# ISEA WEBINAR
# A CASE STUDY OF A MIXED-LEVEL OMARS DESIGN

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W. L. Gore & Associates

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*Together, improving life*

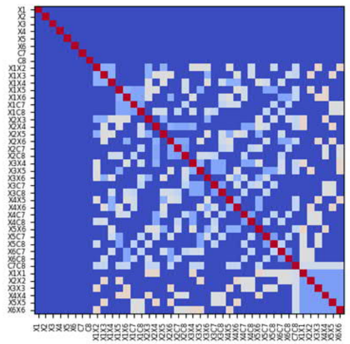
# What is the talk about?

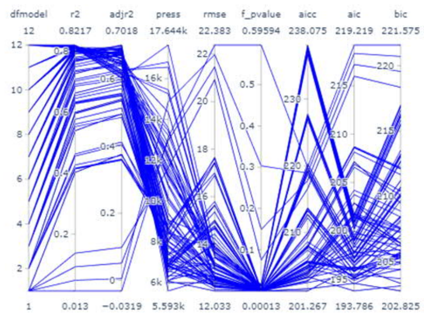
From the perspective of a statistical consultant

The company that allows me to do
such a valued and enjoyable body of work

The journey of applying a(n OMARS) design

- Insight into the application behind
- My way to, and success factors of, experimental planning
- What is an OMARS design, also relative to other designs?
- Results from the DOE and how they were analyzed in a new software prototype (EFFEX)

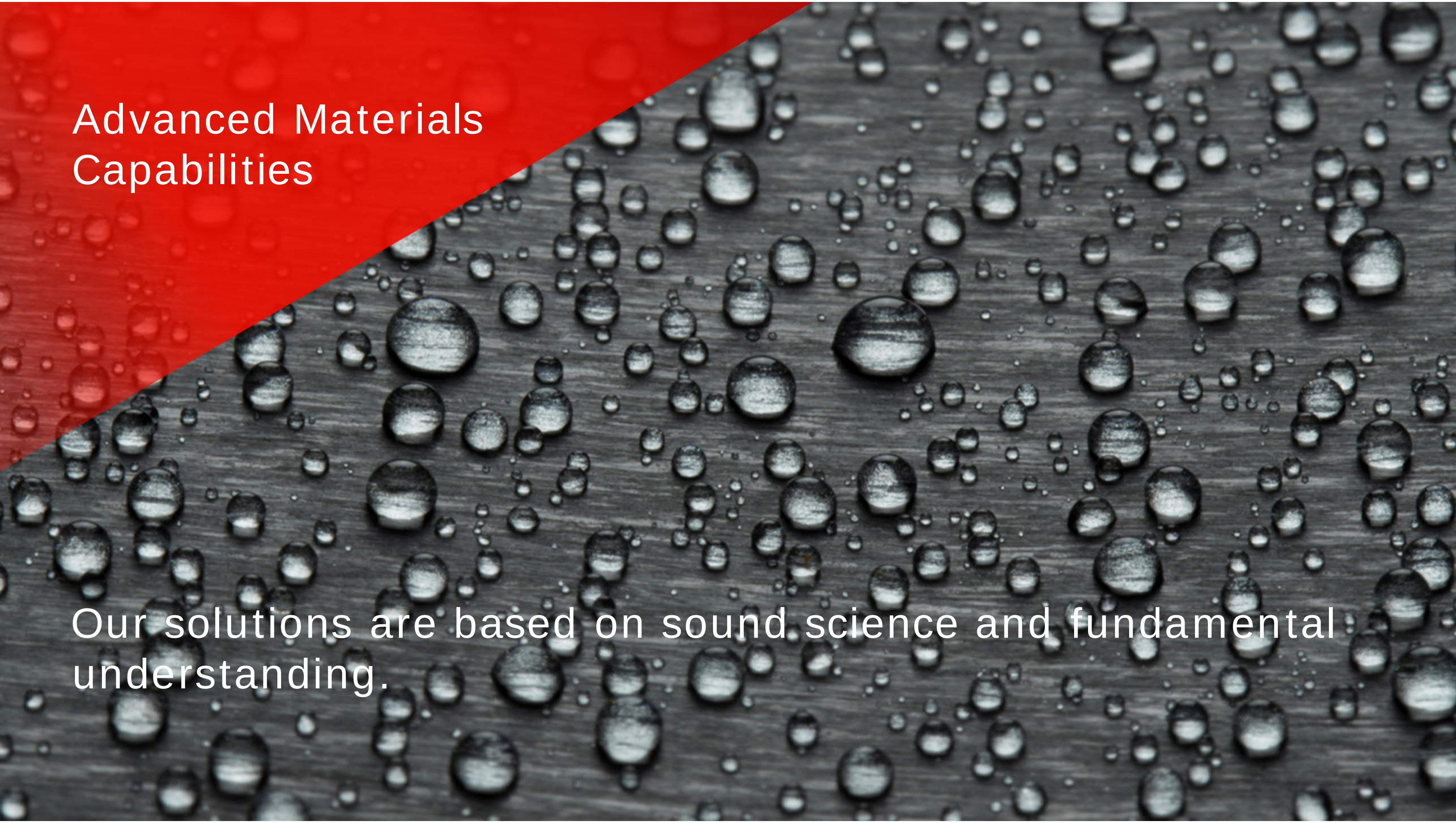




# Agenda

1. W. L. Gore & Associates
2. Product and process
3. Experimental design
4. Statistical analysis
5. Summary

# Advanced Materials Capabilities



Our solutions are based on sound science and fundamental understanding.

# 13,000

Community of over 13,000 Associates

# Global

Gore has manufacturing facilities in the United States, Germany, United Kingdom, the Netherlands, Japan and China, and sales offices around the world.

# $4.8

Billion of annual revenues

# One of the 200 largest

privately-held U.S. companies

# >3,500

unique inventions

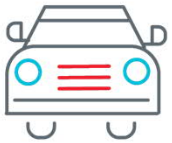
# Our culture

Globally recognized as a great place to work.

# Gore innovates across 15+ diverse industries

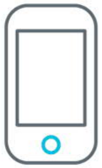
Healthcare &
Life Sciences

Aerospace



Automotive

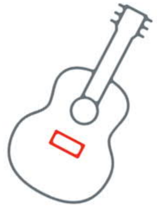
Industrial &
Chemical



Mobile
Electronics

Public Safety

Apparel &
Textiles



Consumer
Products

# INNOVATION

Gore innovates with purpose to deliver meaningful solutions that solve the challenges of our customers and communities.

# The case study

## WHAT

- Chemical product
- Functionalized fine powder copolymer

- Used for liquid filtration applications within the semiconductor industry
- Strong surface properties of the filtration element improve filtration efficiency

# The case study

## WHY

- Supplier shutdown
- Move production from external to internal

## CHALLENGE

- “Copy that product as similarly as possible”
- Improvements welcome
- Timelines short
- Materials extremely expensive

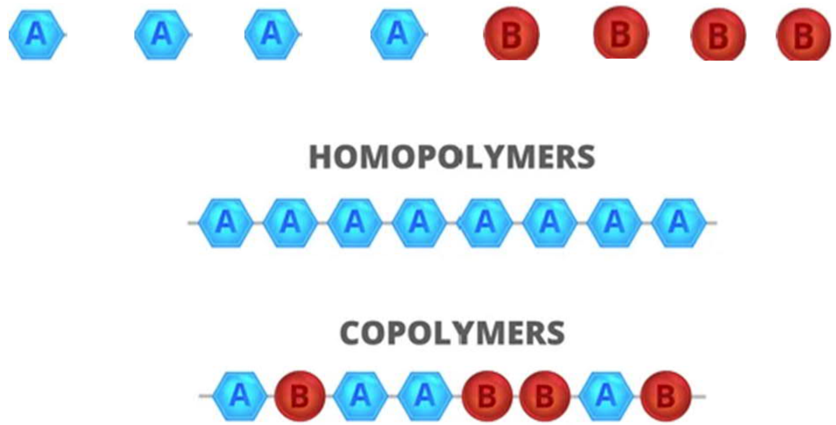
# The production process

Chemical view

- Two different monomers result in one co-polymer

Polymerization → Coagulation → Drying

Finishing




- Polymer is made in first step
- The following two steps are for extracting and drying of the product

# The production process

Properties and constraints

- Batch process
  - In one batch, all factor settings remain the same
  - Batch = production unit = experimental unit
  - No blocking, no split-plot structure
  - Full randomization
  - Material can be tested after each step
- One batch takes 30 hours of making on a full-scale production line
- Raw material cost
- DOE size limited due to the cost
- Execution of the DOE took several months

# EXPERIMENTAL DESIGN

# Experimental design

## BUDGET

- Maximum 24 runs

## GOALS

- Validate from development stage to commercial production
- Meet all customer specifications reliably

- Identify key process factors – screening
- Predict future process capability – optimization/ response-surface modeling
- Combine screening phase and optimization into ONE single experiment

# Process and Factors (X)

Polymerization → Finishing

1. Setting T
2. Setting P
3. Chemical S (2 levels)
4. Chemical E

5. Acid amount
6. Composition ratio
7. Polishing time (2 levels)
8. Drying temperature

# Two-factor interactions evaluation

Factor names standardized

| | Poly factors (process step 1) | | | | Finishing factors (process step 2) | | | |
|---|---|---|---|---|---|---|---|---|
| Factor | s1_x1 | s1_x2 | s1_x3 | s1_x4 | s2_x5 | s2_x6 | s2_x7 | s2_x8 |
| s1_x1 | | high | medium | high | 0 | high | ? | 0 |
| s1_x2 | | | medium | ? | 0 | high | 0 | 0 |
| s1_x3 | | | | high | 0 | medium | medium | 0 |
| s1_x4 | | | | | 0 | high | 0 | 0 |
| s2_x5 | | | | | | 0 | ? | high |
| s2_x6 | | | | | | | ? | high |
| s2_x7 | | | | | | | | 0 |
| s2_x8 | | | | | | | | |

- Helps combine statistical properties with subject matter expertise
- Brings knowledge, assumptions, and uncertainties to the table

# Statistical model

## FACTORS

- All factors continuous
- 6 on three levels each
- 2 on two levels each

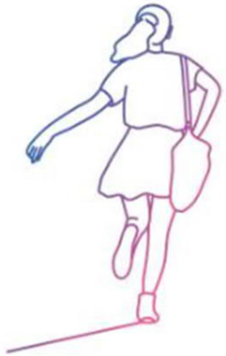
## MINIMUM VIABLE MODEL

- 8 main effects
- 6 quadratic effects
- 8 key two-factor interactions selected by applying subject matter expertise

# Statistical model

Full response surface model

- 1 constant term (intercept)
- 8 main effects
- 6 quadratic effects
- 3 main effects of covariates
- 28 two-factor interactions
  ---------------------------------------
- 46 model degrees of freedom → $2^{46}$ = 70,368,744,177,664 potential models !



Covariates on top...

- Several covariates were recorded and considered influential
- Not part of the design or model setup
- 3 of them were included in the analysis later on

# Design choice

## REQUIREMENTS

- As many as 8 factors → typically use a screening design
- Optimization & prediction → typically use a response surface design

→ Minimum 3 levels per factor required

→ How to handle that many model effects?

## CHOICE

What designs with 3 levels per factor exist that can cover both,
screening and optimization,
in one step instead of two and hence save a full DOE?

# Design choice

An OMARS design was considered the best choice

# OMARS designs?

Courtesy of Peter Goos, KU Leuven

- Experimental designs for quantitative factors
- Every factor is studied at three levels

→ Therefore, they are called response surface designs

- All main effects are orthogonal to each other

→ Therefore, they are called orthogonal designs

- All main effects are orthogonal to
  - all two-factor interactions
  - all quadratic effects

→ Therefore, they are called minimally aliased

Orthogonal

Minimally

Aliased

Response

Surface

Designs

# OMARS designs that you might know already

Courtesy of Peter Goos, KU Leuven

## Traditional response surface designs

- Central composite designs (axials on face)
- Box-Behnken designs

They are “strong” OMARS designs

- Interaction effects are orthogonal to each other
- Interaction effects are orthogonal to quadratic effects

SUPER LARGE

NO ALIASING

## Definitive screening designs (DSD)

- 3 levels per factor
- Much smaller than traditional RS designs
- Still screening designs but with the potential to do response surface modeling, IF effect sparsity applies
- Substantial aliasing among interactions
- Substantial aliasing between interactions and quadratics

SMALL

SUBSTANTIAL ALIASING

# Catalog of original OMARS designs

Courtesy of Peter Goos, KU Leuven

- Bridge the gap between small DSDs and large traditional RS designs

- Screening and response surface experiment in one, guaranteed
- Good projection properties
- Less aliasing
- Better power for quadratic effects

# Catalog of OMARS designs

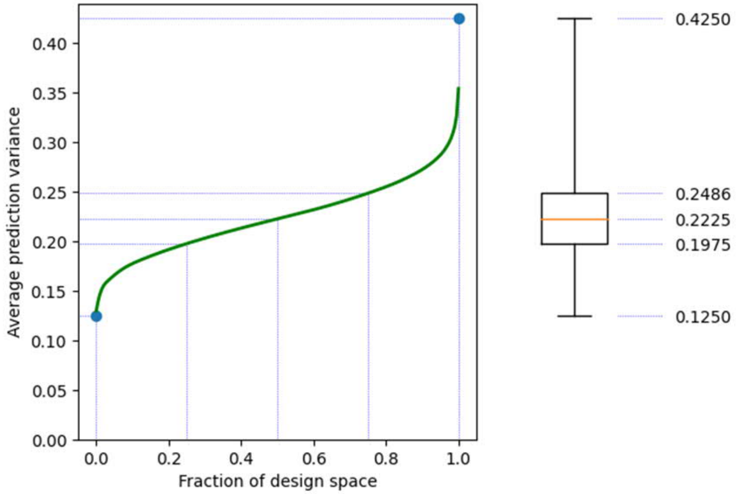
What’s in there today?

- Three level designs for quantitative factors
- Mixed-levels: Quantitative factors can have 2 or 3 levels
  – Categorical factors with 2 levels possible
- Orthogonally blocked designs
- Orthogonality and minimal aliasing property are kept

→ Mixed-level OMARS design

# Properties of the chosen design

- Uniform precision
- Good powers – just weaker for quadratics
  - price to pay for a small design size
- Prediction variances below 0.5




| Effect | Relative estimate error |
|---|---|
| intercept | 0.204 |
| X1 | 0.223 |
| X2 | 0.223 |
| X3 | 0.223 |
| X4 | 0.223 |
| X5 | 0.223 |
| X6 | 0.223 |
| C7 | 0.204 |
| C8 | 0.204 |

Powers for alpha=0.05

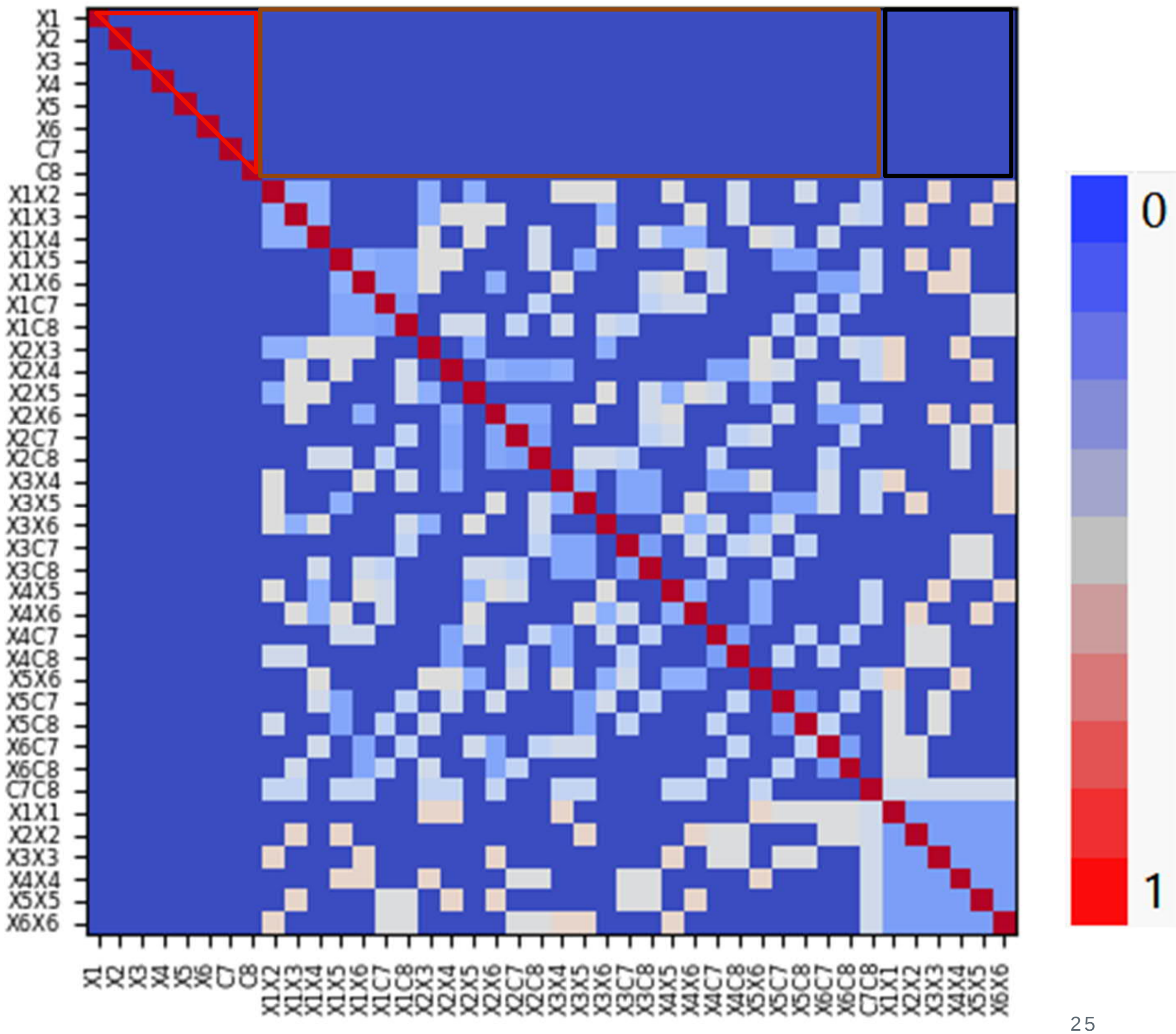
| model | effects | SNR=1 | SNR=1.5 |
|---|---|---|---|
| Intercept | main | 0.989 | 0.999 |
| ME | main | 0.986 | 0.999 |
| ME | interaction | 0.96 | 0.995 |
| ME | quadratic | 0.403 | 0.556 |

# Color map on correlations

Main effects orthogonal to

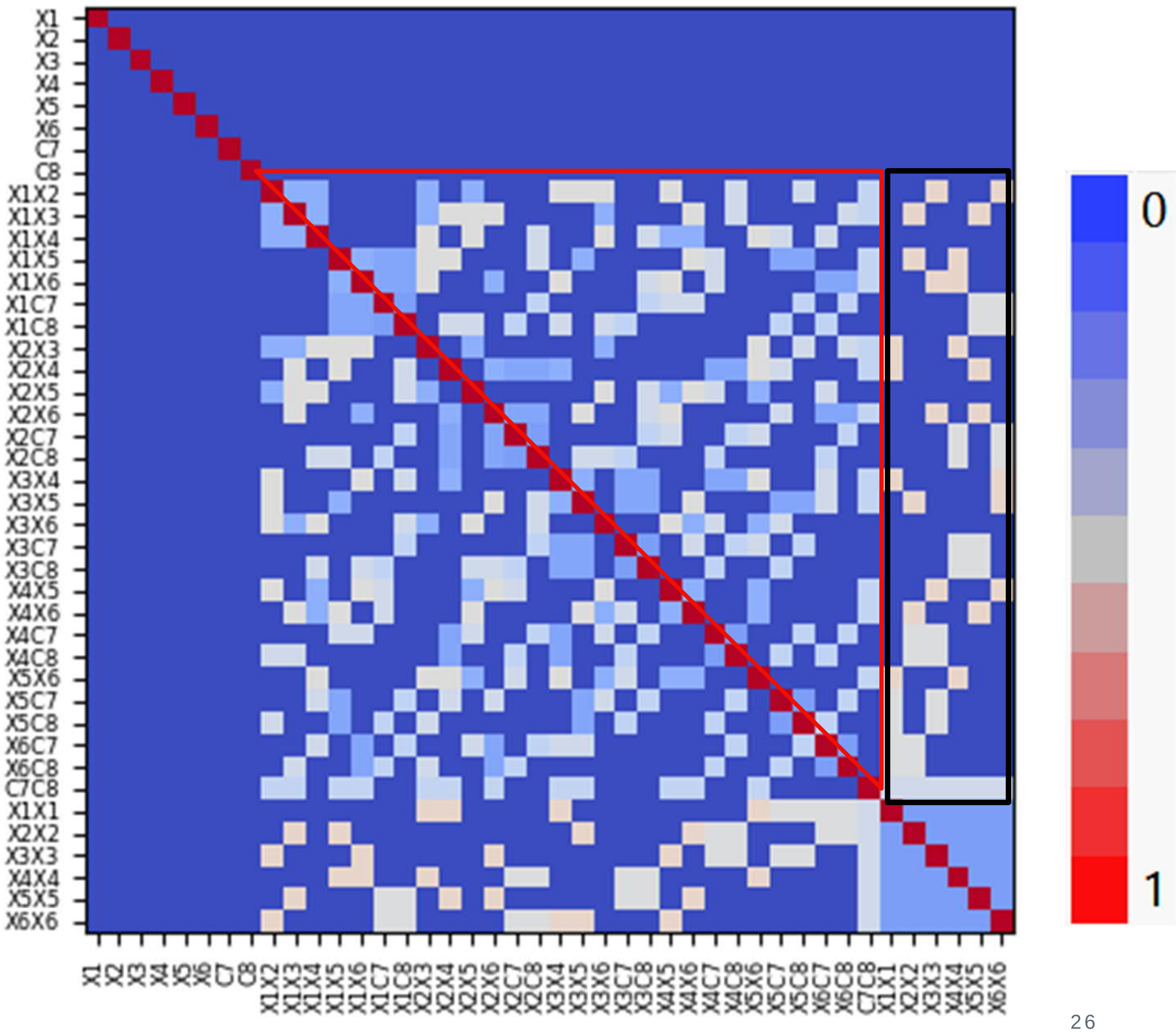
- Other main effects
- Two-factor interactions
- Quadratic effects

Please note:
Factors on 3 levels are labeled X (X1 – X6),
factors on 2 levels are labeled C (C7, C8)



# Color map on correlations

- Correlations between any pair of 2-factor interactions are <= 0.5
- Correlations between 2-factor interactions and quadratic effects are <= 0.54



| | Polymerization factors | | | | Finishing factors | | | |
|---|---|---|---|---|---|---|---|---|
| EU | Process Setting T | Process Setting P | Chemical S amount | Chemical E amount | Acid amount | Composition ratio | Polishing time | Drying temperature |
| | s1_x1 | s1_x2 | s1_x3 | s1_x4 | s2_x5 | s2_x6 | s2_x7 | s2_x8 |
| 1 | -1 | 1 | -1 | 1 | 1 | 1 | -1 | 0 |
| 2 | -1 | -1 | -1 | 0 | 1 | -1 | 1 | -1 |
| 3 | 1 | 0 | 1 | -1 | 1 | 1 | -1 | -1 |
| 4 | 1 | 0 | -1 | -1 | -1 | 1 | 1 | 1 |
| 5 | 0 | -1 | [illegible] | -1 | 1 | 1 | 1 | -1 |
| 6 | -1 | 1 | [illegible] | [illegible] | 0 | 1 | -1 | 1 |
| 7 | 1 | 1 | [illegible] | [illegible] | [illegible] | 0 | -1 | 1 |
| 8 | 1 | -1 | [illegible] | [illegible] | [illegible] | 0 | -1 | -1 |
| 9 | 1 | -1 | [illegible] | [illegible] | [illegible] | [illegible] | 1 | -1 |
| 10 | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 1 | 1 |
| 11 | 1 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 1 | 1 |
| 12 | 1 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | -1 | -1 |
| 13 | 1 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | -1 | 1 |
| 14 | -1 | -1 | [illegible] | [illegible] | [illegible] | [illegible] | 1 | 1 |
| 15 | 0 | 1 | -1 | [illegible] | [illegible] | [illegible] | 1 | -1 |
| 16 | -1 | 0 | -1 | 1 | [illegible] | [illegible] | 1 | 1 |
| 17 | 1 | 1 | 1 | -1 | -1 | [illegible] | 1 | 0 |
| 18 | -1 | -1 | 1 | -1 | -1 | [illegible] | 1 | -1 |
| 19 | -1 | 0 | 1 | 1 | 1 | -1 | -1 | -1 |
| 20 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 |
| 21 | 0 | 1 | 1 | 1 | -1 | -1 | -1 | 1 |
| 22 | -1 | 1 | 1 | -1 | 1 | 0 | 1 | 1 |
| 23 | -1 | 1 | 1 | 0 | -1 | 1 | -1 | -1 |
| 24 | -1 | -1 | -1 | -1 | -1 | -1 | -1 | 0 |

Experimental plan

| EU | Polymerization factors | | | | Finishing factors | | | | Covariates | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Process Setting T | Process Setting P | Chemical S amount | Chemical E amount | Acid amount | Composition ratio | Polishing time | Drying temperature | Drying oven | Particle size | TGA after Poly |
| | s1_x1 | s1_x2 | s1_x3 | s1_x4 | s2_x5 | s2_x6 | s2_x7 | s2_x8 | s2_z5 | s2_z6 | s2_z7 |
| 1 | -1 | 1 | -1 | 1 | 1 | 1 | -1 | 0 | 1 | 197 | 6,50 |
| 2 | -1 | -1 | -1 | 0 | 1 | -1 | 1 | -1 | 2 | 234 | 4,13 |
| 3 | 1 | 0 | 1 | -1 | 1 | 1 | -1 | -1 | 1 | 128 | 6,21 |
| 4 | 1 | 0 | -1 | -1 | -1 | 1 | 1 | 1 | 2 | 133 | 6,26 |
| 5 | 0 | -1 | | -1 | 1 | 1 | 1 | -1 | 1 | 171 | 6,20 |
| 6 | -1 | 1 | | | 0 | 1 | -1 | 1 | 2 | 247 | 5,06 |
| 7 | 1 | 1 | | | | 0 | -1 | 1 | 1 | 170 | 5,99 |
| 8 | 1 | -1 | | | | [illegible] | -1 | -1 | 2 | 153 | 6,56 |
| 9 | 1 | -1 | | | | | [illegible] | -1 | 1 | 170 | 6,80 |
| 10 | 0 | -1 | | | | | | 1 | 2 | 238 | 5,76 |
| 11 | 1 | -1 | | | | | | 1 | 1 | 153 | 6,02 |
| 12 | 1 | 1 | | | | | | -1 | 2 | 299 | 5,41 |
| 13 | 1 | -1 | | | | | | 1 | 1 | 182 | 7,44 |
| 14 | -1 | -1 | [illegible] | | | | | 1 | 2 | 265 | 6,73 |
| 15 | 0 | 1 | -1 | | | | | -1 | 1 | 256 | 6,29 |
| 16 | -1 | 0 | -1 | 1 | | | [illegible] | 1 | 2 | 154 | 4,99 |
| 17 | 1 | 1 | 1 | -1 | -1 | | 1 | 0 | 1 | 287 | 5,67 |
| 18 | -1 | -1 | 1 | -1 | -1 | 0 | 1 | -1 | 2 | 222 | 5,76 |
| 19 | -1 | 0 | 1 | 1 | 1 | -1 | -1 | -1 | 1 | 124 | 5,33 |
| 20 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 2 | 195 | 7,28 |
| 21 | 0 | 1 | 1 | 1 | -1 | -1 | -1 | 1 | 1 | 194 | 5,53 |
| 22 | -1 | 1 | 1 | -1 | 1 | 0 | 1 | 1 | 2 | 128 | 5,03 |
| 23 | -1 | 1 | 1 | 0 | -1 | 1 | -1 | -1 | 1 | 119 | 5,78 |
| 24 | -1 | -1 | -1 | -1 | -1 | -1 | -1 | 0 | 2 | 165 | 5,06 |

Experimental plan plus covariates

# Where do you get OMARS designs?

EFFEX

- Spin-off company for OMARS designs
- 500,000 OMARS Pareto-optimal designs are available in the web-based EFFEX™ software
  - Three-level OMARS designs
  - Mixed-level OMARS designs
  - Orthogonally blocked OMARS designs
- Multi-criteria design selection
- José Nuñez Ares and Peter Goos

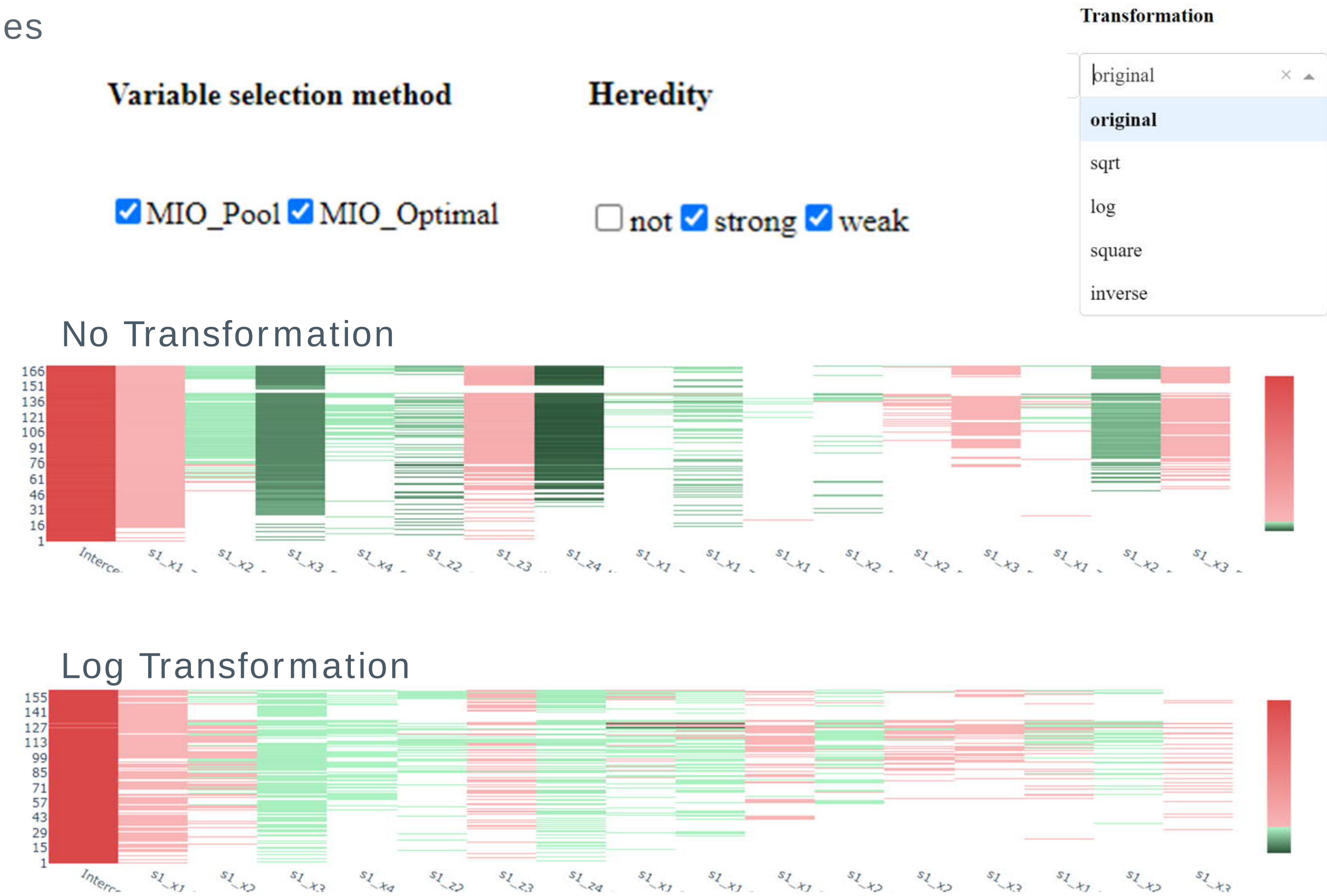
WANNA TAKE A BREATH?
I DO!

# STATISTICAL ANALYSIS

# Model selection for a single response (particle size) after process step 1 (polymerization)

Plots with courtesy of José Nunez Ares

- Effects in the generated raster plot show how often and how strong each effect appears in the many models adjusted (166 and 155, resp.)
- Frequency approach for model selection
- Model candidates also available with standard transformations
- Here: clearer picture with no transformation

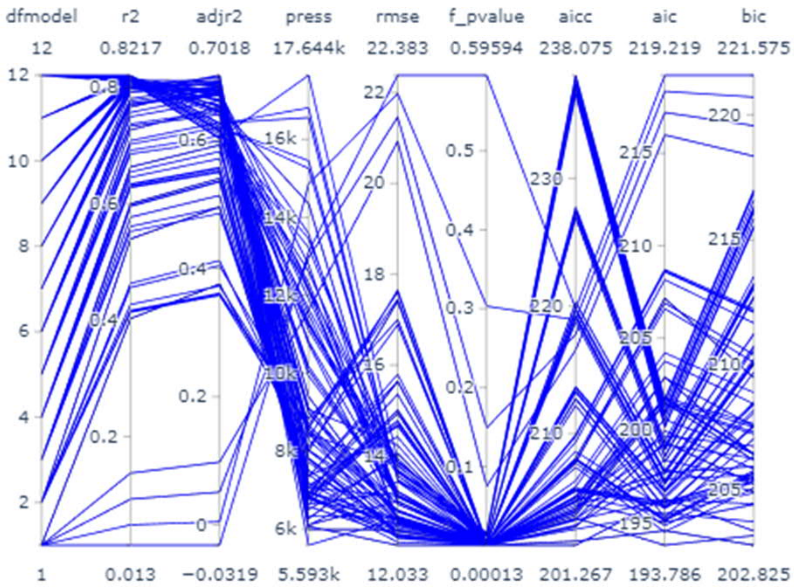


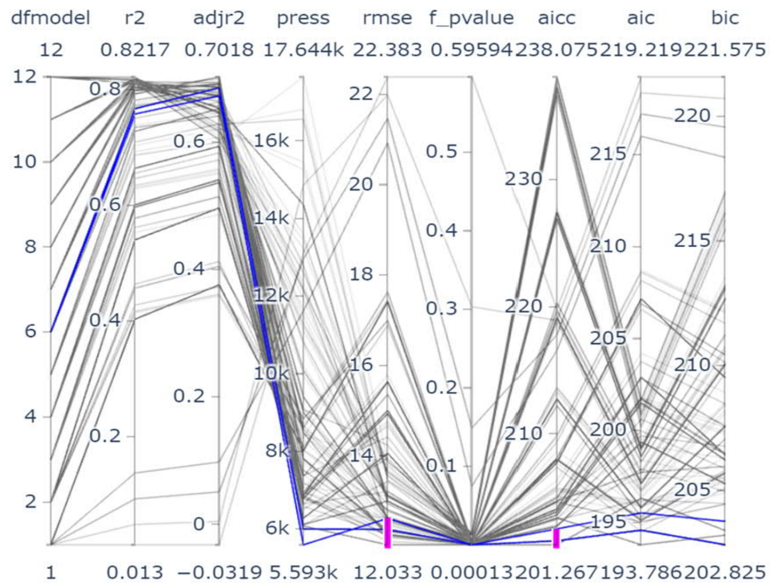
# Model selection for a single response (particle size) after process step 1 (polymerization)

Plots with courtesy of José Nunez Ares

Model quality parallel coordinates plot

- Nine model performance criteria for filtering
- All in one graph

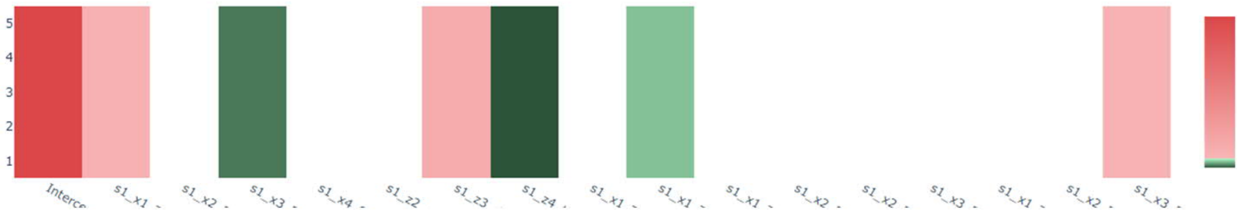


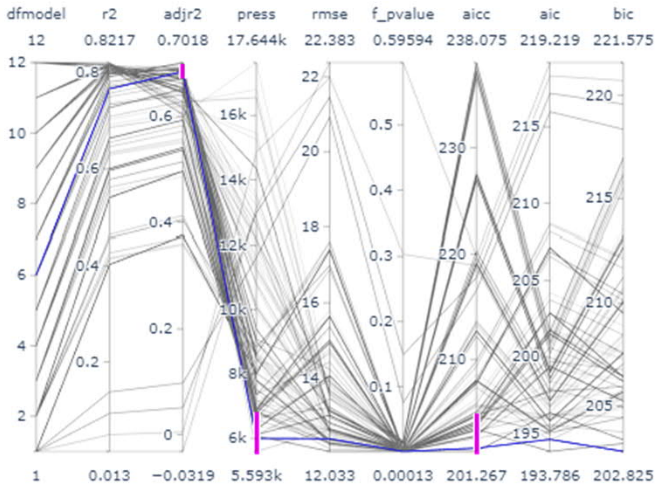



# Model selection for a single response (particle size) after process step 1 (polymerization)

Plots with courtesy of José Nunez Ares

- Now: down-select one final model candidate from the many
- Two model selection criteria applied

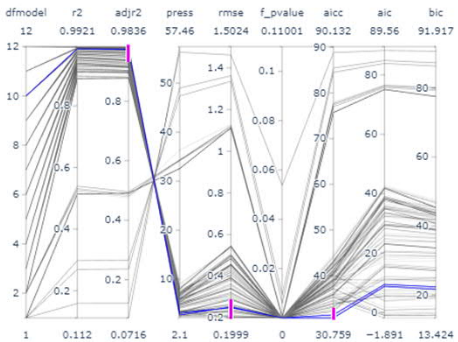




# Model selection for a single response (TGA after finishing) after process step 2 (finishing)

Plots with courtesy of José Nunez Ares

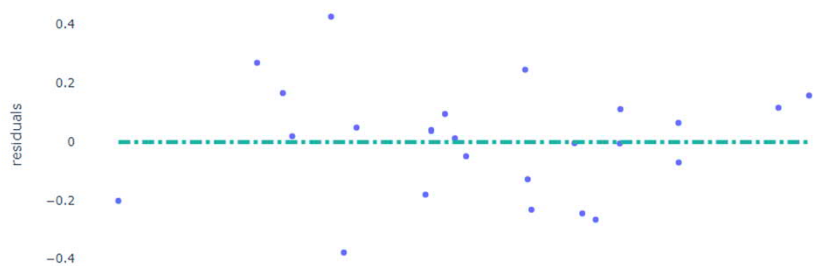
Same procedure after process step 2 – just with even more model effects

# Model selection for a single response (TGA after finishing) after process step 2 (finishing)

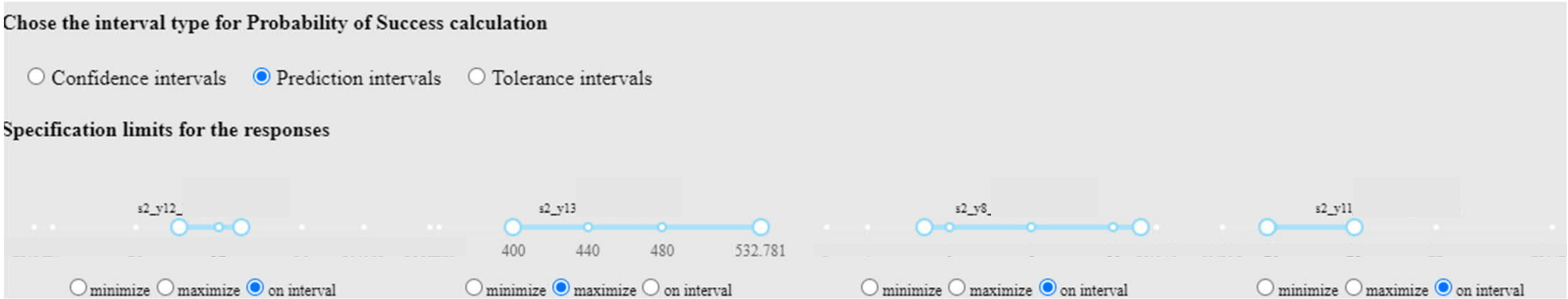
Plots with courtesy of José Nunez Ares


residual vs predicted plot



# Optimization for multiple responses

Plots with courtesy of José Nunez Ares



# Optimization for multiple responses

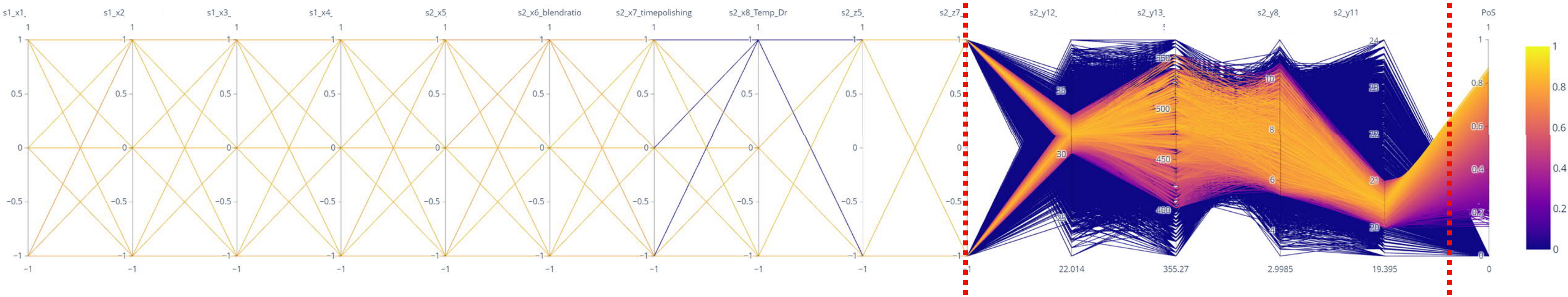
Plots with courtesy of José Nunez Ares

POS = probability of success to meet all customer specifications

Factor level settings

Customer specifications

POS



# Optimization for multiple responses

Plots with courtesy of José Nunez Ares

Factor level settings for the five process stetting conditions with the highest POS (best)

| s1_x1 | s1_x2 | s1_x3 | s1_x4 | s2_x5 | s2_x6 | s2_x7 | s2_x8 | s2_z5 | s2_z7 | s2_y8 | s2_y12 | s2_y13 | PoS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| -1 | -1 | 1 | -1 | -1 | -1 | 1 | -1 | -1 | -1 | | | | 0.87 |
| -1 | 1 | 1 | -1 | -1 | 1 | 1 | -1 | -1 | -1 | | | | 0.87 |
| 1 | -1 | -1 | -1 | 1 | 1 | -1 | -1 | 1 | -1 | | response data blinded | | 0.86 |
| -1 | -1 | -1 | -1 | -1 | -1 | 1 | -1 | -1 | -1 | | | | 0.86 |
| 1 | -1 | -1 | 1 | 1 | 1 | -1 | -1 | -1 | -1 | | | | 0.85 |

# Summary

- In a challenging case of product development,
  we found an efficient and powerful OMARS DOE
  that allowed us to combine screening and optimization steps into ONE,
  with a guarantee to get a clear analysis of the effects.
- In the analysis, we could study much more effects
  than in a more traditional DOE.
- The engineers could even learn about the effect of covariates
  that were not part of the design.

# Acknowledgements to

My colleagues

- Philipp Meier, PE
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- Mihir Khadilkar, data scientist

Partners in finding and analyzing OMARS designs

- José Nunez Ares, EFFEX
- Peter Goos, KU Leuven

# THANK YOU

*Together, improving life*