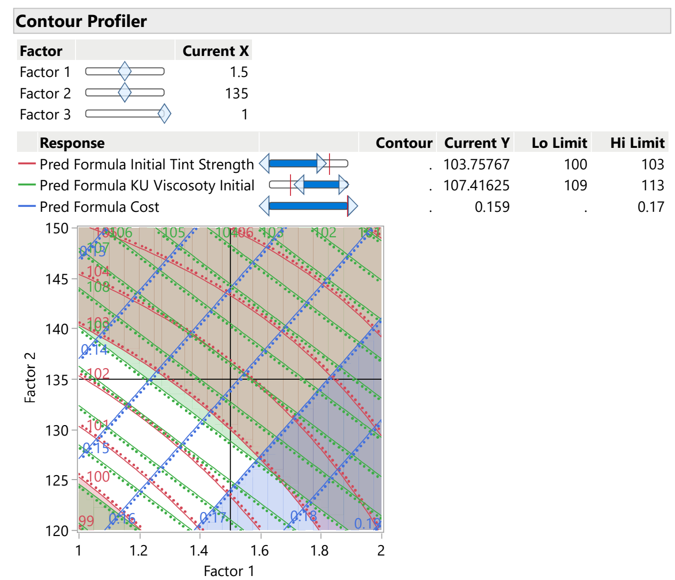
Example 1 – New Resin Design for Architectural Coatings

Goal	New resin for white base paints that meets performance requirements for several global product lines
Initial Status	No single resin meets all requirements. Early Prototypes struggled with low tint strength, poor heat age stability, and poor reproducibility.



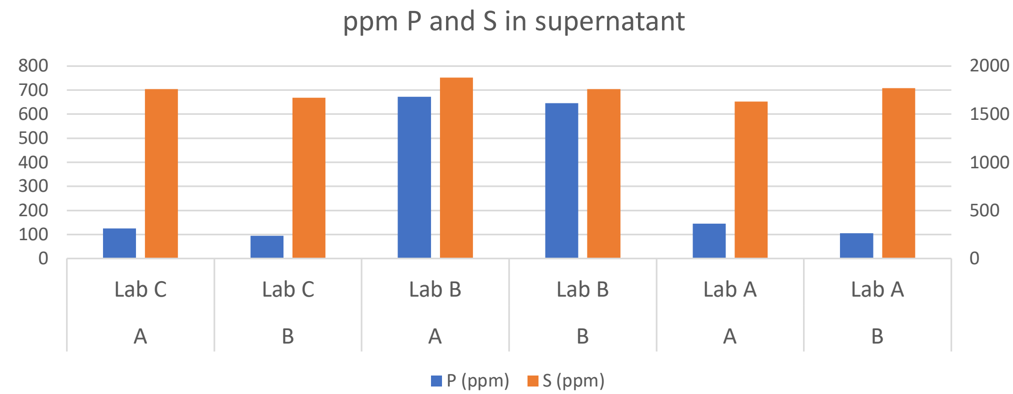
Example1 – Resin Location DOE

- **Goal**
  - Confirm and quantify previously observed correlation between particle size and tint strength
  - Does co-surfactant addition point affect key properties?
  - Can the resin synthesis be reproduced across three different locations?

	Resin formulation	Target PSz	Co-surfactant addition	reactor location
1	A	150	INITIAL CHARGE	Lab C
2	C	110	INITIAL CHARGE	Lab C
3	D	150	FEED	Lab C
4	B	130	INITIAL CHARGE	Lab C
5	E	130	FEED	Lab C
6	F	110	FEED	Lab C
7	A	150	INITIAL CHARGE	Lab B
8	C	110	INITIAL CHARGE	Lab B
9	D	150	FEED	Lab B
10	B	130	INITIAL CHARGE	Lab B
11	E	130	FEED	Lab B
12	F	110	FEED	Lab B
13	A	150	INITIAL CHARGE	Lab A
14	C	110	INITIAL CHARGE	Lab A
15	D	150	FEED	Lab A
16	B	130	INITIAL CHARGE	Lab A
17	E	130	FEED	Lab A
18	F	110	FEED	Lab A

Example 1 – Resin Location DOE - Conclusions

- What could cause higher conductivity and pH at 1 hr?
  - Analysis of supernatant after precipitation of the polymer

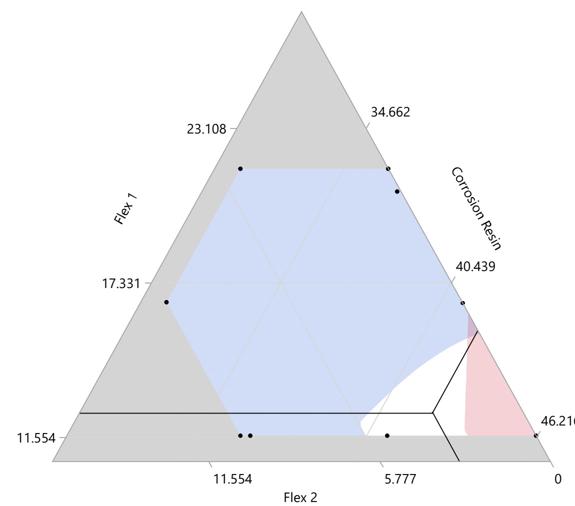


- Resins from Lab B have about 4x the level of P than other labs.
- Only one raw material brings in P
- Further investigation revealed that material supplied to Lab B was too concentrated.

- Auxiliary data from the DOE (little ys) allowed the problem to be identified very quickly.
- Project stayed on track.
- Bonus – a new method of influencing tint strength was identified.

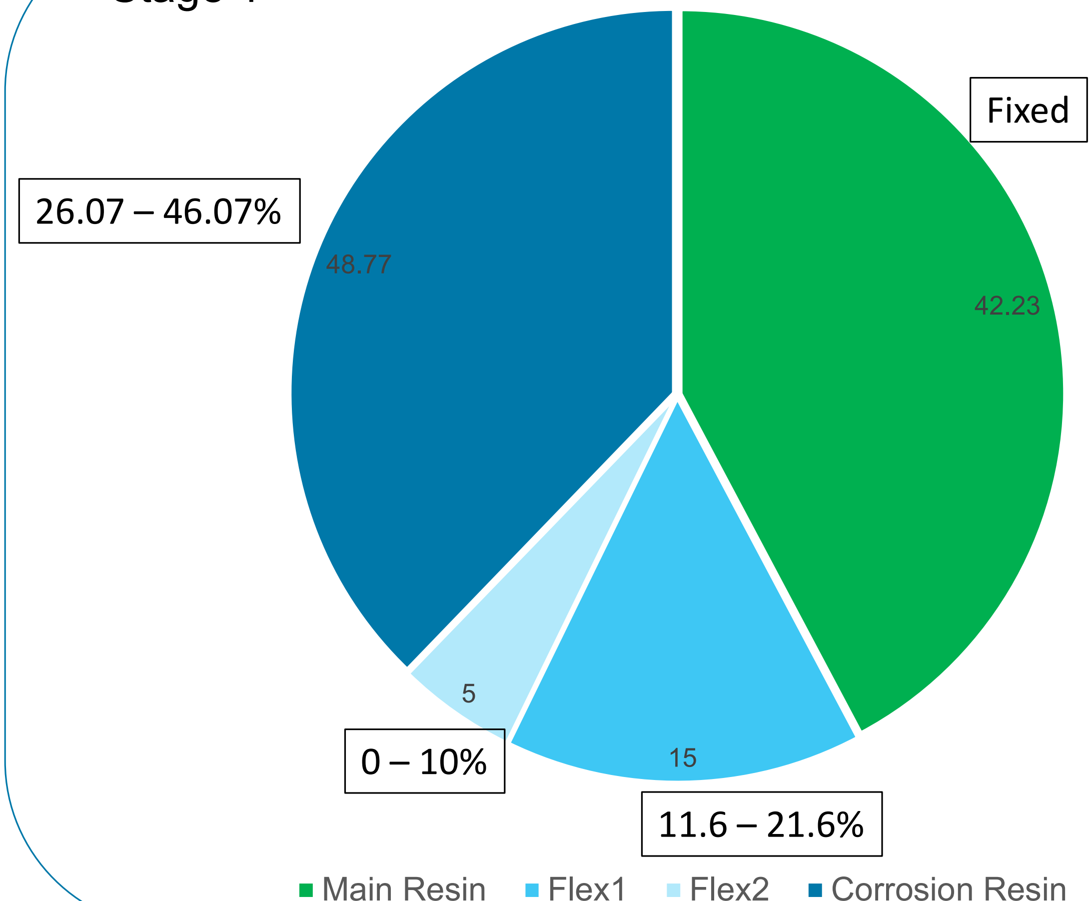
Example 2 – Protective Coating

- Five resin components to be investigated- Corrosion Resin, Flex1, Flex2, Flex3, Flex4
- First three are components incorporated during stage 1 of the coating prep. The other two are added later in a separate step.
- How do the resin components affect corrosion and flexibility?
- What resin levels deliver the best combination of these properties?



Example 2 - Design

Stage 1



Stage 2

\+ Flex3

10 – 20%

\+ Flex4

0 – 15%

Flex2 + Flex3 >10%

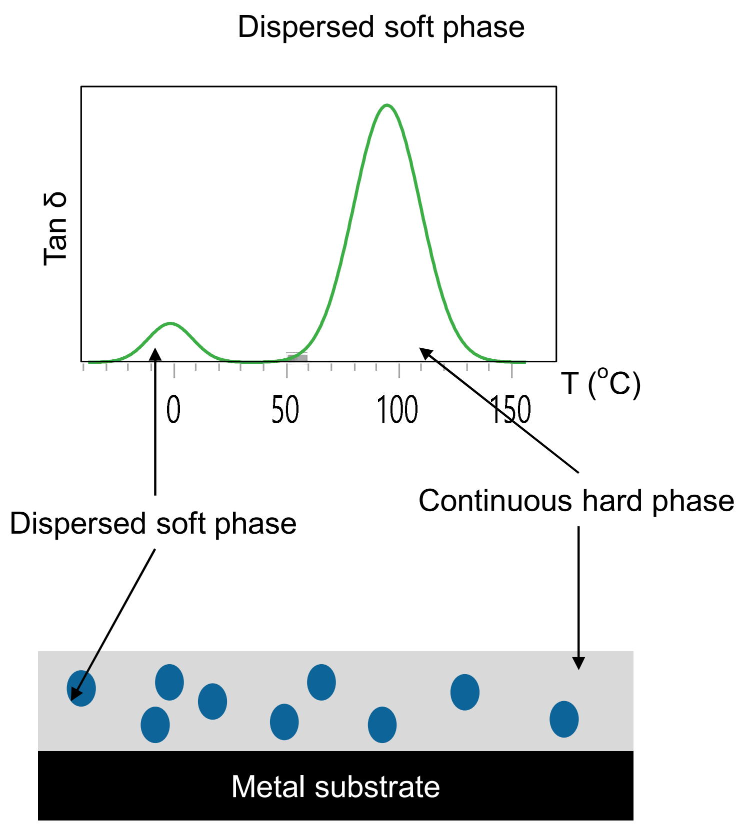
Flex2 + Flex3 <30%

Flex1 + Flex2 + Flex 3 + Flex4 < 41.6%

Flex1 + Flex2 < 26.6%

Example 2 - Learnings

- Multiple Tgs are usually a sign of a multi-phase material
  - Confirmed by microscopy
- High Tg of the continuous phase provides good corrosion resistance
- Soft dispersed phase contributes to flexibility



Conclusions

- It is possible to carry out successful DOEs where only the critical responses are measured (Ys), but…

- Including carefully selected auxiliary responses (ys) can often be very valuable.
  - Bring clarity to unexpected results
  - Build scientific knowledge
  - Simpler or better test methods

- JMP provides many tools to help with this

- We thank the many associates at PPG's Coatings Innovation Centre who contributed to this work.