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# Metodologi Riset dan Analisis Data Kuantitatif

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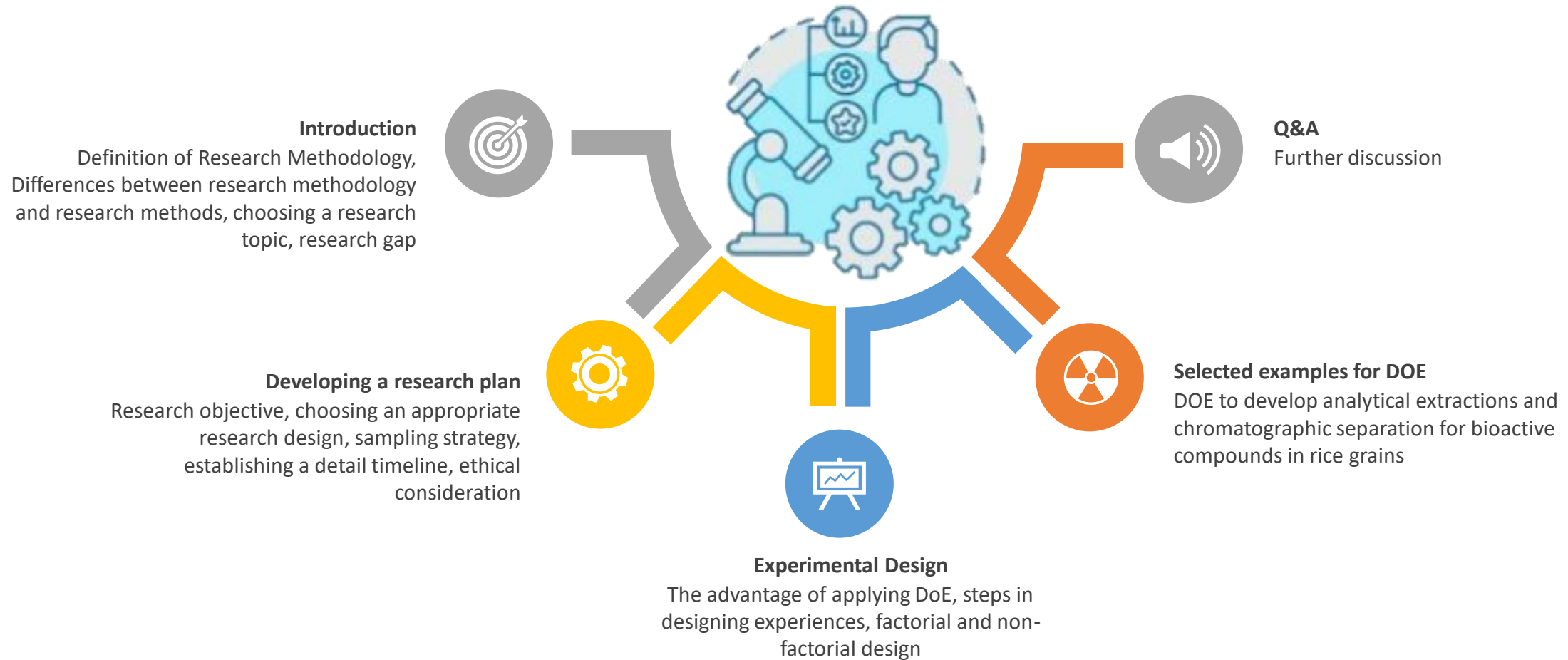
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Disampaikan pada acara  
Workshop Metodologi Riset Kuantitatif dan Kualitatif  
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29 Februari 2024



# Content-Index



# Introduction



## Research Methodologies

the science of study of how research is done systematically

*Is the specific techniques used to identify, select, process, and analyze information about a topic*

### QUALITATIVE RESEARCH

Qualitative research investigates human experiences and behavior through non-numerical data collection and analysis.

### QUANTITATIVE RESEARCH

Quantitative research examines relationships between variables using numerical data and statistical techniques.

### MIXED METHODS RESEARCH

Mixed methods research combines qualitative and quantitative approaches to provide a more holistic understanding of a research question.

## + Key point

Research methodology and research methods are distinctly different things

### + Research methodology

- + Methodology is a theoretical aspect on how research should be undertaken.
- + Research methodology is a systematic way to solve a problem. It is a science of studying how research is to be carried out.
- + Essentially, the procedures by which researchers go about their work of describing, explaining and predicting phenomena are called research methodology.
- + It is also defined as the study of methods by which knowledge is gained. Its aim is to give the work plan of research.
- + Research methodology seeks to answer: how did the researcher complete his study.

### + Research Methods/design

- Research methods are the various procedures, schemes, steps and algorithms used in research.
- All the methods used by a researcher during a research study are termed as research methods.
- They are essentially planned, scientific and value-neutral. They include observations, theoretical procedures, experimental studies, numerical schemes, statistical approaches, etc. Research methods help us collect samples, data and find a solution to a problem.
- A subset of research methodology
- Research method seeks to answer: what did the researcher use to complete his research.



# Choosing a Research Topic

It sets the foundation for your entire study and determines the direction in which you will proceed



*The Do's of choosing a research topic*

- Choose a topic that interests you
- Ensure the topic is relevant
- Keep the topic focused
- Ensure you have enough resources to support your research

What is examined thus far in the available literatures?

What is not studied yet?



The deficiency in the literature

- Exhaustive reading
- Critical review
- Future research direction
- Lack of understanding
- Limitations of previous study



# Developing a Research Plan

A research plan serves as a roadmap, guiding researchers through the various stages of their study and ensuring that they stay focused and organized.



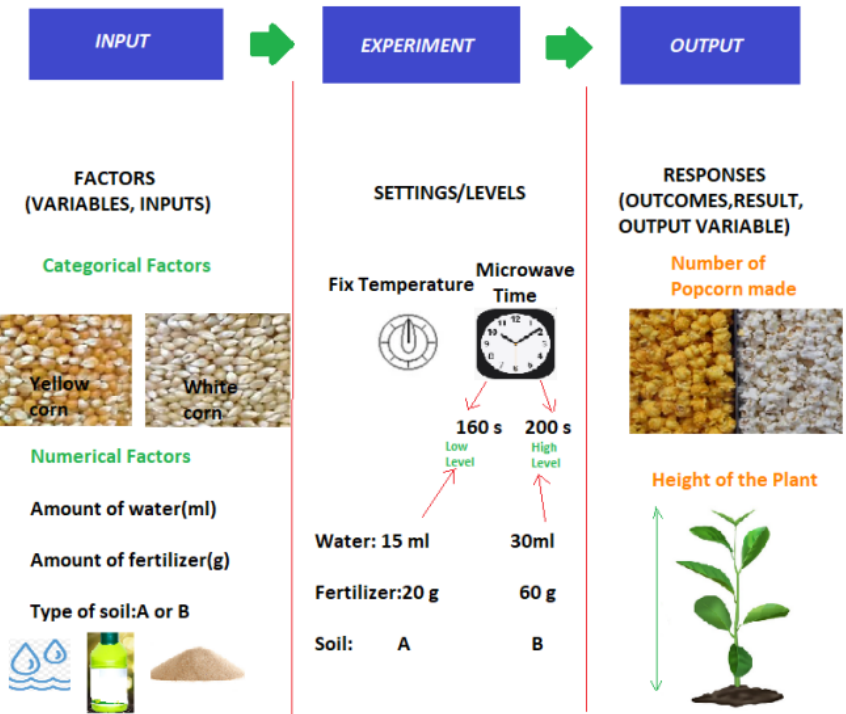
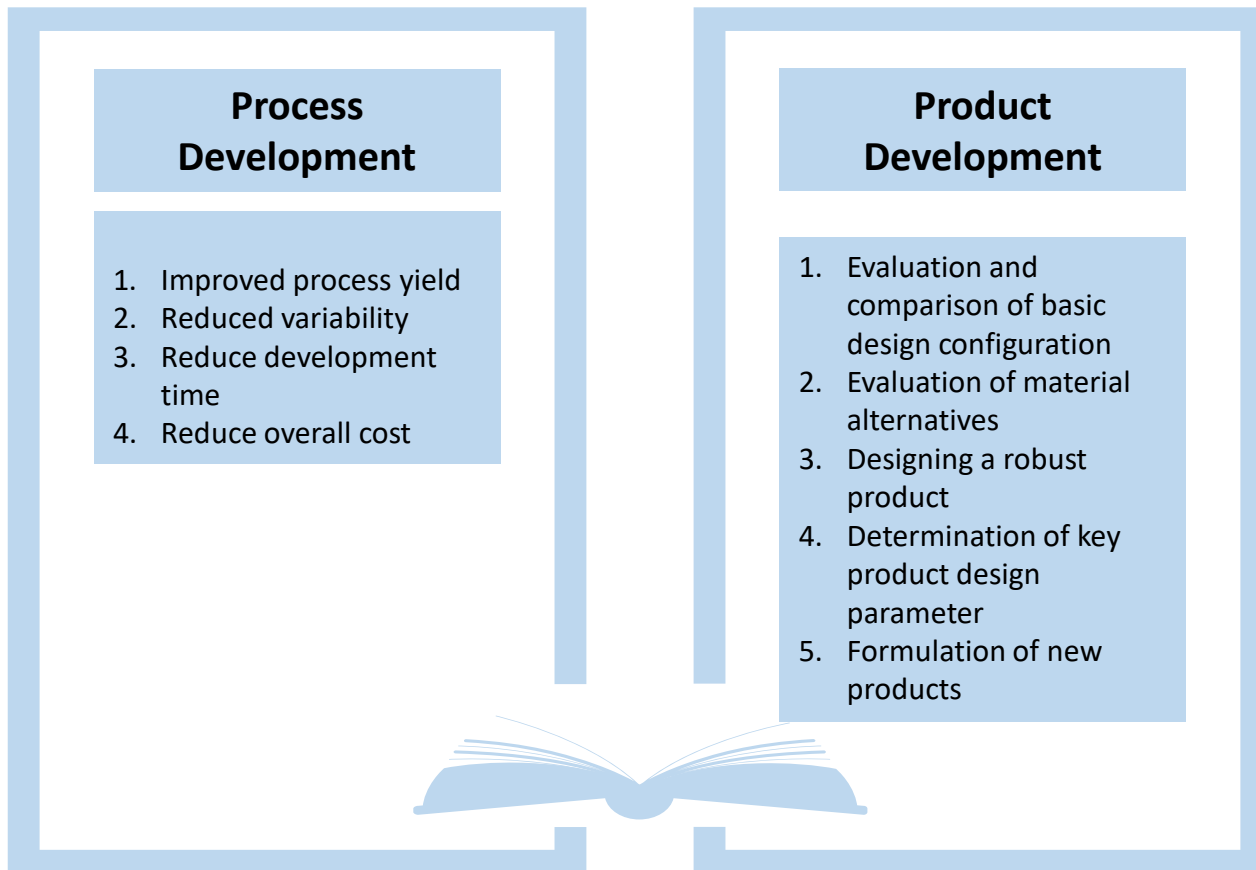
Strategic plan or outline that lays out the direction, goals, and milestones for a specific area of research over a defined period of time.

## Research Road Map

By outlining the key objectives, methods, and timeline, a research plan helps researchers to stay on track and avoid unnecessary detours.

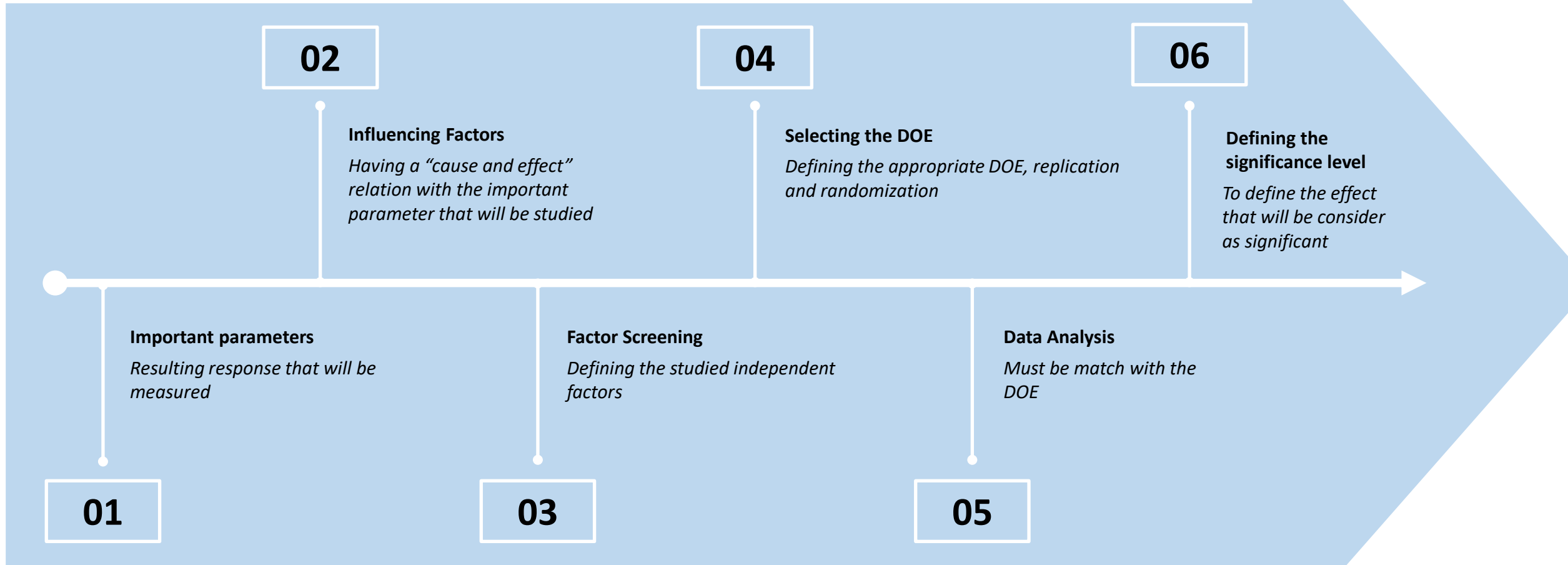


# Application of DOE

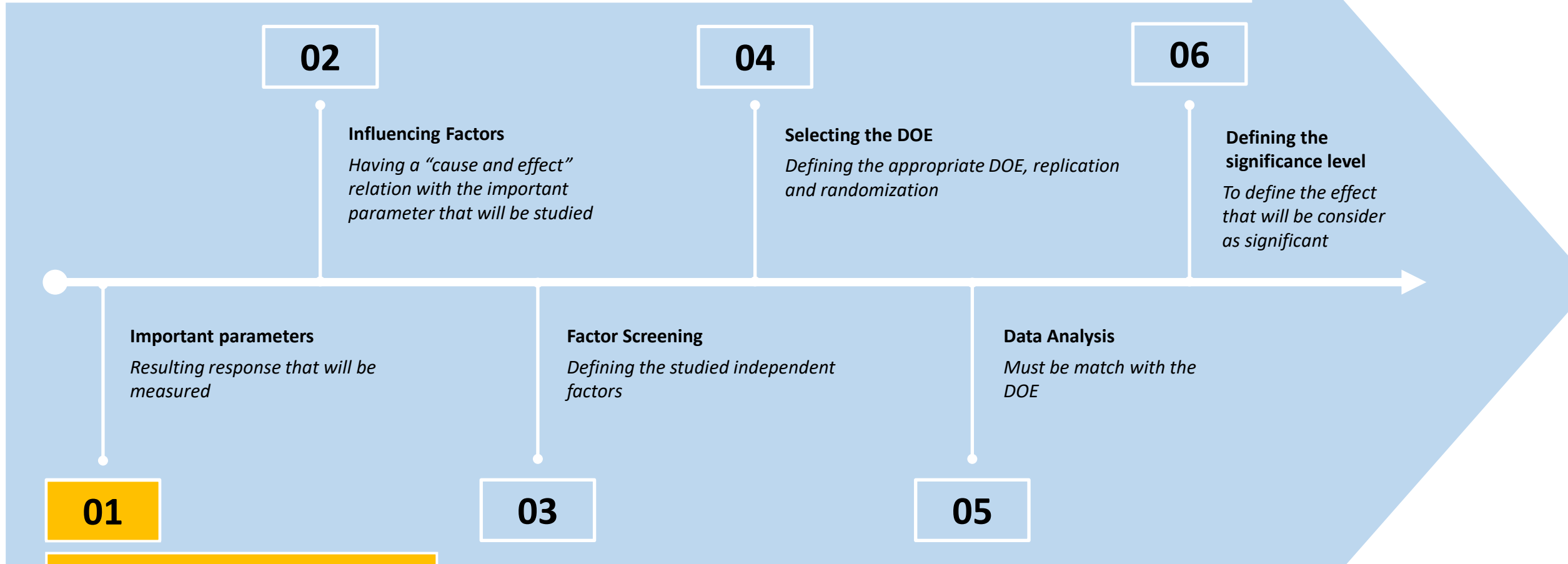


DOE is widely applied in various applications

# Steps in designing experiments



# Steps in designing experiments



**Dependent Variable**





**Independent Variable**

**Controlled Variable**

**Uncontrolled Variable**

**02**

**Influencing Factors**

*Having a "cause and effect" relation with the important parameter that will be studied*

***cause-and-effect diagram***

**04**

**Selecting the DOE**

*Defining the appropriate DOE, replication and randomization*

**06**

**Defining the significance level**

*To define the effect that will be consider as significant*

**Important parameters**

*Resulting response that will be measured*

**01**

**Dependent Variable**

**03**

**Factor Screening**

*Defining the studied independent factors*

**05**

**Data Analysis**

*Must be match with the DOE*



**Independent Variable**

**Controlled Variable**

**Uncontrolled Variable**

*Basic principle: Blocking*

**02**

**Influencing Factors**

*Having a "cause and effect" relation with the important parameter that will be studied*

**cause-and-effect diagram**

**04**

**Selecting the DOE**

*Defining the appropriate DOE, replication and randomization*

**Basic principle: Replication and Randomization**

**06**

**Defining the significance level**

*To define the effect that will be considered as significant*

**Important parameters**

*Resulting response that will be measured*

**01**

**Dependent Variable**

**Factor Screening**

*Defining the studied independent factors*

**03**

**Independent Variable**

**Data Analysis**

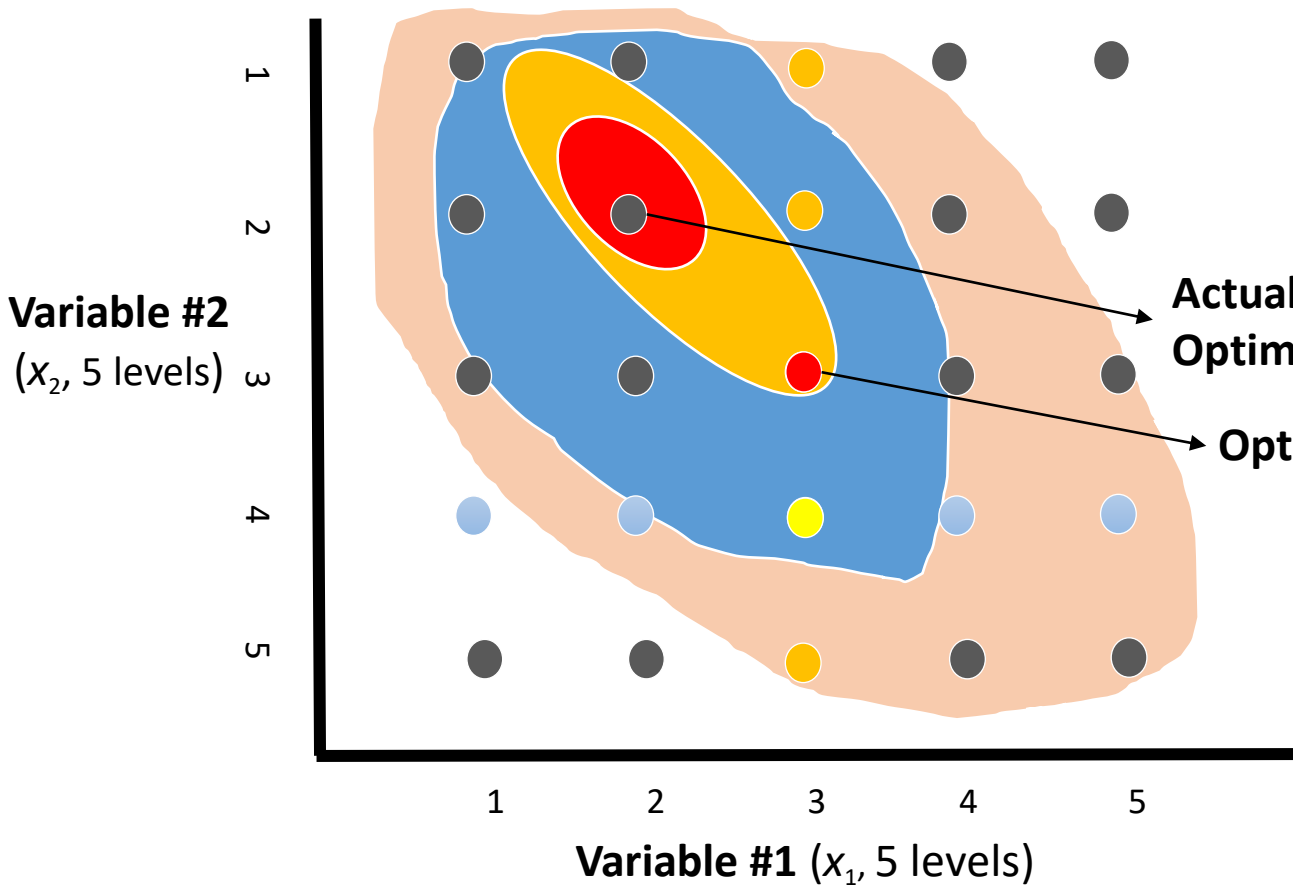
*Must be match with the DOE*

**05**

# Factorial and non-factorial designs,



## Optimization of multiple variables



**Non-Factorial**  
Using a One-factor-at-a-time approach

**Factorial**  
The variables are evaluated and included in the DOE simultaneously

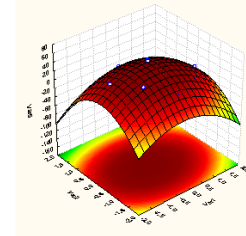
### Full Factorial Design

$5 \times 5 = 25$  → 2 variables – 25  
3 variables – 125

### Interaction

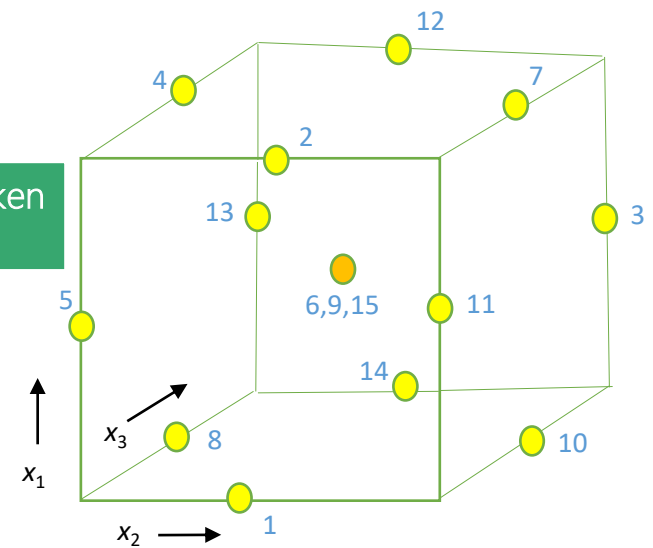
**Cartesiano x Superficie**

2	25	11
3	125	17



**Actual Optimum**  
**Optimum**

### Box-Behnken Design



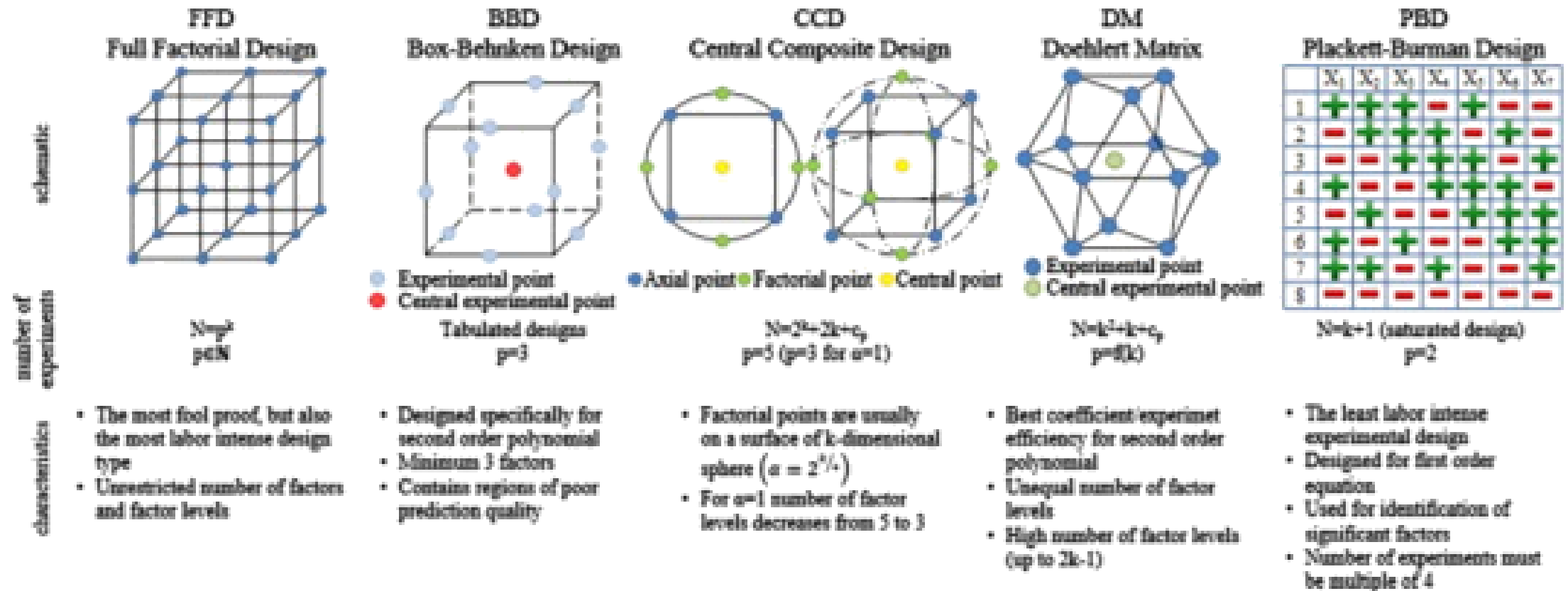
### Non-Factorial Design

$5 + 5 = 10$



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## DESIGN OF EXPERIMENTS



LOCALLY ROOTED, GLOBALLY RESPECTED



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## Examples

DOE to develop analytical extractions and chromatographic separation for **bioactive compounds** in rice grains

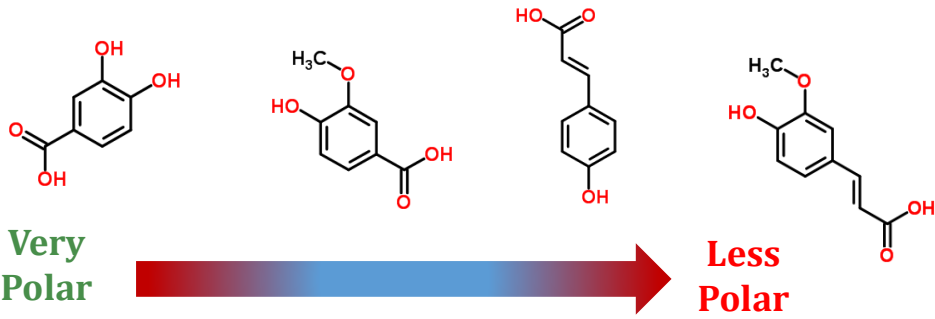
LOCALLY ROOTED, GLOBALLY RESPECTED

# Introduction

Research Problems



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Potent antioxidant activities

A huge variety of phenolic compounds

Amphipathic property

Complex matrix



Very low levels of analytes



ppb Levels

ppm Levels

# Materials and Methods

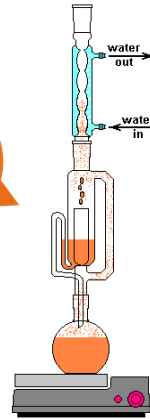


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## Analytical Techniques

### EXTRACTION

>24 h



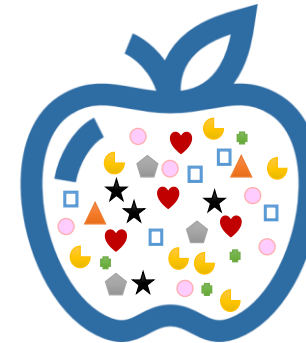
MAE

Ethos 1600 (Milestone S.r.l., Sorisole, Italy)



PLE

Dionex ASE 200 extractor (Dionex, Sunnyvale, CA, USA)



Analyte



Interference



### SEPARATION



HPLC-PDA

Dionex P680 coupled with Dionex PDA-100 Photodiode Array Detector (Dionex, Sunnyvale, CA, USA).



UPLC-PDA-FD

ACQUITY UPLC® H-Class system (Waters Corporation, Milford, MA, USA).



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## Example #1

DOE to develop analytical extractions for  
**melatonin and phenolic compounds** in  
**rice**: microwave-assisted and pressurised  
liquid extraction

LOCALLY ROOTED, GLOBALLY RESPECTED



## EXTRACTION FACTORS

Temperature

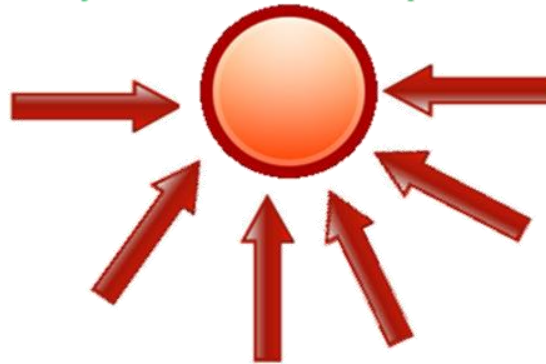
Microwave power

Solvent

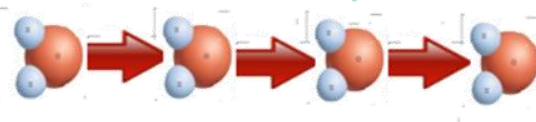
Sample:solvent ratio

Extraction time

Temperature outer >>>> temperature center



Heat is transferred molecule by molecule from outside



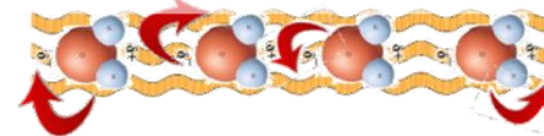
Overheating can occur on the outside

*Heating by Conduction*

Microwave penetrate the material and create rapidly changing fields



Dipoles continually react Attempting to align in the field which generates heat



The heat is uniformly distributed throughout the material

*Heating by Microwaves*



## Central Composite Design

### EXTRACTION FACTORS

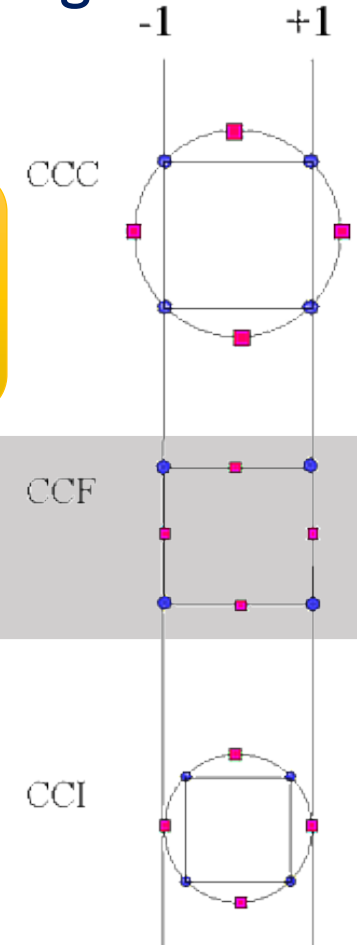
	-1	0	+1	Unit
Temperature	125	150	175	°C
Microwave power	500	750	1000	Watt
Solvent	5	15	10	min
Sample:solvent ratio	10	50	90	%
Extraction time	10	15	20	-

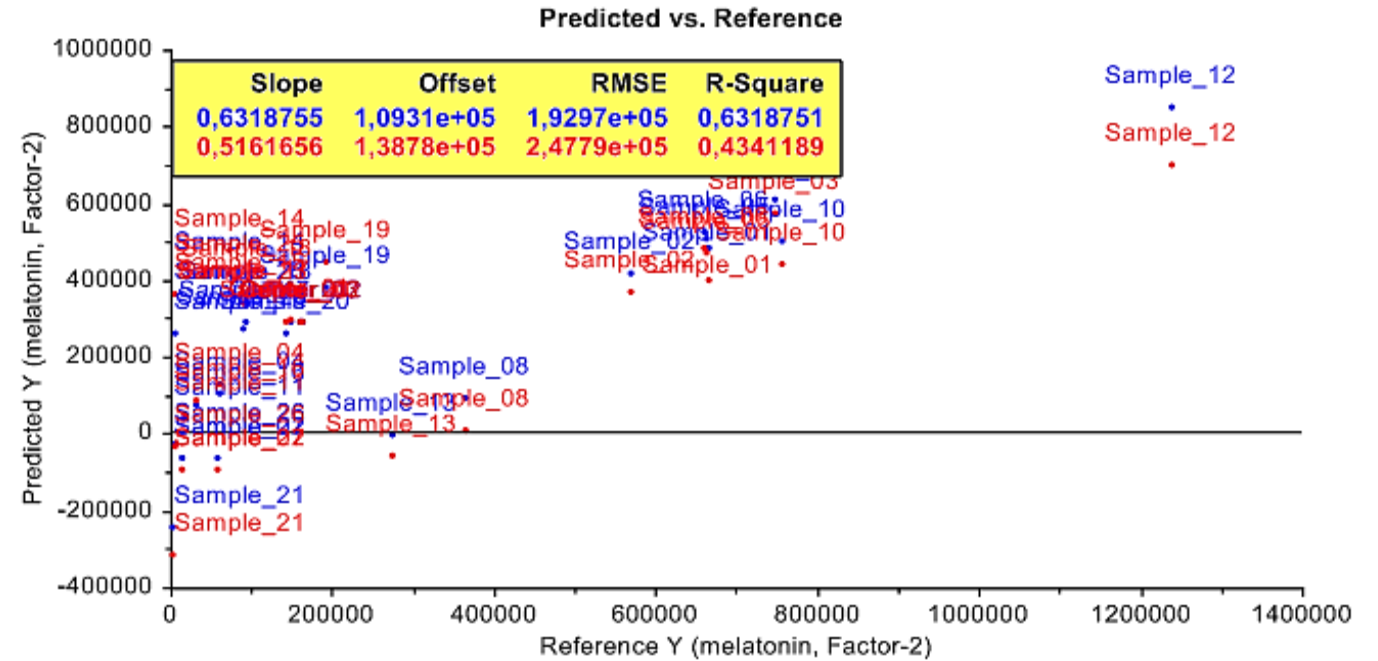
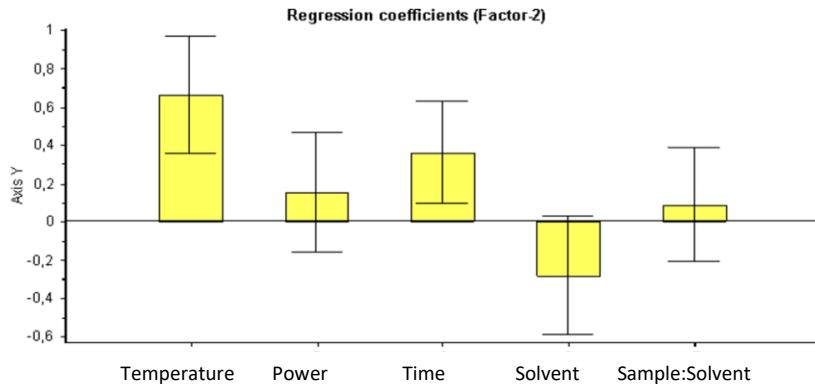
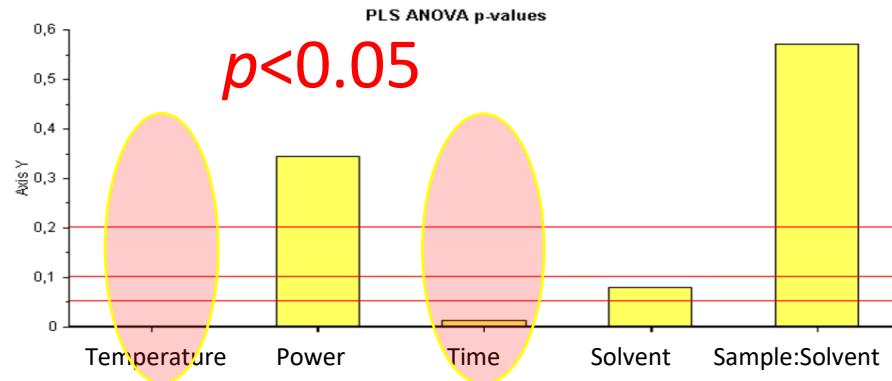
DOE: 26

+

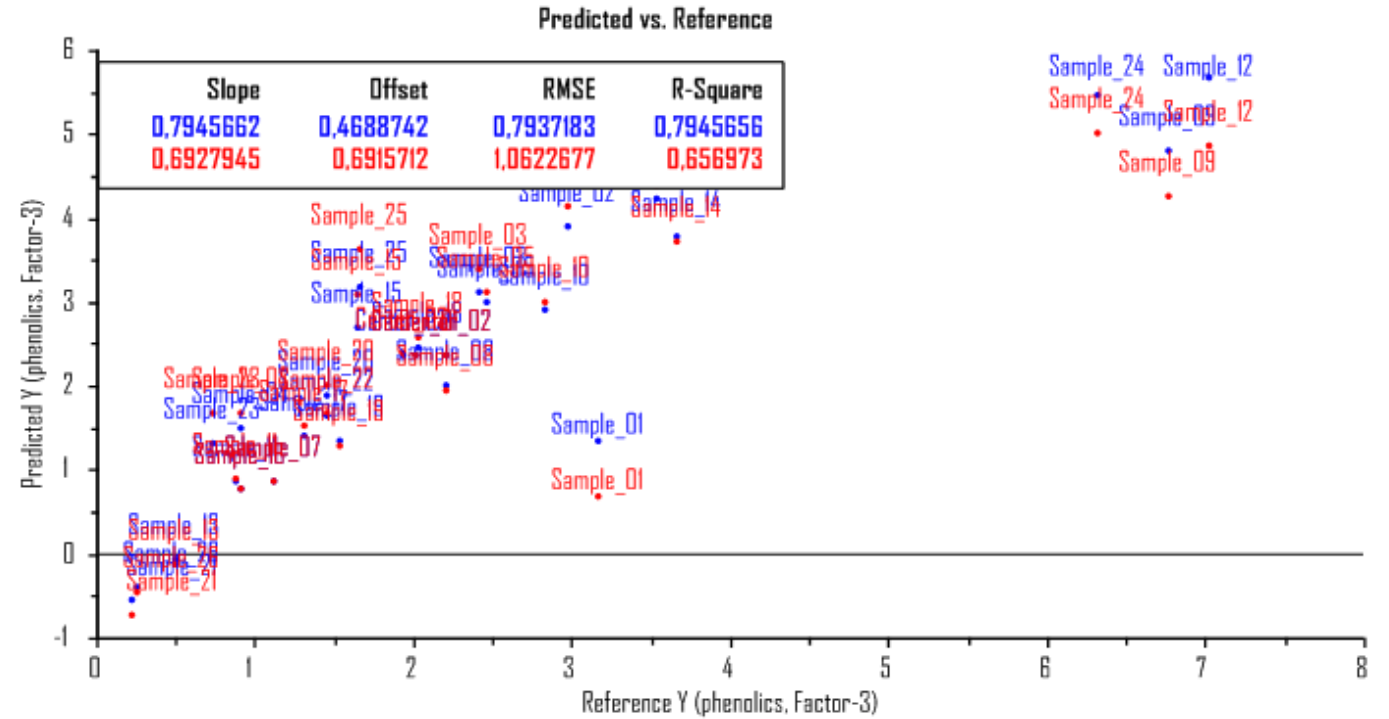
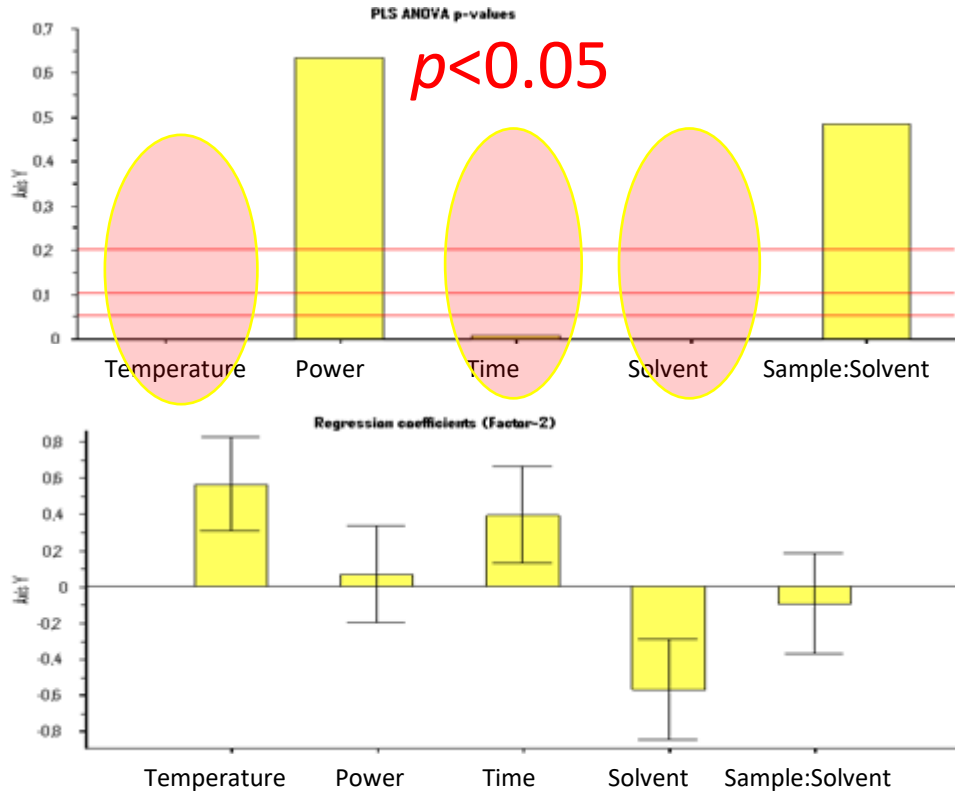
Centre points: 3

Factors: 5  
Levels: 3





# MAE Method Development for tryptophan related compound



# MAE Method Development for phenolic compounds



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Contents lists available at SciVerse ScienceDirect

## Journal of Cereal Science

journal homepage: [www.elsevier.com/locate/jcs](http://www.elsevier.com/locate/jcs)



# A new microwave-assisted extraction method for melatonin determination in rice grains

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*Department of Analytical Chemistry, Faculty of Sciences, University of Cadiz, Campus de Excelencia Internacional Agroalimentario (CeIA3), Campus del Rio San Pedro, 11510 Puerto Real, Cádiz, Spain*



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## Food Chemistry

journal homepage: [www.elsevier.com/locate/foodchem](http://www.elsevier.com/locate/foodchem)



Analytical Methods

### Pressurized liquid extraction of phenolic compounds from rice (*Oryza sativa*) grains



W. Setyaningsih<sup>a,b</sup>, I.E. Saputro<sup>b</sup>, M. Palma<sup>b,\*</sup>, C.G. Barroso<sup>b</sup>

<sup>a</sup> Department of Food and Agricultural Product Technology, Faculty of Agricultural Technology, Gadjah Mada University, Jalan Flora, Bulaksumur, 55281 Yogyakarta, Indonesia

<sup>b</sup> Department of Analytical Chemistry, Faculty of Sciences, University of Cadiz, Campus de Excelencia Internacional Agroalimentario (CeIA3), Campus del Rio San Pedro, 11510 Puerto Real, Cádiz, Spain



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## Example #2

DOE to develop chromatographic separation for **Tryptophan related compounds and phenolic compounds** in rice extracts

LOCALLY ROOTED, GLOBALLY RESPECTED



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# Food Chemistry

journal homepage: [www.elsevier.com/locate/foodchem](http://www.elsevier.com/locate/foodchem)



## Analytical Methods

### Multiresponse optimization of a UPLC method for the simultaneous determination of tryptophan and 15 tryptophan-derived compounds using a Box-Behnken design with a desirability function



Widiastuti Setyaningsih<sup>a,b</sup>, Irfan E. Saputro<sup>b</sup>, Ceferino A. Carrera<sup>b</sup>, Miguel Palma<sup>b,\*</sup>, Carmelo G. Barroso<sup>b</sup>

<sup>a</sup> Department of Food and Agricultural Product Technology, Faculty of Agricultural Technology, Gadjah Mada University, Jalan Flora, Bulaksumur, 55281 Yogyakarta, Indonesia

<sup>b</sup> Department of Analytical Chemistry, Faculty of Sciences, Instituto de Investigación Vitivinícola y Agroalimentaria (IVAGRO), University of Cadiz, Campus de Excelencia Internacional Agroalimentario (CeIA3), Campus del Rio San Pedro, 11510, Puerto Real, Cádiz, Spain

UHPLC

Separation for 16 TD analytes



# Results and Discussion

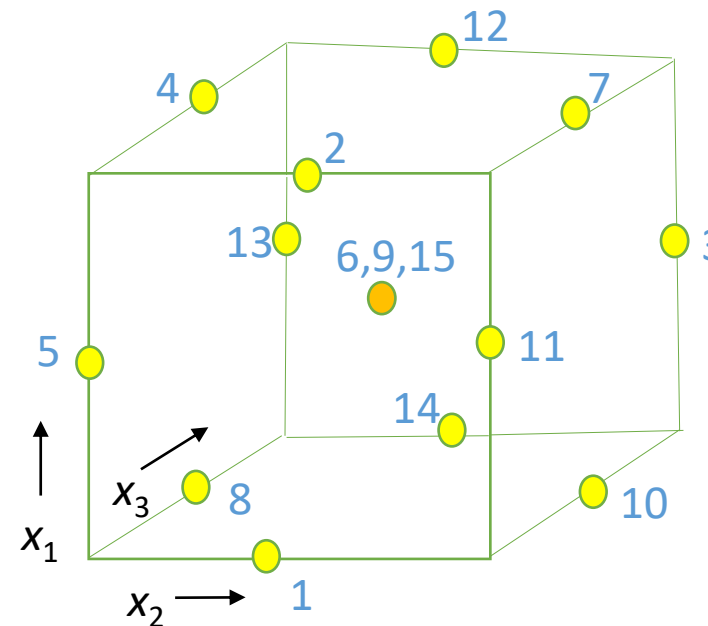


## UHPLC Factors and the levels

Variables	-1	0	+1	Units
A:%B Start	0	5	10	% Phase B
B:%B End	50	75	100	% Phase B
C:Flow Rate	0.5	0.6	0.7	mL/min PB
	0.45	0.55	0.65	mL/min SCB



## Experimental Design points



15 Runs  
Inc. 3 center points

UHPLC

Separation for 20 Phenolics

# Results and Discussion



## Box-Behnken design and the responses

## 1st Column : PB

## 6 Responses to be optimized

BLOCK	A	B	C	RS 1-2	RS 2-3	RS 3-4	RS 4-5	RS 5-6	RS 6-7	RS 7-8	RS 8-9	RS 9-10	RS 10-11	RS 11-12	RS 12-13	RS 13-14	RS 14-15	RS 15-16	Analysis Time
1	-1	0	-1	1.1699	4.3597	6.4253	2.6264	0.7728	1.1012	0.9012	1.9657	2.5455	9.5745	1.1240	2.5582	0.8714	7.7156	3.1558	3.7460
				0.5169	2.7573	4.5930	2.5679	0.0000	1.0748	1.0748	1.9203	2.8351	11.2452	1.6697	3.3865	0.9333	7.8714	3.1885	3.2865
				1.0333	4.5826	8.1021	3.2605	0.7740	1.1032	0.9377	2.8874	4.2733	13.1147	1.1823	3.9370	0.4894	9.1150	2.8586	2.5690
				0.5679	4.3827	6.4607	2.5947	0.0000	1.1762	1.0029	1.7111	2.2134	9.8268	1.2566	2.2422	0.0000	6.4990	3.1265	3.8240
				0.8282	3.1905	5.1933	2.6613	0.9930	1.0029	1.1032	2.5913	4.2404	14.5302	1.1354	4.1600	0.5010	9.6017	2.7275	4.4900
1	1	0	-1	0.9517	5.0864	7.8807	3.2791	0.8432	1.1241	1.1241	2.7016	3.6469	12.1964	1.2674	3.5650	0.5419	8.2046	2.9889	3.2440
1	0	1	1	0.5878	3.2527	5.4270	2.5117	0.0000	1.1821	0.9821	1.9636	2.7891	10.4563	1.3082	2.8434	0.7762	6.9441	3.0796	2.5610
1	1	-1	0	1.1369	5.0928	8.9355	3.0373	1.4291	1.2433	1.2433	3.3331	4.4871	14.1325	0.9600	4.2580	0.0000	9.5501	2.6735	4.4065
1	0	1	-1	1.0339	5.4632	8.1142	3.2453	0.7728	1.0455	1.0455	2.7203	3.6309	12.3645	1.2511	3.6211	0.6814	9.8899	2.9366	3.2435
...	...	...	...	1.0502	4.2489	6.2239	2.6333	0.6409	1.0656	0.8656	1.9741	2.3041	9.9711	1.1431	2.4165	0.9539	8.6644	2.9891	2.9470
...	...	...	...	0.7897	5.2804	7.9339	3.2169	0.4369	1.1856	0.9762	2.7400	3.5867	12.0393	1.3691	3.4690	0.9760	8.2000	2.9713	3.0265
1	1	0	1	0.6953	5.3228	7.9566	3.1510	0.0000	1.1377	1.0751	2.5754	3.1759	11.1242	1.2751	3.1761	0.3310	7.3678	2.8589	2.7725
1	0	-1	1	1.0216	3.6075	5.2721	2.6667	1.2108	1.0751	1.5653	2.4676	3.6333	12.6798	0.7356	3.9195	0.0000	9.0237	2.7196	3.8940
1	-1	0	1	1.1419	5.0771	8.0693	3.3369	1.1114	1.5653	1.0958	4.2112	5.2261	14.6452	1.0089	4.1921	0.0000	9.6888	2.2071	3.2520
1	0	0	0	1.0298	4.8533	7.4903	2.8073	0.8562	1.0958	1.1856	3.0295	3.5864	12.1699	1.2407	3.4952	0.5570	7.7409	2.6842	3.2470

UHPLC

Separation for 16 TD analytes

# Results and Discussion



## Box-Behnken design and the responses

## 2nd Column : SCB

## 6 Responses to be optimized

BLOCK	A	B	C	RS 1-2	RS 2-3	RS 3-4	RS 4-5	RS 5-6	RS 6-7	RS 7-8	RS 8-9	RS 9-10	RS 10-11	RS 11-12	RS 12-13	RS 13-14	RS 14-15	RS 15-16	Analysis Time
1	-1	0	-1	0.587	3.8290	8.5435	4.1487	0.242	2.0816	0.294	2.4112	2.6824	11.5097	1.618	4.4066	0.544	9.1636	4.1243	3.735
				0.000	3.3366	5.8537	4.0653	0.325	1.6661	0.504	2.4000	3.3697	13.0768	1.855	5.0441	0.581	9.4466	3.5812	3.268
				0.000	4.3920	9.3611	5.4556	0.256	2.3805	0.000	3.1161	4.2157	14.6212	1.455	6.2486	0.000	12.5085	3.6214	2.52
				0.000	4.2693	7.9200	3.6548	0.037	1.9844	0.216	2.2696	2.5562	11.3811	1.325	3.8460	0.457	7.2119	3.4849	3.719
				0.000	3.3882	5.6849	4.1362	0.514	1.8000	0.000	2.6504	4.3723	15.6302	1.378	6.9015	0.000	12.9294	3.0204	4.433
1	1	0	-1	0.000	4.2316	8.8851	5.0335	0.363	2.0636	0.000	2.8941	3.7067	13.7143	1.516	5.6335	0.000	10.1720	3.3448	3.188
1	0	1	1	0.000	3.3739	5.8105	4.0357	0.310	1.6408	0.510	2.4282	3.1579	12.7117	1.674	4.6067	0.468	8.7223	3.7770	2.528
1	1	-1	0	0.753	4.7165	10.8554	4.5791	1.330	2.4971	0.133	2.4907	4.3837	14.7556	0.824	5.8766	0.523	11.6509	3.2000	4.333
1	0	1	-1	0.000	4.2947	9.2287	4.9826	0.270	2.1183	0.000	2.8657	3.6331	13.3078	1.443	5.3153	0.000	10.6378	3.6527	3.191
...	...	...	...	0.628	2.1677	7.4816	3.3778	0.000	1.4247	0.480	1.6000	2.2306	10.7608	1.622	2.9681	0.512	5.2031	3.4794	2.92
...	...	...	...	0.000	4.1358	8.6154	5.0637	0.000	2.1486	0.545	2.6916	3.4429	13.7481	1.767	5.4069	0.745	10.3037	3.3280	3.026
1	1	0	1	0.000	4.1532	8.2364	4.6857	0.211	2.0764	0.296	2.8571	3.3060	12.6131	1.453	5.1064	0.000	9.1259	2.9520	2.701
1	0	-1	1	0.000	3.6757	6.2849	4.4384	1.036	1.9660	0.300	2.7236	3.9000	14.7866	0.626	5.3663	1.200	10.8511	3.3576	3.788
1	-1	0	1	0.673	3.8980	8.4843	5.8667	0.873	2.5888	0.000	2.4322	5.1594	17.0331	1.128	6.4859	0.000	12.5198	2.5961	3.188
1	0	0	0	0.000	3.8493	9.5698	4.8312	0.307	1.9253	0.000	3.0202	3.3988	12.8627	1.470	5.1070	0.000	9.9754	3.4361	3.198

UHPLC

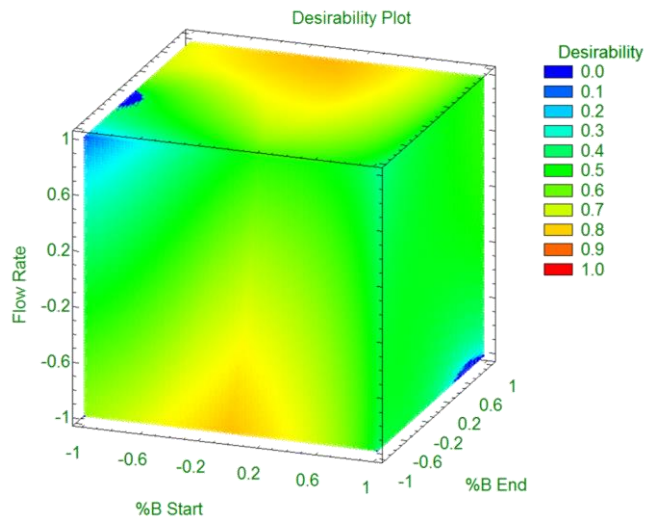
Separation for 16 TD analytes

# Results and Discussion



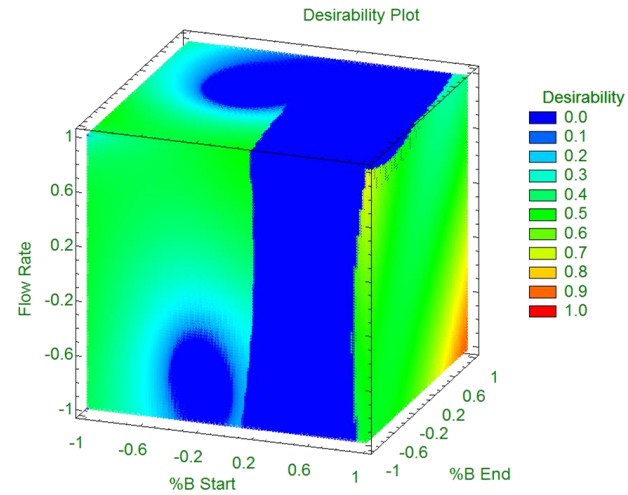
## Multi-response Optimization

PB Column



Factor	Coordinate	Optimum
%B Start	-0.37359	3.13 % Phase B
%B End	0.28690	82.17% Phase B
Flow Rate	-0.99999	0.5 mL min <sup>-1</sup>

SCB Column



Factor	Coordinate	Optimum
%B Start	0.999994	10 % Phase B
%B End	0.999999	100% Phase B
Flow Rate	-0.99999	0.45 mL min <sup>-1</sup>

UHPLC

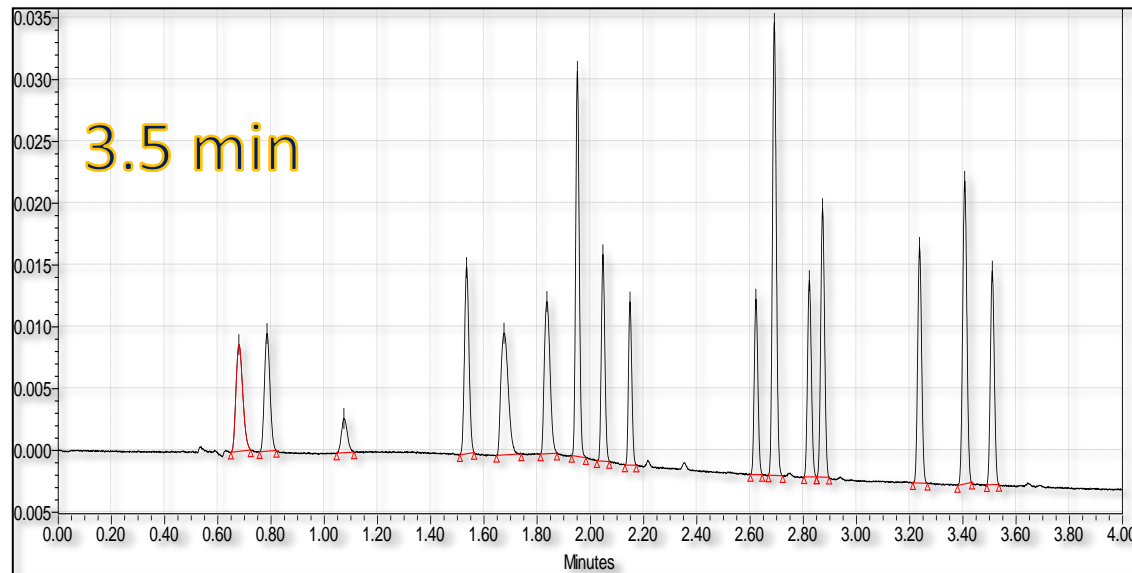
Separation for 16 TD analytes

# Results and Discussion

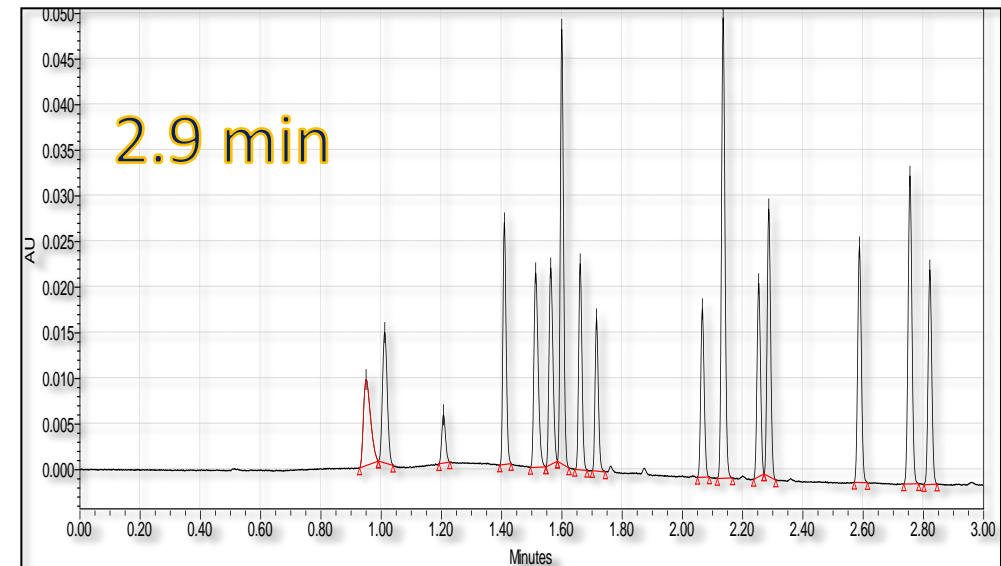


## Multi-response Optimization

PB Column



SCB Column



UHPLC

Separation for 16 TD analytes

# Results and Discussion



## Method Validation

### PB Column

#### Working Range (mg L<sup>-1</sup>)

- 1-100, R<sup>2</sup> : > 0.999

#### Limits

- LOD, 1.23 - 3.47 µg L<sup>-1</sup>
- LOQ, 3.73 µg L<sup>-1</sup> - 10.51 µg L<sup>-1</sup>

#### Precision (RT)

- Repetability, 0.02% - 0.14%
- Intermediate Precision, 0.04% - 0.18%

#### Precision (Area)

- Repetability, 0.30% - 3.29 %
- Intermediate Precision, 0.25% - 0.46%

### SCB Column

#### Working Range (mg L<sup>-1</sup>)

- 1-100, R<sup>2</sup> : > 0.999

#### Limits

- LOD, 2.21 - 5.05 µg L<sup>-1</sup>
- LOQ, 6.67 - 15.30 µg L<sup>-1</sup>

#### Precision (RT)

- Repetability, 0.02% - 0.87%
- Intermediate Precision, 0.03% - 0.46%

#### Precision (Area)


- Repetability, 0.44% - 3.51 %
- Intermediate Precision, 0.23% - 5.62%

UHPLC

Separation for 16 TD analytes



# Fast Determination of Phenolic Compounds in Rice Grains by Ultrapformance Liquid Chromatography Coupled to Photodiode Array Detection: Method Development and Validation

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UHPLC

Separation for 20 Phenolics



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# Thank You



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