

DESIGN OF EXPERIMENT (DOE) & RESPONSE SURFACE METHODOLOGY (RSM)

Present by:

Wan Nor Nadyaini Wan Omar, B.Eng (Chem), M.Eng (Chem)

Faculty of Chemical And Energy Engineering

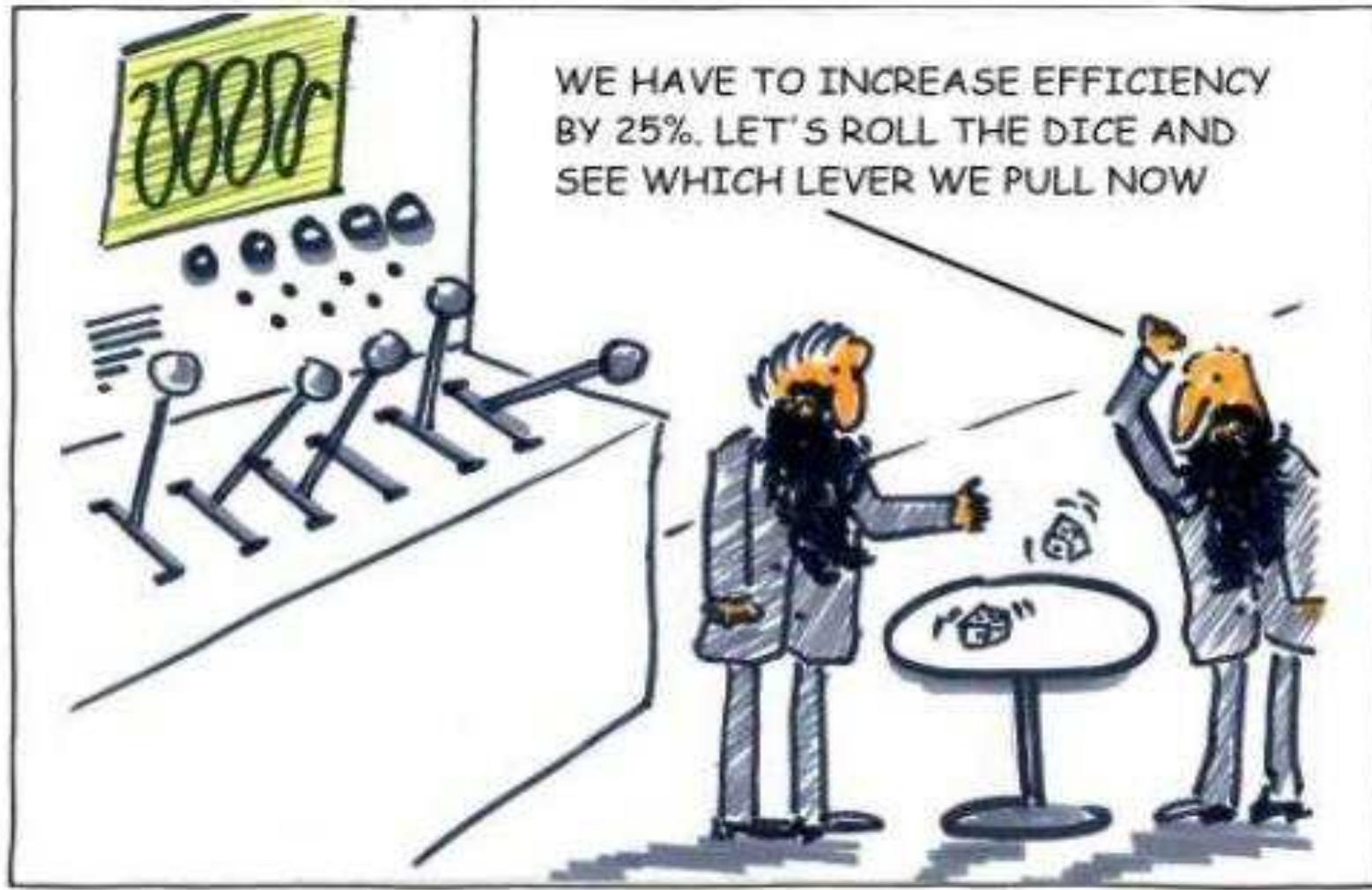
(wnnadyaini@gmail.com)

Date: 3 MARCH 2016

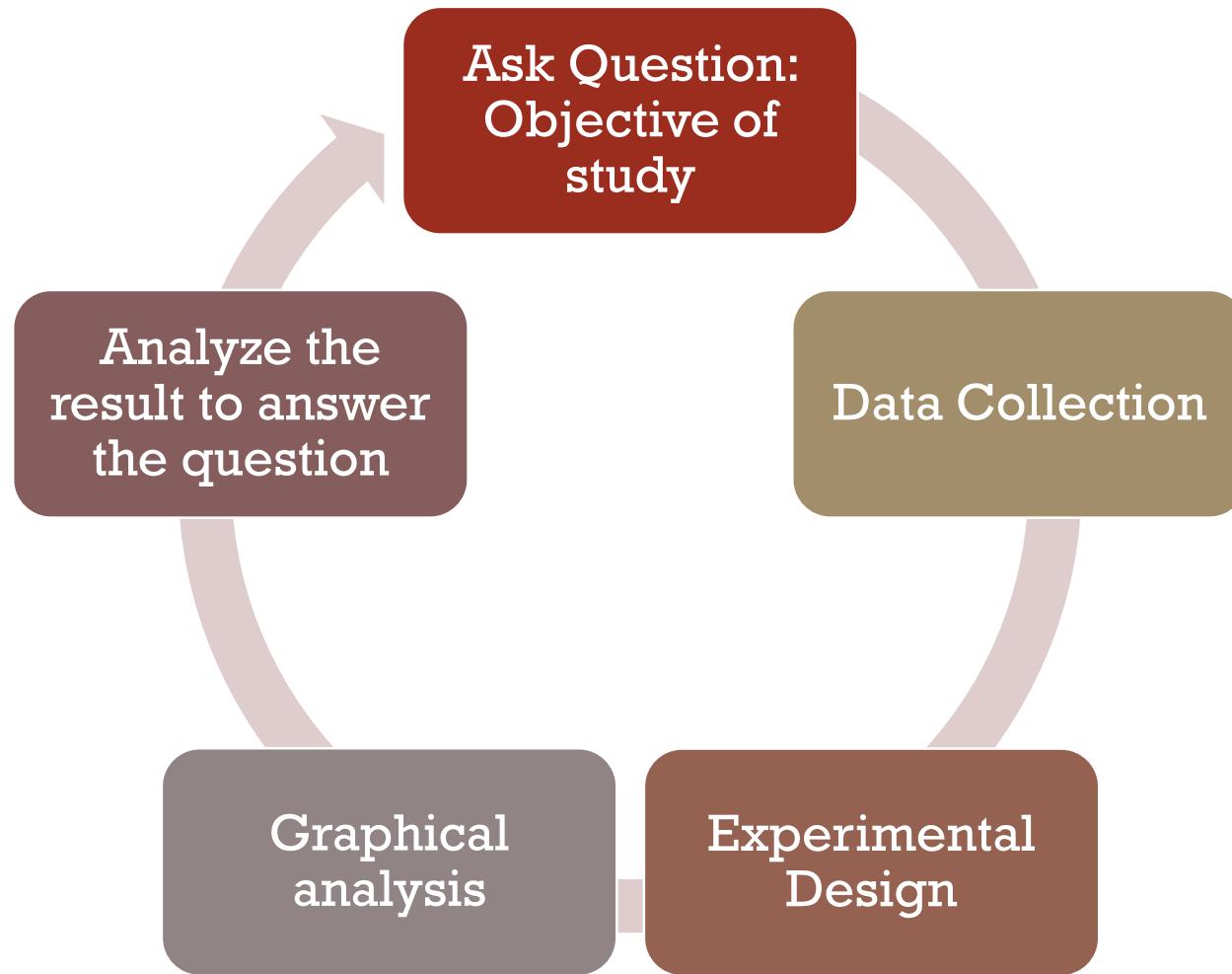
Place: n16, CREG MEETING ROOM, Faculty of Chemical AND ENERGY EngineeriNG



IF??



RESEARCH CYCLE PROCESS



DATA COLLECTION

To clarify the objective of experiment

The experimenter must determine

- What data to be collected?
- How to measure it?
- How the data relates to process performances and experimental objective?

The experimenter must ensure the data collected is represented the process

- Thus, the data could lead to correct conclusion

The experimental design must related to experimental objectives

EXPERIMENTAL DESIGN

Conventional Method

- One factor at a time (OFAT)
- Time consuming
- Cannot interpret the interaction between 2 or more variables

Statistical method

- Known as Design of Experiment (DOE)
- Apply factorial concept
- Use the modelling to predict the behavior of process variables
- RSM, ANN etc.
- Could explain the interaction between the process variables
- Reduce lead time and improve efficiency



What is DOE?

A collection of predetermined process variables setting

What is RSM?

Response Surface Methodology (RSM) is a **statistic techniques employed a regression analysis** to performed for the collective data.

What is STATISTICA, Design Expert, MiniTab and etc?

- **is a tool** to help we designs our experiment and analyses our data.
- RSM is one of the technique that have been programmed in that software.

WHAT DOE & RSM CAN DO?

PREDICTION

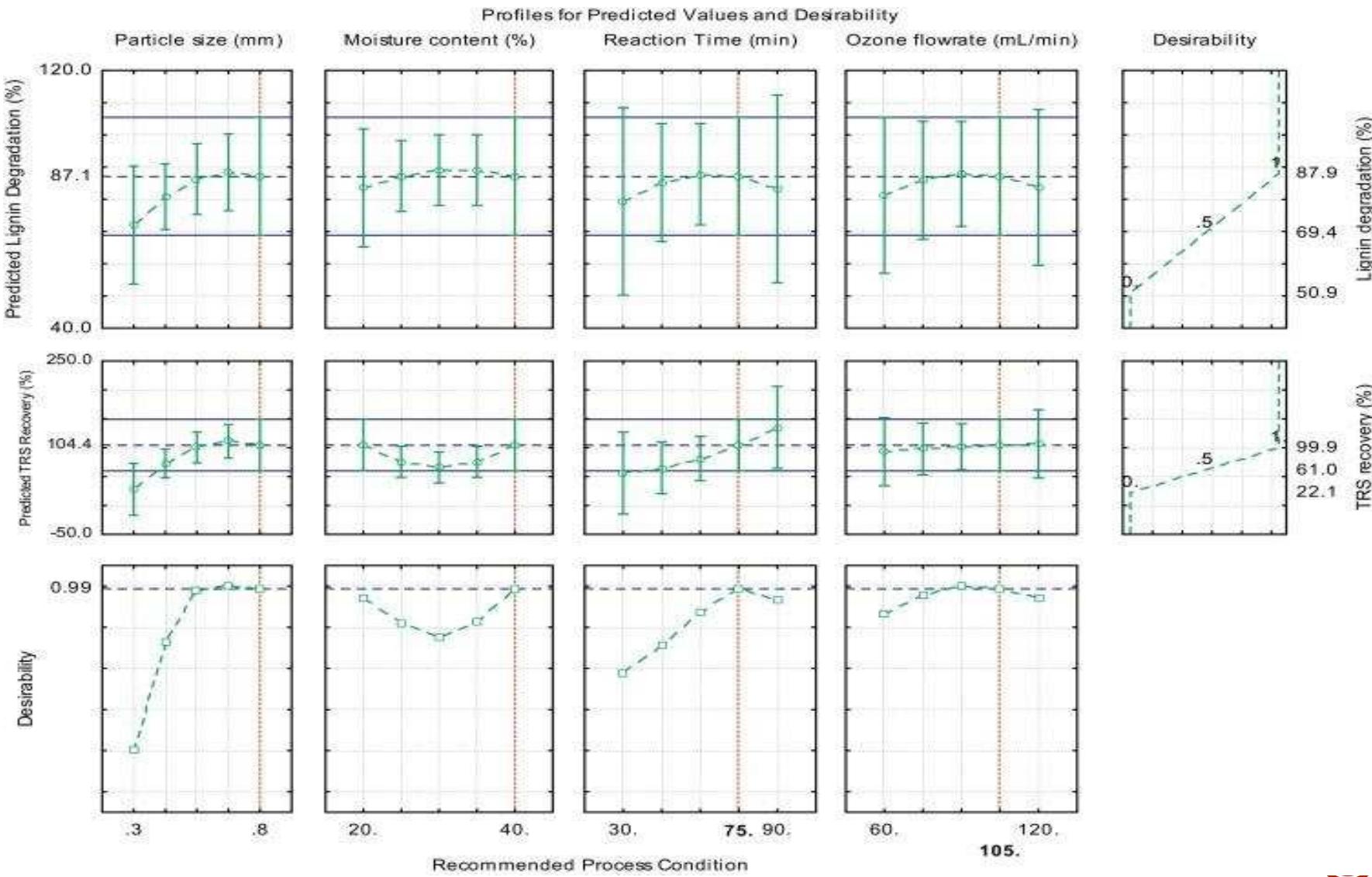
- Could predict the relationship/interaction between the values of some measureable response variable(s) and those of a set of experimental factors presumed to affect the response(s)
- Predict the response value at various process condition

OPTIMIZATION

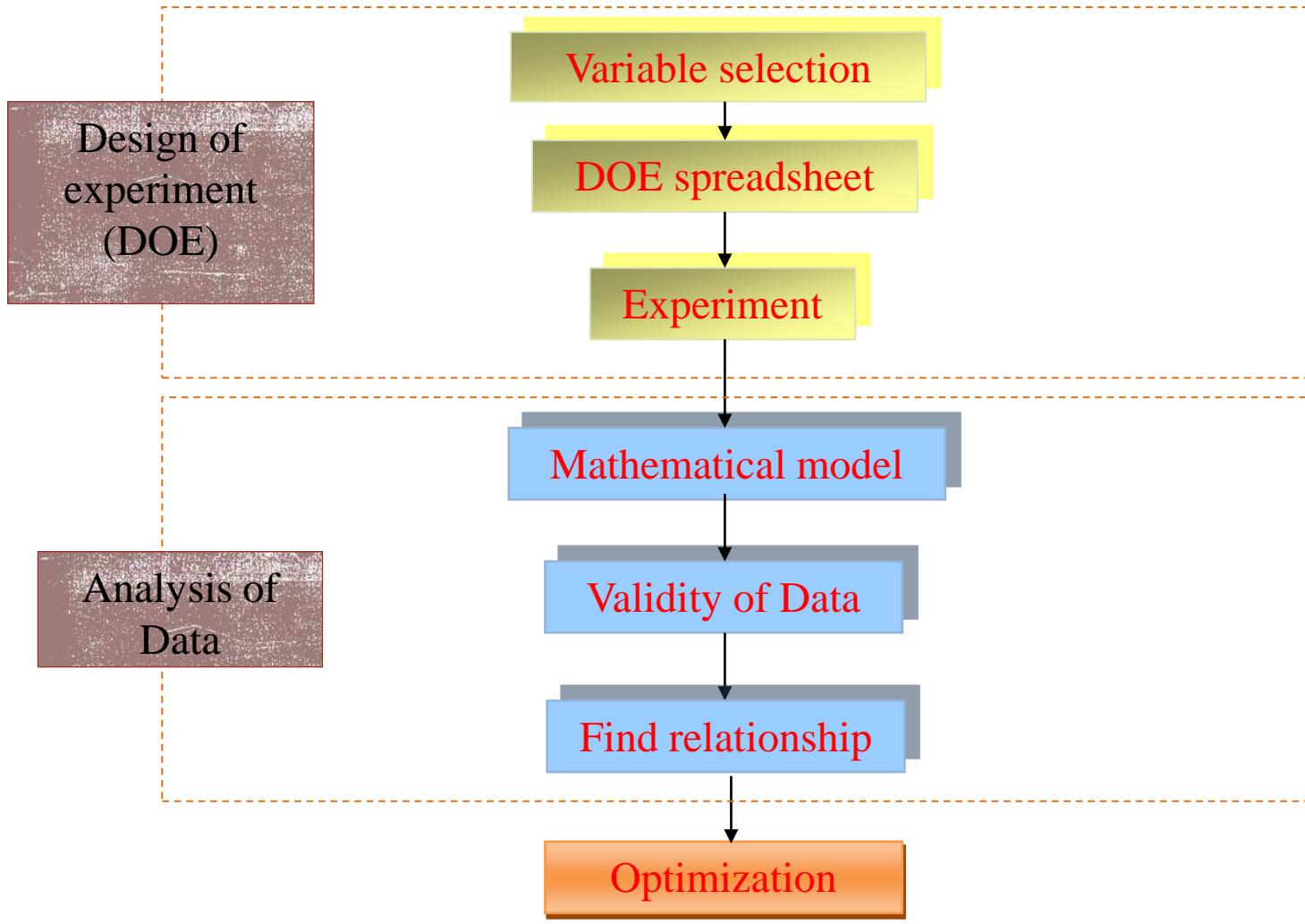
- Could find the values of the factors that produces the best value or values of the response(s).



EXAMPLE



FLOW OF RSM STUDY



STEP IN RSM STUDY

Before: Select the
variable-Design the
experiment



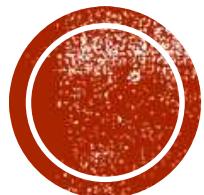
After: Analyze the
data.



During: The actual
experiment will be
carried out



BEFORE EXPERIMENT



1. Selecting the process variables
2. Selecting the level and range for each process variable
3. Selecting the design of experiment (DOE)

PREPARING FOR RSM STUDY

RESPONSE

- What response variables are to be measured, how they will be measured, and in what sequence?

PROCESS VARIABLE

- Which factor are most important and therefore will be included in the experiment, and which are least important and can these factors be omitted? With the important factors, can the desired effects be detected?

DISTURBANCE

- What extraneous or disturbing factors must be controlled or at least have their effects minimized?

EXPERIMENTAL UNIT

- What is the experimental unit, that is to say, what is the piece of experimental material from which a response value is measured? How are the experimental units to be replicated, if at all?

DESICION

- The choice of the factors and level determined the type, size and experimental region. The no. of levels at each factor as well as the no. of replicated experiment units represent the total no. of experiments.

DESIGN OF EXPERIMENT (DOE) PROCESS

Objective

- Screening
- Prediction
- Optimization

Factor

- No of Independent Var.
- Block
- Level
- Range

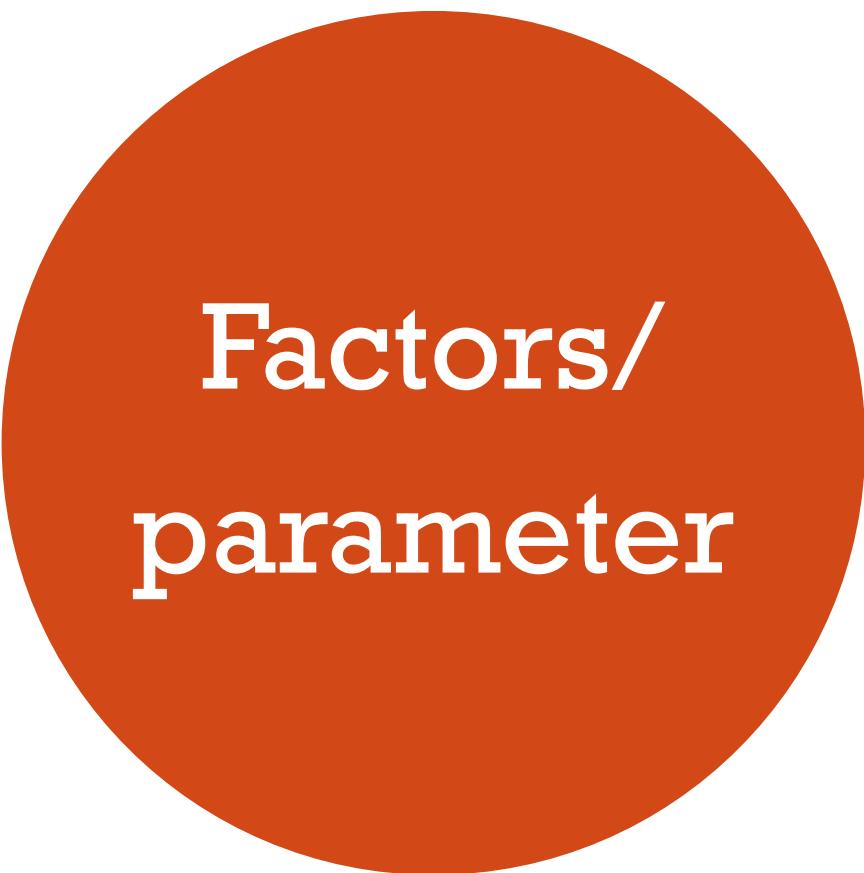
Type of design

- Full factorial
- Fractional factorial
- Placket Burman
- CCD
- Box-behnken
- Taguchi
- Etc.

OBJECTIVES OF STUDY

Screening	Optimization
<ul style="list-style-type: none">• To identify the significant main effect of factors from a list of many potential ones• Not identified the interaction effect• Type of design: 2-level with resolution III or IV, fractional factorial, Plackett-Burman	<ul style="list-style-type: none">• To identify the best process performance, interaction effect, and significant of factors• Type of design: CCD or BBD

1) SELECTING THE PARAMETER



Process conditions that would give an effect on the value of response variable

How to decide?

- Previous Experiment
- Previous experience
- Literature

Blocking parameter

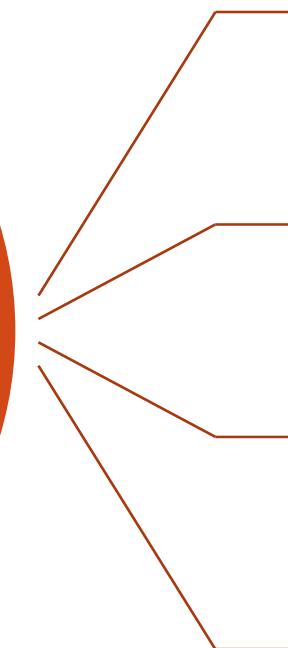
- Qualitative (e.g. type of biomass, type of chemical, type of gas, machine etc)
- Quantitative but in group (e.g. particle size, pH, etc)

Independent parameter

- normally considered in RSM
- E.g. Temp, pressure, flowrate, mass loading etc.



1) SELECTING THE PARAMETER



The measurable quantity whose value is assumed to be affected by changing the levels of the factors and most interested in optimizing.

How much the response will be considered?

Thus, the selected response is affecting with each other

e.g. yield, conversion, selectivity, consumption



2) SELECTING THE LEVEL

What is a level?

- The limits of the factor settings available to run the process.

Why is the level is important?

- To identify region of study
- Because of the optimum condition could be anywhere in the study

How to determine the level?

- Previous experiment
- Previous experience
- Literature



2) SELECTING THE LEVEL

Two level (2^k) - (-1,+1)-first order,

- Two-level factorial design is each factor is evaluated at a “low” setting and at “high” setting.

Three level (3^k) – (-1,0,+1) second or higher order

- Three-level factorial design is each factor is evaluated at a “low”, “center” and at “high” setting.

Five-level (5^k)-(- α , -1,0,+1, + α) second or higher order

- Five-level factorial design is each factor is evaluated at a “Star low”, “low”, “center”, “high” and “star high” setting.



EXAMPLE

- CASE A: In biodiesel production, selected parameter is temperature

Parameter	Coded	Low level, -1	High level, +1
Temperature (°C)	X	70	100

- How to find the center?
- Center, $0 = (\text{low} + \text{high})/2 = (70+100)/2=85$



EXPERIMENTAL REGION

- The region of conceivable factor level values that represents the factor combinations of potential interest.
- Need to determined before the experiment by finding the range of variables.
- If at the end of analysis, the factor value or optimum is out of the range, the experiment need to repeat with the new range.

Factors	Symbol	Range and Levels		
		-1	0	+1
Molar ratio methanol: oil	X ₁	20:1	30:1	40:1
Catalyst loading, wt%	X ₂	2	3	4
Reaction Time, min	X ₃	120	180	240
Reaction Temperature	X ₄	90	120	150



3) SELECTING THE TYPE OF DOE

- Full factorial
- Fractional factorial
- Placket Burman
- CCD
- Box-behnken
- Taguchi
- Etc.

The most popular is CCD and box-behnken design

What are different between CCD and box-behnken design???



FACTORIAL DESIGN

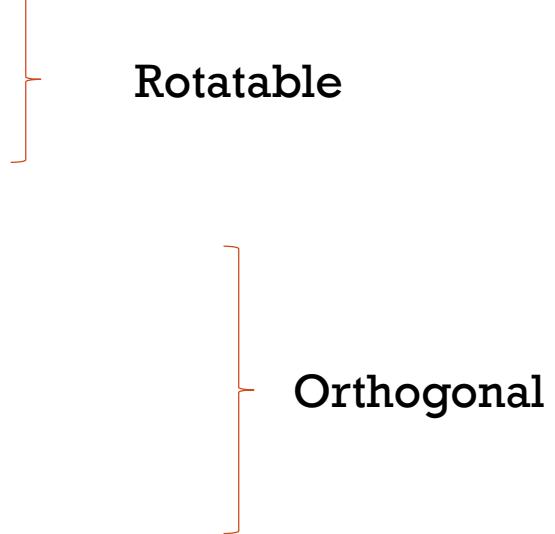
Easy to be used by simply following relatively simple design

Able to meet the majority of the experimental needs and its data analysis can be performed by graphical methods

Require relatively few runs at a reasonable size

If large number of factors is selected, the fractional factorial design can be employed to keep the experimental run at a reasonable size

FULL FACTORIAL & FRACTIONAL FACTORIAL

- Two level Full factorial $(-1,+1) = 2^k$
 - Three level full factorial $(-1,0,+1) = 3^k$
 - Fractional factorial (two level) $= 2^{k-m}$, $m < k$
 - $\frac{1}{2} = 2^{k-1}$
 - $\frac{1}{4} = 2^{k-2}$
 - $\frac{1}{8} = 2^{k-4}$
 - Fractional factorial (three level) $= 3^{k-m}$, $m < k$
- 



BLOCKING ARRANGEMENT, 2-LEVEL

2-level fractional factorial design
(resolution IV)

	A	B	C	D
1	-1	-1	-1	-1
2	1	-1	-1	1
3	-1	1	-1	1
4	1	1	-1	-1
5	-1	-1	1	1
6	1	-1	1	-1
7	-1	1	1	-1
8	1	1	1	1

2-level factorial design (full)

	A	B	C	D
1	-1	-1	-1	-1
2	1	-1	-1	-1
3	-1	1	-1	-1
4	1	1	-1	-1
5	-1	-1	1	-1
6	1	-1	1	-1
7	-1	1	1	-1
8	1	1	1	-1
9	-1	-1	-1	1
10	1	-1	-1	1
11	-1	1	-1	1
12	1	1	-1	1
13	-1	-1	1	1
14	1	-1	1	1
15	-1	1	1	1
16	1	1	1	1



BLOCKING ARRANGEMENT, 3-LEVEL

3***(4-0) full factorial design, 1 block , 81 runs (Spreadsheet1)

	A	B	C	D
1	-1	-1	-1	-1
2	-1	-1	-1	0
3	-1	-1	-1	1
4	-1	-1	0	-1
5	-1	-1	0	0
6	-1	-1	0	1
7	-1	-1	1	-1
8	-1	-1	1	0
67	1	0	0	-1
68	1	0	0	0
69	1	0	0	1
70	1	0	1	-1
71	1	0	1	0
72	1	0	1	1
73	1	1	-1	-1
74	1	1	-1	0
75	1	1	-1	1
76	1	1	0	-1
77	1	1	0	0
78	1	1	0	1
79	1	1	1	-1
80	1	1	1	0
81	1	1	1	1

3***(4-1) fractional factorial design, 9 blocks, 27 runs (Spreadsheet1)

Bloc k	A	B	C	D
1	1	0	1	0
2	1	1	0	1
5	2	1	0	0
6	2	0	1	-1
7	3	1	0	-1
8	3	-1	-1	0
11	4	-1	0	-1
12	4	1	1	1
13	5	-1	0	1
14	5	1	1	0
15	5	0	-1	-1
16	6	1	1	-1
17	6	0	-1	1
18	6	-1	0	0
19	7	-1	1	-1
20	7	0	0	0
21	7	1	-1	1
22	8	0	0	-1
23	8	1	-1	0
24	8	-1	1	1
25	9	0	0	1
26	9	1	-1	-1
27	9	-1	1	0

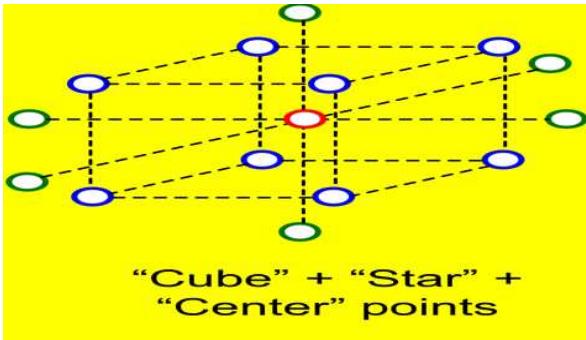
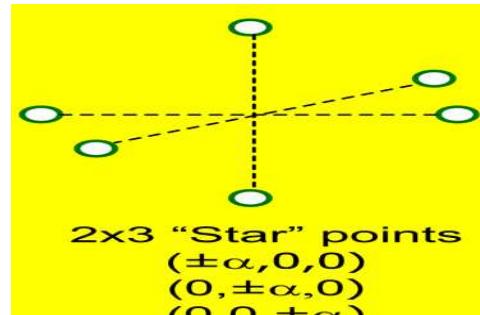
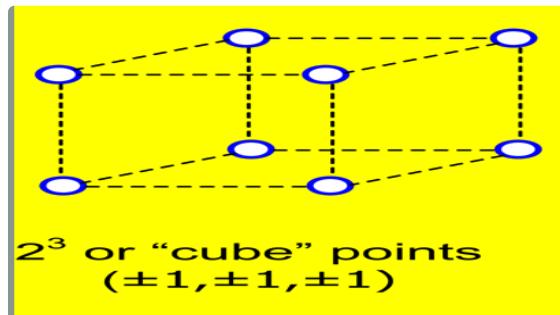
3***(4-1) fractional factorial design, 3 blocks, 27 runs (Spreadsheet1)

Block	A	B	C	D
1	1	1	0	-1
2	1	0	1	0
3	-1	-1	1	0
4	-1	0	-1	1
5	-1	0	0	0
6	-1	0	1	-1
7	-1	1	-1	0
8	-1	1	0	-1
9	-1	1	1	1
10	0	-1	-1	1
11	0	-1	0	0
12	0	-1	1	-1
13	2	0	-1	1
14	2	0	-1	0
15	2	0	-1	1
16	2	1	1	0
17	2	-1	0	-1
18	2	1	1	1
19	3	0	0	1
20	3	0	0	0
21	3	-1	1	-1
22	3	0	0	-1
23	3	1	-1	1
24	3	1	-1	0
25	3	1	-1	0
26	3	-1	1	1
27	3	-1	1	0

3***(4-1) fractional factorial design, 1 block , 27 runs (Spreadsheet1)

	A	B	C	D
1	-1	-1	-1	-1
2	-1	-1	0	1
3	-1	-1	1	0
4	-1	0	-1	1
5	-1	0	0	0
6	-1	0	1	-1
7	-1	1	-1	0
8	-1	1	0	-1
9	-1	1	1	1
10	0	-1	-1	1
11	0	-1	0	0
12	0	-1	1	-1
13	0	1	1	0
14	1	-1	-1	0
15	1	-1	1	1
16	1	0	0	-1
17	1	0	1	0
18	1	1	1	-1
19	1	1	-1	0
20	1	-1	0	-1
21	1	-1	1	1
22	1	0	-1	-1
23	1	0	0	1
24	1	0	1	0
25	1	1	-1	1
26	1	1	0	0
27	1	1	1	-1

CENTRAL COMPOSITE DESIGN (CCD)



2^k vertices of a k -dimensional “cube” (2 -level full factorial design or 2^{k-m} fractional design) → coded as ± 1

$2k$ vertices of a k -dimensional “star”
→ coded as $\pm \alpha$

$n_0 \geq 1$ “center” point replicates → coded as 0



Providing the estimate of pure error and curvature



$$\text{Total run} = 2^k + 2k + n_0 \text{ or } 2^{k-m} + 2k + n_0$$



CCD

The most common design (for the 2nd degree model)

Can be orthogonal or rotatable design

- **orthogonal:** the term of model have to redefined
 - : normally used if the blocking variable is considered.
- **Rotatable:** related to the precision of the predicted value
 - : archieved by selecting appropriate values for n_0 (>0) and $\alpha=4\sqrt{M}$, $M=2^k$

BOX-BEHNKEN DESIGN

The equivalent in the case of $3^{(k-p)}$ designs (3-level full factorial with incomplete block) are the so-called Box-Behnken designs (Box and Behnken, 1960).

These designs do not have simple design generators (they are constructed by combining two-level factorial designs with incomplete block designs), and have complex confounding of interaction.

However, the designs are economical and therefore particularly useful when it is expensive to perform the necessary experimental runs.

DOE BLOCKING ARRANGEMENT

-1.00	-1.00	-1.00	-1.00
-1.00	-1.00	-1.00	1.00
-1.00	-1.00	1.00	-1.00
-1.00	-1.00	1.00	1.00
-1.00	1.00	-1.00	-1.00
-1.00	1.00	-1.00	1.00
-1.00	1.00	1.00	-1.00
-1.00	1.00	1.00	1.00
1.00	-1.00	-1.00	-1.00
1.00	-1.00	-1.00	1.00
1.00	-1.00	1.00	-1.00
1.00	-1.00	1.00	1.00
1.00	1.00	-1.00	-1.00
1.00	1.00	-1.00	1.00
1.00	1.00	1.00	-1.00
1.00	1.00	1.00	1.00
-2.00	0.00	0.00	0.00
2.00	0.00	0.00	0.00
0.00	-2.00	0.00	0.00
0.00	2.00	0.00	0.00
0.00	0.00	-2.00	0.00
0.00	0.00	2.00	0.00
0.00	0.00	0.00	-2.00
0.00	0.00	0.00	2.00
0.00	0.00	0.00	0.00

CCD, 26 run

-1.00	-1.00	-1.00	-1.00
-1.00	-1.00	0.00	1.00
-1.00	1.00	1.00	0.00
-1.00	0.00	-1.00	1.00
-1.00	0.00	0.00	0.00
-1.00	0.00	1.00	-1.00
-1.00	1.00	-1.00	0.00
-1.00	1.00	0.00	-1.00
-1.00	1.00	1.00	1.00
0.00	-1.00	-1.00	1.00
0.00	-1.00	0.00	0.00
0.00	-1.00	1.00	-1.00
0.00	0.00	-1.00	0.00
0.00	0.00	0.00	-1.00
0.00	0.00	1.00	1.00
0.00	1.00	0.00	1.00
0.00	1.00	-1.00	0.00
0.00	1.00	1.00	-1.00
1.00	-1.00	-1.00	0.00
1.00	-1.00	0.00	-1.00
1.00	-1.00	1.00	1.00
1.00	0.00	-1.00	-1.00
1.00	0.00	0.00	1.00
1.00	0.00	1.00	0.00
1.00	1.00	-1.00	1.00
1.00	1.00	0.00	0.00
1.00	1.00	1.00	-1.00

3 fractional factorial, 27 run

-1.00	-1.00	0.00	0.00
1.00	-1.00	0.00	0.00
-1.00	1.00	0.00	0.00
1.00	1.00	0.00	0.00
0.00	0.00	-1.00	-1.00
0.00	0.00	1.00	-1.00
0.00	0.00	-1.00	1.00
0.00	0.00	1.00	1.00
0.00	0.00	0.00	0.00
-1.00	0.00	0.00	-1.00
1.00	0.00	0.00	-1.00
-1.00	0.00	0.00	1.00
1.00	0.00	0.00	1.00
0.00	-1.00	0.00	0.00
0.00	1.00	0.00	0.00
0.00	-1.00	-1.00	0.00
0.00	1.00	-1.00	0.00
0.00	-1.00	1.00	0.00
0.00	1.00	1.00	0.00
0.00	0.00	0.00	0.00
-1.00	0.00	0.00	0.00
1.00	0.00	0.00	0.00
-1.00	0.00	0.00	0.00
1.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00

BBD, 27 run

CCD VS BBD

Criteria	CCD	BBD
Design	2-level factorial, with star point	3-level fractional factorial,
Block	Up to researcher	Limited
Mean effect	Not considered	Considered
Interaction	Linear	Linear, quadratic
Optimization	Yes	Yes



DOE TABLE

Run Manipulated Variables		Response				
	X ₁	X ₂	X ₃			
	Operating temperature,T(°C)	Level ^b	Molar Ratio (meOH: oil)	Level Reaction time,t (h)	Level ^b	Yield, Y ₁ (%)
1	50	-1	3	-1	2	-1
2	50	-1	3	-1	4	+1
3	50	-1	10	+1	2	-1
4	50	-1	10	+1	4	+1
5	70	+1	3	-1	2	-1
6	70	+1	3	-1	4	+1
7	70	+1	10	+1	2	-1



ADDITION NOTE

Randomisation

- The order of run is random
- Can protect us from bias caused by unaware factors, and validates our analysis based on normal mode assumptions

Replication

- Repetition of experiments
- It is essential feature to increase the degree of belief

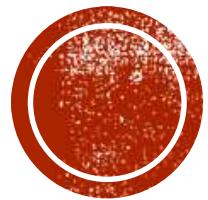
Blocking

- The experimental units are grouped into homogeneous clusterd in an attempt to improve the comparison of treatments with greater precision by randomly allocating the treatments withing each cluster or 'block'
- To detect the effect of treatment from background noise caused by non-homogeneous experimental unit.
- Can eliminate a source of variability from analysis

REMEMBERS

**There are none software
that can help you IF
your DOE is worst or
wrong.**

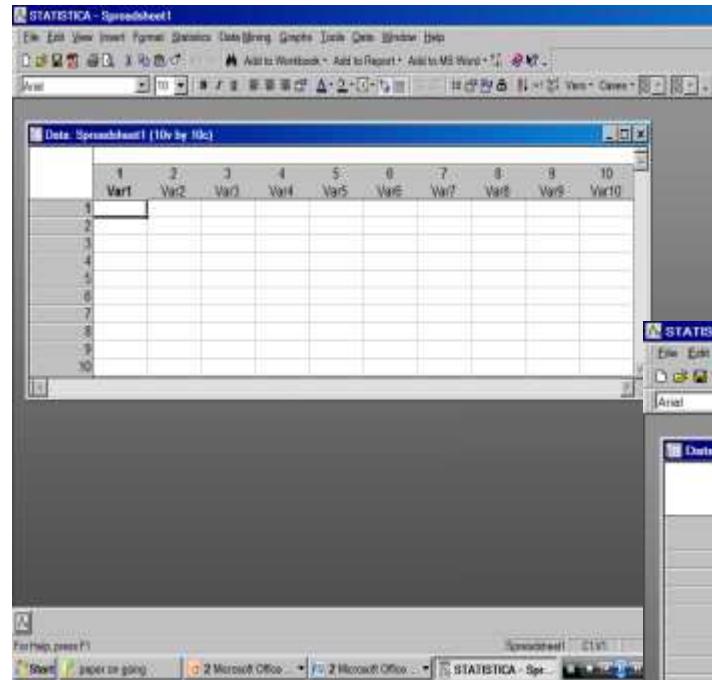




STATISTICA TUTORIAL 1

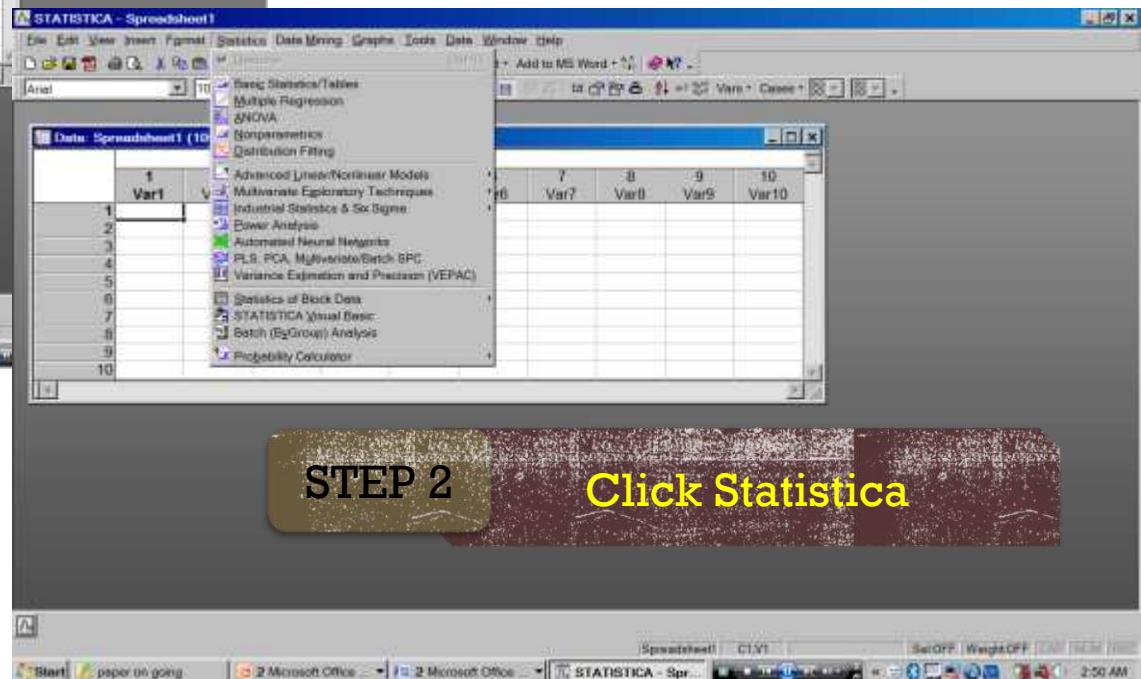
DESIGN OF EXPERIMENT

DOE SPREADSHEET



Open spreadsheet

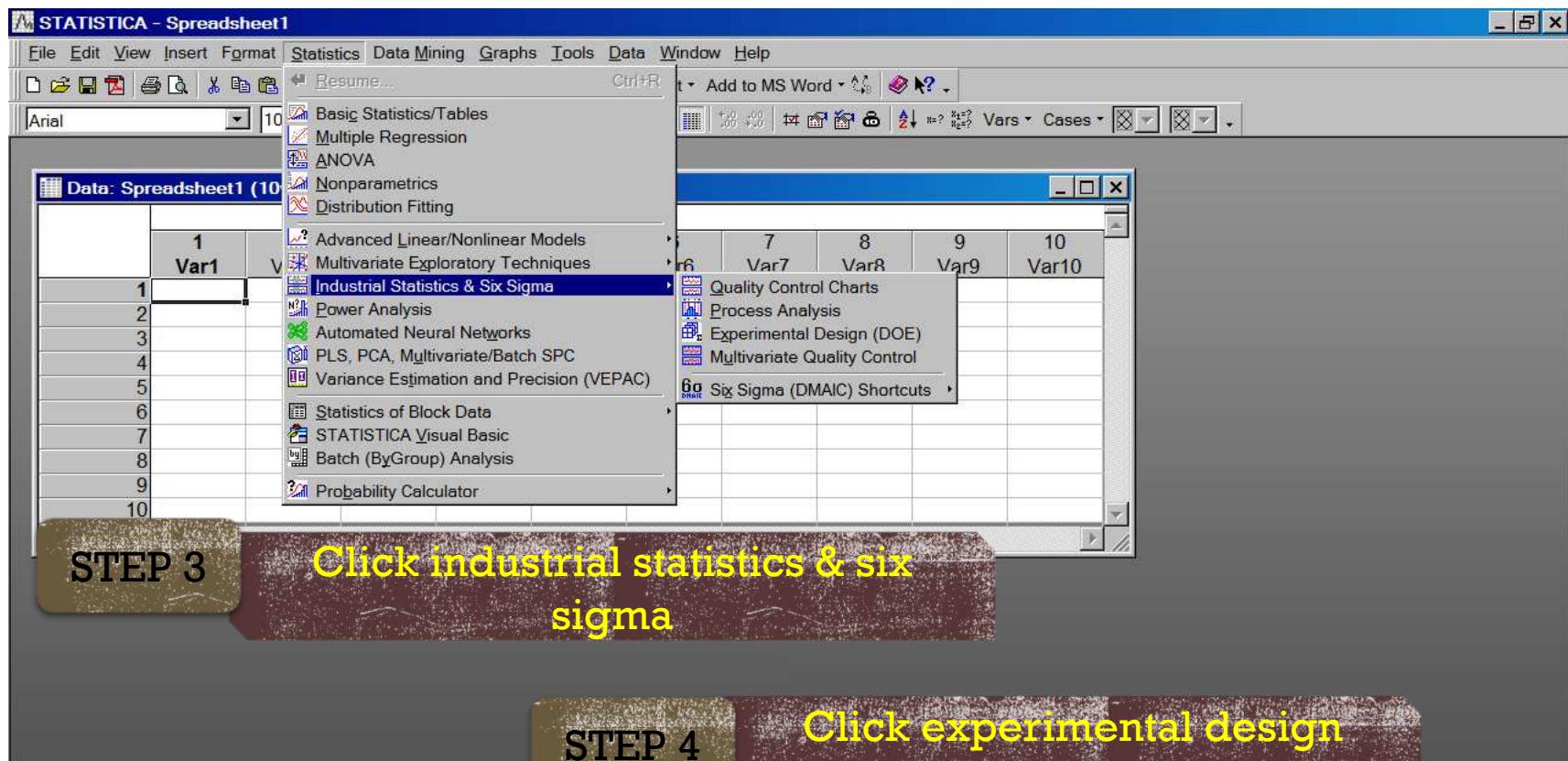
STEP 1



STEP 2

Click Statistica





DESIGN & ANALYSIS OF EXPERIMENT WINDOWS

The screenshot shows the STATISTICA software interface. In the foreground, a dialog box titled "Design & Analysis of Experiments: Spreadsheet1" is open. The dialog has two tabs: "Quick" (selected) and "Advanced". Under the "Quick" tab, several experimental design options are listed:

- 2^{k-p}(K-p) standard designs (Box, Hunter, & Hunter)
- 3^{k-p}(K-p) and Box-Behnken designs
- Mixed 2 and 3 level designs
- Central composite, non-factorial, surface designs
- Mixture designs and triangular surfaces

At the bottom right of the dialog box are buttons for "OK", "Cancel", and "Options". A note below the list states: "For full-factorial designs, hierarchically nested models or designs with unbalanced nesting, and mixed-model (random effect) designs, see also Variance Components and GLM." Below the dialog box, a yellow callout bubble contains the text: "STEP 5 Click central composite, non factorial, surface design→ok".

The background shows the main STATISTICA window with a spreadsheet titled "Data: Spreadsheet1 (10v by 10c)". The spreadsheet has columns labeled "Var1", "Var2", and "Var3". The status bar at the bottom of the screen shows "Ready", "Spreadsheet1 C1,V1", "Sel:OFF Weight:OFF CAP NUM REC", and the system clock "2:51 AM".



STATISTICA - Spreadsheet1

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help

Add to Workbook Add to Report Add to MS Word Vars Cases

Arial 10 B I U Vars Cases

Data: Spreadsheet1 (10v by 10c)

Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsheet

Design experiment Analyze design

Standard design Small design

Factors/blocks/runs:

1/1/82 2/2/10 3/1/16 3/2/16 3/3/17 4/1/26 4/2/26 4/3/27 5/1/27 5/2/28
5/2/44 5/5/47 6/1/46 6/2/46 6/3/47 7/1/80 7/2/80 7/5/83 8/2/82 8/5/82
8/2/82 8/5/82 8/1/30 8/2/30 8/1/40 8/2/40 8/1/54 8/2/54

OK Cancel Options

Replications, additional points, labels, etc., can be specified on the next dialog; see also the 3⁺(K-p) and Box-Behnken design option for additional designs.

SELECT CASES

STEP 6 Pick suitable design →ok



STATISTICA - Spreadsheet1

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help

Add to Workbook Add to Report Add to MS Word Vars Cases

Arial 10 B I U

Data: Spreadsheet1 (10v by 10c)

Design of a Central Composite (Response Surface) Experiment: Spreadsheet1

STANDARD DESIGN SUMMARY: 2**4 cube plus star (central composite design)
Number of factors: 4
Number of blocks: 1
Number of runs: 26 nc=16 ns=8 n0=2
Alpha for rotatability: 2.0000 Alpha for orthogonality: 1.4826

Quick Display design Add to design Design characteristic Generators & aliases

Summary Cancel Summary Box Options

To save the design, use option "Display design," modify the design if necessary, and save the Spreadsheet.

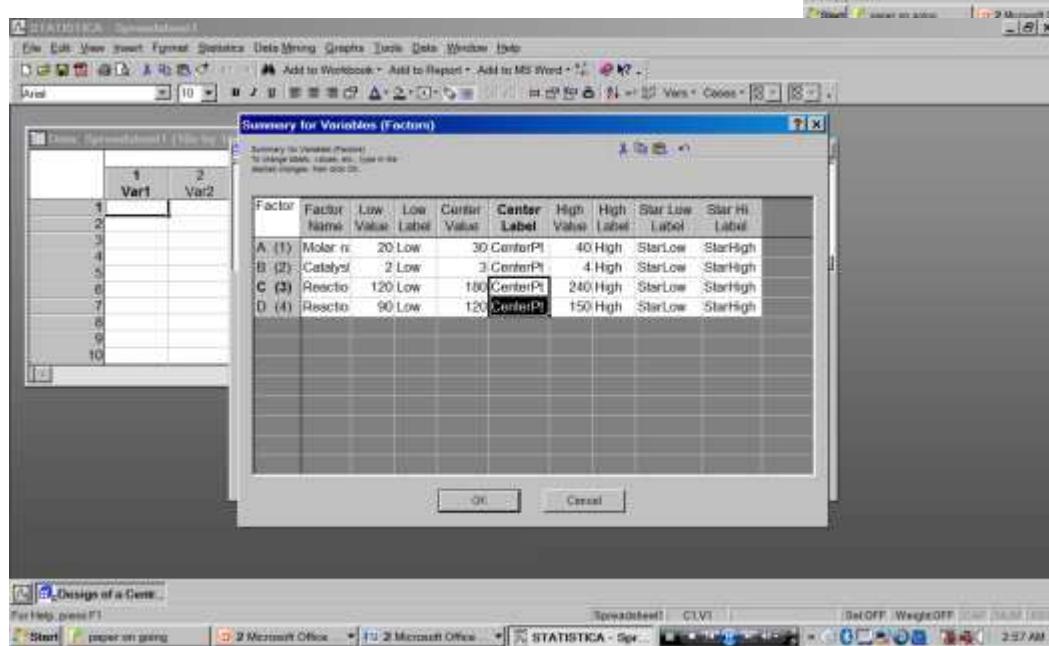
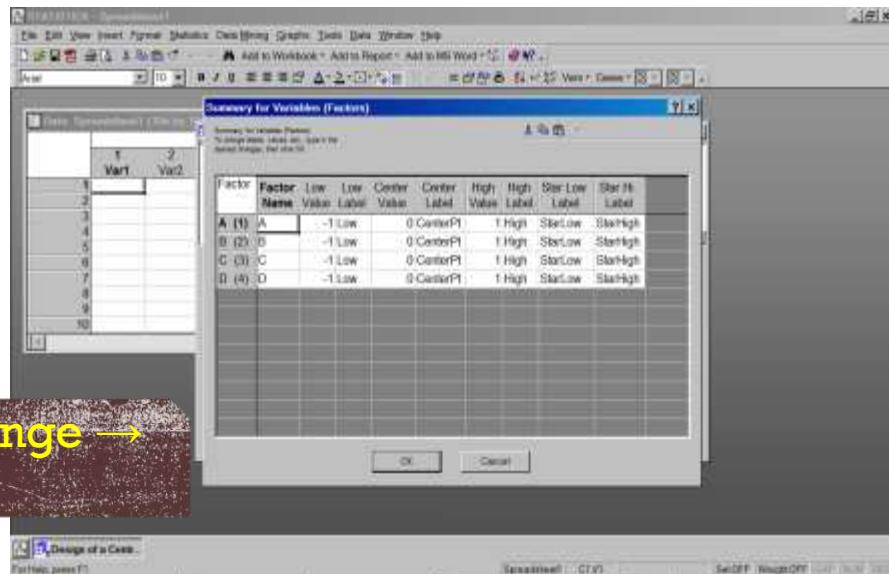
STEP 7 Click change factor value etc



CHANGE VALUE

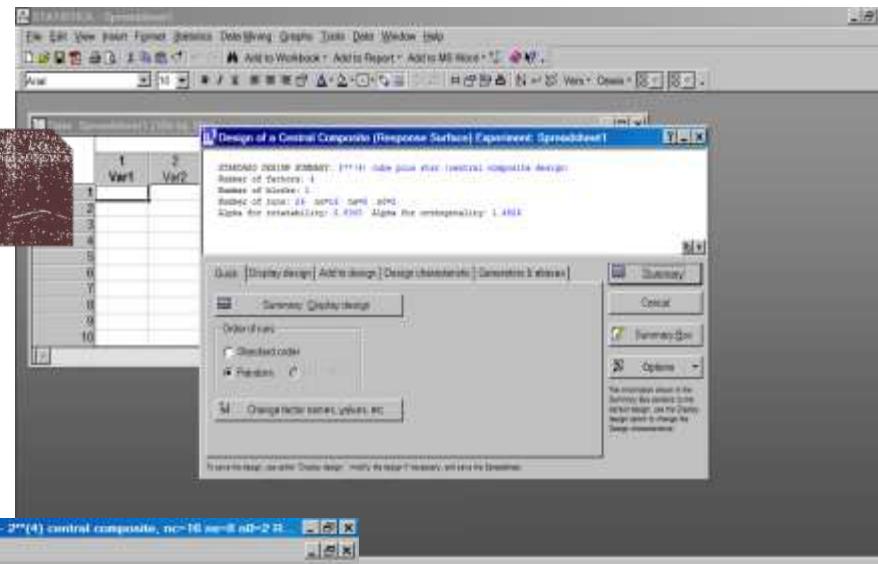
STEP 8

Insert the variable and range →
ok



STEP 9

Click design display
(standard order)



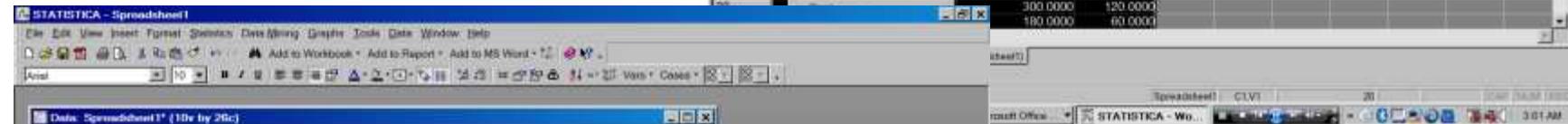
Run	Standard	Molar ratio: methanol: oil	Catalyst loading: wt%	Reaction Time, min	Reaction Temperature
1		20.00000	2.00000	120.0000	90.0000
2		20.00000	2.00000	120.0000	150.0000
3		20.00000	2.00000	240.0000	90.0000
4		20.00000	2.00000	240.0000	150.0000
5		20.00000	4.00000	120.0000	90.0000
6		20.00000	4.00000	120.0000	150.0000
7		20.00000	4.00000	240.0000	90.0000
8		20.00000	4.00000	240.0000	150.0000
9		40.00000	2.00000	120.0000	90.0000
10		40.00000	2.00000	120.0000	150.0000
11		40.00000	2.00000	240.0000	90.0000
12		40.00000	2.00000	240.0000	150.0000
13		40.00000	4.00000	120.0000	90.0000
14		40.00000	4.00000	120.0000	150.0000
15		40.00000	4.00000	240.0000	90.0000
16		40.00000	4.00000	240.0000	150.0000
17		10.00000	3.00000	180.0000	120.0000
18		50.00000	3.00000	180.0000	120.0000
19		30.00000	1.00000	180.0000	120.0000
20		30.00000	5.00000	180.0000	120.0000
21		30.00000	3.00000	60.0000	120.0000
22		30.00000	3.00000	300.0000	120.0000
23		30.00000	3.00000	180.0000	60.0000

Design display on
workbook windows



COPY DOE TO SPREADSHEET

STEP 1 Select all → right click →
copy with headers

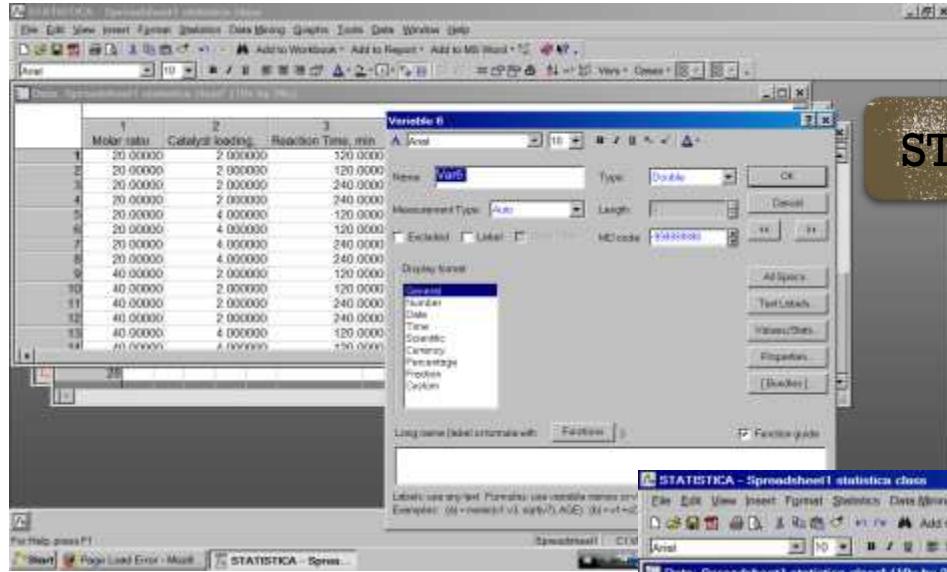


	1	2	3	4	5	6	7
Molar ratio	Catalyst loading	Reaction Time, min	Reaction	Vars	Vars	Vars	Vars
1	20.00000	2.000000	120.0000	90.0000			
2	20.00000	2.000000	120.0000	150.0000			
3	20.00000	2.000000	240.0000	90.0000			
4	20.00000	2.000000	240.0000	150.0000			
5	20.00000	4.000000	120.0000	90.0000			
6	20.00000	4.000000	120.0000	150.0000			
7	20.00000	4.000000	240.0000	90.0000			
8	20.00000	4.000000	240.0000	150.0000			
9	40.00000	2.000000	120.0000	90.0000			
10	40.00000	2.000000	120.0000	150.0000			

STEP 2 Paste on spreadsheet



EDIT, SAVE & PRINT SPREADSHEET

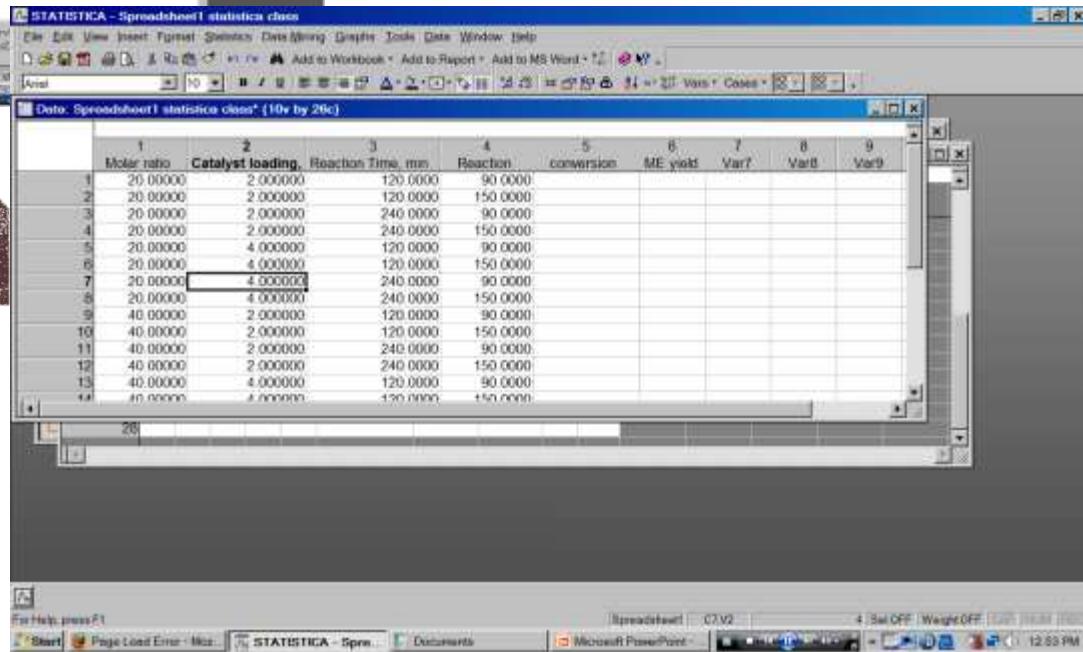


STEP 1

Right click on the column → edit

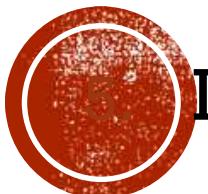
STEP 2

click file → save



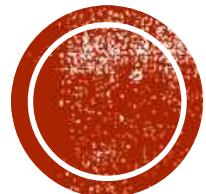
STEP 3

click file → print



1. Insert the complete the data
2. Develop the empirical/predicted model
 3. Statistic analysis of empirical model
 4. Find the importance of process variables
5. Investigate the influence of process variables
6. Optimization of process variables

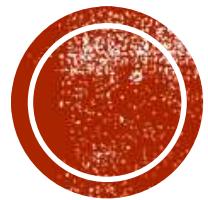
AFTER EXPERIMENT



COMPLETE THE DATA

Insert the collected data into the software.

(refer to tutorial 2)



STATISTICA TUTORIAL 2

INSERT AND ANALYSIS THE DATA

INSERT THE RESULT INTO SPREADSHEET

The screenshot shows the STATISTICA software interface with a menu bar (File, Edit, View, Insert, Format, Statistics, Data Mining, Graphs, Tools, Data, Window, Help) and a toolbar below it. The main window displays a spreadsheet titled "Data: Spreadsheet1 statistica class* (10v by 26c)". The spreadsheet has 10 rows and 9 columns, labeled 1 through 9. Column 5 is specifically labeled "conversion". The data includes various numerical values such as 20.00000, 4.000000, and 59.56. A red box highlights the value "40.00" in cell E5. The status bar at the bottom shows "For Help, press F1", "Page Load Error - ...", "STATISTICA", "heightOFF CAP NUM REC", and the time "12:01 PM". A large callout bubble in the center of the screen contains the text "Open spreadsheet and insert the result" and "STEP 1" in yellow, and "Remember save the spreadsheet" and "(note: spreadsheet is an important in statistica)" in white.

	1 Molar ratio	2 Catalyst loading,	3 Reaction Time, min	4 Reaction	5 conversion	6 ME yield	7 Var7	8 Var8	9 Var9
1	20.00000	2.000000	120.0000	90.0000	48.73	59.56			
2	20.00000	2.000000	120.0000	150.0000	69.02	73.55			
3	20.00000	2.000000	240.0000	90.0000	50.00	67.56			
4	20.00000	2.000000	240.0000	150.0000	40.00	53.76			
5	20.00000	4.000000	120.0000	90.0000	43.88	59.34			
6	20.00000	4.000000	120.0000	150.0000	53.00	69.90			
7	20.00000	4.000000	240.0000	90.0000	56.00	65.45			
8	20.00000	4.000000	240.0000	150.0000	50.00	45.32			
9	40.00000	2.000000	120.0000	90.0000	54.80	56.39			
10	40.00000	2.000000	120.0000	150.0000	68.59	74.56			
11	40.00000	2.000000	240.0000	90.0000	80.00	84.46			
12	40.00000	2.000000	240.0000	150.0000	42.31	34.78			
13	40.00000	4.000000	120.0000	90.0000	40.00	52.89			
14	40.00000	4.000000	120.0000	150.0000	56.44	54.89			



STEP 2

Click Statistica

The screenshot shows the STATISTICA software interface. The menu bar is visible at the top with options like File, Edit, View, Insert, Format, Statistics, Data Mining, Graphs, Tools, Data, Window, and Help. A sub-menu is open under the Statistics option, listing various statistical methods. The 'Industrial Statistics & Six Sigma' option is highlighted with a blue selection bar. Below this, other options like Basic Statistics/Tables, Multiple Regression, ANOVA, Nonparametrics, Distribution Fitting, Advanced Linear/Nonlinear Models, Multivariate Exploratory Techniques, Power Analysis, Automated Neural Networks, PLS, PCA, Multivariate/Batch SPC, Variance Estimation and Precision (VEPAC), Statistics of Block Data, STATISTICA Visual Basic, Batch (ByGroup) Analysis, and Probability Calculator are listed. The main workspace shows a spreadsheet titled 'Data: Spreadsheet1 statistic' with columns labeled 1 through 9. The first column contains values from 1 to 14, and the second column contains 'Molar ratio' values. The third column is partially visible.

STEP 3

Click industrial statistics & six sigma

STEP 4

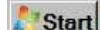
Click experimental design (DOE)



Starts up Design of Experiments (DOE)

Spreadsheet1 C1,V5

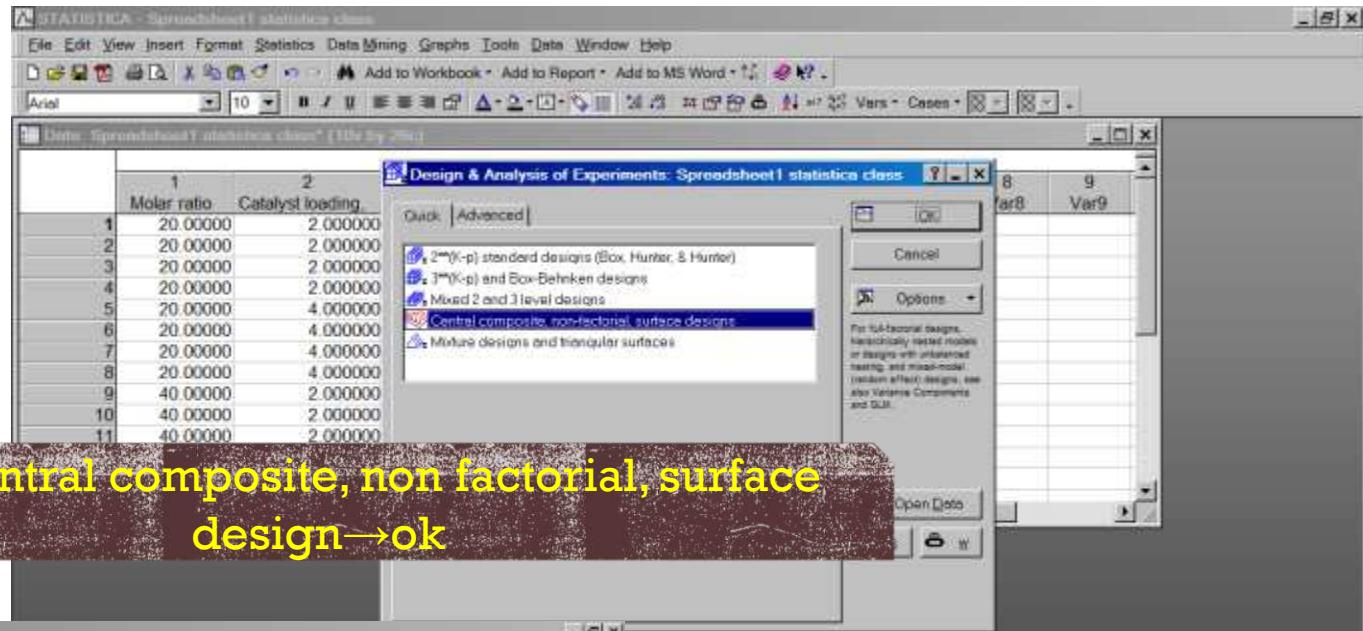
48.73 Sel:OFF Weight:OFF CAP NUM REC



Page Load Error - Moz...

STATISTICA - Spre... Documents Microsoft PowerPoint - ...





STEP 5

Click central composite, non factorial, surface
design → ok

The screenshot shows the STATISTICA software interface again. This time, the 'Design & Analysis of Central Composite (Response Surfaces)' dialog box is open, also under the 'Spreadsheet1' tab. The 'Analyze design' tab is selected. There are two radio buttons: 'Standard design' (selected) and 'Small design'. Below these buttons, there is a section for 'Factors/blocks/runs' with a dropdown menu showing options like '1/1/10 5/1/44 6/1/82', '2/2/10 5/2/44 6/2/82', etc. At the bottom of the dialog box, there is a note about replicates, additional points, levels, etc., and a link to the '2^m(k-p) and Box-Behnken design option for additional designs'. On the right side, there are buttons for 'OK', 'Cancel', and 'Options'. The status bar at the bottom of the window shows 'Spreadsheet1 C1,V5 48.73 SetOFF WeightOFF 11:17 PM'.

STEP 6

Click analyze design tab

STATISTICA - Spreadsheet1 statistica class

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help

Add to Workbook Add to Report Add to MS Word Vars Cases

Arial 10 B I U Vars Cases

Data: Spreadsheet1 statistica class* (10v by 26c)

	1 Molar ratio	2 Catalyst level	9 Varg
1	20.00000		
2	20.00000		
3	20.00000		
4	20.00000		
5	20.00000		
6	20.00000		
7	20.00000		
8	20.00000		
9	40.00000		
10	40.00000		
11	40.00000		
12	40.00000		
13	40.00000		
14	40.00000		

Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsheet1

Design experiment Analyze design

Variables

Dependent: none

Independent (factors): none

Blocking variable: none

To recode factor values (levels), use

Automatically determined factor levels from file

User-defined high/low factor values

Factor levels are recoded as
 $x = (\text{value}-\text{avg.})/(\text{range}/2)$; where
range=HighValue-LowValue, and
avg.= $(\text{HighValue}+\text{LowValue})/2$; press F1 or
click ? for more info.

Use this option (dialog) to analyze central composite and non-factorial
experiments; you can also use the 3*(k-p) and Box-Behnken, 2*(k-p), and mixed
2/3 level designs options to fit response surfaces to designs without star points.

OK Cancel Options

STEP 8 Click variables



STEP 9

Pick variable → ok

Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsheet

Select dependent and independent variables, and (optional) blocking variable

Dependent: conversion

Independent (factors): 1-4

Blocking variable: none

OK Cancel [Bundles]...

Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsheet

Design experiment Analyze design

Variables

Dependent: conversion

Independent (factors): 1-4

Blocking variable: none

To record factor values (levels), use:

Automatically determined factor levels from file

User-defined high/low factor values

OK Cancel Options

STEP 10

Click ok

STATISTICA - Spreadsheet1 statistica class

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help

Add to Workbook Add to Report Add to MS Word

Arial 10 B I U

Data: Spreadsheet1 statistica class* (10v by 26c)

	1 Molar ratio	2 Catalyst loading,	3 Reaction Tin
1	20.00000	2.000000	
2	20.00000	2.000000	
3	20.00000	2.000000	
4	20.00000	2.000000	
5	20.00000	4.000000	
6	20.00000	4.000000	
7	20.00000	4.000000	
8	20.00000	4.000000	
9	40.00000	2.000000	
10	40.00000	2.000000	
11	40.00000	2.000000	
12	40.00000	2.000000	
13	40.00000	4.000000	
14	40.00000	4.000000	

Analysis of a Central Composite (Response Surface) Experiment: Spreadsheet1 statistica class

DESIGN SUMMARY:
Number of factors (independent variables): 4
Total number of runs (cases, experiments): 26
Number of unique runs (cases experiments): 25
Number of blocks: 1
Number of replications: 0 - 1

Variable: conversion Print results All variables

Review/save residuals Residual plots Box-Cox Prediction & profiling
Quick Model Design ANOVA/Effects Means

ANOVA Predicted (estimated) response
Summary: Effect estimates Fitted response surface
ANOVA table Fitted response profiles
Pareto chart of effects Critical values (min, max, saddle)

These results are for the current model; you can change the model (add or remove interaction effects) on the Model tab.

Analysis of a Ce... Ready Spreadsheet1 C1.V5 48.73 Sel:OFF Weight:OFF CAP NUM REC

Start Page Load Error - Moz... STATISTICA - Spre... Documents Microsoft PowerPoint - ... 1:20 PM

Analysis of the central composite (response surface) experiment windows opened.
(note: this windows is an important for analysis since it display all information needed)

SAVE AS PROJECT

STATISTICA - [Data: final spreadsheet adjusted (14v by 16c)]

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help

New... Ctrl+N
Open... Ctrl+O
Open URL...
Open Examples...
Close
Save Ctrl+S
Save As... F12
Save As PDF...
Save Project...
Save Project As...
Open Project...
Get External Data
Add to Workbook
Add to Report
Output Manager...
Print Setup...
Print Preview
Print... Ctrl+P
Properties...
1 final spreadsheet adjusted
2 solid recovery and monomer
3 C:\Users\...\sol
4 final spreadsheet adjusted-beginning
5 final spreadsheet adjusted-ending
6 C:\Users\...\sept7
7 WorkbookOptima dec 2013
8 C:\Users\...\screening2nd

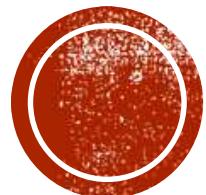
Add to Workbook Add to Report Add to MS Word Vars Cases

3 particle size (mm)	4 Reaction time (min)	5 Ozone flowrate (LPM)	6 Solid Recovery (%)	7 Lignin Degradation (%)	8 WHC (g H ₂ O/g)	9 Mass swollen (g/g OD)	10 Solubility (%)	11 Glucose (g/g material)	12 Xylose (g/g material)	13 Glucos (%)
0.25	30	90	74.80	85.05	7.23	8.03	25.00	1.47	2.19	109
0.25	30	60	68.00	66.74	8.32	9.20	13.21	0.83	0.75	56
0.25	30	60	59.80	48.60	8.20	9.23	-3.23	0.53	0.62	31
0.25	30	90	74.06	56.82	8.23	9.00	30.43	1.10	1.68	81
0.63	30	60	59.20	98.73	5.93	6.90	3.45	1.96	3.56	115
0.63	30	90	63.60	89.11	6.67	7.67	0.00	2.20	3.50	139
0.63	30	90	63.40	73.76	7.63	8.50	15.38	2.10	2.71	133
0.63	30	60	67.40	89.94	7.17	8.17	0.00	1.90	2.26	128
0.25	60	60	66.40	83.64	7.43	8.37	7.14	1.84	2.81	121
0.25	60	90	76.60	80.68	5.97	6.77	25.00	0.10	0.07	7
0.25	60	90	68.50	79.67	8.73	9.67	7.14	1.53	2.53	104
0.25	60	60	73.67	75.30	8.50	9.37	15.38	1.35	2.14	99
0.63	60	90	56.50	98.09	6.43	7.43	0.00	2.20	3.42	124
0.63	60	60	61.70	67.04	5.93	6.93	0.00	1.34	2.56	82
0.63	60	60	54.10	95.54	9.87	7.00	1.03	2.10	3.13	114
								33	3.64	148

final spreadsheet C1,V14 163.641403508772 Set:OFF Weight:OFF CAP NUM REC

6:15 PM 9/5/2015

This statistica project file can be opened anytime and the analysis and workbook could be resume.



- First Order polynomial
- Second Order polynomial

DEVELOP THE MODEL

MATHEMATICAL MODEL (EMPIRICAL/PREDICTED MODEL)

To

- represent the relationship of response function and the factor level
- Predict the response at various combination of process variables

Can be shown by

- First order model (linear, X)
- Second order model (Quadratic, X^2)
- Third model (Cubic, X^3), if using design expert.

Analysis

- the least square method was employed to estimate the response surface model.

Test of significance of model

- t and F-test
- Parity plot
- Pareto chart
- Probability plot

Random Error-(Pure error)- are normal distributed

FIRST ORDER MODEL

■ Fit for

- Limited for small experimental region (two level)
- Response surface is hyperlane
- First approximation of the surface
- Cost of experimentation are held to a minimum
- To locate higher value of the response-steepest ascent
- Screening for the important factor

$$\hat{Y} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3$$

Y
 β_0
 β_1, β_2 and β_3
 X_1, X_2 and X_3

: predicted response (response function)
: intercept coefficient (offset)
: linear terms (first order)
: uncoded independent variables



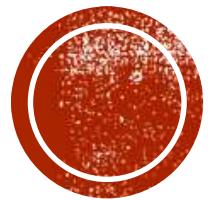
SECOND ORDER MODEL (POLYNOMIAL)

- Normally used for optimization since it is consider the center point

$$\hat{Y} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 \\ + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2$$

Y	: predicted response (response function)
β_0	: intercept coefficient (offset)
β_1 , β_2 and β_3	: linear terms (first order)
β_{11} , β_{22} and β_{33}	: quadratic terms (second order)
β_{12} , β_{13} and β_{23}	: interaction terms
X_1 , X_2 and X_3	: uncoded independent variables





STATISTICA TUTORIAL 3

PREDICTED/EMPIRICAL MODEL

MODEL (COEFFICIENT SELECTION)

Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7

DESIGN SUMMARY:

Number of factors (independent variables): 4
Total number of runs (cases, experiments): 27
Number of unique runs (cases experiments): 25
Number of blocks: 1
Number of replications: 0 - 2

Variable: Lignin degrade ▾

Print results All variables

Review/save residuals | Residual plots | Box-Cox | Prediction & profiling

Quick Model Design ANOVA/Effects Means

Include in model

No interactions
 2-way interactions (linear x linear)
 2-way interactions (linear, quadr.)
 Ignore some effects

Specify the effects that are to be included in the model. All ANOVA results, effect estimates, predicted and residual values, etc. will be computed based on this model.

ANOVA error term

SS residual
 Pure error

The error term will be used in all tests for statistical significance, and in the computation of standard errors.

Summary Cancel Options By Group

STEP 1

STEP 2 Click Anova/effect
→ regression coefficient

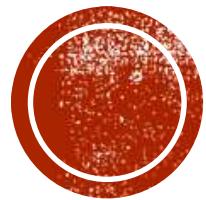
Regr. Coefficients; Var: conversion, R-sqr= 81226, Adj: 57332 (Spreadsheet1 statistica class)

4 factors, 1 Blocks, 26 Runs, MS Residual=59.00487

DV: conversion

Factor	Regress.	Std Err	t(11)	P	-95. % Cnf Limit	+95. % Cnf Limit
Mean/Interc.	Coeff.					
(1)Molar ratio methanol: oil(L)	-192.048	74.73568	-2.56970	0.020064	358.540	-27.5562
Molar ratio methanol: oil(Q)	4.623	1.55151	2.97971	0.012526	1.206	8.0379
(2)Catalyst loading, w%(L)	-0.050	0.01771	-2.83881	0.016116	-0.089	-0.0113
Catalyst loading, w%(Q)	-0.266	15.51509	-0.01711	0.986652	-34.414	33.8830
(3)Reaction Time, min(L)	-0.843	1.77106	-0.47580	0.643528	-4.741	3.0554
Reaction Time, min(Q)	0.774	0.25856	2.99253	0.012242	0.205	1.3430
(4)Reaction Temperature(L)	-0.001	0.00049	-2.52885	0.02031	-0.002	-0.0002
Reaction Temperature(Q)	1.978	0.57997	3.41055	0.005620	0.702	3.2545
1L by 2L	-0.003	0.00197	-1.52765	0.154828	-0.007	0.0013
1L by 3L	-0.198	0.19204	-1.03170	0.324361	-0.621	0.2245
1L by 4L	0.004	0.00320	1.10005	0.294801	-0.004	0.0108
2L by 3L	-0.011	0.00640	-1.74316	0.109151	-0.025	0.0029
2L by 4L	0.056	0.03201	1.76399	0.105449	-0.014	0.1269
3L by 4L	0.001	0.06401	0.01562	0.987816	-0.140	0.1419
	0.009	0.00107	-4.75235	0.000597	-0.007	-0.002?

$$Y_2 = -192.048 + 4.623X_1 - 0.266X_2 + 0.774X_3 + 1.978X_4 - 0.050X_1^2 - 0.843X_2^2 - 0.001X_3^2 - 0.003X_4^2 \\ - 0.198X_1X_2 + 0.004X_1X_3 - 0.011X_1X_4 + 0.056X_2X_3 + 0.001X_2X_4 - 0.005X_3X_4$$



- Regression analysis
- ANOVA
- Hypothesis testing

STATISTIC ANALYSIS OF MODEL

VALIDITY OF MODEL

The adequacy of the fitted model is checked by *ANOVA (Analysis of Variance) using Fisher F-test*

The fit quality of the model can also be checked from their *Coefficient of Correlation (R)* and *Coefficient of Determination (R^2)*



OBSERVED AND PREDICTED TABLE

x_1	x_2	Y_u	\hat{Y}_u	$Y_u - \bar{Y}$	$\hat{Y}_u - \bar{Y}$	$Y_u - \hat{Y}_u$
-1	-1	7.0	6.8375	0.6625	0.5000	0.1625
-1	-1	6.9	6.8375	0.5625	0.5000	0.0625
-1	+1	5.2	5.4125	-1.1375	-0.9250	-0.2125
-1	+1	5.4	5.4125	-0.9375	-0.9250	-0.0125
+1	-1	7.1	7.2625	0.7625	0.9250	-0.1625
+1	-1	7.2	7.2625	0.8625	0.9250	-0.0625
+1	+1	6.1	5.8375	-0.2375	-0.5000	0.2625
+1	+1	5.8	5.8375	-0.5375	-0.5000	-0.0375
$\Sigma Y_u = 50.7$		$\Sigma(Y_u - \bar{Y})^2 = 4.59875$		$\Sigma(\hat{Y}_u - \bar{Y})^2 = 4.4225$	$\Sigma(Y_u - \hat{Y}_u)^2 = 0.17625$	
$\bar{Y} = 6.3375$						

$$\text{SSR} = \sum (\hat{Y}_u - \ddot{Y})^2 = 4.4225$$

$$\text{SST} = \sum (Y_u - \ddot{Y})^2 = 4.59875$$

$$\text{SSE} = \sum (Y_u - \hat{Y})^2 = 0.17625$$

COEFFICIENT OF DETERMINATION (R-SQUARE, R^2)

Coefficient of Determination (R^2): a proportion of total variation of the observed values of activity (Y_i) about the mean explained by the fitted model

- $R^2 = \text{SSR}/\text{SST}$

Coefficient of Correlation (R) : an acceptability about the correlation between the experimental and predicted values from the model.

Adjusted R^2 : Measure the drop of magnitude of the estimate of the error variance

- $\text{adj } R^2 = 1 - \text{MS}_{\text{residual}} / (\text{SST}/N)$ - more smaller more better



HOW TO INTERPRET THE R^2 ?

R^2 value is always in between 0 to 1

The value of 1 , indicated the empirical/predicted model explains all of the variability in the data

The value of 0, indicated that none of the variability in the data can be explained by predicted model.

R^2 is closer to 1, the predicted model is more reliable

$R^2 > 0.75$ acceptable (Haaland), however, > 0.8 is much better



ANALYSIS OF VARIANCE (ANOVA)

- The F -value is a measurement of variance of data about the mean based on the ratio of mean square (MS) of group variance due to error.
- $F\text{-value} = \text{MS}_{\text{regression}}/\text{MS}_{\text{residual}} = (\text{SSR}/\text{DF}_{\text{regression}})/(\text{SSE}/\text{DF}_{\text{residual}})$

where:

SSR = sum of square of regression

SSE = sum of square of error/residual

$\text{DF}_{\text{regression}}$ =degree of freedom of regression

$\text{DF}_{\text{residual}}$ =degree of freedom of residual

- F table $= F(p-1, N-p, \alpha)$
 - $p-1$: $\text{DF}_{\text{regression}}$
 - $N-p$: $\text{DF}_{\text{residual}}$
 - N =total exp
 - P =no of term in fitted model
 - α -value: level of significant

- the calculated F -value should be greater than the tabulated F -value to reject the null hypothesis,



HYPOTHESIS TESTING (F VALUE)

- There are 2 statement is comparing at **significant confident level (95%, $\alpha= 0.05$)**

F-table Can be
find online

Null hypothesis, H_0 : All the coefficient (β) are zero

Alternative hypothesis, H_1 : At least one of coefficient (β) is not zero.

Conclusion: The null is

The surface is
plane

True: $F_{cal} < F_{table}$, cannot be rejected

Rejected: $F_{cal} > F_{table}$.

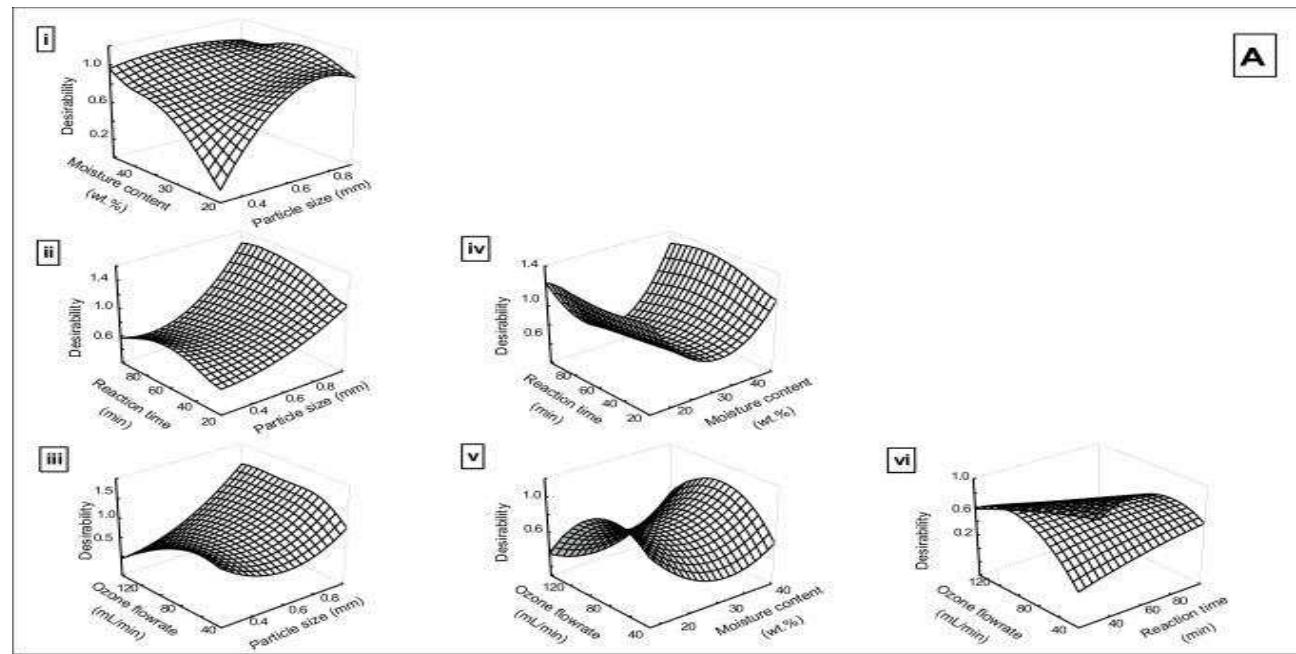
OR

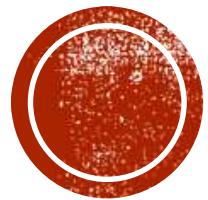
The surface is
twisted.



EXAMPLE

Sources	Sum of Squares (SS)	Degree of Freedom(d.f)	Mean Squares (MS)	F-value	$F_{0.05}$
Lignin Degradation, $R^2=99.0\%$, adj=87.4%					
Regression (SSR)	2055.83	24	85.66	8.51	19.45
Residual	20.12	2	10.06		
Total (SST)	2075.95	26			





STATISTICA TUTORIAL 4

ANOVA

ANOVA/EFFECTS

Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7

DESIGN SUMMARY:

Number of factors (independent variables): 4
Total number of runs (cases, experiments): 27
Number of unique runs (cases experiments): 25
Number of blocks: 1
Number of replications: 0 - 2

Variable: Lignin degrade ▾ Print results All variables

Review/save residuals | Residual plots | Box-Cox | Prediction & profiling

Quick | Model | Design | ANOVA/Effects | Means

Summary | Cancel | Options | By Group

Plots of effects

Normal probability plot
 Half-normal probability plot
 Pareto chart
 Label points in normal plot
 Exclude block effects
 Plot standardized effects

Summary: Effect estimates

Use centered & scaled polynomials

Regression coefficients

Effects sorted by size

Confidence interval: 95.0 %

Alpha (highlighting): .050

ANOVA table

These results are for the current model; you can change the model (add or remove interaction effects) on the Model tab.

n (%); R-sqr=.99031; ... Probability Plot Var.:Lignin degradation (%);

STEP Click ANOVA table tab

ANOVA TABLE

STATISTICA [Workbook2 - ANOVA, Var.: conversion, R-sqr= 81226, Adj. 57332 (Spreadsheet1 statistic class)]

File Edit View Insert Format Statistics Data Mining Graphs Tools Data Workbook Window Help

Add to Report

Analyses

Factor:

	SS	df	MS	F	p
(1) Molar ratio methanol: oil(L)	230.296	1	230.296	3.90299	0.073822
Molar ratio methanol: oil(Q)	475.510	1	475.510	8.05883	0.016116
(2) Catalyst loading, wt% (L)	23.637	1	23.637	0.40059	0.539720
Catalyst loading, wt% (Q)	13.358	1	13.358	0.22638	0.643528
(3) Reaction Time, min (L)	4.919	1	4.919	0.08337	0.778150
Reaction Time, min (Q)	377.341	1	377.341	6.39508	0.02601
(4) Reaction Temperature (L)	3.255	1	3.255	0.05517	0.818622
Reaction Temperature (Q)	137.701	1	137.701	2.33372	0.154828
1L by 2L	62.806	1	62.806	1.06441	0.324361
1L by 3L	71.403	1	71.403	1.21011	0.294801
1L by 4L	179.292	1	179.292	3.03060	0.109151
2L by 3L	183.603	1	183.603	3.11165	0.105449
2L by 4L	0.014	1	0.014	0.00024	0.96701
3L by 4L	1322.615	1	1322.615	22.5948	0.000097
Error	649.054	11	59.005		
Total SS	3457.391	25			

R²>0.75 (Haaland, 1989)

Residual

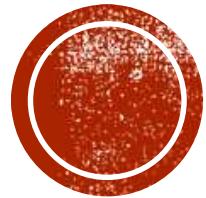
DF

SST

Sources	Sum of Squares(SS)	Degree of Freedom(d.f)	Mean Squares (MS)	F-value	$F_{0.05}$
Regression (SSR)	2807.32	14	200.52	3.39	>2.7
Residual	649.87	11	59.08		4
Total (SST)	3457.29	25			

$$SSR = SST - \text{residual}$$

$$DF_{SSR} = DF_{SST} - DF_{\text{residual}}$$



- T-value and p -value
- Pareto chart
- Probability plot

IMPORTANCE/SIGNIFICANT OF PROCESS VARIABLES

SIGNIFICANT OF THE MODEL COEFFICIENT

T-Value:

- Measure how large the coefficient is in relationship to its standard error
- $T\text{-value} = \text{coefficient} / \text{standard error}$

P-value

- is an observed significance level of the hypothesis test or the probability of observing an F-statistic as large or larger than one we observed.
- The small values of p -value → the null hypothesis is not true.

Can be visualized

- Pareto Chart
- Normal Probability plot

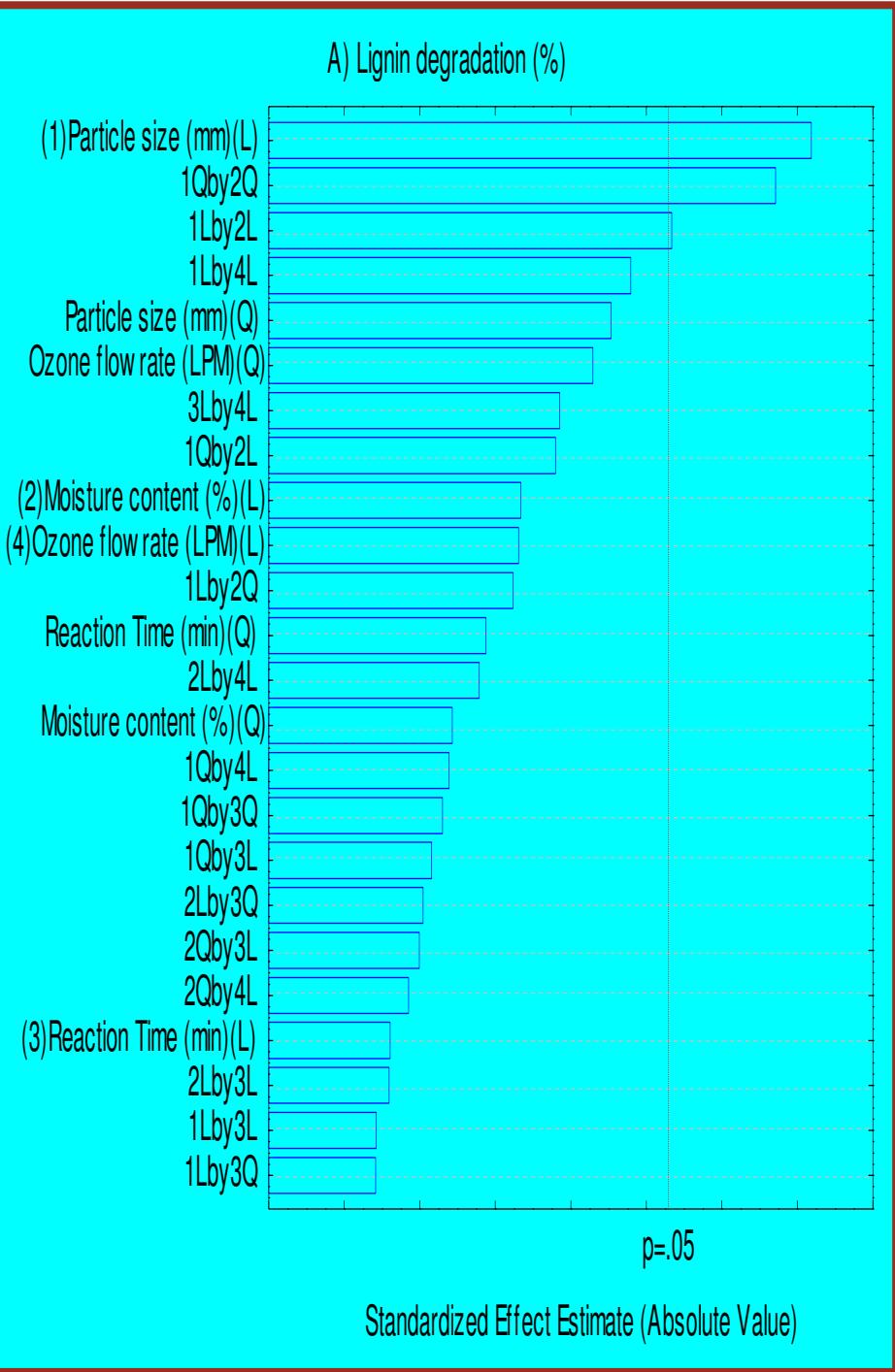
INTERPRETATION?

- If a p -value is ≤ 0.01 , then the H_0 can be rejected at a 1% significance level → “*convincing*” evidence that the H_A is true.
- If a p -value is $0.01 < p\text{-value} \leq 0.05$, then the H_0 can be rejected at a 5% significance level → “*strong*” evidence in favor of the H_A .
- If a p -value is $0.05 < p\text{-value} \leq 0.10$, then the H_0 can be rejected at a 10% significance level. → it is in a “*gray area/moderate*”
- If a p -value is > 0.10 , then the H_0 cannot be rejected. → “*weak*” or “*no*” evidence in support of the H_A .

Design expert:
 P -value sometimes known as P_{prob}

Statistica:
visualize using pareto chart

PARETO CHART



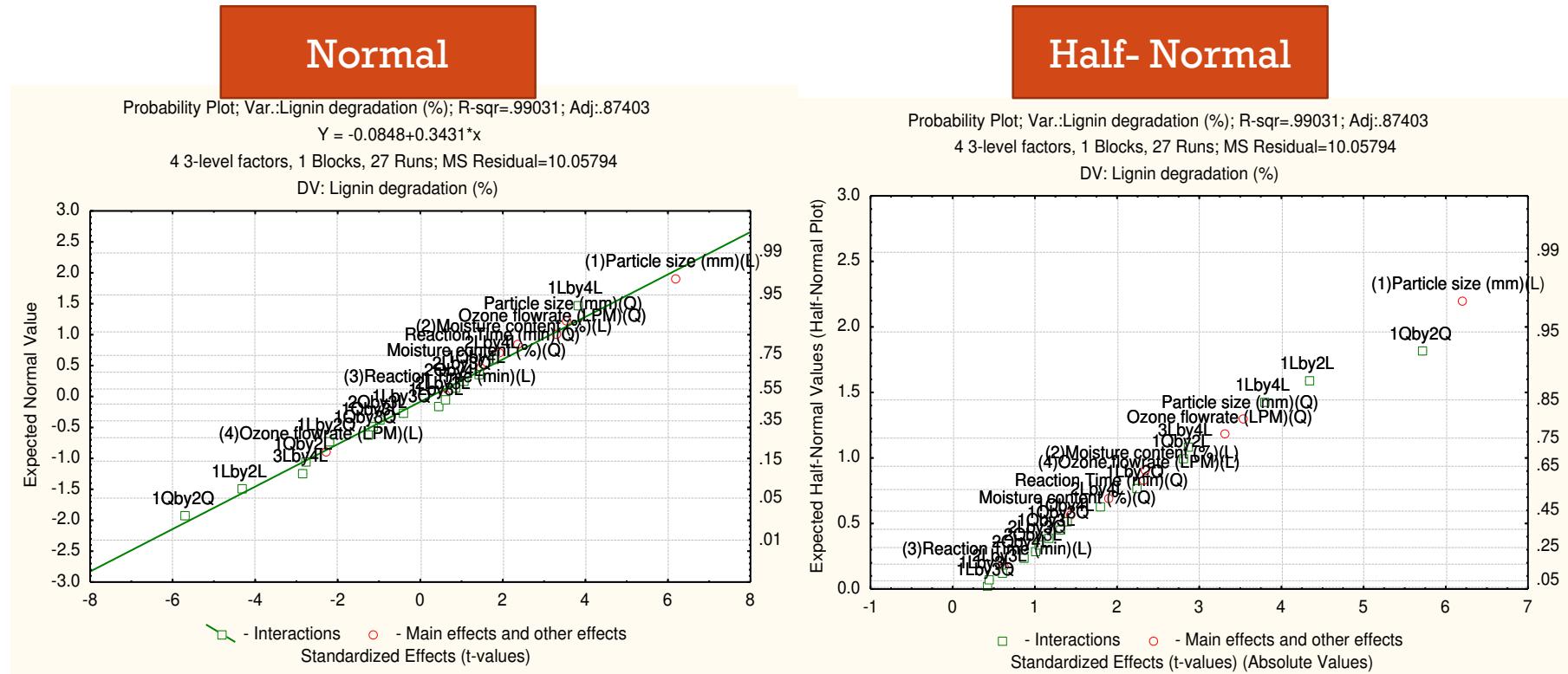
ANOVA effect estimates are sorted from largest to small value

The magnitude of each effect is represented by a column

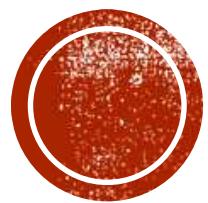
A line going across the column indicates how large and effect has to be statistically significant



PROBABILITY PLOT



- To assess how closely a set of observed values follow a theoretical distribution
 - if all values fall onto straight line, the residual follow the normal distribution
 - The parameter were rank -ordered.



STATISTICA TUTORIAL 5

Effects

TAB OF ANOVA/EFFECTS

Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7

DESIGN SUMMARY:

Number of factors (independent variables): 4
Total number of runs (cases, experiments): 27
Number of unique runs (cases experiments): 25
Number of blocks: 1
Number of replications: 0 - 2

Variable: Lignin degrade ▾ Print results All variables

Review/save residuals | Residual plots | Box-Cox | Prediction & profiling | Summary | Cancel | Options | By Group

Quick | Model | Design | ANOVA/Effects | Means

Summary: Effect estimates
 Use centered & scaled polynomials
 Regression coefficients
 Effects sorted by size
Confidence interval: 95.0 %
Alpha (highlighting): .050
 ANOVA table

Plots or effects

Normal probability plot
 Half-normal probability plot
 Pareto chart
 Label points in normal plot
 Exclude block effects
 Plot standardized effects

These results are for the current model; you can change the model (add or remove interaction effects) on the Model tab.

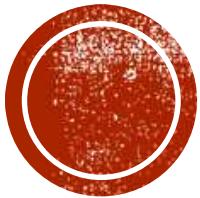
Conf. Limits (95.%) | Probability Plot Var.:Lignin degradation (%); R-sqr=.99031; ... | Probability Plot Var.:Lignin degradation (%);

EFFECT ESTIMATES

Effect Estimates; Var.:Lignin degradation (%); R-sqr=.99031; Adj:.87403 (Spreadsheet optimization sept 7) 4 3-level factors, 1 Block, 27 Runs; MS Residual=10.05794 DV: Lignin degradation (%)										
Factor	Effect	Std.Err.	t(2)	p	-95.% Cnf.Limit	+95.% Cnf.Limit	Coeff.	Std.Err. Coeff.	-95.% Cnf.Limit	+95.% Cnf.Limit
Mean/Interc.	74.7109	1.618268	46.16721	0.000469	67.7481	81.67374	74.71080	1.618268	67.7481	81.67374
(1)Particle size (mm)(L)	19.6213	3.171426	6.18691	0.025144					9	16.63343
Particle size (mm)(Q)	8.1603	2.310647	3.53162	0.071665					8	9.05112
(2)Moisture content (%) (L)	7.4338	3.178466	2.33880	0.144277					0	10.55482
Moisture content (%) (Q)	2.8125	1.965637	1.43078	0.288787					6	5.63507
(3)Reaction Time (min)(L)	1.9354	3.178466	0.60890	0.604542					2	7.80560
Reaction Time (min)(Q)	3.6935	1.965637	1.87896	0.201020					1	6.07556
(4)Ozone flowrate (LPM)(L)	-7.3450	3.178466	-2.31086	0.147050	-2.0208	6.33083	-3.67250	1.589233	-10.5104	3.16542
Ozone flowrate (LPM)(Q)	5.4365	1.650462	3.29390	0.081112	-6.649	12.53783	2.71823	0.825231	-0.8325	6.26891
1L by 2L	-13.7577	3.171426	-4.33802	0.049247	-2.4033	-0.11217	-6.87886	1.585713	-13.7016	-0.05609
1L by 2Q	-5.0159	2.242537	-2.23672	0.154775	-1.6648	4.63292	-2.50797	1.121268	-7.3324	2.31646
1Q by 2L	-6.3382	2.264861	-2.79849	0.107490	-1.0830	3.40669	-3.16909	1.132425	-8.0415	1.70335
1Q by 2Q	-9.1473	1.601401	-5.71174	0.029311	-1.0380	-2.25663	-4.57365	0.800746	-8.0190	-1.12832
1L by 3L	1.3490	3.171426	0.42537	0.711968	-1.2965	14.99456	0.67451	1.585713	-6.1483	7.49728
1L by 3Q	-0.9315	2.242537	-0.41536	0.718199	-1.5803	8.71739	-0.46573	1.121268	-5.2902	4.35870
1Q by 3L	-2.6225	2.264861	-1.15793	0.366486	-1.3674	7.12234	-1.31126	1.132425	-6.1837	3.56117
1Q by 3Q	-2.0845	1.601401	-1.30163	0.322788	-1.9752	4.80611	-1.04227	0.800746	-4.4876	2.40306
1L by 4L	12.0341	3.171426	3.79453	0.062964	-1.6115	25.67960	6.01703	1.585713	-0.8057	12.83980
1Q by 4L	3.1461	2.264861	1.38910	0.299255	-1.5988	12.89098	1.57305	1.132425	-3.2994	6.44549
2L by 3L	1.8843	3.171426	0.59416	0.612662	-1.7612	15.52988	0.94217	1.585713	-5.8806	7.76494
2L by 3Q	2.3447	2.242537	1.04556	0.405508	-1.3041	11.99357	1.17236	1.121268	-3.6521	5.99679

The value is
considered in Pareto
chart



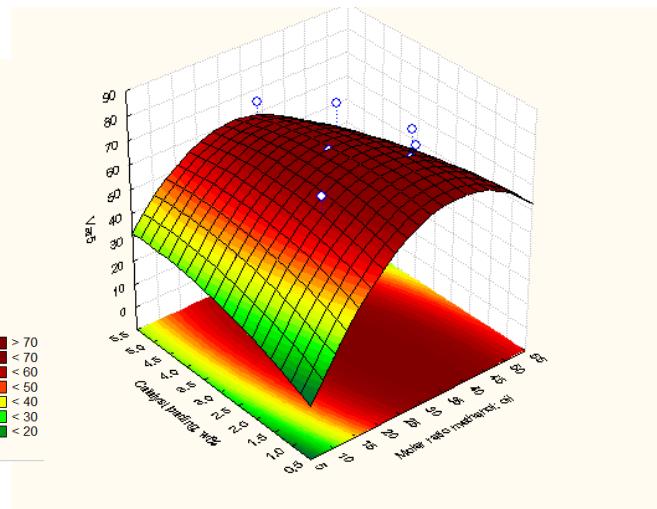
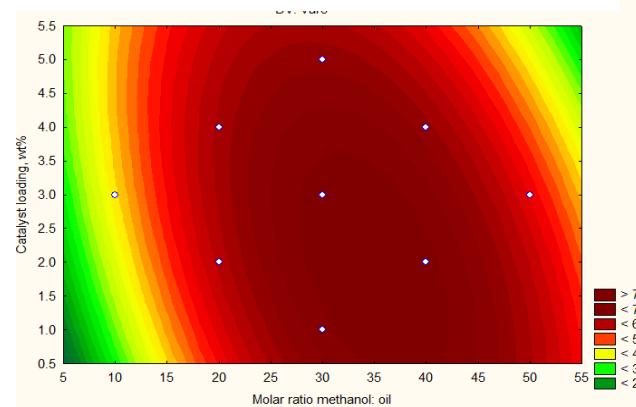
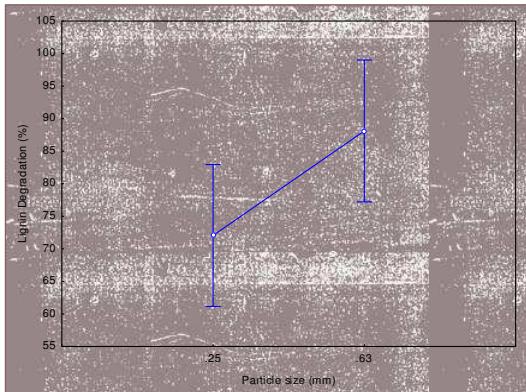


- Contour plot
- Surface (3D)
- Single parameter

INTERACTION/INFLUENCE OF PROCESS VARIABLES

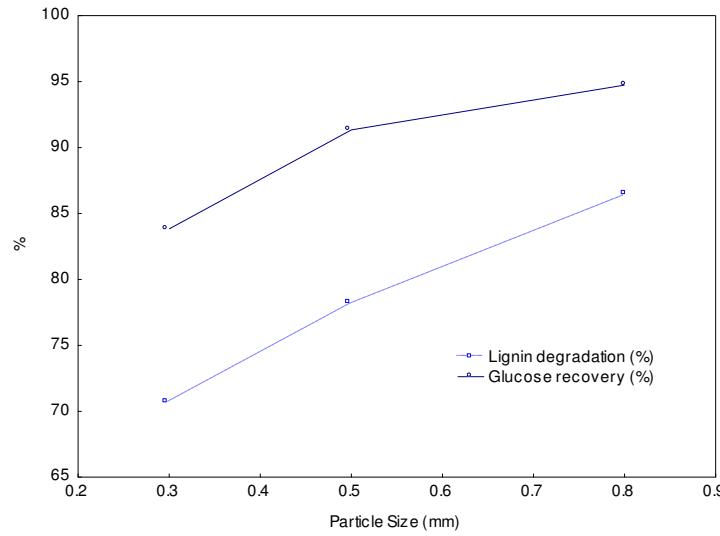
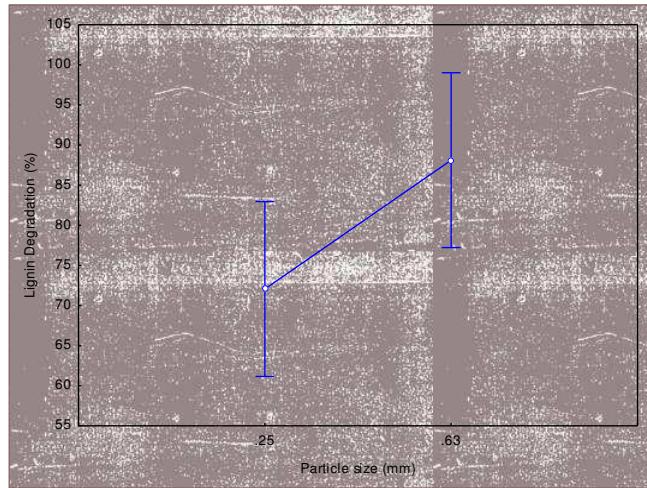
VISUALIZE THE RESULT

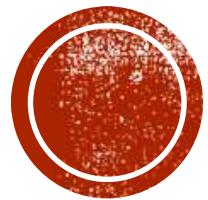
- Predicted response function (\hat{Y}) (read “*Y hat*”):
 - Predict the value of response
- Response surface:
 - Represent the relationship between predicted response function and factor
 - Is visualized in 3D, contour, single parameter;



SINGLE VARIABLES

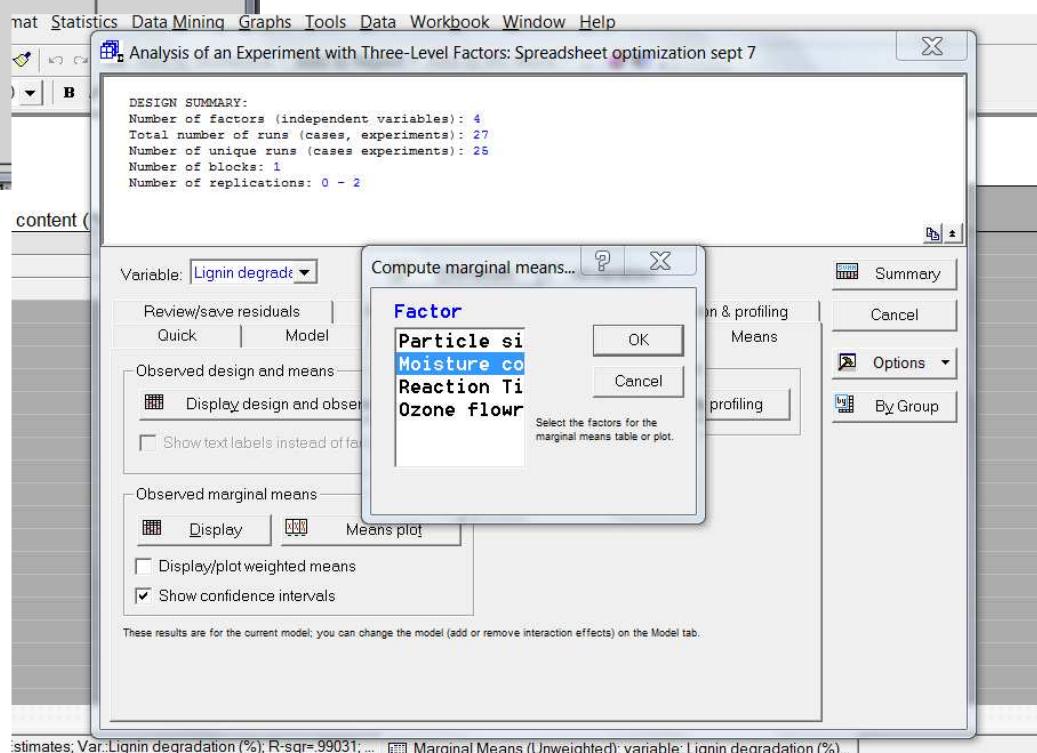
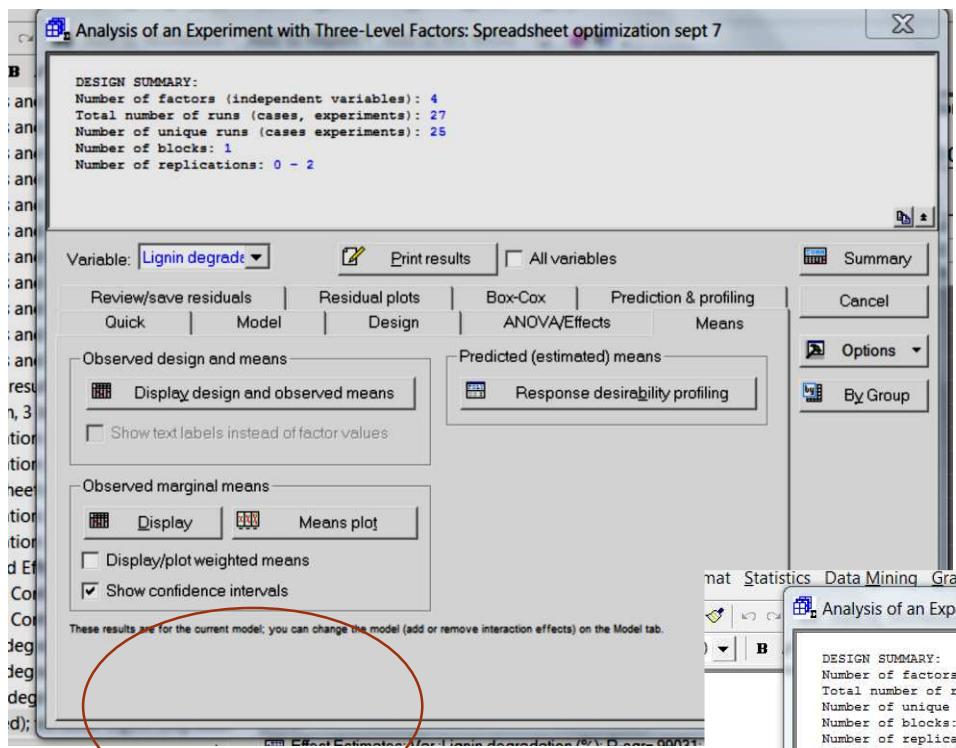
- The graph is plot the predicted Mean of value of process variables





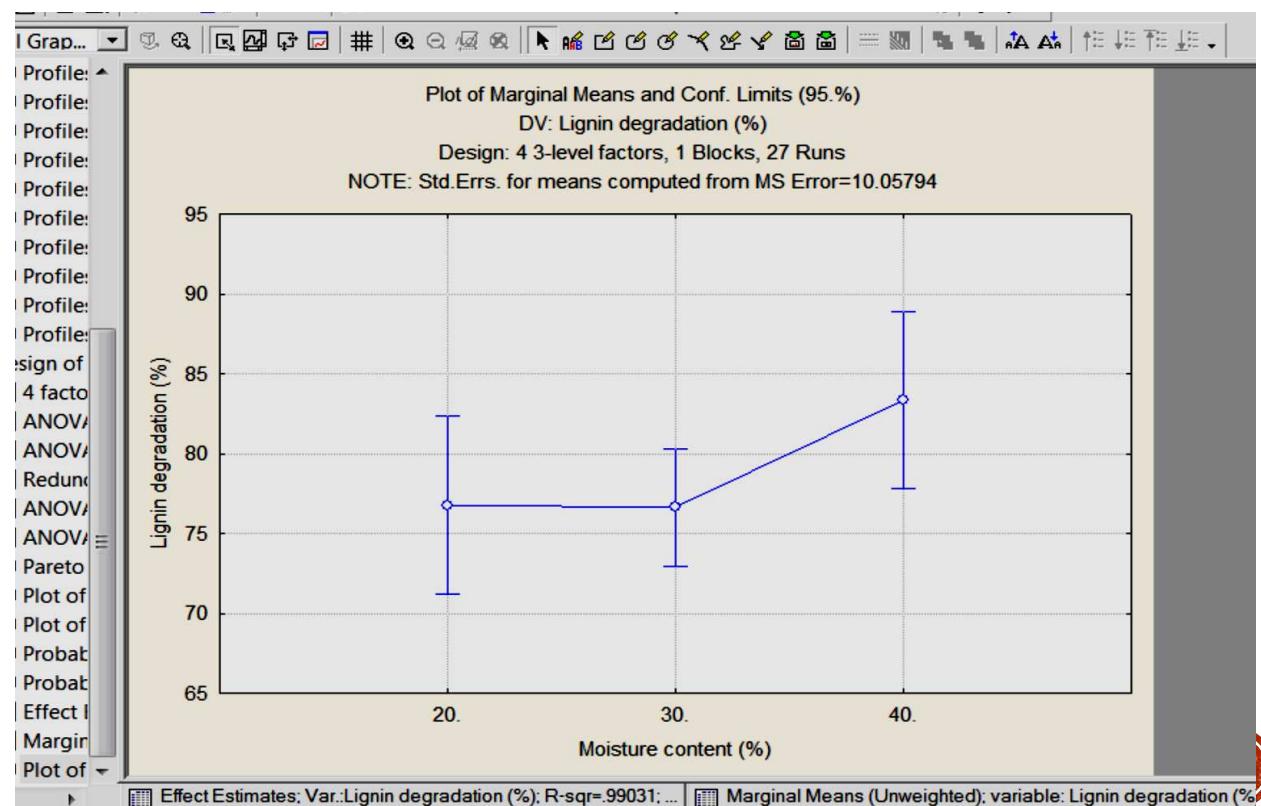
STATISTICA TUTORIAL 6

Mean Effect



Marginal Means (Unweighted); variable: Lignin degradation (%) (Spreadsheet optimization sept 7)
Design: 4 3-level factors, 1 Blocks, 27 Runs
NOTE: Std.Errs. for means computed from MS Error=10.05794

Moisture content (%)	Means	Pooled Std.Dev.	Overall Std.Dev.	N	Std.Err. for Mean	-95.% Cnf.Limt	+95.% Cnf.Limt		
	20.	30.	40.						
	76.75631	0.000000	13.80098	6	1.294729	71.18554	82.32708		
	76.62517	3.171426	7.92173	15	0.856745	72.93890	80.31145		
	83.36216	0.000000	2.74082	6	1.294729	77.79139	88.93293		



CONTOUR

Visualized the shape of the 3D response surface

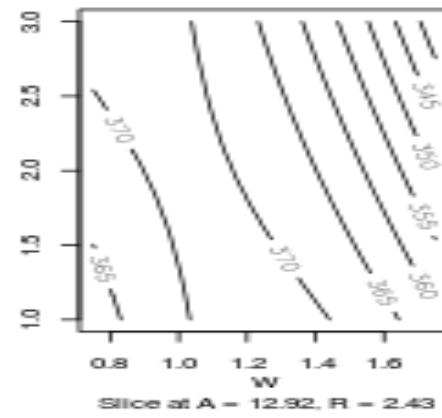
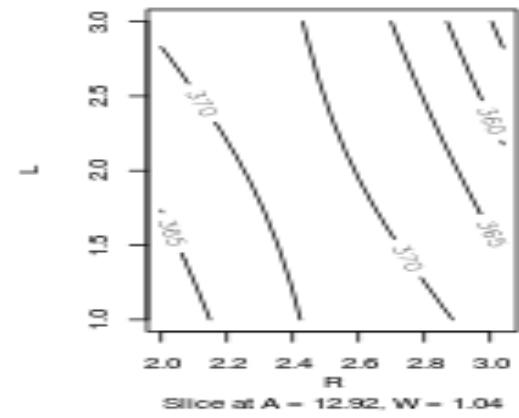
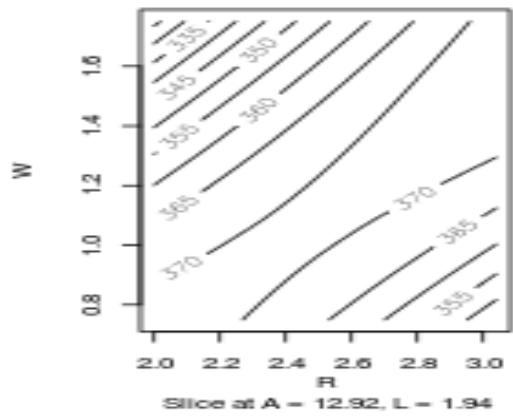
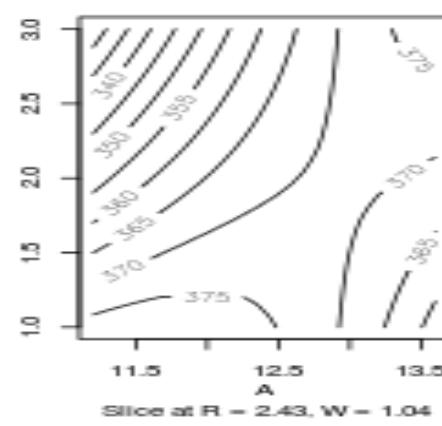
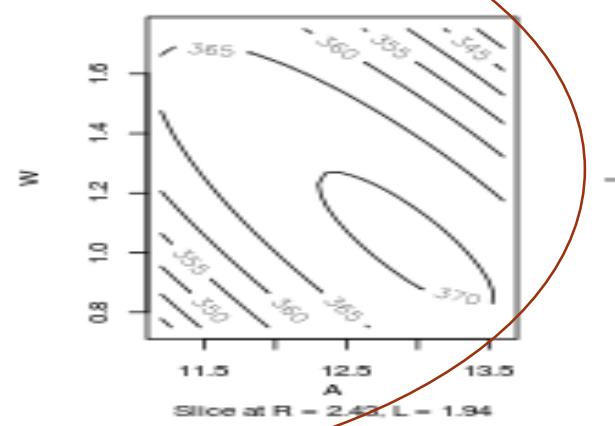
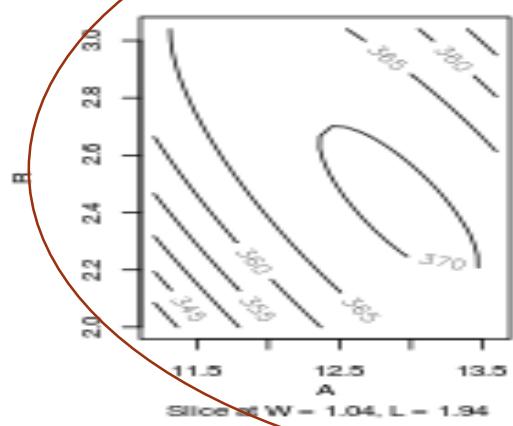
Line or curves (known as contour) represent the surface of response value are drawn on graph or plane whose coordinates represent the level of the factor.

The direction of contour can be used to explained the behavior of interaction for both parameter

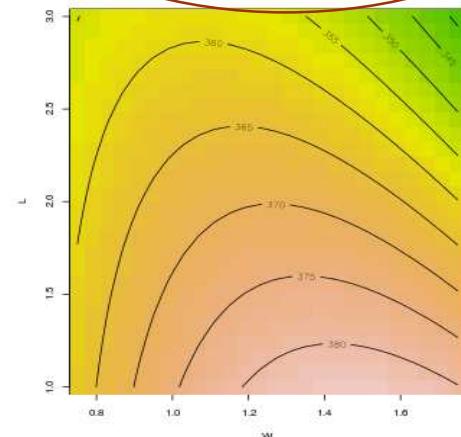
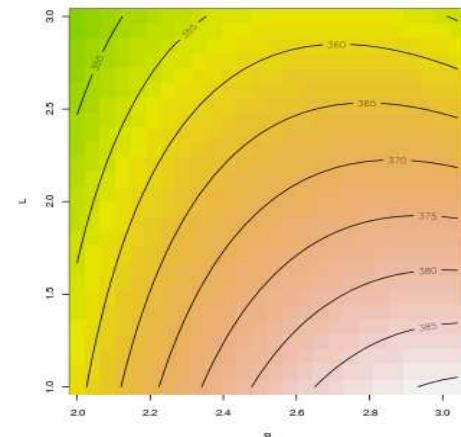
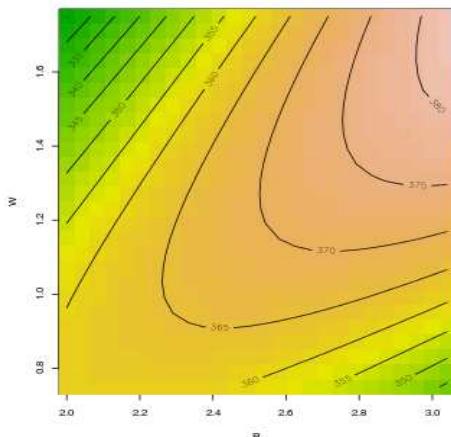
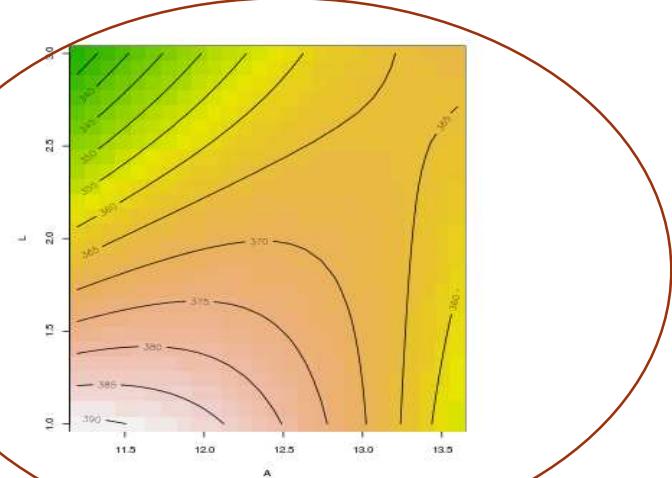
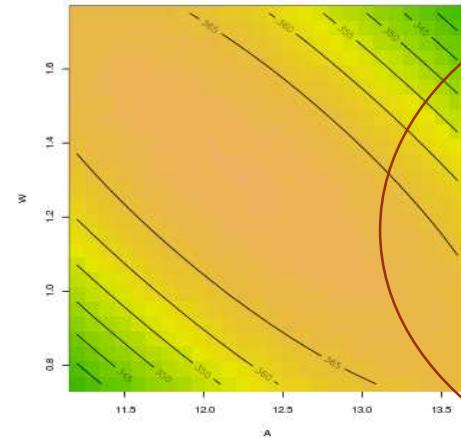
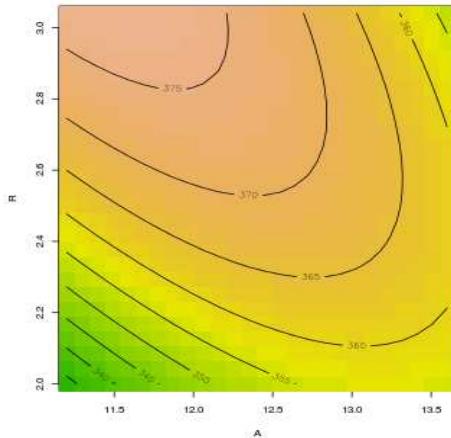
Ellipses, circular or saddle point



EXAMPLES (ELLIPSE)



EXAMPLES (SADDLE POINT)



EXAMPLE (CIRCULAR)

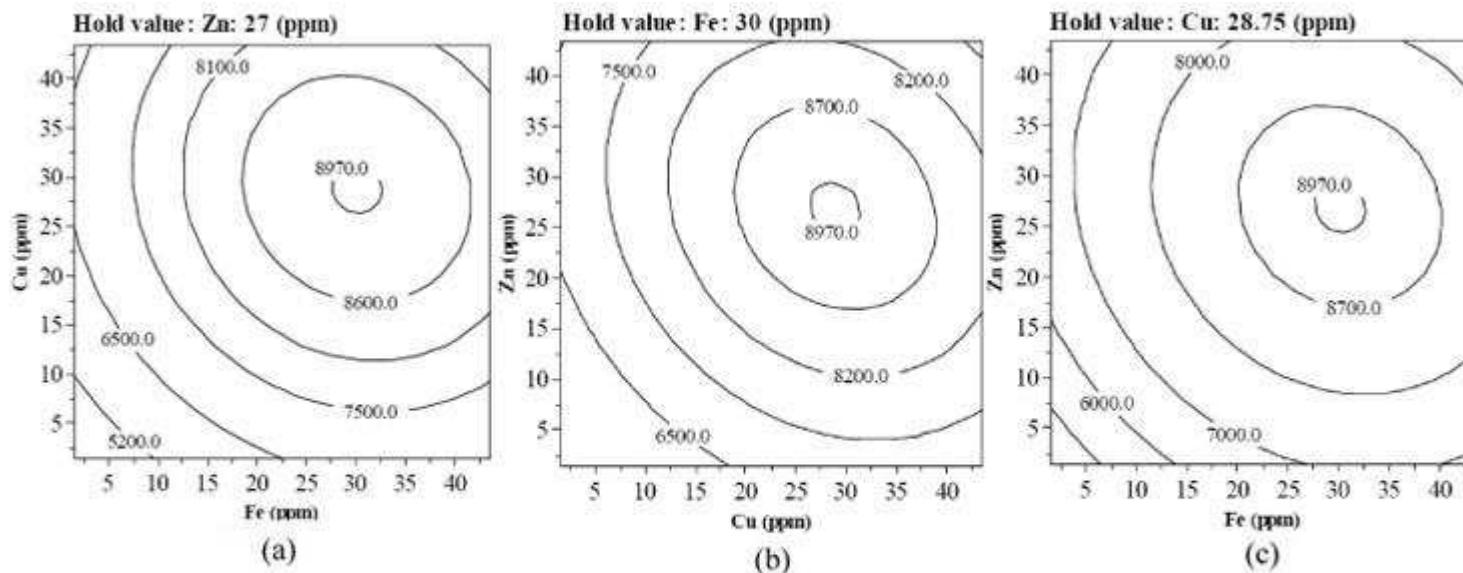
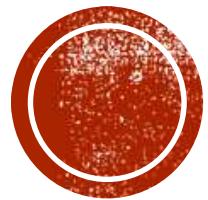


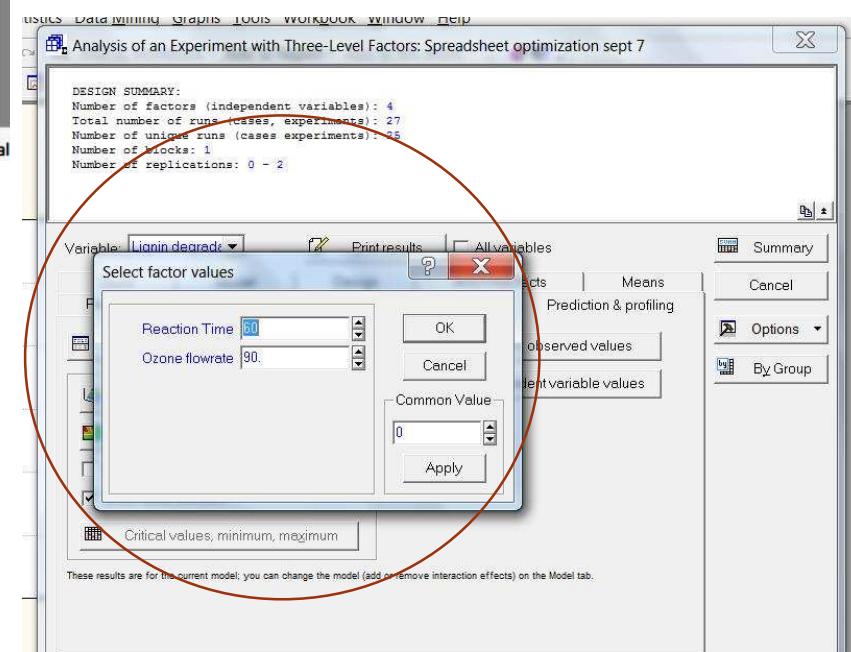
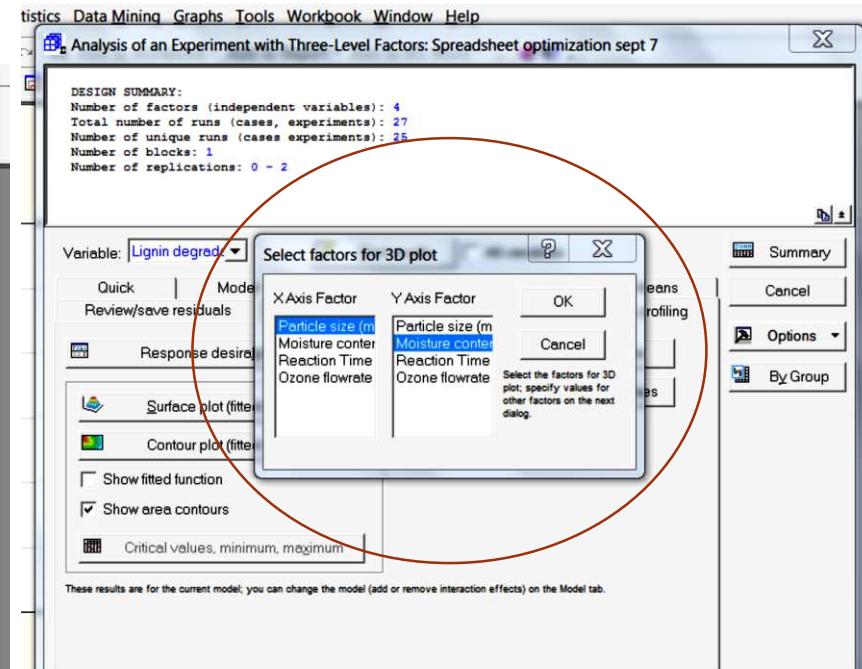
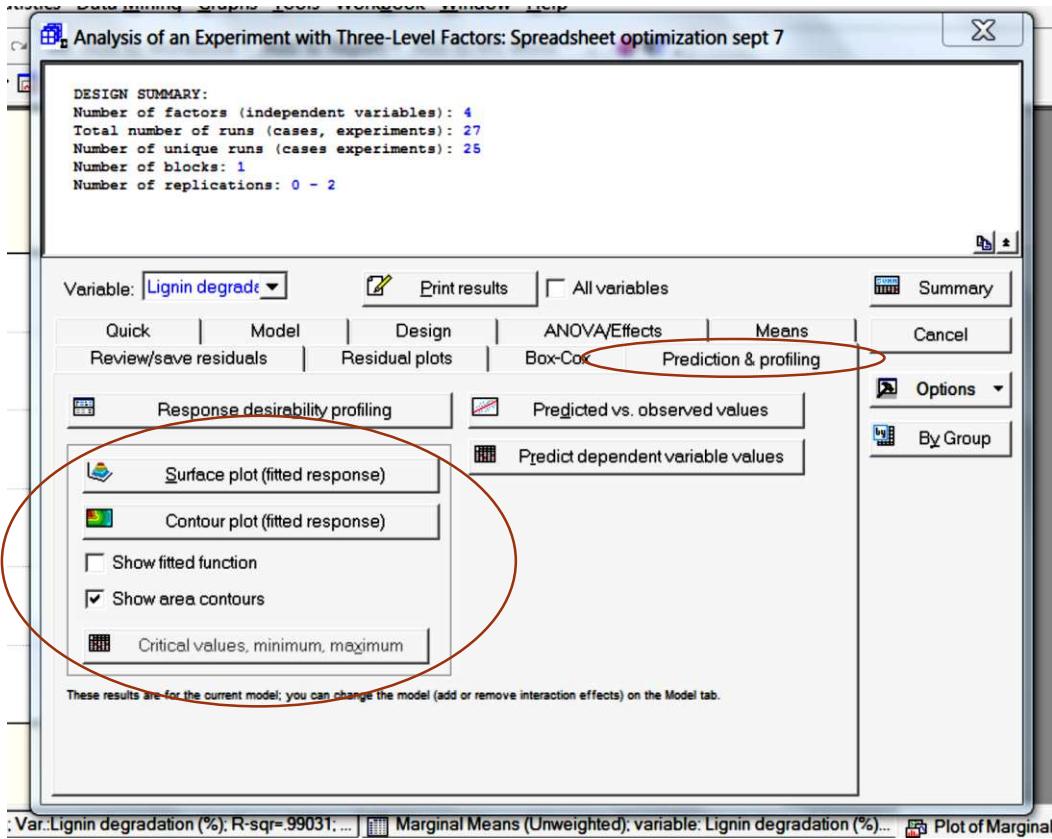
Figure 3: Contour plots (two-dimensional surface plots) of the model equation fitted to the data. (a) interaction of Fe^{3+} and Cu^{2+} concentration, (b) interaction of Fe^{3+} and Zn^{2+} concentration, (c) interaction of Cu^{2+} and Zn^{2+} concentration. (Cu^{2+} (copper, ppm); Fe^{3+} (iron, ppm); Zn^{2+} (zinc, ppm))





STATISTICA TUTORIAL 7

Contour plot



3D SURFACE PLOT

Shows the interaction between two process variables as function of factors.

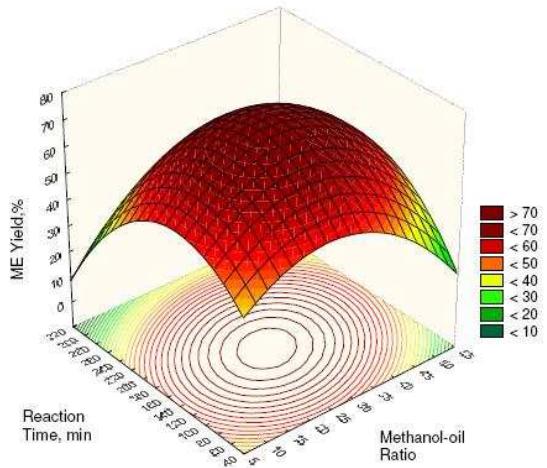
Shape

- Minimum: basin
- Maximum: hill
- Saddle: saddle shape



SURFACE

b)

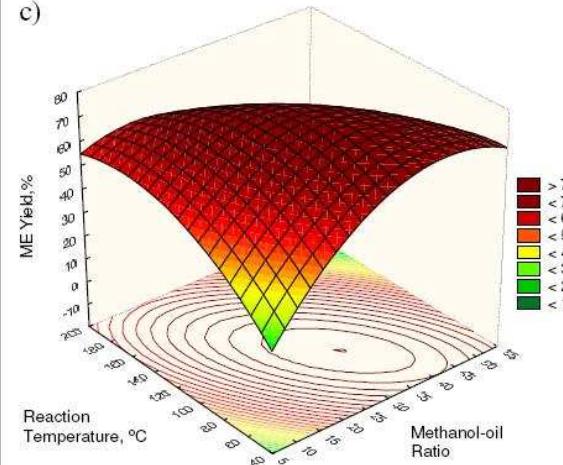


Hyperbola, maximum hill

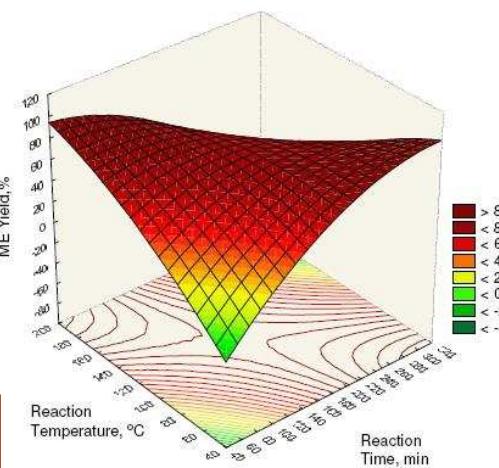
Saddle-shaped

Parabola, maximum hill

c)



f)



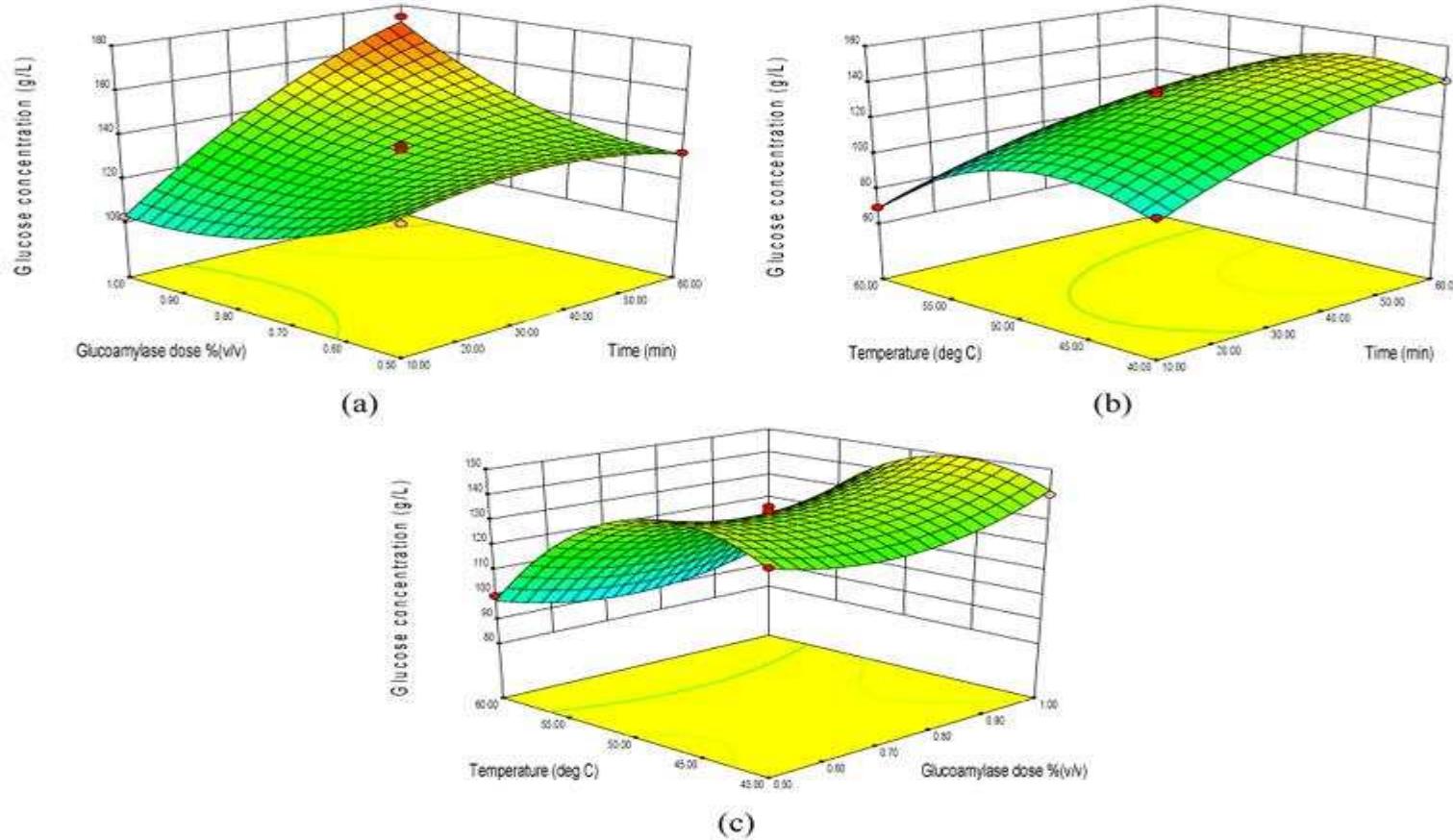
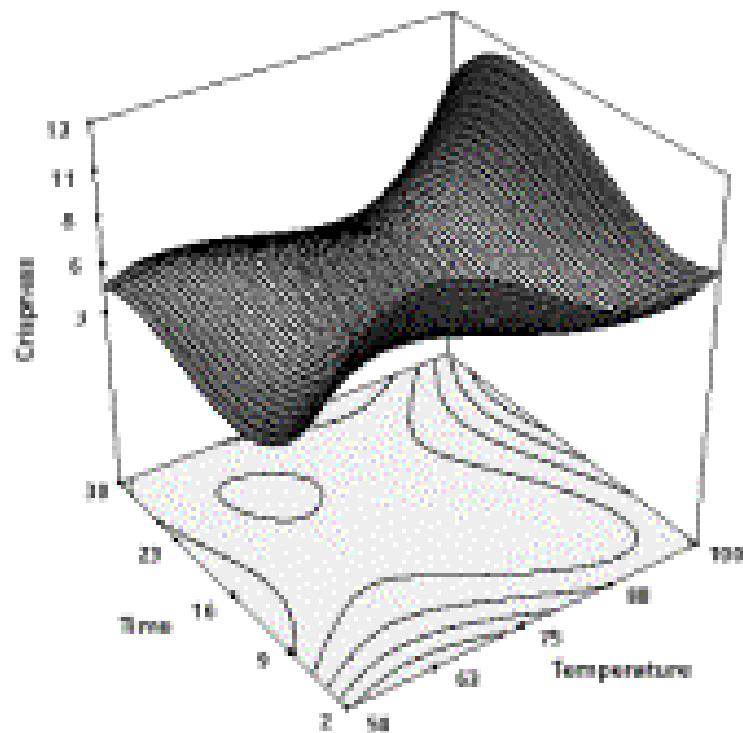


Figure 2(a)-(c): Surface plots for saccharification of sweet potato peel.

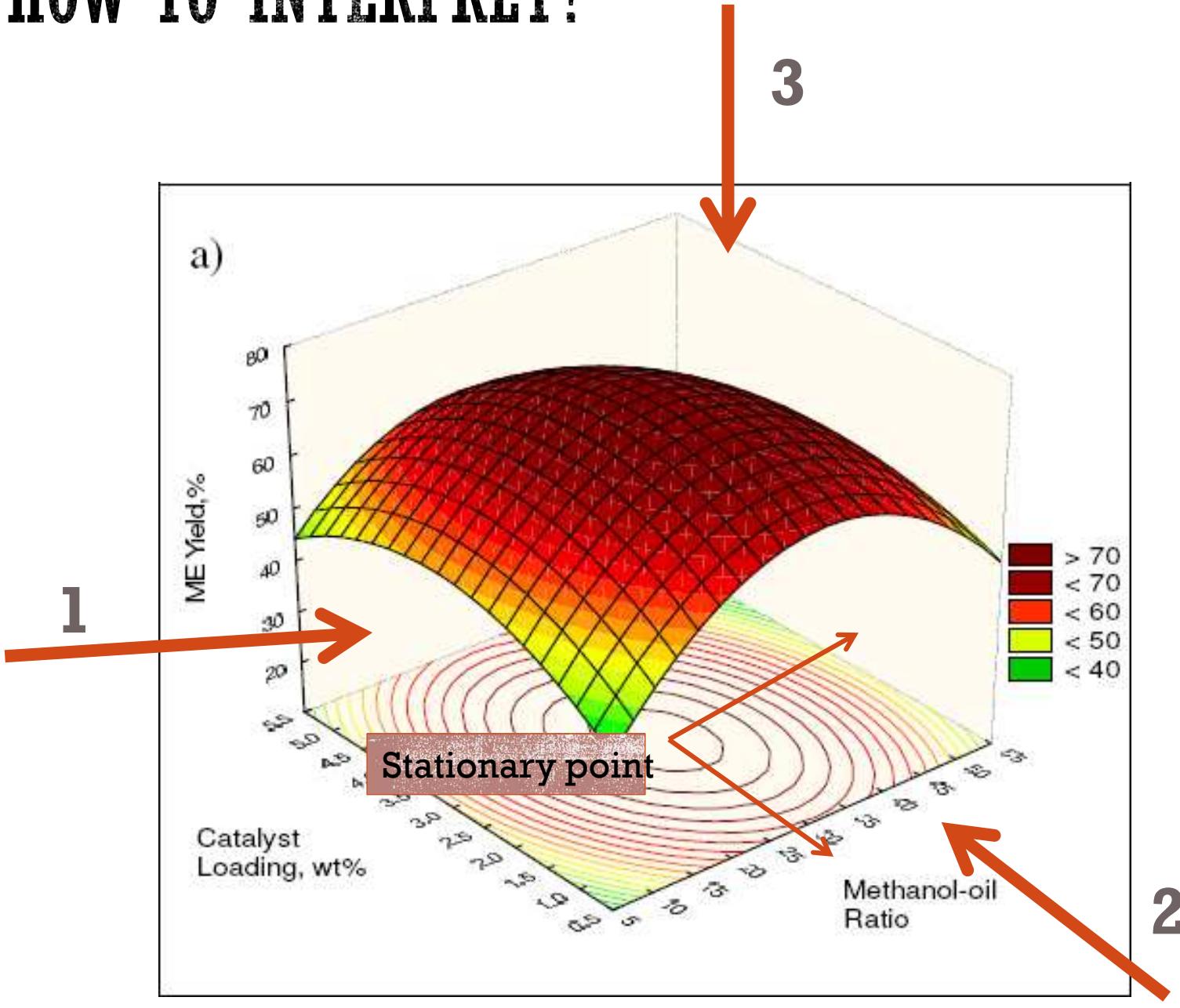
- (a) effect of glucoamylase dose, time and their reciprocal interaction on glucose concentration.
- (b) effect of temperature, time and their reciprocal interaction on glucose concentration.
- (c) effect of temperature, glucoamylase dose and their reciprocal interaction on glucose concentration.

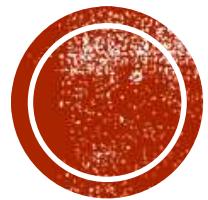


QUADRATIC INTERACTION



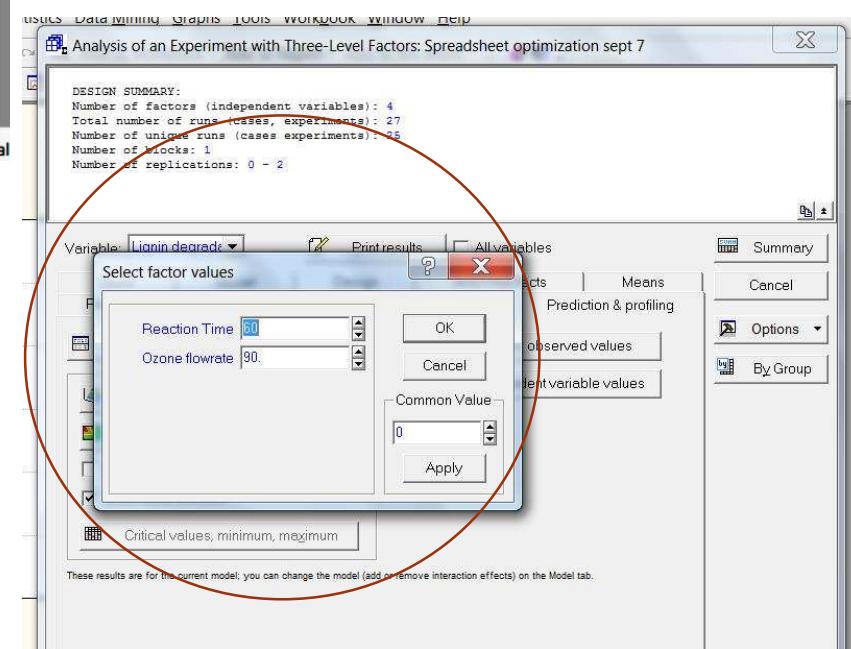
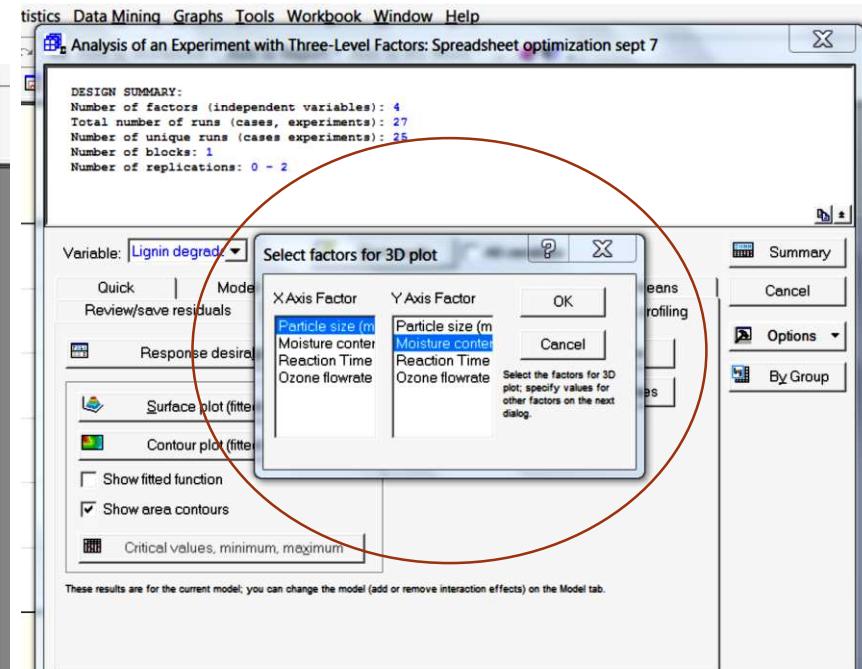
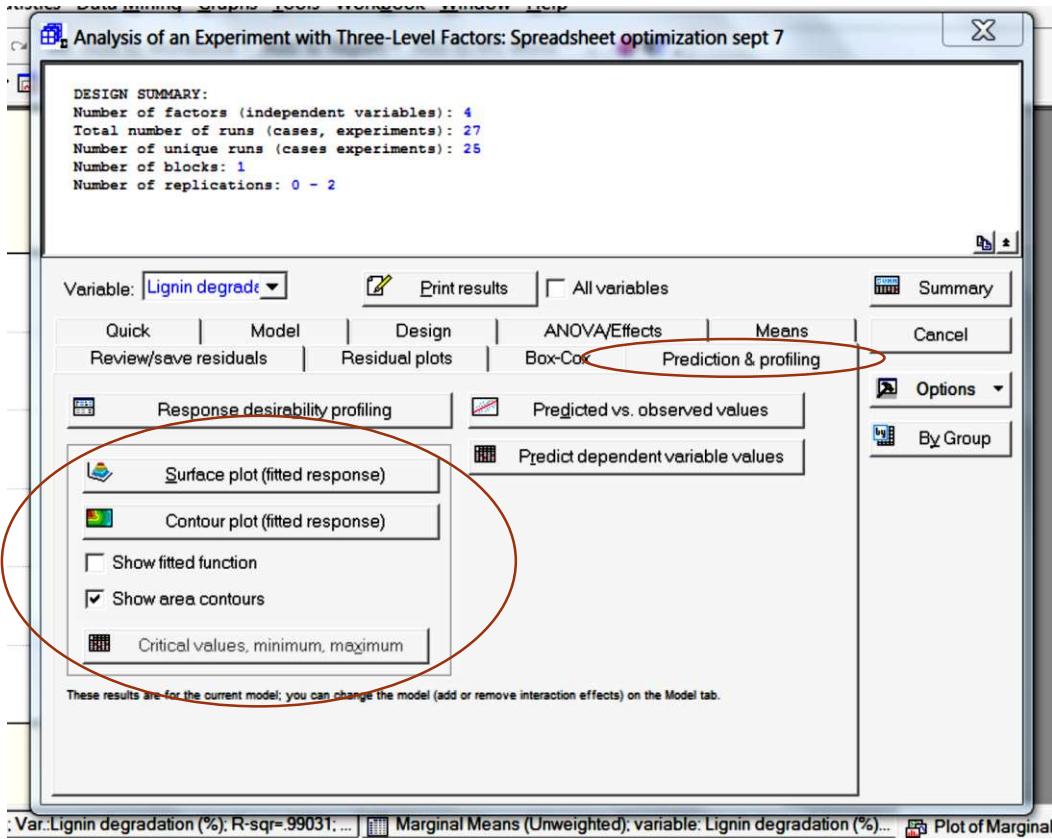
HOW TO INTERPRET?

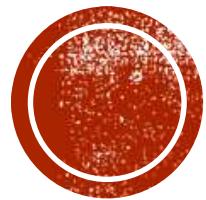




STATISTICA TUTORIAL 8

3D Surface





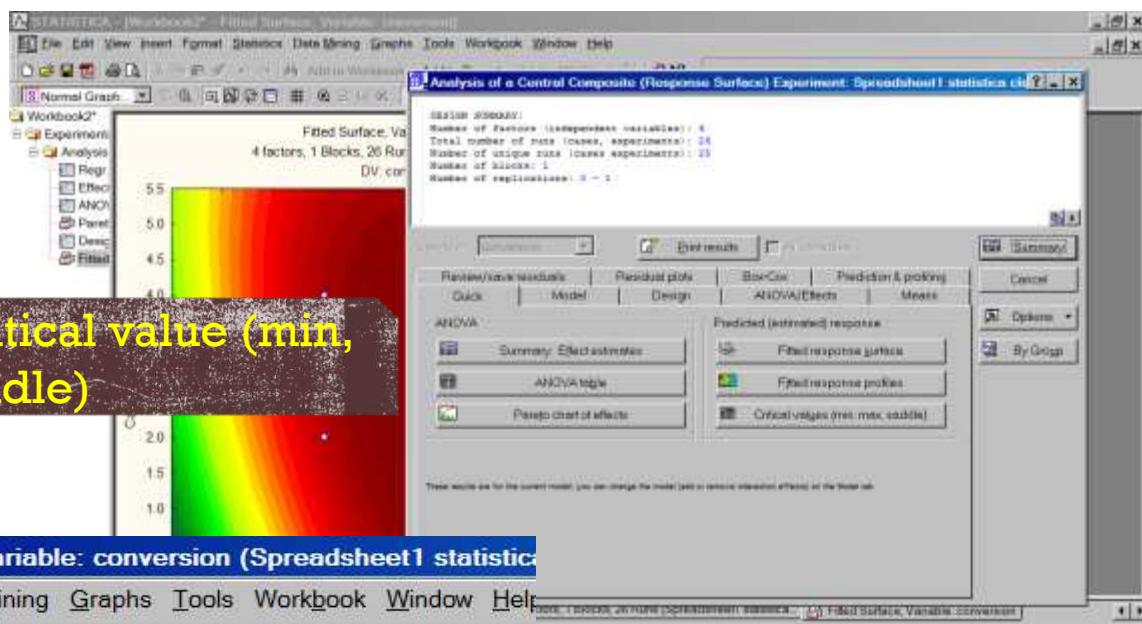
OPTIMIZATION

- Single response
- Multi response

OPTIMIZATION: SINGLE RESPONSES

STEP 1

Click quick tab → critical value (min, max, saddle)



Predicted
response



OPTIMIZATION: MULTI-RESPONSE

- Superimpose of two contour plot.

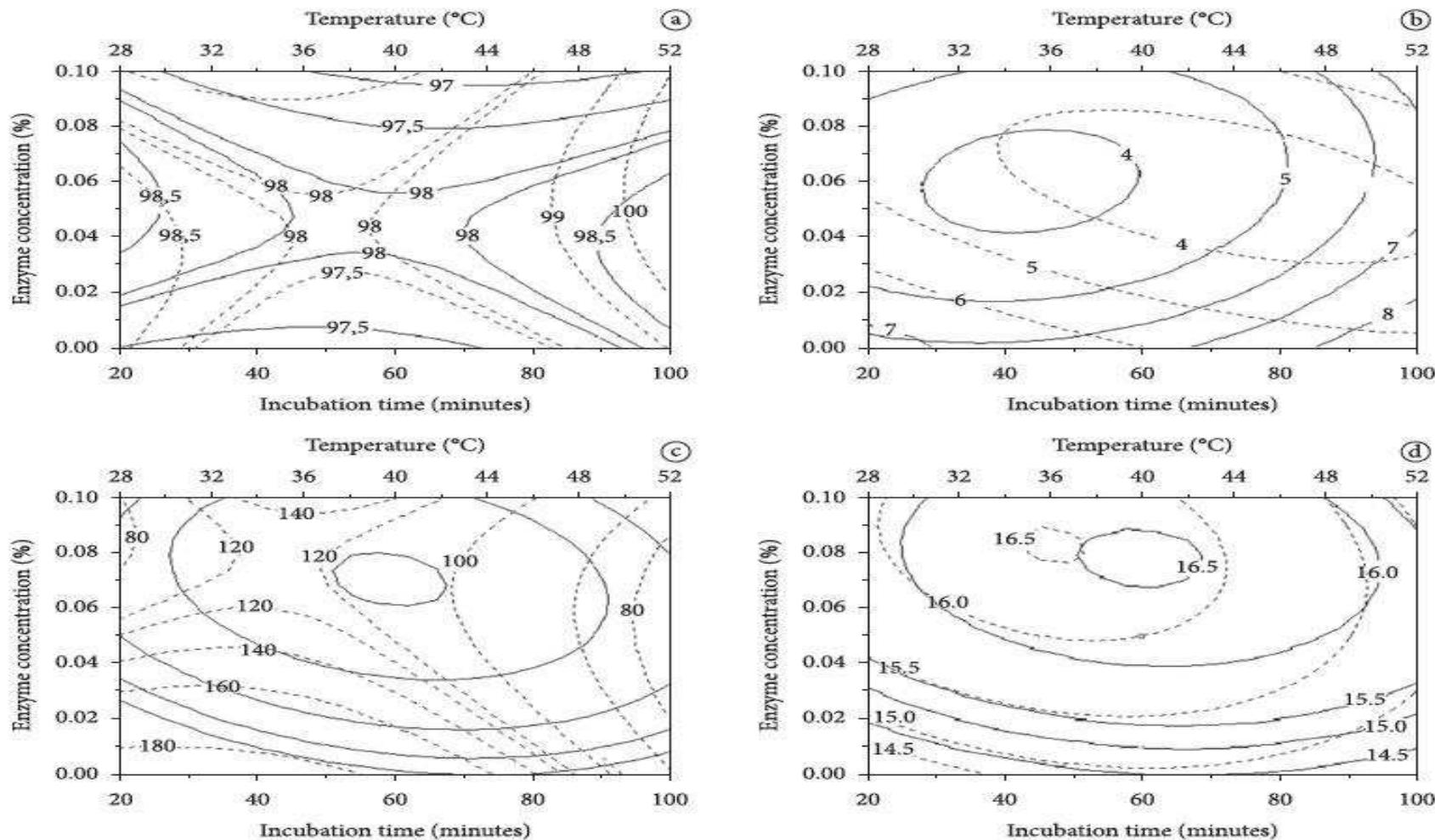


Figure 6. Optimum regions for the contour plots (a- L^* value; b- viscosity; c- turbidity; d- yield, ---- time, — temperature).

OPTIMIZATION: MULTI-RESPONSE VIA DESIRABILITY FUNCTION

A popular and established technique for simultaneous determinization of optimum settings of input variables that can determine optimum performance levels for one or more responses

Converting the estimated response model (Y) into individual desirability function (d) that are then aggregated into a composite function (D).

This composite function is usually a geometric or an arithmetic , which will be maximized or minimized, respectively.



Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7

DESIGN SUMMARY:
 Number of factors (independent variables): 4
 Total number of runs (cases, experiments): 27
 Number of unique runs (cases experiments): 25
 Number of blocks: 1
 Number of replications: 0 - 2

Variable: Lignin degrade ▾ Print results All variables

Quick | Model | Design | ANOVA/Effects | Means | Box-Cox | Prediction & profiling

Response desirability profiling Predicted vs. observed values

Surface plot (fitted response) Contour plot (fitted response)

Show fitted function Show area contours Critical values, minimum, maximum

These results are for the current model; you can change the model (add or remove interaction effects) on the Model tab.

Surf... Can... Light... Runs... Opt... By...

Add to report Add to MS Word X

Profiler: Spreadsheet optimization s...

Dep. vars: 7 11 Cancel

View 1 2

Quick | Save/Open | Options |

Set factors at

Mean values User specified

At optimum value Block: 1

Factor grid: Particle size (mm)

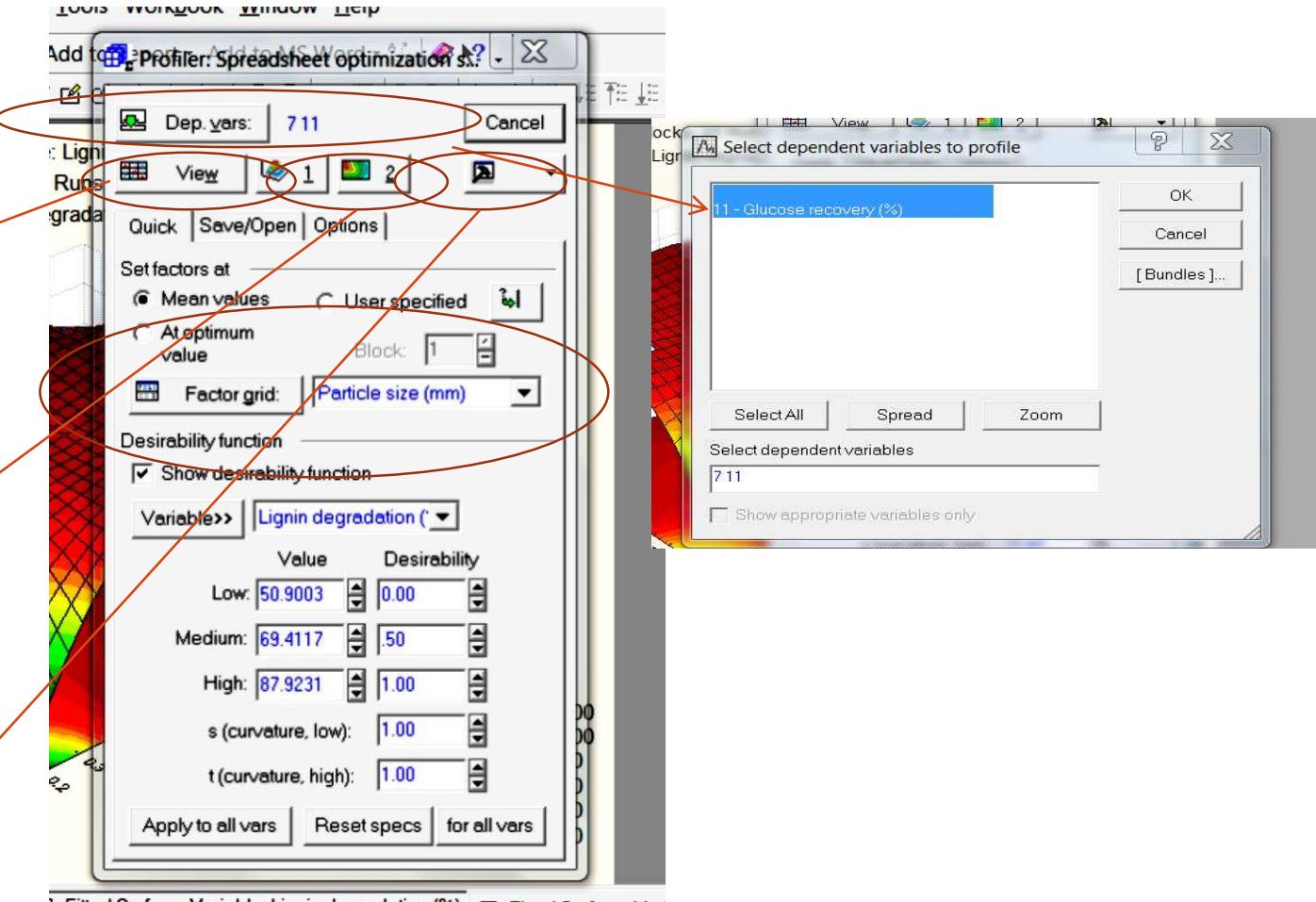
Desirability function

Show desirability function

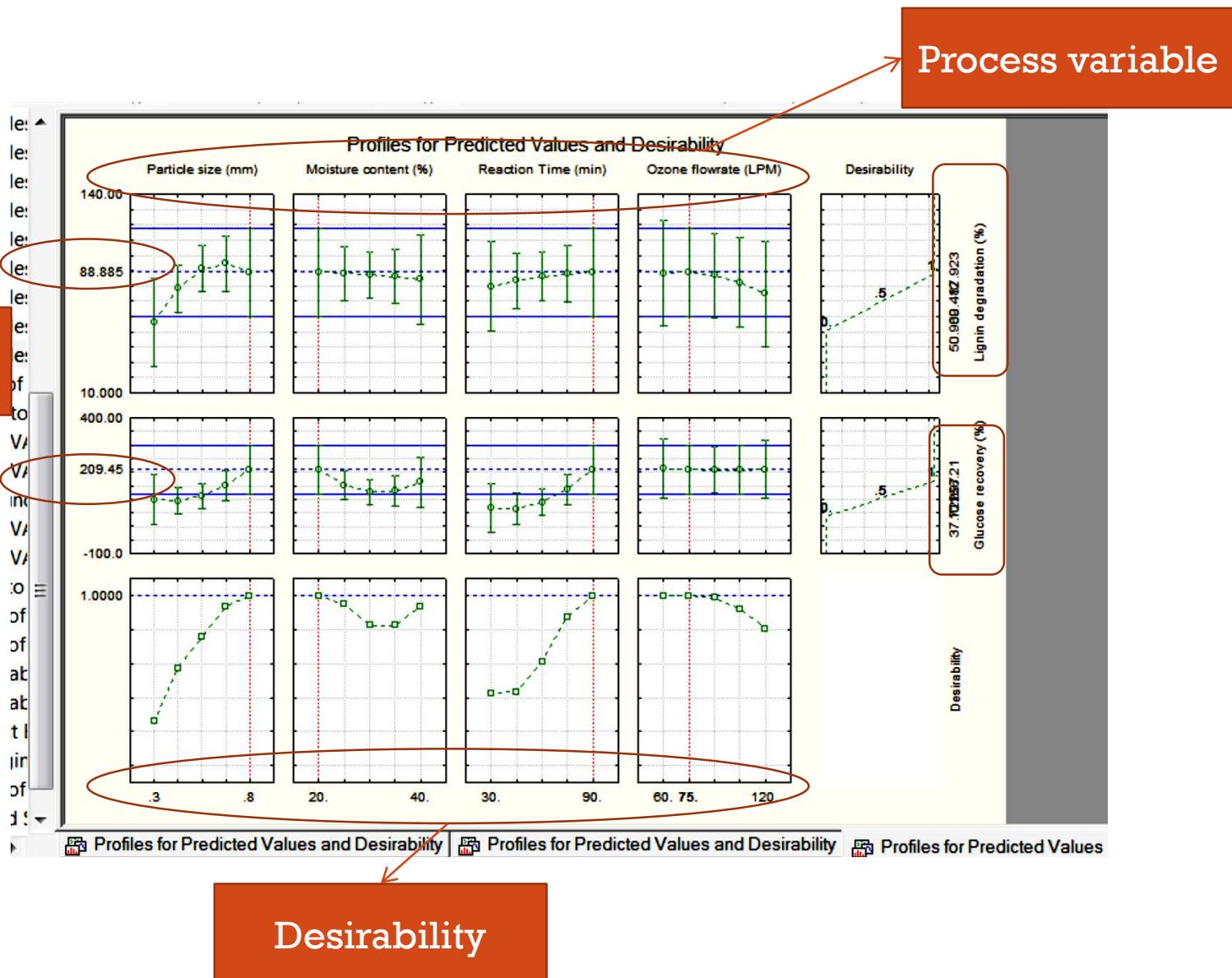
Variable>> Lignin degradation (%)

Value	Desirability
Low: 50.9003	0.00
Medium: 69.4117	.50
High: 87.9231	1.00
s (curvature, low):	1.00
t (curvature, high):	1.00

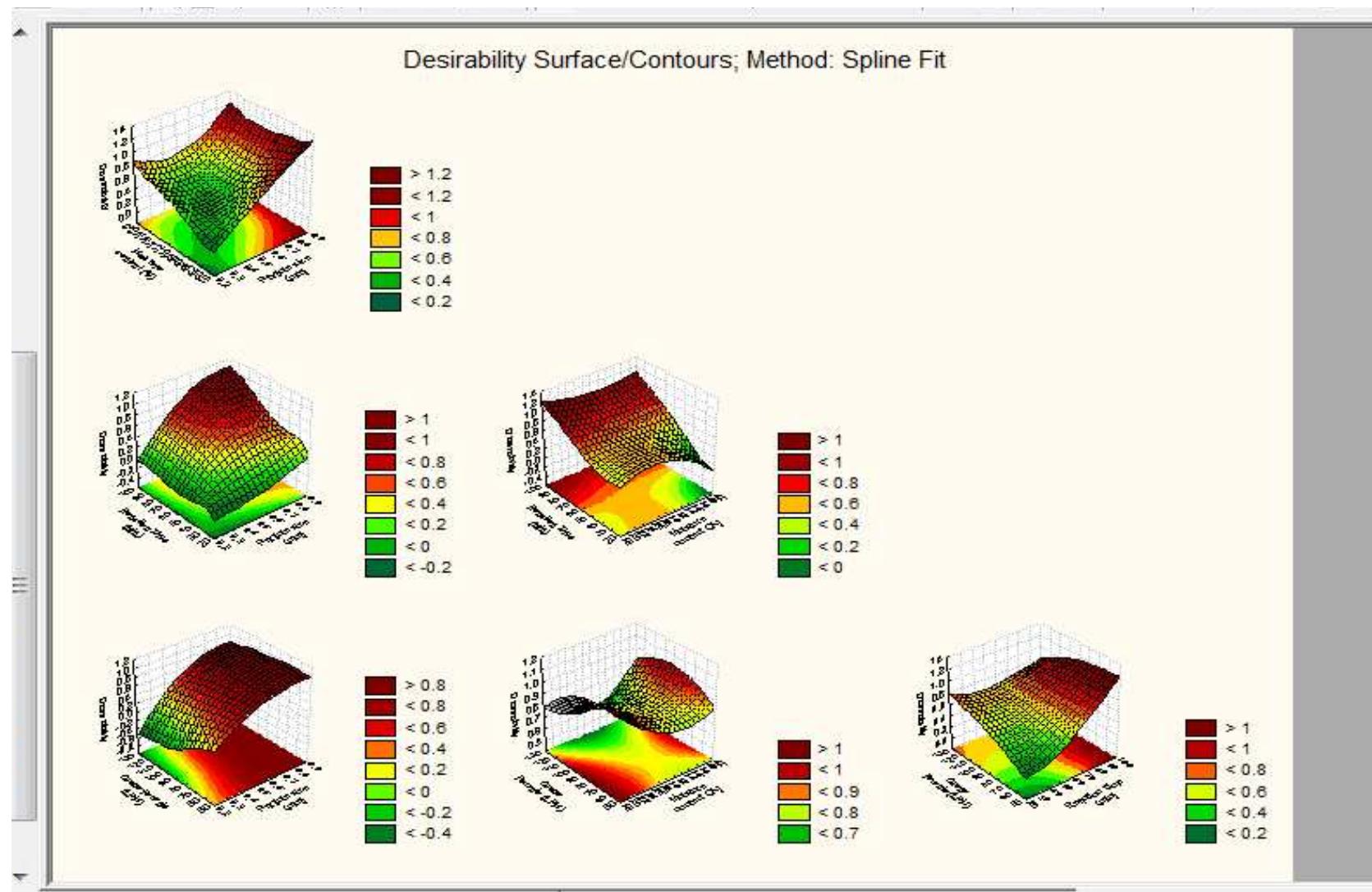
Apply to all vars Reset specs for all vars



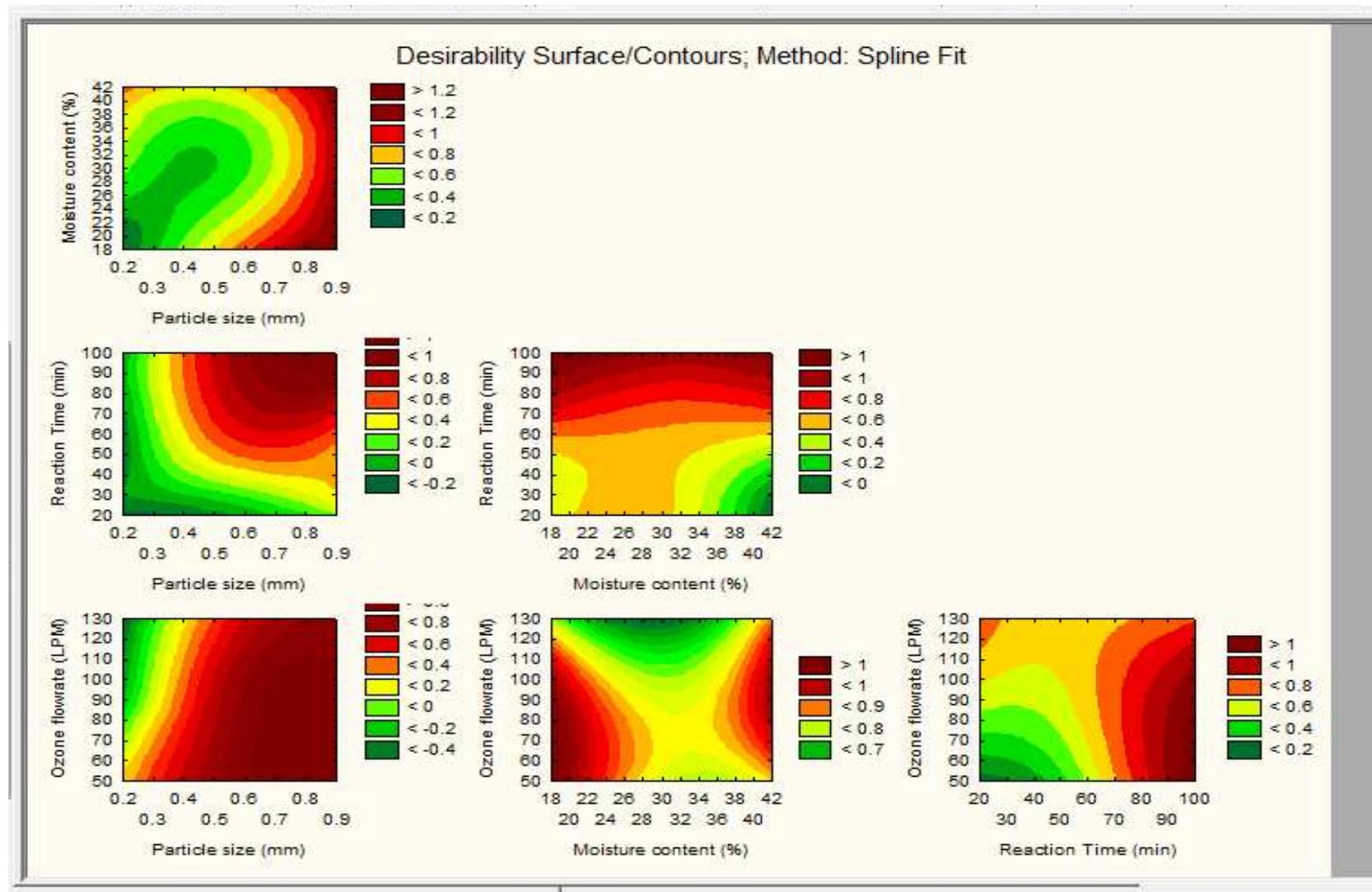
DESIRABILITY PROFILE



3D SURFACE PLOT



CONTOUR PLOT



CONCLUSION

Response Surface Methodology

- A powerful method for design of experimentation, analysis of experimental data, and optimization.

Advantages

- design of experiment, statistical analysis, optimization, and profile of analysis in one step
- Produce empirical mathematical model

Disadvantage

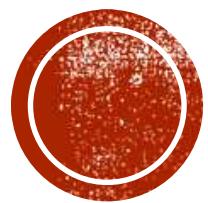
- The prediction only can be determined in range of study.



REFERENCES

- Montgomery, D. C. 1997. **Design and Analysis of Experiment.** Fifth Edition. Wiley, Inc., New York, USA.
- Brown, S. R. and Melemend, L. E. 1990. **Experimental Design and Analysis Quantitative Application in the Social Science.** Sage Publication, California. 74.
- Cornell J.A. 1990. **How to Apply Response Surface Methodology.** America Society For Quality Control: Statistic Devision . US.
- Haaland, P. D. 1989. **Experimental Design in Biotechnology.** Marcel Dekker Inc., New York.





**SLIDE CAN BE FOUND
AT**

<https://teknologimalaysia.academia.edu/DahliaOmar>