Design Of Experiment (DOE) & Response Surface Methodology (RSM)

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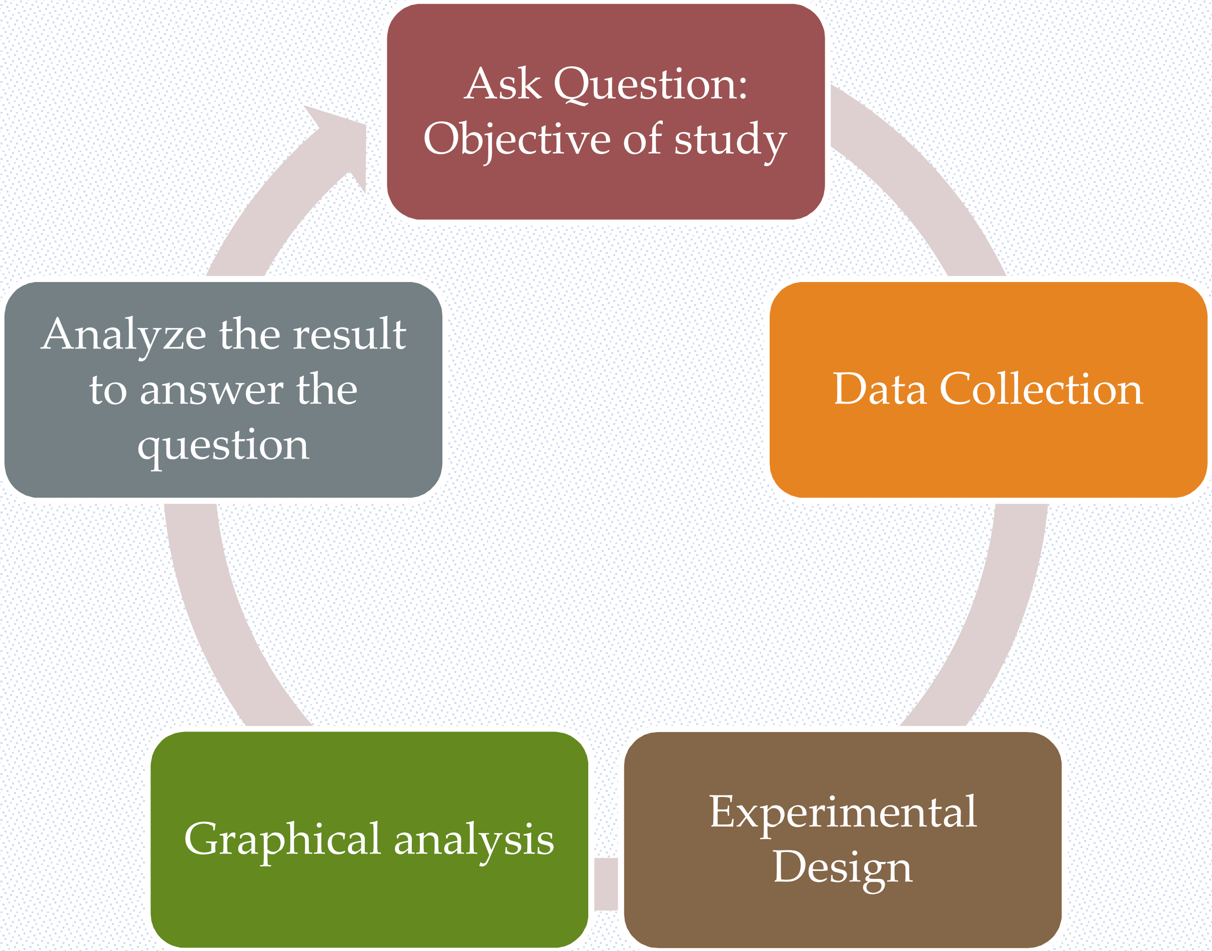
IF??


WE HAVE TO INCREASE EFFICIENCY BY 25%. LET'S ROLL THE DICE AND SEE WHICH LEVER WE PULL NOW

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	Statistical method
• One factor at atime (OFAT) • Time consuming • Cannot interpret the interaction between 2 or more variables	• 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.

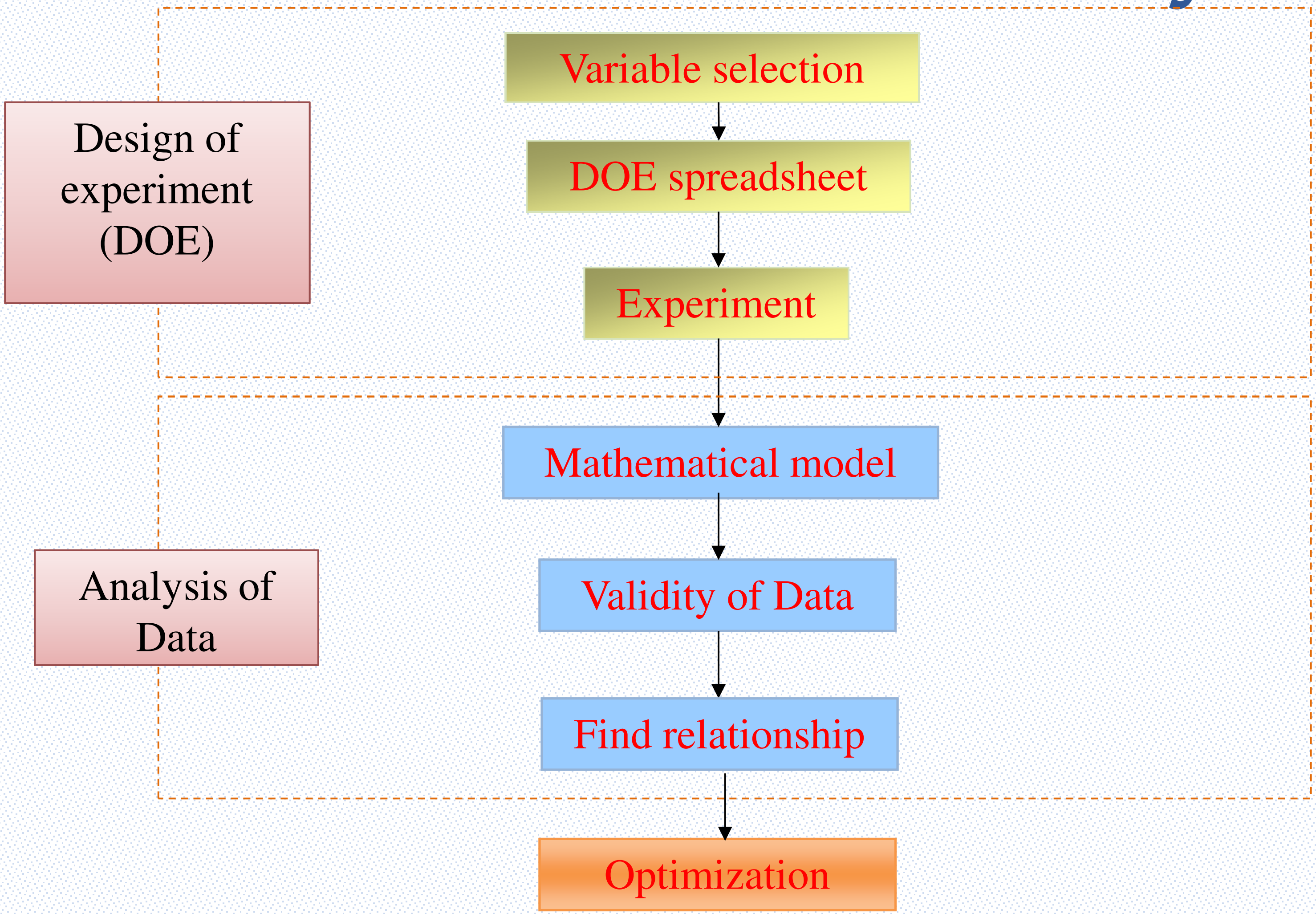
What is STATISTICA, Design Expert, MiniTab and etc?

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

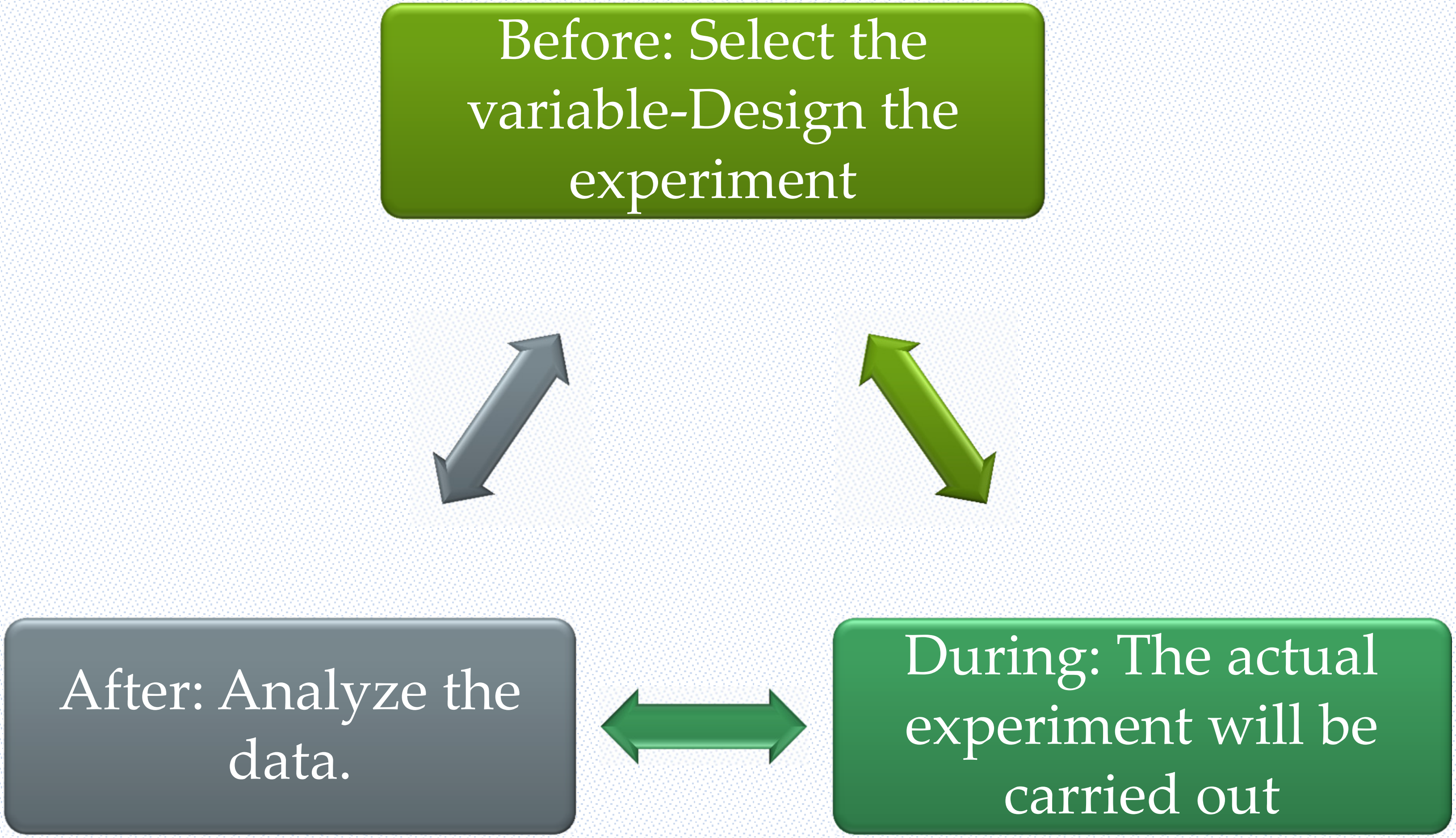
What DOE & RSM can do?

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

Flow of RSM study



Step in RSM study



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 factor 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 level at each factor as well as the no. of replicated experiment units represent the total no. of experiment.

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.

Objective

Screening

- To identify 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

Optimization

- To identify the best process performance, interaction effect, and significant of factors
- Type of design: CCD or BBD

1) Selecting the Parameter

Factors:

- Process conditions influence the value of response variable
- Can be qualitative or quantitative
- Qualitative-**blocking** variables
- Quantitative –normally considered in RSM

Responses:

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

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)-($-\alpha$, -1,0,+1, $-\alpha$) second or higher order

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

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 thea 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

Rotatable

- Fractional factorial (two level) = 2^{k-m}, m<k
  - ½= 2^{k-1}
  - 1/4= 2^{k-2}
  - 1/8= 2^{k-4}
- Fractional factorial (three level) = 3^{k-m}, m<k

Orthogonal

Matrix Arrangement (2-level)

2-level fractional fractorial 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

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

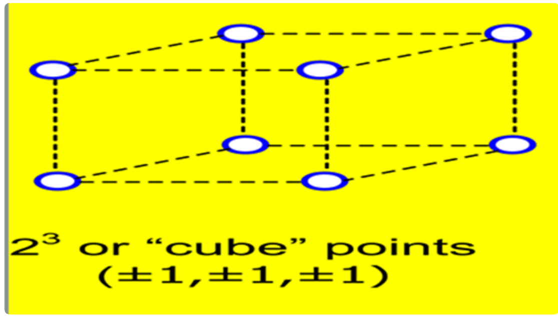
3**(4-1) fractional factorial design, 3 blocks, 27 runs (Spreadsheet1)

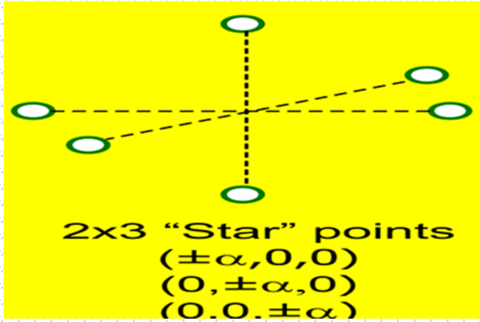
	Block	A	B	C	D
1	**1**	**1**	**0**	**-1**	**-1**
2	1	0	1	0	1
3	1	-1	-1	0	1
4	1	0	1	-1	-1
5	1	1	0	0	1
6	1	-1	-1	1	0
13	2	0	-1	1	-1
14	2	0	-1	0	0
15	2	0	-1	-1	1
16	2	1	1	0	0
17	2	-1	0	-1	1
18	2	1	1	1	-1
19	3	0	0	1	1
20	3	0	0	0	-1
21	3	-1	1	-1	0
22	3	0	0	-1	0
23	3	1	-1	1	1
24	3	1	-1	0	-1
25	3	1	-1	-1	0
26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

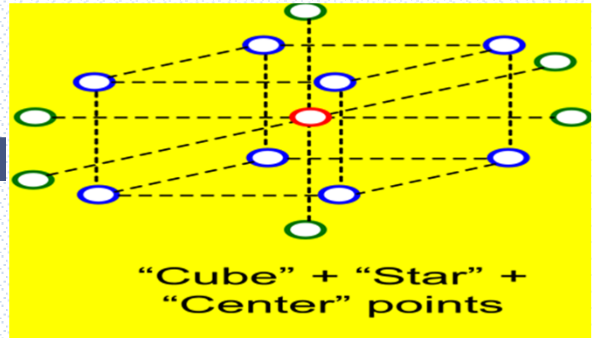
3**(4-1) fractional factorial design, 1 block , 27 runs (Spreadsheet1)

	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
18	0	1	1	0
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 ±α

$n_0 \geq 1$ "center" point replicates ➔ coded as 0

Providing the estimate of pure error and curvature

=

Total run= 2^k+2k+n_o or $2^{k-m}+2k+n_o$

CCD

The most common design (for the 2^{nd} 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_o (>0) and $a=4\sqrt{M}$, $M=2^k$

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 Matrix 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
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	-1.00	-1.00
0.00	1.00	0.00	1.00
0.00	1.00	1.00	0.00
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	-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	-1.00	0.00
1.00	0.00	-1.00	0.00
-1.00	0.00	1.00	0.00
1.00	0.00	1.00	0.00
0.00	-1.00	0.00	-1.00
0.00	1.00	0.00	-1.00
0.00	-1.00	0.00	1.00
0.00	1.00	0.00	1.00
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

Runs	Manipulated Variables						Responses
	X_1		X_2		X_3		
	Operating temperature,T(°C)	Level[b]	Molar Ratio (meOH: oil)	Level[b]	Reaction time,t (h)	Level[b]	Yield, Y_1 (%)
1	50	-1	3	-1	2	-1	91.90
2	50	-1	3	-1	4	+1	84.60
3	50	-1	10	+1	2	-1	65.15
4	50	-1	10	+1	4	+1	95.95
5	70	+1	3	-1	2	-1	63.90
6	70	+1	3	-1	4	+1	94.95
7	70	+1	10	+1	2	-1	87.60

Addition note

Randomisation

- The order of run is random
- Can protect us from bias caused by unware 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.

1. Insert the complete data into DOE
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

1) Complete data

Insert the collected data into the software.

(refer to tutorial 2)

- First Order polynomial
- Second Order polynomial

…

2) 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

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

Y	**: predicted response (response function**
β_o	***: intercept coefficient (offset)***
β_1 , β_2 **and** β_3	***: linear terms (first order)***
X_1 , X_2 **and** X_3	**: uncoded independent variables**

Second order model (polynomial)

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

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

Y	**: predicted response (response function**
β_o	***: 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**

- Regression analysis
- ANOVA
- Hypothesis testing

• • •

3) 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
		$\Sigma Y_u = 50.7$ $\bar{Y} = 6.3375$		$\Sigma(Y_u - \bar{Y})^2 =$ 4.59875	$\Sigma(\hat{Y}_u - \bar{Y})^2 =$ 4.4225	$\Sigma(Y_u - \hat{Y}_u)^2 =$ 0.17625

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

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

$$SST=\sum(Yu-\hat{Y})^2 =0.17625$$

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=SSR/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

- adj R^2=1- Msresidual/(SST/N)-more smaller more better

How to interpret the R^2 ?

R2 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-value = $MS_{regression}/MS_{residual} = (SSR/DF_{regression})/(SSE/DF_{residual})$

where:
SSR= sum of square of regression
SSE= sum of square of error/residual
$DF_{regression}$=degree of freedom of regression
$DF_{residual}$=degree of freedom of residual

- F table $=F(p-1, N-p, a)$
  - $p-1$:$DF_{regression}$
  - $N-p$:$DF_{residual}$
  - *N=total exp*
  - *P=no of term in fitted model*
  - *a*-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.

The surface is plane

Conclusion: The null is

True: Fcal< F table, cannot be rejected

OR

Rejected: F cal>F table.

The surface is twisted.

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

4) 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-value = coefficient/ 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_o can be rejected at a 1% significance level ➔ "*convincing*" evidence that the H_A is true.

- If a *p*-value is 0.01<p-value≤0.05, then the H_o can be rejected at a 5% significance level ➔ "*strong*" evidence in favor of the H_A.

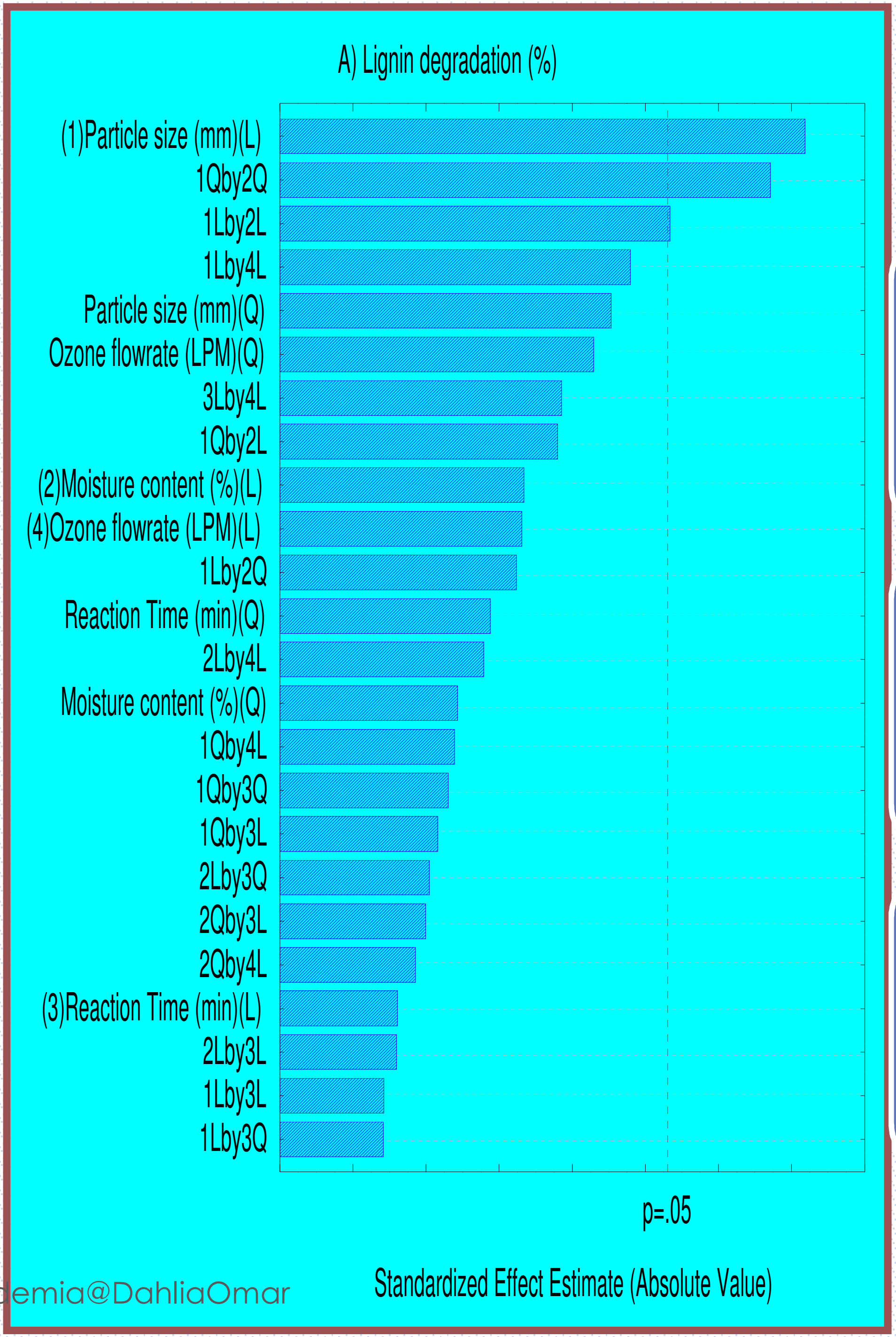
- If a *p*-value is 0.05<p-value≤0.10, then the H_o can be rejected at a 10% significance level. ➔it is in a "*gray area/moderate*"

- If a *p*-value is >0.10, then the H_o 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



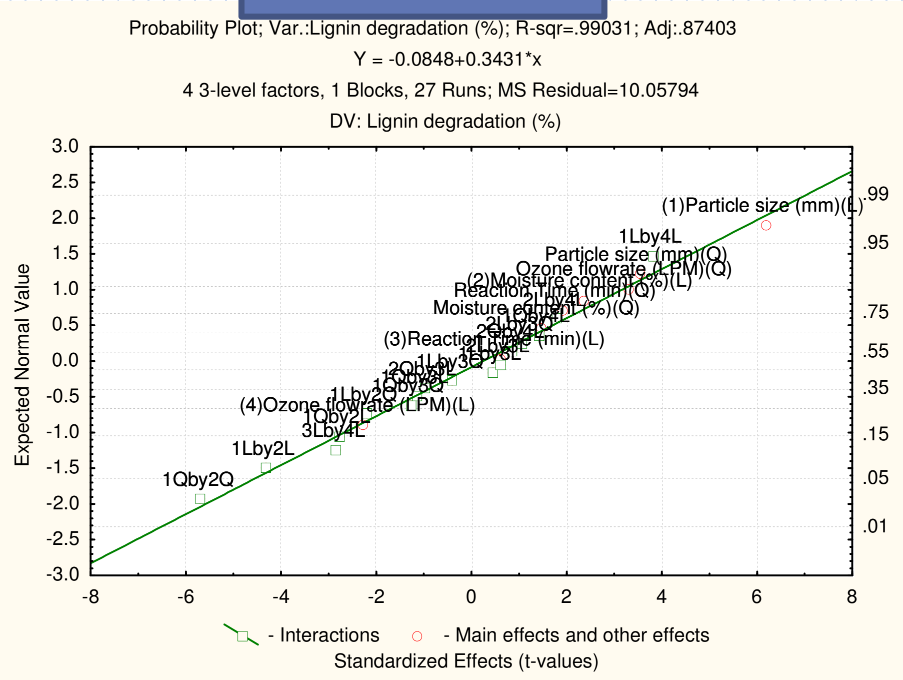
ANOVA effect estimates are sorted from largest to small value

The magnitude of each effect is represented by a column

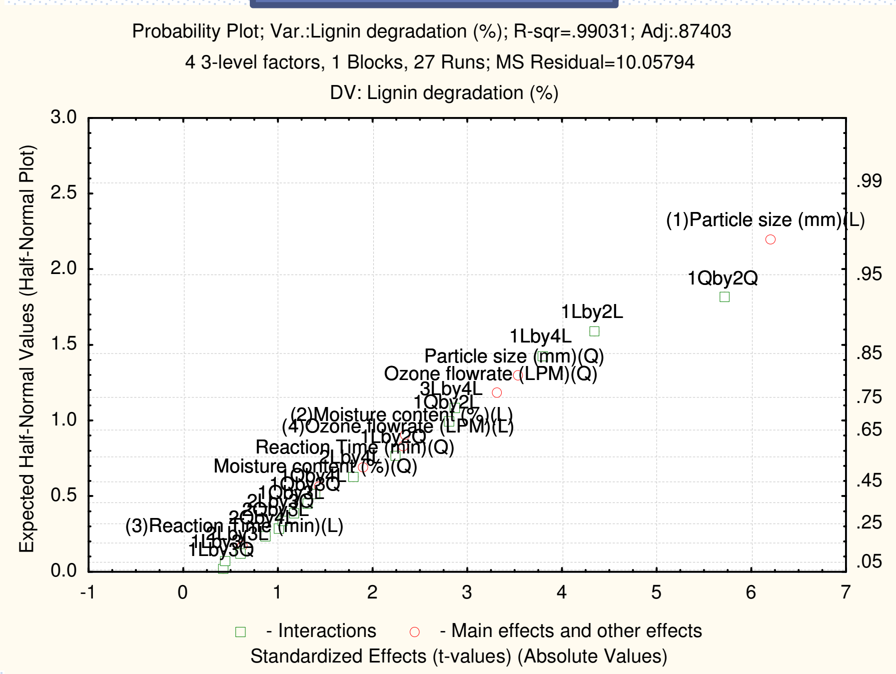
A line going across the column indicates how large and effect has to be statistically significant

Probability plot

Normal



Half- Normal



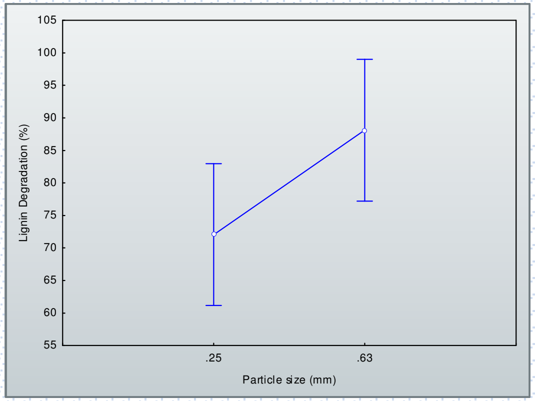
- To assess how closely a set of observed values follow a theoretical distribution
- if all values fall onto stright line, the residual follow the normal distribution
- The parameter were rank –ordered.

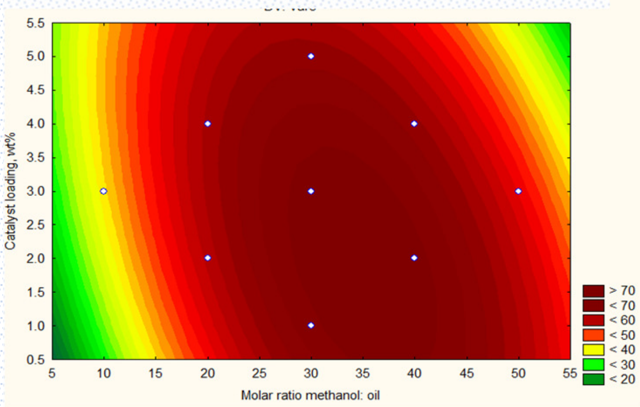
- Contour plot
- Surface (3D)
- Single parameter

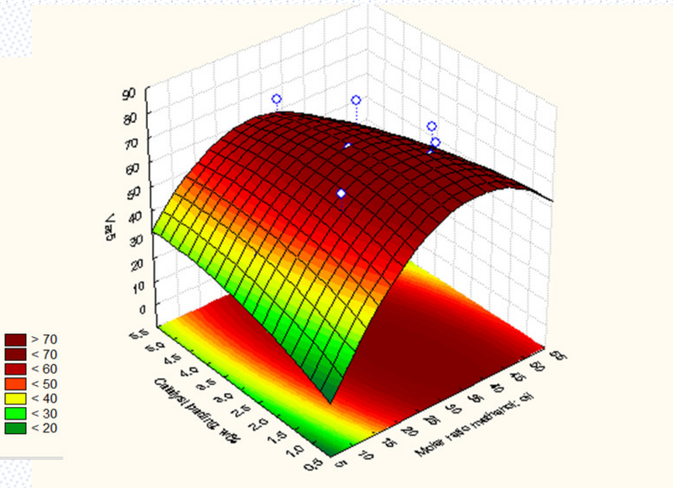
5) Interaction/influence of process variables

Visualize the result

- Predicted response function (Ŷ) *(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;

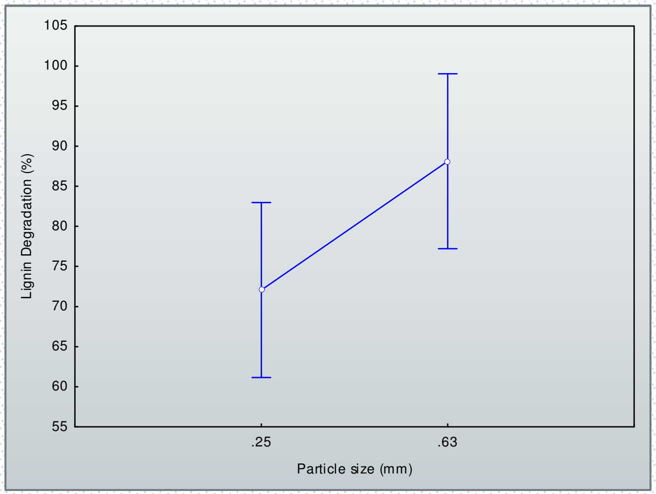


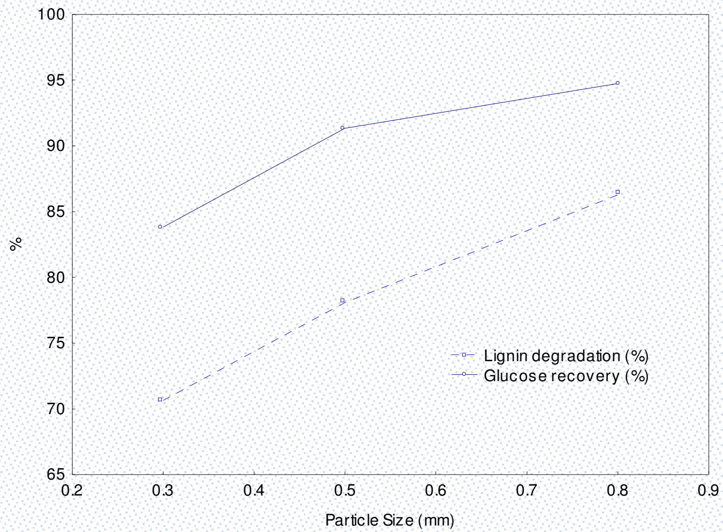




Single Variables

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





Contour

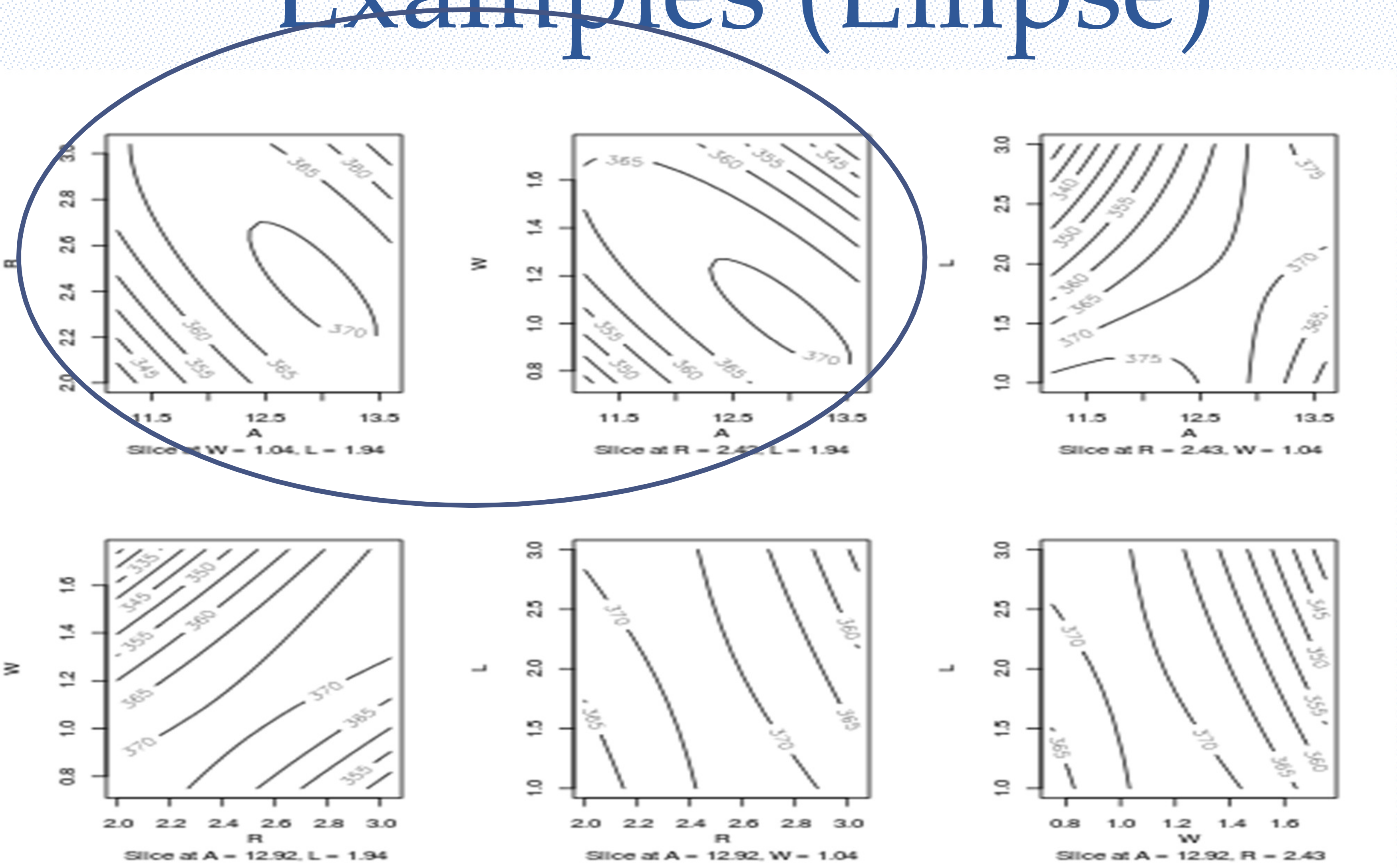
Visualized the shape of the 3D response surface

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.

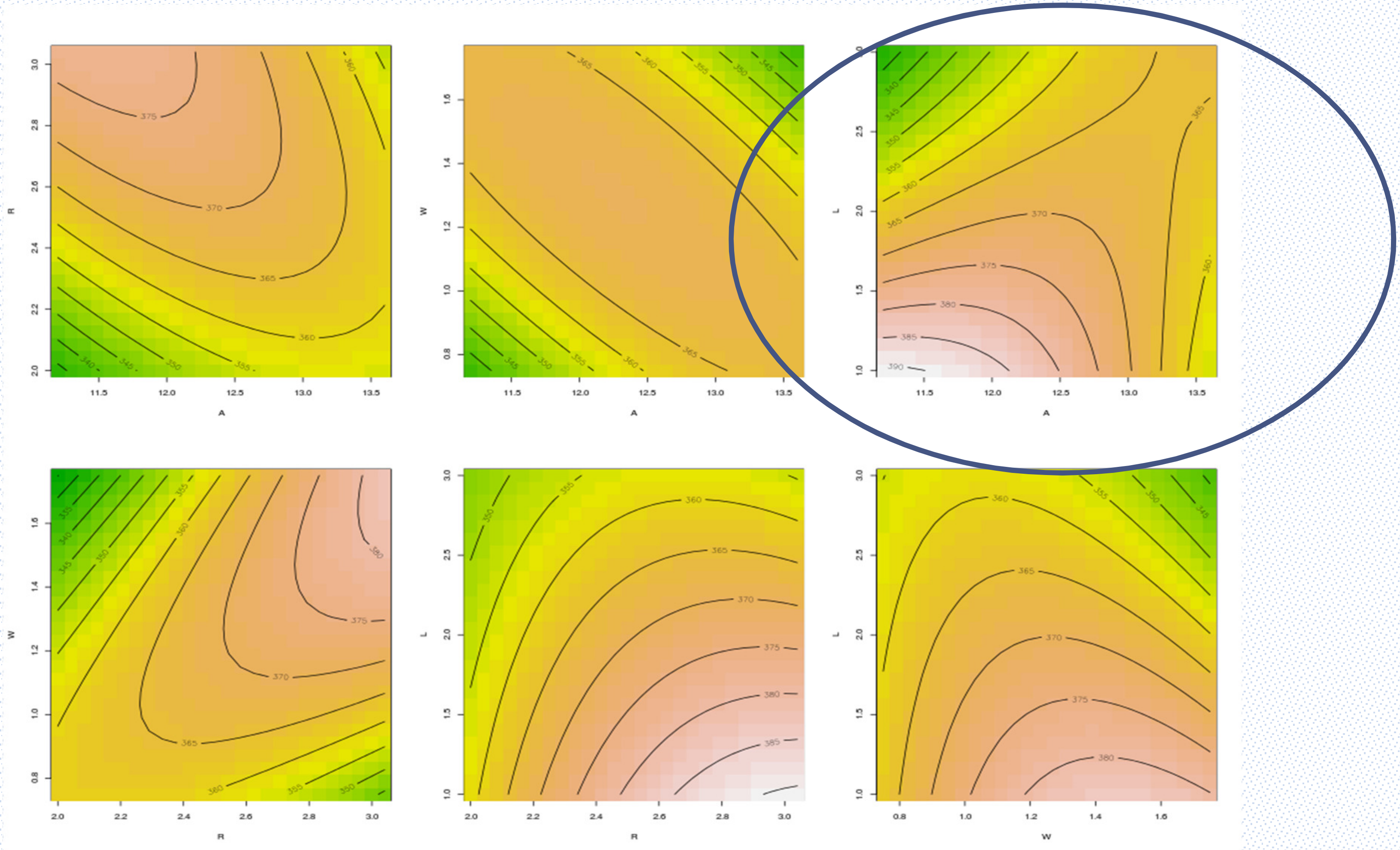
The direction of contour can be used to explained the behavior of interaction for both parameter

Ellipses, circular or saddle point

Examples (Ellipse)



Examples (saddle point)



Example (Circular)

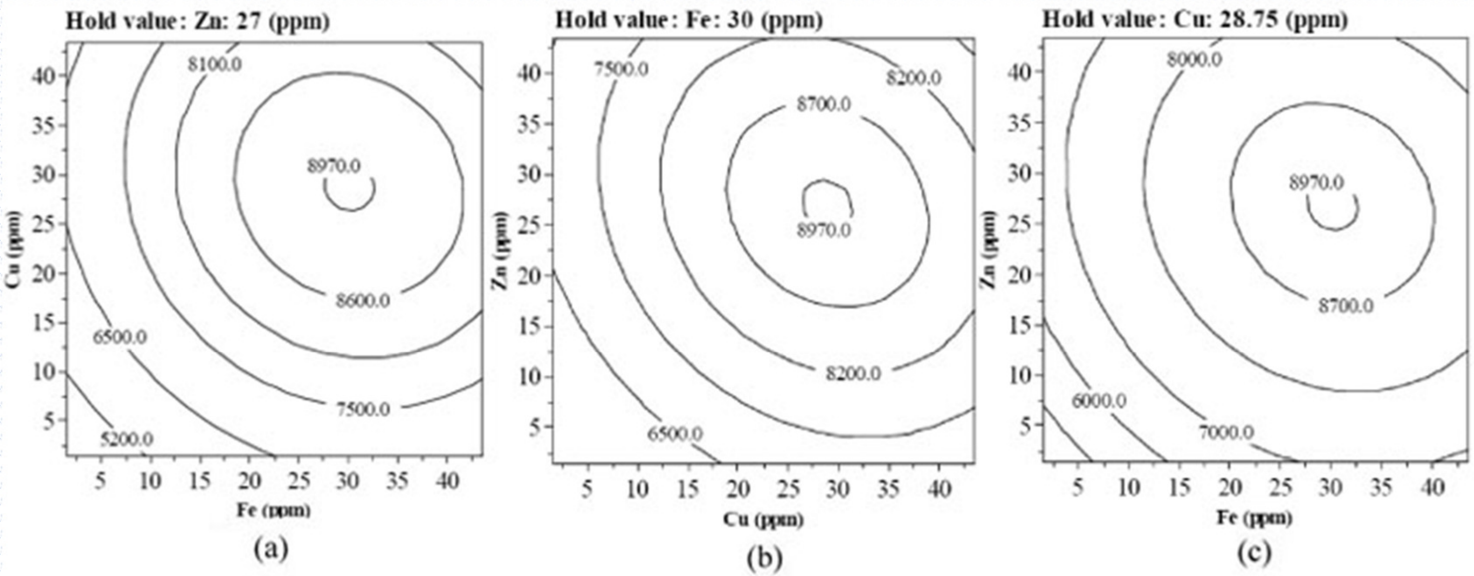


Figure 3: Contour plots (two-dimensional surface plots) of the model equation fitted to the data. (a) interaction of Fe^{3+} and Cu^{2+} concentration, (b) interaction of Fe^{3+} and Zn^{2+} concentration, (c) interaction of Cu^{2+} and Zn^{2+} concentration. (Cu^{2+} (copper, ppm); Fe^{3+} (iron, ppm);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

3D surface plot

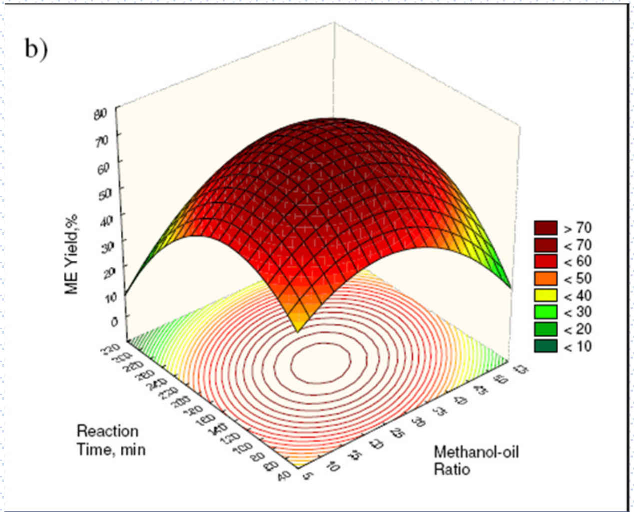
Shows the interaction between two process variables as function of factors.

Shape

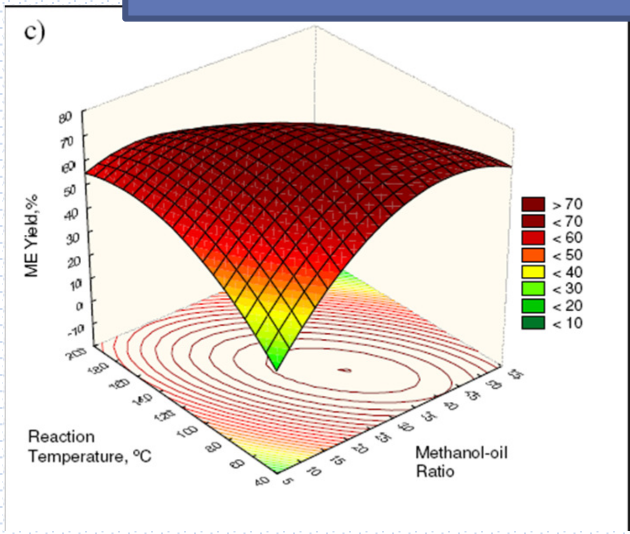
- Minimum: basin
- Maximum: hill
- Saddle: saddle shape

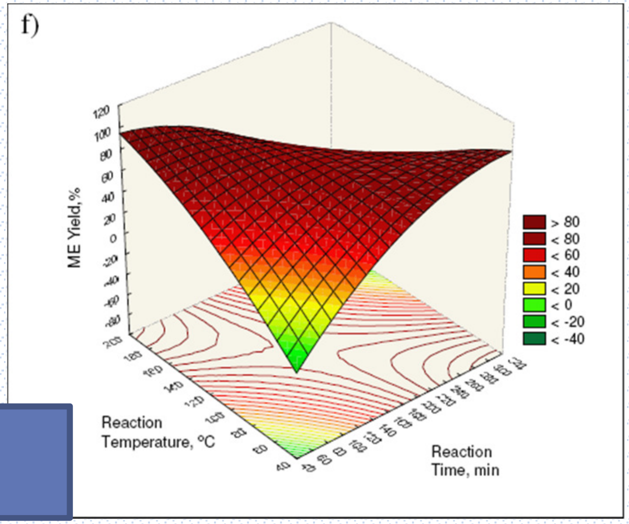
Surface

Parabola, maximum hill



Hyperbola, maximum hill





Saddle-shaped

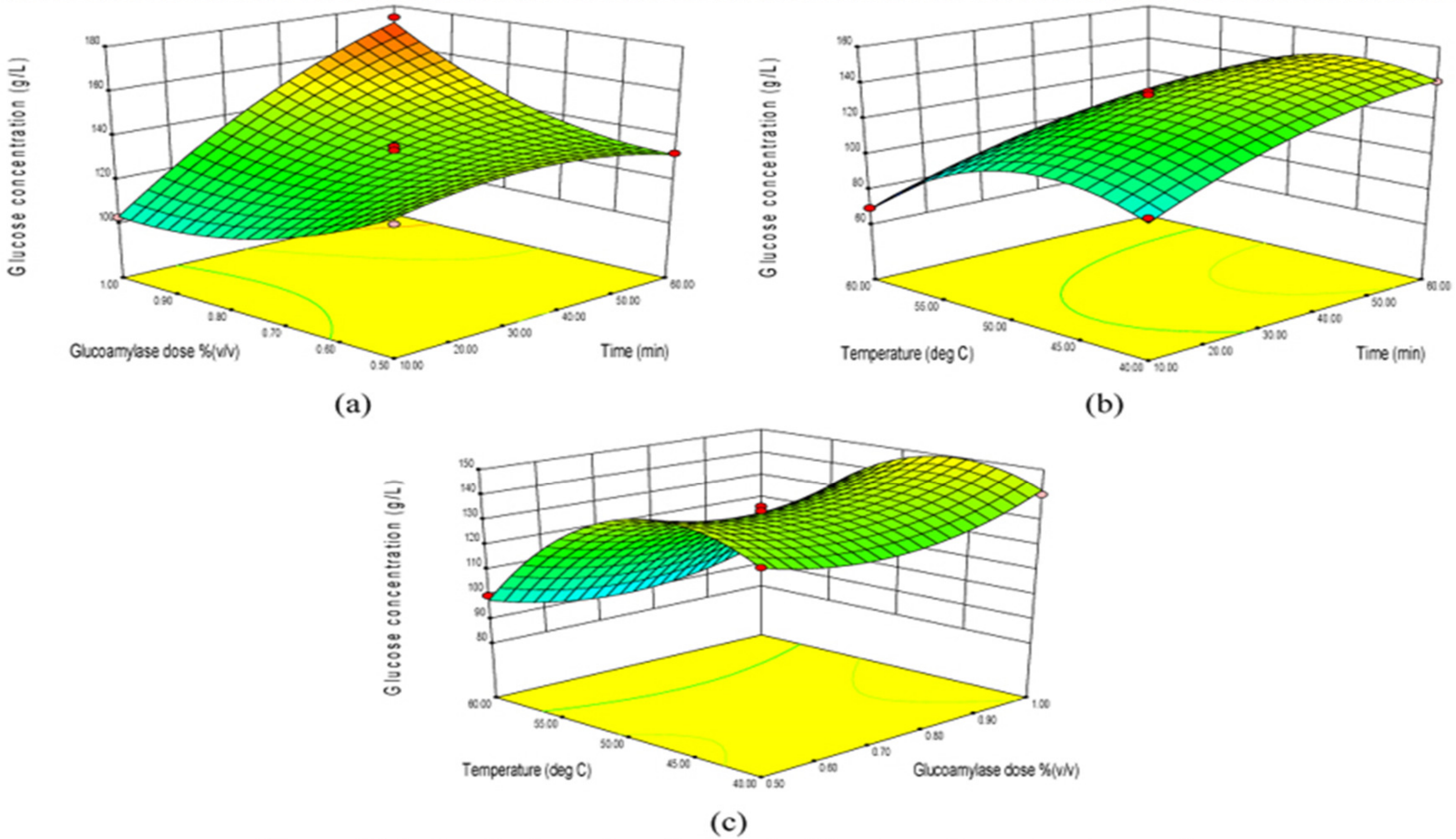
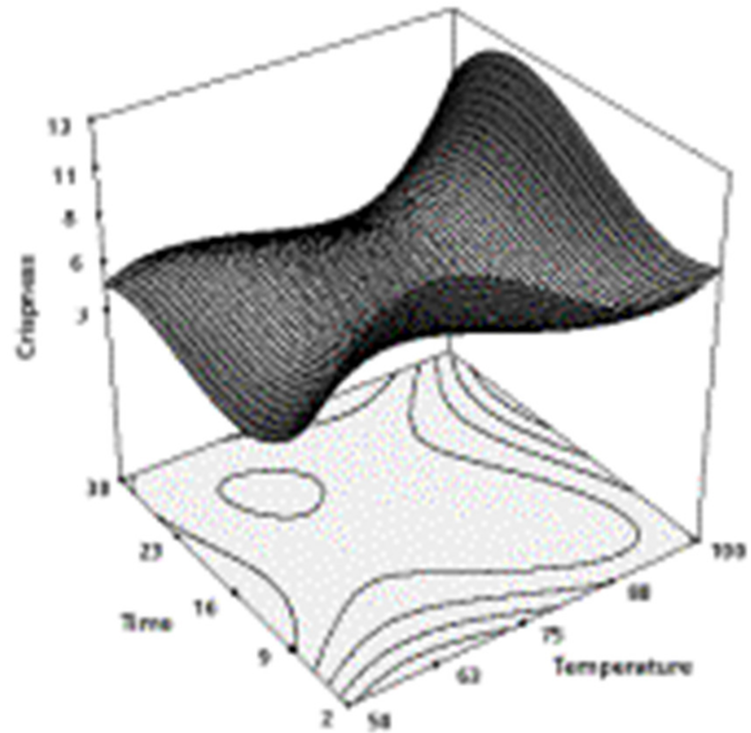
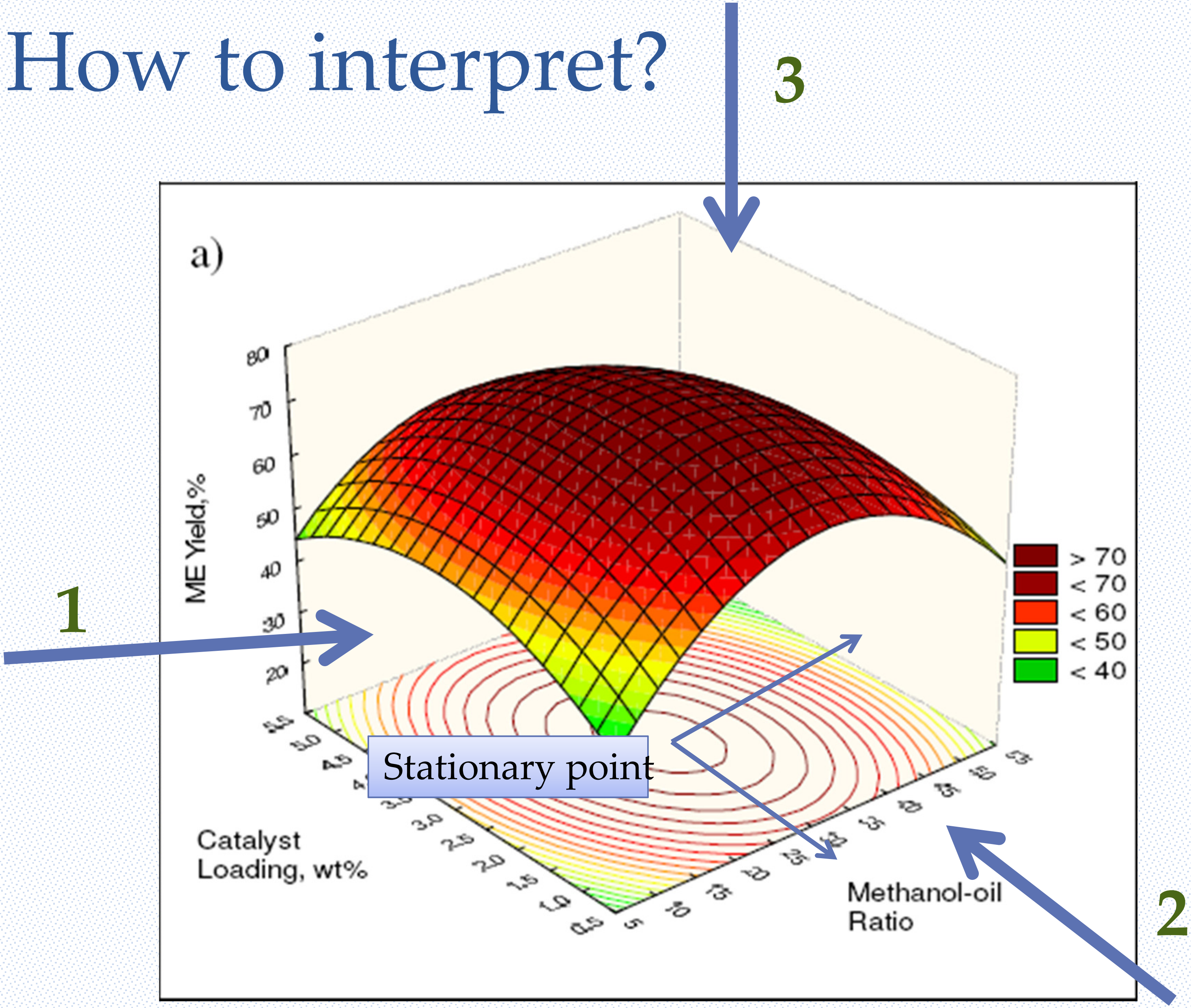


Figure 2(a)-(c): Surface plots for saccharification of sweet potato peel.
(a) effect of glucoamylase dose, time and their reciprocal interaction on glucose concentration.
(b) effect of temperature, time and their reciprocal interaction on glucose concentration.
(c) effect of temperature, glucoamylase dose and their reciprocal interaction on glucose concentration.

Quadratic interaction



How to interpret?



6) Optimization

- Single response
- Multi response

Optimization: Single response-Critical value

Will identified the point on the quadratic response surface either it the minimum, maximum, or saddle point of the surface.

The critical values for the independent variables are the coordinates of the origin of the quadratic response surface.

Shown the predicted value of the dependent variable (response) at the critical values for each of the independent variables.

OPTIMIZATION: Multi-response

- Superimpose of two contour plot.

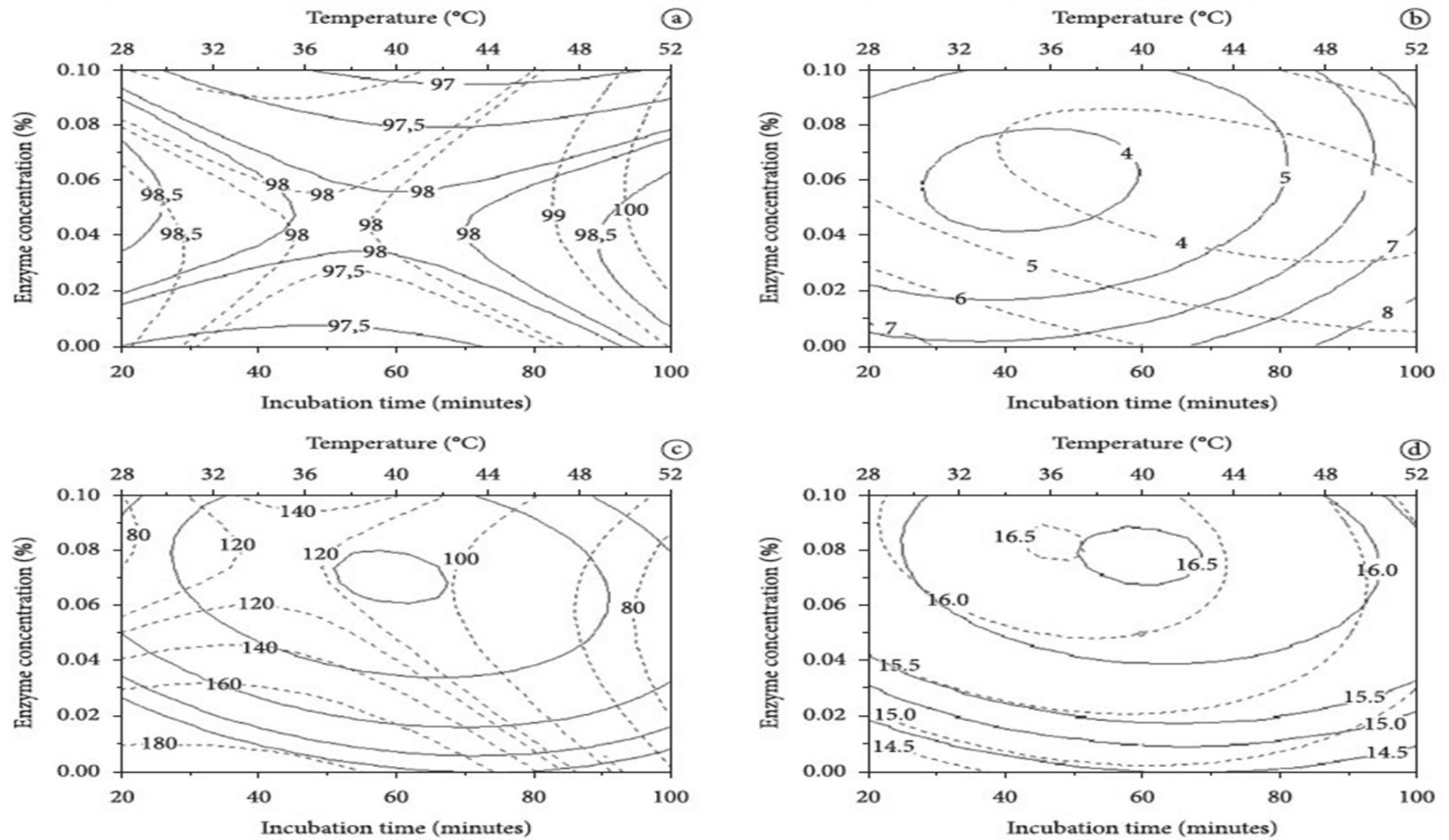


Figure 6. Optimum regions for the contour plots (a- L^* value; b- viscosity; c- turbidity; d- yield, ---- time, —temperature).

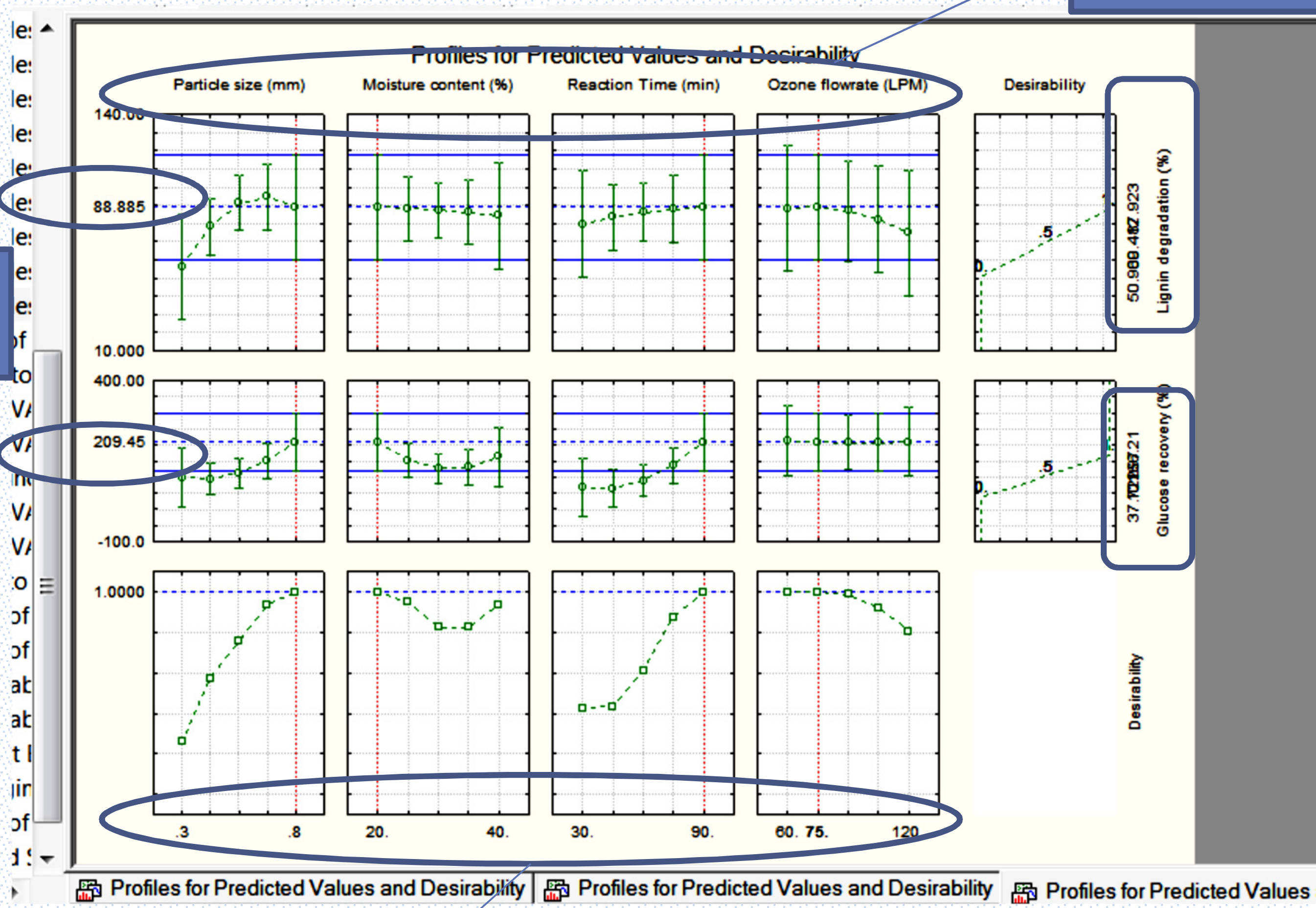
OPTIMIZATION: Multi-response via Desirability Function

A popular and established technique for simultaneous determization 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.

Desirability Profile

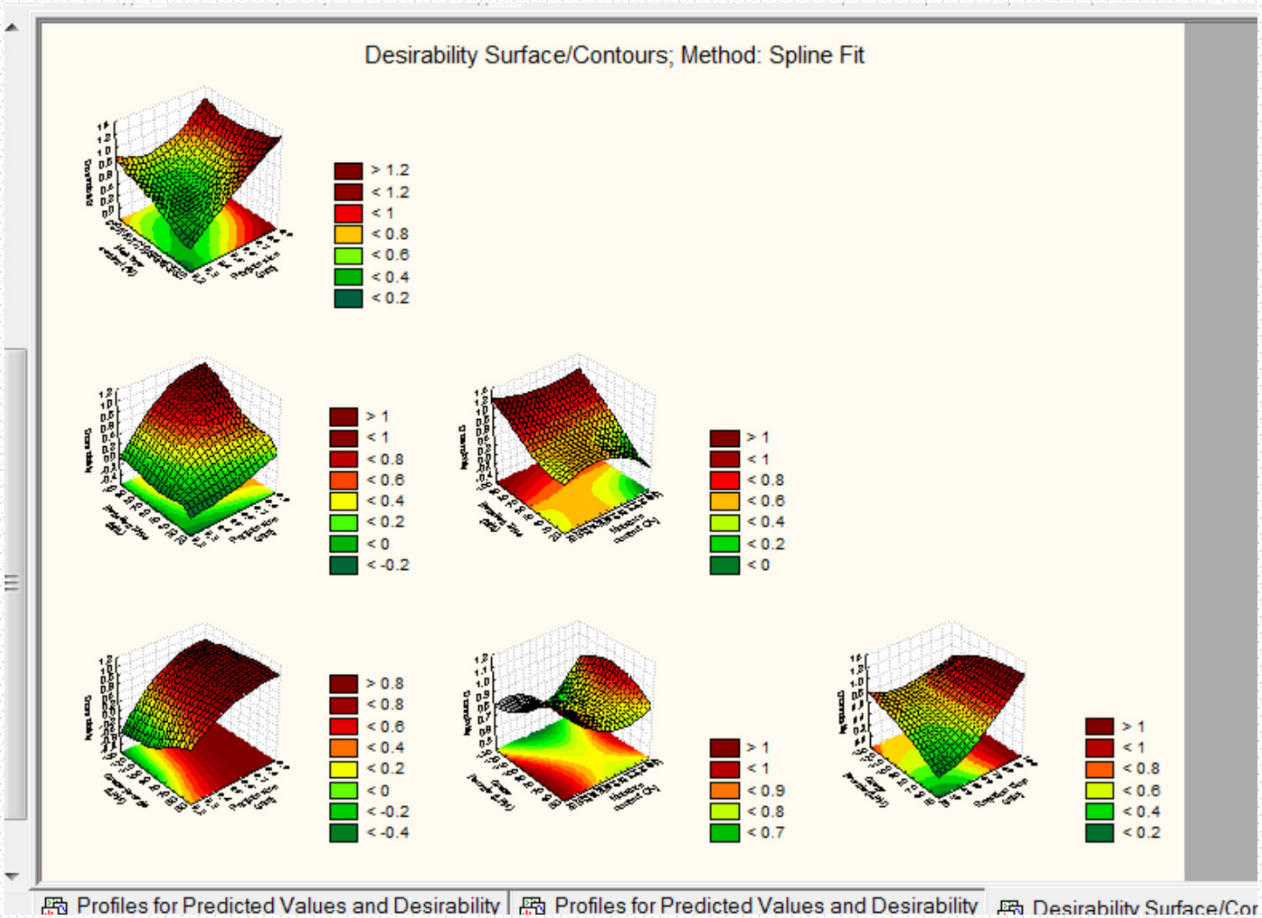


Process variable

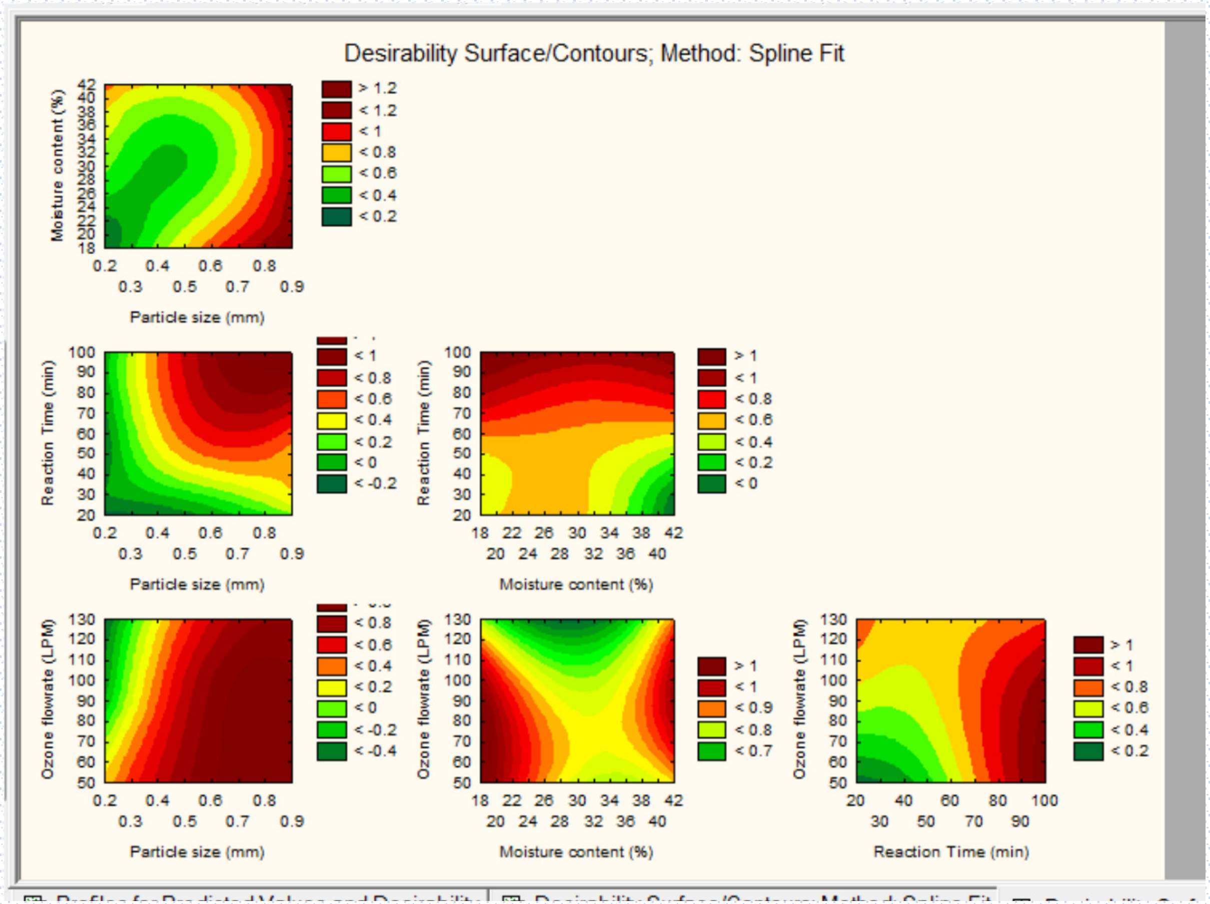
Predicted value

Desirability

3D surface plot



Contour plot



Conclusion

DOE and RSM

- 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.

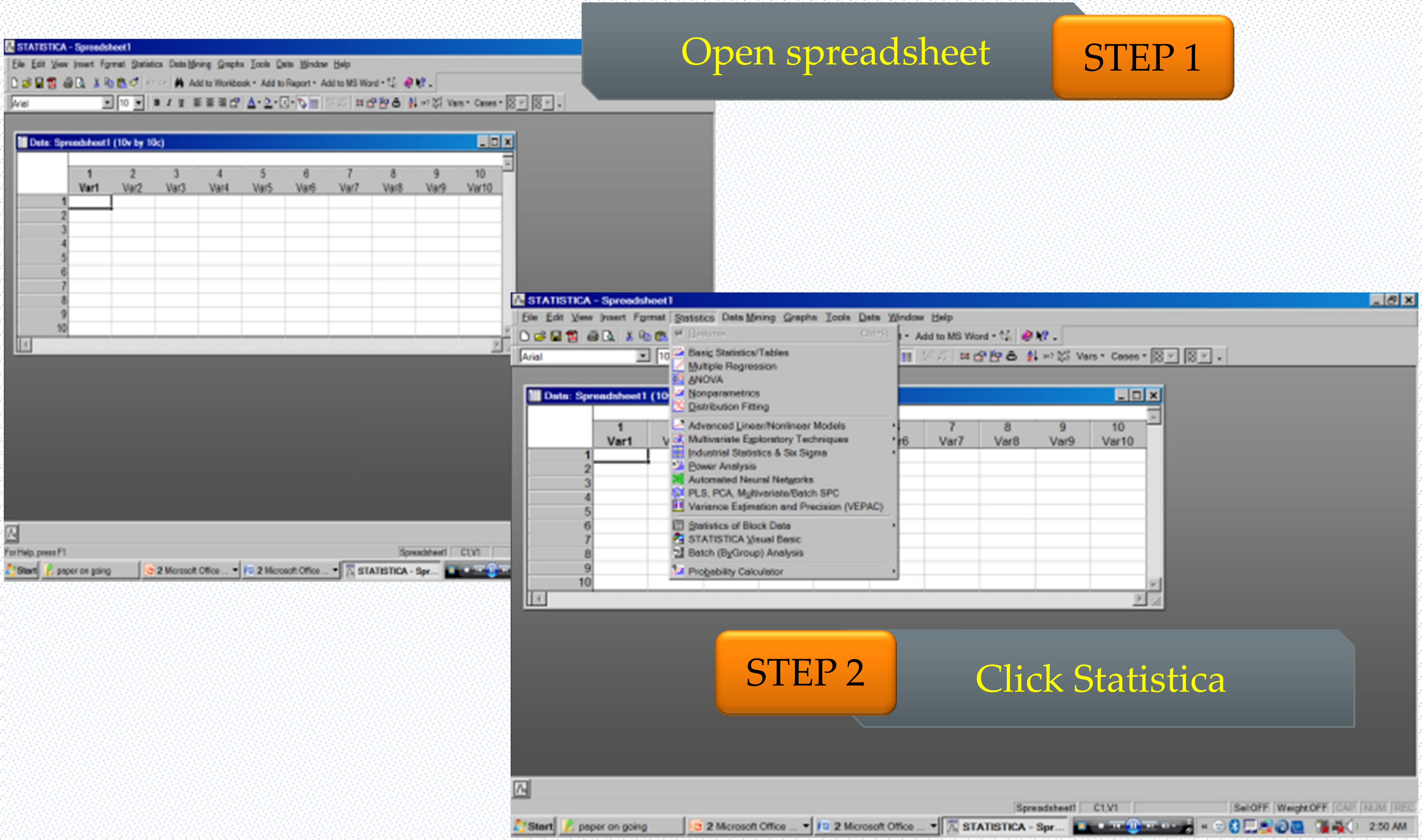
Slide can be found at

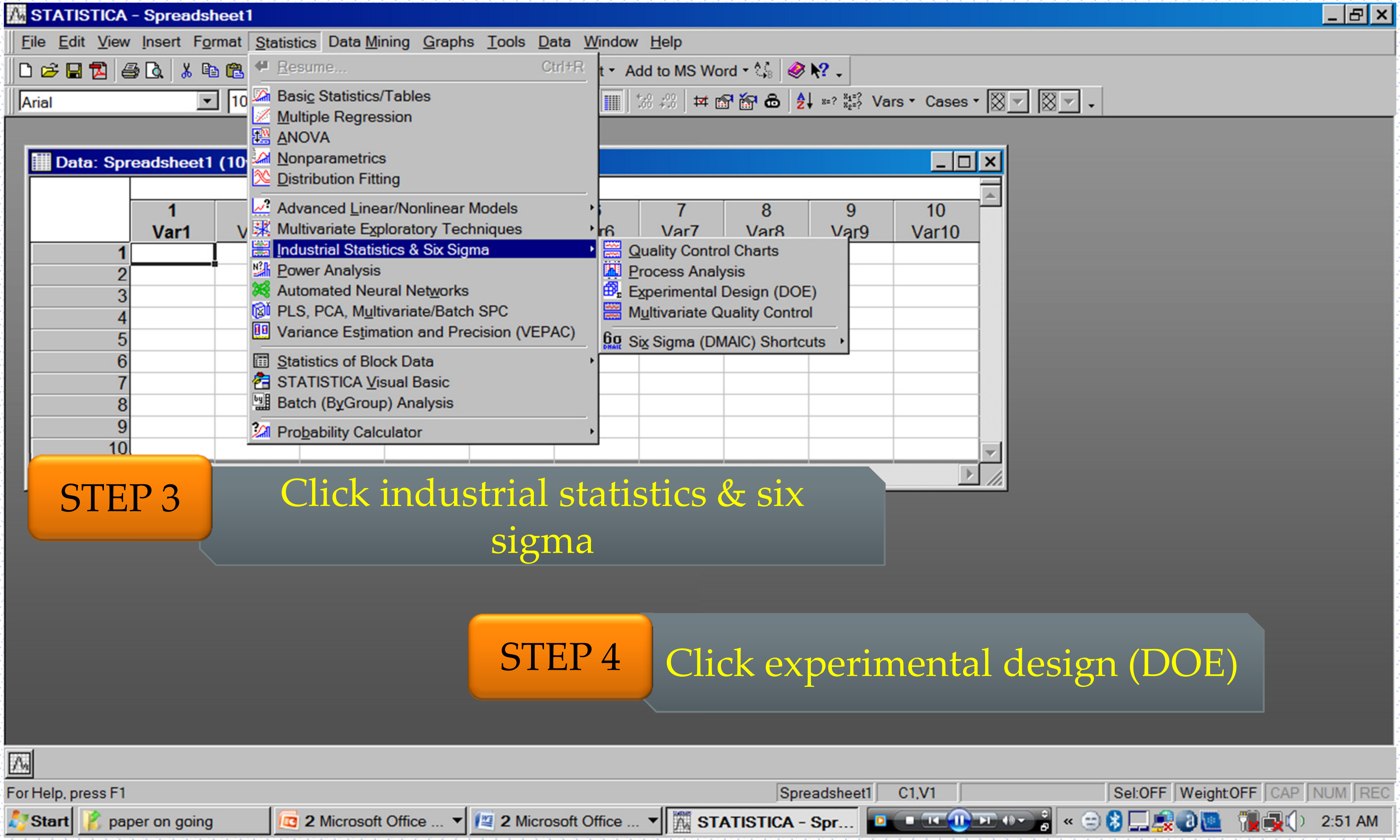
https://teknologimalaysia.academia.edu/DahliaOmar

Statistica Tutorial 1

DESIGN OF EXPERIMENT

DOE spreadsheet

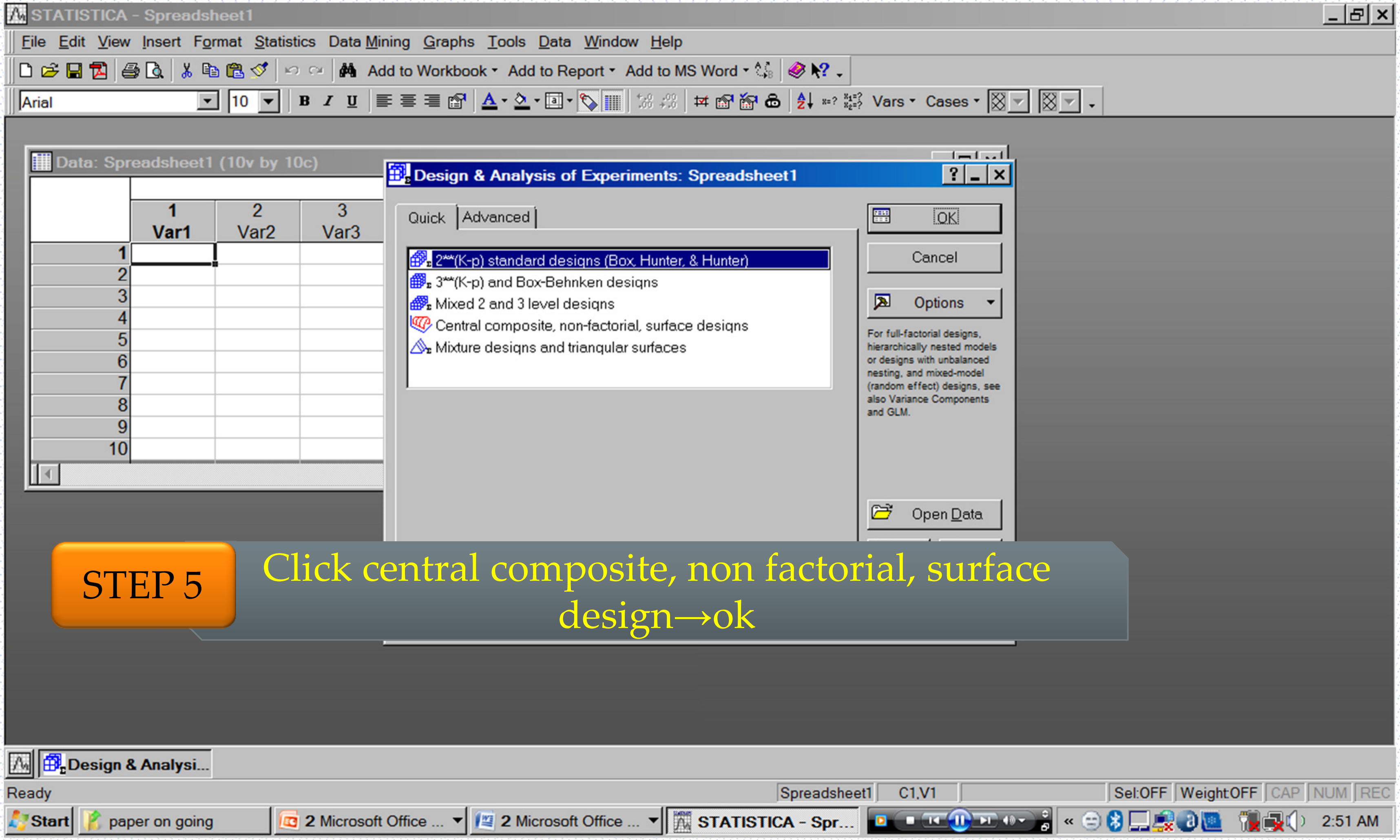


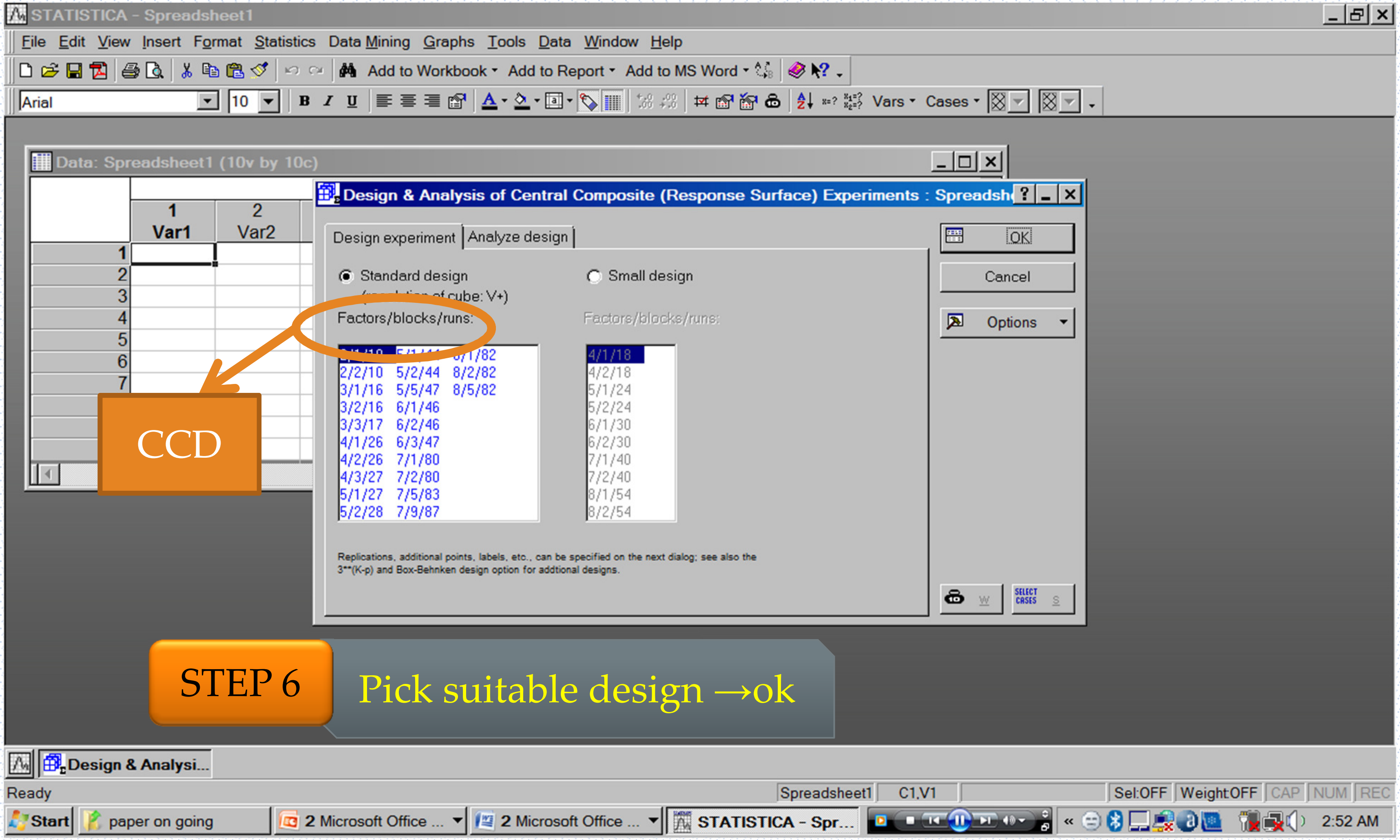


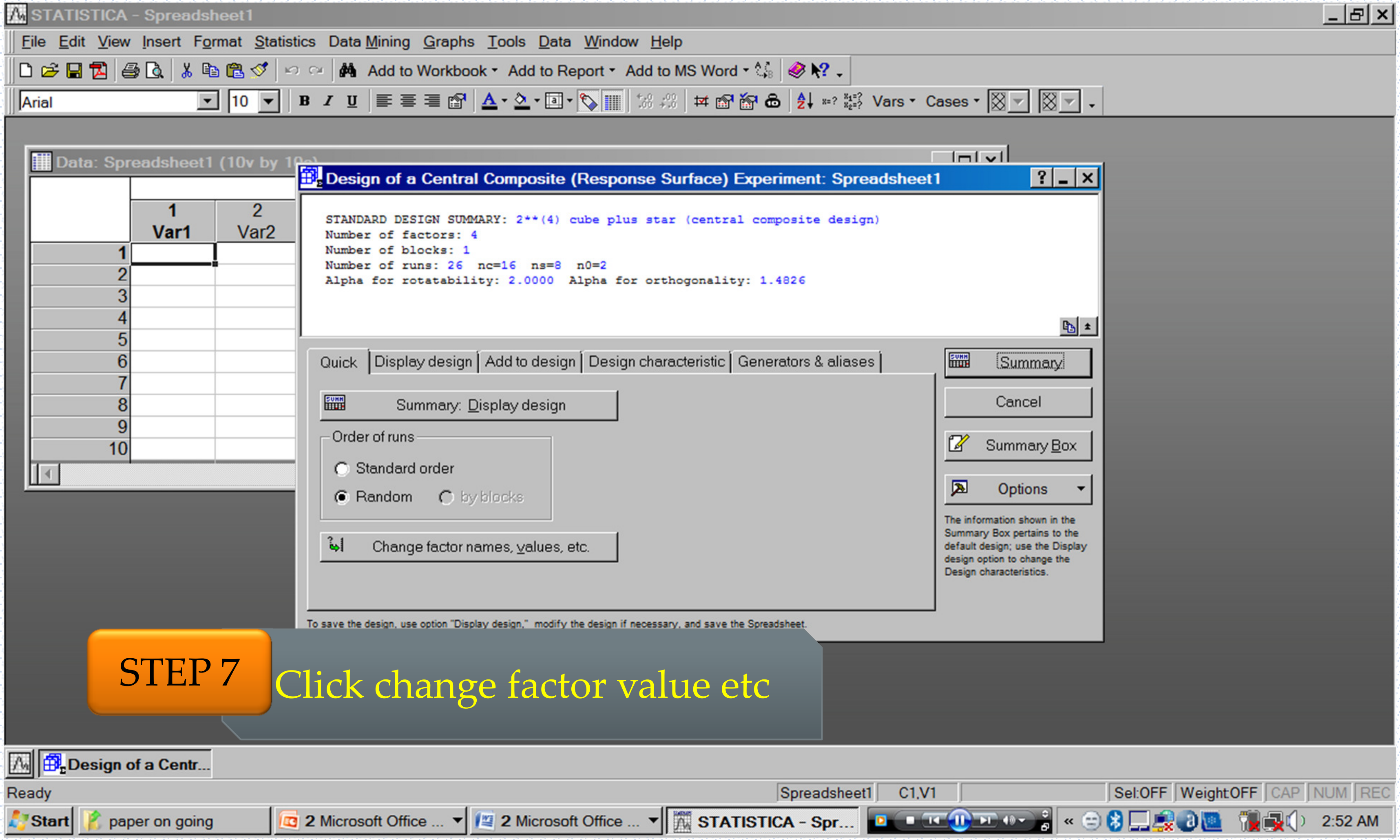
STEP 3 Click industrial statistics & six sigma

STEP 4 Click experimental design (DOE)

Design & analysis of experiment windows



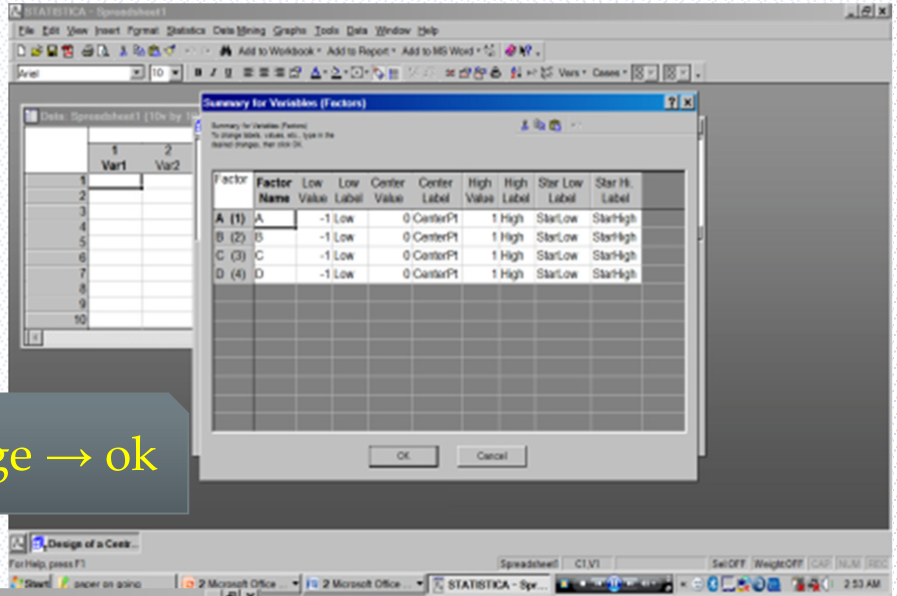

STATISTICA - Spreadsheet1
File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help
Add to Workbook Add to Report Add to MS Word
Data: Spreadsheet1 (10v by 10c)
Var1
Var2
Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsh
Design experiment
Analyze design
Standard design
Small design
Factors/blocks/runs:
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 addtional designs.
CCD
STEP 6
Pick suitable design →ok



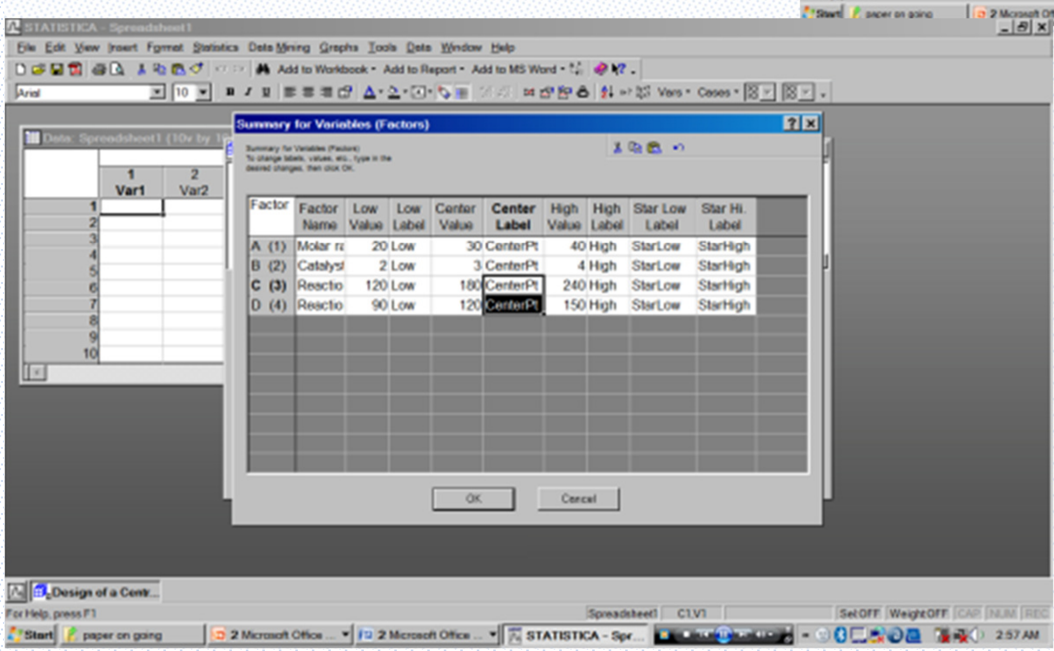
STEP 7

Click change factor value etc

Change value

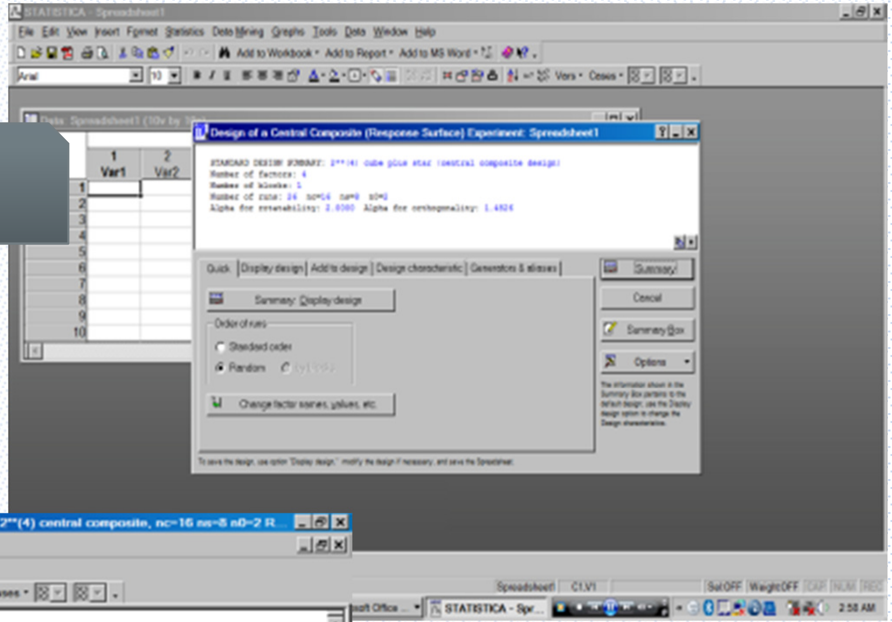


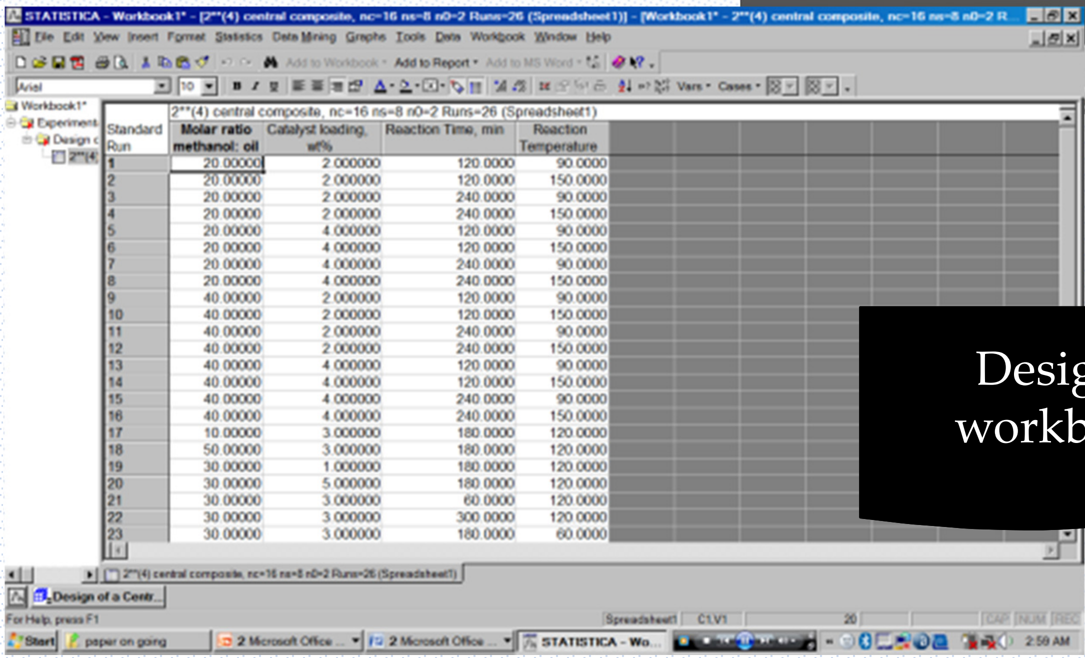
STEP 8 Insert the variable and range → ok



STEP 9

Click design display (standard order)



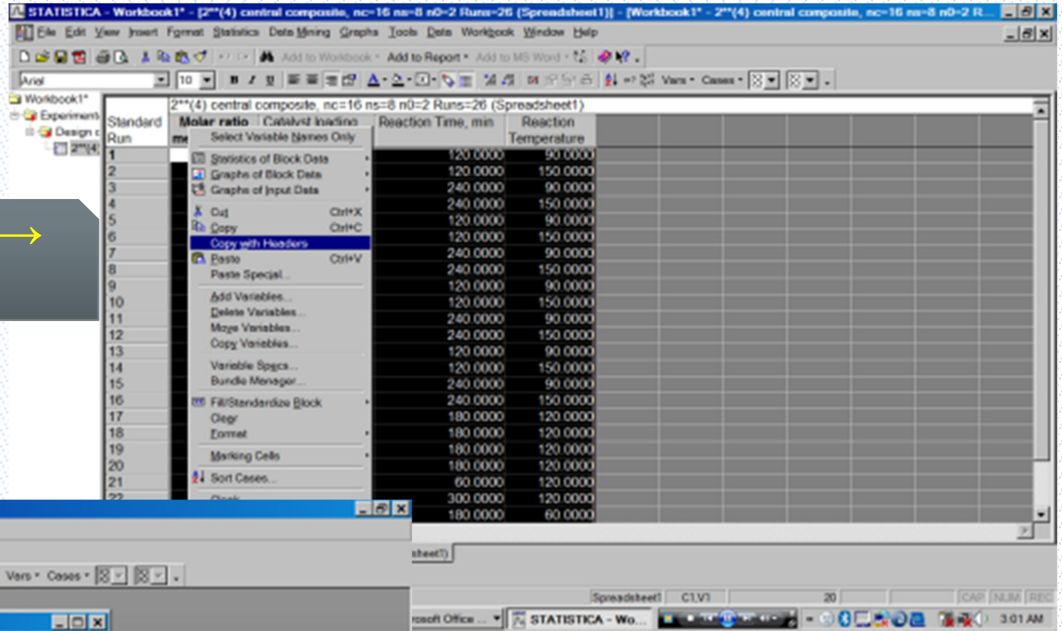


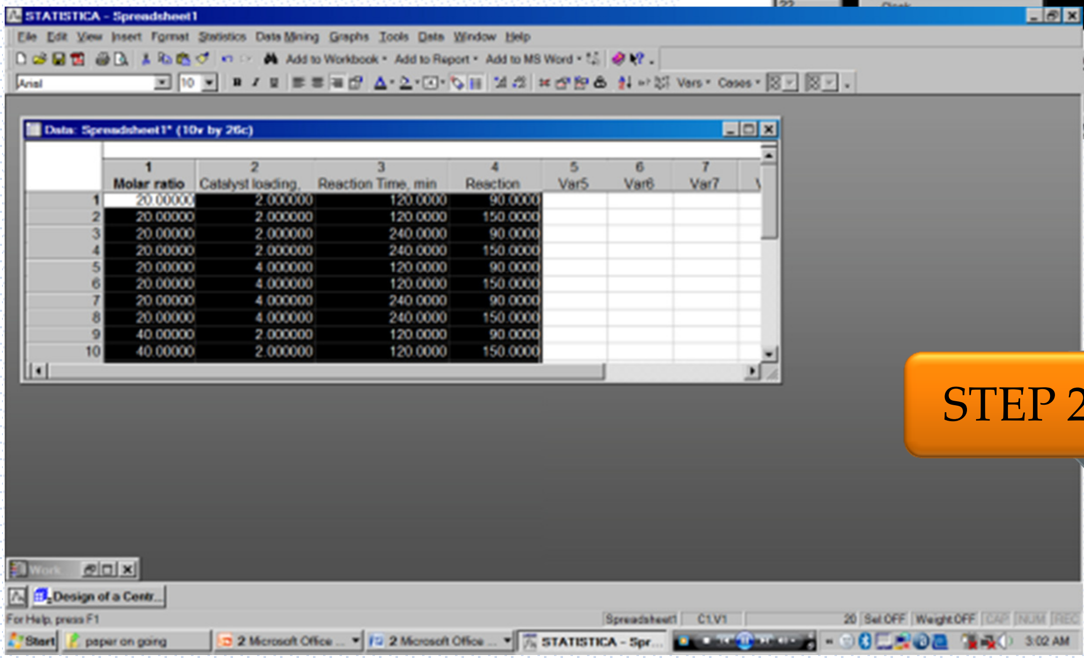
Standard Run	Molar ratio methanol: oil	Catalyst loading, wt%	Reaction Time, min	Reaction Temperature
1	20.00000	2.000000	120.0000	90.0000
2	20.00000	2.000000	120.0000	150.0000
3	20.00000	2.000000	240.0000	90.0000
4	20.00000	2.000000	240.0000	150.0000
5	20.00000	4.000000	120.0000	90.0000
6	20.00000	4.000000	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
11	40.00000	2.000000	240.0000	90.0000
12	40.00000	2.000000	240.0000	150.0000
13	40.00000	4.000000	120.0000	90.0000
14	40.00000	4.000000	120.0000	150.0000
15	40.00000	4.000000	240.0000	90.0000
16	40.00000	4.000000	240.0000	150.0000
17	10.00000	3.000000	180.0000	120.0000
18	50.00000	3.000000	180.0000	120.0000
19	30.00000	1.000000	180.0000	120.0000
20	30.00000	5.000000	180.0000	120.0000
21	30.00000	3.000000	60.0000	120.0000
22	30.00000	3.000000	300.0000	120.0000
23	30.00000	3.000000	180.0000	60.0000

Design display on workbook windows

Copy DOE to spreedsheet

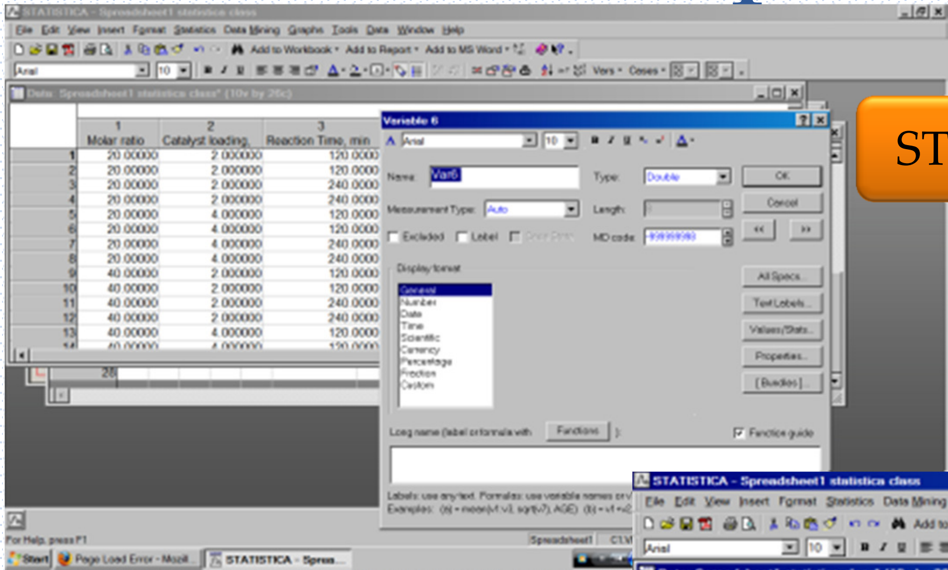
STEP 1 Select all → right click→ copy with headers





STEP 2 Paste on spreadsheet

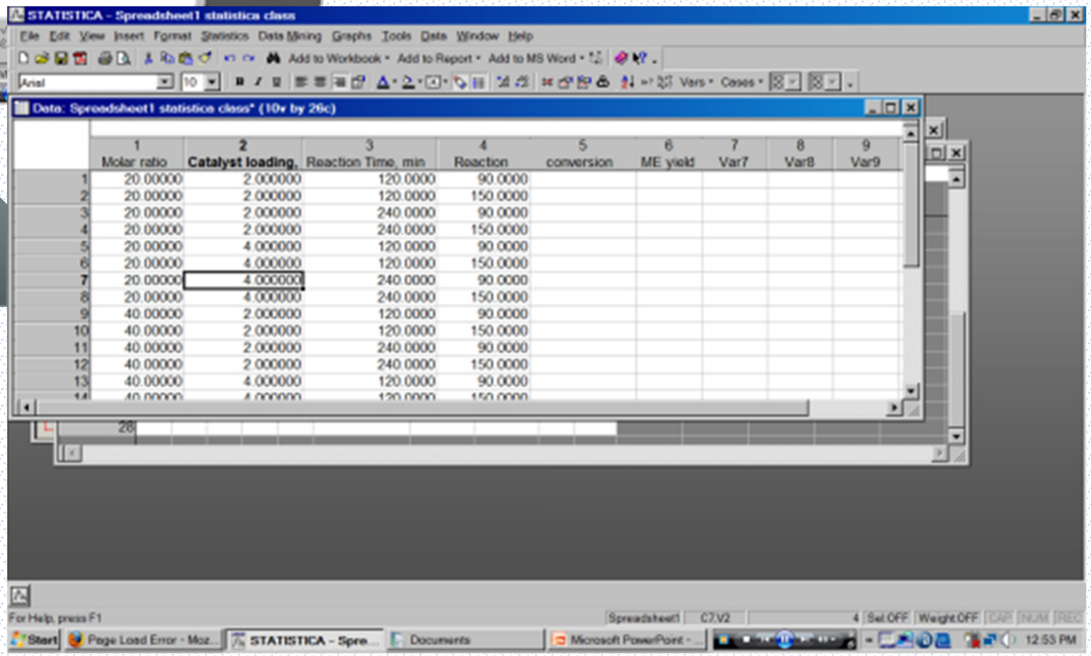
Edit, save & print spreedsheet



STEP 1 Right click on the column→edit

STEP 2 click file→save

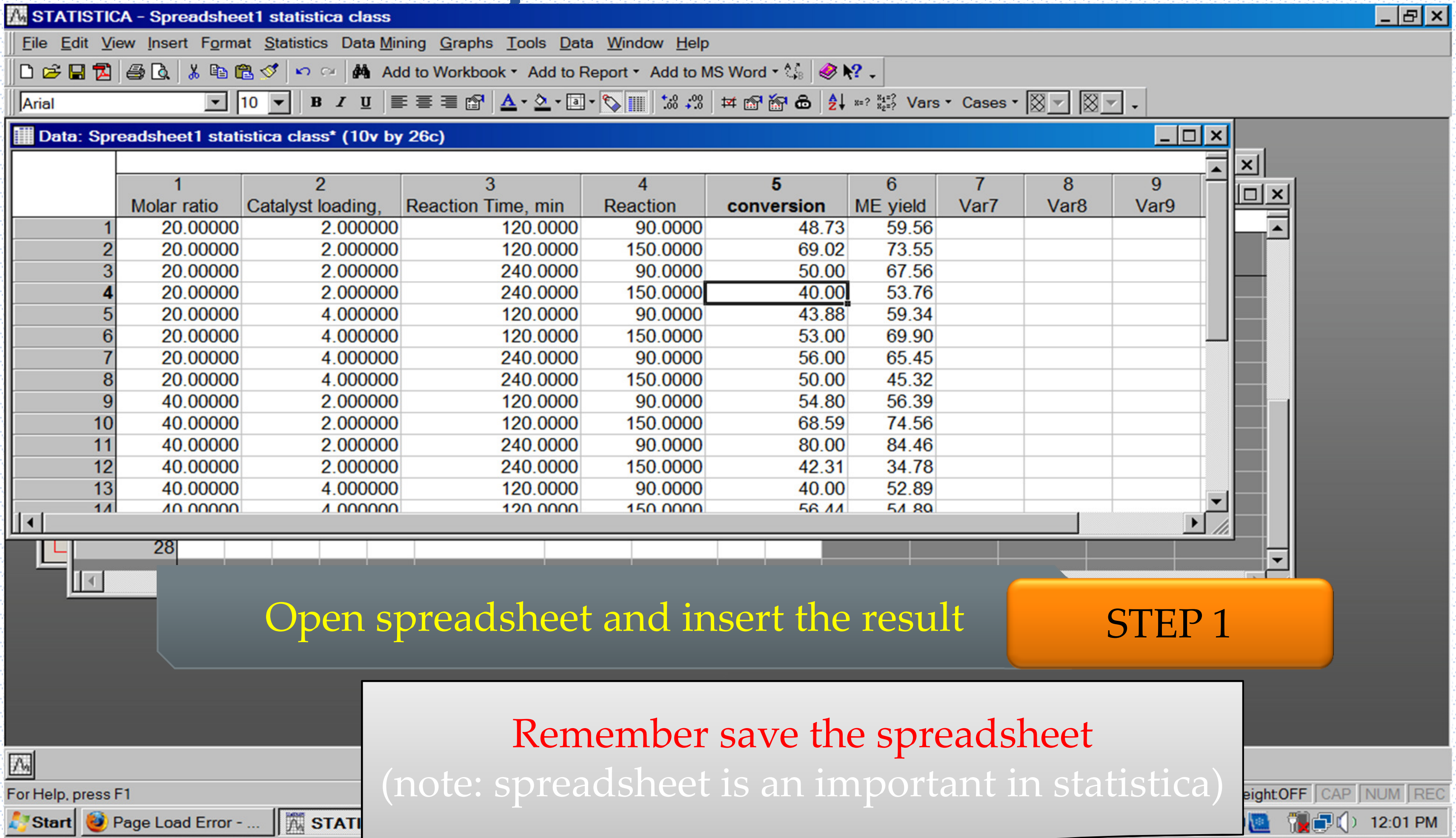
STEP 3 click file→print



Statistica tutorial 2

INSERT AND ANALYSIS THE DATA

Insert the result into spreadsheet

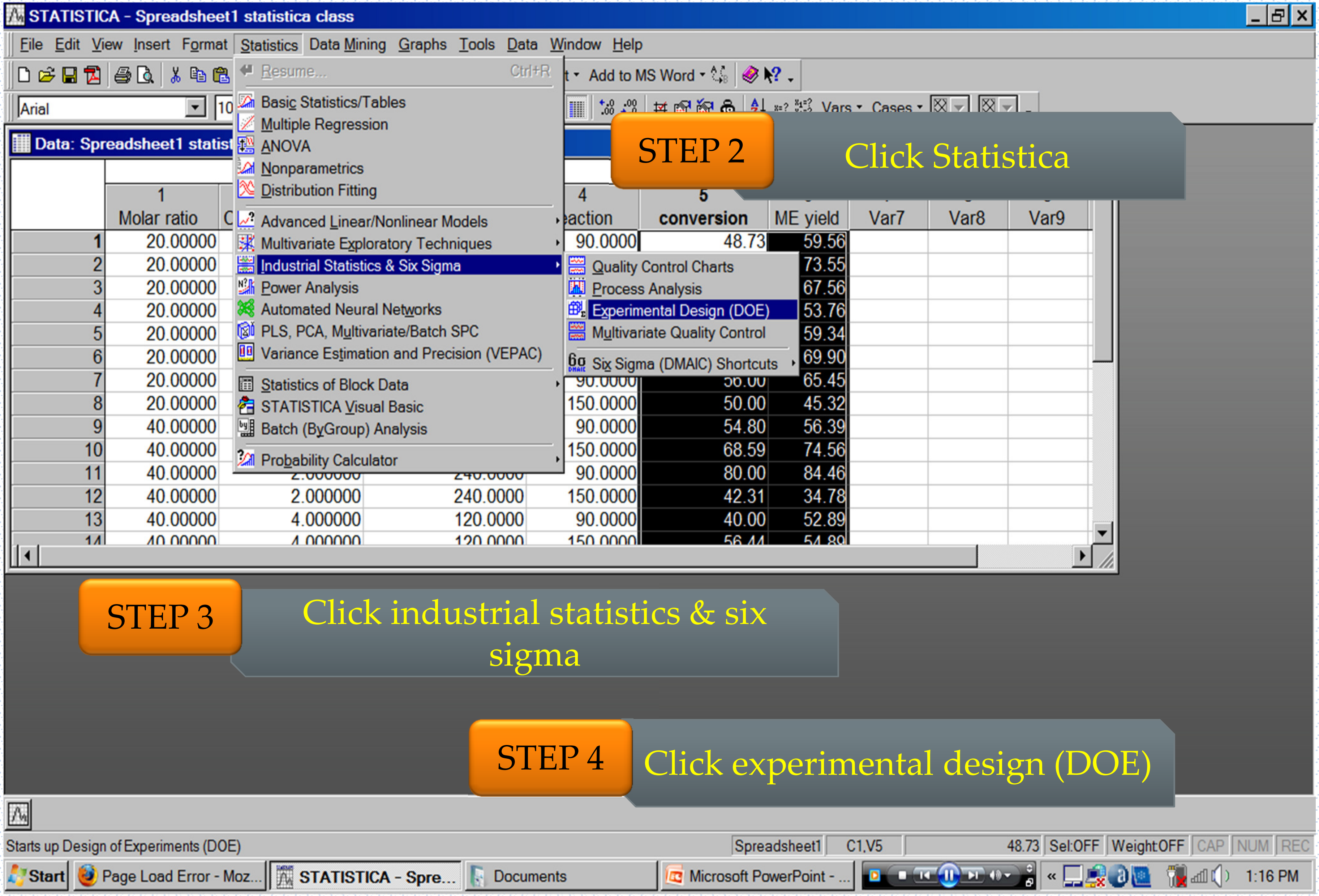


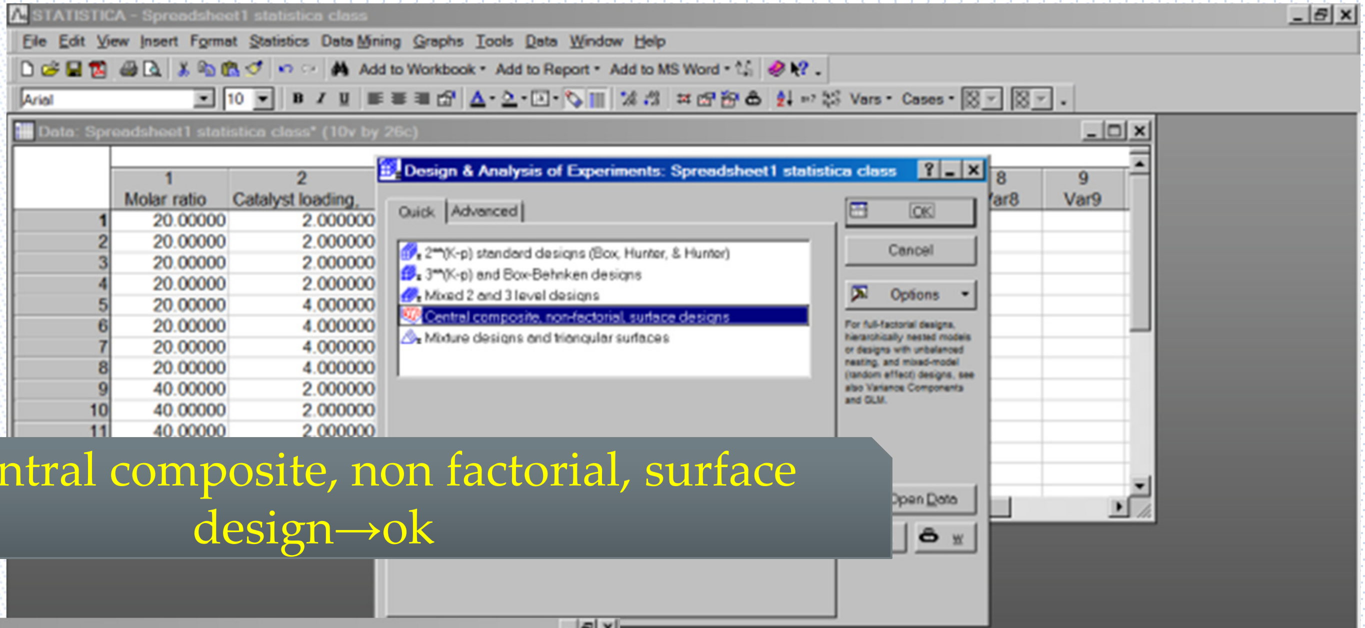
	1 Molar ratio	2 Catalyst loading,	3 Reaction Time, min	4 Reaction	5 conversion	6 ME yield	7 Var7	8 Var8	9 Var9
1	20.00000	2.000000	120.0000	90.0000	48.73	59.56			
2	20.00000	2.000000	120.0000	150.0000	69.02	73.55			
3	20.00000	2.000000	240.0000	90.0000	50.00	67.56			
4	20.00000	2.000000	240.0000	150.0000	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			

Open spreadsheet and insert the result

STEP 1

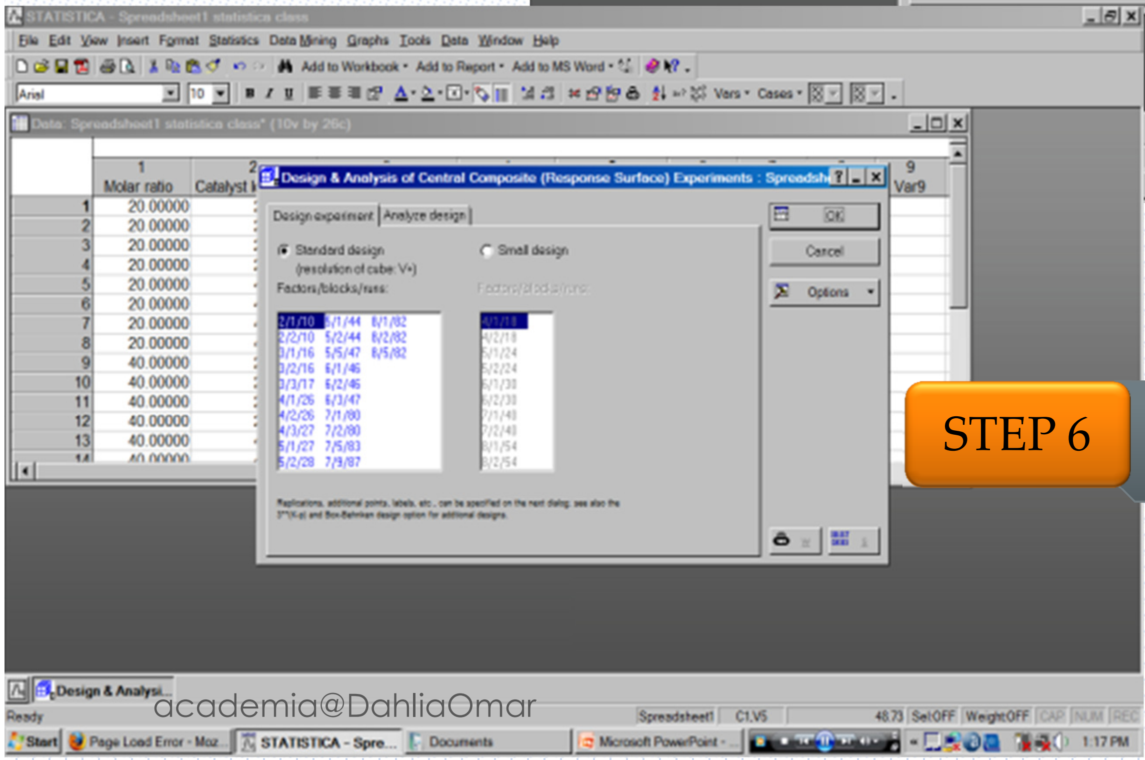
Remember save the spreadsheet
(note: spreadsheet is an important in statistica)


STATISTICA - Spreadsheet1 statistica class
File Edit View Insert Format Statistics Data Mining Graphs Tools Data Window Help
Basic Statistics/Tables
Multiple Regression
ANOVA
Nonparametrics
Distribution Fitting
Advanced Linear/Nonlinear Models
Multivariate Exploratory Techniques
Industrial Statistics & Six Sigma
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
Probability Calculator
Quality Control Charts
Process Analysis
Experimental Design (DOE)
Multivariate Quality Control
Six Sigma (DMAIC) Shortcuts
STEP 2
Click Statistica
STEP 3
Click industrial statistics & six sigma
STEP 4
Click experimental design (DOE)
Starts up Design of Experiments (DOE)



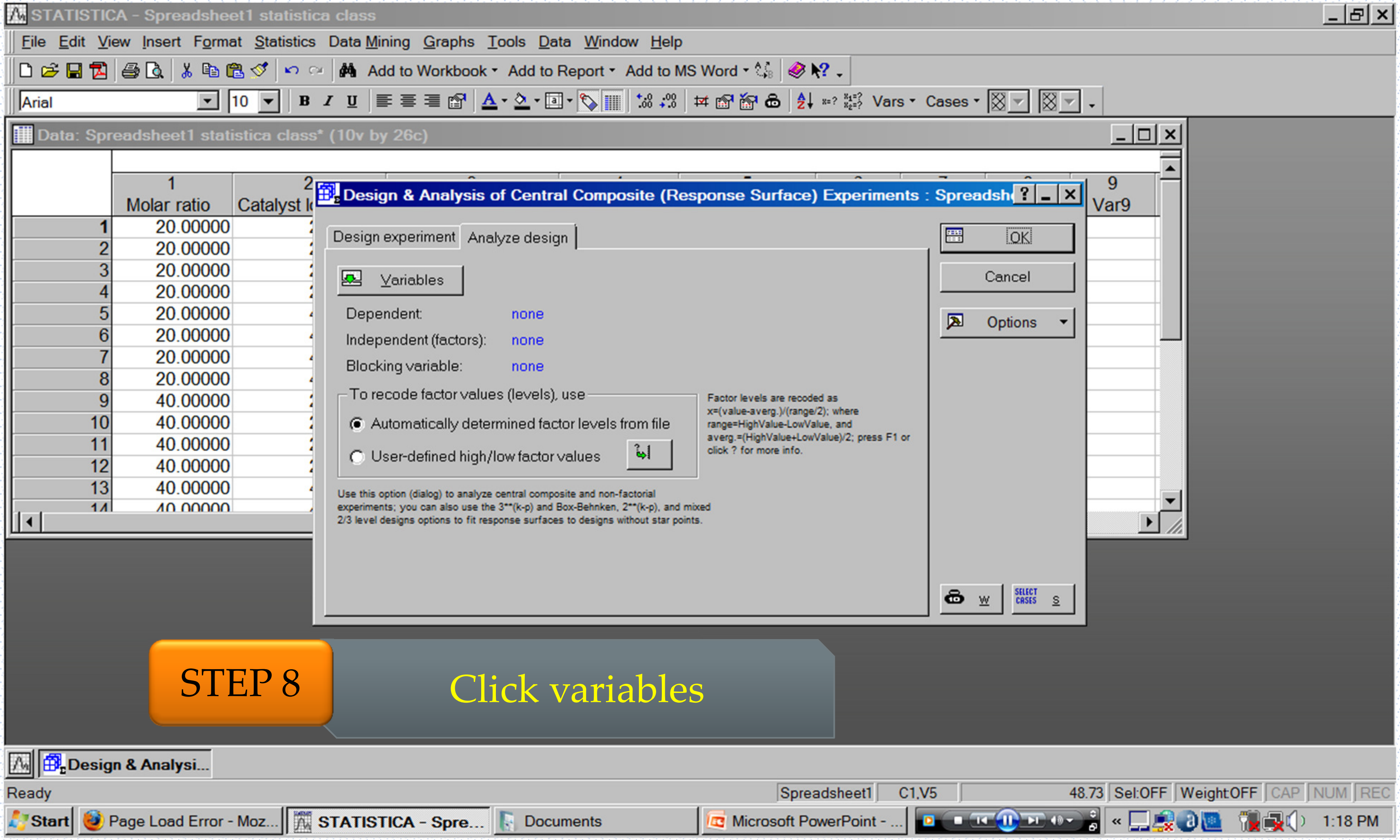
STEP 5

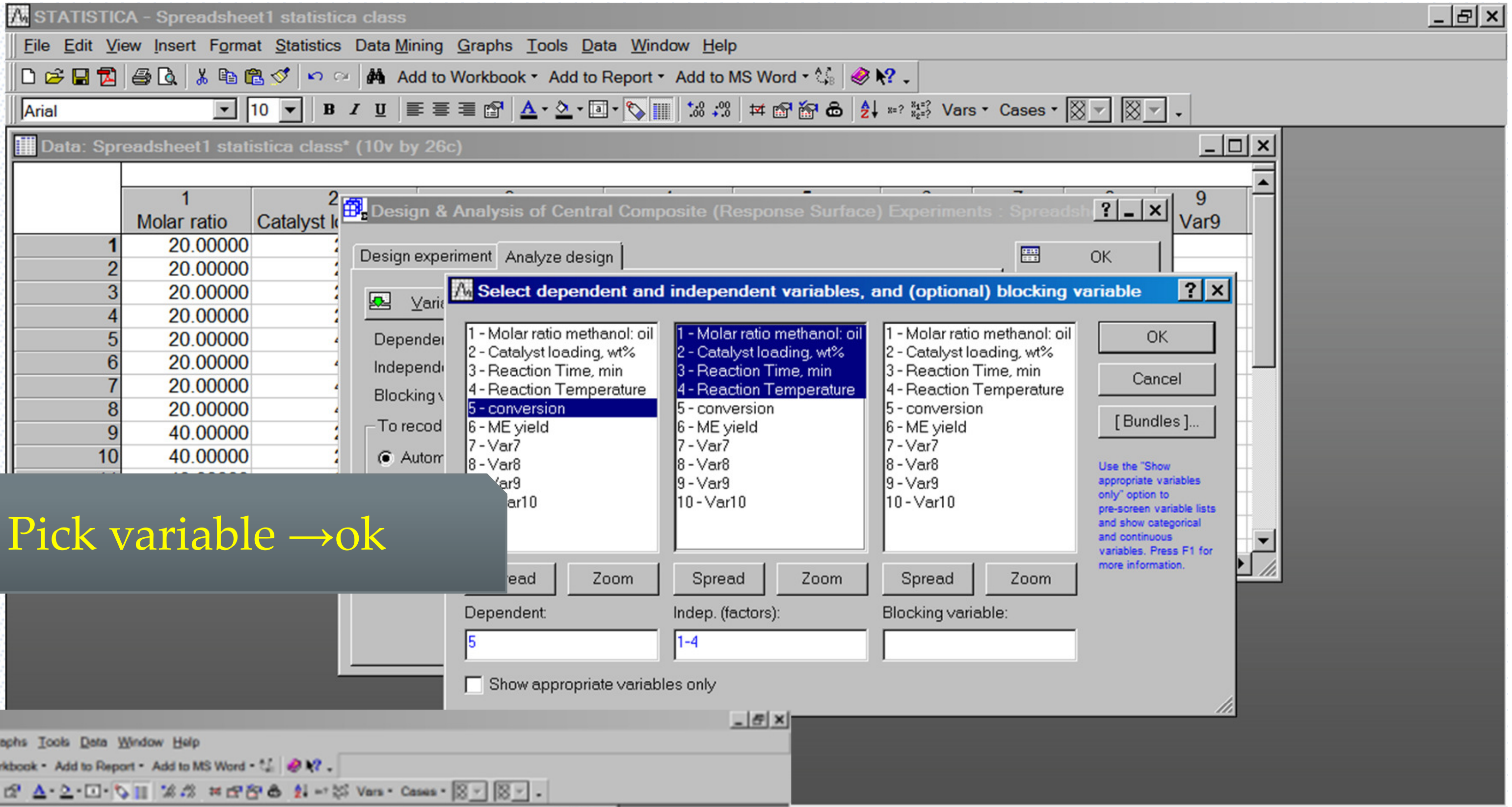
Click central composite, non factorial, surface design→ok



STEP 6

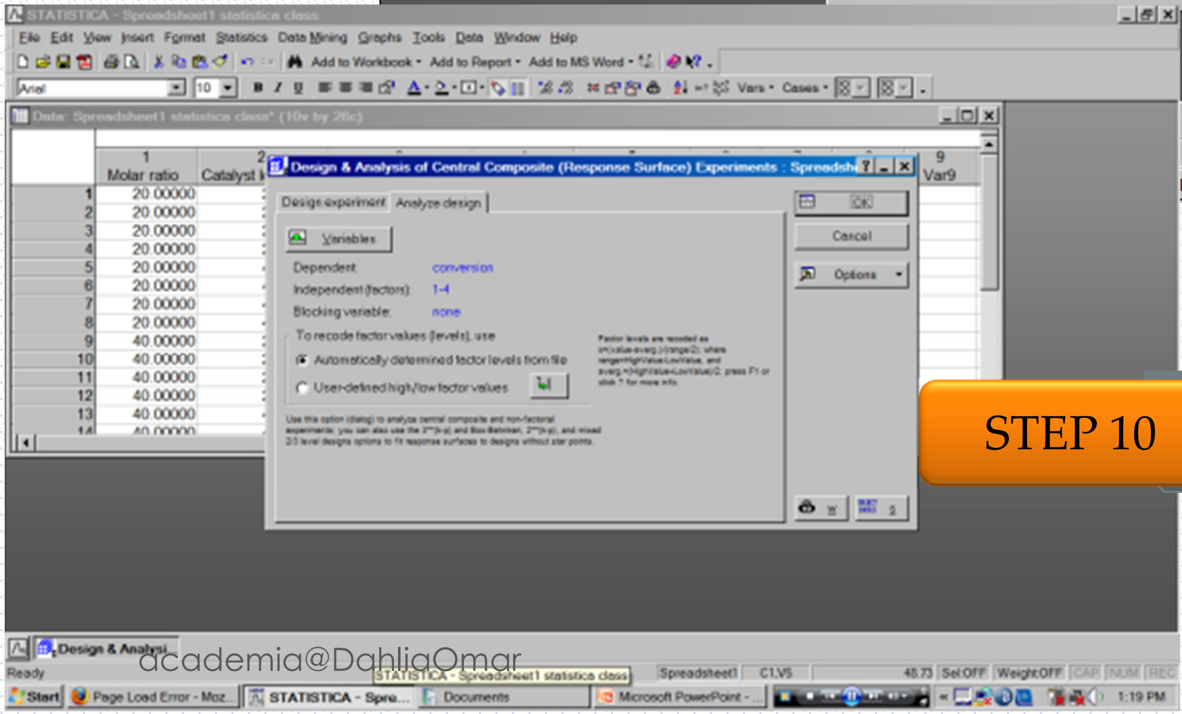
Click analyze design tab


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
Data: Spreadsheet1 statistica class* (10v by 26c)
Molar ratio
Catalyst l
Var9
Design & Analysis of Central Composite (Response Surface) Experiments : Spreadsh
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=(value-averg.)/(range/2); where range=HighValue-LowValue, and averg.=(HighValue+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
Design & Analysi...
Ready
Start
STATISTICA - Spre...
Documents
Microsoft PowerPoint - ...
1:18 PM



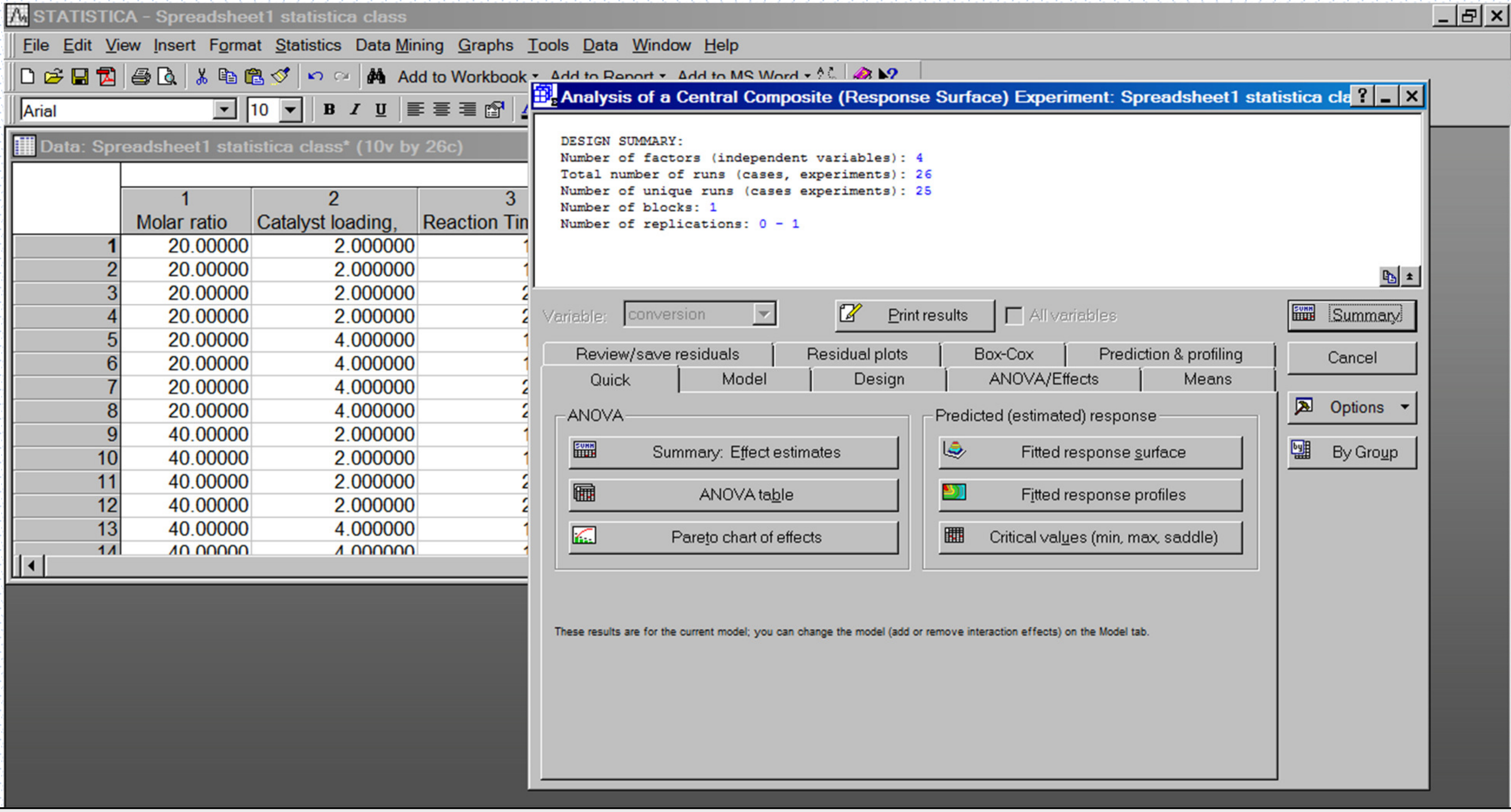
STEP 9

Pick variable →ok



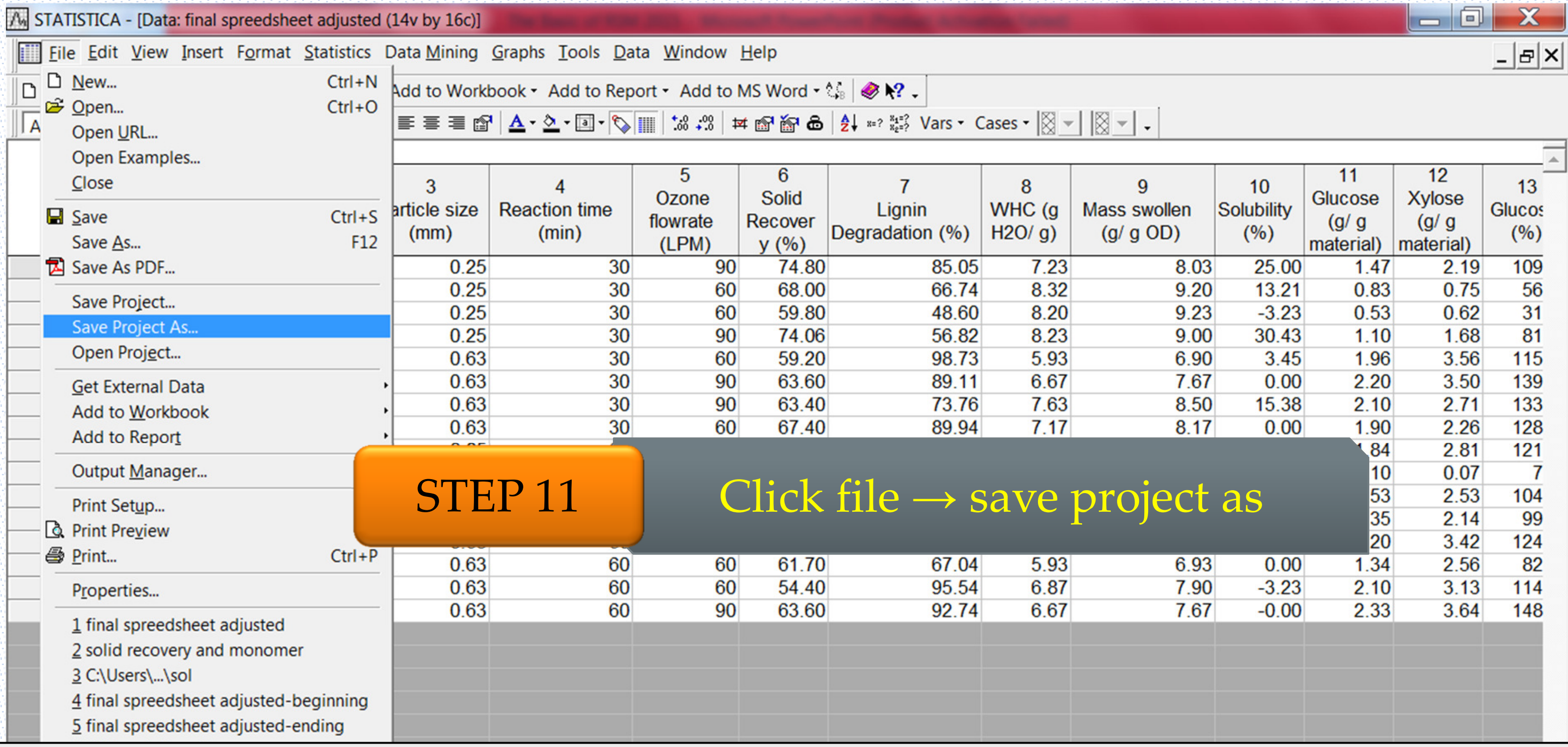
STEP 10

Click ok



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



STEP 11

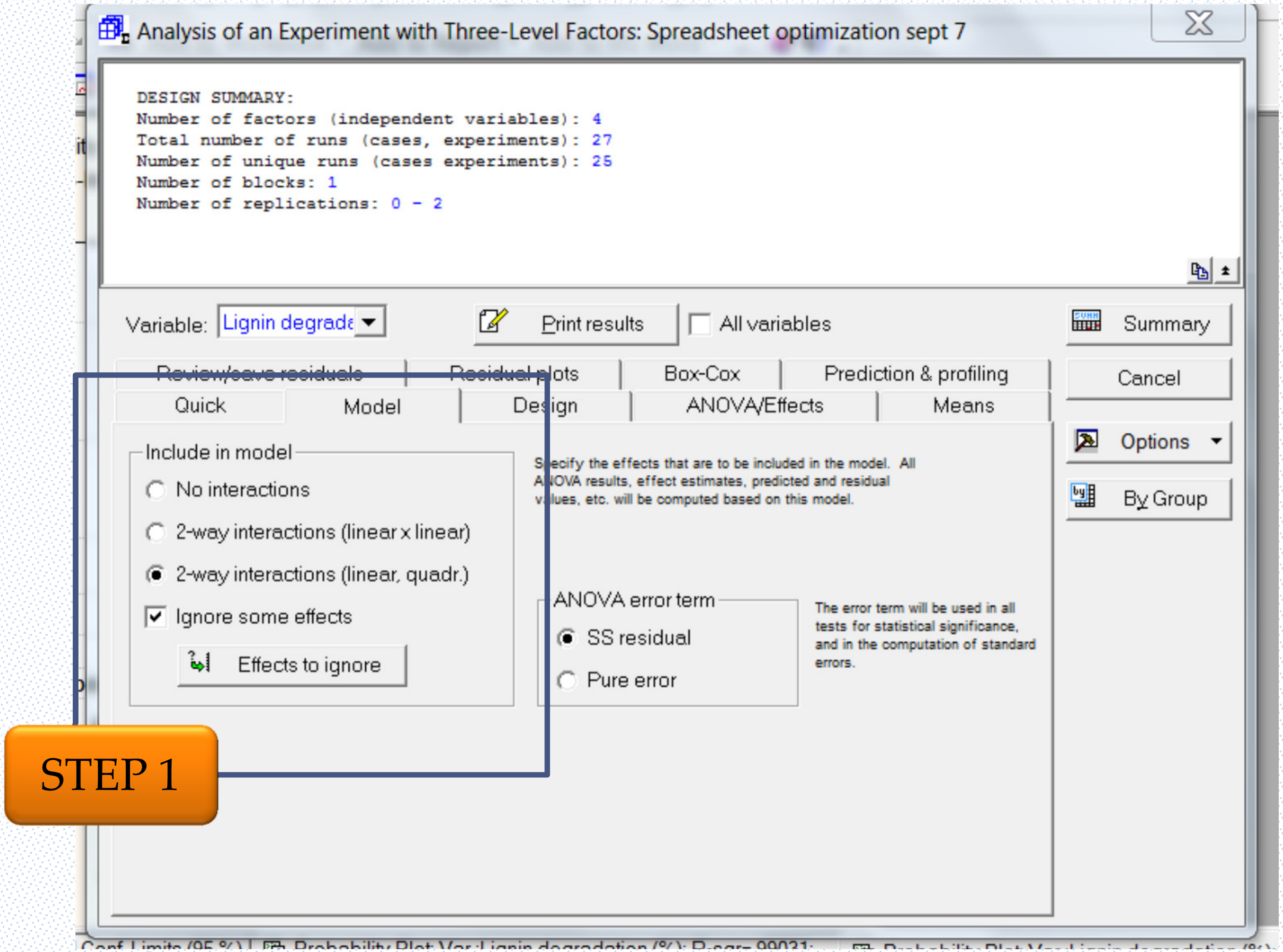
Click file → save project as

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

STATISTICA TUTORIAL 3

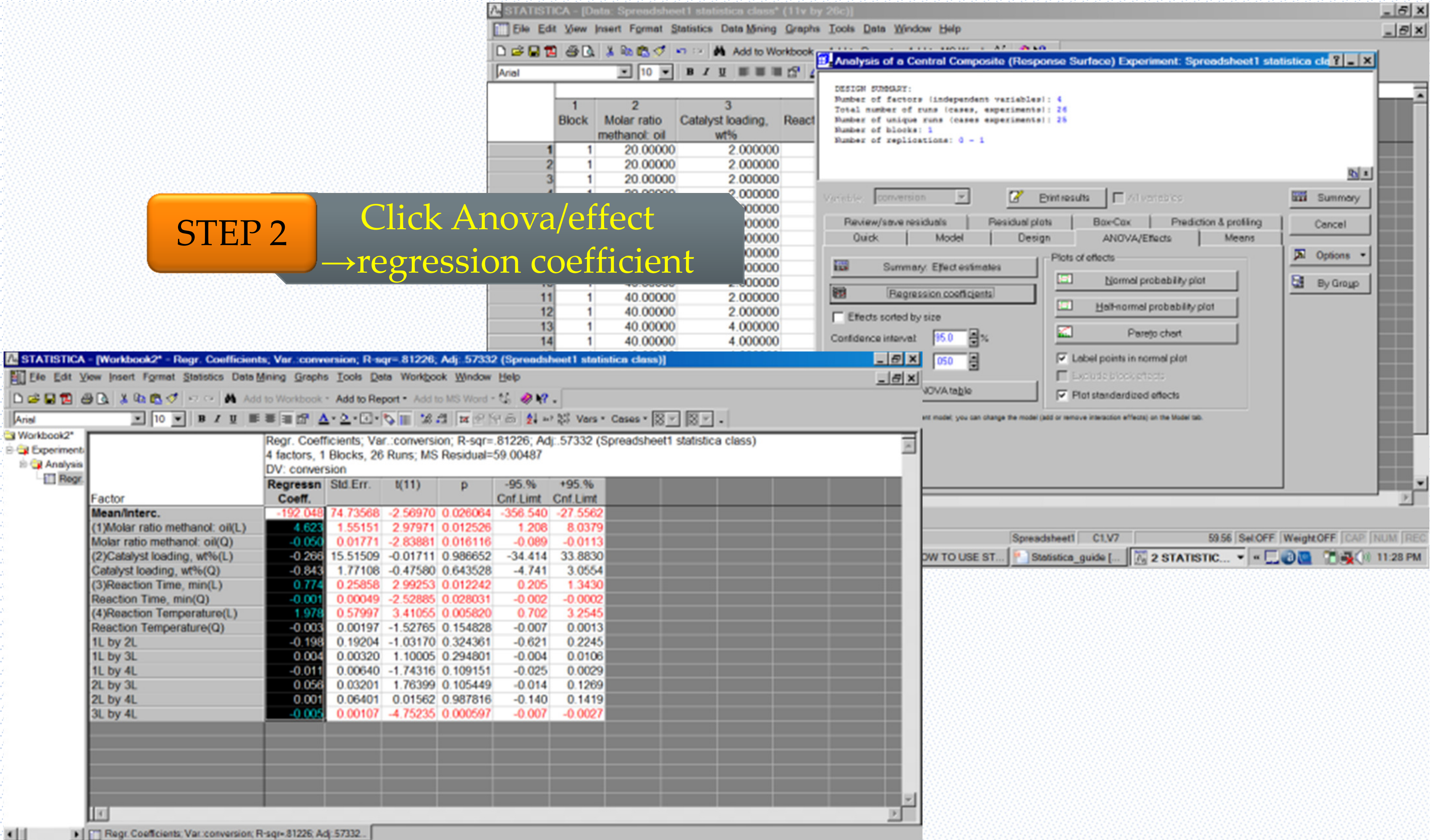
PREDICTED/EMPIRICAL MODEL

Model (Coefficient selection)



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

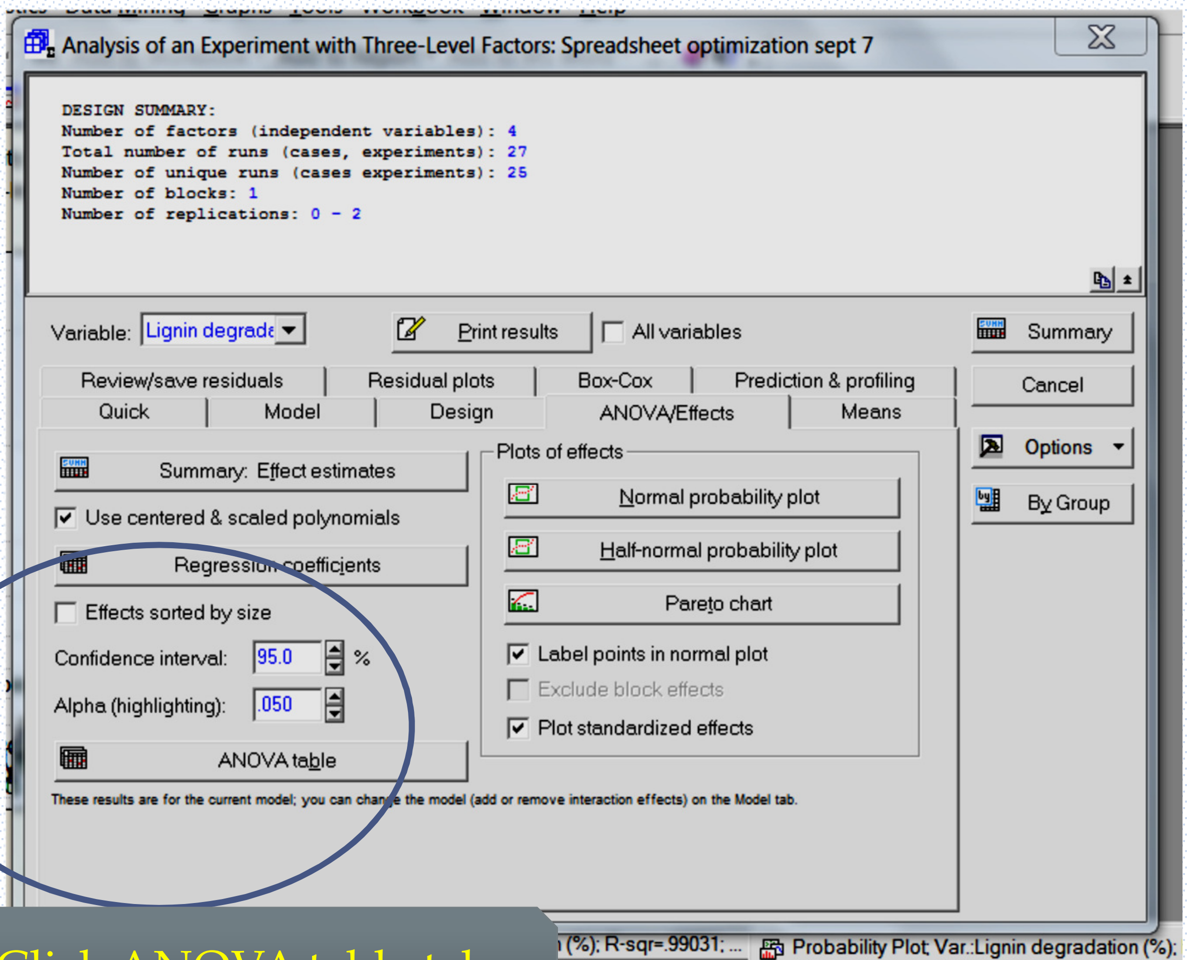
Factor	Regressn Coeff.	Std.Err.	t(11)	p	-95.% Cnf.Limt	+95.% Cnf.Limt
Mean/Interc.	-192.048	74.73568	-2.56970	0.026064	-356.540	-27.5562
(1)Molar ratio methanol: oil(L)	4.623	1.55151	2.97971	0.012526	1.208	8.0379
Molar ratio methanol: oil(Q)	-0.050	0.01771	-2.83881	0.016116	-0.089	-0.0113
(2)Catalyst loading, wt%(L)	-0.266	15.51509	-0.01711	0.986652	-34.414	33.8830
Catalyst loading, wt%(Q)	-0.843	1.77108	-0.47580	0.643528	-4.741	3.0554
(3)Reaction Time, min(L)	0.774	0.25858	2.99253	0.012242	0.205	1.3430
Reaction Time, min(Q)	-0.001	0.00049	-2.52885	0.028031	-0.002	-0.0002
(4)Reaction Temperature(L)	1.978	0.57997	3.41055	0.005820	0.702	3.2545
Reaction Temperature(Q)	-0.003	0.00197	-1.52765	0.154828	-0.007	0.0013
1L by 2L	-0.198	0.19204	-1.03170	0.324361	-0.621	0.2245
1L by 3L	0.004	0.00320	1.10005	0.294801	-0.004	0.0106
1L by 4L	-0.011	0.00640	-1.74316	0.109151	-0.025	0.0029
2L by 3L	0.056	0.03201	1.76399	0.105449	-0.014	0.1269
2L by 4L	0.001	0.06401	0.01562	0.987816	-0.140	0.1419
3L by 4L	-0.005	0.00107	-4.75235	0.000597	-0.007	-0.0027

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

STATISTICA TUTORIAL 4

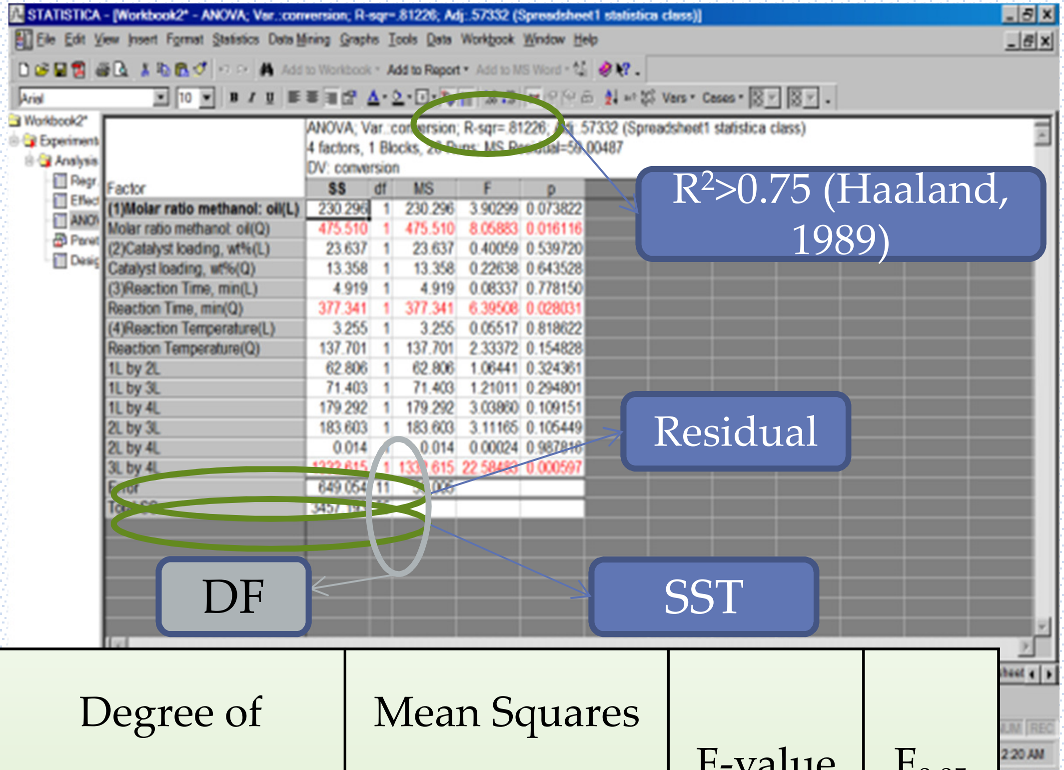
ANOVA

ANOVA/Effects



STEP Click ANOVA table tab

ANOVA table



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	>2.74
Residual	649.87	11	59.08		
Total (SST)	3457.29	25			

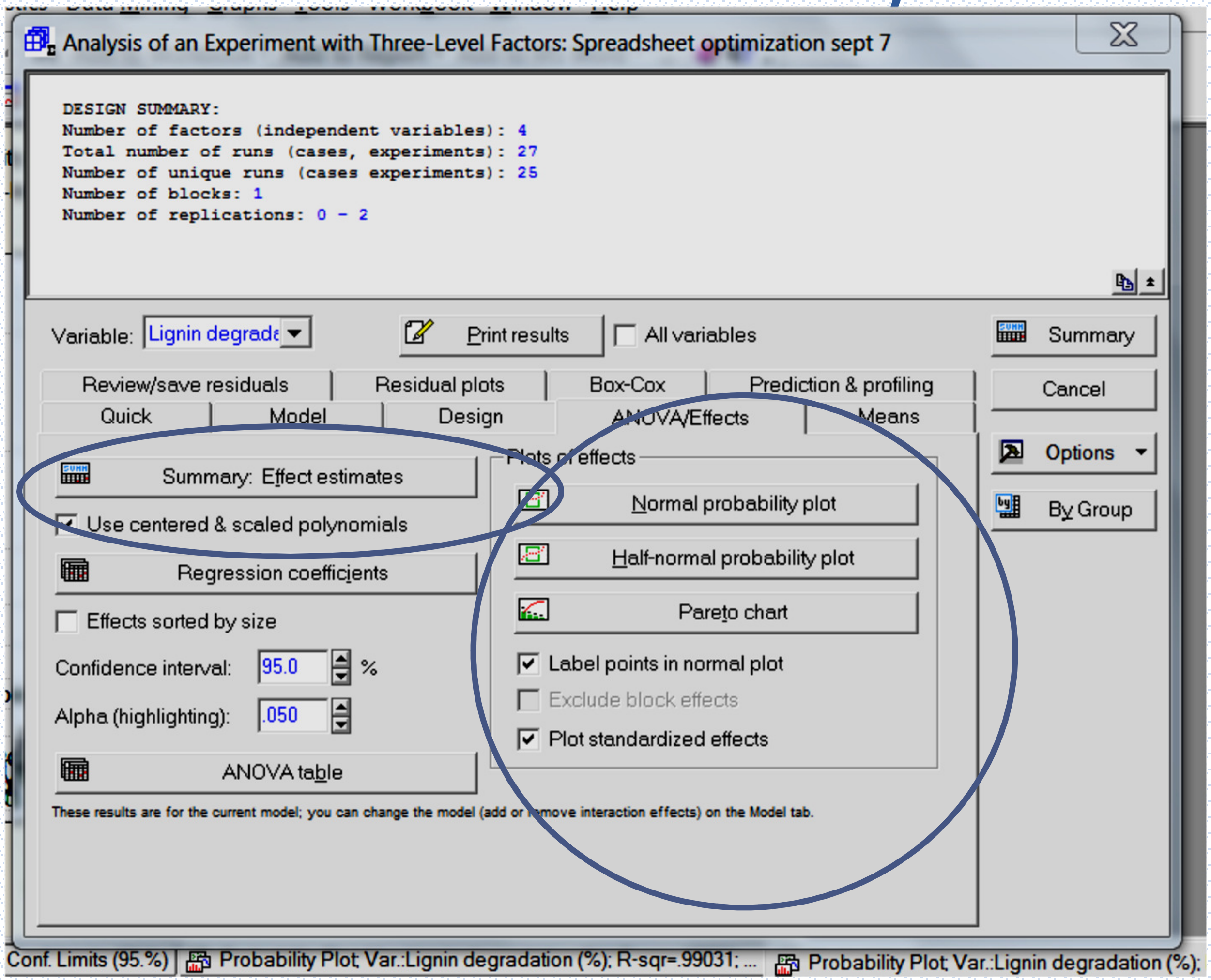
SSR= SST-residual

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

STATISTICA TUTORIAL 5

Effects

Tab of ANOVA/Effects



Effect estimates

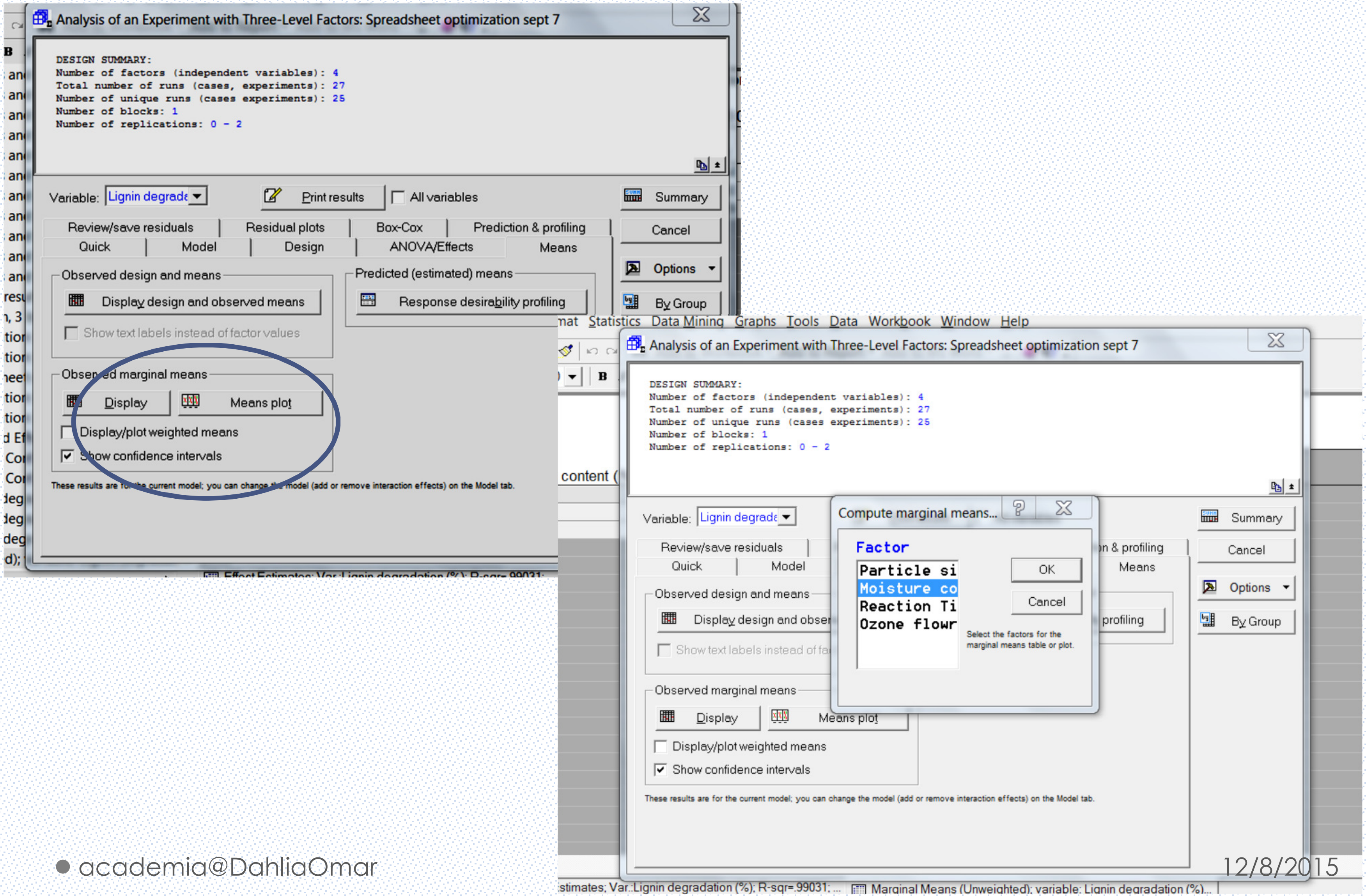
Effect Estimates; Var.:Lignin degradation (%); R-sqr=.99031; Adj:.87403 (Spreadsheet optimization sept 7)
4 3-level factors, 1 Blocks, 27 Runs; MS Residual=10.05794
DV: Lignin degradation (%)

Factor	Effect	Std.Err.	t(2)	p	-95.% Cnf.Limt	+95.% Cnf.Limt	Coeff.	Std.Err. Coeff.	-95.% Cnf.Limt	+95.% Cnf.Limt
Mean/Interc.	74.7109	1.618268	46.16721	0.000469	67.7481	81.67374	74.71089	1.618268	67.7481	81.67374
(1)Particle size (mm)(L)	19.6213	3.171426	6.18691	0.025144	5.9758	33.26686	9.81066	1.585713	2.9879	16.63343
Particle size (mm)(Q)	8.1603	2.310647	3.53162	0.071665	-1.7816	18.10225	4.08017	1.155324	-0.8908	9.05112
(2)Moisture content (%)(L)	7.4338	3.178466	2.33880	0.144277	-6.2420	21.10963	3.71690	1.589233	-3.1210	10.55482
Moisture content (%)(Q)	2.8125	1.965687	1.43078	0.288787	-5.6452	11.27014	1.40623	0.982844	-2.8226	5.63507
(3)Reaction Time (min)(L)	1.9354	3.178466	0.60890	0.604542	-11.7405	15.61119	0.96768	1.589233	-5.8702	7.80560
Reaction Time (min)(Q)	3.6935	1.965687	1.87896	0.201020	-4.7642	12.15112	1.84673	0.982844	-2.3821	6.07556
(4)Ozone flowrate (LPM)(L)	-7.3450	3.178466	-2.31086	0.147050	-21.0208	6.33083	-3.67250	1.589233	-10.5104	3.16542
Ozone flowrate (LPM)(Q)	5.4365	1.650462	3.29390	0.081112	-1.6649	12.53783	2.71823	0.825231	-0.8325	6.26891
1L by 2L	-13.7577	3.171426	-4.33802	0.049247	-27.4033	-0.11217	-6.87886	1.585713	-13.7016	-0.05609
1L by 2Q	-5.0159	2.242537	-2.23672	0.154775	-14.6648	4.63292	-2.50797	1.121268	-7.3324	2.31646
1Q by 2L	-6.3382	2.264851	-2.79849	0.107490	-16.0830	3.40669	-3.16909	1.132425	-8.0415	1.70335
1Q by 2Q	-9.1473	1.601491	-5.71174	0.029311	-16.0380	-2.25663	-4.57365	0.800746	-8.0190	-1.12832
1L by 3L	1.3490	3.171426	0.42537	0.711968	-12.2965	14.99456	0.67451	1.585713	-6.1483	7.49728
1L by 3Q	-0.9315	2.242537	-0.41536	0.718199	-10.5803	8.71739	-0.46573	1.121268	-5.2902	4.35870
1Q by 3L	-2.6225	2.264851	-1.15793	0.366486	-12.3674	7.12234	-1.31126	1.132425	-6.1837	3.56117
1Q by 3Q	-2.0845	1.601491	-1.30163	0.322788	-8.9752	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	2.264851	1.38910	0.299255	-6.5988	12.89098	1.57305	1.132425	-3.2994	6.44549
2L by 3L	1.8843	3.171426	0.59416	0.612662	-11.7612	15.52988	0.94217	1.585713	-5.8806	7.76494
2L by 3Q	2.3447	2.242537	1.04556	0.405508	-7.3041	11.99357	1.17236	1.121268	-3.6521	5.99679

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

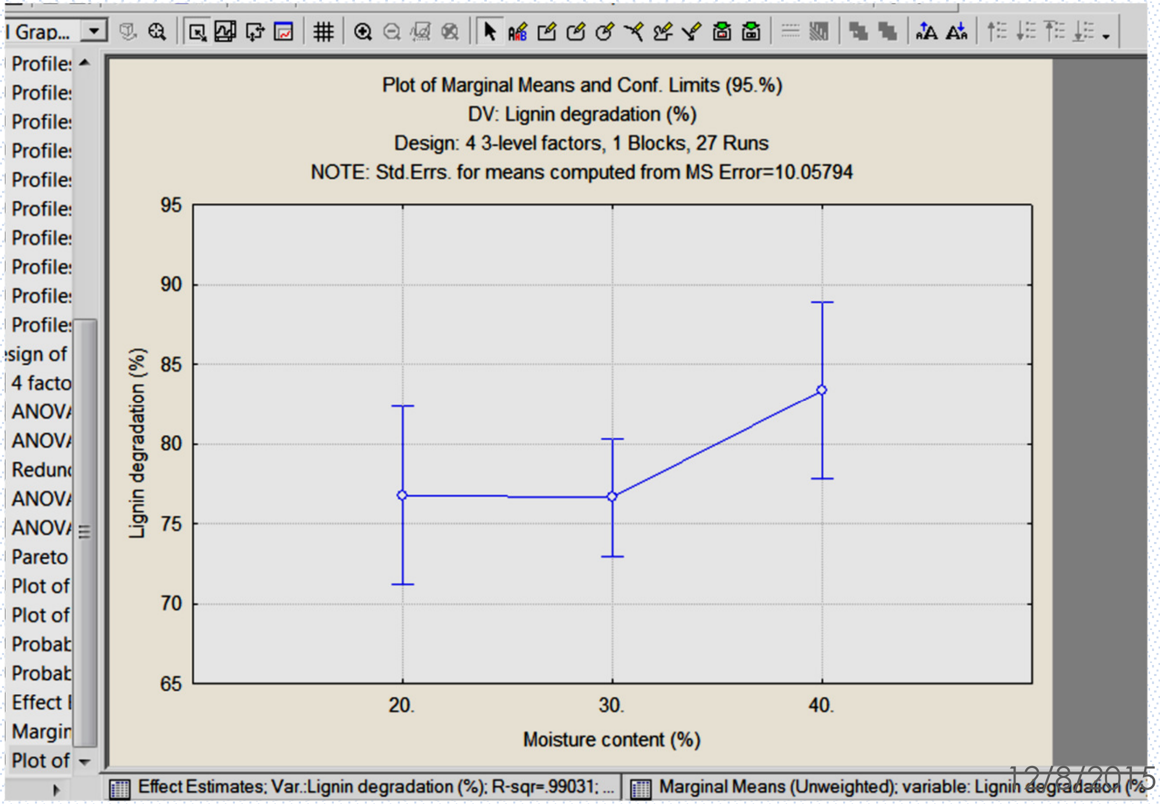
STATISTICA TUTORIAL 6

Mean Effect


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
Summary
Cancel
Options
By Group
Review/save residuals
Residual plots
Box-Cox
Prediction & profiling
Quick
Model
Design
ANOVA/Effects
Means
Observed design and means
Display design and observed means
Show text labels instead of factor values
Predicted (estimated) means
Response desirability profiling
Observed marginal means
Display
Means plot
Display/plot weighted means
Show confidence intervals
These results are for the current model; you can change the model (add or remove interaction effects) on the Model tab.
Statistics Data Mining Graphs Tools Data Workbook Window Help
Compute marginal means...
Factor
Particle si
Moisture co
Reaction Ti
Ozone flowr
OK
Cancel
Select the factors for the marginal means table or plot.
Marginal Means (Unweighted); variable: Lignin degradation (%)...

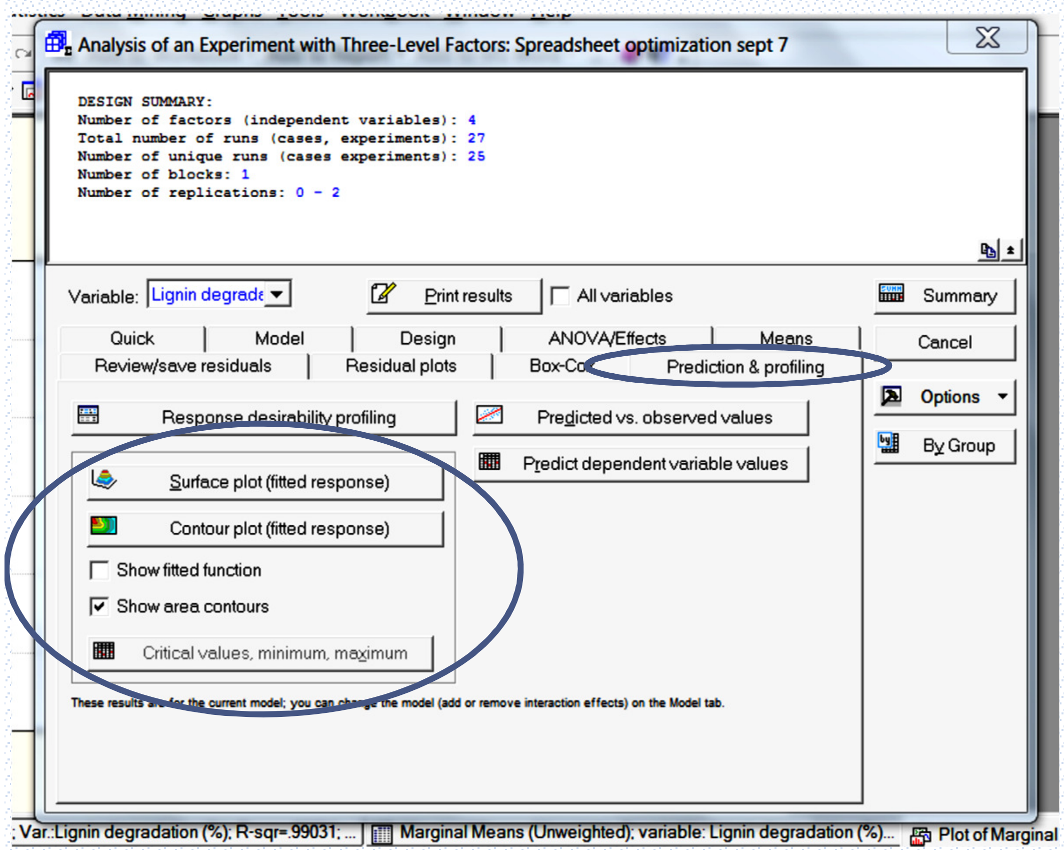
Marginal Means (Unweighted); variable: Lignin degradation (%) (Spreadsheet optimization sept 7)
Design: 4 3-level factors, 1 Blocks, 27 Runs
NOTE: Std.Errs. for means computed from MS Error=10.05794

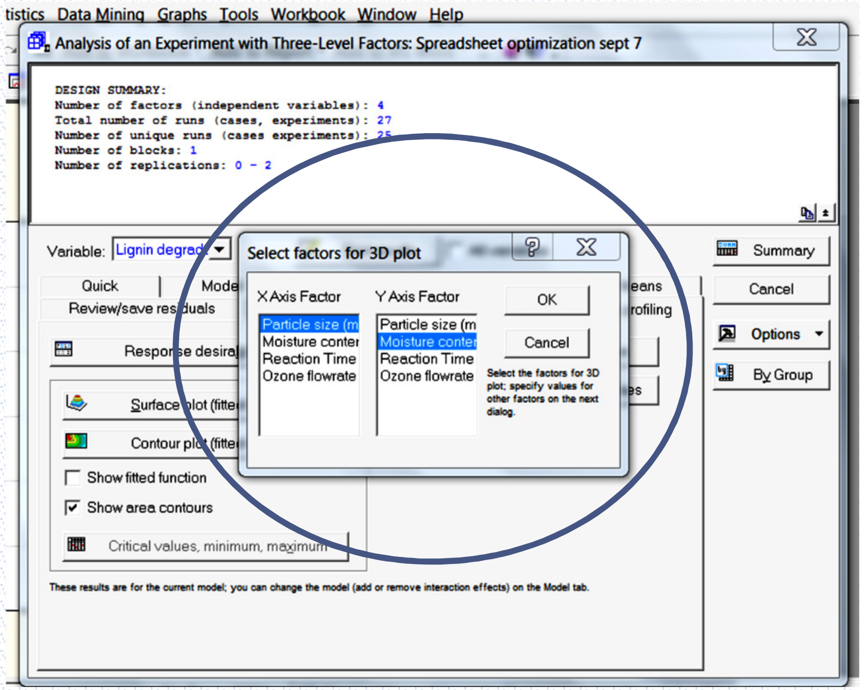
Moisture content (%)	Means	Pooled Std.Dev.	Overall Std.Dev.	N	Std.Err. for Mean	-95.% Cnf.Limt	+95.% Cnf.Limt
20.	76.75631	0.000000	13.80098	6	1.294729	71.18554	82.32708
30.	76.62517	3.171426	7.92173	15	0.856745	72.93890	80.31145
40.	83.36216	0.000000	2.74082	6	1.294729	77.79139	88.93293

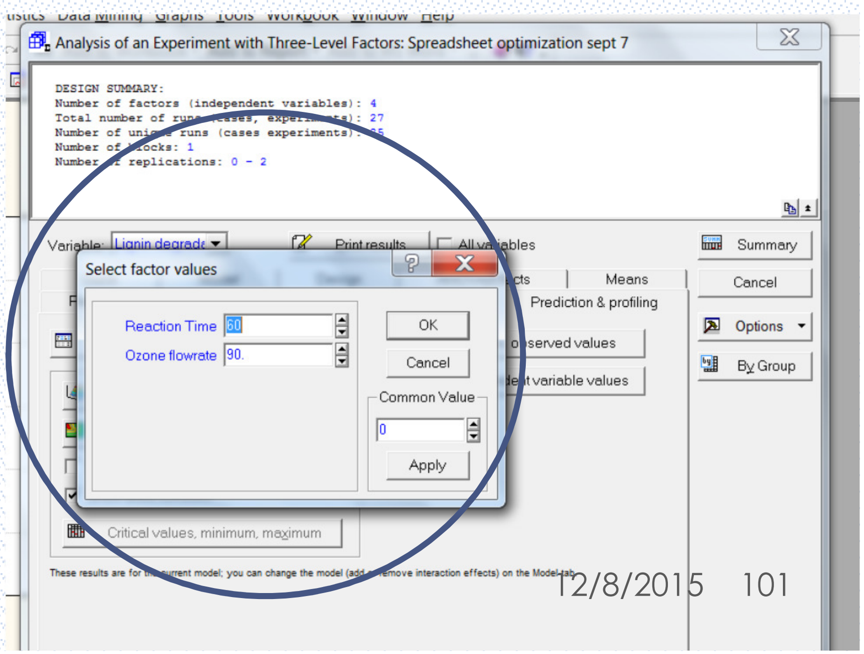


STATISTICA TUTORIAL 7

Contour plot

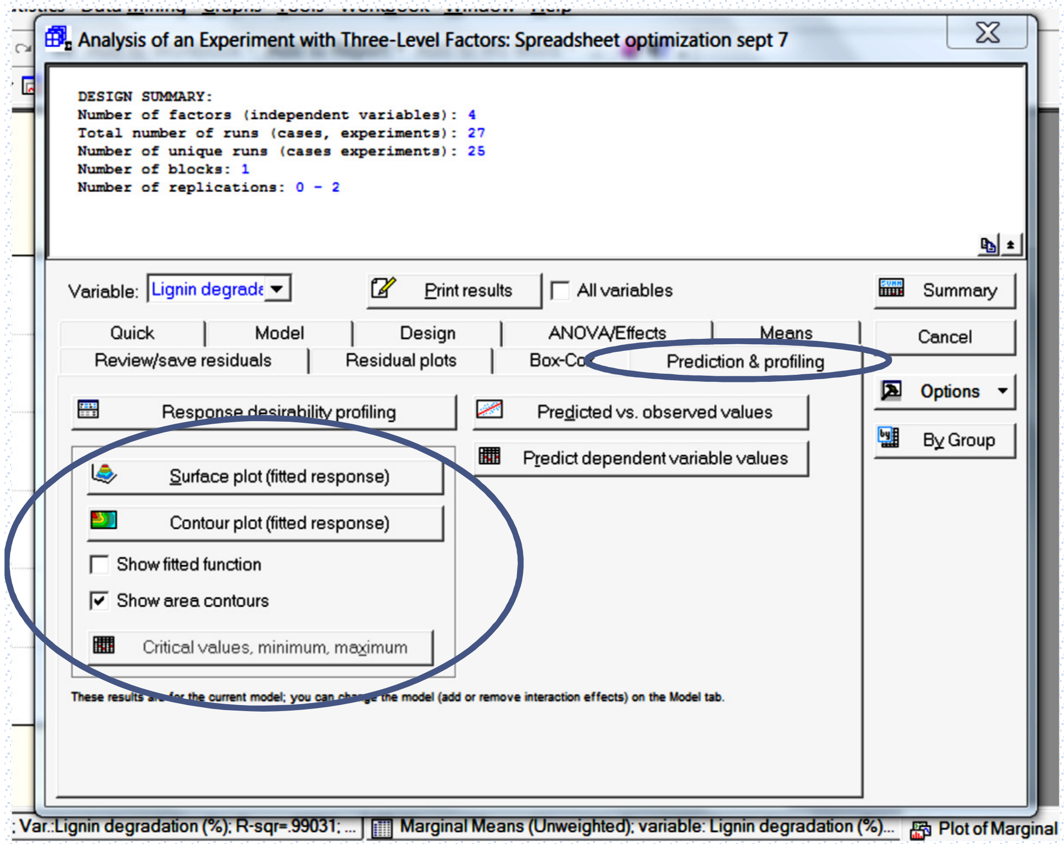

Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7
DESIGN SUMMARY:
Number of factors (independent variables): 4
Total number of runs (cases, experiments): 27
Number of unique runs (cases experiments): 25
Number of blocks: 1
Number of replications: 0 - 2
Variable: Lignin degrade
Print results
All variables
Summary
Quick
Model
Design
ANOVA/Effects
Means
Review/save residuals
Residual plots
Prediction & profiling
Cancel
Options
By Group
Response desirability profiling
Predicted vs. observed values
Predict dependent variable 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.
Marginal Means (Unweighted); variable: Lignin degradation (%)...
Plot of Marginal

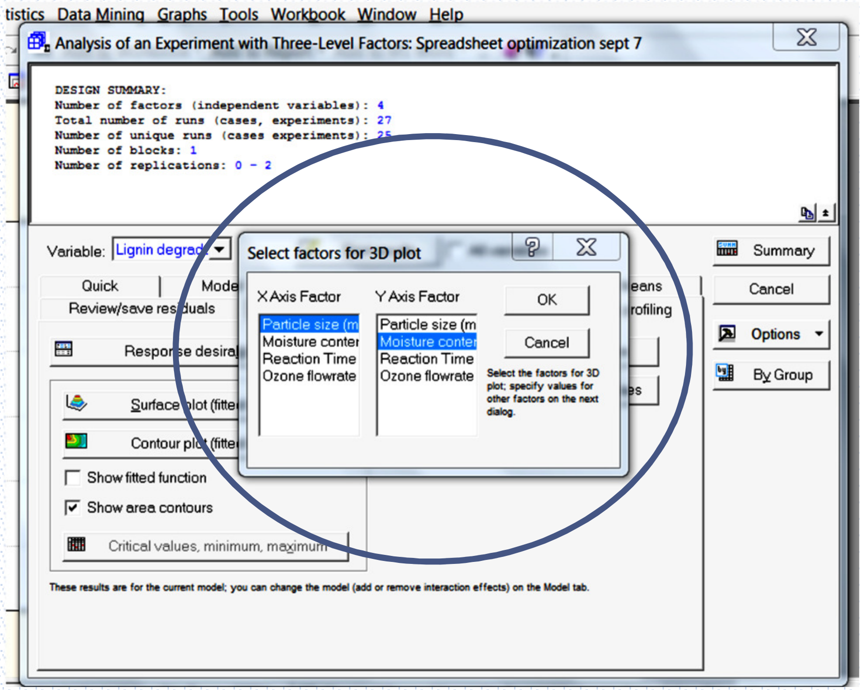

Select factors for 3D plot
X Axis Factor
Y Axis Factor
Particle size (m
Moisture conter
Reaction Time
Ozone flowrate
OK
Cancel
Select the factors for 3D plot; specify values for other factors on the next dialog.

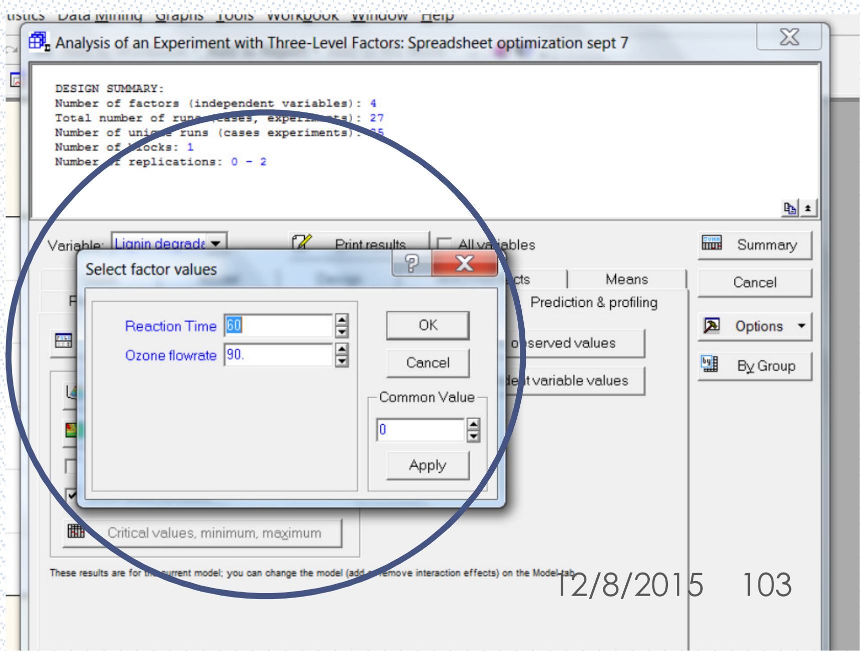

Select factor values
Reaction Time 60
Ozone flowrate 90.
OK
Cancel
Common Value
0
Apply

STATISTICA TUTORIAL 8

3D Surface


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
Summary
Quick
Model
Design
ANOVA/Effects
Means
Cancel
Review/save residuals
Residual plots
Box-Co
Prediction & profiling
Options
Response desirability profiling
Predicted vs. observed values
Predict dependent variable values
By Group
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.
; Var.:Lignin degradation (%); R-sqr=.99031; ...
Marginal Means (Unweighted); variable: Lignin degradation (%)...
Plot of Marginal


tistics Data Mining Graphs Tools Workbook Window Help
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
Select factors for 3D plot
X Axis Factor
Y Axis Factor
Particle size (m
Moisture conter
Reaction Time
Ozone flowrate
OK
Cancel
Select the factors for 3D plot; specify values for other factors on the next dialog.
Summary
Cancel
Options
By Group
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.

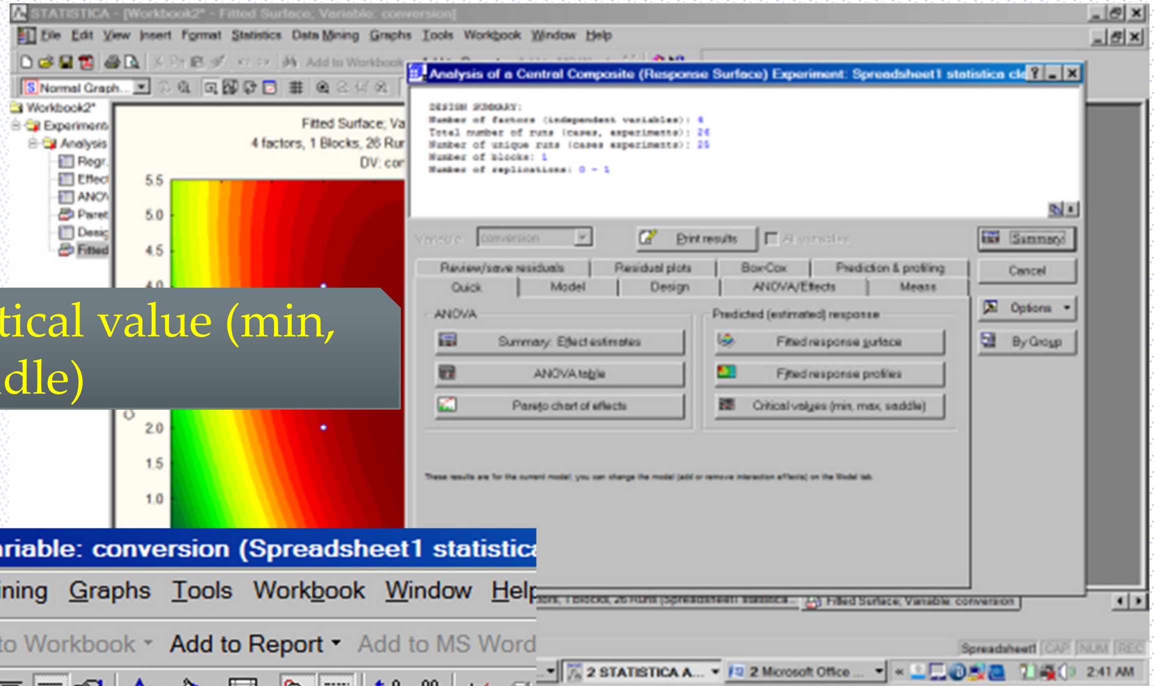

Analysis of an Experiment with Three-Level Factors: Spreadsheet optimization sept 7
Select factor values
Reaction Time 60
Ozone flowrate 90.
OK
Cancel
Common Value
0
Apply
Summary
Cancel
Options
By Group
Means
Prediction & profiling
Critical values, minimum, maximum

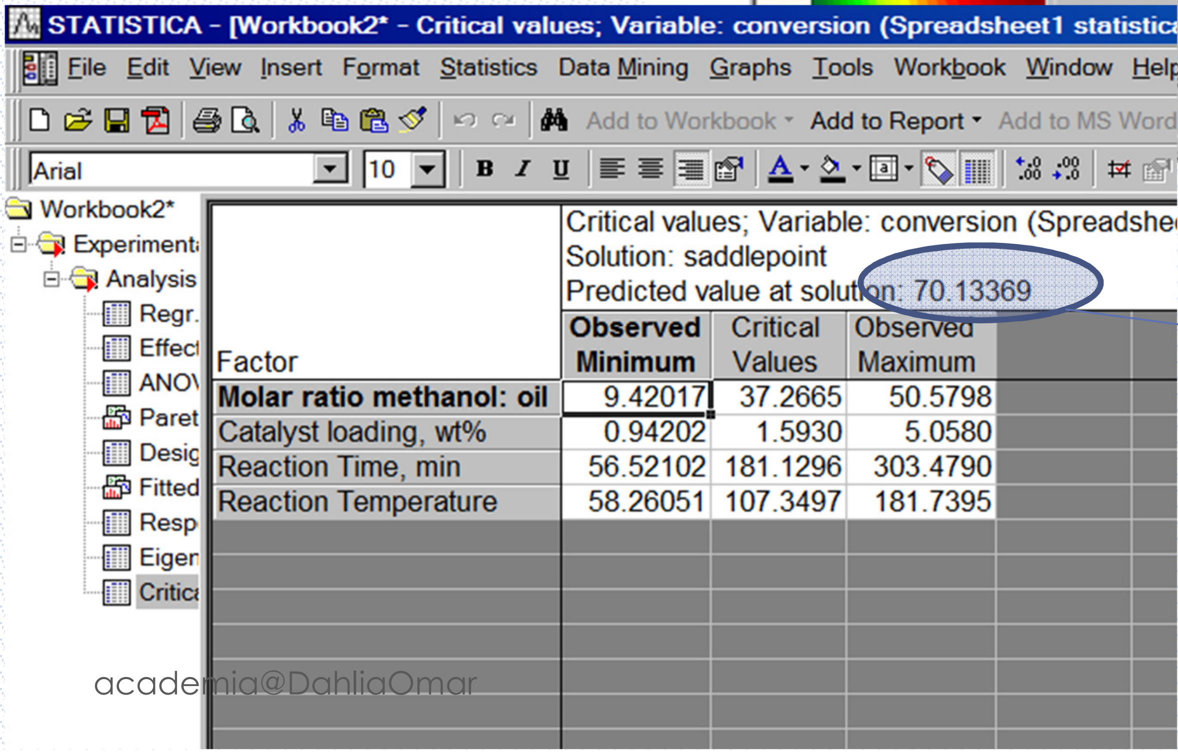
STATISTICA TUTORIAL 9

Optimization: Single response

OPTIMIZATION: Single responses

STEP 1 Click quick tab→ critical value (min, max, saddle)



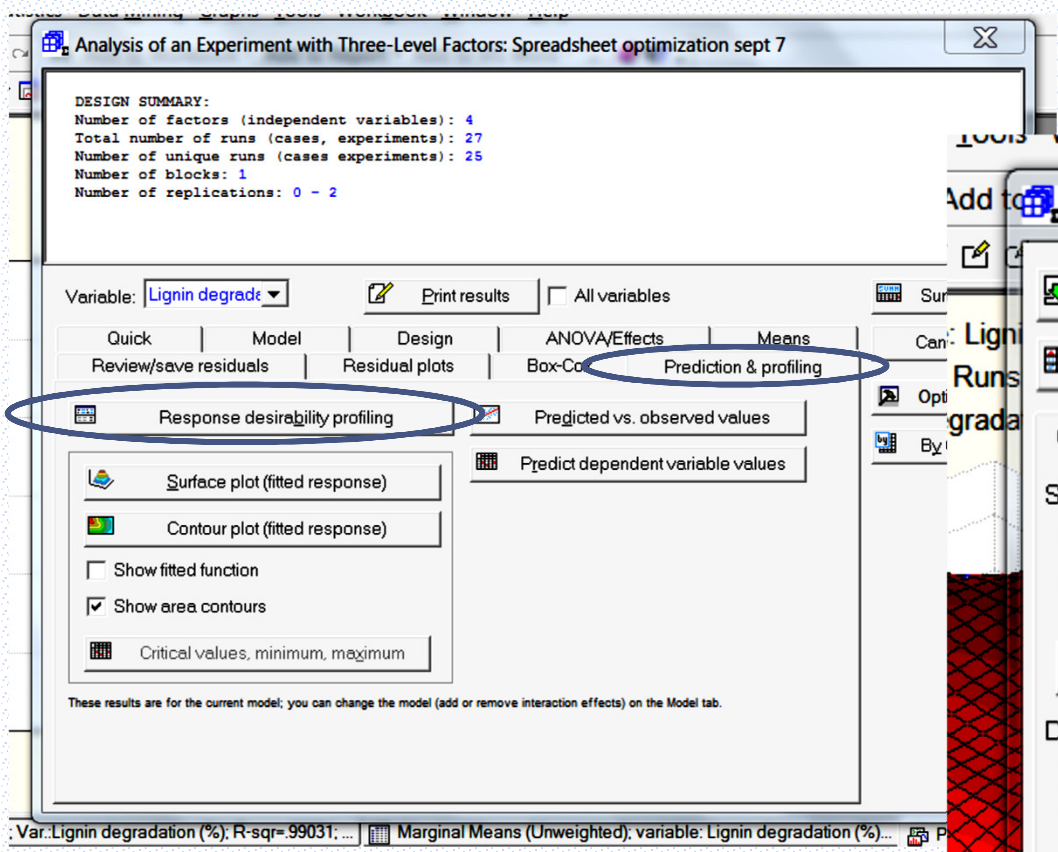


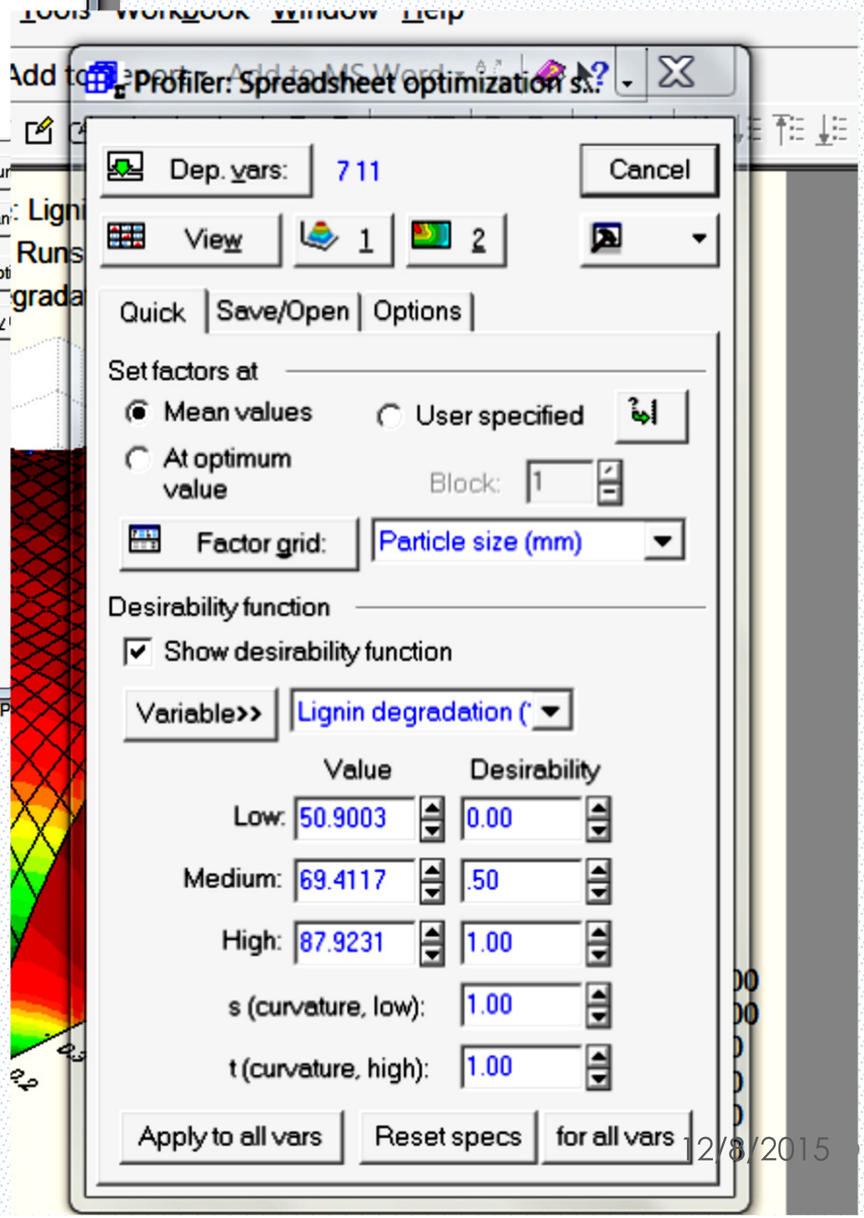
Factor	Observed Minimum	Critical Values	Observed Maximum
Molar ratio methanol: oil	9.42017	37.2665	50.5798
Catalyst loading, wt%	0.94202	1.5930	5.0580
Reaction Time, min	56.52102	181.1296	303.4790
Reaction Temperature	58.26051	107.3497	181.7395

Predicted response

STATISTICA TUTORIAL 10

Optimization: Desirability function


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
Review/save residuals
Residual plots
Prediction & profiling
Response desirability profiling
Predicted vs. observed values
Predict dependent variable 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.
Var.:Lignin degradation (%); R-sqr=.99031; ...
Marginal Means (Unweighted); variable: Lignin degradation (%)...


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