

Design of Experiments for Research and Development

Prepared for
Midwest SCC

by
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Outline

- ◆ What is DOE?
- ◆ Structure of Designed Experiments
- ◆ Examples
 - Factorial Experiments
 - Screening Studies
 - Optimization Studies
 - Mixture Experiments
- ◆ Summary

What is Design of Experiments?

- ◆ Design of Experiments (DOE) is:
 - an efficient, systematic approach
 - » Minimum number of runs to get an answer
 - » Resource commitment known in advance
 - » Controllable level of precision
 - to study the impacts of multiple, controllable factors
 - » Ingredient types and levels
 - » Process conditions
 - » Packaging characteristics
 - on key measures of product quality
 - » Objective: Physical/chemical/sensory
 - » Subjective: Consumer acceptance
 - » One-at-a-time or simultaneously

When to Use DOE?

- ◆ EARLY -- Screening Studies.
 - *So many variables, so little time.*
- ◆ MIDDLE -- Factorial Experiments.
 - *It's never that simple.*
- ◆ LATE -- Optimization Studies.
 - *Just tell me what's the best and why?*

Why Use DOE?

- ◆ Efficiency
 - Minimum number of samples to get the answer.
- ◆ Sensitivity
 - “Hidden Replications” add power at no extra cost.
- ◆ Robust Findings
 - Effects of each variable are assessed at multiple levels of all other variables.

DOE Examples

- ◆ Factorial Experiments
- ◆ Screening Studies
- ◆ Optimization Studies
- ◆ Mixture Experiments

Factorial Experiments

Objective

- ◆ **OBJECTIVE:** Assess the impact of four production variables on consumers' acceptance of a shampoo.
 - Silicone Type (Type A or Type B)
 - Silicone Level (0.1% or 2.0%)
 - Pearlizer (Without or With)
 - Polymer Level (0.1% vs. 1.0%)

Technical Approach

- ◆ A factorial experiment comprised of the 16 possible combinations of factor levels was developed to assess the impact of the four formula variables.
- ◆ 400 consumers evaluated four of the sixteen samples in a Home Use Test using a BIBD.

Sixteen Experimental Runs

Run	Silicone Type	Silicone Level	Pearlizer	Polymer Level	OVR Liking
1	B	2.0	Without	0.1	81
2	A	0.1	Without	0.1	58
3	B	0.1	Without	0.1	50
4	B	0.1	With	1.0	59
5	A	2.0	With	1.0	85
6	A	0.1	With	0.1	58
7	A	0.1	With	1.0	68
8	A	2.0	Without	0.1	90
9	A	0.1	Without	1.0	71
10	A	2.0	Without	1.0	91
11	B	0.1	With	0.1	51
12	B	2.0	With	1.0	76
13	B	2.0	Without	1.0	81
14	B	2.0	With	0.1	78
15	A	2.0	With	0.1	85
16	B	0.1	Without	1.0	62

Selecting Effects for the Model

- ◆ One option for fitting the factorial model is to use a probability plot to identify potentially significant effects.

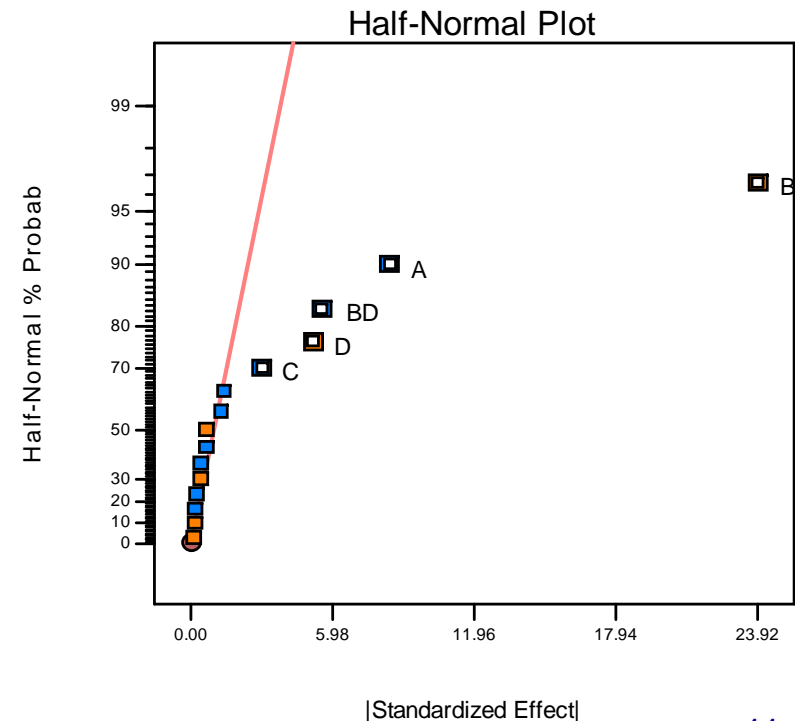
- ◆ Points that fall off the line, high and to the right are likely to be significant.

Design-Expert® Software
OVR Liking

Shapiro-Wilk test
W-value = 0.949
p-value = 0.654

A: Silicone Type
B: Silicone Level
C: Pearlizer
D: Polymer Level

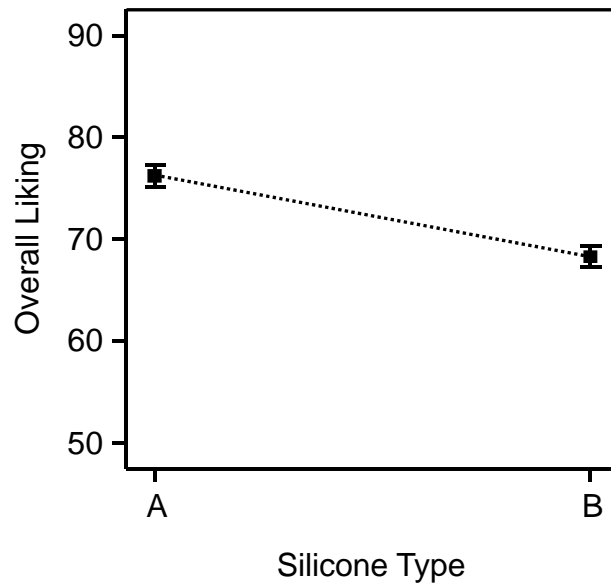
■ Positive Effects
■ Negative Effects



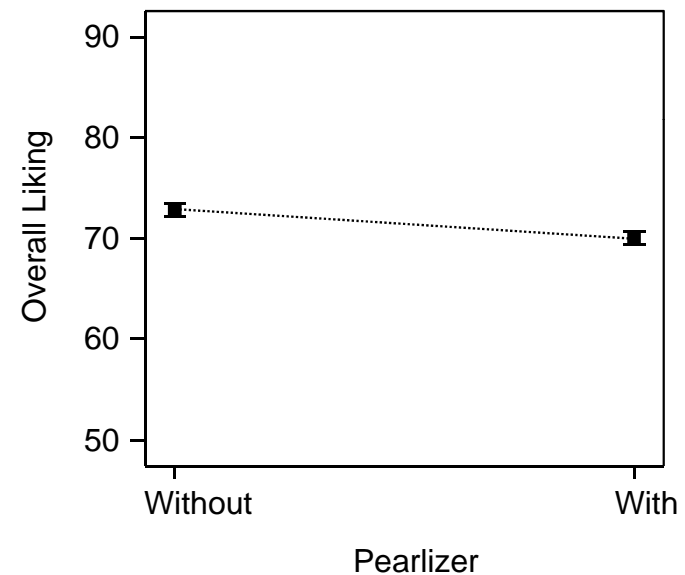
Analysis of Variance Confirms the Significance of the Effects

Source	df	Sum of Squares	Mean Square	F-Value	P-Value
Total	15	2852.36			
A-Silicone Type	1	281.25	281.25	147.5	< 0.0001
B-Silicone Level	1	2289.20	2289.20	1200.3	< 0.0001
C-Pearlizer	1	35.70	35.70	18.7	0.0015
D-Polymer Level	1	106.06	106.06	55.6	< 0.0001
BD	1	121.08	121.08	63.5	< 0.0001
Residual	10	19.07	1.91		

Significant Main Effects

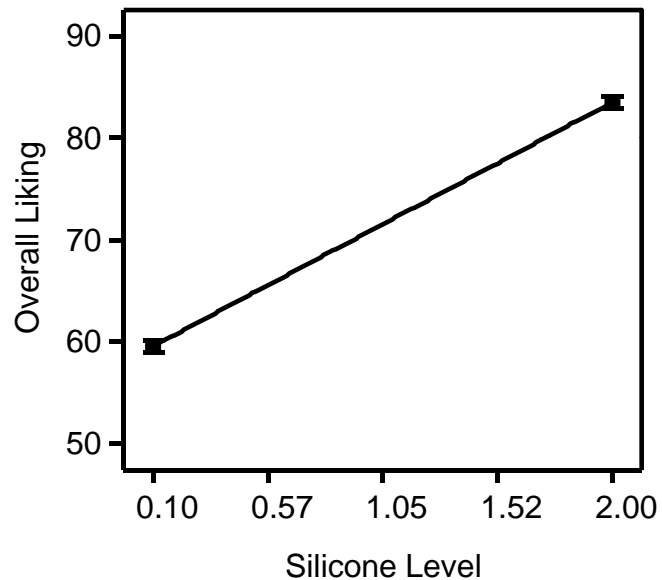


Silicone Type A is more well liked.

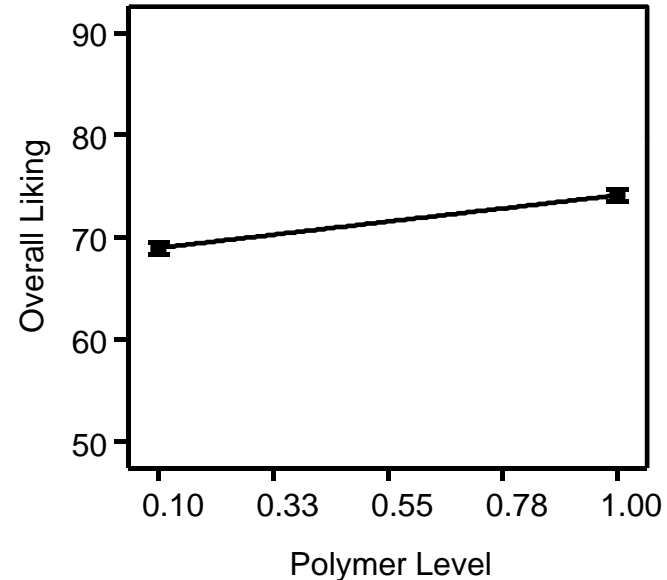


Without Pearlizer is more well liked (slightly).

Significant Main Effects



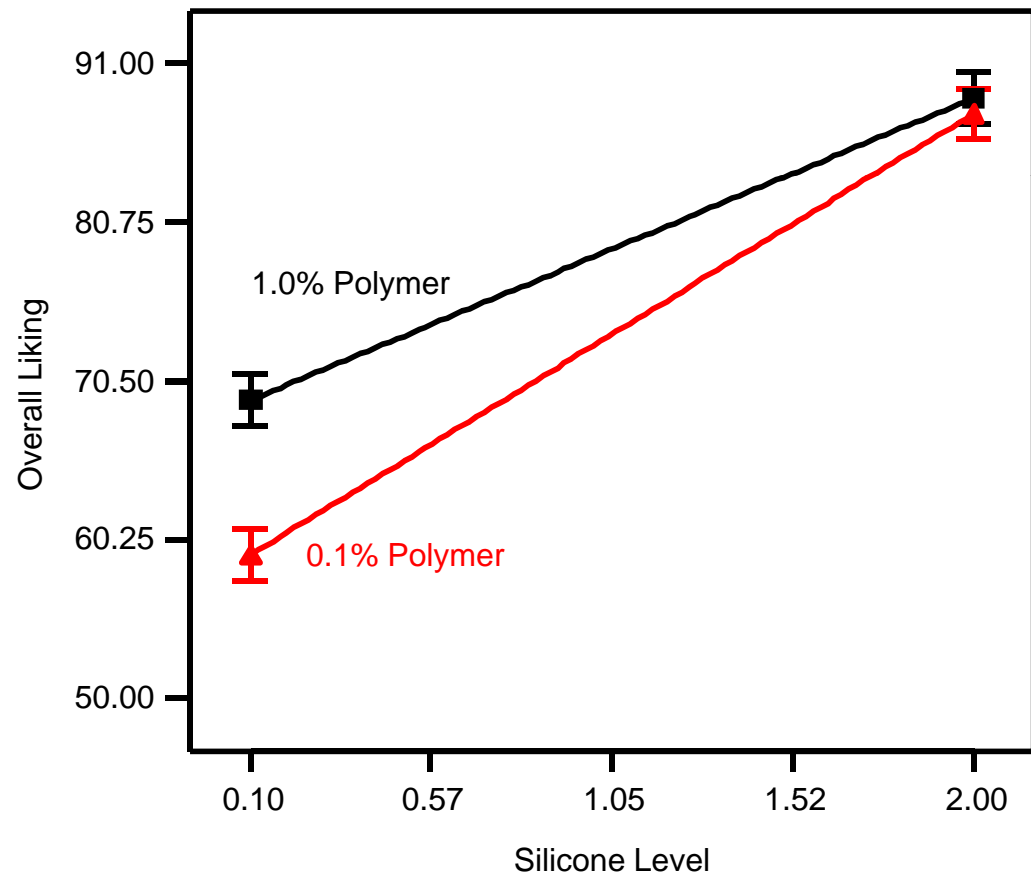
2.0% Silicone is more well liked than 0.1%.



1.0% Polymer is more well liked than 0.1%.

Significant Silicone-by-Polymer Interaction

- ◆ 2.0% Silicone is more well liked than 0.1% Silicone.
- ◆ At 2.0% silicone, polymer level does not matter.
- ◆ At 0.1% Silicone, 1.0% Polymer is more well liked than 0.1% Polymer



Results & Recommendations

- ◆ Most well liked shampoo is made at:
 - Silicone Type A
 - High Silicone Level (2.0 %),
 - Without Pearlizer, and
 - Either low or high Polymer (0.1% or 1.0%).
- ◆ Confirmatory study including both polymer levels should be conducted.
 - Why incur the added cost of 1.0% polymer if you do not need to?

Screening Experiments

Screening Experiments

- ◆ Early stages of research
- ◆ Many factors
- ◆ Unknown effects
- ◆ Looking for factors with big effects
- ◆ Save resources for fine-tuning experiments run later

How Screening Studies Save Runs

- ◆ Four-way, Five-way and Six-way interactions are almost always impossible to interpret.
- ◆ Why invest resources in your experimental designs to be able to estimate effects that you will not be able to understand?
- ◆ Run a specially selected subset of the full factorial to save resources but still be able to study the effects that you are interested in.

Main Effects and Interactions in a 2^6 Factorial Experiment

EFFECT	Number of Effects	Number of Runs Needed
Intercept	1	22
Main Effects	6	
2-way Interactions	15	
3-way Interactions	20	42
4-way Interactions	15	
5-way Interactions	6	
6-way Interactions	1	

Objective

- ◆ Assess the impact of six production variables on consumers' impressions of a sweet snack.
 - Diameter (Small vs. Large)
 - Mouthfeel Ingredient (0.0% vs. 0.4%)
 - Moisture (2.25% vs. 4.50%)
 - Particulates (0.0% vs. 0.5%)
 - Color (3.5% vs. 7.0%)
 - Mold Position (Open vs. Closed)

Technical Approach

- ◆ A statistically design variable-screening study comprised of eight experimental sample was developed to assess the impact of the six production variables
- ◆ 108 consumers evaluated all eight samples.
- ◆ Statistical analyses were conducted to measure the impact of each product variable on all consumer responses.

The Experimental Design

- ◆ For each factor, both levels are replicated four times.
- ◆ Within each level of one factor, the levels of the other factors are changing. The effect of a factor is not connected to an arbitrary set of initial conditions.

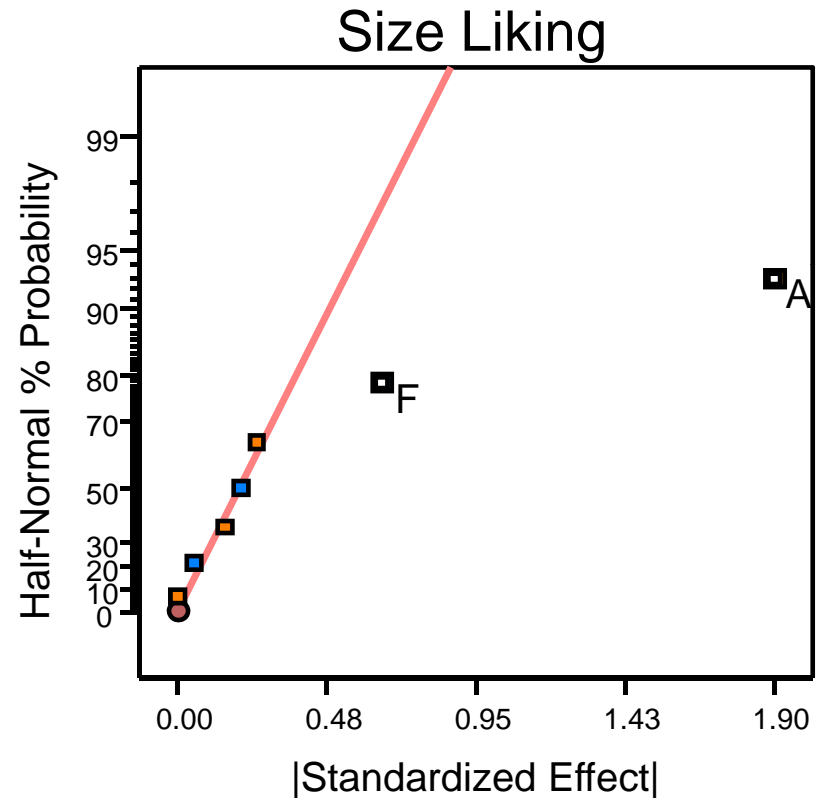
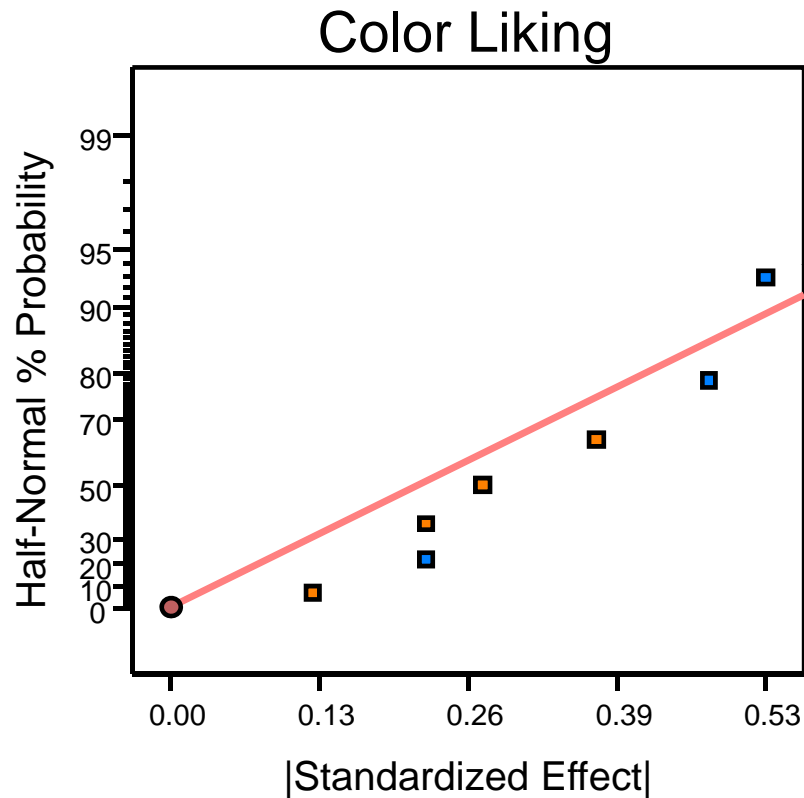
Run	Diameter	Mouthfeel Ingredient	Moisture	Particulates	Color	Mold Position
1	Small	0.4	2.25	0.0	7.0	Open
2	Small	0.0	2.25	0.5	7.0	Closed
3	Large	0.4	2.25	0.5	3.5	Open
4	Large	0.4	4.50	0.5	7.0	Closed
5	Small	0.4	4.50	0.0	3.5	Closed
6	Small	0.0	4.50	0.5	3.5	Open
7	Large	0.0	2.25	0.0	3.5	Closed
8	Large	0.0	4.50	0.0	7.0	Open

Alternative One-at-a-Time Study

- ◆ Requires one less run but provides only one comparison for each factor.
 - Screening Study provides four.
- ◆ Also, comparisons may be influenced by choice of the baseline conditions.

Run	Diameter	Mouthfeel Ingredient	Moisture	Particulates	Color	Mold Position
1	Small	0.4	2.25	0.0	7.0	Open
2	Large	0.4	2.25	0.0	7.0	Open
3	Small	0.0	2.25	0.0	7.0	Open
4	Small	0.4	4.50	0.0	7.0	Open
5	Small	0.4	2.25	0.5	7.0	Open
6	Small	0.4	2.25	0.0	3.5	Open
7	Small	0.4	2.25	0.0	7.0	Closed

Analysis is Simple and Direct



Some responses clearly
have no significant effects.

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Other responses clearly
have significant effects.

Results - Impact of Factors

Response	Diameter	Mouthfeel Ingredient	Moisture	Parti- culates	Color	Mold Position
Acceptance						
Color						
Size	+					-
Thickness	+					-
APP Coating						
OVR Appear	+					-
OVR Liking	-		-			-
PI	-					-
Flavor						
Mouthfeel	-					-
Crispness			-			-
Crunchiness			-			-
Intensity						
Color	+				+	
Size	+					-
Thick	-					-
Amt Coating					+	
Fruity Flavor						
Creaminess						
Crispness	+					+
Crunchiness	+					+

Results

- ◆ Mold Position had greatest impact.
 - Closed is superior to open.
 - » More well liked overall.
 - » More well liked in key attributes.
- ◆ Diameter and Finished Moisture also important.
 - Larger diameter more well liked for size, thickness and overall appearance.
 - Smaller diameter more well liked overall, for purchase intent and mouthfeel.
 - Low finished moisture more well liked for crispness, crunchiness and overall.
- ◆ Mouthfeel Ingredient, Particulates and Color are not important.

Optimization Designs

Optimization Studies

- ◆ Response Surface Methodology (RSM).
 - Designed regression analysis.
 - Built on simple factorial experiments.
 - Predict responses at points between those run in the study.
 - » The response surface.
- ◆ Mixture Experiments
 - An RSM study in which the levels of all variables have to sum to a constant.

RSM Example

◆ PROJECT OBJECTIVE

- Determine the formula for an orange flavored beverage with the current flavor system that has the greatest acceptability among consumers.

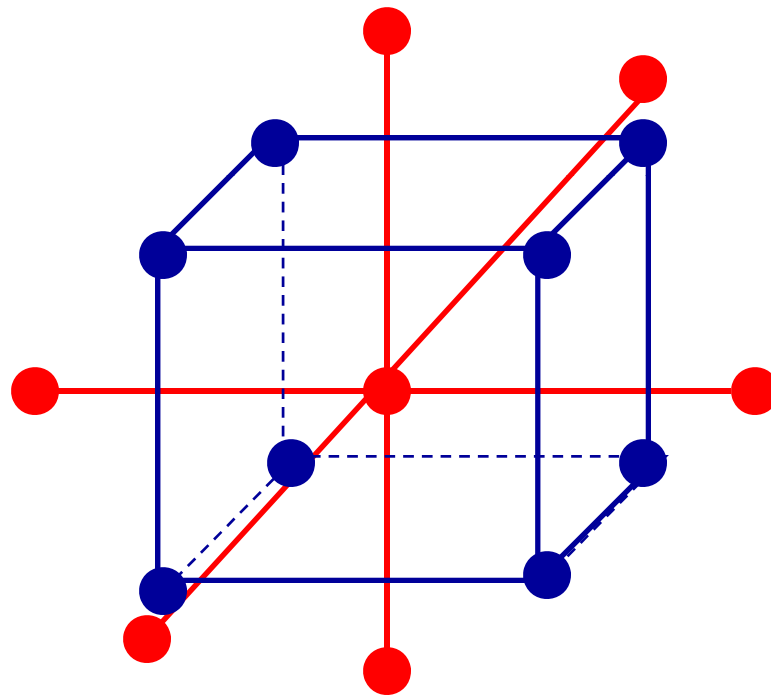
◆ RESEARCH OBJECTIVE

- Model the impact of sweetener, acid and flavor levels on the overall liking for the product in order to determine the levels that yield the most well liked product.

Approach

- ◆ Statistically Designed Study
 - Systematic changes to the levels of sweetener, acid and flavor.
 - 15 experimental samples.
- ◆ Designed Consumer Acceptance Test
 - Each respondent evaluates 3 of the 15 samples in a balanced incomplete block design (BIBD).
- ◆ Analysis
 - Link formula and acceptability.
 - Identify the most well liked formula.

Three-Factor RSM



Samples and Data

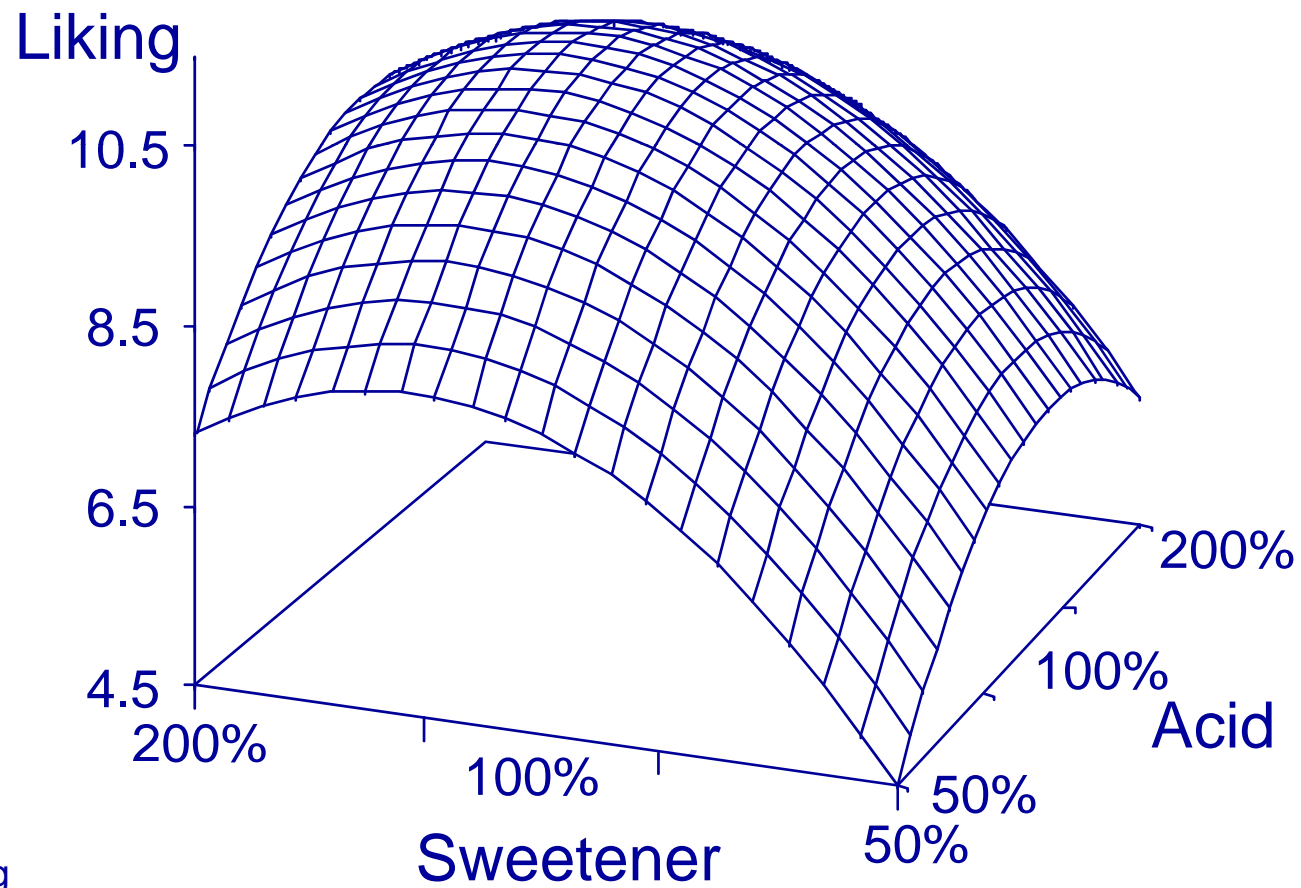
<u>Sweetener</u>	<u>Acid</u>	<u>Flavor</u>	<u>Liking</u>
66.2	66.2	66.2	7.2
66.2	66.2	151	7.0
66.2	151	66.2	8.5
66.2	151	151	8.1
151	66.2	66.2	9.2
151	66.2	151	9.4
151	151	66.2	9.8
151	151	151	10.9
50	100	100	7.4
200	100	100	8.7
100	50	100	8.0
100	200	100	8.7
100	100	50	10.4
100	100	200	9.9
100	100	100	9.3

RSM Model

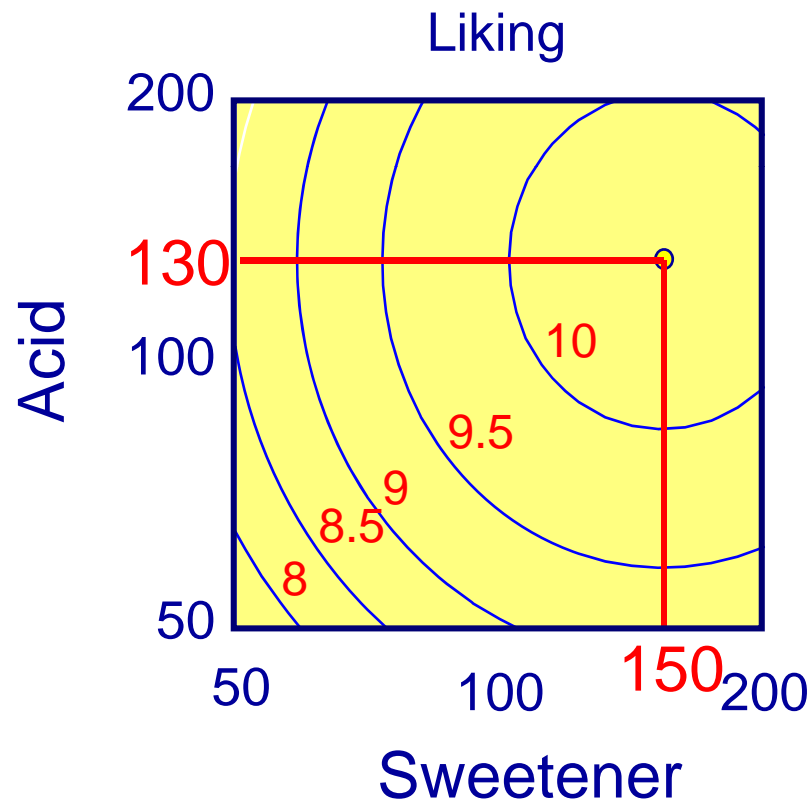
◆ The Link between Formula and Acceptance

$$\begin{aligned} \text{Liking} = & 9.89 + 0.78 \left\{ \frac{\text{LOG (Swt) - 2}}{0.3} \right\} - 0.63 \left\{ \frac{\text{LOG (Swt) - 2}}{0.3} \right\}^2 \\ & + 0.42 \left\{ \frac{\text{LOG (Acid) - 2}}{0.3} \right\} - 0.53 \left\{ \frac{\text{LOG (Acid) - 2}}{0.3} \right\}^2 \end{aligned}$$

Response Surface



Contour Plot



Results

- ◆ Overall Liking is maximized with a 50% increase in sweetener and a 30% increase in acid.
- ◆ Flavor level has no significant impact on liking.

Summary

- ◆ Statistical DOE
 - Easy to use.
 - Applicable at all stages.
 - Applicable to all research.
 - Efficient, powerful & rich in information

Mixture Experiments

Mixture Experiment

- ◆ OBJECTIVE:

- Determine the relative proportions of three components in a blend that deliver a desired set of sensory properties.

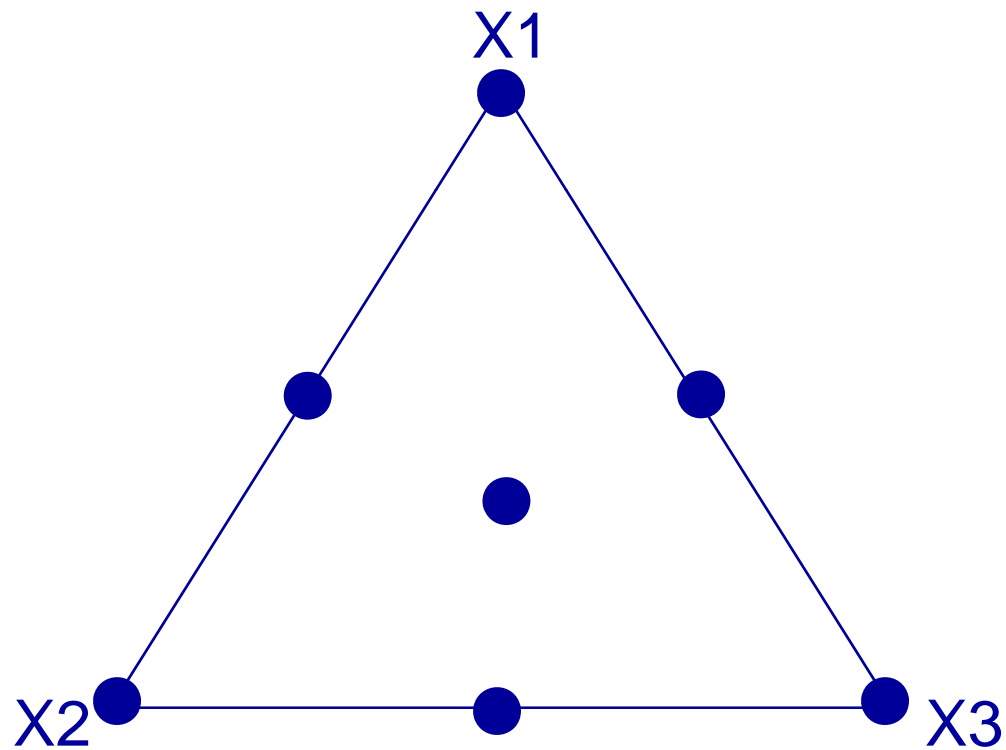
- ◆ RESEARCH OBJECTIVE:

- Model the sensory properties of the product as a function of its composition. Identify the region of blend ratios within which all action standards are satisfied simultaneously.

Approach

- ◆ Seven experimental samples:
 - Three “Pure Blends”.
 - Three 50:50 Blends.
 - One 33:33:33 Blend.
- ◆ Evaluated by 175 assessors using a 7-Pick-4 BIBD (yields 100 evaluations/sample).
- ◆ Sensory responses modeled using proportions of the components as predictors.

Three Component Mixture Design



Samples and Data

X1	X2	X3	Liking
100	0	0	5.9
0	100	0	4.7
0	0	100	4.2
50	50	0	5.8
50	0	50	4.9
0	50	50	5.6
33	33	33	5.5

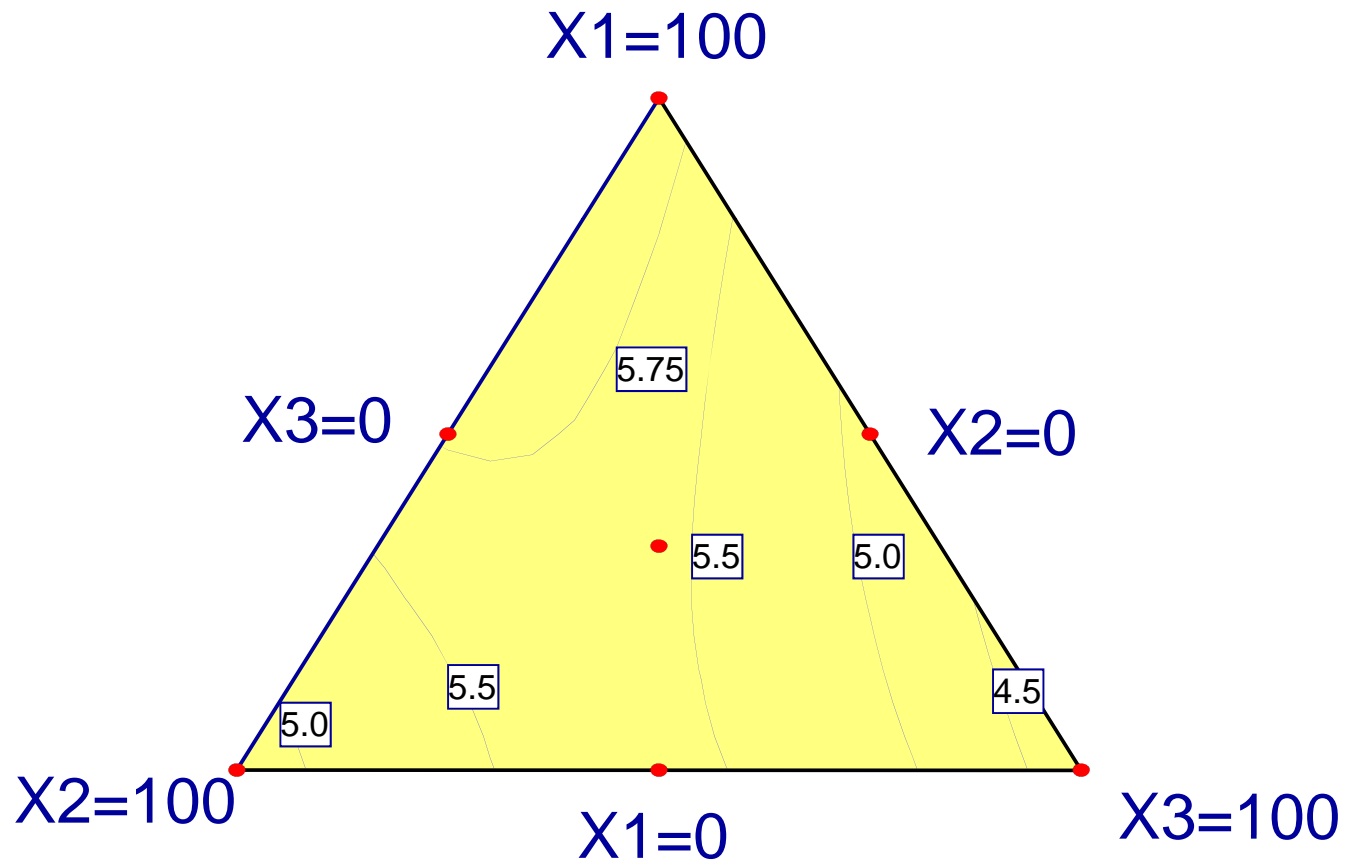
Mixture Model

Predictive Model Relating Overall Liking to Blend

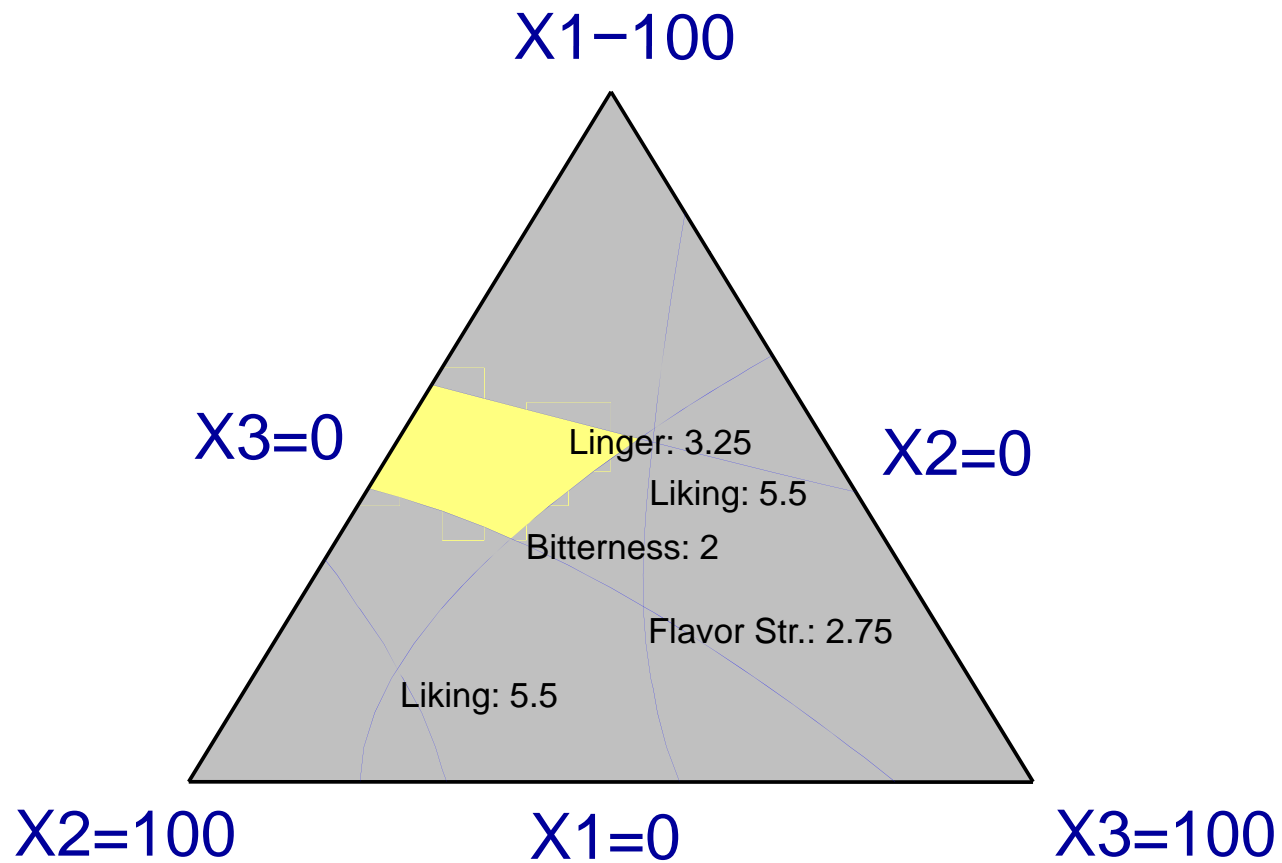
$$\begin{aligned}\text{Liking} &= 5.9*X1 \\ &+ 4.7*X2 \\ &+ 4.2*X3 \\ &+ 1.9*X1*X2 \\ &- 0.7*X1*X3 \\ &+ 4.5*X2*X3\end{aligned}$$

$$R^2=0.98$$

Contour Plot of Overall Liking



Optimized Blend Region



Summary

- ◆ Statistical DOE
 - Easy to use.
 - Applicable at all stages.
 - Applicable to all research.
 - Efficient, powerful & rich in information